JANA98

THE CRYSTALLOGRAPHIC COMPUTING SYSTEM

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Contents

About This Manual

The first part of this manual describes the installation of the program and shows how to use *JANA98* and its basic common tools. In the next part programs used for data processing and setting the basic crystal information are explained. The third part about structure solution and refinement can be understood a heart of the manual. It describes in details structure parameters, programs for their changing and refinement and Fourier program for calculation of electron density maps. An underlying theory is given for each structure parameter. The last part is dedicated to the structure interpretation based on distances and angles, electron density maps and modulation parameters.

This version of the manual is the definitive description of *JANA98*. The successor of *JANA98* is the program *JANA2000* and we recommend using it instead of *JANA98*. However, most information given here is still valid for *JANA2000*. New manual for *JANA2000* is expected during year 2001.

The manual is available in the ftp server ftp.fzu.cz in the PDF format. The authors welcome any remark concerning both this manual and the functionality of *JANA98/2000*.

1

Basic Features

This part describes the installation of JANA98 and its basic concepts and gives a cursory overview of the system.

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1

Basic Features

JANA98 is a system for solving and refinement of regular, modulated and composite structures from single crystal diffraction data. It is compatible with previous version *JANA96* but its functionality is extended.

Basic features:
JANA98 covers the basic tasks of structure analysis from data reduction to the solution of the phase problem, refinement of the structure model and presentation of results. Two parts of the system - direct methods and plotting program - are not yet available. The three dimensional and higher dimensional crystals are treated uniquely in one system. In addition to regular structure parameters (occupancy, atomic position and anisotropic temperature parameters) the system allows description of atomic displacements by ADP (anharmonic displacement parameters).

Advanced features:
Both regular and modulation parameters can be refined within the rigid body approximation. Molecules can have a local symmetry different of the site symmetry. Special modulation functions (c*renel functions* for occupancy and *sawtooth functions* for position) can be used for refinement of non-harmonic modulations. They can also be used together with harmonic functions using the orthogonalized modulation waves. *JANA98* can plot higher dimensional electron density maps and joined probability density functions. Commensurate structures can be solved in the superspace. *JANA98* supports work with complicated twins and partially overlapped structures.

Platforms:
JANA98 runs with SunOS (Solaris), Irix, HPUX, AIX, LINUX, DEC UNIX and Windows9x MSDOS emulation. For UNIX platforms it is distributed as a source code and needs FORTRAN and C compiler. The package is freely available from anonymous ftp server
ftp://ftp.fzu.cz/pub/cryst.

The authors welcome any remark, suggestion or question about this program.

1.1 Installation and user support

JANA98 is portable to all basic UNIX platforms: SunOS, Irix, HPUX, AIX, LINUX and DEC UNIX. For UNIX platforms it is distributed as a source code and requires FORTRAN77 or FORTRAN90, C compiler and X11 library. The PC version is a compiled program for MSDOS or Windows9x MSDOS emulation. It does not work[1] with WindowsNT MSDOS emulation. VMS, OS2 and Macintosh are not supported. The 32 bit version for Windows95/98 is under development.

JANA98 is freely available from anonymous ftp://ftp.fzu.cz/pub/cryst. The jana98 directory contains the following files:

`README.TXT`	this downloading and installation notes
`jana98.tar.Z`	the UNIX version compressed by compress
`jana98.tar.gz`	the UNIX version compressed by gzip
`janainst.exe`	the self extracting installation file for DOS version
`jana98Pack.exe`	the self extracting installation file for UNIX version
`manual`	the directory containing this manual

Any file can also be copied through the *JANA98* homepage http://www-xray.fzu.cz/jana/jana.html. The following commands are for people using command line ftp:

An example of ftp commands:

ftp ftp.fzu.cz	Connects to ftp.fzu.cz
cd pub/cryst/jana98	Changes remote directory to jana98
binary	Sets binary transfer
get jana98.tar.Z	Copies the compressed archive (for UNIX workstation in this example) to the current directory
quit	Exits ftp

---

[1] *JANA98* under WindowsNT has an unpredictable graphical behaviour. The calculation (for instance refinement) usually runs well. Some users of WindowsNT reported they have been using *JANA98* without any difficulty.

1.1.2 Installation of *JANA98* on UNIX workstations

Automatic installation from jana98Pack.exe

The automatic installation can be executed by the command

```
source jana98Pack.exe
```

from csh or tcsh shell. If you are running other shell (sh,bash, ksh etc.) you should use the following commands:

```
csh                       starts csh
source jana98Pack.exe  starts automatic installation
exit                      exits csh
```

The installation procedure creates the directory jana98 and extracts all files to this directory. This is a difference from installation of the DOS version where the user must create a directory and execute here the installation file.

Note for multiple installations:

The file jana98Pack.exe is changed during installation so that it contains the full path to the csh interpreter. It cannot be easily edited as a part of the file is binary. Please make a backup copy of jana98Pack.exe before making multiple installations of *JANA98* on computers with different path to csh.

Manual installation from jana98.tar.gz or jana98.tar.Z

Here are ***step by step instructions*** for manual installation. It is necessary when the compilation requires special settings in the Makefile that are not included in the automatic installation file.

gunzip jana98.tar	Replaces compressed file jana98.tar.gz with the uncompressed jana98.tar. Jana98.tar.Z should be uncompressed by "uncompress"
tar xf jana98.tar	Creates directory jana98. Extracts all files from the archive jana98.tar to the directory jana98. The files of the same name will be overwritten without any warning. The tar creates the following directory structure: Jana98 -> source -> fg -> test
	The directory jana98 contains Makefile – the description file for the compilation tool *make*. The directory source and fg contains source files. The testing examples are in the directory test.
cd jana98	Changes directory to jana98.
edit Makefile	Makefile must be edited before the compilation. Find editable section corresponding to your computer and remove "#" from the first column.
make	Executes commands from the Makefile. Builds the libraries and compiles all programs of the package.
make clean_up	Removes object files, libraries and temporary files. This command is optional.
cd test	Changes directory to testing examples
../jana98 testa	Runs the testing example 1

The environmental variable JANADIR

The installed program can be used without setting of JANADIR[1] assuming that this variable is undefined and that the compiled program stays in the directory where it has been created. by make. JANADIR is necessary when the compiled program is moved after the compilation.

Copying installed JANA98 to a different directory

Compiled *JANA98* cannot be simply moved to other destination as it needs access to several files from the installation directory. This is the way how to move the compiled program:

1. Copy the whole directory jana98 to a new destination
2. Add the following line to your .cshrc file:

```
setenv JANADIR new_destination_of_compiled_jana98_file
```

Troubleshooting

- The installation was not successful

If you used the automatic installation try the manual one instead. If it also fails here is a list of several possible reasons:

1. The cc, f77 or f90 compiler is not installed on your workstation or is not accessible. This can be confirmed by the command like:
   `which f77` or `whereis f77`

2. You are using f90 compiler. Try f77 instead (i.e. disable the section for f90 in your Makefile and enable that one for f77)

3. Your compiler cannot find X11 include files or X11 library. The information about the path to X11 include files is in the Makefile after -I option (look for lines starting "INCLUDE = -I …". The information about the path to X11 library is written in Makefile in the LINKLIBS macro (look for lines starting "LINKLIBS = …".

4. If it does not help please send a message to dusek@fzu.cz.

- The installation was successful but the program did not work correctly

Common source of difficulties **after** installation is wrong definition of JANADIR variable. It often contains a path to a different version of Jana (JANA96 etc.).

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[1] This is difference of the DOS version where setting of JANADIR is mandatory.

The initialisation file jana98.ini

The initialisation file jana98.ini contains user settings and can be created interactively by Tools->Preferences. This is the hierarchy how *JANA98* searches for jana98.ini:

1. The directory written to JANAPATH (if this variable exists)
2. The user home directory
3. The starting directory[1]

The last initialisation file has the highest priority. This means that the same option included in all processed initialisation files is taken from the last one. This feature is useful if a multi-user environment. The initialisation file in JANAPATH can contain options set by the administrator and each user can redefine these options in own configuration files.

The initialisation file for UNIX version of *JANA98* can be configured separately for various display addresses. For instance there can be setting in the initialisation file making the program running in the full screen mode on a small display and in the reduced window mode on a large display. This may cause difficulties when using the secure shell - see page 22.

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[1] In *JANA98* the directory can be changed by File manager. Thus the starting directory is not necessarily the current directory.

1.1.3 Installation of *JANA98* on a PC with Windows9x/NT or DOS

The PC version is distributed as self-extracting installation file janainst.exe containing the compiled program jana98.exe and all other necessary files. It does not contain the source code. The program is compiled by LF90 (LAHEY FORTRAN 90) version 4.00. The graphic interface is based on INTERACTER 4.10 graphic library. The JANA98 source code for LF90 can be obtained on the request to petricek@fzu.cz.
The installation procedure creates also the initialisation file jana98.ini with basic setting of display, colours etc. The file can be interactively changed by user with Tools->Preferences. In rare cases the program does not work properly with mouse and needs another configuration. In such cases please contact petricek@fzu.cz.

Step by step instructions

1.	Create new directory for *JANA98.* ("janadir"). The recommended name is c:\jana98.
2.	Copy the installation file janainst.exe into "janadir"
3.	Run janainst.exe.
4.	Add the following line into your autoexec.bat : SET JANADIR="janadir"
5.	Add "janadir" into the path command in your autoexec.bat.
6.	Restart the computer
7.	Change directory to "janadir"\test
8.	Run the testing examples: jana98 testa jana98 testb jana98 testc jana98 testm **Important note for users of Windows95/98**: The program is NOT Windows application. It runs in protected memory mode of MSDOS in full screen mode. The launch of the program on older PC's can take between 1-2 minutes. See the following paragraph for more information.

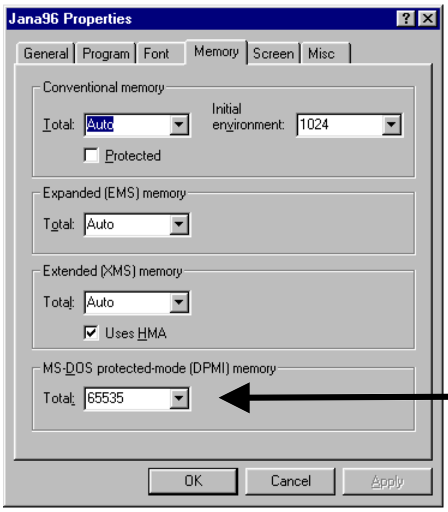
According to our experience 16 MB of operating memory is minimal, 32 MB sufficient and 64 MB optimal for running *JANA98.*

Program properties for Windows95/98

In many cases *JANA98* runs well immediately after installation and restarting the computer. Sometimes it is necessary to set properties for *JANA98* DOS environment. The window with "Jana98 properties" can be opened from a menu appearing after clicking the right mouse button on the file jana98.exe in the windows explorer or in the "My computer" window.

The following pictures show two tabs important for running of *JANA98*.

Figure 1 *The protected memory setting for Windows95*



Conventional memory

Total: Auto

Initial environment: 1024

☐ Protected

Expanded (EMS) memory

Total: Auto

Extended (XMS) memory

Total: Auto

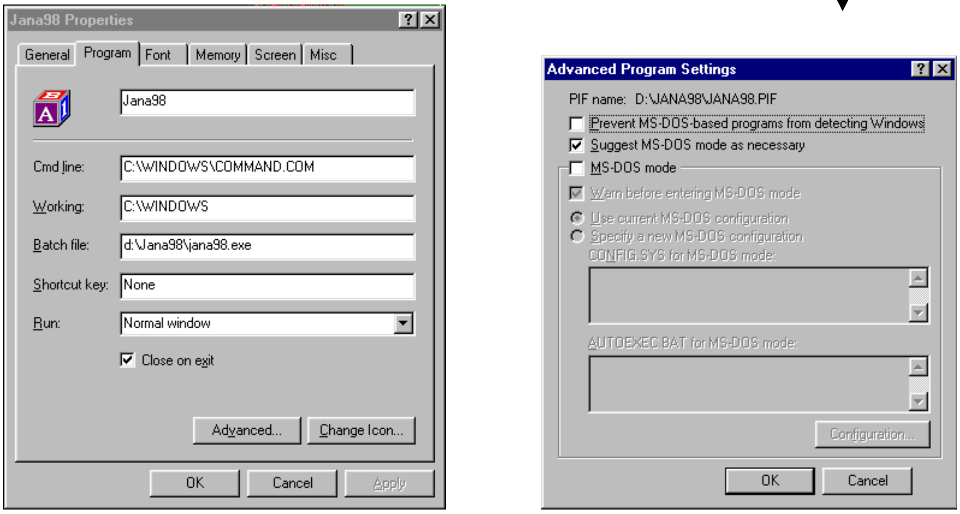
☑ Uses HMA

MS-DOS protected-mode (DPMI) memory

Total: 65535

The settings recommended for *JANA98*.

Figure 2 *The Advanced Program Settings for Windows95*



Jana98

Cmd line: C:\WINDOWS\COMMAND.COM

Working: C:\WINDOWS

Batch file: d:\Jana98\jana98.exe

Shortcut key: None

Run: Normal window

☑ Close on exit

PIF name: D:\JANA98\JANA98.PIF

☐ Prevent MS-DOS-based programs from detecting Windows

☑ Suggest MS-DOS mode as necessary

☐ MS-DOS mode

☑ Warn before entering MS-DOS mode

● Use current MS-DOS configuration

○ Specify a new MS-DOS configuration

CONFIG.SYS for MS-DOS mode:

AUTOEXEC.BAT for MS-DOS mode:

1.1.4 Adjustable parameters

The number of atoms, molecules etc. in *JANA98* is limited. Users of UNIX version can control some of these limits by the corresponding `parameter` statement in the source code. After changing the limits the program must be recompiled. For DOS version an adapted compiled program can be supplied by request.

The following table lists the adjustable parameters of *JANA98*. All parameters are defined in the file params.cmn.

Table 1 *The adjustable parameters of JANA98*

name	initial value	purpose
MXA	200	maximal number of atoms
MXM	5	maximal number of molecules
MXP	15	maximal number of the positions of single molecule
MXW	16	maximal number of modulation waves
MXREF	150000	maximal number of the reflections for the refinement
MXE	500	maximal number of equations in the m50
MXEP	10	maximal number of the parameters in single equation
MXPARRF	1000	maximal number of the refined parameters

1.1.5 Location of files

The directory structure

The following figures show the directory structure of *JANA98* for PC and UNIX version. **The tree is relative to *JANADIR*** (see page 11).
The filenames shown in the figures are necessary for running of *JANA98*.

Figure 3 *The Directory Structure of PC Installation*

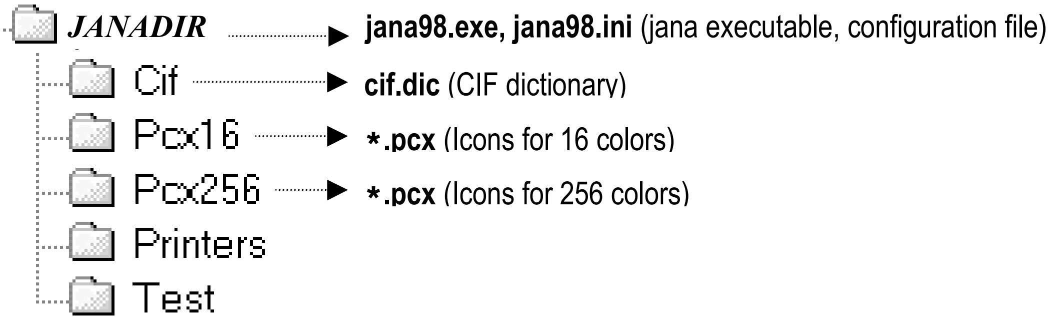
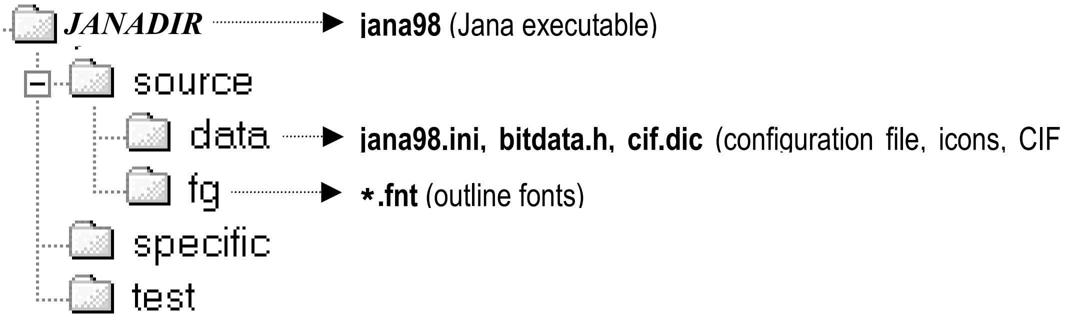


Figure 4 *The Directory Structure of UNIX Installation*



Temporary files

JANA98 uses two types of temporary files.

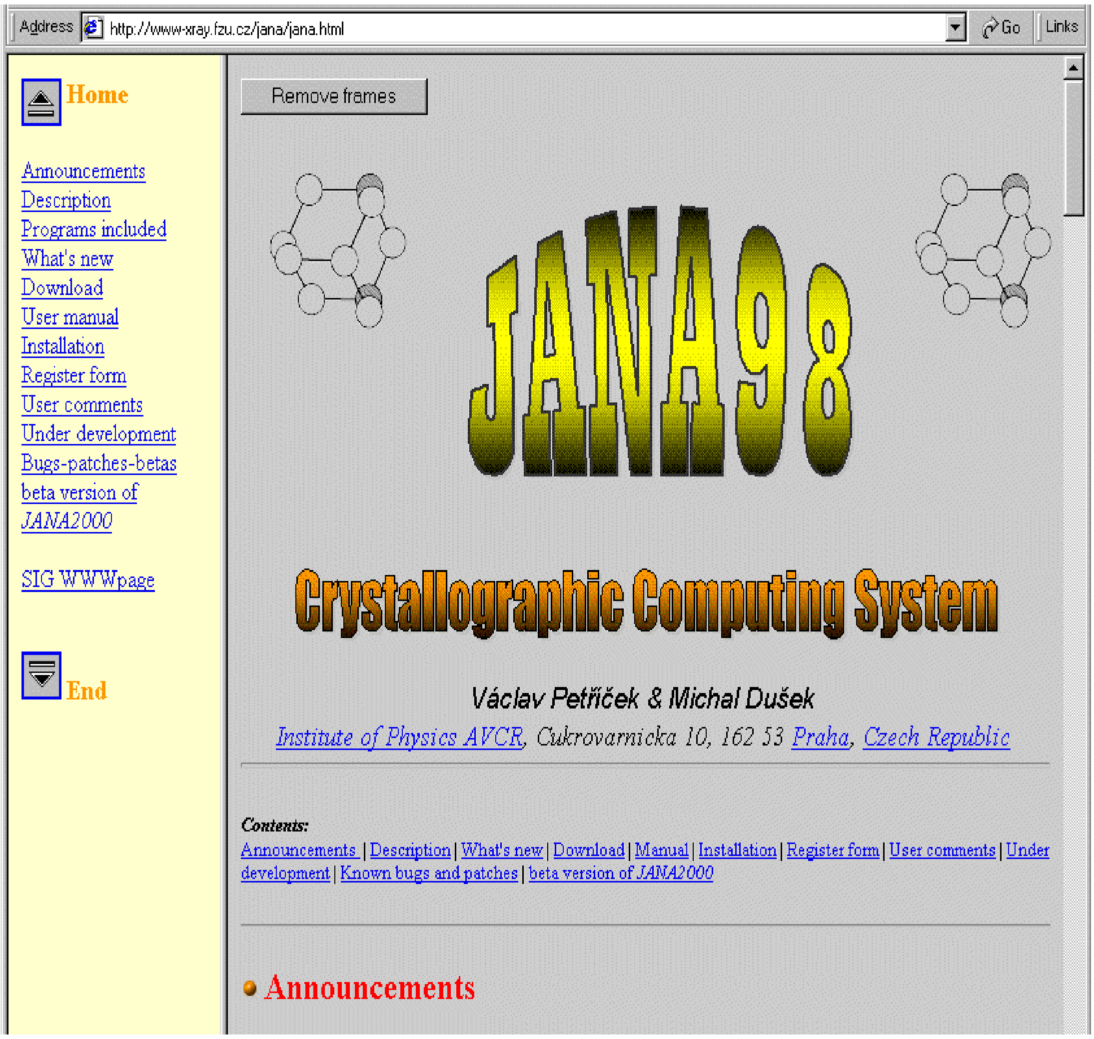
- The first type has names like `name.l??` and `*.pcx`, where `name` is name of the structure, * and ? are wildcards. They are saved in the directory containing the used structure. Their names are not unique so that two sessions of *JANA98* cannot run in the same directory.
- The second type has names like `cmd*.tmp`. In the case of DOS version they are saved in the tmp directory, which is read from jana98.ini. The default tmp directory is set to C:\ and it can be modified by tools→properties. The UNIX version searches for directories /scratch, /var/tmp ,/tmp and $(HOME) (in this order) and saves the temporary files to the first accessible one.

1.1.6 User Support

WWW Page of JANA98

The home page of *JANA98* is accessible in the address
http://www-xray.fzu.cz/jana/jana.html.
The page contains the latest information about the changes of *JANA98*, list of known bugs and available patches, link for download and a registration form.

Figure 5 *The JANA98 WWW Home Page*



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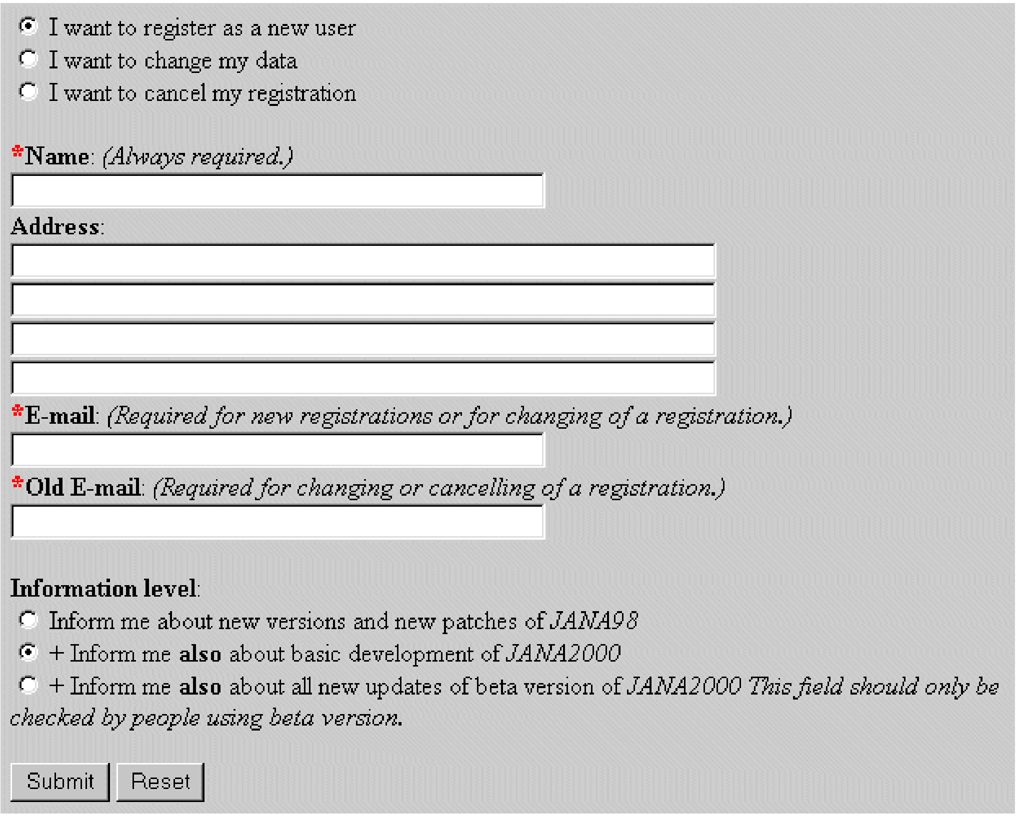
Registered Users

The WWW home page contains also the form for registering of users. Although *JANA98* is a free software, which is fully functional without registering, the registered users take advantage of detailed e-mail messages about upgrades. For the authors this is valuable feedback information.

Figure 6 *The registration form for JANA98*



● I want to register as a new user
○ I want to change my data
○ I want to cancel my registration

***Name**: *(Always required.)*

Address:

***E-mail**: *(Required for new registrations or for changing of a registration.)*

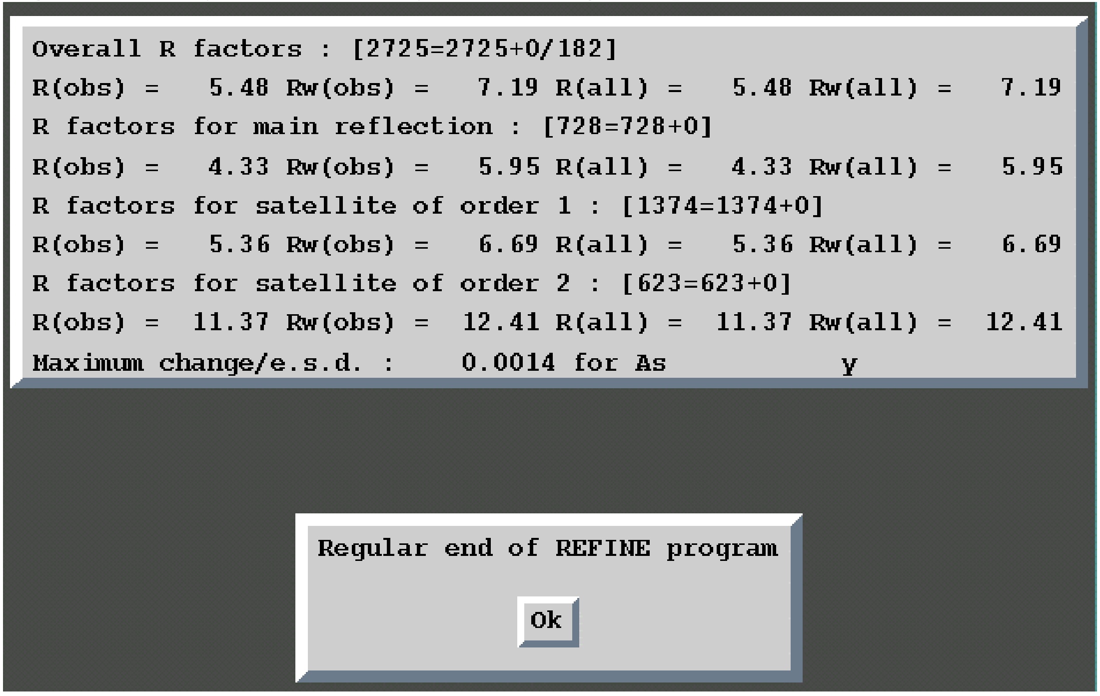
***Old E-mail**: *(Required for changing or cancelling of a registration.)*

Information level:
○ Inform me about new versions and new patches of *JANA98*
● + Inform me **also** about basic development of *JANA2000*
○ + Inform me **also** about all new updates of beta version of *JANA2000* *This field should only be checked by people using beta version.*

1.1.7 Results of testing refinements

After the system has been successfully installed the user should run refinements of the testing examples to see if it is working properly. The resulting R values should be close to the ones listed here.

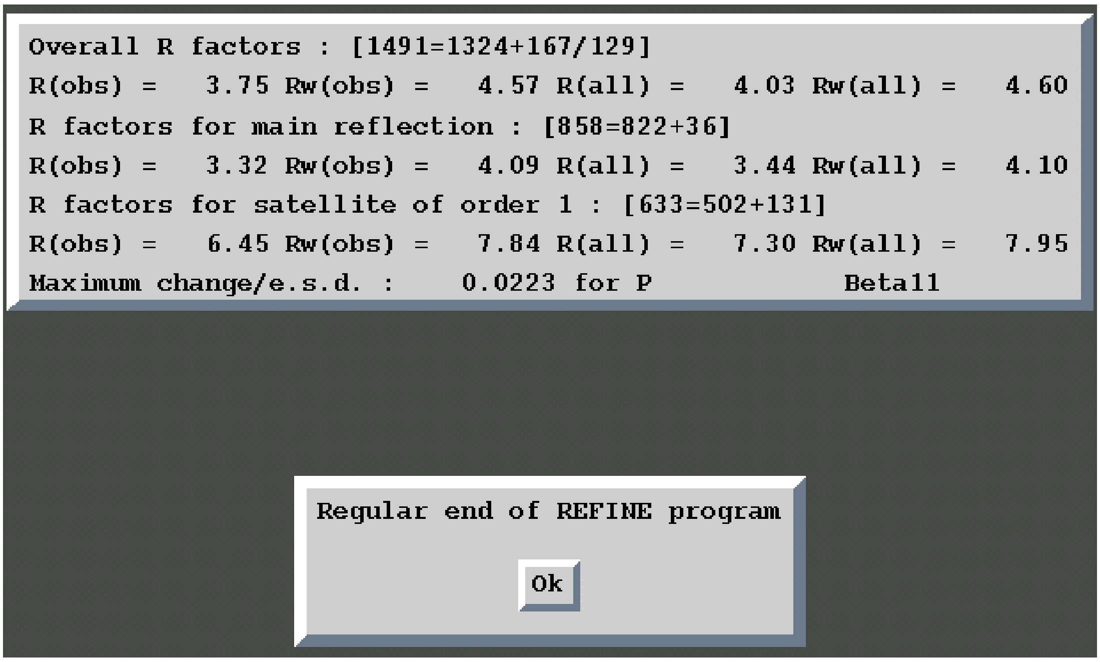
Figure 7 *Refinement results for the testing example* `testa`



```
Overall R factors : [2725=2725+0/182]
R(obs) =    5.48 Rw(obs) =    7.19 R(all) =    5.48 Rw(all) =    7.19
R factors for main reflection : [728=728+0]
R(obs) =    4.33 Rw(obs) =    5.95 R(all) =    4.33 Rw(all) =    5.95
R factors for satellite of order 1 : [1374=1374+0]
R(obs) =    5.36 Rw(obs) =    6.69 R(all) =    5.36 Rw(all) =    6.69
R factors for satellite of order 2 : [623=623+0]
R(obs) =   11.37 Rw(obs) =   12.41 R(all) =   11.37 Rw(all) =   12.41
Maximum change/e.s.d. :     0.0014 for As             y
```

Regular end of REFINE program

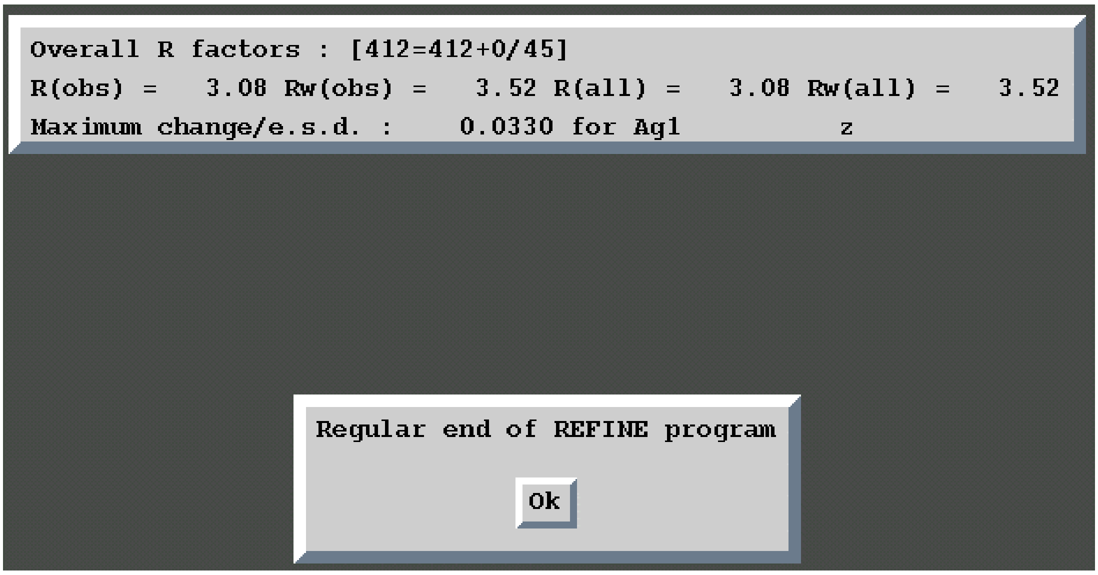
Figure 8 *Refinement results for the testing example* `testb`



```
Overall R factors : [1491=1324+167/129]
R(obs) =    3.75 Rw(obs) =    4.57 R(all) =    4.03 Rw(all) =    4.60
R factors for main reflection : [858=822+36]
R(obs) =    3.32 Rw(obs) =    4.09 R(all) =    3.44 Rw(all) =    4.10
R factors for satellite of order 1 : [633=502+131]
R(obs) =    6.45 Rw(obs) =    7.84 R(all) =    7.30 Rw(all) =    7.95
Maximum change/e.s.d. :     0.0223 for P              Beta11
```

Regular end of REFINE program

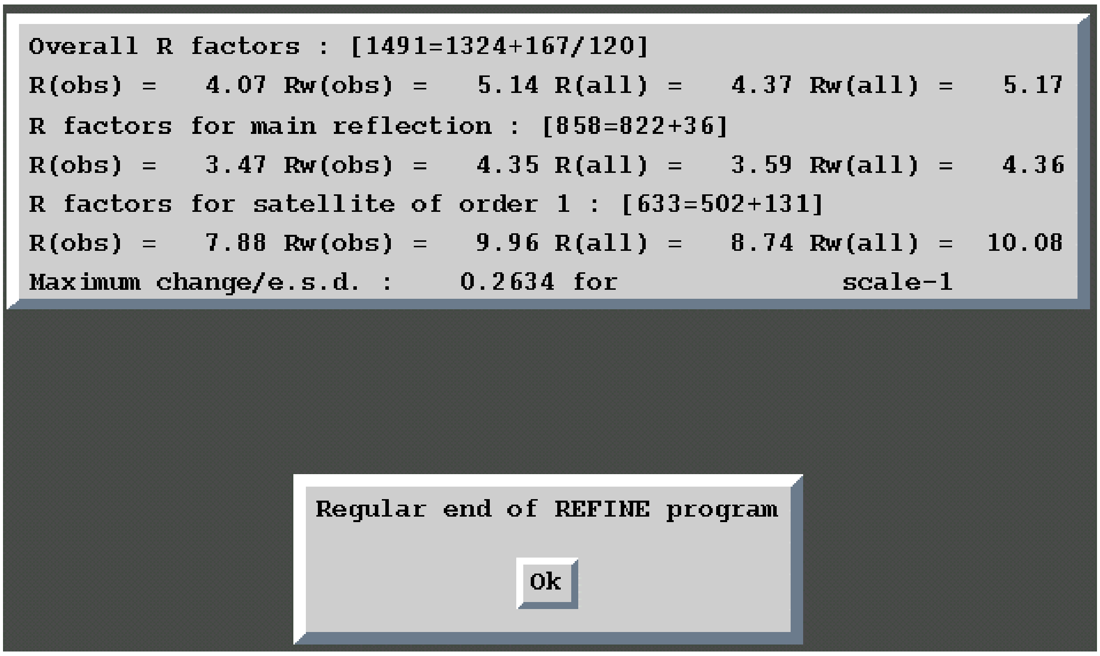
Figure 9 *Refinement results for the testing example* `testc`



```
Overall R factors : [412=412+0/45]
R(obs) =   3.08 Rw(obs) =   3.52 R(all) =   3.08 Rw(all) =   3.52
Maximum change/e.s.d. :    0.0330 for Ag1           z
```

```
Regular end of REFINE program
```

Figure 10 *Refinement results for the testing example* `testm`



```
Overall R factors : [1491=1324+167/120]
R(obs) =   4.07 Rw(obs) =   5.14 R(all) =   4.37 Rw(all) =   5.17
R factors for main reflection : [858=822+36]
R(obs) =   3.47 Rw(obs) =   4.35 R(all) =   3.59 Rw(all) =   4.36
R factors for satellite of order 1 : [633=502+131]
R(obs) =   7.88 Rw(obs) =   9.96 R(all) =   8.74 Rw(all) =  10.08
Maximum change/e.s.d. :    0.2634 for              scale-1
```

```
Regular end of REFINE program
```

1.2 Executing *JANA98*

The PC version of *JANA98* runs as a MSDOS application in the full screen mode. It is started from the DOS command line or by clicking the *JANA98* executable or an appropriate shortcut.
The UNIX version is started from the command line.
Simultaneous run of several *JANA98* sessions is possible, but ***it is strongly recommended to start each session in a special directory***. Running two sessions of *JANA98* in one directory could cause a conflict when using the temporary files.

1.2.1 The command line syntax

The command line for the PC version

The syntax for starting *JANA98* from the MSDOS command line is

```
jana98 [name]
```

The command line for the UNIX version

The syntax for starting *JANA98* from the UNIX command line is

```
jana98 [name] [options] [&]
```

The parameter `name` is a reference title of the structure, which is used in all the filenames of the job. The “&” at the end of the command is used to start the program on the background.

The options start by a dash followed by a keyword and a value (if required).

Table 2 *The command line options available in UNIX version of JANA98*

option	description
`-iconic`	*JANA98* starts as an icon instead of normal window
`-scale` *`number`*	the height of *JANA98* window will be *`number`* percents of the height of the display

1.2.2 The window size in UNIX version

If the option `-scale` is not used, the program chooses the window size based on the display size. With a small display the window is set to full screen, with the diagonal greater then 15 inches the window uses 65% of the display height. Some installations of the X11 library return bad information about the display size. The returned size is usually less than the real one. This problem can be solved by the `-scale` option. Another possibility is Preferences (see page 47).

The window of *JANA98* can be also resized by the mouse but only if the program stays in the basic window. Otherwise it is automatically backed to the original size.

1.2.3 Using UNIX version of *JANA98* with ssh

In some laboratories connection to an X-server is preferably done through the secure shell (ssh). The X11 graphics coming from the X-server to a user's display is encrypted and this may considerably slow down all graphic operations (like selection from a program menu, moving of a process indicator etc.)[1].

Whether the graphics is encrypted can be recognised from the DISPLAY environmental variable. For a non encrypted connection DISPLAY takes a simple form like bigstar.ulima.com:0. With encrypted connection the display address is changed by ssh to a form like bigstar.ulima.com:11.0 (the number is not necessarily 11). If we set DISPLAY back to the original address (i.e. bigstar.ulima.com:0) the X11 graphics stops to be encrypted. An elegant way is to create an alias like[2]

```
alias jana98 (setenv DISPLAY jana98 bigstar.ulima.com:0; jana98 \!*)&
```

which starts *JANA98* without encryption but leaves the parent window in the encrypted mode.

---

[1] We found this problem with PC connected to an X-server through teraterm with ssh and using eXceed or Xwin32 like an X11 emulator.

[2] This example is written in csh shell.

1.2.4 File name conventions

Each filename used by *JANA98* consists of the name of the structure and two or three character extension**. In this manual the files are referenced only by their extensions.**

Table 3 *Basic file extensions in JANA98*

group	extension
input/output files	m+number (`m40`,`m50` etc.)
listing	first three characters of the program name (`ref`,`fou` etc.)
configuration	`ini`
CIF	`cif, smr`
scratch files	l+number (`l81`,`l82` etc.)
	`tmp`
	`pcx`
graphic output	`ps,hgl,pcx`

Example:		
Name of the structure:	testa	
Refinement reflection file:	testa.m91	(`m91` in this manual)
Basic crystal information file:	testa.m50	(`m50` in this manual)
Refinement parameter file:	testa.m40	(`m40` in this manual)
Refinement listing:	testa.ref	

1.2.5 Elements of *JANA98* graphics interface

JANA98 provides the following basic communication tools:

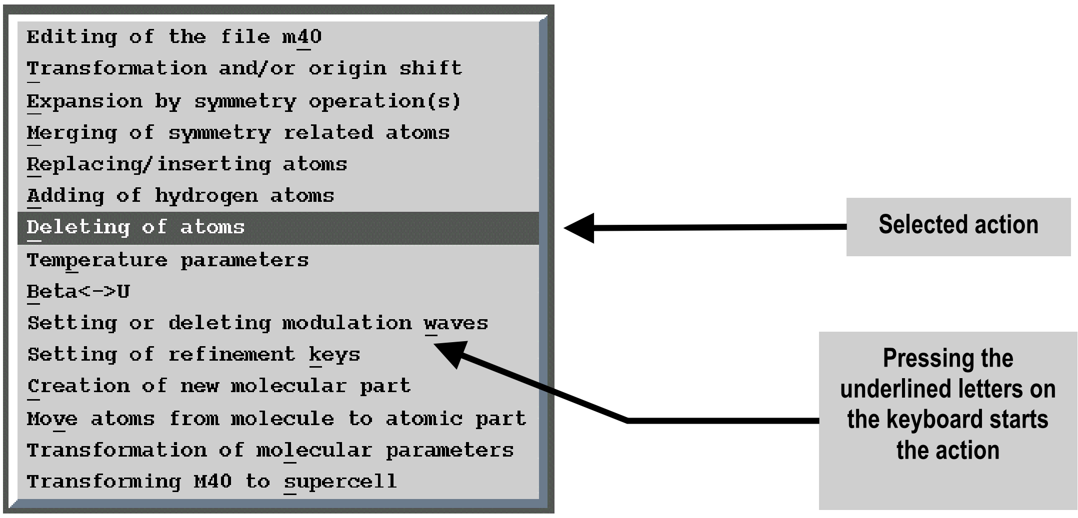
- Pull down menu
- Form
- Dialogue box
- Message box
- Special tools

All of them can be controlled by the mouse and from the keyboard. The special tools (for instance the File manager, the Select Atoms form etc.) will be described elsewhere.

Pull down menu

The pull down menu is used to select and start the desired action from the list.

Figure 11 *An Example of the Pull Down Menu*

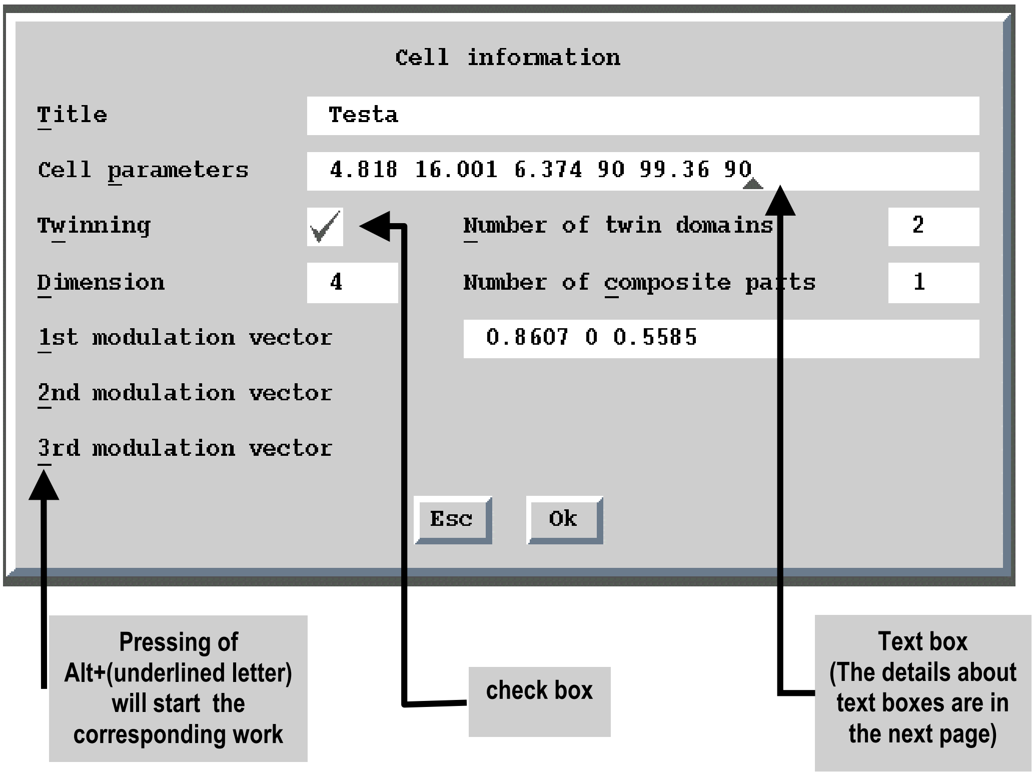


🖱	⌨
➢ **Click** *Selects and starts the action*	➢ **Enter** *Selects and starts the action*
➢ **Double-click** *Not useful*	➢ **Esc** *Cancels the menu*
➢ **Click out of the menu** *Closes the menu*	➢ **Arrows** *Select the action*
➢ **Mouse movement** *Selects the action*	➢ **Key** *Selects and starts the item containing the underlined letter*

Form

Form is used for gathering information necessary for some action. It contains the following objects: text boxes check boxes, selection boxes and buttons.

Figure 12 *An Example of the Form*



Cell information

Title: Testa

Cell parameters: 4.818 16.001 6.374 90 99.36 90

Twinning: ☑

Number of twin domains: 2

Dimension: 4

Number of composite parts: 1

1st modulation vector: 0.8607 0 0.5585

2nd modulation vector:

3rd modulation vector:

Pressing of Alt+(underlined letter) will start the corresponding work

check box

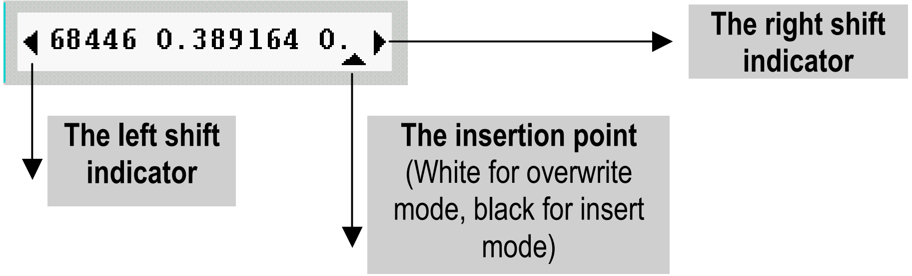
Text box (The details about text boxes are in the next page)

➢ **Click** *Selects an object*	➢ **Enter** *Selects OK button*
	➢ **Double-Enter** *Closes the form and starts the work*
	➢ **Esc** *Cancels the form*
	➢ **Arrows** *Move the insertion point inside the text boxes*
	➢ **Key** *Writes to text boxes*
	➢ **Alt-Key** *Selects the object denoted by underlined letter*
	➢ **Tab** *Changes the focus between text boxes. Some forms check the content of the text box for correctness before changing the focus.*

Text box

The text boxes are used for entering a text from the keyboard. Unlike standard windows programs the text cannot be cut or pasted.

Figure 13 *Example of the Text Box*

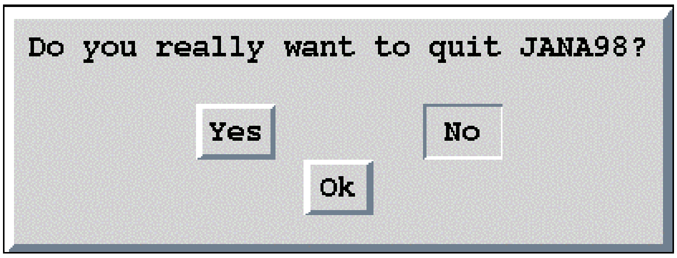


🖱	⌨
➢ **Click** *Selects the text box* *Sets the insertion point inside the text box* *Activates the buttons, check and selection boxes.*	➢ **Tab** *Changes the focus between text boxes and check boxes* ➢ **Arrows** *Moves the insertion point inside the text box* ➢ **Home** *Moves the insertion point to the beginning of the text box* ➢ **End** *Moves the insertion point to the end of the text box* ➢ **Insert** *Toggles between the insert and overwrite mode* ➢ **Enter** *Leaves the text box and selects the OK button if the text box is part of a form* ➢ **Ctrl-Y** *Deletes the contents of the text box*

Dialogue box

The Yes/No buttons can be selected by mouse, TAB key or by arrows.

Figure 14 *Example of a dialogue box*



Do you really want to quit JANA98?

1.2.6 Atom names in *JANA98*

Each atom of the structure model has a name listed in the refinement parameter file `m40`. The length of the name is limited to 8 characters, but ***the recommended length is 5 characters*** as in some cases *JANA98* appends another characters to the end of the atom name. The names are ***case insensitive***.

Wildcards

In the text boxes of some forms and also in the `restric`, `equation` and `fixed` commands of *REFINE* (see page 245) and the `center` command of *FOURIER* (see page 273) groups of atoms can be defined using the wild cards. The wildcards have usual meaning:

`Sn*`	denotes all names starting with string “sn”.
`S?`	denotes all names starting with “s” and containing two characters .
`?a*`	denotes names having the letter “a” in the second position.

Molecular positions

If some atom is a part of a molecule, a character denoting the molecular position is appended to the name. For instance, atom As of a model molecule has name Asa in the 1st position, Asb in the 2nd one etc. See page 140 and 171 for more information about molecules).

These extended names can be used for definition of a general plane (page 299), in the *DIST* `select` control command (page 312) etc They cannot be used in the refinement restriction commands (see page 245) because they are not present in the `m40` file.

The internal symmetry codes

Some tools of *JANA98* accept the internal symmetry codes denoting symmetry position of the atom. The internal symmetry code is separated from the basic name by “#” character.

Syntax: `sn` `cm` `ti1i2i3` (typed without spaces)

`sn`	specifies the |`n`|th symmetry operator from the `m50` file. If `n` is negative, the operation is combined with the center of symmetry[1].
`cm`	specifies that the `m`th centring vector will be added to the result of the symmetry transformation s_n (The centring vectors are listed in the basic crystal information part of any *JANA98* listing)
`ti1i2i3`	specifies the additional cell translation defined by three integers i1, i2 and i3.

---

[1] if it exists in the structure - otherwise an error message occurs

Examples :

```
Si3#s-3c2t1,-1,0
Na1#s2
Cr4#t1,0,-1
```

An atom name together with an internal symmetry code can exceed the length of 8 characters because it is never present in the `m40` file. The internal symmetry codes can be used for the definition of the general section plane and are also present in the *DIST* listing `m61`.

1.3 Basic parts of *JANA98*

1.3.1 List of programs

The following table shows basic programs of *JANA98*.

Table 4 *The basic programs of JANA98: purpose, input and output files.*
(The input files in the square brackets are not necessary for the program.)

name reference	input/output files purpose
DATRED (Page 55)	**Input:** diffractometer output file or [`m94`, -m95] **Output:** `m94`, `m95` **M50 control keywords:** none *DATRED* (the Data Reduction Program) reads the diffractometer output file, calculates LP and absorption corrections and creates the *basic reflection file* `m95`. If `m95` already exists, *DATRED* can either be used to modify the absorption correction or make transformation of cell parameters etc. or to import another diffractometer output file. *DATRED* also creates or updates the *header file of* *`m95`,* `m94`, containing information about orientation matrix, cell parameters and their transformations etc. **`M95` and `m94` MUST be used together.**
PRELIM (Page 67)	**Input:** `m94`,`m95` or a reflection file from other system(SHELX etc.), [`m50`] **Output:** `m50`,`m91`,[`m94`,`m95`],`pre` **M50 control keywords:** none *PRELIM* prepares interactively the *basic crystal information file* `m50` and then creates the *refinement reflection file* `m91`. If `m50` already exists, *PRELIM* reads the crystal information and offers its editing. The `m91` is created from data included in the *basic reflection file* `m95` and in the *header file of* *`m95`,* `m94`, by averaging reflections according the symmetry and by deleting the systematically absent reflections. *PRELIM* also contains a tool for reading reflection files of other systems, for instance SHELX. It doesn't convert them directly to `m91` but it creates `m95` and `m94` instead that can be used for creation of `m91` or can be further processed by *DATRED*. *PRELIM* can combine data sets of several measurements.
REFINE (Page 219)	**Input:** `m50`,`m91`,[`m40`] **Output:** `m40`,`m80`,`ref` **M50 control keywords:** the section between the keywords `refine` and `end` *REFINE* reads parameters from the *refinement parameter file* `m40` and refines them according to the commands in `m50` and the refinement keys in `m40`. It *REFINE* saves the results of the refinement to `m40` after each refinement cycle. During the last refinement cycle it creates the *Fourier reflection file* `m80`. If the input file `m40` doesn't exist or if it doesn't contain any atoms, *REFINE* automatically prepares `m80` file for the calculation of the Patterson map.

***Table 4** (continued)*

EDITM40 (Page 153)	**Input:** `m50`,[`m40`],[`m48`],[`m45`] **Output:** `m40` **M50 control keywords:** none *EDITM40* creates or edits the *refinement parameter file* `m40`. It can be used for adding or deleting atoms, creating molecules, setting of structure parameters, their initial values and refinement keys, etc. In addition two special input files `m48` and `m45` can be used in *EDITM40*, the former for adding of Fourier maxima to `m40`, the latter for importing of a model molecule.
DIST (Page 311)	**Input:** `m40`,`m50` ,[`m47`],[`m48`] **Output:** dis, `m61` **M50 control keywords:** the section between the keywords `dist` and `end` *DIST* calculates distances, angles, torsion angles and best planes for a structure model defined in `m40`. It can also work with coordinates of Fourier maxima and minima stored by *FOURIER* in the `m47` and `m48` file. For modulated structures *DIST* calculates distances and angles as a function of the `t` coordinate. The results are written to the FORTRAN listing `dis` in the newspaper columns form and to the file `m61` containing a complementary information.
FOURIER (Page 265)	**Input:** `m40`,`m50`,`m80` **Output:** `m81`,`m47`,`m48`,`fou` **M50 control keywords:** the section between the keywords `fourier` and `end` *FOURIER* calculates multidimensional electron density maps and stores them in the *Fourier map file* `m81`. It creates files `m47` and `m48` with a list of Fourier minima and maxima, respectively. In case of modulated structures `m48` also contains positional modulation amplitudes calculated from the maps. Note: The Fourier reflection file `m80` has to be created by *REFINE* before running *FOURIER* except the case when the Patterson map is needed. *REFINE* creates `m80` automatically during the last cycle so that a refinement interrupted by Break button does not yield `m80`. must not be interrupted by Break button. Zero refinement cycles is sufficient for creation of `m80`.
CONTOUR (Page 291)	**Input:** [`m40`],`m50`,[`m81`] **Output:** [files for SciAn] **M50 control keywords:** none *CONTOUR* interprets multidimensional electron density maps calculated by *FOURIER* and stored in the `m81` file. It makes contours plots of sections through the maps in the direct space for regular and modulated structures and electron/probability density plots based on a set of refined parameters including Gram-Charlier expansion terms up to 6th order. It can also generate input files for the program SciAn for 3d visualisation of electron/probability density. Error maps based on the refined parameters and the covariance matrix from the refinement can be calculated by applying the Monte Carlo method.
GRAPHT (Page 331)	**Input:** `m40`,`m50` **Output:** none **M50 control keywords:** none *GRAPHT* plots various quantities as a function of the `t` coordinate.

1.3.2 List of files

JANA98 uses basic, derived and temporary files. The basic files (`m95`, `m94`, `m50`, `m40` and `m91`[1]) contain all necessary information about the crystal data and structure model. The derived files contain information, which can be reconstructed from the basic files. The temporary files are used internally by the program and are deleted before exit.

Table 5 *The basic files of JANA98*

name reference	purpose
`m95` (Page 58)	**Name:** Basic reflection file **Created by:** *DATRED* or *PRELIM* **Input for:** *DATRED* or *PRELIM* The *basic reflection file* `m95` contains diffractometer data in a common format.In an ideal case it contains all measured reflections corrected only for LP factor. Every time the user changes symmetry of the structure model, transforms cell parameters or needs to recalculate the absorption correction *JANA98* uses data from this file. Then the *new refinement reflection file* `m91` can be created. `M95` is usually created by *DATRED* from the diffractometer output file. It can also be created by *PRELIM* by importing the reflection files of various formats (SHELX, JANA94, M94/M95 etc.) but in this case its usage may be limited (for instance, because the imported file may contain only symmetrically independent reflections). `M95` is used together with `m94`.
`m94`	**Name:** Header file of m95 **Created by:** *DATRED* or *PRELIM* **Input for:** *DATRED* or *PRELIM* The *Header file of m95*, m94 contains information about orientation matrix, cell parameters, possible user transformations etc.
`m50` (Page 77)	**Name:** Basic crystal information file **Created by:** *PRELIM* **Input for:** all programs of *JANA98* except *DATRED* The first part of the *basic crystal information file* `m50` contains cell parameters, symmetry information, atomic form factors etc. The basic information is common for all programs of *JANA98* and is separated from another part by the keyword `end`. The second part of `m50` contains keywords specific for basic programs of *JANA98*. A section for a particular program starts with the name of the program and ends with the keyword `end` . Note: *changing the basic crystal information part of m50 "by hand" is a bad practice*. The recommended tool is program *PRELIM.* The second section can be edited in a text editor or – more conveniently – by the Set Commands tool of *JANA98*.

---

[1] `m91` can be created from `m95` and `m50` but it is nevertheless called basic for its close relation to the structure model. With `m40`, `m50` and `m91` the user can immediately start refinement.

Table 6 *The derived files of JANA98*

`m40` (Page 93)	**Name:** Refinement parameter file **Created by:** *EDITM40* or *REFINE* **Input for:** all programs of *JANA98* except *DATRED* and *PRELIM* The *refinement parameter file* `m40` contains parameters of a structure model and corresponding refinement keys. `M40` can be created or edited by *EDITM40*. The refinement program uses `m40` as an input/output file, i.e. it is read as an input of the refinement and it is then updated after each refinement cycle. The backup copy of `m40` is named `s40` and is created automatically when *REFINE*. is started. Note: `m40` is the most complex file of *JANA98*. *EDITM40* contains a set of tools for changing parameters in `m40` but it is not editor of `m40`. Some changes has to be done in a text editor. Therefore the user should understand the structure of `m40`.
`m91` (Page 87)	**Name:** Refinement reflection file **Created by:** *PRELIM* **Input for:** *REFINE* The file `m91` contains reflections for the refinement. It is created by *PRELIM* from `m95` using information in `m50`.
`m93`	**Name:** FoFc table **Created by:** *REFINE* The table of observed and calculated structure factors is created by *REFINE* if the refinement control command `fofc` in `m50` is set to 1.
`m80`	**Name:** Fourier reflection file **Created by:** *REFINE* **Input for:** *FOURIER* The Fourier reflection file contains reflections for calculation of Fourier or Patterson maps. It is created in the last cycle of *REFINE*. If `m40` used by *REFINE* does not contain any atoms, *REFINE* prepares `m80` for the Patterson calculation. Note: `m80` is a binary file
`m48` (Page 282)	**Name:** Fourier maxima file **Created by:** *FOURIER* **Input for:** *EDITM40* The Fourier maxima file `m48` contains an ordered list of Fourier maxima together with the positional modulation amplitudes calculated from the maps. *EDITM40* can be used for adding the maxima as new atoms to the `m40` file.
`m47` (Page 282)	**Name:** Fourier minima file **Created by:** *FOURIER* **Input for:** *EDITM40* The Fourier minima file `m47` contains an ordered list of Fourier minima. The file has the same format as `m48` except modulation parameters.

Table 7 *The derived files of JANA98*

`m45` (Page 177)	**Name:** Model molecule file **Created by:** user **Input for:** *EDITM40* The *model molecule file* `m45` contains coordinates of a model molecule. It can be used for creation of a new molecule by *EDITM40.*
`m81`	**Name:** Fourier map file **Created by:** *FOURIER* **Input for:** *CONTOUR* The Fourier map file contains a set of Fourier maps represented by two-dimensional nets of numerical values of electron density. The *CONTOUR* program interprets m81. Note: `m81` is a binary file
`m61` (Page 330)	**Name:** Long listing of *DIST* **Created by:** *DIST* The `m61` contains the results of *DIST* in a one column form together with symmetry codes used for the calculation of a distance or an angle.
`pre` (Page 88)	Listing of *PRELIM*
`ref` (Page 260)	Listing of *REFINE*
`fou` (Page 283)	Listing of *FOURIER*
`dis` (Page 318)	Listing of *DIST*

1.3.3 Basic steps with *JANA98*

This part describes the basic common steps with *JANA98*. More information about the programs mentioned here is available in Table 4, the input and output files are explained in Table 5.

- ***Preparing the basic reflection file `m95` and m94***

There are two ways how to create `m95` and `m94`. If the output file from the data collection is available m95 and `m94` are created by *DATRED* by processing this file. In this case `m95` and `m94` contains complete information about the data set. Another way is the import of a reflection file created by other system by *PRELIM*. The usage of `m95` and `m94` created by *PRELIM* is limited as they may not contain full information.

- ***Preparing the basic crystal information file `m50` and the refinement reflection file `m91`***

Both `m50` and `m91` are created by *PRELIM*. `M50` is prepared interactively by supplying information requested by *PRELIM*. Then the user can create `m91` from `m95` through "Creation of refinement reflection file".

- ***Preparing the refinement parameter file `m40`***

The initial m40 can be prepared by two ways:

1. By solving the phase problem by the heavy atom method. *FOURIER* calculates the Patterson synthesis based on `m80` prepared by running zero refinement cycles with `m40` without atoms. If `m40` already contains atoms *FOURIER* uses ordinary `m80` and the type of map is chosen through the SetCommands tool The Patterson map can be interpreted using *CONTOUR* and the listing of *FOURIER*. The initial structure model can be added to `m40` by *EDITM40*.
2. By starting from the structure model solved by other systems. *JANA98* contains tools for reading SHELX and CIF files. Both are accessible in the Tools menu in the basic window of *JANA98*.

- ***Refining the structure***

Once `m91`, `m50` and `m40` are prepared the user can refine the structure by *REFINE*. The SetCommands tool can set the parameters of the refinement. The refined model is saved in `m40` and new input file for Fourier calculation is available in `m80`.

- ***Calculating Fourier and Patterson maps***

The Fourier and Patterson maps are calculated by *FOURIER*. The SetCommands tool controls the type of the map and other characteristics. The input file for *FOURIER* is the file `m80` created by *REFINE*. The Fourier maxima and (for modulated structures) modulation parameters found in the map are saved in `m48`, which can be directly used as an input for *EDITM40*.

- ***Interpretation of the Fourier maps by the* CONTOUR *program***

CONTOUR interprets the maps stored by *FOURIER* in `m81`.

- ***Calculation of the distances and angles***

DIST calculates distances and angles from the parameters written by *REFINE* to `m40`. It also works with the Fourier maxima stored by *FOURIER* `in m48`.

The following three examples illustrate relation between *DATRED* and *PRELIM*.

- ***Changing the symmetry***

The user can change the symmetry information in `m50` through the Symmetry form of *PRELIM*. This change has no influence to `m40` and `m91`, which must be updated by the user. New `m91` is created from `m95` by Creation of the refinement reflection file. The necessary change of `m40` depends on the new symmetry.

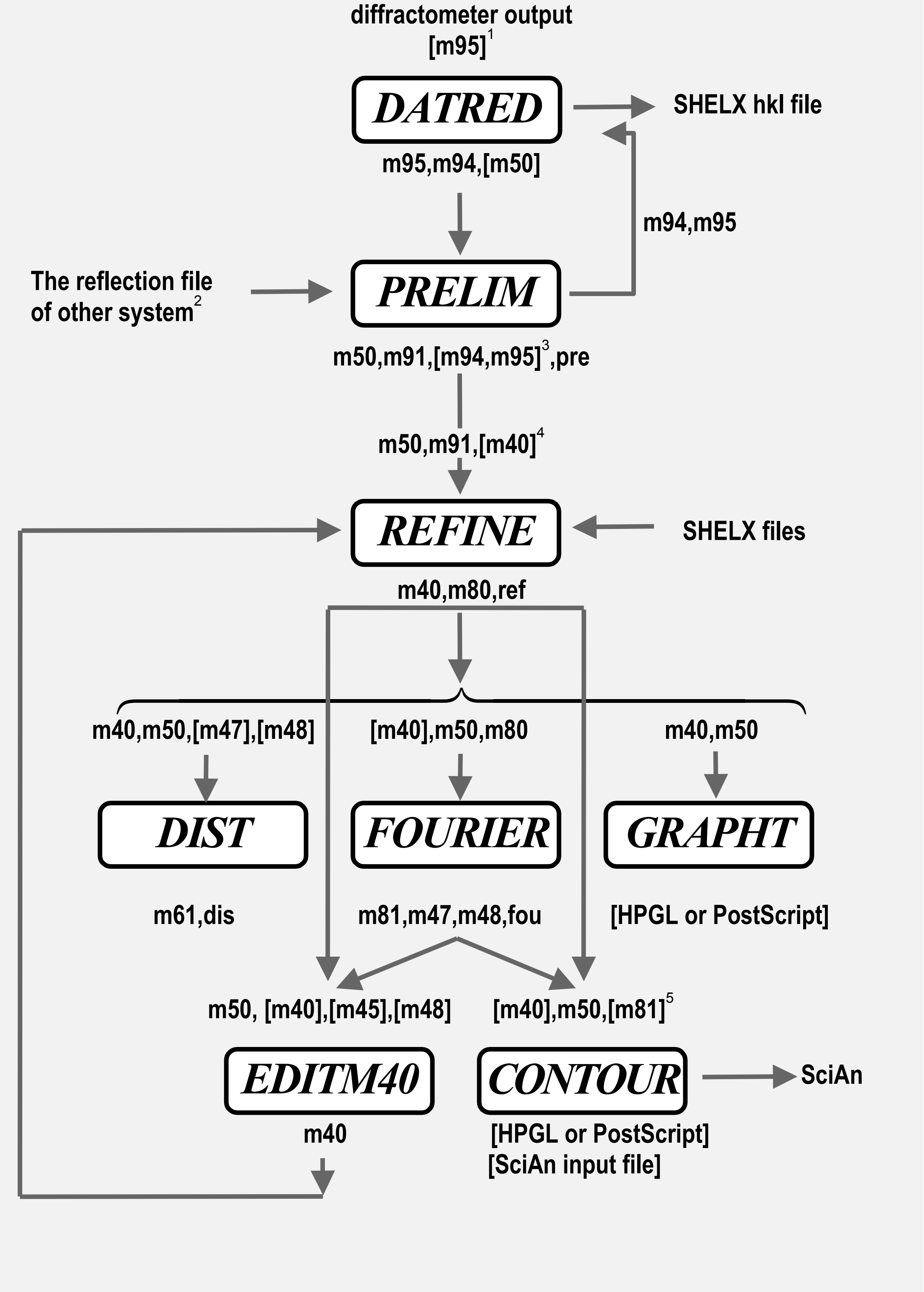
- ***Changing the absorption correction***

The absorption correction can be changed by *DATRED*. The necessary condition is that `m95` contains desired information, i.e. that it has been created directly from the diffractometer file. *DATRED* calculates the new correction and creates the new *basic reflection file* `m95`. Then the user can create new `m91` by *PRELIM*.

- ***Merging reflections of several measurements***

JANA98 can work simultaneously with data from several measurements of the same crystal. The necessary condition is that the data are of the same type - X-ray and neutron data cannot be mixed. First the user processes each data set by *DATRED* using different job names for each run. *DATRED* creates the basic reflection files like `name1.m95`, `name2.m95` … and corresponding files `m94`. Then the user starts *JANA98* again with the definitive job name and joins all `m95's` by the “Import of reflections” tool of *PRELIM* into one single `m95`. The last step is creation of new `m91` by *PRELIM*. Each data set can have its own scale in `m91. If` there are some common reflections in all sets the sets can be rescaled to a common scale.

Figure 15 *The basic programs communication scheme*



---

[1] The diffractometer output is not used if `m95` already exists.

[2] See p. 72 for more information about supported formats.

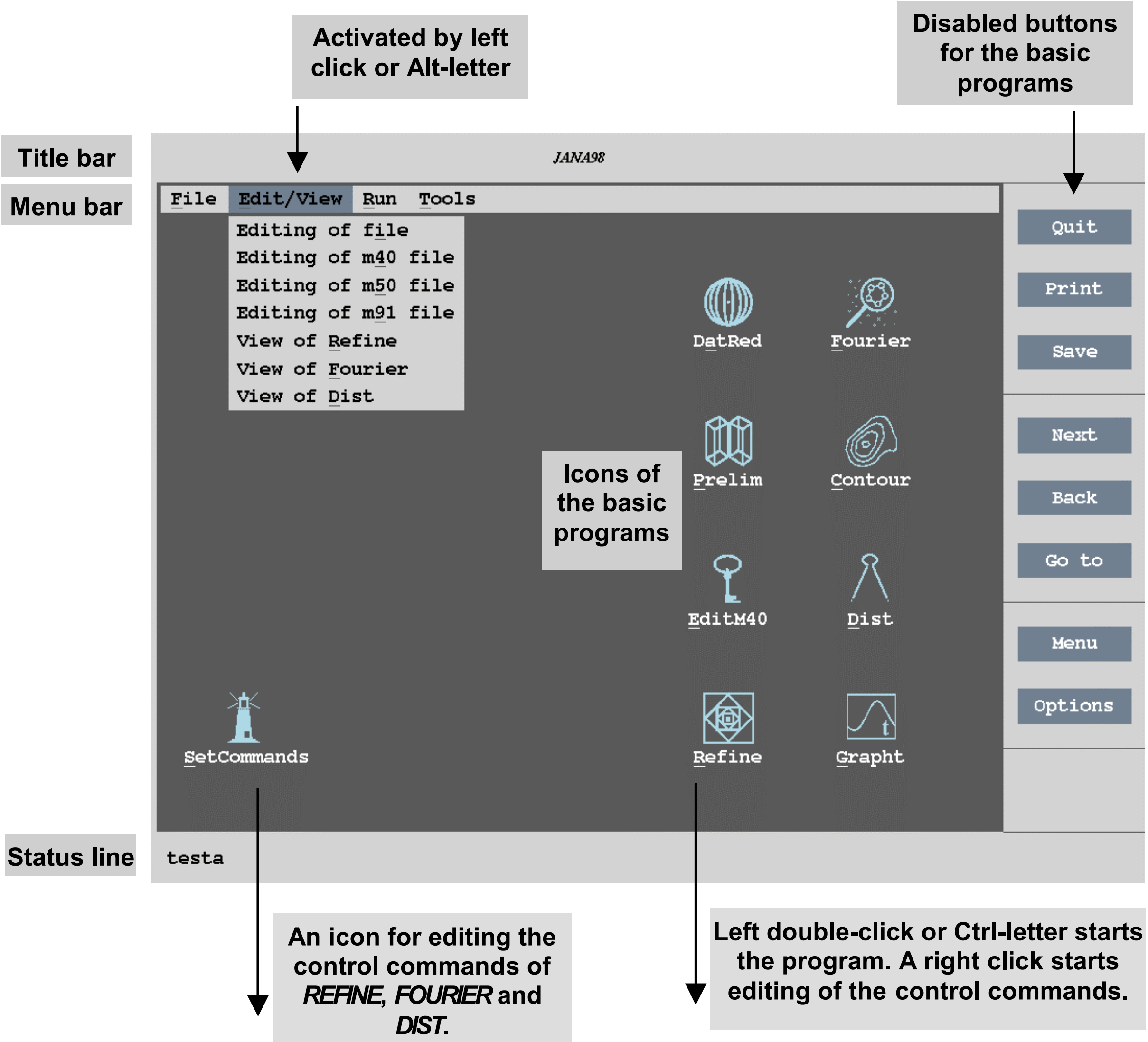
[3] *PRELIM* creates or changes `m94` and creates or extends `m95` if it imports data from other system or already existing file.

[4] If the `m40` is missing or if it doesn't contain atoms, *REFINE* prepares the `m80` file for the Patterson synthesis.

[5] The `m81` is not necessary if *CONTOUR* is used for plotting of probability density functions.

1.4 The basic window and common tools

Figure 16 *The basic window of JANA98*



Activated by left click or Alt-letter

Disabled buttons for the basic programs

Title bar

Menu bar

Icons of the basic programs

Status line

An icon for editing the control commands of *REFINE*, *FOURIER* and *DIST*.

Left double-click or Ctrl-letter starts the program. A right click starts editing of the control commands.

Starting of *JANA98* is explained in page 21. After loading of *JANA98* the user see the basic window with a menu bar, a status bar, icons and buttons. The ***icons*** are used for starting the basic programs of *JANA98* or for the modification of their control commands. The ***menu bar*** is used for the actions, which are not included in the basic programs. The ***buttons*** on the right are all disabled and reserved for basic programs. The **status line** shows the name of the current structure.

Table 8 *Control of the JANA98 basic window*

🖱	⌨
➢ **Left click** *Activates a pull down menu* *Starts actions from the pull down menus* *Selects an icon*	➢ **Enter** *Starts the item selected in a pull down menu or a program associated with a selected icon*
➢ **Left double-click on a icon** *Starts a program associated with the icon*	➢ **Esc** *Not useful (The basic window can be closed only by File->exit)*
➢ **Right click on a icon** *Starts editing of the control commands of a program associated with the icon*	➢ **Arrows** *Changes the focus between icons if one of them has been already selected.* *Selects items of an activated pull down menu*
➢ **Click out of the pull down menu** *Closes the pull down menu*	➢ **Alt-Key** *Activates the pull down menu denoted by underlined letter*
➢ **Mouse movement** *Selects an item of the active pull down menu* *Changes focus between the pull down menus if one of them is already activated*	➢ **Ctrl-Key** *Starts a program containing the underlined letter in the name of its icon.*
	➢ **Key** *Starts an item of the pull down menu containing the underlined letter*
	➢ **Tab** *Not useful*

1.4.1 The SetCommands Tool

This tool is used for editing user commands of basic programs of *JANA98*. The user commands are saved in `m50`, their syntax is explained page 77 and also in the chapters describing the basic programs.
SetCommands can be started by clicking the corresponding icon in the basic window of *JANA98*. In this case it offers a list of programs for which the tool is available. It can be also started directly for the relevant program by clicking the right mouse button on the program icon.

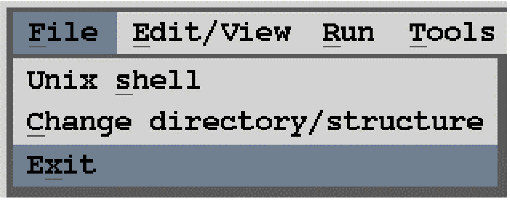
The SetCommands works for *REFINE*, *FOURIER* and *DIST*. First it sets the default options of the program, then it reads possible user commands from `m50` and finally it opens the graphics interface for editing of the commands. After quitting and user confirmation it writes the commands back to `m50`, but only the ones different of the default values.

1.4.2 The File Menu

The `Unix shell` item of the File menu is used for executing of a Unix shell command. Typically it starts the xterm window but the action is configurable via user preferences (see page 46). In the DOS version the item is called `Skip to DOS` and starts a DOS window or a DOS command line.

The `Change directory/structure` item starts the File manager of *JANA98*, which is described in the next page.

Figure 17 *The File Menu*

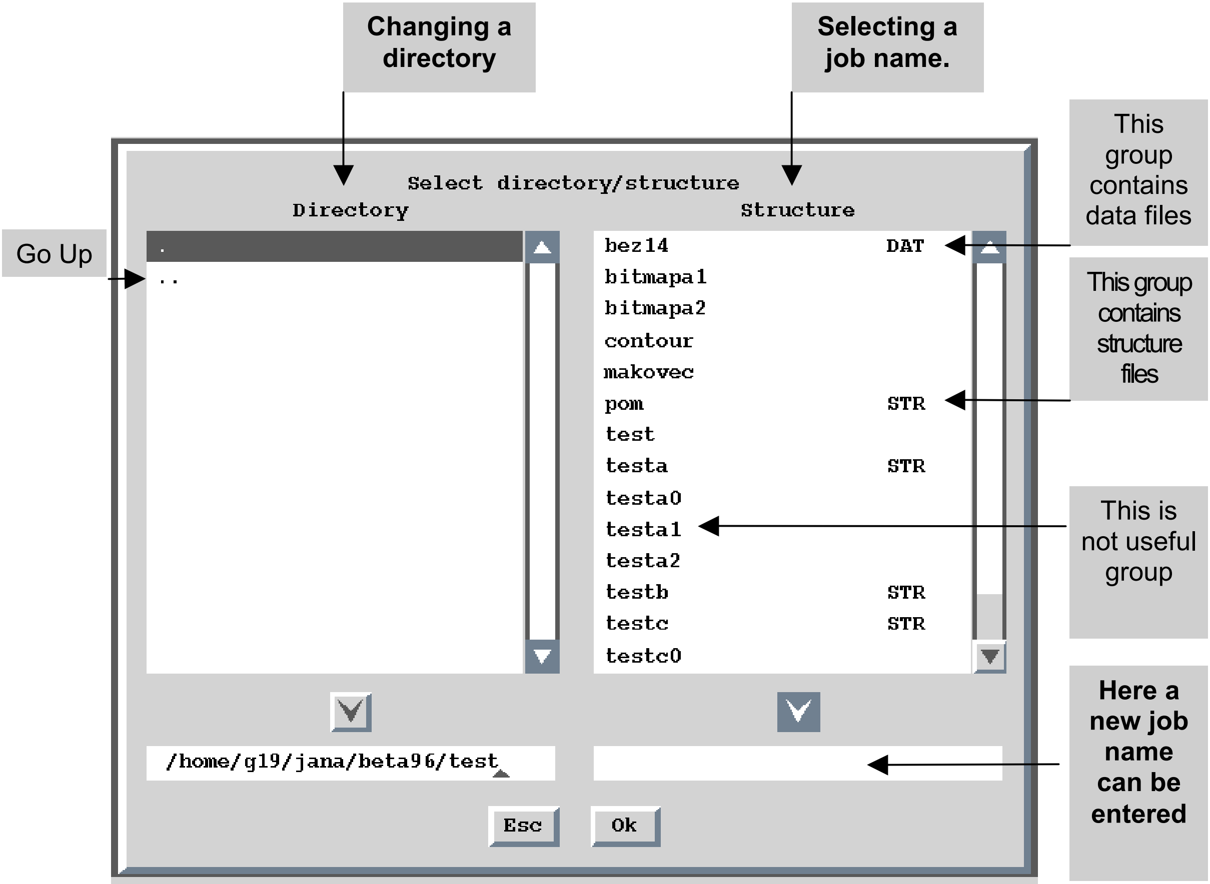


This starts the File manager working in the group mode

The file manager

The file manager is used for selecting or changing the current job name. The left window is used for selecting the directory, the right one lists the files. If some files are recognised as belonging to one job name they are grouped together and given a flag STR or DAT. This feature occurs when the file manager is started in the group mode.
The user can choose an existing job name or type a name of a new job in the textbox on the right. In both cases after quitting the File manager *JANA98* skips to the selected directory and uses it as the new working directory.

Figure 18 *The file manager working in the group mode*



Changing a directory

Selecting a job name.

Go Up

This group contains data files

This group contains structure files

This is not useful group

Here a new job name can be entered

```
Select directory/structure
Directory                      Structure
.                              bez14          DAT
..                             bitmapa1
                               bitmapa2
                               contour
                               makovec
                               pom            STR
                               test
                               testa          STR
                               testa0
                               testa1
                               testa2
                               testb          STR
                               testc          STR
                               testc0

/home/g19/jana/beta96/test
```

How to change a directory:

- **double click the line with the directory name**
- **or select the line and press Alt-V**
- **or select the line and press** ∨

How to change a job name:

- **double click the line with the job name**
- **or select the line and press Alt-V**
- **or select the line and press** ∨
- **or type the job name to the right text box. This is the way to create a new job name.**

Note: A structure name selected in the right panel by a single click but not sent to the textbox by a double-click is ignored.

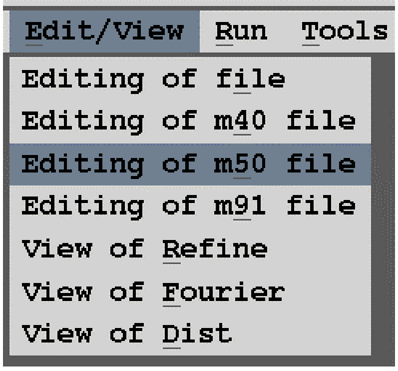
The Edit Menu

`Editing of file` starts the file manager without the group mode, i.e. showing all files in a selected directory.

The item `Editing of m40/m50/m91 file` opens a file directly in the text editor. The default text editor is `edit.com` for the case of DOS and `vi` for Unix. In both cases the editor command can be set by the user in preferences.

The item `View of Refine/Fourier/Dist` activates the listing viewer, which is described in the next paragraph.

Figure 19 *The Edit Menu*

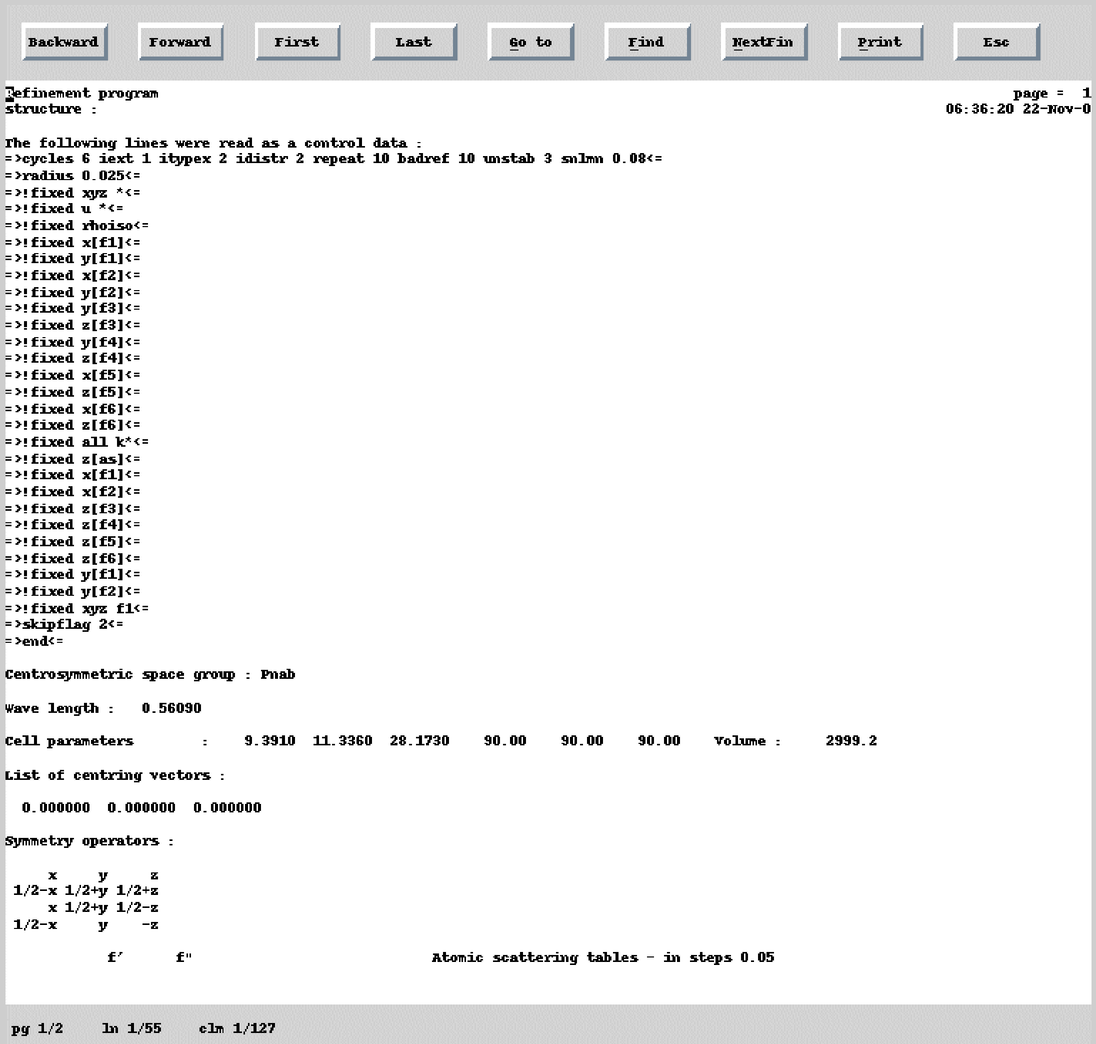


The Listing Viewer

The listing viewer shows the listing of *REFINE*, *FOURIER* or *DIST*. The advantage over using an ordinary text editor is that the listing is properly formatted showing 1 page in one screen. This is especially useful for *DIST* that prints the distances in the newspaper column format.

In the user preferences the listing viewer can be replaced by a text editor defined by the user.

Figure 20 *The listing viewer*

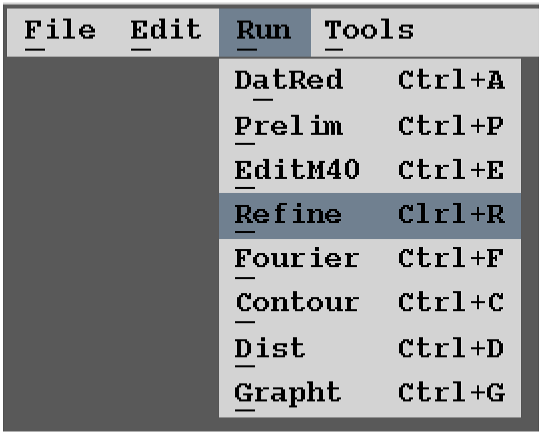


```
Refinement program                                                                                                   page =   1
structure :                                                                                                  06:36:20 22-Nov-0

The following lines were read as a control data :
=>cycles 6 iext 1 itypex 2 idistr 2 repeat 10 badref 10 unstab 3 snlmn 0.08<=
=>radius 0.025<=
=>!fixed xyz *<=
=>!fixed u *<=
=>!fixed rhoiso<=
=>!fixed x[f1]<=
=>!fixed y[f1]<=
=>!fixed x[f2]<=
=>!fixed y[f2]<=
=>!fixed y[f3]<=
=>!fixed z[f3]<=
=>!fixed y[f4]<=
=>!fixed z[f4]<=
=>!fixed x[f5]<=
=>!fixed z[f5]<=
=>!fixed x[f6]<=
=>!fixed z[f6]<=
=>!fixed all k*<=
=>!fixed z[as]<=
=>!fixed x[f1]<=
=>!fixed x[f2]<=
=>!fixed z[f3]<=
=>!fixed z[f4]<=
=>!fixed z[f5]<=
=>!fixed z[f6]<=
=>!fixed y[f1]<=
=>!fixed y[f2]<=
=>!fixed xyz f1<=
=>skipflag 2<=
=>end<=

Centrosymmetric space group : Pnab

Wave length :   0.56090

Cell parameters        :    9.3910  11.3360  28.1730    90.00    90.00    90.00    Volume :     2999.2

List of centring vectors :

  0.000000  0.000000  0.000000

Symmetry operators :

     x     y     z
 1/2-x 1/2+y 1/2+z
     x 1/2+y 1/2-z
 1/2-x     y    -z

           f'      f"                              Atomic scattering tables - in steps 0.05

pg 1/2     ln 1/55     clm 1/127
```

1.4.3 The Run Menu

The Run menu is used for starting the basic programs of *JANA98*.

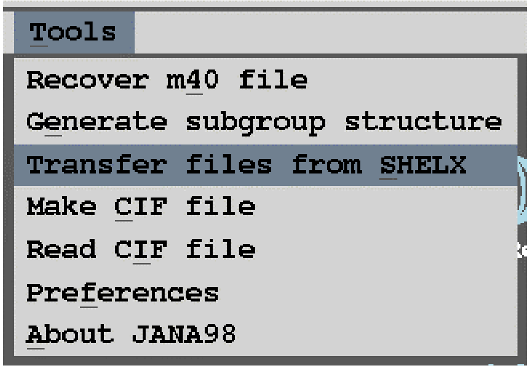
Figure 21 *The Run Menu*



1.4.4 The Tools Menu

The Tools menu contains various jobs not made by basic programs.

Figure 22 *The Tools Menu*



Recovering `m40` *file*

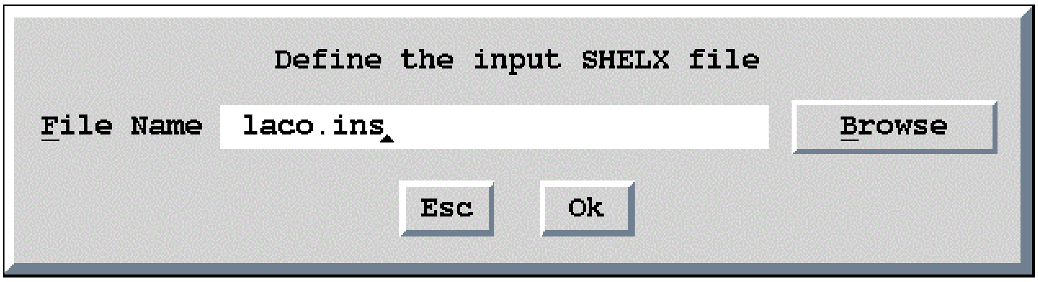
At the beginning of each refinement program *REFINE* makes `m40` backup file named `s40`. This tool copies the `s40` back to the `m40`.

In the current version of *JANA98* there is only one level of backup. If the refinement runs with the refinement repeat greater that 1 the `s40` is overwritten after each refinement repeat (see page 231).

Transferring files from SHELX

This tool reads the `name.hkl` and `name.ins` SHELX files and creates files `m40`, `m50`, `m91`, `m94` and `m95`. The job name of the created files is the one *JANA98* has been started with.

Figure 23 *Importing SHELX files*


Define the input SHELX file

File Name: laco.ins

Creating CIF Files

JANA98 supports the core CIF dictionary version 2.1beta3 (the last update 8/12/1997) and the CIF for modulated structures (G.Madariaga, 1994) version 0.4 (the last update 27/9/1998).

This tool creates CIF file `name.cif` by combining the files `m40`, `m50` and `smr`. The file `name.smr` is used for collecting necessary information during the run of the basic programs of *JANA98*. Each program has its own header in `name.smr`..

In order to have as complete CIF file as possible the user should

- check the basic crystallographic information like the cell contents in *PRELIM* .
- run several cycles of the refinement with the option for creation of the FoFc file
- calculate difference Fourier map for the independent cell volume
- run *DIST* with carefully selected bond limits and other options

If some information needed for the CIF file is missing *JANA98* fills the missing fields with "?". Some fields, like `_cell_measurement_temperature` are always filled with "?" and the user is expected to complete them in a text editor. The information from *DIST* usually contains superfluous items and needs some editing, too.

The CIF concept, syntax and tools are described in http://www.iucr.ac.uk. The CIF files can be checked or printed with help of the following automatic services[1]:

- The services for checking of the syntax and completeness of the CIF files are checkcif@iucr.ac.uk and http://www.iucr.org/iucr-top/journals/acta/c/services/checkcif.html**.** If the mail serves is used the CIF file should be sent as the body of the message.
- The services for printing the CIF are printcif@iucr.ac.uk and http://www.iucr.org/iucr-top/journals/acta/c/services/printcif.html**.** In the case of the mail server the printed CIF is returned as a PostScript file. The WWW service returns a PDF file.

Reading CIF files

This tool reads CIF files and converts them to `m40`, `m50` , `m91`, `m94` and `m95`. The initial job name is arbitrary as the tool gives the possibility to select new name for the converted structure. The files `m91`, `m94` and `m95` are created from the FoFc table by the standard procedure described in page 83.

---

[1] Unfortunately CIF for modulated structures is not supported. However, this version of CIF dictionary is accepted in the database of modulated structures in http://www.cryst.ehu.es/icsdb.

Generating a subgroup structure

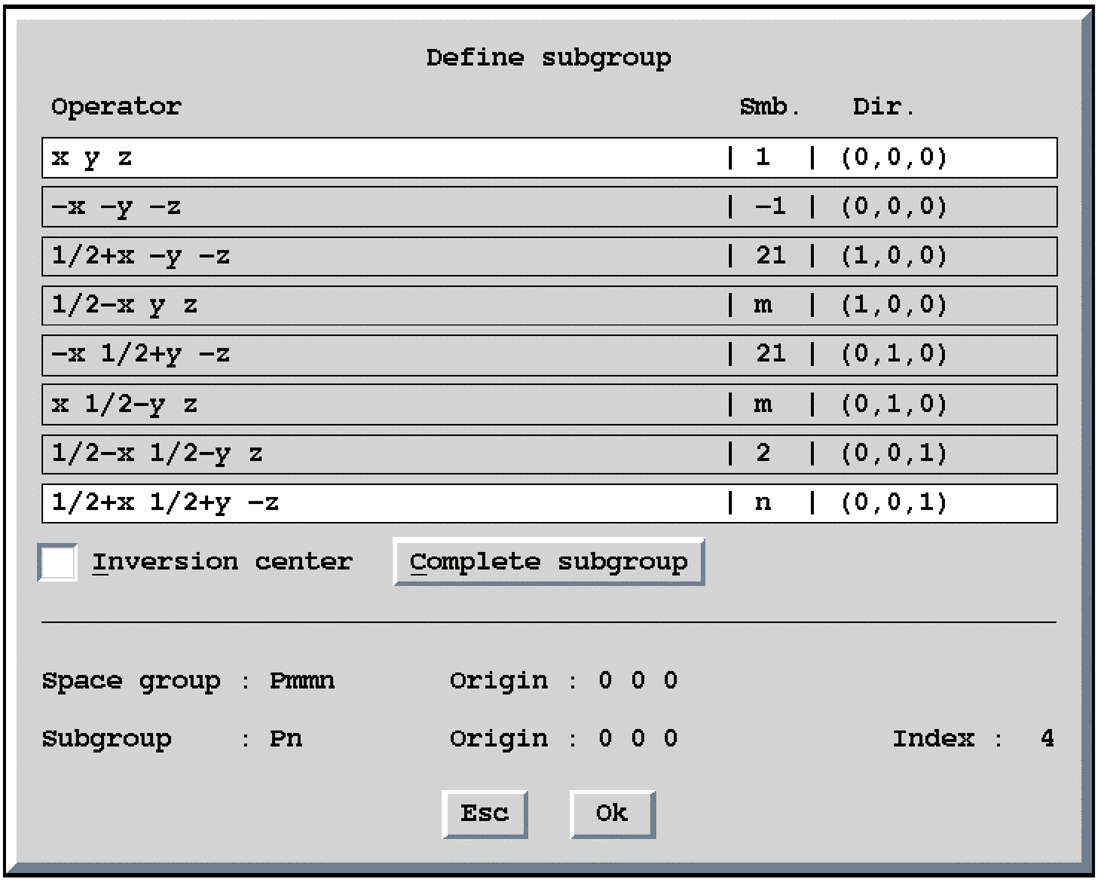
One of the typical tasks of the structure analysis is changing of a structure model with higher symmetry to a lower symmetrical one. This tool enables to do such transformation easily and consistently. The necessary condition is that the new space group is a subgroup of the original one.

- In the first step the user selects from the list of symmetry operators which ones should be present in the new structure model. After pressing the `Complete subgroup` button the program completes the subgroup (this means that only necessary generators can be selected) and tries to derive the new space group symbol from the selected operators.

- When the subgroup is successfully completed and the form is closed the procedure continues by giving the user possibility of choosing operators which will be used for expanding the structure model or generation of twinning operations. This selection cannot influence results but can be convenient for instance in cases when several structure models are to be compared.

- In the last step the user decides if the removed symmetry operators are to be replaced by twinning operators and also selects the job name for the new structure model. Then the procedure creates the basic files of the lower symmetrical model.

Figure 24 *Generation of the subgroup structure*



Define subgroup

Operator	Smb.	Dir.
x y z	1	(0,0,0)
-x -y -z	-1	(0,0,0)
1/2+x -y -z	21	(1,0,0)
1/2-x y z	m	(1,0,0)
-x 1/2+y -z	21	(0,1,0)
x 1/2-y z	m	(0,1,0)
1/2-x 1/2-y z	2	(0,0,1)
1/2+x 1/2+y -z	n	(0,0,1)

☐ Inversion center

Space group : Pmmn Origin : 0 0 0

Subgroup : Pn Origin : 0 0 0 Index : 4

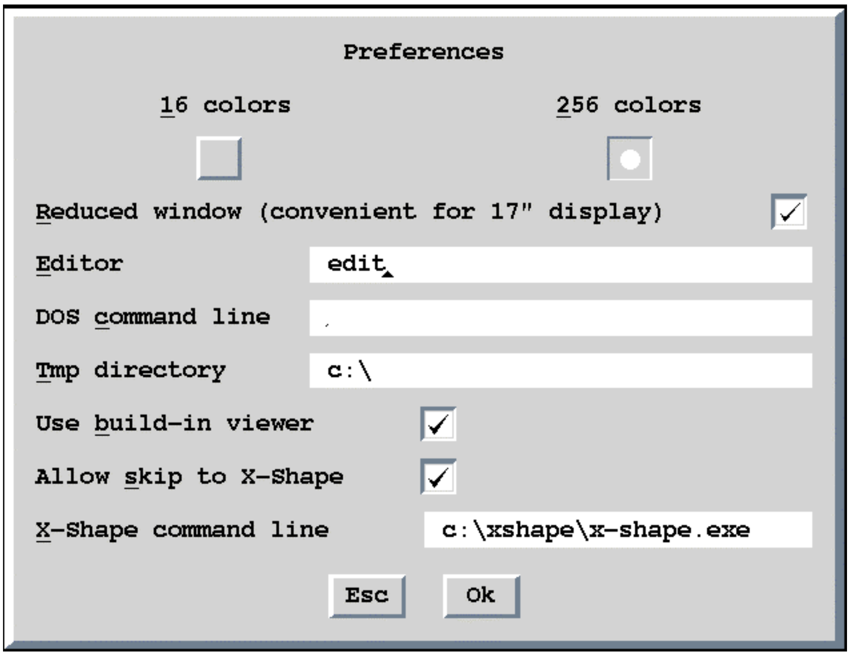
User preferences

Both PC and UNIX version of *JANA98* uses the configuration file jana96.ini (see page 12 for details about UNIX initialisation file with the user preferences. This tool is the interface to this file.

DOS preferences

- The `Editor` line contains the text editor command - edit is default as it is always available.
- The `DOS command` line contains the command executed by File->Skip to DOS. Implicitly it is the DOS window.
- `Tmp directory` is used for temporary files - see page 16 for details.
- The `Use built-in viewer` option is used for choosing between the built-in viewer or a text editor for viewing the listings of the basic programs.
- X-Shape is a program of STOE + CIE GmbH for optimisation of the crystal shape for absorption correction. Its usage in connection with *JANA98* is explained in page 60.

Figure 25 *The User Preferences for the PC Version*



16 colors ☐
256 colors ●
Reduced window (convenient for 17" display) ☑
Editor: edit
DOS command line:
Tmp directory: c:\
Use build-in viewer ☑
Allow skip to X-Shape ☑
X-Shape command line: c:\xshape\x-shape.exe

UNIX preferences

Unix preferences are much more complicated than in the case of DOS due to the existence of wide variety of workstations and flexibility of the environment. The most important features are

- hierarchy of initialisation files (see page 12 for details)
- possibility to define display specific settings, i.e. settings valid for a specific internet display address.

UNIX general preferences

The following figure shows the General preferences, which are executed regardless of the display address.

- `Start as icon` means that *JANA98* will be automatically iconised after the start up.
- `Set icon position` defines the coordinates (in pixels) of the upper left corner of the icon window. If the x coordinate is negative it is understood as the distance between the right side of the icon window and the right side of the display. If the y coordinate is negative it is understood as the distance between the bottom of the icon window and the bottom of the display.
- The `Window position` enables to define the coordinates (in pixels) of the upper left corner of the *JANA98* window. For negative coordinates the same rules are valid like in the case of `Set icon position`. If the check box is activated and the user moves the window of *JANA98* by mouse (with open Preferences form) the current coordinates of the upper left corner appear automatically in the text boxes.
- The double click speed is given in tenth of the second.
- The `editor and printer commands` contain special characters %d and %f. The %d stands for the name of the X11 monospaced font used like *JANA98* system font. The %f stands for the name of the file, which will be printed or edited. The %f must not be omitted.
- The `Unix shell command` is used by File->Unix shell
- The `viewer command` is used for editing of listings of the basic programs in case the user does not prefer the built-in viewer.

Examples of printer commands

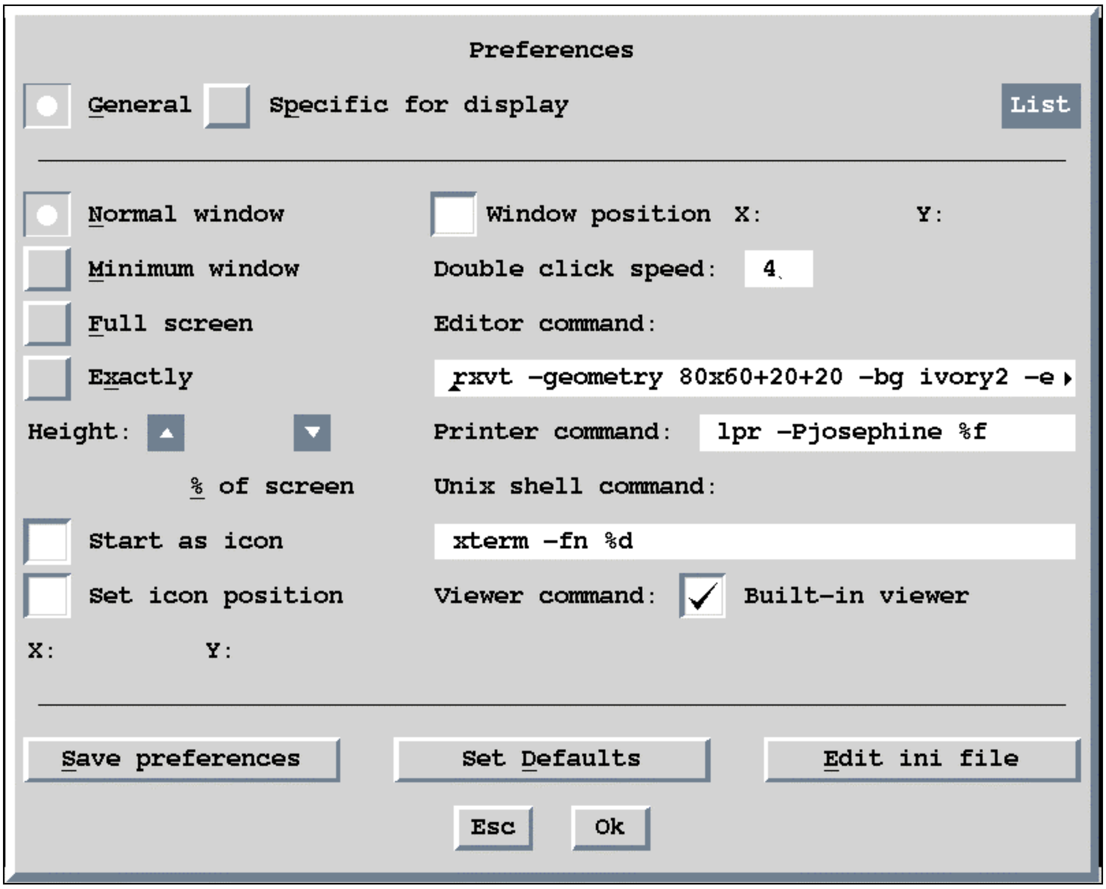
`lpr  %f`	sends the output to the default printer.
`lpr -Pname %f`	sends the output to the printer named `name`.
`lpr -Pname -s %f`	sends the output to the printer named `name`. The option -s causes the file, which has to be printed, is not copied to the spool area so that large file can be printed.
`rcp %f my.dear.cz:work/pppp; rsh my.dear.cz "lpr -Pmyprinter work/pppp"`	This command is useful in case that the user runs *JANA98* on a fast central computer but needs printing on a local printer which is not directly accessible. The postscript print file (`%f`) is copied to the local workstation `my.dear.cz` to the directory `work` and named `pppp`. Then the print command for the local printer `myprinter` is executed. The user must be allowed to execute the `rcp` and

	`rsh` commands on the local workstation - please contact the system administrator for more details.
`xterm -e prjana %f`	The last command can help in the special case that a user dialogue precedes the printing. Typical situation is if the print server asks for login and password. The command opens the `xterm` window and immediately starts the procedure `prjana` in the window. `Prjana` should contain commands for connecting the print server and printing. The `xterm` window is closed automatically when `prjana` is finished.

Examples of editor commands

`textedit -fn %d %f`	Typical command for SUN workstation
`emacs -font %d %f`	The command for emacs editor.
`xterm -fn %d -e vi %f`	The command for vi editor. This editor does not open its own window and in this command it uses the xterm window.
`nedit %f`	The command for nedit editor. This is one of the most user friendly editors in Unix world.

Figure 26 *The General User Preferences for UNIX Version*



Preferences

● General ☐ Specific for display

● Normal window
☐ Minimum window
☐ Full screen
☐ Exactly
Height:
% of screen
☐ Start as icon
☐ Set icon position
X: Y:

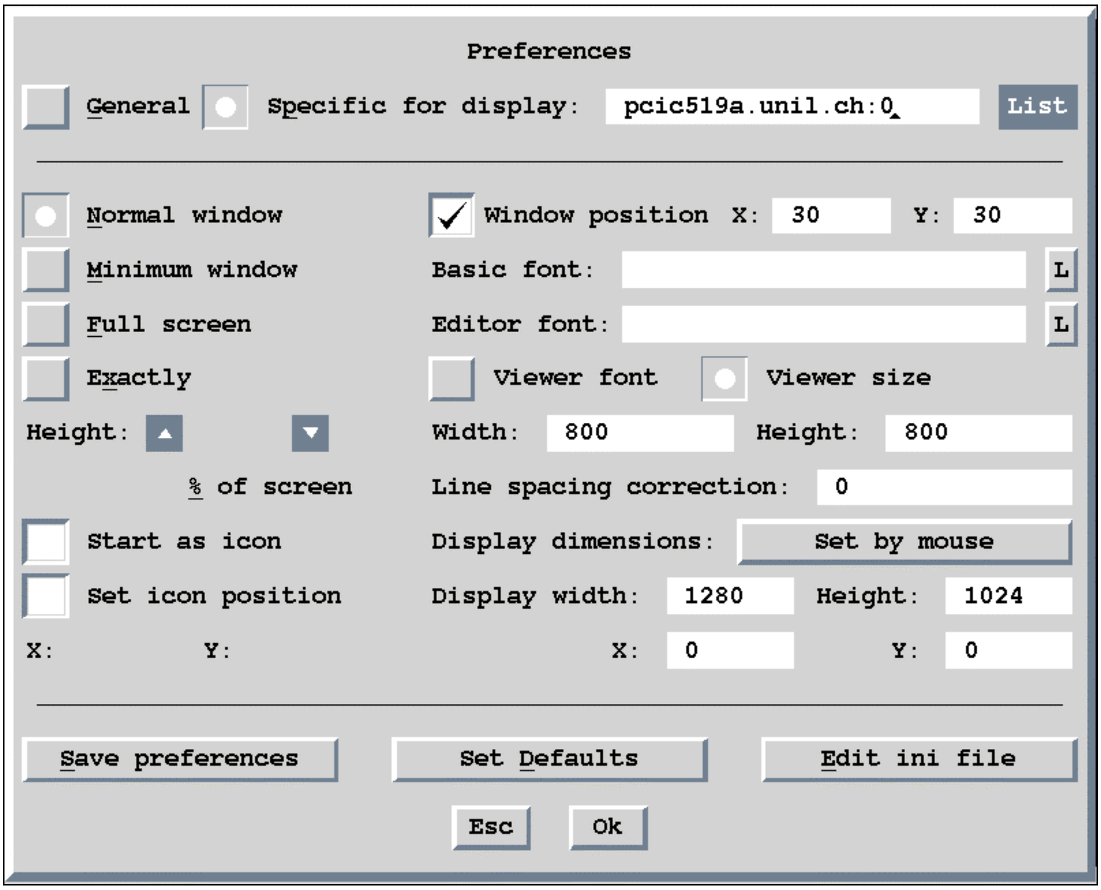
☐ Window position X: Y:
Double click speed: 4
Editor command: rxvt -geometry 80x60+20+20 -bg ivory2 -e
Printer command: lpr -Pjosephine %f
Unix shell command: xterm -fn %d
Viewer command: ☑ Built-in viewer

UNIX display-specific preferences

JANA98 automatically recognises an internet address of the display. If the address matches the one written to the initialisation file the corresponding preferences are used. The initialisation file can contain options for several displays. Problems with display-specific settings may arise when using ssh for connection to the Unix workstation because the secure shell changes the display address. See page 22 for more information.

- The options in the left part of the form are the same like the ones for General preferences. The Display specific preferences take priority over the General preferences.
- The `Basic font` is the one used for *JANA98* menus, forms etc. It can be selected by `L` button but the user rarely needs to change it. Proportional fonts are not allowed.
- The `editor font` is used for editing of files. It can be selected by `L` button and must not be proportional.
- The `Viewer size` and the `Viewer font` are used for the built in listing viewer. In the case the user does not use the built in viewer the listings are opened in a text editor and displayed with editor font. The `Viewer size` and the `Viewer font` cannot be set independently. They should be only used if the default viewer settings do not give satisfactory results, for instance if the viewer font is too small. In this case the most effective way is changing the `Viewer size`. The size units are given in pixels.
- `Line spacing correction` is used for the listing viewer. It is given in pixels and a positive value enlarges the line spacing.
- The `Display dimensions` setting is used for the definition of a virtual display. The user does not need this option under normal circumstances. Some X servers, however, returns invalid information about display dimensions. The virtual display defines the visible area of the screen and the window position and size are defined with respect to this rectangle. If the display dimensions are changed the position and window size are immediately recalculated in order to have the same window at the same place.

Figure 27 *The Display-Specific User Preferences for UNIX version*



Preferences

☐ General ● Specific for display: pcic519a.unil.ch:0

● Normal window ☑ Window position X: 30 Y: 30

☐ Minimum window Basic font:

☐ Full screen Editor font:

☐ Exactly ☐ Viewer font ● Viewer size

Height: ▲ ▼ Width: 800 Height: 800

% of screen Line spacing correction: 0

☐ Start as icon Display dimensions: Set by mouse

☐ Set icon position Display width: 1280 Height: 1024

X: Y: X: 0 Y: 0

Resizing JANA98 basic window

The PC version of *JANA98* runs in the full screen mode and the user can only select the reduced or full window size.

UNIX users can define the size of the *JANA98* basic window by three ways:

- By the command line option `-scale` (see page 21).
- Through the "Preferences" form.
- By dragging the corner of the window by mouse

The "Normal window" in the Preferences is the default one or the one defined by the `-scale` option. The "Minimal window" is the smallest window *JANA98* can properly work in.

The resizing of the window by mouse works only in the basic window mode. The size of the window is changed and the X11 fonts are reloaded. The window manager is instructed to keep the aspect ratio unchanged. If *JANA98* is not in the basic window mode, the window size is automatically backed to the original value.

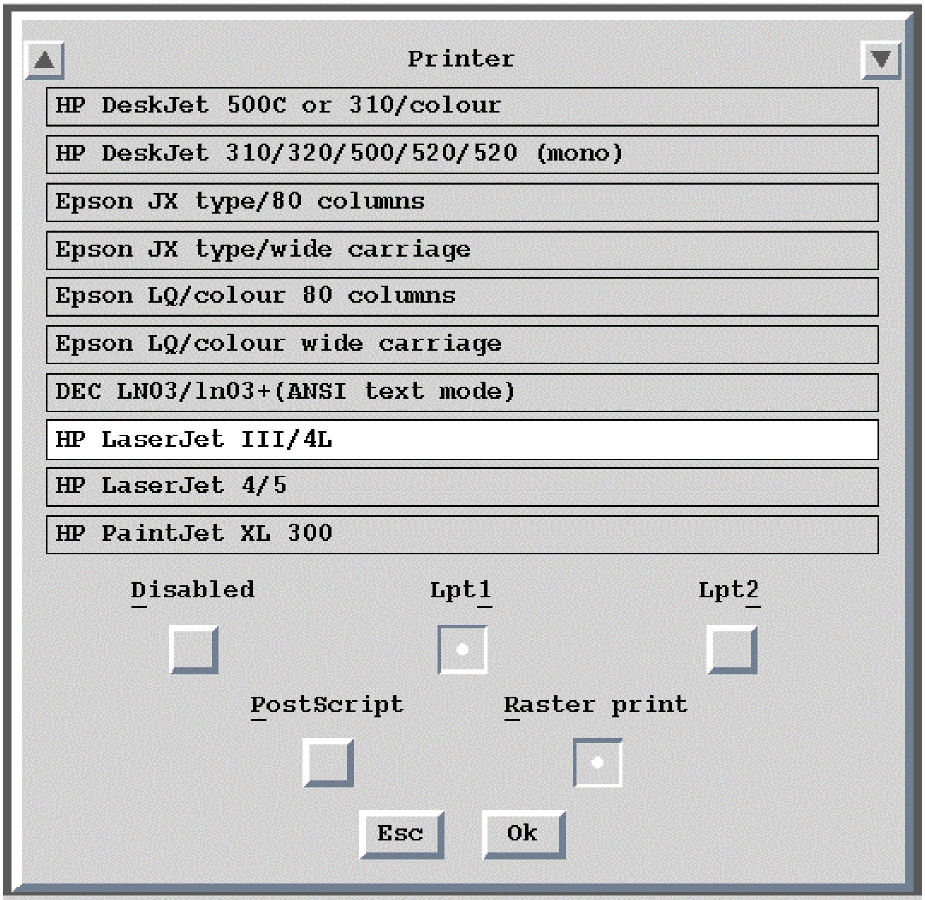
Printer settings

Some basic programs of *JANA98* and also the listing viewer can send an output directly to a printer through a Print button. Printing works only the printer settings in Preferences is correct.

The printer settings for UNIX discussed in page 47. The output format of the print files is always PostScript.

The PC printer settings can be defined via the `Printer` item of the Tools menu, which is available only for DOS version of *JANA98*. The PC version uses PostScript or raster output. Both of them can be directed to the ports Lpt1 or Lpt2. In the Printer form there is a list of printer drivers, which is important only for the raster output. In the case of the PostScript output there is only one PostScript driver available which is expected to work properly with every PostScript printer.

Figure 28 *The printer settings for the PC version of JANA98.*



Printer

- HP DeskJet 500C or 310/colour
- HP DeskJet 310/320/500/520/520 (mono)
- Epson JX type/80 columns
- Epson JX type/wide carriage
- Epson LQ/colour 80 columns
- Epson LQ/colour wide carriage
- DEC LN03/ln03+(ANSI text mode)
- HP LaserJet III/4L
- HP LaserJet 4/5
- HP PaintJet XL 300

Disabled ☐ Lpt1 ● Lpt2 ☐

PostScript ☐ Raster print ●

2

Preliminary Work

This part describes how to prepare the basic data files for JANA98.

IN THIS CHAPTER:

Next Chapter: Structure Solution and Refinement, page 91.
Previous chapter: Basic Features, page 7.

2
Preliminary Work

This part describes programs *DATRED* and *PRELIM* for preparation of the reflection file and the basic crystal information. This work precedes the structure determination.

- *DATRED* is used for reading of a diffractometer output, making the data reduction, testing the symmetry of the diffraction pattern and transformation of the cell and modulation vector.
- *PRELIM* is used for preparation or changes of the crystal information file `m50` and for creation of the refinement reflection file `m91`.

The cursory overview of the basic programs and their communication is given in page 29.

2.1 Program *DATRED*

Input: diffractometer file or [`m94` and `m95`]
Output: `m95,m94,` SHELX diffractometer file

DATRED (the Data Reduction Program) is used for three basic tasks:

- reading of a diffractometer output
- making the data reduction
- testing the symmetry of the diffraction pattern and making transformation of the cell and modulation vector

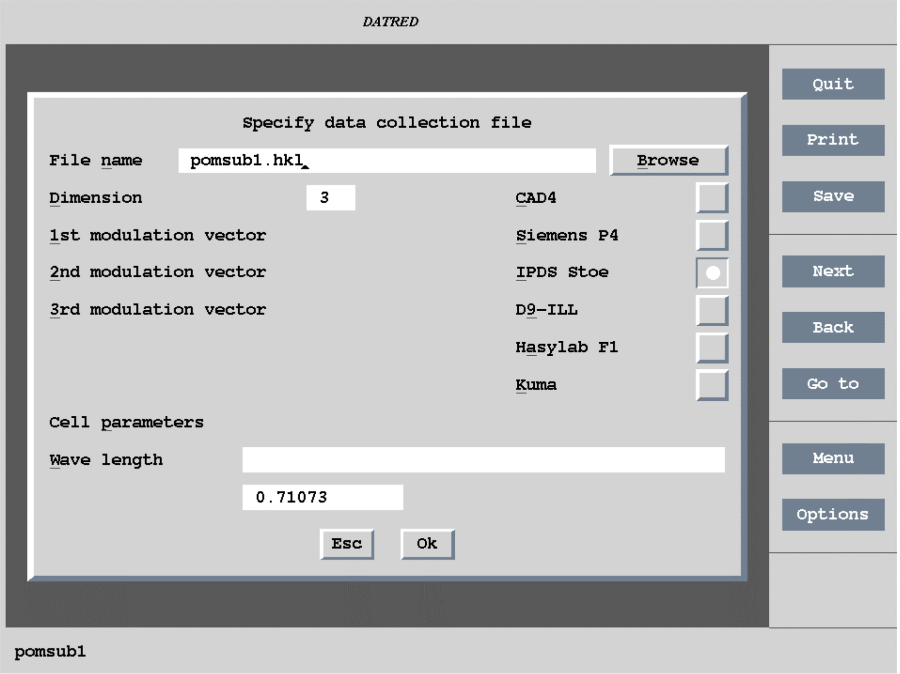
DATRED transforms each supported diffractometer output to the common diffractometer format and saves it in files `m95` and `m94`. These files are used for another tools of *DATRED* and also for creating of the reflection file `m91` by *PRELIM*. If reflections are imported by *PRELIM* (because a diffractometer file is not available) they are converted to `m95` and `m94` to enable *DATRED* tools.

The common diffractometer file can be used for restoring original data. This is for instance used when changing symmetry - the data are restored from `m95` and then a new symmetry is applied. The changes made by *DATRED* do not affect the original data in `m95` as they only change additional coefficients.

2.1.1 Reading of diffractometer files

Figure 29 shows a screen appearing when starting *DATRED* for a job without `m94` and `m95`. The user should select a diffractometer[1], supply a necessary information and start the import.

Figure 29 *The basic window in the case that* `m95` *does not exist*



Specify data collection file

File name: pomsub1.hkl
Dimension: 3
1st modulation vector
2nd modulation vector
3rd modulation vector
Cell parameters
Wave length: 0.71073

CAD4 ☐
Siemens P4 ☐
IPDS Stoe ●
D9-ILL ☐
Hasylab F1 ☐
Kuma ☐

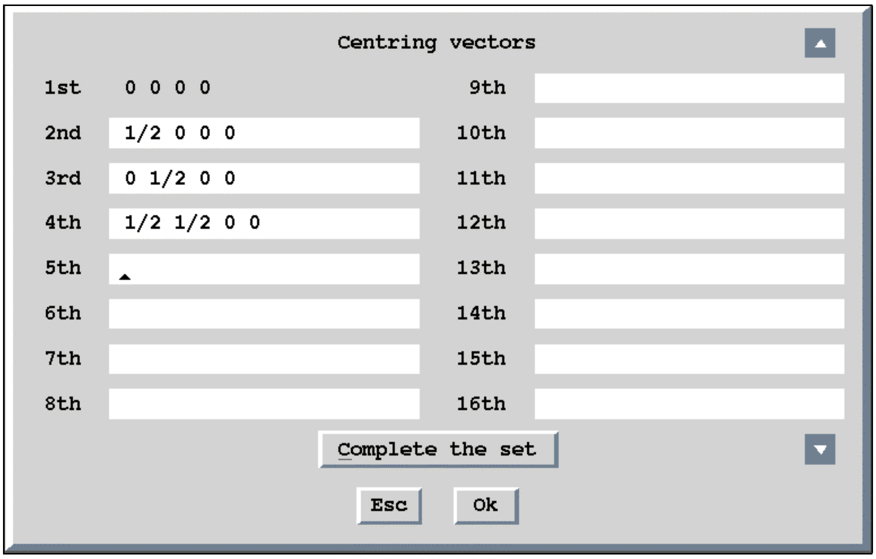
pomsub1

---

[1] The list of the diffractometer types can be extended by request of the users.

The next form is used to define the centring vectors which are necessary to recover (3+d) dimensional indices of modulated crystals from setting diffractometer angles and the orientation matrix. The form is activated only if the structure is modulated and the diffractometer cannot handle (3+d) dimensional indices directly (CAD4[1] and Siemens P4). As the vectors need not to be independent there is the `Complete the set` button for adding vectors derived from the already existing ones. In the example in Figure 30 the 4th line can be added by `Complete the set`.

Figure 30 *The Centring Vectors Form*

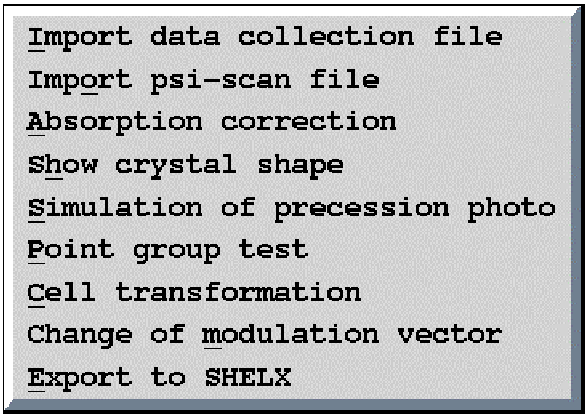


Centring vectors

1st 0 0 0 0
2nd 1/2 0 0 0
3rd 0 1/2 0 0
4th 1/2 1/2 0 0

After quitting the Centring vectors form *DATRED* reads the diffractometer output file, makes the LP corrections and (for some diffractometer types) the correction for the standard reflections decay. When the transformation of the diffractometer file to `m94` and `m95` formal is successfully finished *DATRED* opens the main menu (see Figure 31).

Figure 31 *The main menu of DATRED.*



---

[1] CAD4 data file collected in the `file` mode can be used without using any additional information.

Appending another diffractometer file

With the first item of the main menu of *DATRED* the existing `m95` and `m94` can be overwritten or appended by another data collection file. This tool can be used only for appending files of the same measurement. A procedure for joining data sets with different scales or twin domain numbers is described in chapters page 34 and 72.

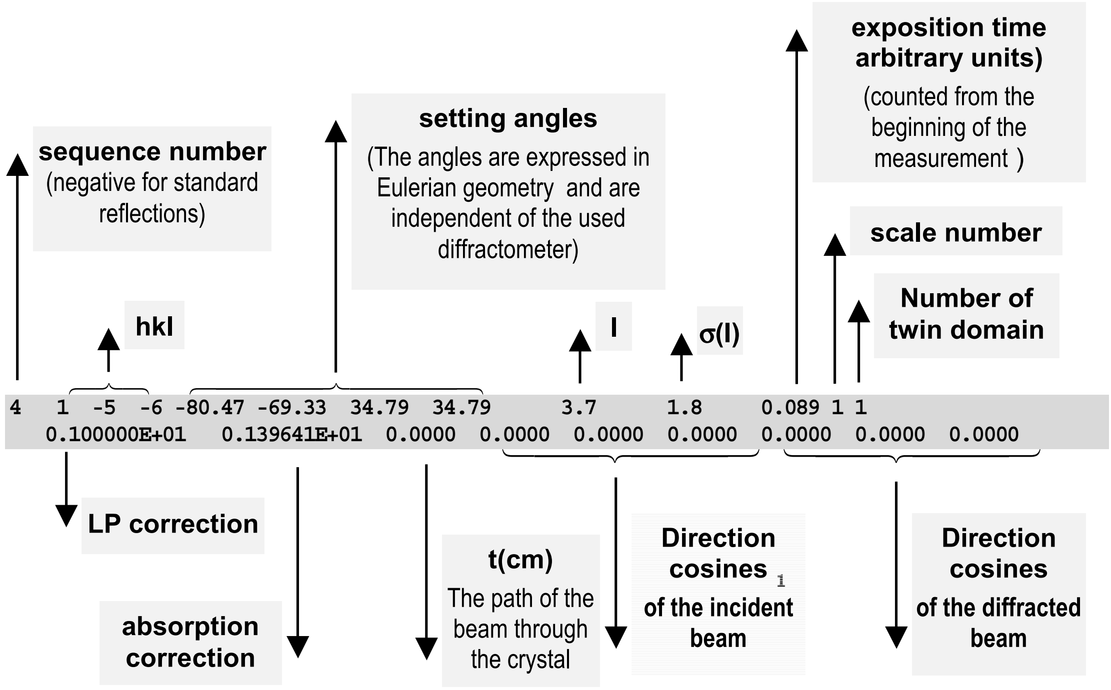
2.1.2 The basic reflection file `m95`

The *basic reflection file* `m95` contains all reflections from the measurement and their LP and absorption correction coefficients. Depending on the way it has been created `m95` can also contain diffractometer angles and direction cosines. `M95` can be created by *DATRED* from the diffractometer output file or by *PRELIM* by importing the reflection file of a different system (SHELX etc.).

`M95` can include joined reflections from several measurements or from several twin domains. The scale factor and the serial number of the twin domain depends on a data set/twin domain the reflection belongs to. The joining of data set from several measurements or twin domains is described in chapters page 34 and 72.

Figure 32 *The Basic reflection File* `m95`

```
  -1   3   0   0   7.95   7.54   7.59   7.46   8253.2     32.2     0.028 1 1
 0.100000E+01   0.100000E+01  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000
  -2   0   3   0  68.07   2.47   7.53   7.46   7538.2     30.8     0.041 1 1
 0.100000E+01   0.100000E+01  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000
  -3   3  -3  -2 -45.24 -48.54  12.77  12.74   1221.2     12.7     0.055 1 1
 0.100000E+01   0.100000E+01  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000
   4   1  -5  -6 -80.47 -69.33  34.79  34.79      3.7      1.8     0.089 1 1
 0.100000E+01   0.100000E+01  0.0018  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000
```



```
4   1  -5  -6 -80.47 -69.33  34.79  34.79      3.7      1.8     0.089 1 1
 0.100000E+01   0.139641E+01  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000  0.0000
```

---

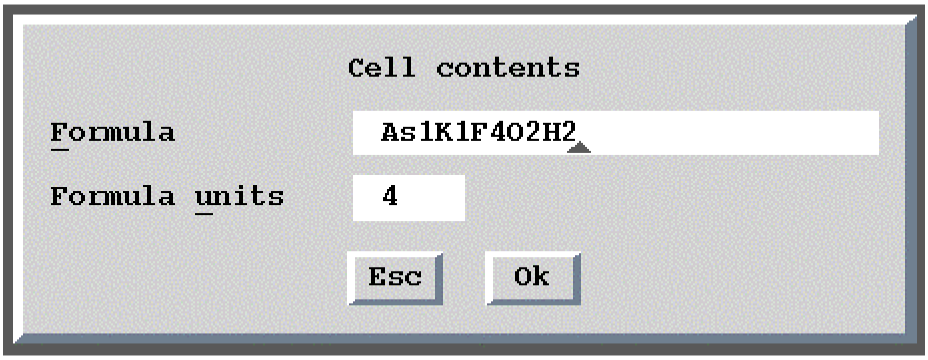
[1] Let us denote the direction cosines in `m95` like D11, D12, D13, D21, D22, D23. Then the relationship to the direction cosines S11, S21, S12, S22, S13, S23 from SHELX diffraction files is the following: S11 = -D11; S21 = D22; S12 = -D12; S22 = D22; S13 = -D13; S23 = D23.
The vector of the incident beam in the JANA system is supposed to be directed from the source to the crystal

2.1.3 Absorption correction

Absorption correction is calculated for a spherical or cylindrical sample or for a general shape defined by the bounding crystal faces. The absorption coefficients are saved in `m95` but the rest of the file remains unchanged so that the absorption correction can be arbitrarily repeated. The program needs the direction cosines to make the correction; in the case of four circle diffractometer they are calculated from the setting angles, for other diffractometer types they must be present in the diffractometer file.

Before calculating the absorption correction the program asks for a chemical formula and number of the formula units in the unit cell. The chemical formula can be entered in an arbitrary case but **a number must separate each chemical element**.

Figure 33 *The Cell Contents Form*



Cell contents

Formula: As1K1F4O2H2

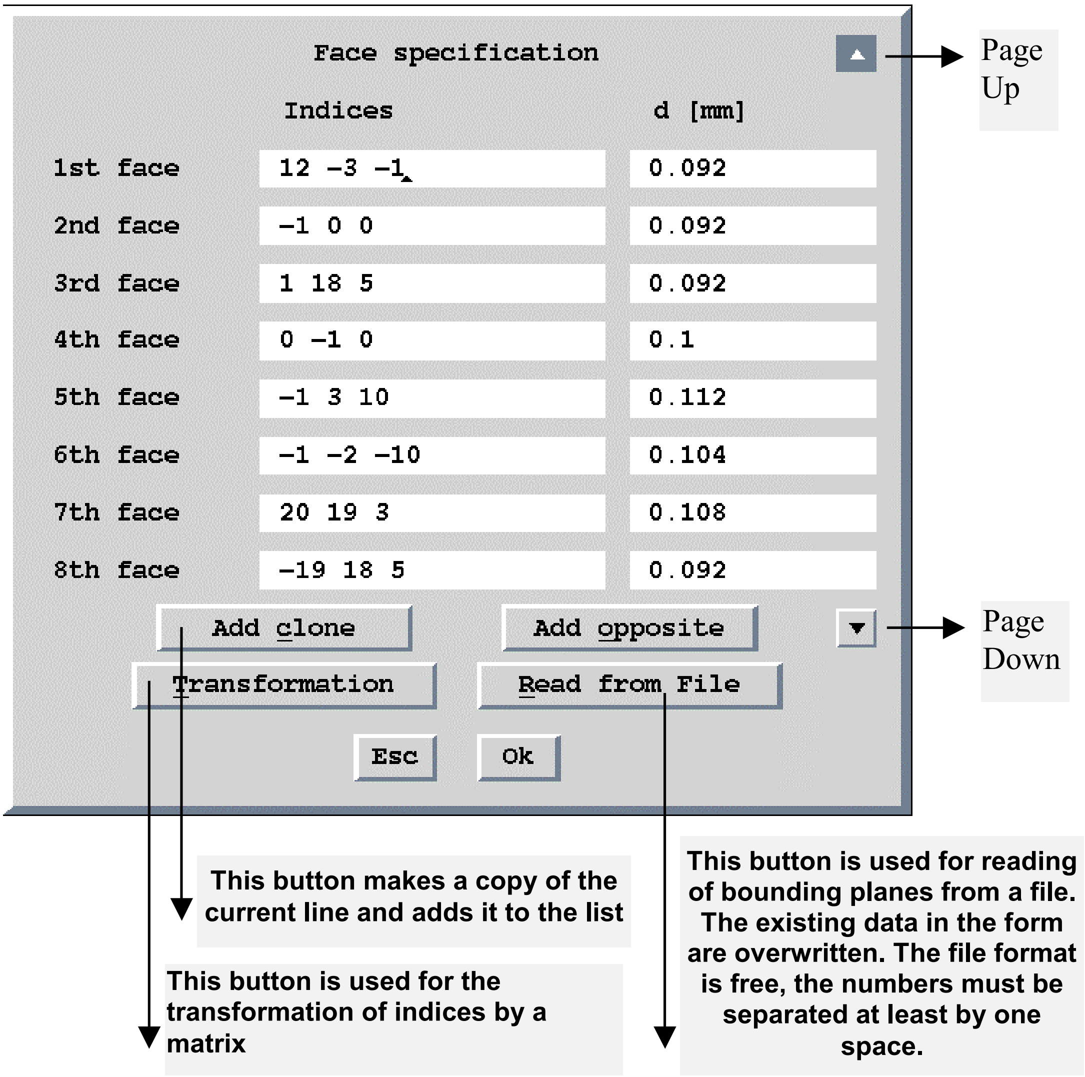
Formula units: 4

Absorption correction for a general shape

The absorption correction for a general shape is calculated by the Gaussian integration method. The accuracy of the absorption correction calculated with a default Gaussian grid “10 10 10” is usually sufficient.

The crystal shape is defined in the `Crystal shape` form in terms of the face indices and their perpendicular distances from an arbitrary common point in the crystal.

Figure 34 *The Crystal Shape Form*



	Indices	d [mm]
1st face	12 -3 -1	0.092
2nd face	-1 0 0	0.092
3rd face	1 18 5	0.092
4th face	0 -1 0	0.1
5th face	-1 3 10	0.112
6th face	-1 -2 -10	0.104
7th face	20 19 3	0.108
8th face	-19 18 5	0.092

This button makes a copy of the current line and adds it to the list

This button is used for the transformation of indices by a matrix

This button is used for reading of bounding planes from a file. The existing data in the form are overwritten. The file format is free, the numbers must be separated at least by one space.

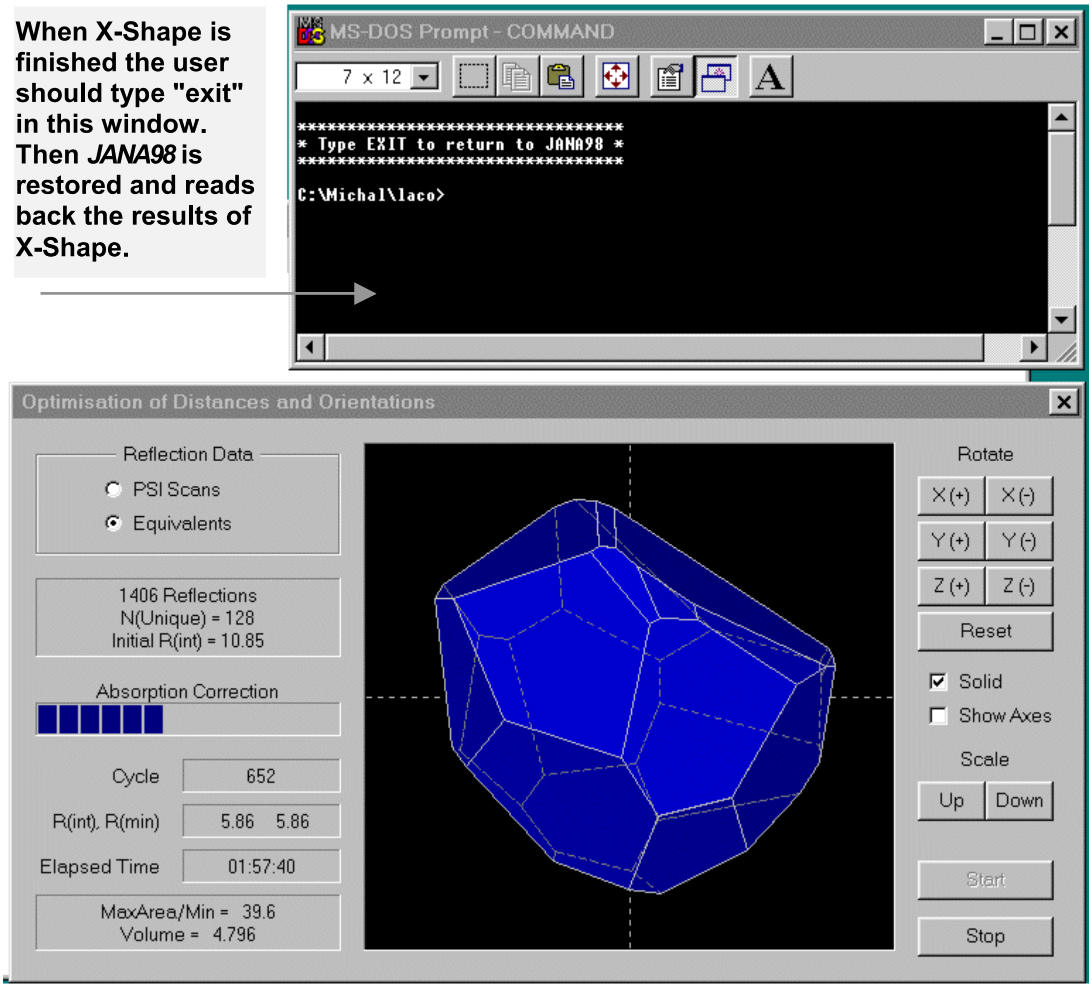
Optimisation of the crystal shape by X-Shape

X-shape is a program of STOE + CIE GmbH for optimisation of the crystal shape. It needs as the input the basic crystallographic information, the list of bounding planes and the file with intensities, direction cosines of equivalent reflections or file with a set of reflections measured by the Ψ-scan.

The DOS version of *JANA98* has an additional item in the main menu of *DATRED* called `Run X-Shape`, which prepares the input files for X-Shape and then swaps *JANA98* in order to allow X-Shape running under windows95/98. When X-Shape is finished *JANA98* restores and reads a new optimised crystal shape so that the user can immediately run the absorption correction.

X-Shape is available in http://stoe.com/.

Figure 35 *Running X-Shape*



When X-Shape is finished the user should type "exit" in this window. Then *JANA98* is restored and reads back the results of X-Shape.

```
**********************************
* Type EXIT to return to JANA98 *
**********************************

C:\Michal\laco>
```

Reflection Data
○ PSI Scans
● Equivalents

1406 Reflections
N(Unique) = 128
Initial R(int) = 10.85

Absorption Correction

Cycle 652
R(int), R(min) 5.86 5.86
Elapsed Time 01:57:40

MaxArea/Min = 39.6
Volume = 4.796

☑ Solid
☐ Show Axes

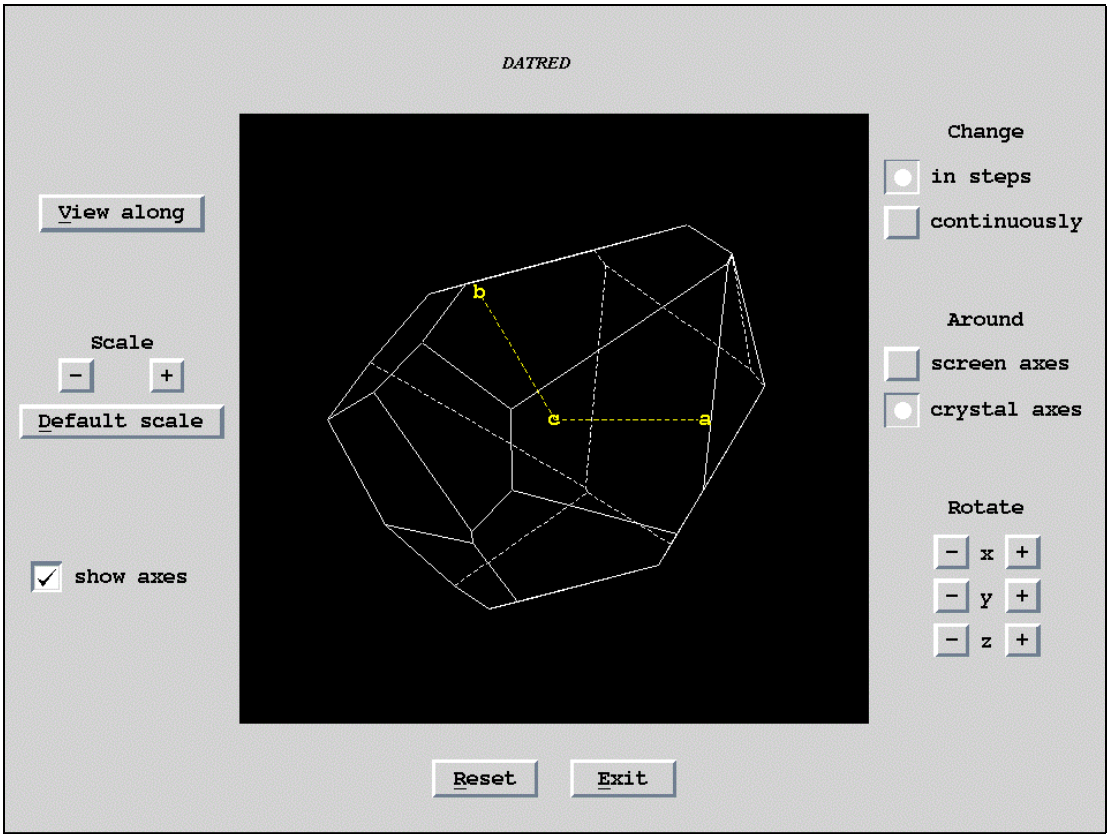
The necessary steps for running X-Shape from* JANA98*:

- First the Point group test has to be executed from the main menu of *DATRED* with the desired point group and its results has to be saved. See page 64 for details about the Point group test.
- The cell contents and optionally also the input crystal faces should be defined in `Absorption correction` in the main menu of DATRED
- X-Shape is executed by `Run X-Shape` item in the main menu of *DATRED*.
- DOS exit command restores *JANA98*. The optimised faces can be either accepted or rejected for a subsequent work.

The Crystal shape viewer

The Crystal shape viewer can examine the crystal shape used for the absorption correction. Before the viewer is started the form showed in page 60 is opened so that the crystal shape can be edited.

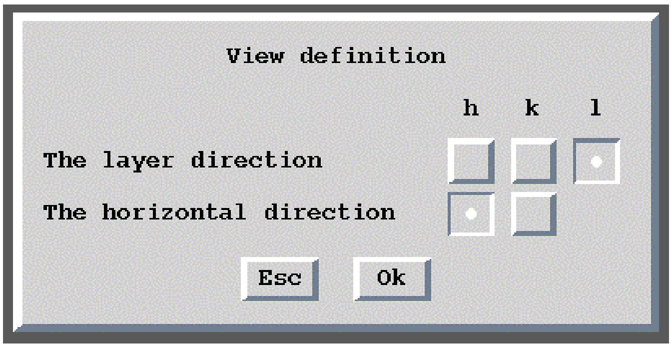
Figure 36 *Crystal Shape Viewer*



2.1.4 Simulation of a precession photo

DATRED calculates a simulated precession photo from reflections from `m95`.

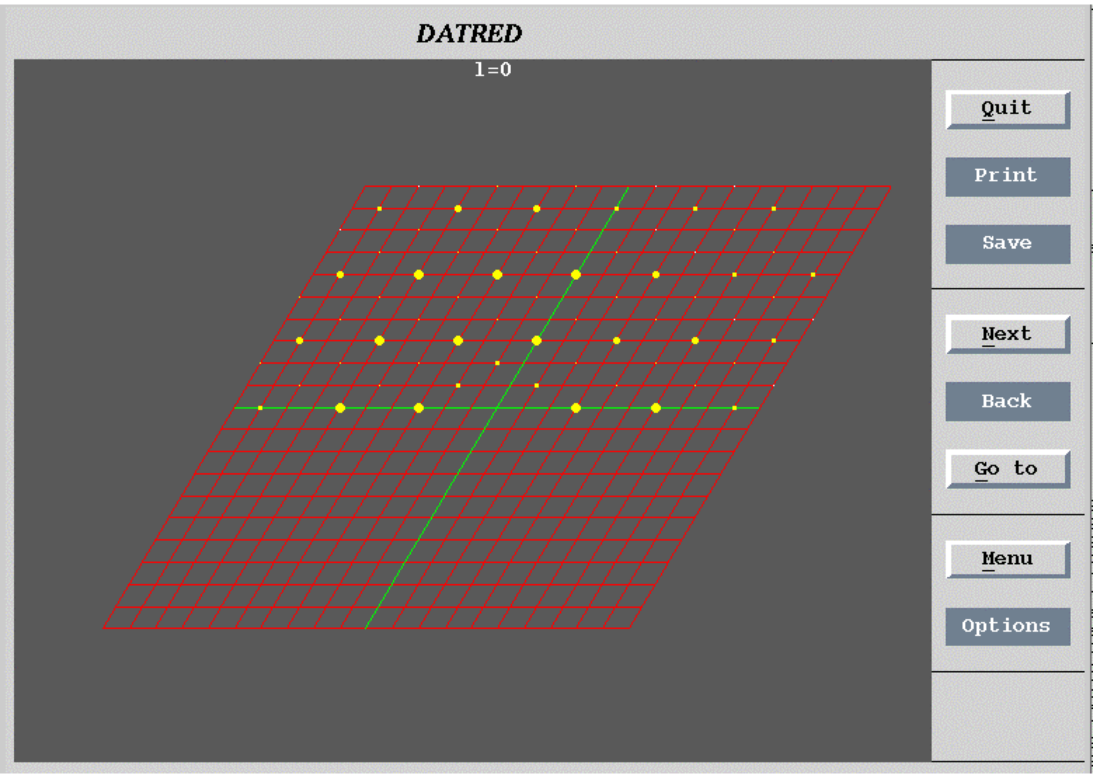
Figure 37 *The View Definition for the Precession Photo*



View definition

	h	k	l
The layer direction	○	○	●
The horizontal direction	●	○	

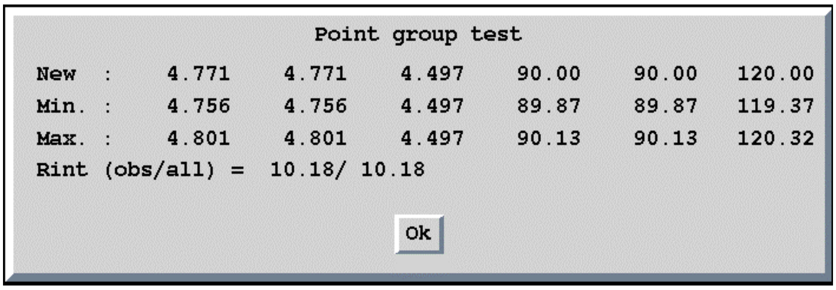
Figure 38 *The Simulation of the Precession Photo*



2.1.5 The Point group test

This tool is used for testing the symmetry of a diffraction pattern. The reflections are averaged according to an interactively chosen point group and then the information showed in the following figure is reported:

Figure 39 *The Point Group Test Results*



```
                      Point group test
New  :     4.771    4.771    4.497    90.00    90.00   120.00
Min. :     4.756    4.756    4.497    89.87    89.87   119.37
Max. :     4.801    4.801    4.497    90.13    90.13   120.32
Rint (obs/all) =  10.18/ 10.18
```

The R_{int} value is calculated for all reflections and for observed reflections with $I > 3\sigma(I)$. The cell parameters are calculated as an average from their values resulting from the transformation by symmetry operations of the tested point group.

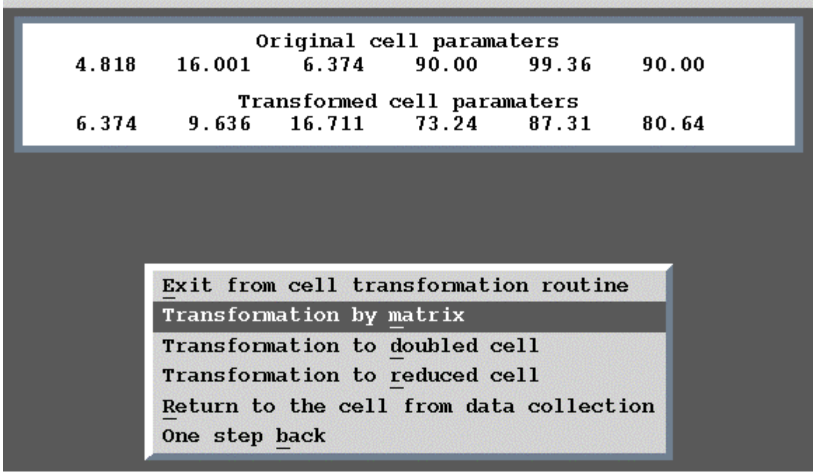
After pressing OK the user can continue with another point group or can quit the test and (optionally) save the results.

2.1.6 Cell transformations

This tool offers various types of cell transformations. The transformation matrix is defined by the following equation:

$$[\mathbf{a}_1' \quad \mathbf{a}_2' \quad \mathbf{a}_3'] = [\mathbf{a}_1 \quad \mathbf{a}_2 \quad \mathbf{a}_3] \begin{bmatrix} T_{11} & T_{12} & T_{13} \\ T_{21} & T_{22} & T_{23} \\ T_{13} & T_{23} & T_{33} \end{bmatrix}$$

Figure 40 *The Cell Transformation Menu*



```
              Original cell paramaters
 4.818   16.001    6.374    90.00    99.36    90.00

             Transformed cell paramaters
 6.374    9.636   16.711    73.24    87.31    80.64
```

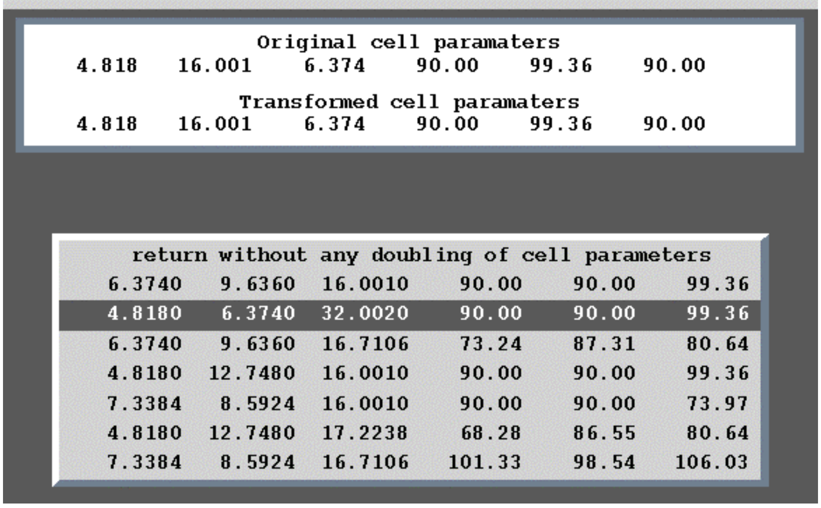
The transformed cell parameters are shown in a small window in the top of the Cell transformation menu. The last transformation can be cancelled by `One step back`. The point `Return to the cell from data collection` cancels all transformations.

If the transformation is accepted it is saved in `m94` and all subsequent reading of `m95` is combined with this operation. The cell parameters in `m50` are also changed. If `m91` already exists its indices are automatically transformed together with coordinates of existing structure model in `m40`.

It should be noted that the point `Return to the cell from data collection` recovers **the last saved** transformation.

The transformation to a doubled unit cell offers all possible doubled cells in order to easily recognise proper centring if it was omitted during the data collection.

Figure 41 *Transformation to Doubled Cell*



```
              Original cell paramaters
 4.818   16.001    6.374    90.00    99.36    90.00

             Transformed cell paramaters
 4.818   16.001    6.374    90.00    99.36    90.00
```

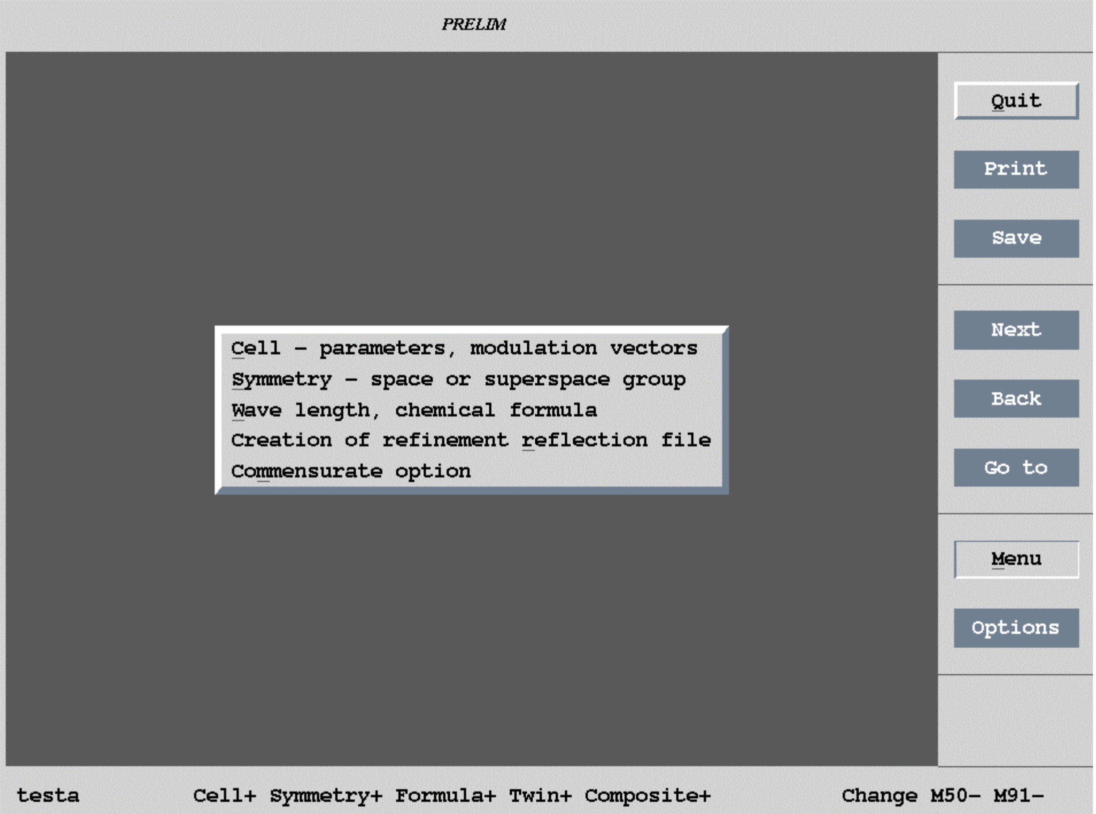
```
     return without any doubling of cell parameters
   6.3740    9.6360   16.0010     90.00     90.00     99.36
   4.8180    6.3740   32.0020     90.00     90.00     99.36
   6.3740    9.6360   16.7106     73.24     87.31     80.64
   4.8180   12.7480   16.0010     90.00     90.00     99.36
   7.3384    8.5924   16.0010     90.00     90.00     73.97
   4.8180   12.7480   17.2238     68.28     86.55     80.64
   7.3384    8.5924   16.7106    101.33     98.54    106.03
```

2.2 Program *PRELIM*

Input: `m94`,`m95` or foreign reflection file (SHELX etc.), [`m50`]
Output: `m50`,`m91`,[`m95`],`pre`
M50 control keywords: none

2.2.1 Description of *PRELIM*

Figure 42 *The Main Menu*



```
testa          Cell+ Symmetry+ Formula+ Twin+ Composite+          Change M50- M91-
```

PRELIM prepares or changes the *crystal information file* `m50` and the *refinement reflection file* `m91`. In the case when `m50` does not exist the crystal information prepared by *DATRED* and stored in `m94` is used to initialise *PRELIM*. `M91` is created from the *basic reflection file* `m95` by applying of correction factors calculated in *DATRED* and stored in `m95` to measured intensities (Lp and absorption). During this process all systematically absent reflections are deleted. Optionally the program makes averaging of the reflections by the used symmetry. If `m95` doesn't exist, it should be created `by Import of reflections`.

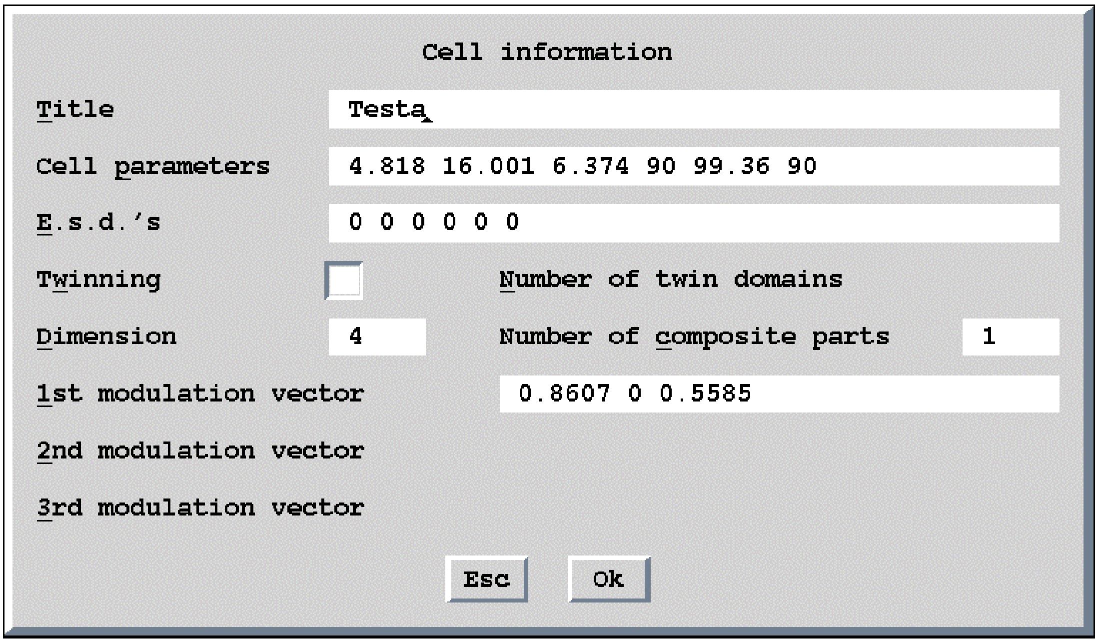
The status line of *PRELIM* contains flags indicating if the corresponding information is known. For instance the flag “`Cell-`" indicates that information listed in the Cell form of *PRELIM* is incomplete or unknown. *PRELIM* checks the completeness of the crystal information before quitting.

The flags in the status line are not independent. For instance if the Cell information is edited and the new cell parameters are not consistent with the symmetry the flag “Symmetry” is set to “-“. The flag “Change m50- m91-” indicates whether the basic crystal information and/or reflection file m91 have been modified.

2.2.2 The Cell form

This form is used for definition of the cell parameters, q-vectors and number of twinning and composite parts.

Figure 43 *The Cell Form*



Cell information

Title: Testa

Cell parameters: 4.818 16.001 6.374 90 99.36 90

E.s.d.'s: 0 0 0 0 0 0

Twinning ☐ Number of twin domains

Dimension: 4 Number of composite parts: 1

1st modulation vector: 0.8607 0 0.5585

2nd modulation vector

3rd modulation vector

2.2.3 The Symmetry form

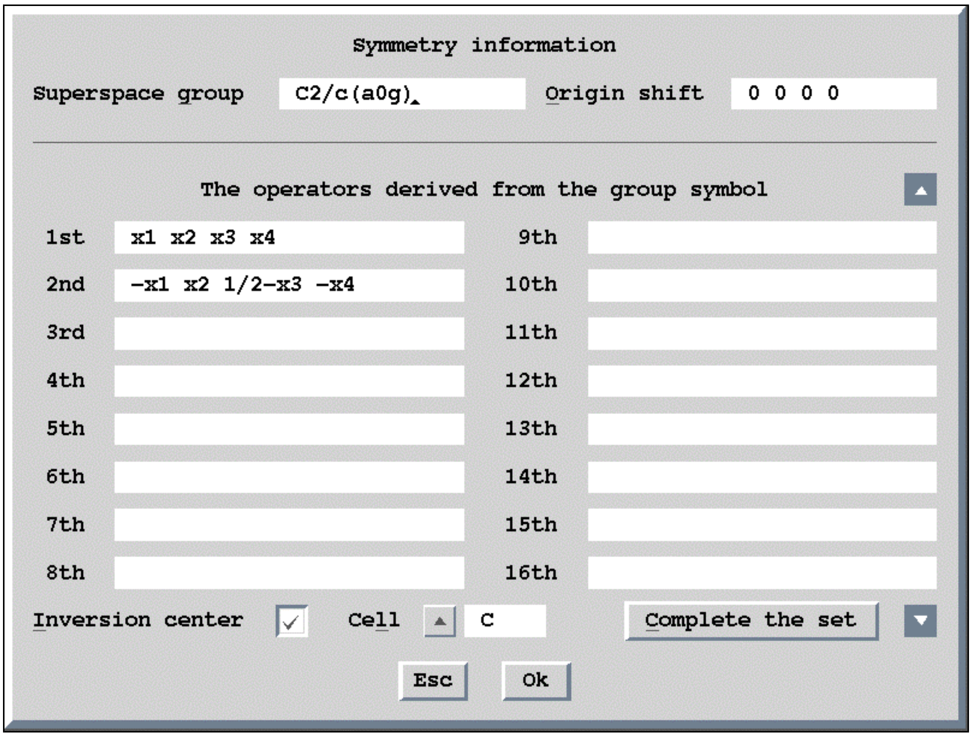
This form is used for entering the symmetry information. This can be done by two ways:

- By entering the (super)space group symbol in the upper part of the form. After finishing the text box by pressing the TAB button or by selecting other text box or by pressing a button the lines in the lower part of the form are automatically filled in with the generated symmetry operators and the following text appears in the form: “`The operators derived from the group symbol`”.
- By entering symmetry operators in the lower part of the form. After pressing the `Complete the set` button the operators are completed to form a (super)space symmetry group. In the case that operators correspond to a standard setting of some (super)space group the text box in the upper part of the form is filled in with that symbol and the following text appears in the form: “`The group symbol derived from the operators`”.

The flexibility of this tool can be appreciated with the tasks like adding/removing of the symmetry center or changing the centring of the cell. In both cases the user makes changes in the lower part of the form and new symmetry operators and a (super)space group symbol are derived simply by pressing `Complete the set`. It is also possible to remove some symmetry operators by pressing Ctrl-Y in the corresponding text box or to edit them. Pressing `Complete the set` again finishes the work.

It should be noted that for changing the (super)space group of an existing structure model to a subgroup there is more complex tool available in Tools-> `Generating of subgroup structure`, see page 45.

Figure 44 *The Symmetry Form*



Symmetry information

Superspace group C2/c(a0g)

Origin shift 0 0 0 0

The operators derived from the group symbol

1st x1 x2 x3 x4

2nd -x1 x2 1/2-x3 -x4

Inversion center ☑

Cell C

The (super)space group symbols

The (super)space group symbols can be entered either in the short form or as Hall's symbols (Hall(1981)) by the following rules:

- The coding is case insensitive (P1 is the same as p1)
- The bar over a character is coded as minus (P□ is coded as P-1)
- Superscripts and subscripts are not distinguished (P2$_1$/c is coded as P21/c)
- The generators of the Hall's symbols are separated by semicolons (P□$_c$□$_c$ is coded as P-6c;-2c)

Four-dimensional superspace groups

The symbol of a four-dimensional superspace group can be expressed in both Hall's notation and the notation from the International Tables vol. C 9.8. The α,β and γ are coded as alpha, beta, gamma and can be arbitrarily abbreviated to the shortest form a, b, g. Both the older and newer notation of the superspace groups are allowed.

Example: `W:Pban:qq-1` or `Pban(1/21/2gamma)qq`

The same superspace group can be expressed using Hall's notation.

Example: `W:-p-2xb;-2ya:qq` or `-p-2xb;-2ya(1/21/2gamma)qq`

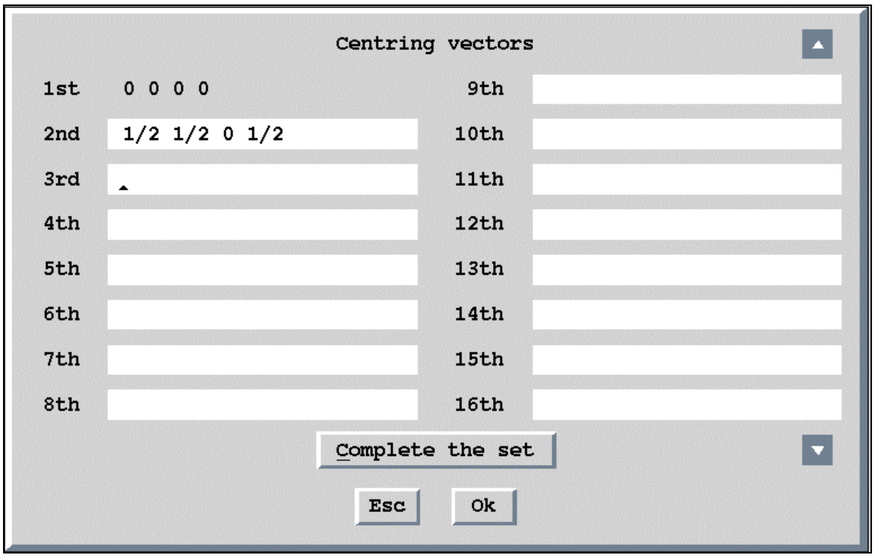
Permutation of basic vectors

For the monoclinic and orthorhombic crystal systems *PRELIM* enables all possible permutations of the basic vectors **a**, **b**, **c**. The sequence of the irrational components of the modulation vector is therefore not necessarily (αβ0) or (00γ) as used by De Wolff, Janssen & Janner(1981) .

Non-standard centring

PRELIM enables a non-standard centring of the unit cell, which is useful for example if the form of the modulation vector leads to a centring in the superspace. If the centring symbol of the cell in the Cell form is chosen as "X" another form is opened for definition of the centring vectors:

Figure 45 *The Centring Vectors Form*



1st 0 0 0 0

2nd 1/2 1/2 0 1/2

Transformation of q_r

PRELIM checks the consistency between the rational part of the q vector entered in the Cell form and the one following from the superspace group symbol. If the $\mathbf{q}_r$ in the Cell form is zero and the derived $\mathbf{q}_r$ from the superspace group symbol is a non-zero the superspace group is transformed in order to get $\mathbf{q}_r = (0,0,0)$.

3d+2 and 3d+3 symmetry

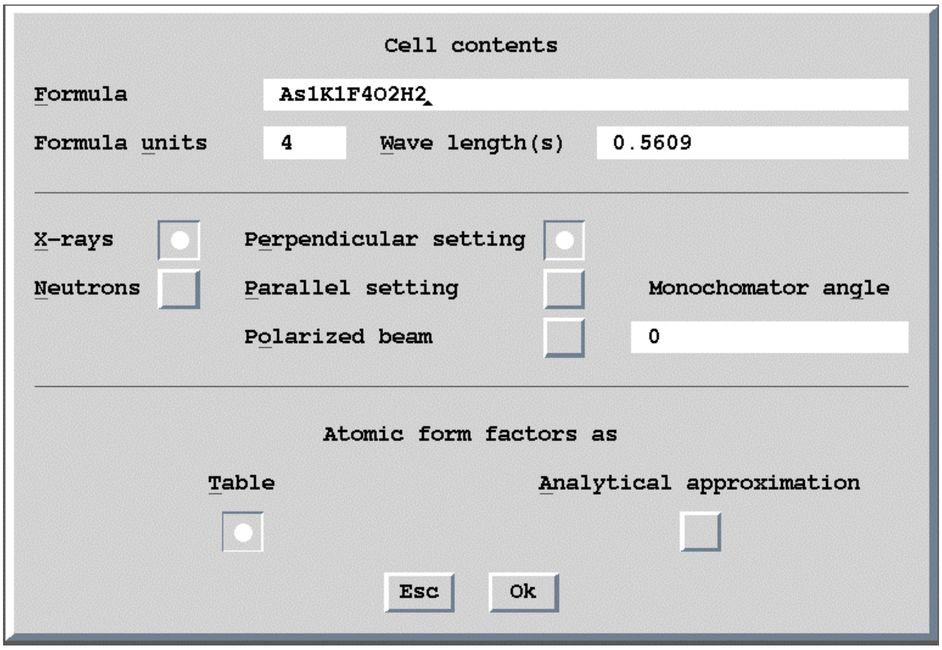
In this case the user enters to the symmetry form only 3d space group symbol. Then *PRELIM* opens a form for submitting remaining information.

2.2.4 Wave length and chemical formula

This form is used to enter the chemical formula, wavelength and atomic form factors.

- The chemical formula is case insensitive; the numbers are used as delimiters of chemical elements. The information about chemical elements is saved in `m50` in the same order they appears in the chemical formula. The sequence number of chemical elements is then assigned to the atoms of the structure model in `m40`. From this the important rule follows that ***if there is already some structure model in `m40`, the order of the chemical elements in the chemical formula must not be changed***.
- For case of X-ray data both the parallel and perpendicular setting of the monochromator are available. Data collected with a source having the polarised beam can be handled as well. The atomic form factors can be prepared from a built-in table or from analytical expression. The atomic form factors for ions are not available and the user should type them directly to `m50` (see the `formtab` keyword in Figure 55, page 78).

Figure 46 *The Atoms Form*



Cell contents

Formula: As1K1F4O2H2

Formula units: 4

Wave length(s): 0.5609

X-rays ●

Neutrons ☐

Perpendicular setting ●

Parallel setting ☐

Polarized beam ☐

Monochomator angle: 0

Atomic form factors as

Table ●

Analytical approximation ☐

2.2.5 Import of reflections

Import of reflections by *PRELIM* is used in cases that data cannot be imported from a diffractometer file. Typical case is a structure in the SHELX format. Like in *DATRED* (page 55) the reflection file is also transformed to the common format and saved as `m94` and `m95`. The information in `m94` and `m95` prepared by *PRELIM* is necessarily reduced but they can still be used for most tools in *DATRED* except for corrections. The difference between the two approaches is shown in the following two figures

Figure 47 *The data reduction scheme starting from the diffractometer file*

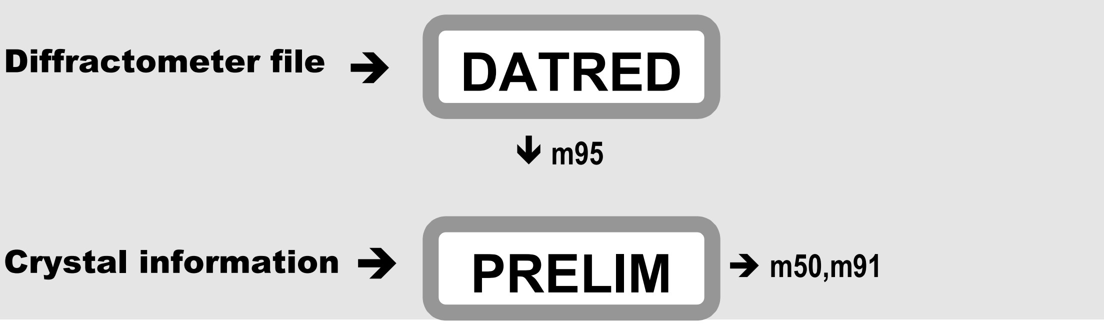
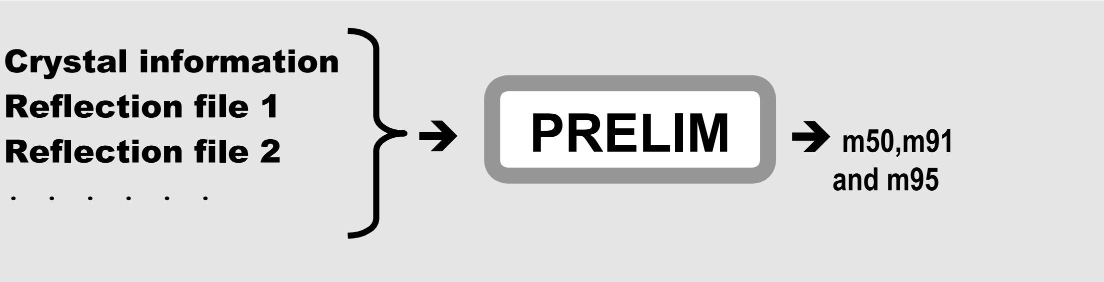


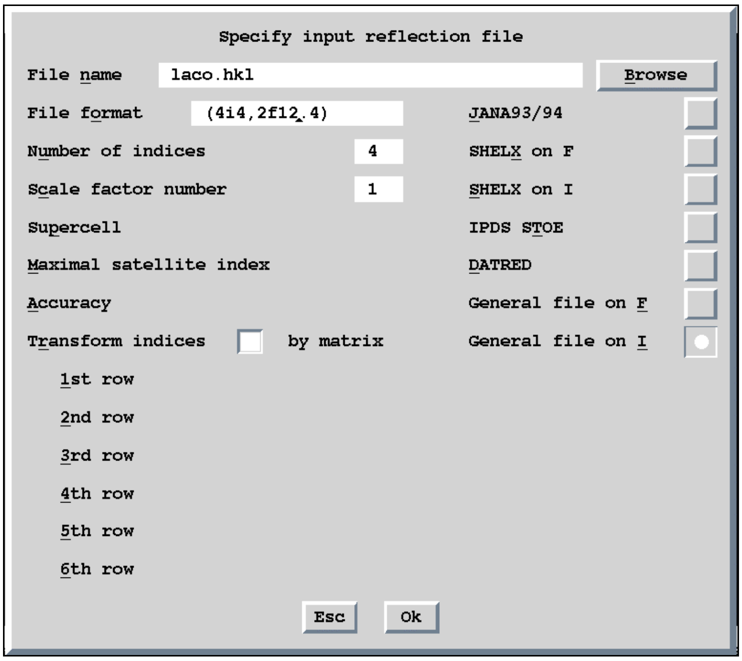
Figure 48 *The data reduction scheme starting from other reflection file*



Importing a single data set

If `m95` does not exist *PRELIM* opens immediately a form shown in the next figure. In this example the user is going to read a reflection file `laco.hkl` with four integer indices followed by the intensity and its e.s.d written in the FORTRAN format `(4i4,2f12.4)`. The format can be changed, a free format (*) is allowed as well. The scale factor in the case of single data set should be one. If the `Transform indices` checkbox is activated the user has possibility do define a matrix which will be applied to the indices before saving to `m95`. After quitting the form the reflection file is read in and `m94` and `m95` are created.

Figure 49 *The Import form*



File name: laco.hkl

File format: (4i4,2f12.4)

Number of indices: 4

Scale factor number: 1

Transform indices ☐ by matrix

JANA93/94 ○

SHELX on F ○

SHELX on I ○

IPDS STOE ○

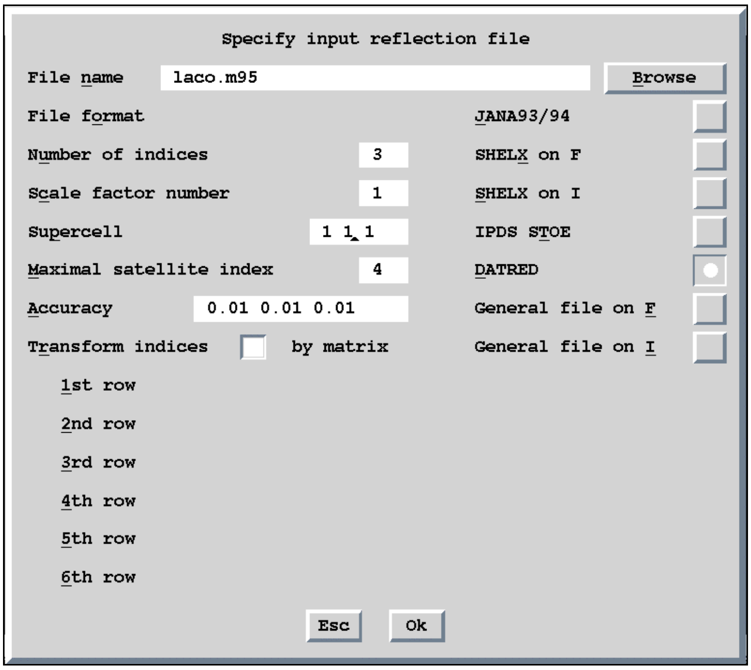
DATRED ○

General file on F ○

General file on I ●

In this first example the inserted reflections have 4 indices and the structure is 3+1 dimensional. In the case the input file has only 3 indices but the structure is still 3+1 dimensional the dialogue takes form shown in Figure 50 with textboxes for definition of the supercell, maximal satellite index and desired accuracy. The reflections that cannot be indexed with four indices within the limit of the desired accuracy are listed after the import.

Figure 50 *The Import form if the* 4^{th} *Index has to be generated*



File name: laco.m95

Number of indices: 3

Scale factor number: 1

Supercell: 1 1 1

Maximal satellite index: 4

Accuracy: 0.01 0.01 0.01

Transform indices ☐ by matrix

JANA93/94 ○

SHELX on F ○

SHELX on I ○

IPDS STOE ○

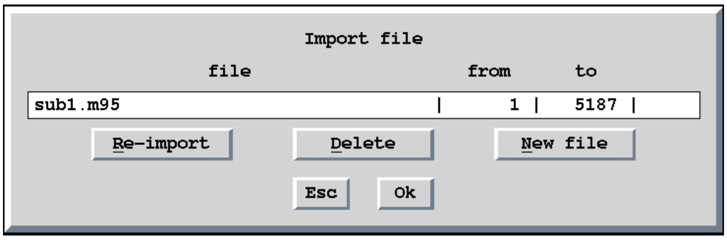
DATRED ●

General file on F ○

General file on I ○

Another possibility is starting the import tool when `m95` already exists. In this case *PRELIM* opens a form in Figure 51 enabling to repeat the import of the same file, delete reflections previously imported from the highlighted file or import a new file. The new file is appended to `m95`. `Re-import` opens the Import form already filled in with the information entered in the previous session.

Figure 51 *The Import list for single data set.*



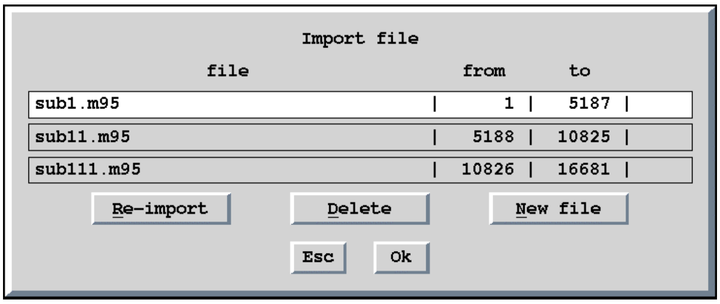
Import file

file	from	to
sub1.m95	1	5187

Importing multiple data sets

With `New file` (Figure 51) several import files can be read and appended to m95. The Import list with several files already imported is in Figure 52.

Figure 52 *The Import list for multiple data sets.*



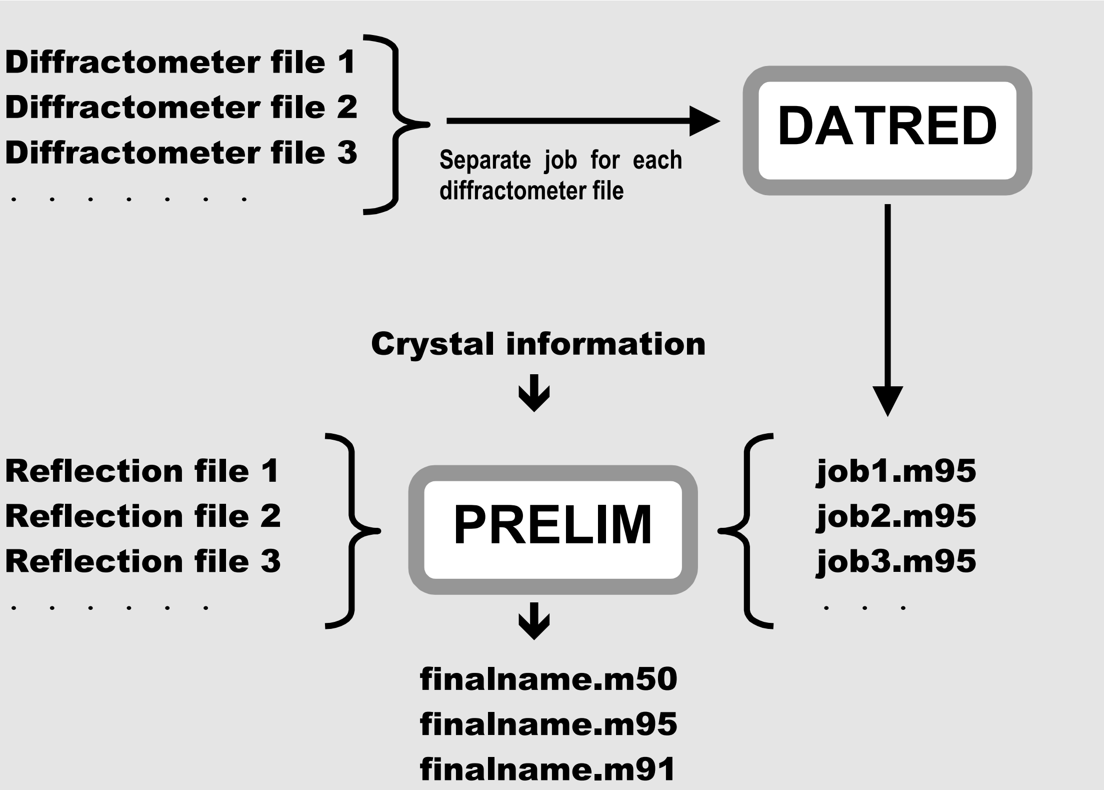
Import file

file	from	to
sub1.m95	1	5187
sub11.m95	5188	10825
sub111.m95	10826	16681

`M95` prepared by *DATRED* from a diffractometer file and data imported by *PRELIM* **cannot** be appended. When joining data sets from several measurements the user should

1. Process each data set by *DATRED*, each of them with a unique job name. The result is the set of `m94` and `m95` named `name1`, `name2`, ….
2. Start *PRELIM* with a definitive job name and import files `name1`, `name2`, … with the Import tool. An individual scale factor can be assigned to each data set.

Figure 53 *Joining multiple data sets*



Importing data sets of several twin domains

Before the import the twin matrices must be defined in `m50` through the Twin option of *PRELIM*. The importing of data sets collected from several twin domains of the same crystal is then an analogy of the procedure described in the previous paragraph. The data sets can be imported with the same scale number. All reflections which can be indexed in the first domain are automatically labelled as belonging to this domain; with the rest of reflections the same procedure is applied with respect to the second, third etc. domain.

In the case when the twinning leads to a diffraction pattern composed from several not fully overlapped (3+d) dimensional lattices the domain to which a particular data set belongs must be specified explicitly.

2.2.6 The Twin option

For a twin the user should first define number of twin domains in the Cell form (see Figure 43). Then the twinning matrices can be defined via the `Twin option` of the main menu. The maximal number of twin domains is 18[1].

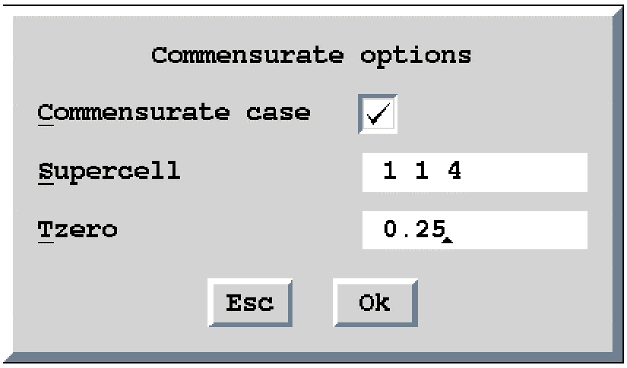
The twinning matrices are applied to the hkl indices defined as row vectors.

The refinement techniques used for various types of twins are discussed in 255.

2.2.7 The Commensurate option

The following form is used for setting of options for commensurate structures.

Figure 54 *The Commensurate Options*



Commensurate options

Commensurate case ☑

Supercell 1 1 4

Tzero 0.25

---

[1] See page 108 for explanation about scale parameters.

2.2.8 The Basic crystal information file `m50`

The first part of `m50` contains information about cell parameters, symmetry operators, form factor tables etc. **The second part** contains control commands for basic programs of *JANA98*.

The user usually doesn't need editing of `m50` in a text editor. The first part of `m50` is prepared interactively by *PRELIM* and the user interface for the second part is the SetCommands Tool (see page 39).

Both parts of `m50` contain keywords followed by one or more values. The keywords are case insensitive. **The second part** of `m50` is created by the following rules:

- The command section for a program starts with the program name and ends with the keyword `end`.
- The command consists of a keyword followed by one ore more numerical values written in a free format.
- Several commands in one line are permitted.
- The length of the command line is limited to 80 characters.
- The commands are case insensitive and their order inside the section is arbitrary.
- The keywords can be shortened until they are unique.
- The line starting with "*" or the part of the line starting with "!" is regarded as a comment.
- The commands between the `end` keyword and beginning of the next command section are not interpreted

The syntax of the **first part** is summarised in Table 9. The command of the basic programs of *JANA98* will be explained in the corresponding chapters.

Figure 55 *The basic crystal information file* `m50` *- Part I*
The commands are explained in Table 9.

```
title Testa
cell 4.818 16.001 6.374 90 99.36 90
lambda 0.5609 radtype 1 lpfactor 1 monangle 0
esdcell 0 0 0 0 0 0
ndim 4
ncomp 1
qi 0.8607 0 0.5585
qr 0 0 0
spgroup C2/c(a0g)  15  22
lattice C
centro
symmetry     x1     x2     x3     x4
symmetry    -x1     x2 1/2-x3    -x4
unitsnumb 4
atom As
atweight 74.922 dmax 3 formula 1
f' 0.276 f" 1.331
fneutron 6.58
formtab 56
   33.000   32.278   30.491   28.301   26.218   24.386   22.739   21.196
   19.722   18.313   16.976   15.717   14.540   13.451   12.454   11.552
   10.744   10.030    9.403    8.858    8.386    7.978    7.626    7.320
    7.053    6.817    6.606    6.415    6.239    6.076    5.922    5.774
    5.633    5.497    5.364    5.235    5.109    4.986    4.867    4.750
    4.637    4.527    4.420    4.317    4.217    4.120    4.027    3.938
    3.852    3.770    3.692    3.617    3.545    3.477    3.412    3.351
atom K
```

. . . .

```
atom O
atweight 15.999 dmax 3 formula 2
f' 0.006 f" 0.004
fneutron 5.803
formtab 56
    8.000    7.798    7.246    6.472    5.623    4.808    4.089    3.489
    3.006    2.628    2.337    2.115    1.946    1.816    1.715    1.634
    1.568    1.512    1.463    1.419    1.377    1.337    1.298    1.260
    1.221    1.183    1.145    1.108    1.070    1.033    0.997    0.961
    0.926    0.891    0.857    0.824    0.792    0.761    0.731    0.702
    0.674    0.647    0.621    0.597    0.573    0.551    0.530    0.509
    0.490    0.472    0.455    0.439    0.424    0.410    0.397    0.384
atom H
atweight 1.008 dmax 3 formula 2
f' 0 f" 0
fneutron -3.739
formtab 56
    1.000    0.960    0.854    0.713    0.568    0.438    0.331    0.248
    0.184    0.138    0.103    0.078    0.060    0.046    0.036    0.028
    0.022    0.018    0.014    0.011    0.009    0.007    0.006    0.005
    0.005    0.004    0.004    0.003    0.003    0.003    0.003    0.003
    0.003    0.003    0.003    0.003    0.003    0.003    0.003    0.003
    0.003    0.003    0.003    0.003    0.003    0.003    0.003    0.003
    0.003    0.003    0.003    0.003    0.003    0.003    0.003    0.003
noofref 4351
slimits 0.362376 0.453337 0.520215 0.574811 0.622404 0.665116 0.712413 0.91344
flimits 43.2 70 119.9 188 311 544.6 1136.6 56184.4
end
******************************************************************************
```

Figure 56 *The basic crystal information file* `m50` *- Part II*
The commands are explained together with description of the basic programs.

`refine`	**The beginning of the section for program *REFINE***
`first sample`	**title of the section**
`cycles 3`	**control commands**
`iext 1 itypex 1 idistr 2`	**control commands**
`restric o1* 1`	**control commands**
`end`	**The end of the section for program *REFINE***
`xxxxxxxxxxxxxxx`	**not interpreted**
	not interpreted
`xxxxxxxxxxxxxxx`	**not interpreted**
`fourier`	**The begin of the section for program *FOURIER***
	title (missing, but the line cannot be used for commands)
`mapt 2`	**control commands**
`* center na2`	**comment**
`scope 0 1 0  ! This is a comment`	**control commands + comment**
`or 2413`	**control commands**
`x4lim 0 1 0.05`	**control commands**
`end`	**The end of the section for program *FOURIER***
`refine`	**This second occurrence of the section for *REFINE* will be ignored**
`cycles 18 damp 0.25`	
`end`	

Table 9 *The Keywords in the First Part of* `m50`

keyword	Syntax + explanation
atom	**atom** `chemical_type` Introduces a section for a chemical element. The keywords between this header and the keyword **formtab** concern this chemical type. The order of chemical types in `m50` is defined by order of atoms in the chemical formula given in *PRELIM* (see page 71). Chemical types of atoms in `m40` are defined by the sequence number the chemical type has in `m50`.
atweight	**atweight** `atomic_weight`
cell	**cell** `a b c alpha beta gamma` Cell parameters in angstroms and degrees
centro	**centro** If present, the structure is centrosymmetric.
commen	**commen** `na nb nc` Definition of the supercell for the commensurate refinement.
dmax	**dmax** `distances_limit` Sets the maximal limit (Å) for calculation of distances for this chemical type.
esdcell	**esdcell** `esda esdb esdc esdalpha esdgamma esdbeta` The e.s.d's of cell parameters. This information is not used in the current version of *JANA98*.
f'	**f’** `fprime` The anomalous dispersion term f’.
f"	**f”** `fdoubleprime` The anomalous dispersion term f”.
flimits	**flimits** `flim1 flim2 . . . . flim8` Limits for reflection groups sorted by |F|. Thy are used for the calculation of the R statistics by *REFINE* (see page 261).
fneutron	**fneutron** `scatlength` The scattering length for neutrons. It is always present in `m50` but it is only used if the neutron radiation is selected by the user (see page 71).

formtab	**formtab** `number_of_values`
	`val1  val2 . . . .  val8`
	`val9  val10 . . . . val16`
	`. . . . . . . . . . valn`
	Atomic form factors for X-ray. They are always present in `m50` but they are only used if the X radiation is selected by the user (see page 71).

formula	**formula** `number` The number of atoms of this chemical type in the formula unit.

Table 9 *The Keywords in the First Part of* `m50` *(Continued)*

lambda	**lambda** `wawe_length` The wave length. If the number is negative its sign is changed, so that the older type of `m50` where the negative wavelengths were used for the neutron radiation can be directly used.
lattice	**lattice** `symbol` The cell centring symbol. In the case of the non-standard centring X the the list of the centring vectors has to be defined by the **lattvec** command.
lattvect	**lattvect** `vt1 vt2` … `vt`$_{ndim}$ Non-primitive centring vectors for the case of the non-standard centring. Each vector is introduced by the **lattvec** command.
lpfactor	**lpfactor** `number` 1 for perpendicular setting of a monochomator 2 for parallel setting of a monochromator 3 for polarised beam (synchrotron, neutron) This option is prepared for a future use in Rietveld's refinement.
ncomp	**ncomp** `number` The number of the composite parts (1 for non-composite structure).
ndim	**ndim** `number` The number of dimensions (3 for standard structure).
noofref	**noofref** `number` The number of reflections in `m91` .
qi	**qi** `alpha beta gamma` The irrational part of the q vector.
qr	**qi** `alpha abeta gamma` The rational part of the q vector.
radtype	**radtype** `number` The radiaton type. 1 for X-rays, parallel setting; 2 for neutrons.
sgshift	**sgshift** `sh1 sh2` … `shndim` Origin shift of the structure according to the conventions in the International tables.

Table 9 *The Keywords in the First Part of* `m50` *(Continued)*

slimits	**slimits** `slim1 slim2 . . . . slim8` Limits for reflection groups sorted by $\sin\theta/\lambda$. Thy are used for calculation of the R statistics by *REFINE* (see page 261).
spgroup	**spgroup** `symbol number1 number2` The space group symbol. `Number1` is the space group number. `Number2` specifies the crystal system: 1 triclinic system 12 monoclinic system with monoclinic angle α 22 monoclinic system with monoclinic angle β 32 monoclinic system with monoclinic angle γ 3 orthorhombic system 4 tetragonal system 5 rhombic system 6 hexagonal system 7 cubic system
symmetry	**symmetry** `x1 x2 x3 . . .` The symmetry operation. The symmetry operations generated by the inversion center are not listed.
title	title
twin	**twin** `ntw` $T^{2}(11)$ $T^{2}(12)$ $T^{2}(13)$ `. . . . . . . . . . .` $T^{2}(31)$ $T^{2}(32)$ $T^{2}(33)$ `. . . . . . . . . . .` $T^{ntw}(11)$ $T^{ntw}(12)$ $T^{ntw}(13)$ `. . . . . . . . . . .` $T^{ntw}(31)$ $T^{ntw}(32)$ $T^{ntw}(33)$ The twinning matrices. `ntw` is the number of twin domains. $T^{n}(ij)$ is an element of a 3x3 twinning matrix. Real numbers are allowed. The first (unit) matrix is not present.
tzero	tzero `number` The value of the internal coordinate `t` for a commensurate refinement.
unitsnumb	**unitsnumb** `number` The number of chemical units in the elementary cell.

wmatrix	**wmatrix**
	`W2(11)        W2(12) . . . . . W2(1,ndim)`
	`. . . . . . . . . . . . . . . . . . . . . . . . .`
	`W2(ndim,1)    W2(ndim,2) . . . W2(ndim,ndim)`
	`. . . . . . . . . . . . . . . . . . . . . . . . .`
	`Wncomp(1,1) Wncomp(1,2). . Wncomp(1,ndim)`
	`. . . . . . . . . . . . . . . . . . . . . . . . .`
	`Wncomp(ndim,1) Wncomp(ndim,2). . Wncomp(ndim,ndim)`
	
	The composite matrices. `ndim` is number of dimension of the structure, `ncomp` is number of composite parts. The first (unit) matrix is not present.

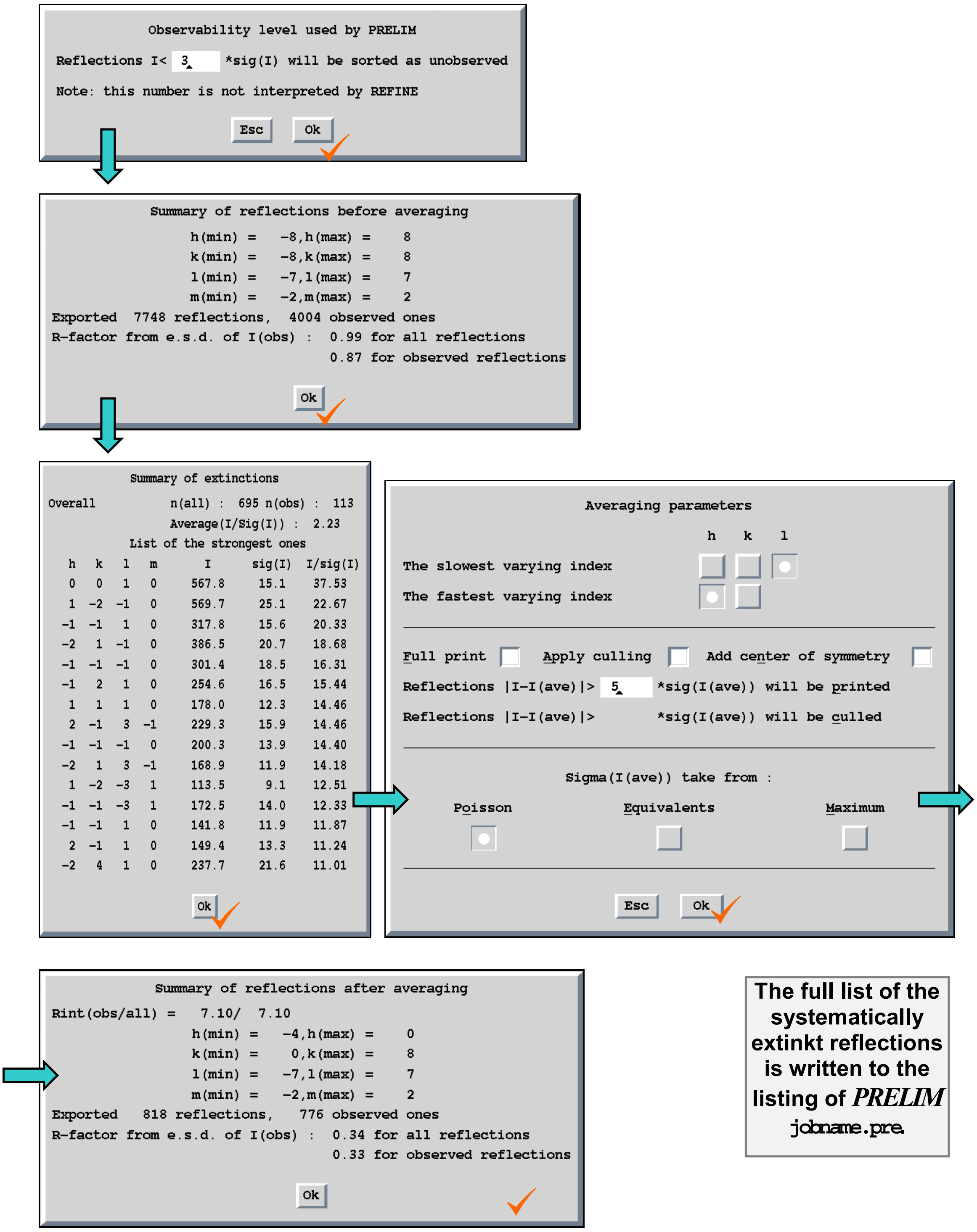
2.2.9 Creation of the Refinement reflection file

This tool creates the refinement reflection file `m91` by combining the basic crystal information and the data from `m94` and `m95`. The most important (optional) action here is the averaging of symmetrically equivalent reflections. This procedure does not influence the data in `m94` and `m95`.

Here are the basic steps leading to creation of `m91`:

- *PRELIM* asks for the observability limit, which is used for calculation of R values for observed and unobserved reflections. This limit is used only locally - *REFINE* has its own observability limit that can be edited with the SetCommands tool.
- *PRELIM* reads reflections from `m95`.
- *PRELIM* checks if the strongest intensity fits with the format of `m91`. Then it offers rescaling of intensities. If some intensities has been found to be out of format the user must rescale them in order to avoid the read error. Otherwise the rescaling is optional. By rescaling the precision of data cannot be enlarged because the intensities and e.s.d's in `m95` are stored with one decimal place.
- *PRELIM* prints summary of reflection read from `m95` and summary of systematically extinct reflections.
- *PRELIM* opens the Averaging form. When it is completed, it starts the averaging.
- *PRELIM* prints the final reflection summary. The internal factor R_{int} that is part of this information can be used as a measure if the symmetry used for the averaging is correct. However, the reliability of its value depends on the number of averaged reflections.
- After pressing `Quit` *PRELIM* asks if the changes of `m50` and `m91` are to be saved.

Figure 57 *Creation of* `m91`



```
Observability level used by PRELIM
Reflections I< 3 *sig(I) will be sorted as unobserved
Note: this number is not interpreted by REFINE
```

```
Summary of reflections before averaging
                h(min) =   -8,h(max) =    8
                k(min) =   -8,k(max) =    8
                l(min) =   -7,l(max) =    7
                m(min) =   -2,m(max) =    2
Exported  7748 reflections,  4004 observed ones
R-factor from e.s.d. of I(obs) :  0.99 for all reflections
                                  0.87 for observed reflections
```

```
           Summary of extinctions
Overall             n(all) :  695 n(obs) :  113
                    Average(I/Sig(I)) :  2.23
           List of the strongest ones
  h   k   l   m       I      sig(I)  I/sig(I)
  0   0   1   0     567.8     15.1    37.53
  1  -2  -1   0     569.7     25.1    22.67
 -1  -1   1   0     317.8     15.6    20.33
 -2   1  -1   0     386.5     20.7    18.68
 -1  -1  -1   0     301.4     18.5    16.31
 -1   2   1   0     254.6     16.5    15.44
  1   1   1   0     178.0     12.3    14.46
  2  -1   3  -1     229.3     15.9    14.46
 -1  -1  -1   0     200.3     13.9    14.40
 -2   1   3  -1     168.9     11.9    14.18
  1  -2  -3   1     113.5      9.1    12.51
 -1  -1  -3   1     172.5     14.0    12.33
 -1  -1   1   0     141.8     11.9    11.87
  2  -1   1   0     149.4     13.3    11.24
 -2   4   1   0     237.7     21.6    11.01
```

```
                     Averaging parameters
                                             h   k   l
The slowest varying index                    ☐   ☐   ●
The fastest varying index                    ●   ☐

Full print ☐   Apply culling ☐   Add center of symmetry ☐
Reflections |I-I(ave)|> 5  *sig(I(ave)) will be printed
Reflections |I-I(ave)|>    *sig(I(ave)) will be culled

                 Sigma(I(ave)) take from :
   Poisson             Equivalents             Maximum
      ●                     ☐                     ☐
```

```
Summary of reflections after averaging
Rint(obs/all) =   7.10/  7.10
                h(min) =   -4,h(max) =    0
                k(min) =    0,k(max) =    8
                l(min) =   -7,l(max) =    7
                m(min) =   -2,m(max) =    2
Exported   818 reflections,   776 observed ones
R-factor from e.s.d. of I(obs) :  0.34 for all reflections
                                  0.33 for observed reflections
```

The full list of the systematically extinkt reflections is written to the listing of *PRELIM* `jobname.pre`

R values

R factor calculated from e.s.d's is defined by the expression

$$R_{e.s.d} = \frac{\sum \sigma(F_i)}{\sum F_i}.$$

The internal R factor is defined as

$$R_{\text{int}} = \sum_i \sum_j \frac{\left(I_j - \bar{I}_i\right)}{\bar{I}_i},$$

where i runs over all independent reflections and j over all equivalent reflections corresponding to the i-th independent reflection. Note that j need not to be the same for all independent reflections.

Calculation of estimated standard deviations ***PRELIM***

The averaging procedure assigns new e.s.d's to the resulting intensities. In the case a reflection is unique, i.e. its intensity has not been calculated like an average value of a group of symmetrically equivalent reflections, the e.s.d. is taken from `m95` and it is only multiplied by the rescaling factor. In the case a reflection belongs to a group of symmetrically equivalent reflections there are three optional ways how to calculate its e.s.d.:

- using Poisson statistics
- using equivalents
- using maximum

The Poisson method is based on the Poisson distribution and takes into account the e.s.d's of the individual intensities known from the measurement.

$$\sigma\left(\bar{I}_i\right) = \frac{1}{n}\sqrt{\sum_j \sigma^2\left(I_j\right)},$$

where n is the number of reflections in the symmetrically equivalent group.

The method using equivalents calculates new e.s.d's independently on the measured ones.

$$\sigma\left(\bar{I}_i\right) = \sqrt{\frac{\sum_j \left(I_j - \bar{I}_i\right)^2}{n(n-1)}}$$

The third method calculates both types of the e.s.d. and takes the larger one.

The culling procedure

The culling procedure is used in cases when some reflections in a symmetrically equivalent group are considerably different from the average value This option can eliminate some strong random errors during the data collection. If the "Apply culling" check box is selected and the limit for culling is set *PRELIM* uses the following procedure:

1. It finds the reflection differing most of the average value.
2. If the difference is less than or equal to the limit the culling is not applied.
3. If the difference is greater than the limit the reflection is discarded and the procedure is repeated with the new average value.

The culling procedure should be used with care and only in well-founded cases.

Listing from averaging

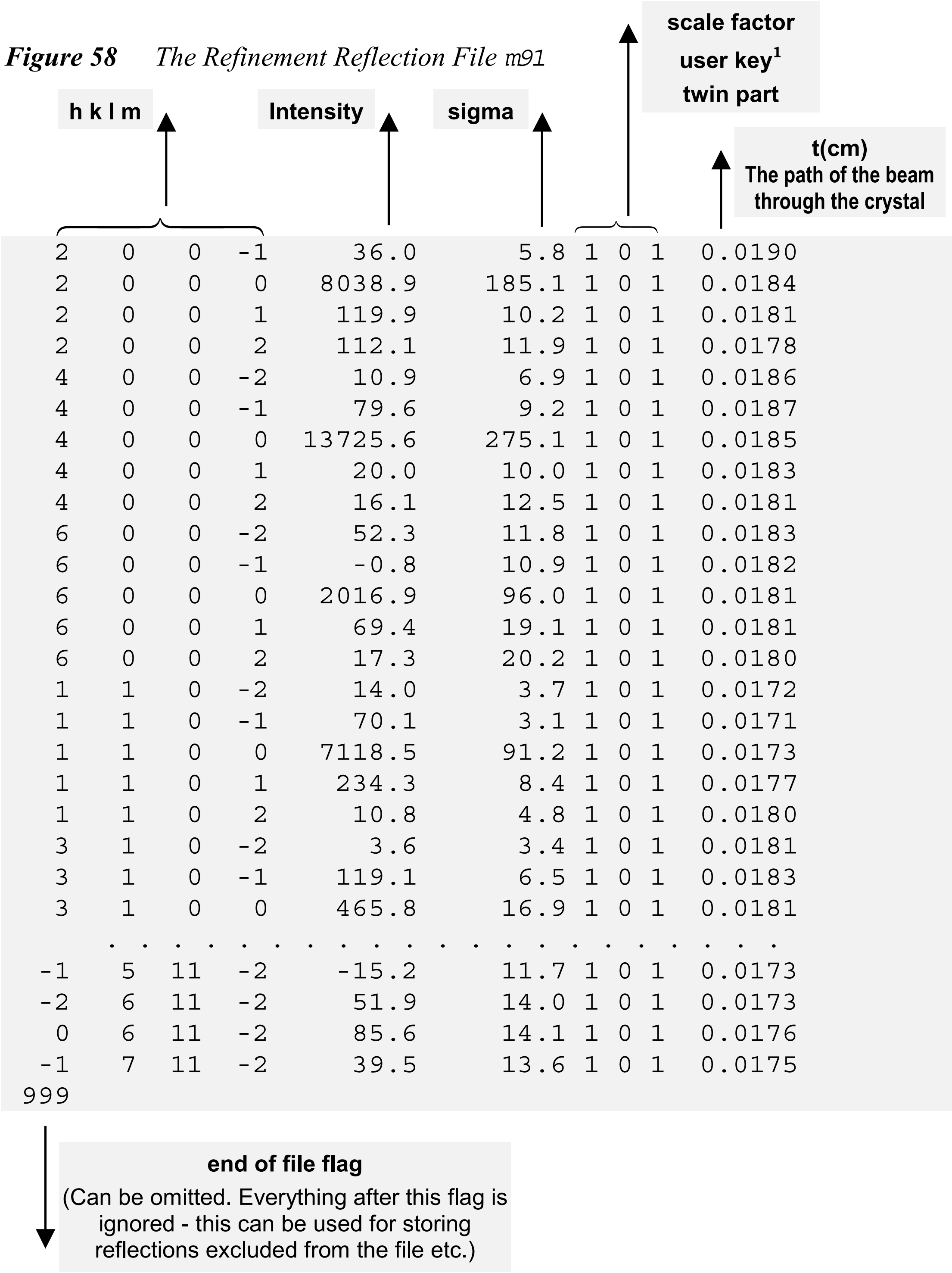
The detailed information about averaging is printed to the listing of *PRELIM* (see page 88). If the `Full print` check box is selected, each reflection from `m95` can be found in the listing. The restricted output contains only the symmetrically equivalent groups where the difference of one or more reflections from the average value is greater that e.s.d. of the average reflection multiplied by the value of the limit. The limit number is optional and can be set *via* the Average form.

Groups of the symmetrically equivalent reflections are printed in the listing together with a flag consisting of asterisks and indicating the extent of the deviation of a single reflections from the average value.

2.2.10 The refinement reflection file `m91`

The file `m91` contains reflections used for the refinement. It is created by *PRELIM* from the basic reflection file `m95` by deleting systematically absent reflections and (optionally) by averaging of reflections according to the used symmetry.

Figure 58 *The Refinement Reflection File* `m91`



h k l m

Intensity

sigma

scale factor
user key[1]
twin part

t(cm)
The path of the beam through the crystal

```
  2   0   0  -1     36.0      5.8 1 0 1  0.0190
  2   0   0   0   8038.9    185.1 1 0 1  0.0184
  2   0   0   1    119.9     10.2 1 0 1  0.0181
  2   0   0   2    112.1     11.9 1 0 1  0.0178
  4   0   0  -2     10.9      6.9 1 0 1  0.0186
  4   0   0  -1     79.6      9.2 1 0 1  0.0187
  4   0   0   0  13725.6    275.1 1 0 1  0.0185
  4   0   0   1     20.0     10.0 1 0 1  0.0183
  4   0   0   2     16.1     12.5 1 0 1  0.0181
  6   0   0  -2     52.3     11.8 1 0 1  0.0183
  6   0   0  -1     -0.8     10.9 1 0 1  0.0182
  6   0   0   0   2016.9     96.0 1 0 1  0.0181
  6   0   0   1     69.4     19.1 1 0 1  0.0181
  6   0   0   2     17.3     20.2 1 0 1  0.0180
  1   1   0  -2     14.0      3.7 1 0 1  0.0172
  1   1   0  -1     70.1      3.1 1 0 1  0.0171
  1   1   0   0   7118.5     91.2 1 0 1  0.0173
  1   1   0   1    234.3      8.4 1 0 1  0.0177
  1   1   0   2     10.8      4.8 1 0 1  0.0180
  3   1   0  -2      3.6      3.4 1 0 1  0.0181
  3   1   0  -1    119.1      6.5 1 0 1  0.0183
  3   1   0   0    465.8     16.9 1 0 1  0.0181
     . . . . . . . . . . . . . . . . . . . .
 -1   5  11  -2    -15.2     11.7 1 0 1  0.0173
 -2   6  11  -2     51.9     14.0 1 0 1  0.0173
  0   6  11  -2     85.6     14.1 1 0 1  0.0176
 -1   7  11  -2     39.5     13.6 1 0 1  0.0175
999
```

end of file flag
(Can be omitted. Everything after this flag is ignored - this can be used for storing reflections excluded from the file etc.)

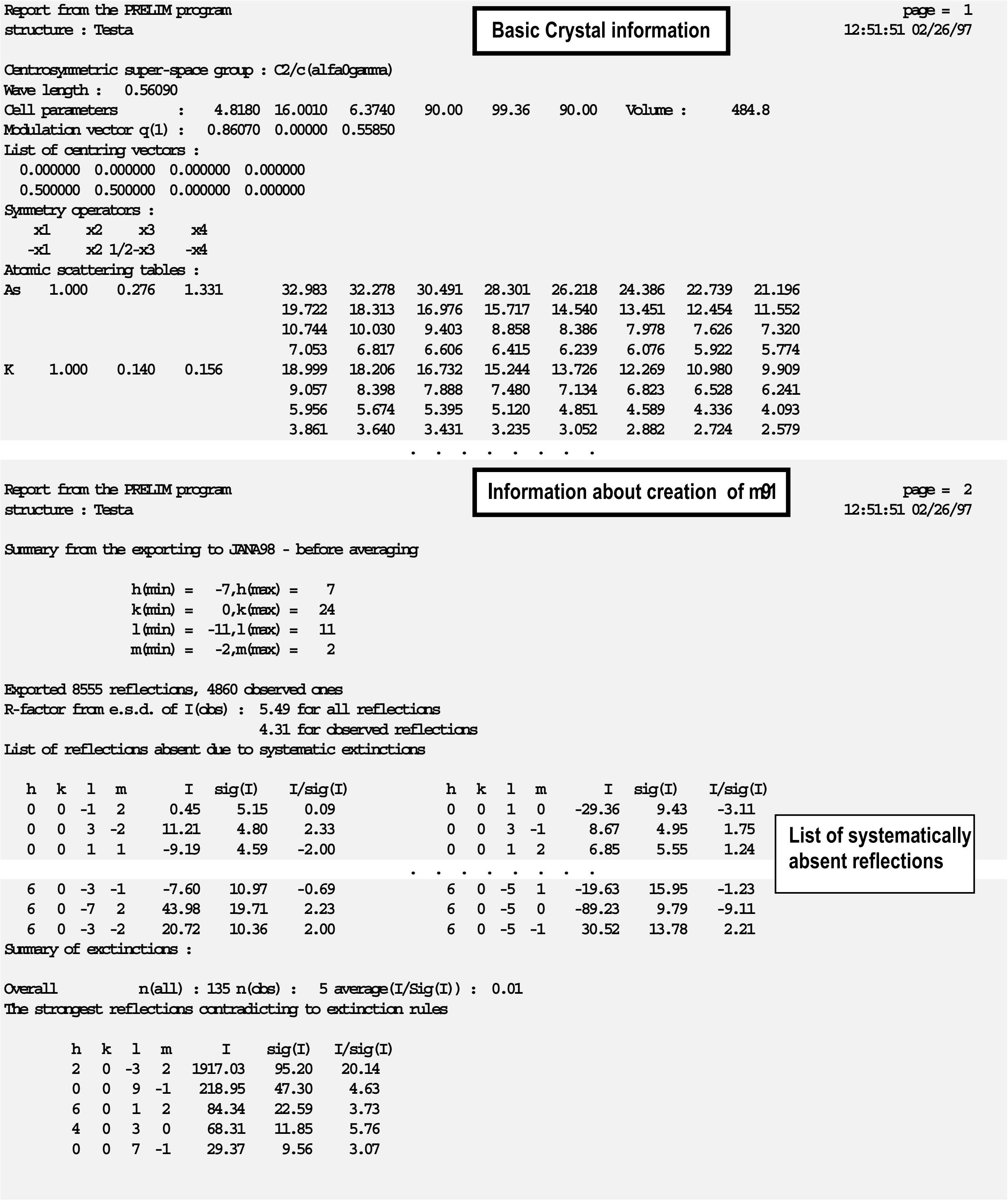
---

[1] The key is used by the refinement control command `skipflag` (see page 240)

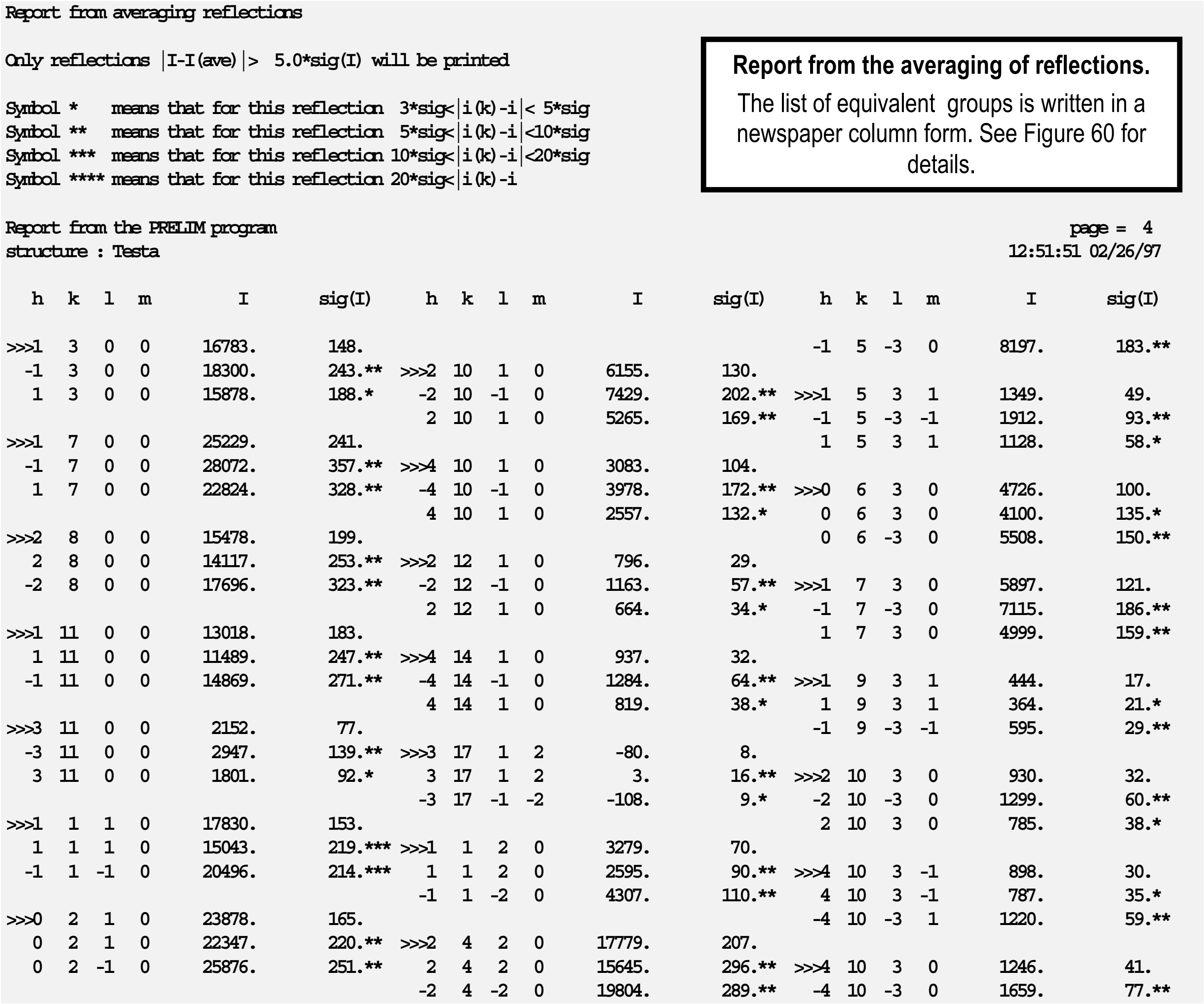
2.2.11 Listing of *PRELIM*

PRELIM creates listing `jobname.pre` containing information about creation of `m50` and `m91`. It can be viewed and printed by the listing viewer (see page 41).

Figure 59 *Listing of PRELIM*



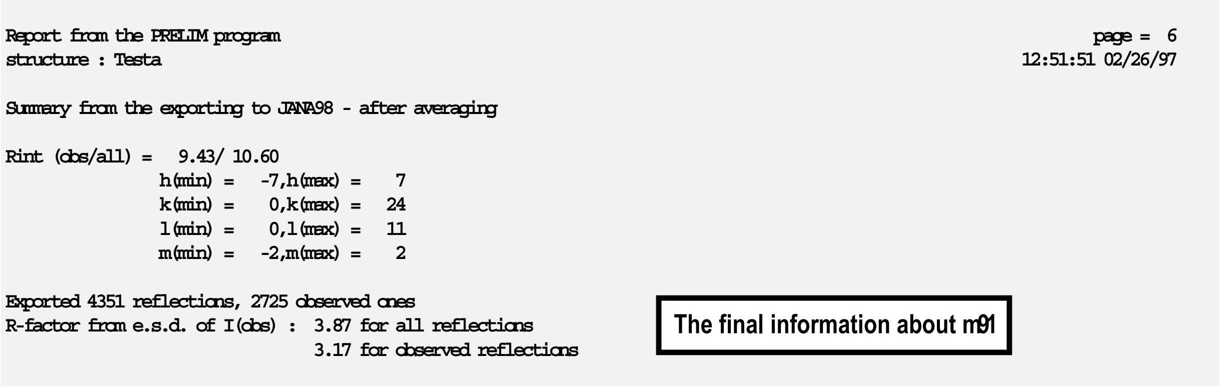
```
Report from the PRELIM program                                                    page =  1
structure : Testa                                                         12:51:51 02/26/97

Centrosymmetric super-space group : C2/c(alfa0gamma)
Wave length :   0.56090
Cell parameters        :   4.8180  16.0010   6.3740    90.00    99.36    90.00    Volume :      484.8
Modulation vector q(1) :   0.86070  0.00000  0.55850
List of centring vectors :
  0.000000  0.000000  0.000000  0.000000
  0.500000  0.500000  0.000000  0.000000
Symmetry operators :
    x1     x2     x3     x4
   -x1     x2 1/2-x3    -x4
Atomic scattering tables :
As    1.000    0.276    1.331        32.983   32.278   30.491   28.301   26.218   24.386   22.739   21.196
                                     19.722   18.313   16.976   15.717   14.540   13.451   12.454   11.552
                                     10.744   10.030    9.403    8.858    8.386    7.978    7.626    7.320
                                      7.053    6.817    6.606    6.415    6.239    6.076    5.922    5.774
K     1.000    0.140    0.156        18.999   18.206   16.732   15.244   13.726   12.269   10.980    9.909
                                      9.057    8.398    7.888    7.480    7.134    6.823    6.528    6.241
                                      5.956    5.674    5.395    5.120    4.851    4.589    4.336    4.093
                                      3.861    3.640    3.431    3.235    3.052    2.882    2.724    2.579
                                   .  .  .  .  .  .  .  .

Report from the PRELIM program                                                    page =  2
structure : Testa                                                         12:51:51 02/26/97

Summary from the exporting to JANA98 - before averaging

               h(min) =   -7,h(max) =    7
               k(min) =    0,k(max) =   24
               l(min) =  -11,l(max) =   11
               m(min) =   -2,m(max) =    2

Exported 8555 reflections, 4860 observed ones
R-factor from e.s.d. of I(obs) :  5.49 for all reflections
                                  4.31 for observed reflections
List of reflections absent due to systematic extinctions

   h   k   l   m        I    sig(I)    I/sig(I)          h   k   l   m        I    sig(I)    I/sig(I)
   0   0  -1   2     0.45     5.15      0.09             0   0   1   0   -29.36     9.43     -3.11
   0   0   3  -2    11.21     4.80      2.33             0   0   3  -1     8.67     4.95      1.75
   0   0   1   1    -9.19     4.59     -2.00             0   0   1   2     6.85     5.55      1.24
                                   .  .  .  .  .  .  .  .
   6   0  -3  -1    -7.60    10.97     -0.69             6   0  -5   1   -19.63    15.95     -1.23
   6   0  -7   2    43.98    19.71      2.23             6   0  -5   0   -89.23     9.79     -9.11
   6   0  -3  -2    20.72    10.36      2.00             6   0  -5  -1    30.52    13.78      2.21
Summary of exctinctions :

Overall          n(all) : 135 n(obs) :   5 average(I/Sig(I)) :  0.01
The strongest reflections contradicting to extinction rules

        h   k   l   m        I     sig(I)   I/sig(I)
        2   0  -3   2   1917.03    95.20    20.14
        0   0   9  -1    218.95    47.30     4.63
        6   0   1   2     84.34    22.59     3.73
        4   0   3   0     68.31    11.85     5.76
        0   0   7  -1     29.37     9.56     3.07
```



```
Report from averaging reflections

Only reflections |I-I(ave)|>  5.0*sig(I) will be printed

Symbol *    means that for this reflection  3*sig<|i(k)-i|< 5*sig
Symbol **   means that for this reflection  5*sig<|i(k)-i|<10*sig
Symbol ***  means that for this reflection 10*sig<|i(k)-i|<20*sig
Symbol **** means that for this reflection 20*sig<|i(k)-i

Report from the PRELIM program                                                                          page =  4
structure : Testa                                                                                 12:51:51 02/26/97

   h   k   l   m         I     sig(I)     h   k   l   m         I     sig(I)     h   k   l   m         I     sig(I)

>>>1   3   0   0    16783.     148.                                              -1   5  -3   0     8197.     183.**
  -1   3   0   0    18300.     243.**  >>>2  10   1   0     6155.     130.
   1   3   0   0    15878.     188.*     -2  10  -1   0     7429.     202.**  >>>1   5   3   1     1349.      49.
                                          2  10   1   0     5265.     169.**    -1   5  -3  -1     1912.      93.**
>>>1   7   0   0    25229.     241.                                               1   5   3   1     1128.      58.*
  -1   7   0   0    28072.     357.**  >>>4  10   1   0     3083.     104.
   1   7   0   0    22824.     328.**    -4  10  -1   0     3978.     172.**  >>>0   6   3   0     4726.     100.
                                          4  10   1   0     2557.     132.*      0   6   3   0     4100.     135.*
>>>2   8   0   0    15478.     199.                                               0   6  -3   0     5508.     150.**
   2   8   0   0    14117.     253.**  >>>2  12   1   0      796.      29.
  -2   8   0   0    17696.     323.**    -2  12  -1   0     1163.      57.**  >>>1   7   3   0     5897.     121.
                                          2  12   1   0      664.      34.*     -1   7  -3   0     7115.     186.**
>>>1  11   0   0    13018.     183.                                               1   7   3   0     4999.     159.**
   1  11   0   0    11489.     247.**  >>>4  14   1   0      937.      32.
  -1  11   0   0    14869.     271.**    -4  14  -1   0     1284.      64.**  >>>1   9   3   1      444.      17.
                                          4  14   1   0      819.      38.*      1   9   3   1      364.      21.*
>>>3  11   0   0     2152.      77.                                              -1   9  -3  -1      595.      29.**
  -3  11   0   0     2947.     139.**  >>>3  17   1   2      -80.       8.
   3  11   0   0     1801.      92.*      3  17   1   2        3.      16.**  >>>2  10   3   0      930.      32.
                                         -3  17  -1  -2     -108.       9.*     -2  10  -3   0     1299.      60.**
>>>1   1   1   0    17830.     153.                                               2  10   3   0      785.      38.*
   1   1   1   0    15043.     219.*** >>>1   1   2   0     3279.      70.
  -1   1  -1   0    20496.     214.***    1   1   2   0     2595.      90.**  >>>4  10   3  -1      898.      30.
                                         -1   1  -2   0     4307.     110.**     4  10   3  -1      787.      35.*
>>>0   2   1   0    23878.     165.                                              -4  10  -3   1     1220.      59.**
   0   2   1   0    22347.     220.**  >>>2   4   2   0    17779.     207.
   0   2  -1   0    25876.     251.**     2   4   2   0    15645.     296.**  >>>4  10   3   0     1246.      41.
                                         -2   4  -2   0    19804.     289.**    -4  10  -3   0     1659.      77.**
```

Report from the averaging of reflections.
The list of equivalent groups is written in a newspaper column form. See Figure 60 for details.

. . . .



```
Report from the PRELIM program                                                                          page =  6
structure : Testa                                                                                 12:51:51 02/26/97

Summary from the exporting to JANA98 - after averaging

Rint (obs/all) =   9.43/ 10.60
                   h(min) =   -7,h(max) =    7
                   k(min) =    0,k(max) =   24
                   l(min) =    0,l(max) =   11
                   m(min) =   -2,m(max) =    2

Exported 4351 reflections, 2725 observed ones
R-factor from e.s.d. of I(obs) :  3.87 for all reflections
                                  3.17 for observed reflections
```

The final information about m91

Figure 60 *The details about the listing from averaging*

```
     h   k   l   m          I     sigma(I)+Flag
>> -1   3  -6   2         11.          2.
   -1  -2  -6   2         26.          8.**
    1  -3  -6   2          3.          7.*
   -2   3  -6   2          5.          7.
   -2  -1  -6   2          4.          7.*
   -2  -1  -6   2         15.          7.
   -1  -2  -6   2          9.         10.
   -3   2  -6   2         -2.          4.**
   -3   2  -6   2          3.          4.*
   -2   3  -6   2          9.          6.
   -3   1  -6   2         43.         12.***
    1  -3  -6   2          5.          5.
   -1  -2  -6   2          6.          7.
   -3   1  -6   2         16.          9.
   -2  -1  -6   2         14.          7.
    2  -3  -6   2          6.         14.
```

The values written to m91

A group of symmetrically equivalent reflections. The flag “*” means that the deviation of the intensity from the average value is between 3*sigma and 5*sigma. The flag “**” means the same for the interval <5*sigma, 10*sigma> etc. The lowest deviation under which the printing is suppressed can be set *via* the Average form.

3

Structure Solution and Refinement

This chapter describes basic programs of JANA98, their input and output files and the control keys.

IN THIS CHAPTER:

Next Chapter: Structure Interpretation, page 289.
Previous chapter: Preliminary Work, page 53.

3

Structure Solution and Refinement

This part describes the programs and files necessary for solution of the phase problem, setting up the structure model and its refinement provided that the preliminary work like preparation of the reflection file and setting of the basic crystal information has been already done.

The structure solution and refinement means that a proper set of structure parameters is found and refined. The first part of this chapter describes the structure of `m40` containing the structural parameters and gives a brief theoretical background for each refinable parameter.

After having the basic knowledge about `m40` program *EDITM40* for setting and changing parameters of a structure model is introduced. This is a set of tools for tasks like changing isotropic temperature parameters to the anisotropic ones, adding or deleting modulation waves etc. The next part describes program *REFINE* for refinement of the structure model. *REFINE* is a hart of the system enabling large variety of refinement strategies. The last program in this chapter is *FOURIER* used for calculation of 3+d electron density maps.

3.1 Structure parameters

Structure parameters in *JANA98* are written in the Refinement parameter file `m40`. Usually the user prepares `m40` with *EDITM40* interface or imports a starting model from SHELX or CIF format (see description of Tools, §1.4.4, page 43). There is also possibility to prepare the starting model *ab initio* using the Heavy atom method. In every case the model is further refined with *REFINE*, which overwrites the initial model in `m40` with the refined one. See § 3.3 page 219 for more information about *REFINE* and § 3.2 page 153 for details about *EDITM40*.

In the next section `m40` will be presented both in the symbolic form and in examples. The parameter names shown in the symbolic form are the same as used by *REFINE* in equations, constraints, fixed commands and in the listing. The refinable parameters are followed by the refinement keys, which equal 1 for refined and 0 for fixed parameters. The setting of the refinement keys is fully automatic in *JANA98* except rare cases. See more information about the refinement keys in §3.3.3 page 223.

3.1.1 The refinement parameter file `m40`

The basic parts of `m40`

Commands
Header numbers
Scale parameters
Extinction parameters
Atomic part of the 1st composite part
Molecular[1] part of the 1st composite part
Atomic part of the 2nd composite part
Molecular part of the 2nd composite part
Atomic part of the 3rd composite part
Molecular part of the 3rd composite part
E.s.d's in Atomic part of the 1st composite part
E.s.d's in Molecular part of the 1st composite part
E.s.d's in Atomic part of the 2nd composite part
E.s.d's in Molecular part of the 2nd composite part
E.s.d's in Atomic part of the 3rd composite part
E.s.d's in Molecular part of the 3rd composite part

---

[1] Molecule in *JANA98* is a group of atoms having some common parameters. It is not necessarily molecule in the chemical meaning. The rigidity of the molecule is determined by the type of parameters being common for all atoms of the molecule and for all its positions.

Typical examples of `m40`

Figure 61 *The example of* `m40` *containing one composite part without molecules.*

```
commands
. . . .
. . . .
end
    5    0    0    0
 1.263756 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
As        1  2     0.500000 0.000000 0.631039 0.250000      000  0  2  2
 0.016070 0.019152 0.018667 0.000000 0.003317 0.000000      0010111010
-0.011058 0.000000-0.026379 0.000000 0.013897 0.000000      101010
-0.001426 0.000000 0.003925 0.000000 0.001400 0.000000      101010
 0.000000 0.000000 0.000000-0.001399 0.000000 0.001612      000101
 0.001689-0.000003 0.000386 0.000000-0.000072 0.000000      111010
 0.000000 0.000000 0.000000 0.000264 0.000000 0.001977      000101
 0.000283-0.000805-0.000181 0.000000 0.000856 0.000000      111010
 0.000000                                                   0
                                            . . . . . .
O3        4  2     1.000000 0.259540 0.704262 0.334582      000  0  2  2
 0.033601 0.045856 0.050422-0.018620 0.018743-0.023155      0111111111
 0.001588 0.012132 0.014724 0.049204-0.017804 0.060715      111111
 0.007225-0.002704 0.000271-0.004372-0.002479 0.004139      111111
 0.001844 0.003826 0.005253-0.001272 0.001209-0.002663      111111
-0.008377 0.003772 0.004731 0.005381-0.003017-0.006556      111111
-0.002579 0.015193 0.003039-0.005828-0.001797-0.012093      111111
-0.003972-0.014366-0.017415 0.013566-0.014126 0.018702      111111
 0.000000                                                   0
As                 0.000000 0.000000 0.000023 0.000000
 0.000194 0.000194 0.000193 0.000000 0.000120 0.000000
 0.000110 0.000000 0.000089 0.000000 0.000034 0.000000
 0.000156 0.000000 0.000110 0.000000 0.000042 0.000000
 0.000000 0.000000 0.000000 0.000172 0.000000 0.000175
 0.000206 0.000238 0.000215 0.000000 0.000164 0.000000
 0.000000 0.000000 0.000000 0.000245 0.000000 0.000217
 0.000343 0.000272 0.000294 0.000000 0.000236 0.000000
 0.000000
                                            . . . . . .
O3                 0.000000 0.000581 0.000180 0.000472
 0.001374 0.001606 0.001792 0.001219 0.001288 0.001398
 0.000776 0.000273 0.000719 0.000752 0.000225 0.000618
 0.000999 0.000306 0.000761 0.001031 0.000310 0.000811
 0.002027 0.002303 0.002737 0.001787 0.001852 0.001985
 0.001743 0.001858 0.001986 0.001432 0.001420 0.001470
 0.002445 0.002647 0.002694 0.001990 0.002036 0.002197
 0.002550 0.002628 0.002897 0.002092 0.002125 0.002218
 0.000000
```

Commands

Header numbers Scale parameters Extinction parameters

Parameters of atom As

Parameters of atom O3

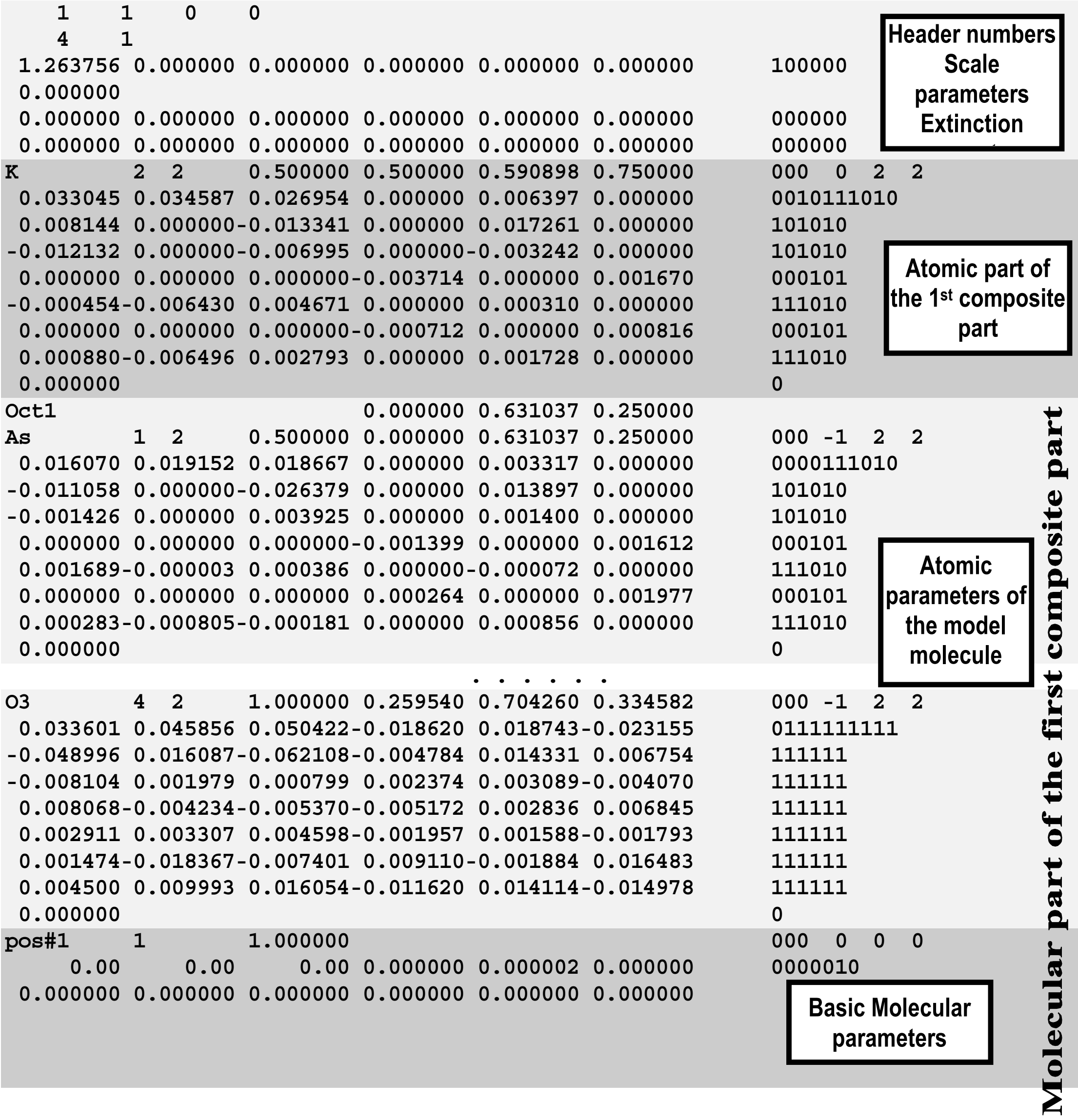
Atomic part of the first composite part

The e.s.d's concerning atom As

The e.s.d's concerning atom O3

Esd's in atomic part of the first composite part

Figure 62 *The example of* `m40` *containing one composite part with a molecule*[1].

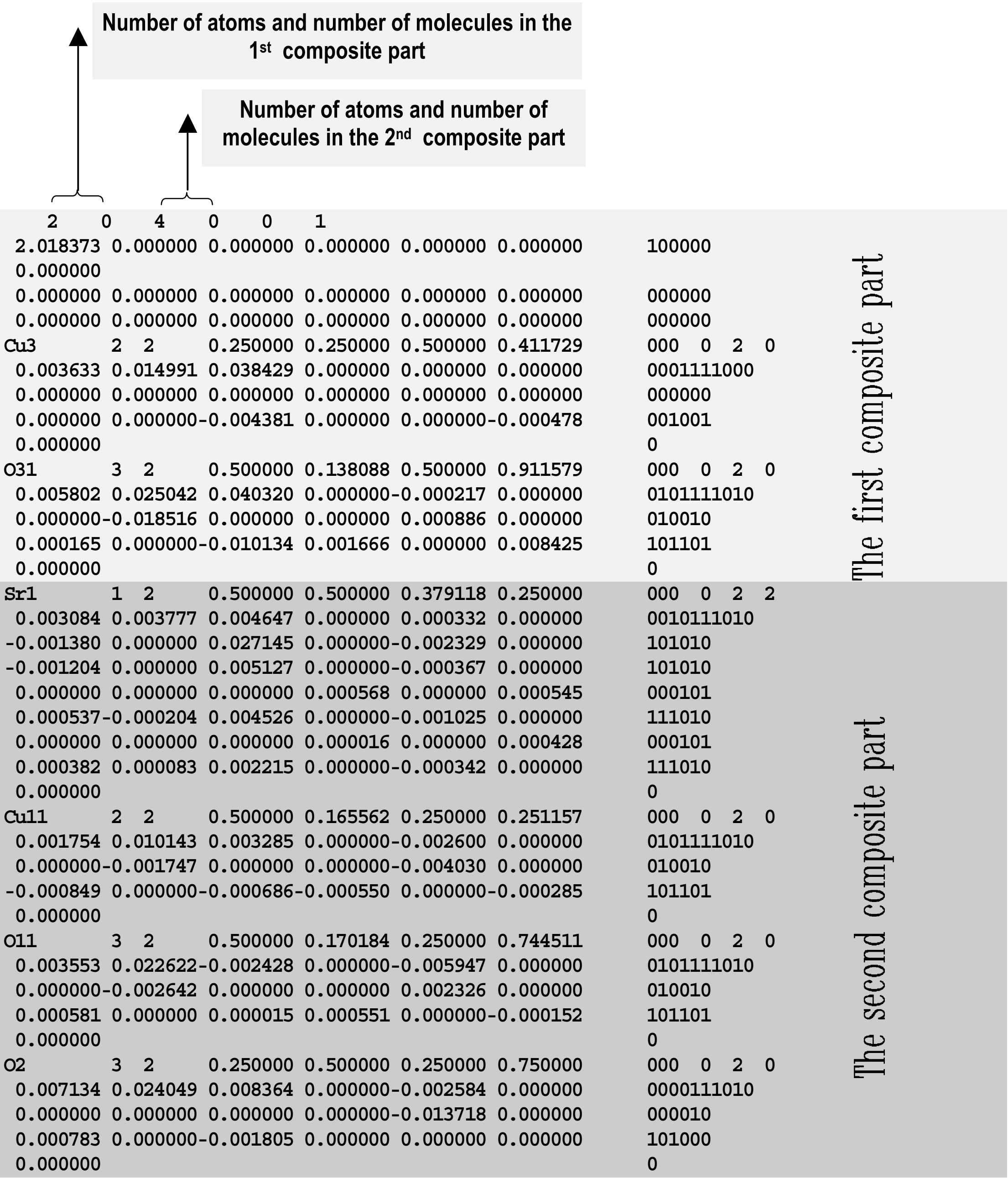


```
    1    1    0    0
    4    1
 1.263756 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K          2  2     0.500000 0.500000 0.590898 0.750000      000  0  2  2
 0.033045 0.034587 0.026954 0.000000 0.006397 0.000000      0010111010
 0.008144 0.000000-0.013341 0.000000 0.017261 0.000000      101010
-0.012132 0.000000-0.006995 0.000000-0.003242 0.000000      101010
 0.000000 0.000000 0.000000-0.003714 0.000000 0.001670      000101
-0.000454-0.006430 0.004671 0.000000 0.000310 0.000000      111010
 0.000000 0.000000 0.000000-0.000712 0.000000 0.000816      000101
 0.000880-0.006496 0.002793 0.000000 0.001728 0.000000      111010
 0.000000                                                   0
Oct1                                 0.000000 0.631037 0.250000
As         1  2     0.500000 0.000000 0.631037 0.250000      000 -1  2  2
 0.016070 0.019152 0.018667 0.000000 0.003317 0.000000      0000111010
-0.011058 0.000000-0.026379 0.000000 0.013897 0.000000      101010
-0.001426 0.000000 0.003925 0.000000 0.001400 0.000000      101010
 0.000000 0.000000 0.000000-0.001399 0.000000 0.001612      000101
 0.001689-0.000003 0.000386 0.000000-0.000072 0.000000      111010
 0.000000 0.000000 0.000000 0.000264 0.000000 0.001977      000101
 0.000283-0.000805-0.000181 0.000000 0.000856 0.000000      111010
 0.000000                                                   0
                                            . . . . . . .
O3         4  2     1.000000 0.259540 0.704260 0.334582      000 -1  2  2
 0.033601 0.045856 0.050422-0.018620 0.018743-0.023155      0111111111
-0.048996 0.016087-0.062108-0.004784 0.014331 0.006754      111111
-0.008104 0.001979 0.000799 0.002374 0.003089-0.004070      111111
 0.008068-0.004234-0.005370-0.005172 0.002836 0.006845      111111
 0.002911 0.003307 0.004598-0.001957 0.001588-0.001793      111111
 0.001474-0.018367-0.007401 0.009110-0.001884 0.016483      111111
 0.004500 0.009993 0.016054-0.011620 0.014114-0.014978      111111
 0.000000                                                   0
pos#1      1        1.000000                                000  0  0  0
     0.00      0.00      0.00 0.000000 0.000002 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
```

---

[1] Work with molecules is explained in page 171.

Figure 63 *The example of* `m40` *with two composite parts.*



```
   2    0    4    0    0    1
 2.018373 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000                                                    0
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
Cu3       2  2     0.250000 0.250000 0.500000 0.411729      000  0  2  0
 0.003633 0.014991 0.038429 0.000000 0.000000 0.000000      0001111000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000-0.004381 0.000000 0.000000-0.000478      001001
 0.000000                                                    0
O31       3  2     0.500000 0.138088 0.500000 0.911579      000  0  2  0
 0.005802 0.025042 0.040320 0.000000-0.000217 0.000000      0101111010
 0.000000-0.018516 0.000000 0.000000 0.000886 0.000000      010010
 0.000165 0.000000-0.010134 0.001666 0.000000 0.008425      101101
 0.000000                                                    0
Sr1       1  2     0.500000 0.500000 0.379118 0.250000      000  0  2  2
 0.003084 0.003777 0.004647 0.000000 0.000332 0.000000      0010111010
-0.001380 0.000000 0.027145 0.000000-0.002329 0.000000      101010
-0.001204 0.000000 0.005127 0.000000-0.000367 0.000000      101010
 0.000000 0.000000 0.000000 0.000568 0.000000 0.000545      000101
 0.000537-0.000204 0.004526 0.000000-0.001025 0.000000      111010
 0.000000 0.000000 0.000000 0.000016 0.000000 0.000428      000101
 0.000382 0.000083 0.002215 0.000000-0.000342 0.000000      111010
 0.000000                                                    0
Cu11      2  2     0.500000 0.165562 0.250000 0.251157      000  0  2  0
 0.001754 0.010143 0.003285 0.000000-0.002600 0.000000      0101111010
 0.000000-0.001747 0.000000 0.000000-0.004030 0.000000      010010
-0.000849 0.000000-0.000686-0.000550 0.000000-0.000285      101101
 0.000000                                                    0
O11       3  2     0.500000 0.170184 0.250000 0.744511      000  0  2  0
 0.003553 0.022622-0.002428 0.000000-0.005947 0.000000      0101111010
 0.000000-0.002642 0.000000 0.000000 0.002326 0.000000      010010
 0.000581 0.000000 0.000015 0.000551 0.000000-0.000152      101101
 0.000000                                                    0
O2        3  2     0.250000 0.500000 0.250000 0.750000      000  0  2  0
 0.007134 0.024049 0.008364 0.000000-0.002584 0.000000      0000111010
 0.000000 0.000000 0.000000 0.000000-0.013718 0.000000      000010
 0.000783 0.000000-0.001805 0.000000 0.000000 0.000000      101000
 0.000000                                                    0
```

The symbolic form of `m40`

Table 10 shows `m40` in symbolic form. Both constants and refinable parameters are explained in the section following the table.

Notation used in the table:

Name of a section of m40
Keywords
Non-refinable constants
`Refinable parameters`
`kkkkkk`

"kkkkkk" denotes the refinement keys. Each "k" stands for a refinement key 0 or 1. The order of refinement keys is the same like the order of parameters in the same line, i.e. the first "k" in some line is the refinement key of the first parameter in the same line. A few exceptions from this rule which will be mentioned later. The setting of the refinement keys is fully automatic in *JANA98* except rare cases. See more information about the refinement keys in §3.3.3 page 223.

Figure 64 *The location of refinement keys in* `m40`

The numbers inside the box are the refinement keys except the line "000 0 2 2" which has different meaning (see Table 10).

```
    5    0    0    0
 1.263756 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
As        1  2     0.500000 0.000000 0.631039 0.250000      000  0  2  2
 0.016070 0.019152 0.018667 0.000000 0.003317 0.000000      0010111010
-0.011058 0.000000-0.026379 0.000000 0.013897 0.000000      101010
-0.001426 0.000000 0.003925 0.000000 0.001400 0.000000      101010
 0.000000 0.000000 0.000000-0.001399 0.000000 0.001612      000101
 0.001689-0.000003 0.000386 0.000000-0.000072 0.000000      111010
 0.000000 0.000000 0.000000 0.000264 0.000000 0.001977      000101
 0.000283-0.000805-0.000181 0.000000 0.000856 0.000000      111010
 0.000000                                                   0
```

Table 10 *File* `m40` *in the symbolic form*

Commands section (see page 105)					
Commands					
maxsc (page 105)	nsc				
ortho[1] (page 106)	name	delta	x_{40}	n	
n1sin	n1cos	n2sin	n2cos	......	epsilon
wave[2] (page 105)	nwave	nv_1	nv_2	......	
end					

Header numbers (see page 107)								
Nat_1	$Nmol_1$	Nat_2[3]	$Nmol_2$	Nat_3[4]	$Nmol_3$	Itemp	Irot	
$Natm_1$	$Npos_1$							
$Natm_2$	$Npos_2$	$Nmol_1$ lines for the 1st composite subsystem						
......								
$Natm_1$	$Npos_1$							
$Natm_2$	$Npos_2$	$Nmol_2$ lines for the 2nd composite subsystem (if exists)						
......								
$Natm_1$	$Npos_1$							
$Natm_2$	$Npos_2$	$Nmol_3$ lines for the 3rd composite subsystem (if exists)						
......								

Scale parameters[5] (see page 108)						
`scale1`	`scale2`	`scale3`	`scale4`	`scale5`	`scale6`	`kkkkkk`
`scale7`	`scale8`	`scale9`	`scale10`	`scale11`	`scale12`	`kkkkkk`
`scale13`	`scale14`	`scale15`	`scale16`	`scale17`	`scale18`	`kkkkkk`
Overall isotropic temperature parameter (see page 110)						
Otemp						
Extinction parameters (see page 110)						
`rho11/` `rhoiso`	`rho22`	`rho33`	`rho12`	`rho13`	`rho23`	`kkkkkk`
`g11/giso`	`g22`	`g33`	`g12`	`g13`	`g23`	`kkkkkk`

---

[1] This command is repeted for each atom needing the orthogonalisation method.

[2] This command is repeated for every wave needing a user definition of the wave vector.

[3] The parameters Nat_2,$Nmol_2$ are present only when the number of composite parts is ≥ 2.

[4] The parameters Nat_3,$Nmol_3$ are present only when the number of composite parts equals 2.

[5] The 2nd and 3rd line of scale parameters is only present when the default number of scale parameters is changed by **maxsc** command (see page 105)

Table 10 *(Continued)*

THE FIRST COMPOSITE PART - BEGINNING ***The atomic part of the 1st composite part - beginning*** (This example is for 1 atom, i.e. Nat$_1$ = 1)								
Atomic header parameters (see page 116)								
Name	Chtype	Ttype	`ai`	`x`	`y`	`z`	$S_oS_pS_t$ W_o W_p W_t [1]	
Name[2]							$S_{t3}S_{t4}S_{t5}S_{t6}$ W_{t3} W_{t4} W_{t5} W_{t6}	
Temperature parameters (see page 120)								
`U11`	`U22`	`U33`	`U12`	`U13`	`U23`	kkkkkkkkkk		
Anharmonic displacement parameters (ADP) [3] (see page 121)								
`C111`	`C112`	`C113`	`C122`	`C123`	`C133`	kkkkkk		3rd
`C222`	`C223`	`C233`	`C333`			kkkk		
`D1111`	`D1112`	`D1113`	`D1122`	`D1123`	`D1133`	kkkkkk		4th
`D1222`	`D1223`	`D1233`	`D1333`	`D2222`	`D2223`	kkkkkk		
`D2233`	`D2333`	`D3333`				kkk		
`E11111`	`E11112`	`E11113`	`E11122`	`E11123`	`E11133`	kkkkkk		5th
`E11222`	`E11223`	`E11233`	`E11333`	`E12222`	`E12223`	kkkkkk		
`E12233`	`E12333`	`E13333`	`E22222`	`E22223`	`E22233`	kkkkkk		
`E22333`	`E23333`	`E33333`				kkk		
`F111111`	`F111112`	`F111113`	`F111122`	`F111123`	`F111133`	kkkkkk		6th
`F111222`	`F111223`	`F111233`	`F111333`	`F112222`	`F112223`	kkkkkk		
`F112233`	`F112333`	`F113333`	`F122222`	`F122223`	`F122233`	kkkkkk		
`F122333`	`F123333`	`F133333`	`F222222`	`F222223`	`F222233`	kkkkkk		
`F222333`	`F223333`	`F233333`	`F333333`			kkkk		
Occupation modulation parameters, harmonic functions[4] (see page 127)								
`o`						k		
`osin1`	`ocos1`					kk		
`...`	`...`					...		
`osin16`	`ocos16`					kk		
`...`	`...`					...		
Occupation modulation parameters, crenel function[5] (see page 127)								
`o`						k		
`osin1`	`ocos1`					kk		

---

[1] This cell is only present for modulated structures.

[2] This line is omitted for non-modulated atoms. With modulated atoms it is omitted if Ttype is less or equal to 2, i.e.for atoms without anharmonic displacement parameters, (see page 116).

[3] These lines are present if Ttype > 2, (see page 116).

[4] These lines are present in the case of occupation modulation described by harmonic functions, i.e. for $W_o > 0$ and $S_o = 0$, (see page 116).

[5] These lines are present in the case of occupation modulation described by crenel function, i.e. for $W_o > 0$ and $S_o = 1$, (see page 116).

***Table 10** (Continued)*

Position modulation parameters, harmonic functions[1] (see page 123)						
xsin1	ysin1	zsin1	xcos1	ycos1	zcos1	kkkkkk
...	...	...	...	...	...	...
xsin16	ysin16	zsin16	xcos16	ycos16	zcos16	kkkkkk
...	...	...	...	...	...	...
Position modulation parameters, sawtooth function[2] (see page 123)						
xsin$[3]	ysin$	zsin$	xcos$	ycos$		kkkkk0
Temperature modulation parameters[4] (see page 131)						
U11sin1 Beta11sin1[5]	U22sin1 Beta22sin1	U33sin1 Beta33sin1	U12sin1 Beta12sin1	U13sin1 Beta13sin1	U23sin1 Beta23sin1	kkkkkk
U11cos1 Beta11cos1	U22cos1 Beta22cos1	U33cos1 Beta33cos1	U12cos1 Beta12cos1	U13cos1 Beta13cos1	U23cos1 Beta23cos1	kkkkkk
...	...	...	...	...	...	...
U11sin16 Beta11sin16	U22sin16 Beta22sin16	U33sin16 Beta33sin16	U12sin16 Beta12sin16	U13sin16 Beta13sin16	U23sin16 Beta23sin16	kkkkkk
U11cos16 Beta11cos16	U22cos16 Beta22cos16	U33cos16 Beta33cos16	U12cos16 Beta12cos16	U13cos16 Beta13cos16	U23cos16 Beta23cos16	kkkkkk
...	...	...	...	...	...	...
Modulation of ADP[6] (see page 137)						
C111sin1	C112sin1	C113sin1	C122sin1	C123sin1	C133sin1	kkkkkk
C222sin1	C223sin1	C233sin1	C333sin1			kkkk
C111cos1	C112cos1	C113cos1	C122cos1	C123cos1	C133cos1	kkkkkk
C222cos1	C223cos1	C233cos1	C333cos1			kkkk
...	...	...	...	...	...	...
C111sin16	C112sin16	C113sin16	C122sin16	C123sin16	C133sin16	kkkkkk
...	...	...	...	...	...	...
D1111sin1	D1112sin1	D1113in1	D1122sin1	D1123sin1	D1133sin1	kkkkkk
D1222sin1	D1223sin1	D1233sin1	D1333sin1	D2222sin1	D2223sin1	kkkkkk
D2233sin1	D2333sin1	D3333sin1				kkk
D1111cos1	D1112cos1	D1113cos1	D1122cos1	D1123cos1	D1133cos1	kkkkkk
D1222cos1	D1223cos1	D1233cos1	D1333cos1	D2222cos1	D2223cos1	kkkkkk
D2233cos1	D2333cos1	D3333cos1				kkk

---

[1] These lines are present in the case of position modulation described by harmonic functions, i.e. for $w_p > 0$ and $s_p = 0$, (see page 116).

[2] This line is present in the case of position modulation described by crenel function, i.e. for $w_p > 0$ and $s_p = 1$, (see page 116). It is always the last line of position modulation parameters.

[3] $ symbolises the sawtooth function parameters are always in the place of the last wave - see page 123 for details.

[4] These lines are present in the case of temperature modulation, i.e. for $w_t > 0$, (see page 116).

[5] The name of the modulation temperature parameters can be referenced to as U or Beta regardless of the current setting in `m40`.

[6] This line is present in the case of ADP modulation, i.e. for Ttype > 2 and ($w_{t3} > 0$ or $w_{t4} > 0$ or $w_{t5} > 0$), (see page 116).

Table 10 *(Continued)*

…	…	…	…	…	…	...
D1111sin16	D1112sin16	D1113sin16	D1122sin16	D1123sin16	D1133sin16	kkkkkk
E11111sin1	E11112sin1	E11113sin1	E11122sin1	E11123sin1	E11133sin1	kkkkkk
E11222sin1	E11223sin1	E11233sin1	E11333sin1	E12222sin1	E12223sin1	kkkkkk
E12233sin1	E12333sin1	E13333sin1	E22222sin1	E22223sin1	E22233sin1	kkkkkk
E22333sin1	E23333sin1	E33333sin1				kkk
E11111cos1	E11112cos1	E11113cos1	E11122cos1	E11123cos1	E11133cos1	kkkkkk
E11222cos1	E11223cos1	E11233cos1	E11333cos1	E12222cos1	E12223cos1	kkkkkk
E12233cos1	E12333cos1	E13333cos1	E22222cos1	E22223cos1	E22233cos1	kkkkkk
E22333cos1	E23333cos1	E33333cos1				kkk
…	…	…	…	…	…	...
E11111sin16	E11112sin16	E11113sin16	E11122sin16	E11123sin16	E11133sin16	kkkkkk
…	…	…	…	…	…	...
F111111sin1	F111112sin1	F111113sin1	F111122sin1	F111123sin1	F111133sin1	kkkkkk
F111222sin1	F111223sin1	F111233sin1	F111333sin1	F112222sin1	F112223sin1	kkkkkk
F112233sin1	F112333sin1	F113333sin1	F122222sin1	F122223sin1	F122233sin1	kkkkkk
F122333sin1	F123333sin1	F133333sin1	F222222sin1	F222223sin1	F222233sin1	kkkkkk
F222333sin1	F223333sin1	F233333sin1	F333333sin1			kkkk
F111111cos1	F111112cos1	F111113cos1	F111122cos1	F111123cos1	F111133cos1	kkkkkk
F111222cos1	F111223cos1	F111233cos1	F111333cos1	F112222cos1	F112223cos1	kkkkkk
F112233cos1	F112333cos1	F113333cos1	F122222cos1	F122223cos1	F122233cos1	kkkkkk
F122333cos1	F123333cos1	F133333cos1	F222222cos1	F222223cos1	F222233cos1	kkkkkk
F222333cos1	F223333cos1	F233333cos1	F333333cos1			kkkk
…	…	…	…	…	…	...
F111111sin16	F111112sin16	F111113sin16	F111122sin16	F111123sin16	F111133sin16	kkkkkk
…	…	…	…	…	…	...
Phason (see page 139)						
phason						k
The atomic part of the 1st composite part - end						

Table 10 (Continued)

The molecular part[1] of the 1st composite part - beginning This example is for 1 molecule consisting of 1 atom and having 1 position, i.e. Nmol$_1$ = 1, Natm$_1$ = 1 and Npos$_1$ = 1. The introduction to the concept of molecules in *JANA98* is in page 140.						
Molecular header parameters (see page 141)						
Mname	IR	PointG	Mx	My	Mz	
Parameters of the model molecule (see page 142)						
See Atomic part in this table.						
The first molecular position - beginning						
Basic molecular parameters (see page 142)						
Pname	Sig	aimol	sm$_o$sm$_p$sm$_t$ wm$_o$ wm$_p$ wm$_t$ [2]			
phi	chi	psi	x-trans	y-trans	z-trans	kkkkkkk
rmx	rmy	rmz	rpx	rpy	rpz	
TLS tensors (see page 145)						
T11	T22	T33	T12	T13	T23	kkkkkk
L11	L22	L33	L12	L13	L23	kkkkkk
S11	S21	S31	S12	S22	S32	kkkkkk
S13	S23	S33				kkk
Molecular occupation modulation parameters, harmonic functions[3] (see page 149)						
om						k
omsin1	omcos1					kk
...	...					...
omsin16	omcos16					kk
...	...					...
Molecular occupation modulation parameters, crenel function[4] (see page 149)						
om						k
omsin1	omcos1					kk

---

[1] The concept of molecules in *JANA98* is described in page 140.

[2] This cell is only present for modulated structures.

[3] These lines are present in the case of molecular occupation modulation, i.e. for wm$_o$ > 0 (see page 142).

[4] These lines are present in the case of occupation modulation described by crenel function, i.e. for wm$_o$ > 0 and sm$_o$ = 1, (see page 142).

Table 10 (Continued)

Molecular position modulation parameters[1] (see page 148)						
xtsin1	ytsin1	ztsin1	xtcos1	ytcos1	ztcos1	kkkkkk
...	...	...	...	...	...	...
xtsin16	ytsin16	ztsin16	xtcos16	ytcos16	ztcos16	kkkkkk
xrsin1	yrsin1	zrsin1	xrcos1	yrcos1	zrcos1	kkkkkk
...	...	...	...	...	...	...
xrsin16	yrsin16	zrsin16	xrcos16	yrcos16	zrcos16	kkkkkk
Modulation of TLS tensors[2] (see page 150)						
T11sin1	T22sin1	T33sin1	T12sin1	T13sin1	T23sin1	kkkkkk
T11cos1	T22cos1	T33cos1	T12cos1	T13cos1	T23cos1	kkkkkk
...	...	...	...	...	...	...
T11sin16	T22sin16	T33sin16	T12sin16	T13sin16	T23sin16	kkkkkk
T11cos16	T22cos16	T33cos16	T12cos16	T13cos16	T23cos16	kkkkkk
L11sin1	L22sin1	L33sin1	L12sin1	L13sin1	L23sin1	kkkkkk
L11cos1	L22cos1	L33cos1	L12cos1	L13cos1	L23cos1	kkkkkk
...	...	...	...	...	...	...
L11sin16	L22sin16	L33sin16	L12sin16	L13sin16	L23sin16	kkkkkk
L11cos16	L22cos16	L33cos16	L12cos16	L13cos16	L23cos16	kkkkkk
S11sin1	S21sin1	S31sin1	S12sin1	S22sin1	S32sin1	kkkkkk
S13sin1	S23sin1	S33sin1				kkk
S11cos1	S21cos1	S31cos1	S12cos1	S22cos1	S32cos1	kkkkkk
S13cos1	S23cos1	S33cos1				kkk
...	...	...	...	...	...	...
...	...	...				...
S11sin16	S21sin16	S31sin16	S12sin16	S22sin16	S32sin16	kkkkkk
S13sin16	S23sin16	S33sin16				kkk
S11cos16	S21cos16	S31cos16	S12cos16	S22cos16	S32cos16	kkkkkk
S13cos16	S23cos16	S33cos16				kkk
Phason (see page 151)						
phasonm						k

---

[1] These lines are present in the case of molecular position modulation, i.e. for $wm_p > 0$ (see page 142).

[2] These lines are present in the case of molecular temperature modulation, i.e. for $wm_t > 0$ (see page 142). Note that only the atoms from the model molecule having $w_t < 0$ (see page 116) take part in the molecular temperature modulation refinement.

Table 10 (Continued)

The first molecular position - end
Here would begin the next molecular position
The molecular part[1] of the 1st composite part - end
THE FIRST COMPOSITE PART - END
THE SECOND COMPOSITE PART - BEGINNING
....
THE FIRST COMPOSITE PART - END
THE SECOND COMPOSITE PART ***The same parameters like for the 1st composite part***
THE THIRD COMPOSITE PART ***The same parameters like for the 1st composite part***
ESD'S OF THE FIRST, THE SECOND AND THE THIRD COMPOSITE PART ***Here are the estimated standard deviations listed in the same order like the parameters.***

---

[1] The concept of molecules in *JANA98* is described in page 140.

3.1.2 Commands section

This part of `m40` is optional and it is used only in special cases. The section starts with the keyword *commands* and ends with *end*. Inside the section there are keywords of commands followed by constants.

Maximal number of scale parameters

Usually `m40` contains six scale parameters in one line of the file. They are reserved for refinement of scales of various data sets or twin domains. If the number of scale parameters is insufficient it can be enlarged by command

maxsc nsc

, where nsc is typically 12 or 18, resulting in 2 or 3 lines of scale parameters. Note that if you change nsc you should also change correspondingly the number of scale parameters lines and placement of the scale refinement keys. These changes should be done by a text editor.

Definition of wave vectors

The harmonic modulation functions (see for instance equation E 20) are defined with respect to selected linear combinations of the modulation vectors, $\overline{\mathbf{q}}_i = \sum_{j=1}^{d} \alpha_{ij} \mathbf{q}_j$, where α_{ij} are integers. The combination can be set by the user with command

wave nwave nv_1 nv_2 ....

The wave vector with a wave number nwave is defined by nv_1*q1 + nv_2*q2 + ..., where q1, q2 ... are **q**-vectors. In the case of four-dimensional structures the default setting

wave 1 1
wave 2 2
wave 3 3
etc.

is usually the optimal one. For 5 or 6 dimensional structures the wave vectors often need to be redefined. The user interface of *EDITM40* for definition of wave vectors is shown in page 168.

Definition of orthogonalization parameters

The orthogonalization procedure is necessary in cases where the strong occupational modulation having step-like character is combined with harmonic modulations of other parameters (position, temperature parameters, …). The calculation of structure factors for the atom having this type of modulation requires proper selection and orthogonalization of the basic set of harmonic functions.

Theoretical

The selection and orthogonalization procedure is explained in page 133.

Setting or deleting orthogonalization parameters

The basic parameters (Δ, x_4^0) together with selection keys defining which `sin` and `cos` components of modulation waves are allowed in the calculation are defined in the header of `m40` by command

ortho name delta x40 n
[n1sin n1cos n2sin n2cos ..... n$_i$sin n$_i$cos [epsilon]],

where name is name of the atom, delta is width of the crenel or sawtooth function, n is a parameter reserved for future use, n$_i$sin and n$_i$cos are the selection keys for wave components and epsilon is defined in equation E 38.

In case that the second line is omitted all waves will be used in the orthogonalization procedure. With the second line present the functions having n$_i$sin or n$_i$cos equal to 0 will be excluded and only the remaining set of functions will be orthogonalized and used in the refinement.

The second line can be edited in a text editor or *via EDITM40* user interface. The parameter epsilon is saved for a successive use of *EDITM40*. However, epsilon is not used by the refinement and can be therefore omitted if the second line is edited with a text editor, i.e. without the *EDITM40* interface.

The following example shows the ortho command for atom As:

```
ortho As        0.500000  0.000000  1.000000
11100111100110011001111001100110    0.950
```

The numbers 111001111.... mean the sin component of the 1st wave will be used, cos of the 1st wave will be used, sin of the 2nd wave will be used, cos of the 2nd wave will not be used, sin of the 3rd wave will not be used .... etc. up to 2* `mxw` wave components (see § 1.1.4 page 15 for details about `mxw` parameter).

3.1.3 Header numbers

Header numbers *(This is part of table in page 98)*								
Nat$_1$	Nmol$_1$	Nat$_2$[1]	Nmol$_2$	Nat$_3$[2]	Nmol$_3$	Itemp	Irot	
Natm$_1$	Npos$_1$							
Natm$_2$	Npos$_2$	} Nmol$_1$ lines for the 1st composite subsystem						
......								

The header of `m40` contains number of atoms in atomic and molecular parts, number of molecules and molecular positions. In the case of a composite these numbers are listed repeatedly for each composite part.

The number of composite parts is given in `m50` (see the key **ncomp,** Table 9, page 80) and can be defined with *PRELIM* user interface (see § 2.2.2, page 68). The numbers for non-existing composite parts are omitted.

Meaning of parameters

Nat$_1$	Number of atoms in the 1st composite part.
Nmol$_1$	Number of molecules[3] in the 1st composite part
Nat$_2$	Number of atoms in the 2nd composite part.
Nmol$_2$	Number of molecules in the 2nd composite part
Nat$_3$	Number of atoms in the 3rd composite part.
Nmol$_3$	Number of molecules in the 3rd composite part
Itemp	Type of temperature parameters (0 for U, 1 for beta)
Irot	Key of molecular rotation (0 for Eulerian, 1 for axial). See page 143 for more information.

Natm$_1$	Number of atoms in the 1st molecule of the 1st composite part
Npos$_1$	Number of positions of the 1st molecule of the 1st composite part
Natm$_2$	Number of atoms in the 2nd molecule of the 1st composite part
Npos$_2$	Number of positions of the 2nd molecule of the 1st composite part

.... *etc. for all molecules of all composite parts*

The total number of symmetrically independent atoms in `m40` is a sum of atoms in all atomic parts + sum of atoms in all molecular parts:

$$\mathrm{Nall} = \mathrm{Nat}_1 + \mathrm{Nat}_2 + \mathrm{Nat}_3 + (\mathrm{Natm}_1 + \mathrm{Natm}_2 + \ldots.)_{1.\ \text{composite part}} + (\mathrm{Natm}_1 + \mathrm{Natm}_2 + \ldots.)_{2.\ \text{composite part}} + (\mathrm{Natm}_1 + \mathrm{Natm}_2 + \ldots.)_{3.\ \text{composite part}}$$

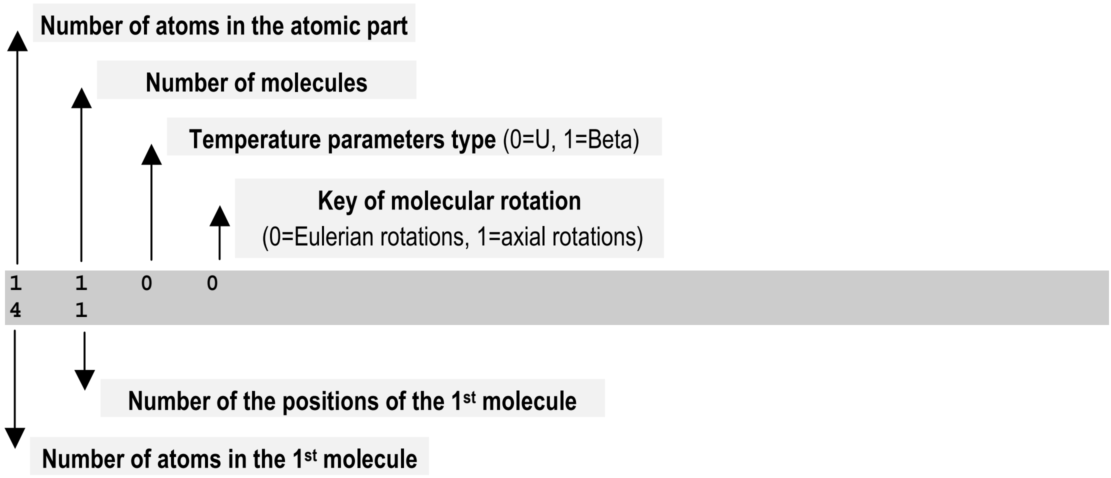
---

[1] The parameters Nat$_2$,Nmol$_2$ are present only when the number of composite parts is ≥ 2.

[2] The parameters Nat$_3$,Nmol$_3$ are present only when the number of composite parts equals 3.

[3] For the concept of molecules in *JANA98* see page 140.

Figure 65 *Example of the header of* `m40` *with 1 molecule and 1 composite part*



```
1   1   0   0
4   1
```

3.1.4 Scale parameters

Scale parameters[1] *(This is part of table in page 98)*						
`scale1`	`scale2`	`scale3`	`scale4`	`scale5`	`scale6`	kkkkkk
`scale7`	`scale8`	`scale9`	`scale10`	`scale11`	`scale12`	kkkkkk
`scale13`	`scale14`	`scale15`	`scale16`	`scale17`	`scale18`	kkkkkk

Usually `m40` contains six scale parameters in single line of the file. They are reserved for refinement of scales of various data sets or twin domains. If the number of scale parameters is insufficient it can be enlarged by command **mxsc** in the Commands section of `m40` (see page 105). The scale parameters are of two types: *Data scales* and *Fractional volumes*.

Meaning of parameters

`scale1,scale2,.....`	data scales
`scale18,scale17,....`	fractional volumes; for **mxsc** 12 they start with 12, for **mxsc** 6 they start with 6.

Data scales

The scale parameters for data sets or groups of reflections are allocated from the left of the `m40` line, i.e. the scale factor of the first data set is `scale1`. If there is no another scale factor `scale1` is the overall scale.

Typically the data scales are assigned when joining diffractometer or reflection files from various sources *via* the Import procedure of *PRELIM* (see page 72). In the Import form (see

---

[1] The 2nd and 3rd line of scale parameters is only present when the default number of scale parameters is changed by **maxsc** command (see page 105)

Figure 49, page 73) there is the `Scale factor number` textbox. The number typed here is valid for the imported data set and is saved in `m95` (see page 58) and `m91` (see page 87). It must correspond to the serial number of the Data scale in `m40`, i.e. the scale factor number 3 is refined as `scale3` etc.

Another way how to assign a Data scale is the **scale** command (see page 239) of *REFINE*. It is assigned via the SetCommands tool for *REFINE* (see page 230) and sets a special scale factor number for user-defined group of reflections. The number must again correspond to the serial number of the Data scale in `m40` but it is not connected with the scale numbers in `m91` or `m95`.

Fractional volumes

The scales of twin domains (fractional volumes) are allocated from the right of the `m40` scale line(s). The first fractional volume is a complementary quantity and is not present in `m40`. Therefore (for default number of 6 scales) the second fractional volume is refined as `scale6`. If the number of the scale parameters is enlarged by **mxsc** command the second fractional volume is `scalen`, where n is the maximal number of scale parameters.

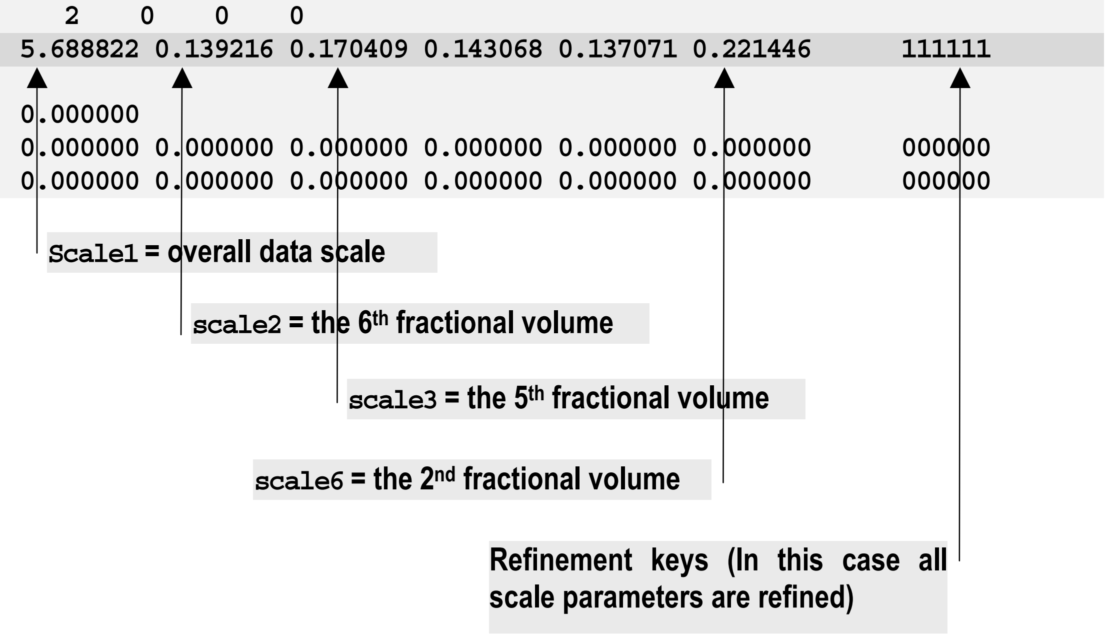
The number of fractional volumes is given by number of twin domains - see § 2.2.6, page 76. The assignment of the fractional volume numbers to the twin domains is done *via* the Import procedure of *PRELIM* (see page 72 and 75) during reading of the diffractometer or reflection file. The numbers are saved in `m95` (see page 58) and `m91` (see page 87) and must correspond with the serial numbers of the fractional volumes in `m40`, i.e. the fractional volume number 1 is not refined, the fractional volume number 2 is refined as `scale6` (assuming that maximal number of the scale parameters is 6), the fractional volume number 3 is refined as `scale5` etc.

The different fractional volume numbers in `m91` and `m95` are only necessary in the case of non-meroedric twinning. If all reflections can be indexed in all used domains, i.e. each index is transformed by every twinning matrix to another but integer index, then all reflections in `m91` and `m95` have the same fractional volume number. The Fractional volumes in `m40` are then independent of the fractional volume number in `m91` and the only condition the user should keep is that their number must be by 1 less than the number of twin domains (for instance for a 6-fold twin there is 5 Fractional volumes in `m40`).

Refinement keys of scale factors

The initial values of scale parameters and corresponding refinement keys (see page 97) are always set manually by the user. The only exception is `scale1`, which is always refined in the automatic mode. The details about refinements keys and automatic mode are explained in § 3.3.3, page 223. With default setting for refinement keys the user can follow a simple rule that scale1 is automatic but for another scales it is necessary to set manually both the initial values and the refinement keys.

Figure 66 *Example of* `m40` *with scale parameters for a 6-fold twin.*



```
   2    0    0    0
5.688822 0.139216 0.170409 0.143068 0.137071 0.221446      111111

0.000000
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
```

`Scale1` = overall data scale

`scale2` = the 6th fractional volume

`scale3` = the 5th fractional volume

`scale6` = the 2nd fractional volume

Refinement keys (In this case all scale parameters are refined)

3.1.5 Overall isotropic thermal parameter

This is constant, which is not refined and is added to all isotropic thermal parameters.

3.1.6 Extinction parameters

Extinction parameters *(This is part of table in page 98)*						
`rho11/` `rhoiso`	`rho22`	`rho33`	`rho12`	`rho13`	`rho23`	kkkkkk
`g11/giso`	`g22`	`g33`	`g12`	`g13`	`g23`	kkkkkk

Meaning of parameters

`rhoiso`	The ratio of the particle size (μm) to the wavelength (Å). This is Type II isotropic extinction correction.
`rho11, ... , rho23`	The anisotropic components of the Type II extinction correction.
`giso`	The width of the Lorentzian/Gaussian distribution function. This is Type I isotropic extinction correction.
`g11,...,g23`	The anisotropic components of the Type I extinction correction.
kkkkkk	refinement keys belonging to parameters in the same line

JANA98 works with several types of the secondary extinction correction:

- Isotropic Type I, Gaussian distribution.
- Isotropic Type I, Lorentzian distribution.
- Isotropic Type II
- Isotropic mixed types, Gaussian distribution.
- Isotropic mixed types, Lorentzian distribution.
- Anisotropic Type I, Gaussian distribution.
- Anisotropic Type I, Lorentzian distribution.
- Anisotropic Type II

The anisotropic extinction has not been completely finished and its use should be first consulted with the authors.

The refined anisotropic extinction parameters are `g11, g22, g33, g12, g13, g23` for the case of Gaussian distribution and `rho11, rho22, rho33, rho12, rho13, rho23` for Lorentzian distribution. The isotropic parameters are `giso` and `rhoiso` for the Gaussian and Lorentzian distribution, respectively. In `m40` the parameter `g11` shares place with `giso`, while `rho11` is at the same place like `rhoiso`.

The extinction parameters are interconnected with the extinction keys in the section for *REFINE* in `m50`. They can be edited *via* the Extinction form (page 243) of the SetCommands tool of *REFINE*.

Figure 67 *Example of* `m40` *with isotropic extinction of type I with Lorentzian distribution.*

```
   5    0    0    0
1.263756 0.000000 0.000000 0.000000 0.000000 0.000000      100000
0.000000
6.524139 0.000000 0.000000 0.000000 0.000000 0.000000      100000

0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
                                                 . . . .
```

`rhoiso`

The refinement key of `rhoiso`

Theoretical

JANA98 allows to extinction correction according to the theoretical models and formulas derived by P.J.Becker and P. Coppens, (1974) in Acta Cryst, **A30,** 129-147. The notation used here is taken from that paper and it also well corresponds to that defined by W.H.Zachariasen, (1967) in Acta Cryst, **23**, 558-564.

The integrated intensity P_k of the Bragg reflection according to the kinematical approximation can be calculated from the intensity of the incident beam I_0 and the volume of the irradiated volume v:

$$\mathsf{P}_\mathsf{k} = \mathsf{I}_0 v Q \tag{E1}$$

The value Q is the average scattering cross section per unit volume:

$$Q = \left|\frac{aFK}{V}\right|^2 \lambda^3 / \sin 2\theta \tag{E2}$$

F is the structure factor, K is polarisation factor (1 for neutron or synchrotron radiation and for the parallel component of X-ray electric field and $\cos\theta$ for the perpendicular component of X-ray electric field), V is the unit cell volume, θ is the Bragg angle, λ is the wavelength of the used radiation. The constant factor a is 10^{-12} cm for neutrons and $\frac{e^2}{mc^2} \approx 2.81776.10^{-13}$ cm for X-rays.

JANA98 can only refine the secondary extinction correction. Its influence can be expressed by the factor y:

$$\mathsf{P} = \mathsf{P}_\mathsf{k} . y \tag{E3}$$

The secondary correction is dependent on the used assumption concerning angular distribution W of the crystalline domains in the crystal. There are two most generally used models with Gaussian and Lorentzian distribution:

$$W_G(\varepsilon) = \sqrt{2}g\exp\left(-2\pi g^2\varepsilon^2\right) \qquad W_L(\varepsilon) = 2g/\left(1+4\pi^2\varepsilon^2 g^2\right) \tag{E4}$$

, where g is the width of the distribution.

The correction factor y can be according to P.Becker and P.Coppens approximated by the formula:

$$y(x,\theta) = \left\{1 + \xi_{G,L}x + \frac{A(\theta)x^2}{1+B(\theta)x}\right\}^{-1/2} \qquad x = \frac{2}{3}Q\alpha_{G,L}\bar{t} \tag{E5}$$

$$\alpha_G = \bar{\alpha}\Big/\left(1 + \frac{\bar{\alpha}^2}{2g^2}\right)^{1/2} \qquad \xi_G = 2.12 \tag{E6}$$

$$\alpha_L = \bar{\alpha}\Big/\left(1 + \frac{2\bar{\alpha}}{3g}\right) \qquad \xi_L = 2 \tag{E7}$$

The parameter $\bar{\alpha} = \frac{3}{2}\frac{r}{\lambda}\sin 2\theta = \frac{3}{2}\rho\sin 2\theta$, where r is the particle size.

The program allows to correct for three different types of isotropic secondary extinction for which there are some differences in used functions $A(\theta), B(\theta)$.

Mixed type

The mixed type makes no other assumptions and uses the expressions for x as defined above. The functions $A(\theta), B(\theta)$ have the following form:

Gaussian distribution:

$$\begin{aligned} A(\theta) &= 0.58 + 0.48\cos 2\theta + 0.24\cos^2 2\theta \\ B(\theta) &= 0.02 - 0.025\cos 2\theta \end{aligned} \tag{E8}$$

Lorentzian distribution:

$$\begin{aligned} A(\theta) &= 0.025 + 0.285\cos 2\theta \\ B(\theta) &= 0.15 - 0.2(0.75 - \cos 2\theta)^2 \quad && \cos 2\theta > 0 \\ B(\theta) &= -0.45\cos 2\theta && \cos 2\theta < 0 \end{aligned} \tag{E9}$$

The extinction depends both of the particle size (parameter ρ) and the width of the distribution *g* and both can be refined.

Type I – extinction being dominated by distribution

In the case when $\bar{\alpha} >> g$ the expressions for $\alpha_{G,L}$ are reduced to:

$$\alpha_G = \sqrt{2}\,g \quad \alpha_L = \frac{3}{2}g \tag{E10}$$

The extinction is then independent of the particle size and only parameter to be refined is the width of the distribution *g*.

The functions $A(\theta), B(\theta)$ have the same form as for the mixed type (see above).

Type II – extinction being dominated by particle size

In the case when $\bar{\alpha} << g$ the expressions for $\alpha_{G,L}$ are reduced to:

$$\alpha_{G,L} = \bar{\alpha} \tag{E11}$$

The extinction is then independent of the width of the distribution particle size and only parameter which to be refined is the particle size $\bar{\alpha}$.

The functions $A(\theta), B(\theta)$ have the same form as for the primary extinction, see Becker&Coppens (28):

$$\begin{aligned} A(\theta) &= 0.20 + 0.45\cos 2\theta \\ B(\theta) &= 0.22 - 0.12(0.5 - \cos 2\theta)^2 \end{aligned} \tag{E12}$$

The program *JANA98* allows using all mentioned types. The mean path $\bar{t}$ [cm] for each reflection is recorded in the file `m95` during the numerical absorption correction. Then it is copied to `m91` and used in the refinement. In case that $\bar{t}$ is not at disposal the program uses the radius defined in the form for the extinction and the following equation for a spherical crystal of radius *R*:

$$\bar{t} = \frac{3}{2}R \tag{E13}$$

The parameters ρ and g can be refined by the least square program REFINE. The recorded values are multiplied by 10^4. This means that the parameter ρ gives the ratio of the particle size in micrometers to the wavelength in Angstroms.

The anisotropic extinction has not been completely finished and its use should be first consulted with the authors.

Table 11 *The meaning of the extinction parameters in* JANA98

Type of the correction	**Refined parameters and their meaning**
Isotropic Type I, Gaussian	`giso` the width of the Gaussian distribution
Isotropic Type I, Lorentzian	`giso` the width of the Lorentzian distribution
Isotropic type II	`rhoiso` the ratio of the particle size in micrometers to the wavelength in Angstroms.
Isotropic mixed	`giso` and `rhoiso`
Anisotropic Type I Gaussian	`g11 . . .g23`
Anisotropic Type II, Lorentzian	`g11 . . .g23`
Anisotropic Type II	`rho11 . . .rho23`

3.1.7 Atomic header parameters

Atomic header parameters *(This is part of table in page 98)*							
Name	Chtype	Ttype	`ai`	`x`	`y`	`z`	$S_oS_pS_t$ W_o W_p W_t [1]
Name[2]							$S_{t3}S_{t4}S_{t5}S_{t6}$ W_{t3} W_{t4} W_{t5} W_{t6} [3]

The atomic header lines contain the basic information about the atom whose parameters are refined, the number and type of parameters which follow in the next part of `m40`, occupation and positional parameters. The four refinement keys belonging to refinable parameters `ai`, `x`, `y` and `z` are placed together with the refinement keys of temperature parameters (see page 120 and Figure 68).

Parameters meaning

Name	the name of atom. It is repeated in the second header line for clarity.
Chtype	the reference number of chemical type. This is the serial number of the atom in the atomic form factors list in `m50`, i.e. the first atom in `m50` has Chtype=1 etc. See § 0, page 77 for more information about `m50`.
Ttype	the type of temperature parameters. 1=isotropic, 2=anisotropic, 3,4,5 and 6 = anharmonic atomic displacement tensors of the 3rd, 4th, 5th and 6th order, respectively. If the atom is part of a model molecule (see page 142) Ttype can also be equal to 0 which means that the temperature parameter is refined as molecular TLS parameters (see page 145).
`ai`	site occupation. In convention of *JANA98* `ai` of a symmetric position is reciprocal value of its multiplicity.
`x,y,z`	positional parameters. For a modulated structure refined with modulation waves `x,y,z` is a position in the basic structure. For a modulated structure refined *without* modulation waves on main reflections `x,y,z` is a position in the average structure.
S_o	key of special function for occupancy modulation. 0 means ordinary function, 1 means special function. The special function for occupancy modulation is the crenel function (page 128).
S_p	key of special function for position modulation. 0 means ordinary function, 1 means special function. The special function for position modulation is the sawtooth function (page 124).
S_t	key of special function for temperature modulation. 0 means ordinary function, 1 means special function. Currently no special function for temperature modulation is available.
W_o[4]	number of occupation modulation waves
W_p	number of position modulation waves

---

[1] This cell is only present for modulated structures.

[2] This line is omitted for Ttype less or equal to 2, i.e.for atoms without anharmonic displacement parameters.

[3] This cell is only present for modulated structures.

[4] The maximal number of modulation waves is 16. In Unix version it can be changed during the compilation of the program - see § 1.1.4 page 15.

w_t	number of temperature modulation waves
s_{t3}, s_{t4}, s_{t5},s_{t6}	key of special function for the 3rd , 4th, 5th and 6th order anharmonic displacement tensors. 0 means ordinary function, 1 means special function. Currently no special function for temperature modulation is available.
w_{t3}, w_{t4}, w_{t5},w_{t6}	number of modulation waves for the 3rd , 4th, 5th and 6th order anharmonic displacement tensors.

The atomic header parameters are also used for atoms of a model molecule - see page 142.

Setting the atomic header line parameters

The name, the reference number of chemical type, occupation and position parameters are entered *via* the *Replacing/Inserting atoms* interface of *EDITM40* (see page 159). For setting of the type of temperature parameters (Ttype) there are three tools in *EDITM40*: *Temperature parameters* (page 164) converts between anisotropic and isotropic parameters. *Beta<->U* (page 164) converts between U and beta type and *Adding or deleting anharmonic tensors* (page 165) sets the anharmonic displacement parameters (ADP). The parameters defining the number of modulation waves are set together with the initial values of modulation parameters with *Setting or deleting of modulation waves* (page 168).

Figure 68 *An atom in* `m40` *with anisotropic temperature parameters.*

```
Name  Chtype  Ttype      ai        x         y         z
Ce         3  2     0.250000 0.000000 0.386452 0.250000
 0.002720 0.005093 0.003850 0.000000 0.000000 0.000000      0010111000
   U11      U22       U33      U13      U13      U23       refinement keys for
                                                             ai,x,y,z,Uij
```

Figure 69 *An atom in* `m40` *with ADP of the 3rd order.*

The next two lines have the same meaning like in **Figure 68**.

```
La2        2  3     0.500000 0.000000 0.000000 0.326373
 0.054580 0.022898 0.023297 0.000000 0.000000 0.002195      0001111001
```

The next two lines contain components of the 3rd order anharmonic tensor followed by their refinement keys

```
0.000000-0.031857 0.000651 0.000000 0.000000 0.000000      011000
-0.027452-0.000329-0.000498-0.000050                        1111
```

Figure 70 *An atom in* `m40` *with position and temperature modulation.*

```
                                                         So Sp St   Wo  Wp  Wt
```

The next two lines have the same meaning like in **Figure 68**.

```
O3         4  2     1.000000 0.259540 0.704262 0.334582      000  0  2  2
 0.033601 0.045856 0.050422-0.018620 0.018743-0.023155      0111111111
```

The next two lines contain the position modulation parameters, one line per one modulation wave, followed by their refinement keys.

```
 0.001588 0.012132 0.014724 0.049204-0.017804 0.060715      111111
 0.007225-0.002704 0.000271-0.004372-0.002479 0.004139      111111
```

The next four lines contain the temperature modulation parameters, two lines per one modulation wave, followed by their refinement keys

```
 0.001844 0.003826 0.005253-0.001272 0.001209-0.002663      111111
-0.008377 0.003772 0.004731 0.005381-0.003017-0.006556      111111
-0.002579 0.015193 0.003039-0.005828-0.001797-0.012093      111111
-0.003972-0.014366-0.017415 0.013566-0.014126 0.018702      111111
```

The phason parameter

```
 0.000000                                                   0
```

Figure 71 *An atom in* `m40` *with modulation of the 3rd order ADP.*

S_o S_p S_t
S_{t3} S_{t4} S_{t5} S_{t6}

W_o W_p W_t
W_{t3} W_{t4} W_{t5} W_{t6}

The 1st and the 3rd lines have the same meaning like in the previous fi
The 2nd line is the extended atomic header for the case of modulation of ADP.

```
La2        2  3     0.500000 0.000000 0.000000 0.326373      000  0  2  2
La2                                                          0000  2  0  0  0
 0.054580 0.022898 0.023297 0.000000 0.000000 0.002195      0001111001
```

The next two lines contain components of the 3rd order anharmonic tensor followed by their refinement keys

```
 0.000000-0.031857 0.000651 0.000000 0.000000 0.000000      011000
-0.027452-0.000329-0.000498-0.000050                        1111
```

The next two lines contain the position modulation parameters, one line per one modulation wave, followed by their refinement keys.

```
 0.001376 0.000000 0.000000 0.000000 0.016677-0.001907      100011
-0.010213 0.000000 0.000000 0.000000-0.011741 0.000315      100011
```

The next four lines contain the temperature modulation parameters, two lines per one modulation wave, followed by their refinement keys

```
 0.000000 0.000000 0.000000-0.009427-0.003535 0.000000      000110
 0.010381-0.002141-0.001840 0.000000 0.000000 0.004628      111001
 0.000000 0.000000 0.000000-0.020498-0.003375 0.000000      000110
-0.026560 0.002990-0.010303 0.000000 0.000000-0.011721      111001
```

The next eight lines contain the modulation parameters of the 3rd order anharmonic tensor, four lines per one modulation wave, followed by their refinement keys

```
 0.019411 0.000000 0.000000 0.001126 0.000155-0.000331      100111
 0.000000 0.000000 0.000000 0.000000                        0000
 0.000000 0.005532-0.002968 0.000000 0.000000 0.000000      011000
 0.005339-0.001135-0.000068-0.000182                        1111
-0.042605 0.000000 0.000000 0.000936-0.003285-0.000959      100111
 0.000000 0.000000 0.000000 0.000000                        0000
 0.000000 0.038119-0.000733 0.000000 0.000000 0.000000      011000
-0.037520 0.001431-0.000268 0.000188                        1111
```

The phason parameter

```
 0.000000
```

3.1.8 Temperature parameters

Atomic temperature parameters *(This is part of table in page 98)*						
`U11`	`U22`	`U33`	`U12`	`U13`	`U23`	*kkkk*kkkkkk

This part contains the parameters of the isotropic or anisotropic temperature factor. By default the temperature factor in *JANA98* takes so called "U-form", i.e. it is defined by expression E14 and E15 for the anisotropic and isotropic case, respectively. The alternative definition is so called "beta-form" - see expressions E16 and E17. The key Itemp in the header of `m40` (see page 107) defines whether the temperature parameters are in the U-form or Beta-form. The conversion of `m40` between these two forms can be made *via* the *Beta<->U* command of *EDITM40* (see page 164).

In the isotropic case the refined parameter is `U11` for both the U-form and Beta-form of the temperature factor. The corresponding refinement key is the 5th key from 10 refinement keys in the line containing the temperature parameters because the first four refinement keys belong to the occupation and position(see page 116 and Figure 68, page 118).

In the anisotropic case the refined parameters are `U11` - `U23` for both the U-form and Beta-form and the corresponding refinement keys are the 5th - 10th ones.

The expressions defining various forms of the temperature factor

Anisotropic case, U-form:

$$\exp\left[-2\pi^2\left(U_{11}h^2a^{*2}+U_{22}h^2b^{*2}+U_{33}h^2c^{*2}+U_{12}hka^*b^*+U_{13}hla^*c^*+U_{23}klb^*c^*\right)\right] \tag{E14}$$

Isotropic case, U-form:

$$\exp\left(-8\pi^2U\frac{\sin^2\theta}{\lambda^2}\right) \tag{E15}$$

Anisotropic case, Beta-form:

$$\exp\left(\beta_{11}h^2+\beta_{22}h^2+\beta_{33}h^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl\right) \tag{E16}$$

Isotropic case, Beta-form:

$$\exp\left(-\beta\frac{\sin^2\theta}{\lambda^2}\right) \tag{E17}$$

3.1.9 Anharmonic displacement parameters (ADP)

Anharmonic displacement parameters (ADP) for atomic part *(This is part of table in page 98)*							
C111	C112	C113	C122	C123	C133	kkkkkk	3rd
C222	C223	C233	C333			kkkk	
D1111	D1112	D1113	D1122	D1123	D1133	kkkkkk	4th
D1222	D1223	D1233	D1333	D2222	D2223	kkkkkk	
D2233	D2333	D3333				kkk	
E11111	E11112	E11113	E11122	E11123	E11133	kkkkkk	5th
E11222	E11223	E11233	E11333	E12222	E12223	kkkkkk	
E12233	E12333	E13333	E22222	E22223	E22233	kkkkkk	
E22333	E23333	E33333				kkk	
F111111	F111112	F111113	F111122	F111123	F111133	kkkkkk	6th
F111222	F111223	F111233	F111333	F112222	F112223	kkkkkk	
F112233	F112333	F113333	F122222	F122223	F122233	kkkkkk	
F122333	F123333	F133333	F222222	F222223	F222233	kkkkkk	
F222333	F223333	F233333	F333333			kkkk	

The parameter ltemp (see page 116) determines whether the atomic displacement will be described by isotropic, anisotropic or anharmonic tensor. The order of the ADP and their initial values are set *via* the tool *Setting or deleting anharmonic tensors* of *EDITM40* (see page 165). A simple example of `m40` with the 3rd order ADP is shown in Figure 69).

Meaning of parameters

`C111,...,C333`	The independent contravariant component of tensor C (see equation E 18, page 122) multiplied by 10^3.
`D1111,...,D3333`	The independent contravariant component of tensor D (see equation E 18, page 122) multiplied by 10^4.
`E11111,...,E33333`	The independent contravariant component of tensor E (see equation E 18, page 122) multiplied by 10^5.
`F111111,...,F333333`	The independent contravariant component of tensor F (see equation E 18, page 122) multiplied by 10^6.
kkkkkk	refinement keys belonging to the parameters in the same line. The refinement program (similar as for all parameters) can derive the symmetry restrictions following from the site symmetry automatically.

Theoretical

Non-harmonic ADP (atomic displacement parameters) used in the program are based on the Gram-Charlier expansion of the structure factor:

$$\begin{aligned} F(\mathbf{H}) = \sum_{\nu=1}^{n} f_\nu(|\mathbf{H}|)\exp(2\pi i\mathbf{r}_\nu \cdot \mathbf{H})\exp(-\beta^{ij}h_ih_j)\cdot \\ (1 - iC^{ijk}h_ih_jh_k + D^{ijkl}h_ih_jh_kh_l + iE^{ijklm}h_ih_jh_kh_lh_m - iF^{ijklmn}h_ih_jh_kh_lh_mh_n) \end{aligned} \quad \text{(E 18)}$$

The tensors **C, D, E** and **F** are the symmetrical tensors of order 3,4,5 and 6, respectively.

More details concerning theory of ADP can be found in Kuhs (1992) and the references herein.

Setting or deleting ADP

The anharmonic displacement parameters can be set in *EDITM40* - see page 165.

3.1.10 Position modulation parameters

Position modulation parameters, harmonic functions *(This is part of table in page 98)*						
xsin1	ysin1	zsin1	xcos1	ycos1	zcos1	kkkkkk
...	...	...	...	...	...	...
xsin16	ysin16	zsin16	xcos16	ycos16	zcos16	kkkkkk
Position modulation parameters, sawtooth function						
xsin$[1]	ysin$	zsin$	xcos$	ycos$		kkkkk0

The type of position modulation parameters is defined by s_p and w_p (see the atomic header parameters, page 116). With $s_p = 0$ and $w_p > 0$ the modulation is described as a linear combination of w_p harmonic modulation waves. With $s_p = 1$ and $w_p = 1$ the position modulation is expressed by a simple sawtooth function. For $s_p = 1$ and $w_p > 1$ the modulation is linear combination of harmonic functions (w_p -1 waves) and the sawtooth function.

Theoretical

In the case of a *d*-dimensional modulated crystal the νth atom is located at

$$\mathbf{r}_{\mathbf{n}\nu} = \mathbf{r}_\nu^0 + \mathbf{n} + \mathbf{u}_\nu\{\mathbf{q}_1(\mathbf{g}_\nu + \mathbf{n}),....,\mathbf{q}_d(\mathbf{g}_\nu + \mathbf{n})\}, \qquad (E\ 19)$$

where $\mathbf{r}_\nu^0$ is the average position of atom ν, $\mathbf{n}$ represents the lattice translations, $\mathbf{u}_\nu$ is the *d*-dimensional periodic vector field $\mathbf{u}_\nu(x_1,....,x_d) = \mathbf{u}_\nu(x_1 + n_1,....,x_d + n_d)$, $n_1, ..., n_d$ being integers, $\mathbf{q}_1,....,\mathbf{q}_d$ are incommensurate modulation vectors, which are rationally independent in the basis $\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*$; $\mathbf{g}_\nu$ is the phase reference point, which is equal to the atomic position for an atom not part of a rigidly displaced entity.

Harmonic position modulation

The vector field $\mathbf{u}_\nu$ is the general modulation function of the atomic position. It can be expanded into a truncated Fourier series:

$$\mathbf{u}_\nu = \sum_{i=1}^{l} \mathbf{U}_\nu^s(i)\sin[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n} + \mathbf{g}_\nu)] + \sum_{i=1}^{l} \mathbf{U}_\nu^c(i)\cos[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n} + \mathbf{g}_\nu)], \qquad (E\ 20)$$

where

$$\overline{\mathbf{q}}_i = \sum_{j=1}^{d} \alpha_{ij}\mathbf{q}_j, \qquad (E\ 21)$$

---

[1] $ symbolises the sawtooth function parameters are always in the place of the last wave - see page 123 for details.

α_{ij} being integers, are selected linear combinations of the modulation vectors and $\mathbf{U}_{\nu}^{s}$, $\mathbf{U}_{\nu}^{c}$ are the amplitudes of the sin and cos displacement waves, respectively.

Sawtooth position modulation

Sometimes the displacement $\mathbf{u}_{\nu}$ can be more effectively described as a linear function rather than by harmonic function shown in equation E 20. In *JANA98* the linear position modulation function is called sawtooth function. Its usage is limited to 4 dimensions. The sawtooth function can be expressed like

$$\mathbf{u}_{\nu} = 2\mathbf{u}_{\mathbf{0},\nu}\left[\left(x_4 - x_4^0\right)/\Delta\right] \quad \text{for}\left(x_4^0 - \Delta/2 < x_4^0 < x_4^0 + \Delta/2\right), \qquad (E\ 22)$$

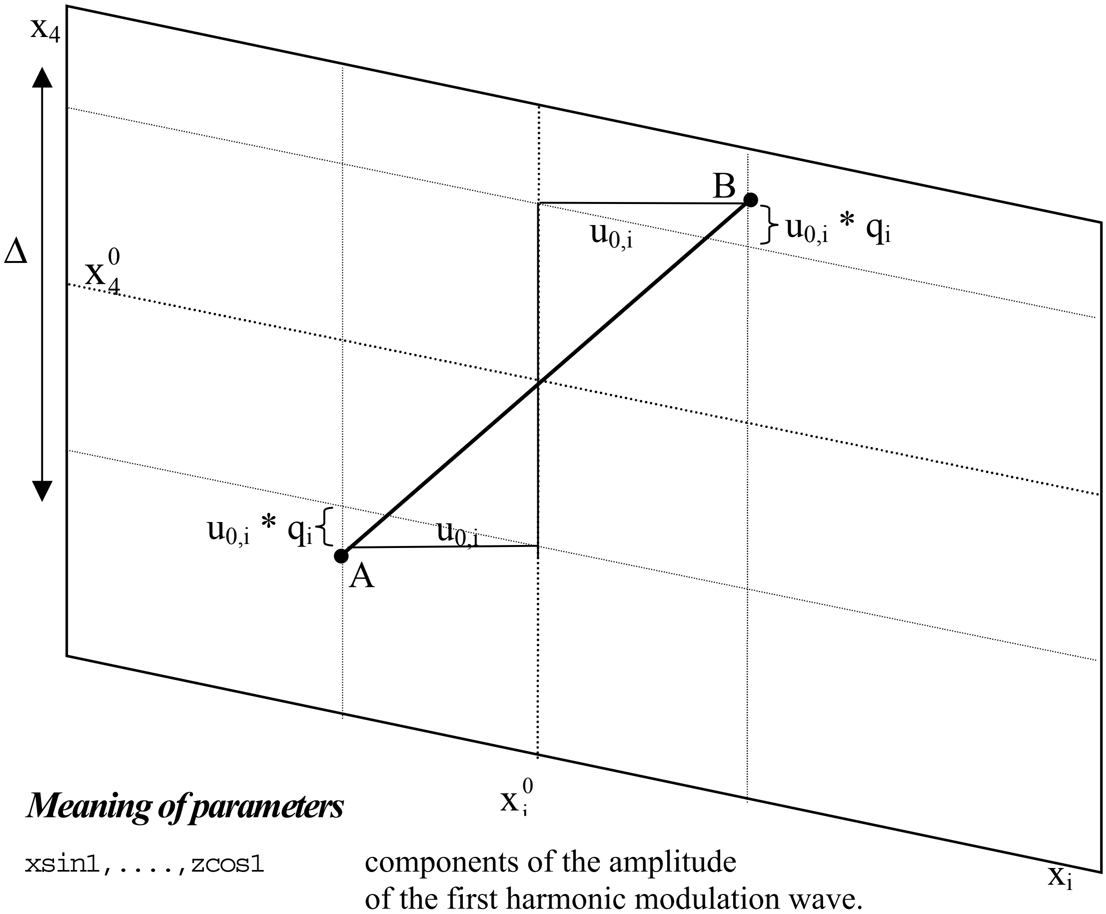
where $\mathbf{u}_{\nu}$ is the displacement of the *ν*th atom, $\mathbf{u_0}$ is the maximal displacement and Δ is the definition interval of the sawtooth function.

An example is given in Figure 72, where the sawtooth function defined by the center x_4^0, by the width Δ and the maximal displacement $\mathbf{u}_0$ is plotted in the x_i-x_4 projection. For $x_4 = x_4^0$ the displacement from the basic atomic position is 0. With increasing x_4, the displacement increases linearly until the maximal value $\mathbf{u_0}$. When x_4 reaches the point A, the atom disappears and does not exist until point B appears again in the next cell with the opposite maximal displacement $\mathbf{u_0}$.

It should be noted that sawtooth function describes simultaneously the position modulation and occupation modulation.

Figure 72 *An example of a sawtooth modulation function (abscissa A-B) in the x_4-x_i projection.*

Δ, x_4^0 and $u_{0,i}$ define the width of the function, the center of the function and the maximal displacement from the basic position, respectively. The occupation of the atom described by this function is equal to Δ.



Meaning of parameters

`xsin1,....,zcos1`	components of the amplitude of the first harmonic modulation wave. They correspond to vector components $U^s_{v,1}(1), U^s_{v,2}(1), U^s_{v,3}(1), U^c_{v,1}(1), U^c_{v,2}(1), U^c_{v,3}(1)$ in equation E 20.
`xsin$`	x_1 component of amplitude of the sawtooth function ($u_{0,1}$ in equation E 22)
`ysin$`	x_2 component of amplitude of the sawtooth function ($u_{0,2}$ in equation E 22)
`zsin$`	x_3 component of amplitude of the sawtooth function ($u_{0,3}$ in equation E 22)
`xcos$`	the center of the sawtooth function in x_4 ($2\,x_4^0$ in equation E 22)
`ycos$`	projection of a sawtooth function to the x_4 axis (Δ in equation E 22)
`zcos$`	unused
kkkkkk	refinement keys belonging to the parameters in the same line

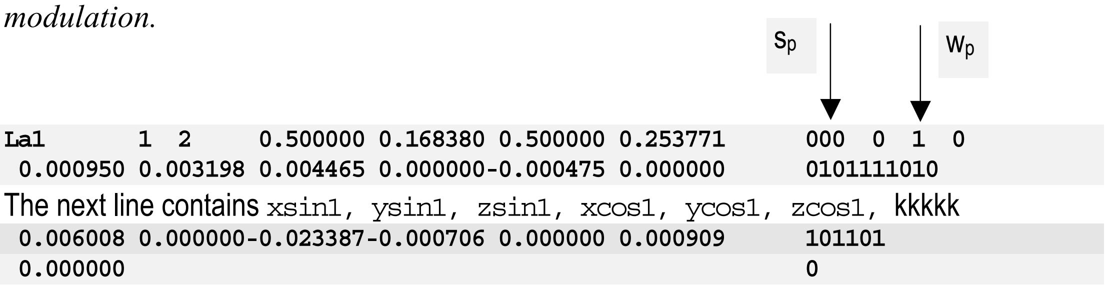
Selection of wave vectors

The vector components $\overline{\mathbf{q}}_i$ (see equation E 21) are linear combinations of q-vectors defined by user. The combination can be defined by the **wave** command (see page 105). In the case of one dimensional modulation there is usually no reason to change the default definition of $\overline{\mathbf{q}}_j = j\mathbf{q}$.

Setting or deleting position modulation

Provided that the dimensions and modulation vector(s) have been properly set in *PRELIM* (see page 68) the number of harmonic waves can be changed in *EDITM40* (page 168). With the automatic setting for refinement keys (see §3.3.3 page 223 for details) the symmetry restrictions of the wave components are made automatically.
The setting of a sawtooth function is described in page 286. It can be applied together with harmonic modulation functions - see page 132 for details.

Figure 73 *Example of an atom in the atomic part of* `m40` *with harmonic position modulation.*

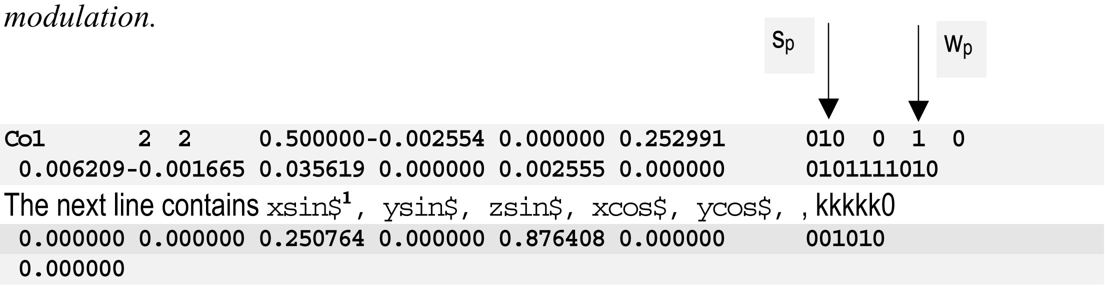


```
La1       1  2     0.500000 0.168380 0.500000 0.253771      000  0  1  0
 0.000950 0.003198 0.004465 0.000000-0.000475 0.000000      0101111010
```

The next line contains `xsin1, ysin1, zsin1, xcos1, ycos1, zcos1,` kkkkk

```
 0.006008 0.000000-0.023387-0.000706 0.000000 0.000909      101101
 0.000000                                                   0
```

Figure 74 *Example of an atom in the atomic part of* `m40` *with sawtooth position modulation.*



```
Co1       2  2     0.500000-0.002554 0.000000 0.252991      010  0  1  0
 0.006209-0.001665 0.035619 0.000000 0.002555 0.000000      0101111010
```

The next line contains `xsin$`[1]`, ysin$, zsin$, xcos$, ycos$,` , kkkkk0

```
 0.000000 0.000000 0.250764 0.000000 0.876408 0.000000      001010
 0.000000
```

---

[1] $ symbolises the sawtooth function parameters are always in the place of the last wave - see page 123 for details.

3.1.11 The occupation modulation parameters

Occupation modulation parameters, harmonic functions *(This is part of table in page 98)*						
o						k
osin1	ocos1					kk
...	...					...
osin16	ocos16					kk
Occupation modulation parameters, Crenel function						
o						k
osin1	ocos1					kk

The type of occupation modulation parameters is defined by s_o and w_o (see the atomic header parameters, page 116). With $s_o = 0$ and $w_o > 0$ the modulation is described by w_o harmonic modulation waves. With $s_o = 1$ and $w_o = 1$ the occupation modulation is expressed by a crenel function. The combinations $s_o \neq 0$ and $w_o \neq 1$ are illegal leading to an unpredictable behaviour of the refinement.

Theoretical

Similarly as for position (see equation E 19) we can write

$$p_{n\nu} = p_\nu^0 + p_\nu\left[\mathbf{q}_1\cdot(\mathbf{g}_\nu+\mathbf{n}),....,\mathbf{q}_d\cdot(\mathbf{g}_\nu+\mathbf{n})\right], \qquad \textit{(E 23)}$$

where $p_{n\nu}$ and p_ν^0 are occupation in the nth cell and the average occupation of atom ν, respectively. $\mathbf{g}_\nu$ and $\mathbf{n}$ are explained in E 19. p_ν is the d-dimensional periodic scalar function

$$p_\nu(x_1,....,x_d) = p_\nu(x_1+n_1,....,x_d+n_d), \qquad \textit{(E 24)}$$

$n_1, ..., n_d$ being integers.

Harmonic occupation modulation

Similarly, as for $\mathbf{u}_\nu$ (see equation E 20), p_ν can be expanded into a Fourier series:

$$p_\nu = \sum_{i=1}^{l} p_\nu^s(i)\sin[2\pi\overline{\mathbf{q}}_i\cdot(\mathbf{n}+\mathbf{g}_\nu)] + \sum_{i=1}^{l} p_\nu^c(i)\cos[2\pi\overline{\mathbf{q}}_i\cdot(\mathbf{n}+\mathbf{g}_\nu)], \qquad \textit{(E 25)}$$

where the summation is over all occupation waves[1]. $\overline{\mathbf{q}}_i$ are selected linear combinations of the modulation vectors (see equation E 21) and p_ν^s, p_ν^c are the amplitudes of the sin and cos occupation waves, respectively. The choice of the upper limit *l* define the actual truncation of the infinite formula.

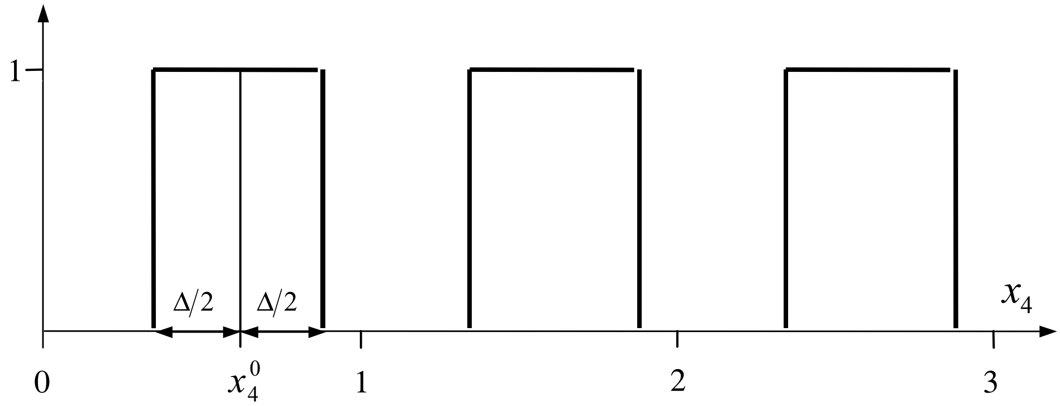
Crenel occupation modulation

Very often modelling the proper shape of the occupation modulation function requires a large number of harmonics. In these cases more efficient way is to use crenel function defined as

$$\begin{aligned} p_\nu(x_4) &= 1 \qquad x_4 \in \left\langle x_4^0 - \Delta/2, x_4^0 + \Delta/2 \right\rangle \\ p_\nu(x_4) &= 0 \qquad x_4 \notin \left\langle x_4^0 - \Delta/2, x_4^0 + \Delta/2 \right\rangle \end{aligned} \tag{E 26}$$

Usage of crenel function in *JANA98* is limited to 4 dimensions. Normally it is applied together with harmonic modulation functions - see page 132 for details.

Figure 75 *The crenel (periodic step-like) occupation wave:*



Meaning of parameters

For harmonic functions

`o`	average occupation of atom *ν;* p_ν^0 in equation E 23.
`osin1,ocos1`	amplitudes of the sin and cos occupation waves (p_ν^s and p_ν^c in equation E 25)

For crenel function

`o`	the width of the crenel function (Δ in equation E 26)
`osin1`	the center of the crenel function (x_4^0 in equation E 26)
`ocos1=1`	Reserved for a future use. Should not be refined or modified.

---

[1] Note that in the case of pure occupation modulation the harmonic wave the *m*th order gives raise to only *m*th order satellites.

Figure 76 *Example of an atom in the atomic part of* `m40` *with harmonic occupation modulation.*

So Wo

```
Na3       4  2     0.500000 0.118274 0.126328 0.500000      000  1  1  1
 0.002500 0.004732 0.022182-0.000051 0.000000 0.000000      0110111100
```

The next line contains `o`, k

```
 0.865520                                                   1
```

The next line contains `osin1`, `ocos1`, kk

```
 0.000000-0.018457                                          01
 0.000000 0.000000-0.002118 0.000349-0.009846 0.000000      001110
 0.000000 0.000000 0.000000 0.000000 0.000500 0.000311      000011
-0.000051 0.000796 0.005622 0.000241 0.000000 0.000000      111100
 0.000000
```

Figure 77 *Example of an atom in the atomic part of* `m40` *with Crenel occupation modulation*

So Wo

```
O         3  2     0.500000 0.155298 0.155298 0.500000      100  1  3  3
 0.003439 0.003439 0.058492-0.000174-0.002425 0.002425      0100101110
```

The next line contains `o`, k

```
 0.500000                                                   0
```

The next line contains `osin1`, `ocos1`, k0

```
 0.250000 1.000000                                          00
 0.002561 0.002561 0.000000-0.002344 0.002344 0.055930      100101
-0.001675 0.001675-0.010607 0.003021 0.003021 0.000000      101100
 0.002421-0.002421 0.003984 0.002846 0.002846 0.000000      101100
-0.001929-0.001929-0.023379 0.000274 0.001268-0.001268      101110
-0.000725 0.000725 0.000000 0.000000 0.004529 0.004529      100010
-0.000574 0.000574 0.000000 0.000000-0.000567-0.000567      100010
 0.001699 0.001699 0.030008 0.002512-0.002588 0.002588      101110
-0.001006 0.001006 0.000000 0.000000 0.000179 0.000179      100010
 0.002198 0.002198 0.031639 0.001819-0.005934 0.005934      101110
 0.000000
```

Setting or deleting occupation modulation

Provided that the dimensions and modulation vector(s) have been properly set in *PRELIM* (see page 68) the number of harmonic waves can be changed in *EDITM40* (see 168). With the automatic setting for refinement keys (see §3.3.3 page 223 for details) the symmetry restrictions of the wave components are made automatically.
The setting of the crenel function is described in page 286. For its application together with harmonic modulation functions see page 132.

The relationship between occupation factor ai and occupation modulation.

The value of the occupancy factor `ai` (see page 116) follows from the local site symmetry (for instance, it is 0.5 for atom located in a mirror or two-fold axis). In case that the position is not fully occupied the maximal value must be reduced. For regular structures or for atoms without occupational modulation it is usually done by refinement of `ai`. In cases where occupational modulation is used the `ai` parameters should not be refined. Instead the zero term o of the occupation wave is refined and the program automatically uses the theoretical value of `ai` following from the site symmetry and reduced by `o`. This value is also assigned to `ai` when converting an atom with occupation modulation to a non-modulated one (by setting 0 occupation modulation waves in *EDITM40*).

3.1.12 The temperature modulation parameters

Temperature modulation parameters *(This is part of table in page 98)*						
U11sin1 Beta11sin1[1]	U22sin1 Beta22sin1	U33sin1 Beta33sin1	U12sin1 Beta12sin1	U13sin1 Beta13sin1	U23sin1 Beta23sin1	kkkkkk
U11cos1 Beta11cos1	U22cos1 Beta22cos1	U33cos1 Beta33cos1	U12cos1 Beta12cos1	U13cos1 Beta13cos1	U23cos1 Beta23cos1	kkkkkk
...	...	...	...	...	...	...
U11sin16 Beta11sin16	U22sin16 Beta22sin16	U33sin16 Beta33sin16	U12sin16 Beta12sin16	U13sin16 Beta13sin16	U23sin16 Beta23sin16	kkkkkk
U11cos16 Beta11cos16	U22cos16 Beta22cos16	U33cos16 Beta33cos16	U12cos16 Beta12cos16	U13cos16 Beta13cos16	U23cos16 Beta23cos16	kkkkkk
...	...	...	...	...	...	...

The temperature modulation in *JANA98* can only be described with harmonic functions. The type of harmonic waves is defined by `Wt` (see the atomic header parameters, page 116).

Theoretical

Let us consider that the positional modulation described in §3.1.10, page 123, is accompanied by the modulation of the mean-square displacement tensors. Similarly as for position (see equation E 19) we can write

$$\beta_{nv} = \beta_v^0 + \beta_v\left[\mathbf{q}_1\cdot(\mathbf{g}_v+\mathbf{n}),....,\mathbf{q}_d\cdot(\mathbf{g}_v+\mathbf{n})\right], \qquad (E\ 27)$$

where β_{nv} and β_v^0 are the tensor of the temperature parameters in the *n*th cell, and the average temperature parameter tensor of atom *v*, respectively. $\mathbf{g}_v$ and $\mathbf{n}$ are explained in E 19. β_v is the *d*-dimensional periodic tensor function

$$\beta_v(x_1,....,x_d) = \beta_v(x_1+n_1,....,x_d+n_d), \qquad (E\ 28)$$

$n_1, ..., n_d$ being integers.

Similarly, as for $\mathbf{u}_v$ (see equation E 20), β_v can be expanded into a truncated Fourier series:

$$\beta_v = \sum_{i=1}^{l}\beta_v^s(i)\sin[2\pi\bar{\mathbf{q}}_i\cdot(\mathbf{n}+\mathbf{g}_v)] + \sum_{i=1}^{l}\beta_v^c(i)\cos[2\pi\bar{\mathbf{q}}_i\cdot(\mathbf{n}+\mathbf{g}_v)], \qquad (E\ 29)$$

where the summation is over all distorsion waves, including higher harmonic terms.

---

[1] The name of the modulation temperature parameters can be referenced to as U or Beta regardless of the current setting in `m40`.

$\overline{\mathbf{q}}_i$ are selected linear combinations of the modulation vectors (see equation E 21) and β_v^s , β_v^c are the amplitudes of the sin and cos displacement waves, respectively.

Parameters meaning

`U11sin1,....,U23cos1`	components of the first harmonic temperature modulation wave. They correspond to tensor components $\beta_{v,1,1}^s(1)$, $\beta_{v,2,2}^s(1)$, $\beta_{v,3,3}^s(1)$, $\beta_{v,1,2}^s(1)$, $\beta_{v,1,3}^s(1)$, $\beta_{v,2,3}^s(1)$ (sinus terms) and $\beta_{v,1,1}^c(1)$, $\beta_{v,2,2}^c(1)$, $\beta_{v,3,3}^c(1)$, $\beta_{v,1,2}^c(1)$, $\beta_{v,,3}^c(1)$, $\beta_{v,2,3}^c(1)$ (cosinus terms) in equation E 29.
kkkkkk	refinement keys belonging to the parameters at the same line

Setting or deleting temperature modulation

Provided that the dimensions and modulation vector(s) have been properly set in *PRELIM* (see page 68) the number of harmonic temperature waves can be changed in *EDITM40* (see page 168). With the automatic setting for refinement keys (see §3.3.3 page 223 for details) the symmetry restrictions of the wave components are made automatically.

3.1.13 Special and harmonic functions applied together

This chapter is based on Petricek, Lee & Evain (1995)

Introduction

The functions describing the displacive, occupation and temperature modulation waves are expressed as periodic functions of the actual position in the crystal:

$$f_{\mathbf{n}\nu} = f_\nu \left[\mathbf{q}_1(\mathbf{g}_\nu + \mathbf{n}),....,\mathbf{q}_d(\mathbf{g}_\nu + \mathbf{n})\right]. \qquad (E\ 30)$$

The quantities g_ν and n are described after equation E 19. For *d*=1 we can write

$$f_{\mathbf{n}\nu} = f_\nu \left[\mathbf{q}(\mathbf{g}_\nu + \mathbf{n}) + t\right] = f_\nu(x_4). \qquad (E\ 31)$$

The periodic function $f_\nu(x_4) = f_\nu(x_4 + 1)$ is usually expanded in a Fourier series

$$f_\nu = A_{0,\nu} + \sum_{n=1}^{\infty} A^s_{n,\nu} \sin(2\pi n x_4) + \sum_{n=1}^{\infty} A^c_{n,\nu} \cos(2\pi n x_4). \qquad (E\ 32)$$

The Fourier coefficients $A_{0,\nu}, A^s_{n,\nu}$ and $A^c_{n,\nu}$ define the set of parameters of the (3+1) dimensional structure.

Combination of harmonic functions and crenel functions

The important advantage of using Fourier series is that the harmonic functions are mutually orthogonal in the sense that the scalar product defined by the integral

$$(g_i \cdot g_j) = \int_0^1 g_i(x) g_j(x) dx = \delta_{ij}, \qquad (E\ 33)$$

where δ_{ij} is the Kronecker delta. The orthogonality condition is necessary to prevent correlations between the Fourier coefficients in the refinement process.

Usually, it suffices to take only a very limited number of harmonics to model the proper shape of the periodic functions defined in E 30. However, in cases that require a large number of harmonics, special functions like sawtooth function for position modulation (see page 124) or crenel functions for an occupation modulation wave (see page 128) can be used with less parameters than would be necessary with a Fourier expansion in E 31.

The discontinuous functions (or functions with discontinuous derivatives) certainly describe an ideal situation that never occurs in a real modulated crystal. We usually use these functions together with harmonic modulation functions (see equation E 31) in order to describe various effects smearing the pure discontinuous character of the modulation function. However, combination of these special functions[1] with harmonic functions may create problem in the refinement. For instance, with a crenel function taking the value 1 for Δ less than 1 (see page 124 for details) the displacive modulation functions are no longer defined for all x_4 and the orthogonality condition (see equation E 33) is no longer warranted, causing severe correlations between the position or temperature Fourier coefficients.

In *JANA98* the problem is solved by ***orthogonalization of the basic functions*** (see the next paragraph). If the set of harmonic functions is nearly linearly dependent the orthogonalization of the complete set of functions is not recommended. In this case the orthogonalization procedure should be preceded by ***selection of basic functions*** (see page 134).

Orthogonalization of basic functions

The orthogonalization procedure in *JANA98* is based on Schmidt orthogonalization procedure. By this method the *n*th orthogonalized function $\gamma_n(x)$ is built from a combination of the first *n* basic functions $g_1(x),\ldots,g_n(x)$:

$$\gamma_n(x) = \sum_{i=1}^{n} T_{ni} g_i(x), \qquad (E\ 34)$$

where *i* runs over the non-selected functions. The procedure is unique if the condition of orthogonality is combined with the normalization of the new functions. It transforms the complete set of the basic functions to a new complete set.

The major problem of the orthogonalization method is that the calculation of structure factors might be troublesome when the initial subset of functions is almost linearly dependent. This is because even small displacements are described as combinations of large partial displacements. In this cases the orthogonalization procedure should be preceded by selection of basic functions (see page 134).

Selection of basic functions

The selection of basic functions is based on their mutual scalar products. A symmetrical **G** matrix is defined from scalar products according to relations

$$G_{ij} = (g_i \cdot g_j)/[(g_i \cdot g_i)(g_j \cdot g_j)]^{1/2}$$

and (E 35)

$$(g_i \cdot g_j) = \int_{x_4^0-\Delta/2}^{x_4^0+\Delta/2} g_i(x) g_j(x)\,dx \ .$$

---

[1] The problem is caused by using a discontinuous function for occupation modulation. It should be noted that sawtooth function for position modulation includes a crenel function for occupation.

The **G** matrix has all diagonal elements equal to 1.00. The off-diagonal elements define cosines of the generalized angle between two elements of the set of functions. The larger the off-diagonal element, the larger is the correlation in the refinement[1]. The function $g_i(x)$ will only be accepted to enlarge the subset $M_n = \{g_1', \ldots, g_n'\}$ of the functions already selected for the refinement if the cosine, ε_i, of its angle to the linear subspace M_n is smaller than the chosen limit λ. To do so, the function $g_i(x)$ can be split into two components, one perpendicular to M_n and one that is a linear combination of the functions of M_n:

$$g_i(x) = g_\perp(x) + g_{M_n}(x) = g_\perp(x) + \sum_{i=1}^{n} \alpha_i g_i'(x). \qquad (E\ 36)$$

The coefficients α_i (i=1,...,n) follow from th matrix equation

$$\alpha = \mathbf{F}^{-1}\beta, \qquad (E\ 37)$$

where $\beta_i = (g \ \cdot g_i')/[(g \ \cdot g \ )(g_i' \cdot g_i')]^{1/2}$ and the matrix **F** is composed from the subset M_n in the same way as the matrix **G** in equation E 35. The length of $g_{M_n}(x)$ divided by the length of g_i represents the cosine of the angle:

$$\varepsilon_i = [\beta^T \mathbf{F}^{-1} \beta / (g_i \cdot g_i)]^{1/2}. \qquad (E\ 38)$$

Thus, g_i is rejected if $\varepsilon_i > \lambda$.

The disadvantage of this method is that some of harmonic functions g_i are skipped and therefore a part of the displacive modulation is not completely described. The quality of the selection can be estimated from the perpendicular components of the non-selected harmonic functions. The larger the perpendicular component, the higher the chance that a serious error occurs in describing the displacive modulation. Thus, the sum of all such contributions, ξ, is a measure of completeness of the selected set of g_i' functions:

$$\xi = \sum_{i=1}^{n} (1 - \varepsilon_i^2)^{1/2}. \qquad (E\ 39)$$

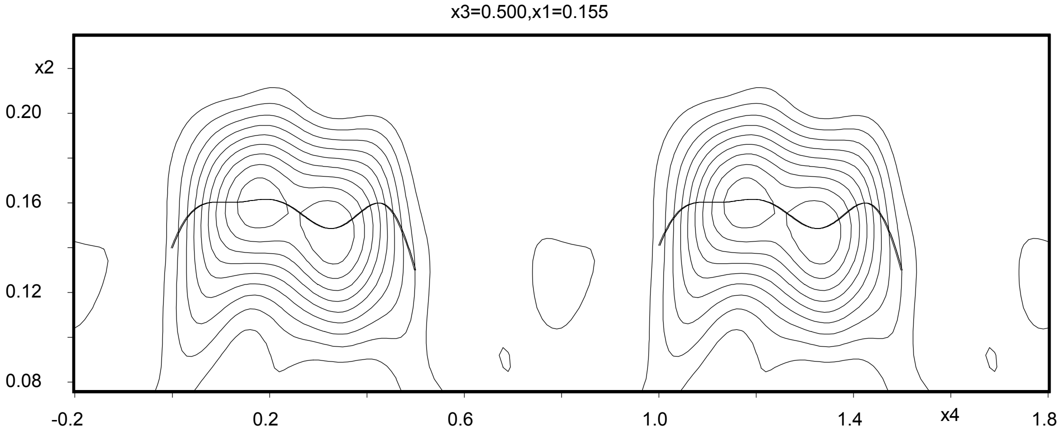
The selection procedure does not ensure orthogonality, but can be used to approach orthogonality. Therefore the selected set of basic functions is not directly used for the refinement but it is orthogonalized (see page 134).

---

[1] The **G** matrix for the orthonormalized set of functions is equal to the unit matrix.

Figure 78 *Example of a crenel function in a Contour plot.*

The position modulation function is made by combination of the crenel occupation modulation and the harmonic position modulation.



The setting and deleting of orthogonalization parameters is described in page 106. The details about this topic can be found in Petricek, Lee & Evain (1995).

3.1.14 The ADP modulation parameters

Modulation of ADP *(This is part of table in page 98)*						
C111sin1	C112sin1	C113sin1	C122sin1	C123sin1	C133sin1	kkkkkk
C222sin1	C223sin1	C233sin1	C333sin1			kkkk
C111cos1	C112cos1	C113cos1	C122cos1	C123cos1	C133cos1	kkkkkk
C222cos1	C223cos1	C233cos1	C333cos1			kkkk
...	...	...	...	...	...	...
C111sin16	C112sin16	C113sin16	C122sin16	C123sin16	C133sin16	kkkkkk
...	...	...	...	...	...	...
D1111sin1	D1112sin1	D1113sin1	D1122sin1	D1123sin1	D1133sin1	kkkkkk
D1222sin1	D1223sin1	D1233sin1	D1333sin1	D2222sin1	D2223sin1	kkkkkk
D2233sin1	D2333sin1	D3333sin1				kkk
D1111cos1	D1112cos1	D1113cos1	D1122cos1	D1123cos1	D1133cos1	kkkkkk
D1222cos1	D1223cos1	D1233cos1	D1333cos1	D2222cos1	D2223cos1	kkkkkk
D2233cos1	D2333cos1	D3333cos1				kkk
...	...	...	...	...	...	...
D1111sin16	D1112sin16	D1113sin16	D1122sin16	D1123sin16	D1133sin16	kkkkkk
...	...	...	...	...	...	...
E11111sin1	E11112sin1	E11113sin1	E11122sin1	E11123sin1	E11133sin1	kkkkkk
E11222sin1	E11223sin1	E11233sin1	E11333sin1	E12222sin1	E12223sin1	kkkkkk
E12233sin1	E12333sin1	E13333sin1	E22222sin1	E22223sin1	E22233sin1	kkkkkk
E22333sin1	E23333sin1	E33333sin1				kkk
E11111cos1	E11112cos1	E11113cos1	E11122cos1	E11123cos1	E11133cos1	kkkkkk
E11222cos1	E11223cos1	E11233cos1	E11333cos1	E12222cos1	E12223cos1	kkkkkk
E12233cos1	E12333cos1	E13333cos1	E22222cos1	E22223cos1	E22233cos1	kkkkkk
E22333cos1	E23333cos1	E33333cos1				kkk
...	...	...	...	...	...	...
E11111sin16	E11112sin16	E11113sin16	E11122sin16	E11123sin16	E11133sin16	kkkkkk
...	...	...	...	...	...	...
F111111sin1	F111112sin1	F111113sin1	F111122sin1	F111123sin1	F111133sin1	kkkkkk
F111222sin1	F111223sin1	F111233sin1	F111333sin1	F112222sin1	F112223sin1	kkkkkk
F112233sin1	F112333sin1	F113333sin1	F122222sin1	F122223sin1	F122233sin1	kkkkkk
F122333sin1	F123333sin1	F133333sin1	F222222sin1	F222223sin1	F222233sin1	kkkkkk
F222333sin1	F223333sin1	F233333sin1	F333333sin1			kkkk
F111111cos1	F111112cos1	F111113cos1	F111122cos1	F111123cos1	F111133cos1	kkkkkk
F111222cos1	F111223cos1	F111233cos1	F111333cos1	F112222cos1	F112223cos1	kkkkkk
F112233cos1	F112333cos1	F113333cos1	F122222cos1	F122223cos1	F122233cos1	kkkkkk
F122333cos1	F123333cos1	F133333cos1	F222222cos1	F222223cos1	F222233cos1	kkkkkk
F222333cos1	F223333cos1	F233333cos1	F333333cos1			kkkk
...	...	...	...	...	...	...
F111111sin16	F111112sin16	F111113sin16	F111122sin16	F111123sin16	F111133sin16	kkkkkk
...	...	...	...	...	...	...

The anharmonic displacement parameters are explained in page 122. The ADP modulation in *JANA98* can only be described for (3+1) modulated structures using harmonic functions. The number of harmonic waves is defined by `Wt3`, `Wt4`, and `Wt5` (see the atomic header parameters, page 116).

For (3+1) modulated structures the non-harmonic ADP parameters can be modulated in similar way as occupancy, position or temperature parameters. Any component of ADP tensor is then a function of the internal coordinate:

$$p_{\mathbf{n}\nu} = p_\nu^0 + p_\nu\{\mathbf{q}(\mathbf{g}_\nu + \mathbf{n})\}, \qquad (E\ 40)$$

where p_ν^0 is the average value of the relevant component of the ADP tensor for atom ν, **n** represents the lattice translations, p_ν is the periodic function $p_\nu(x) = p_\nu(x+n)$, n being integer, **q** is the incommensurate modulation vector, $\mathbf{g}_\nu$ determines the phase reference point of the displaced entity.

The function p_ν is the general modulation function of the atomic position. It can be expanded into a truncated Fourier series:

$$p_\nu = \sum_{i=1}^{l} P_\nu^s(i)\sin[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n} + \mathbf{g}_\nu)] + \sum_{i=1}^{l} P_\nu^c(i)\cos[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n} + \mathbf{g}_\nu)], \qquad (E\ 41)$$

Meaning of parameters

`C111sin1, ...., C333cos1`	Components of the first modulation wave of tensor C (see equation E 18).
`D1111sin1, ...., D3333cos1`	Components of the first modulation wave of tensor D.
`E11111sin1, ...., E33333cos1`	Components of the first modulation wave of tensor E.
`F111111sin1, ...., F333333cos1`	Components of the first modulation wave of tensor F.
kkkkkk	The refinement keys related to parameters present in the same line of `m40`.

Setting or deleting of ADP modulation parameters.

The modulation of anharmonic displacement parameters can be set in *EDITM40* - see page 168.

3.1.15 Phason for atoms

The phason parameter p accounts for reducing of intensities of satellite reflections. The factor as derived by Axe(1980) has an exponential form :

$$F_{corr}(hklm) = \exp[-(m-1)mp]F(hklm)$$

where m is the order of the satellite reflection. Note that there is no reduction for main reflections and first order satellites and therefore it can be applied only if satellites of order higher that 1 were observed and measured. The more complex way how to describe such an effect is modulation of temperature parameters. But this usually needs much more parameters.

3.1.16 The concept of molecules in *JANA98*

Molecule in *JANA98* is a model group of atoms representing one or more molecular positions of this group in the elementary cell. This enables to distinguish ***atomic parameters*** that are defined individually for each atom of the group and reproduced in the molecular positions and ***molecular parameters*** which are defined uniquely for each molecular position. Such treatment enables refinement of less parameters than would result from an atomic model. The molecule as defined in *JANA98* need not be a molecule in the chemical meaning.
The group of atoms shaping the molecule but not yet placed in the molecular position is called the ***model molecule***. The model molecule is transformed to its real positions by rotations defined by three rotational angles and translations defined by a translation vector.

The model and molecular parameters

The parameters of atoms forming the model molecule are called *model parameters.* They are refined independently for each atom of the model and they are common for all molecular positions. Their order and names in `m40` are the same like for atoms in the atomic part.

The *molecular parameters* are refined independently for each position of the model molecule. The ***basic molecular parameters*** are the same for both modulated and ordinary structures. They comprise *rotation parameters*, *translation parameters*, *occupation* and *TLS tensors*. The ***molecular modulation parameters*** can be refined for *occupancy, position and TLS tensors*.

Coordinates of the model molecule are example of model parameters. The rotation and translation parameters are typical molecular parameters. However, for many cases the user can decide whether some parameter type will be refined as model or molecular parameters. For example thermal parameters can be refined as model parameters U11, ...,U23 (i.e. independently for each atom) or as molecular parameters TLS tensors (i.e. independently for each molecular position). The distribution of parameters between the model and molecular part influences the rigidity of the molecule.

The creation of molecules and molecular transformations are described in *EDITM40*, page 171.

3.1.17 Molecular header parameters

Molecular header parameters *(This is part of table in page 98)*						
Mname	IR	PointG	Mx	My	Mz	

The molecular header parameters cannot be refined.

Meaning of parameters

Mname	The name of the molecule.
IR	The type of the reference point (0=explicit, 1=gravity center, 2=geometry center)
PointG	The point group (typed as a string) defining the local symmetry of the model molecule. The point group can be defined either by its Schoenflies or International (Hermann-Mauguin) symbol. This parameter is optional.
Mx, My and Mz	Coordinates of the reference point.

The reference point

In the case of standard structures setting of the reference point does not influence the calculation. Due to the fact *JANA98* uses the rectilinear approximation for calculation of molecular modulations, the distance of the reference point from the center of the molecule should be as short as possible.
In all cases the reference point influences calculation of symmetry restrictions - see page 189 for more information.

Local symmetry

Any of 32 crystallographic point groups and two non-crystallographic (icosahedral I and I_h) can be used as definition of local symmetry of the model molecule. The model molecule in `m40` then contains only symmetrically independent atoms. The setting of local symmetry is optional. See page 207 for examples.

Figure 79 *Example of* `m40` *with the* C_{60} *molecule having the local point group symmetry Ih.*

```
Mname      PointG               Mx       My       Mz
C60       1 Ih                0.000000 0.000000 0.000000
```

The next two lines contain the parameters of the model molecule. Because of the Ih local symmetry the model molecule consists of only 1 atom.

```
C1        1  1     0.500000 0.000000 0.049469 0.246679
 0.010201 0.000000 0.000000 0.000000 0.000000 0.000000      0011100000
pos#1     1        0.942766                                 000  0  0  0
   99.341    0.000    0.000 0.000000 0.000000 0.000000      1100000
 1.000000 1.000000 1.000000 1.000000 1.000000 1.000000
pos#2     1        0.057234                                 000  0  0  0
   39.341    0.000    0.000 0.000000 0.000000 0.000000      0000000
 1.000000 1.000000 1.000000 1.000000 1.000000 1.000000
```

3.1.18 Parameters of the model molecule

The parameters of the model molecule are atomic parameters of atoms forming the molecule. Therefore they are constructed by the same rules as the atomic parameters (see Atomic header parameters, page 116, and consequent paragraphs).

The parameters w_o, w_p and w_t (i.e. the number of occupation, position and temperature waves) should be understood together with corresponding basic molecular parameters of molecular positions. The number of modulation waves equal to zero means that the relevant modulation is refined as molecular parameters. See page 148 for more information.

The parameter Ttype in case of a model molecule can also be equal to 0 meaning that the temperature parameter is refined as molecular TLS parameters (see page 145).

3.1.19 Basic molecular parameters

Basic molecular parameters *(This is part of table in page 98)*						
Pname	Sig	aimol	$sm_o sm_p sm_t$ wm_o wm_p wm_t [1]			
phi	chi	psi	x-trans	y-trans	z-trans	kkkkkkk
rmx	rmy	rmz	rpx	rpy	rpz	

The basic molecular parameters are present for each molecular position. The basic parameter Irot is not present in this part of `m40` and is explained in page 107.

Meaning of parameters

Pname	Name of the molecular position (optional).
Sig	Sign of the rotation. The values -1 and 1 define the improper and proper rotation, respectively.
aimol	Occupation of this molecular position.
$sm_o sm_p sm_t$ wm_o wm_p wm_t	Key for type of molecular modulation function and number of modulation waves. The meaning of parameters is the same like for the atomic header parameters, page 116. The atoms from the model molecule whose modulation parameters are refined in the molecular part must have the corresponding parameters w_o, w_p or $w_t \leq 0$ (see page 116).
phi chi psi	The rotation angles.
x-trans y-trans z-trans	The translation vector.
kkkkkkk	Refinement keys for aimol, phi,chi,psi,x-rans,y-trans,z-trans
rmx rmy rmz	Direction of rotation axis in the model molecule. (0,0,0) means the default direction. Can be entered like a vector coordinates rmx, rmy, rmz or by names of two atoms from

---

[1] This cell is only present for modulated structures.

	`m40` whose positions define the start and end point of the vector.
rpx rpy rpz	Direction of rotation axis in this position. (0,0,0) means the default direction. Can be entered like a vector coordinates rpx, rpy, rpz or by names of two atoms from `m40` whose positions define the start and end point of the vector.

Theoretical

The actual position of the model molecule is calculated according to the formula

$$\mathbf{r} = \mathbf{R} \cdot (\mathbf{r}_m - \rho) + \rho + \mathbf{t}, \qquad (E\ 42)$$

where $\mathbf{r}$ and $\mathbf{r}_\mathrm{m}$ are respectively the actual and model position, ρ is the reference point and **t** is the translation vector.

The matrix **R** comprises the following operations:

- Transformation of the model molecule to the Cartesian system.
- Rotation of the model molecule by angles φ, χ and ψ along the Cartesian axes.
- Transformation back to the fractional coordinate system.

The transformation to Cartesian system is defined as

$$\mathbf{r}' = \begin{pmatrix} a & b\cos\gamma & c\cos\beta \\ 0 & b\sin\gamma & c\sin\beta\cos\alpha^* \\ 0 & 0 & c\sin\beta\sin\alpha^* \end{pmatrix} \mathbf{r}. \qquad (E\ 43)$$

The type of rotations is determined by the parameter Irot in the header of `m40` (see page 107). The ***Eulerian rotations*** (Irot=0) are defined as

$$\mathbf{r}'' = \pm \begin{pmatrix} \cos\varphi & -\sin\varphi & 0 \\ \sin\varphi & \cos\varphi & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\chi & -\sin\chi \\ 0 & \sin\chi & \cos\chi \end{pmatrix} \begin{pmatrix} \cos\psi & -\sin\psi & 0 \\ \sin\psi & \cos\psi & 0 \\ 0 & 0 & 1 \end{pmatrix} \mathbf{r}'. \qquad (E\ 44)$$

The ***axial rotations*** (Irot=1) are defined as

$$\mathbf{r}'' = \pm \begin{pmatrix} \cos\varphi & -\sin\varphi & 0 \\ \sin\varphi & \cos\varphi & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} \cos\chi & 0 & \sin\chi \\ 0 & 1 & 0 \\ -\sin\chi & 0 & \cos\chi \end{pmatrix} \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\psi & -\sin\psi \\ 0 & \sin\psi & \cos\psi \end{pmatrix} \mathbf{r}'. \qquad (E\ 45)$$

The upper and lower sign are used respectively for the proper rotation and for the improper rotation (see the parameter Sig above).

Rotation along predefined axis

Sometimes we are interested in rotation of the molecule along a vector in some important direction. This may be for instance rotation of a terminal CH_3 group, where the rotation axis goes through the carbon of the methyl group and the neighbouring non-hydrogen atom. In these cases the rotation of the model molecule can be limited to the rotation along the user-defined rotation axis.

For this purpose the user should define the rotation axis $\mathbf{r}_m$ of the model molecule (the parameters rmx, rmy, rmz in page 142) and the rotation axes $\mathbf{r}_p$ for each molecular position (rpx, rpy, rpz in page 142).

The matrix **R** then comprises the following operations:

- Transformation of the model molecule to the Cartesian system.
- Rotation bringing the Cartesian z axis and the vector $\mathbf{r}_m$ into coincidence. The rotation is made along the direction perpendicular to the plane defined by the original z axis and the vector $\mathbf{r}_m$ The relevant rotation matrix is:

$$\mathbf{F} = \begin{pmatrix} 1-\dfrac{\cos^2\lambda}{1+\cos\nu} & -\dfrac{\cos\lambda\,.\cos\mu}{1+\cos\nu} & -\cos\lambda \\ -\dfrac{\cos\lambda\,.\cos\mu}{1+\cos\nu} & 1-\dfrac{\cos^2\mu}{1+\cos\nu} & -\cos\mu \\ \cos\lambda & \cos\mu & \cos\nu \end{pmatrix}, \qquad (E\ 46)$$

where cosα, cosμ and cosν are the direction cosines of the vector with respect to the Cartesian axis.

- Rotations of the model molecule by angle φ, χ and ψ along the new Cartesian axes .
- Rotation making coincident the Cartesian z axis and the vector $\mathbf{r}_p$.
- Transformation back to the fractional coordinate system.

3.1.20 TLS tensors

TLS tensors *(This is part of table in page 98)*						
T11	T22	T33	T12	T13	T23	kkkkkk
L11	L22	L33	L12	L13	L23	kkkkkk
S11	S21	S31	S12	S22	S32	kkkkkk
S13	S23	S33				kkk

The TLS tensors are used for refinement of temperature parameters for a molecule assuming that all atoms in the group have amplitudes appropriate to a rigid body and that all atoms move in phase. In *JANA98* one molecule can contain atoms with individual temperature parameters (Ttype > 0) and atoms being part of the TLS group (Ttype = 0). The parameter Ttype is part of the atomic header (see page 116).

Meaning of parameters

T11,...,T23	The independent components of the T tensor (see equation E 50).
L11,...,L23	The independent components of the L tensor (see equation E 50).
S11,...,S33	The independent components of the S tensor (see equation E 50).
kkkkkk	The refinement keys related to parameters present in the same line of m40.

Theoretical

The theory behind the TLS parameterisation has been presented by Schomaker and Trueblood 1968. Any displacement of a rigid body can be described as a rotation about an axis passing through a fixed point, together with a translation of that fixed point. The corresponding displacement of a point at **r** relative to the fixed point is given by

$$\mathbf{u} = \mathbf{t} + \mathbf{D}\cdot\mathbf{r}, \qquad (E\ 47)$$

where **t** is a column vector for the translation and **D** is the rotation matrix. For small displacements, the last term in E 47can be linearised with respect to the amplitude of the rotation to give

$$\mathbf{u} \cong \mathbf{t} + \lambda\times\mathbf{r}, \qquad (E\ 48)$$

where lambda is a vector along the rotation axis with a magnitude equal to the angle of rotation, and x denotes a cross product. We can write

$$\mathbf{u}\mathbf{u}^\mathbf{T} = \mathbf{t}\mathbf{t}^\mathbf{T} + \mathbf{t}\boldsymbol{\lambda}^\mathbf{T}\times\mathbf{r}^\mathbf{T} - \mathbf{r}\times\boldsymbol{\lambda}\mathbf{t}^\mathbf{T} - \mathbf{r}\times\boldsymbol{\lambda}\boldsymbol{\lambda}^\mathbf{T}\times\mathbf{r}^\mathbf{T}. \qquad (E\ 49)$$

A time and spatial average over all displacements yields

$$\mathbf{U} \equiv \left\langle \mathbf{u}\mathbf{u}^\mathbf{T} \right\rangle = \mathbf{T} + \mathbf{S}^\mathbf{T}\times\mathbf{r}^\mathbf{T} - \mathbf{r}\times\mathbf{S} - \mathbf{r}\times\mathbf{L} - \mathbf{r}\times\mathbf{L}\times\mathbf{r}^\mathbf{T}, \qquad (E\ 50)$$

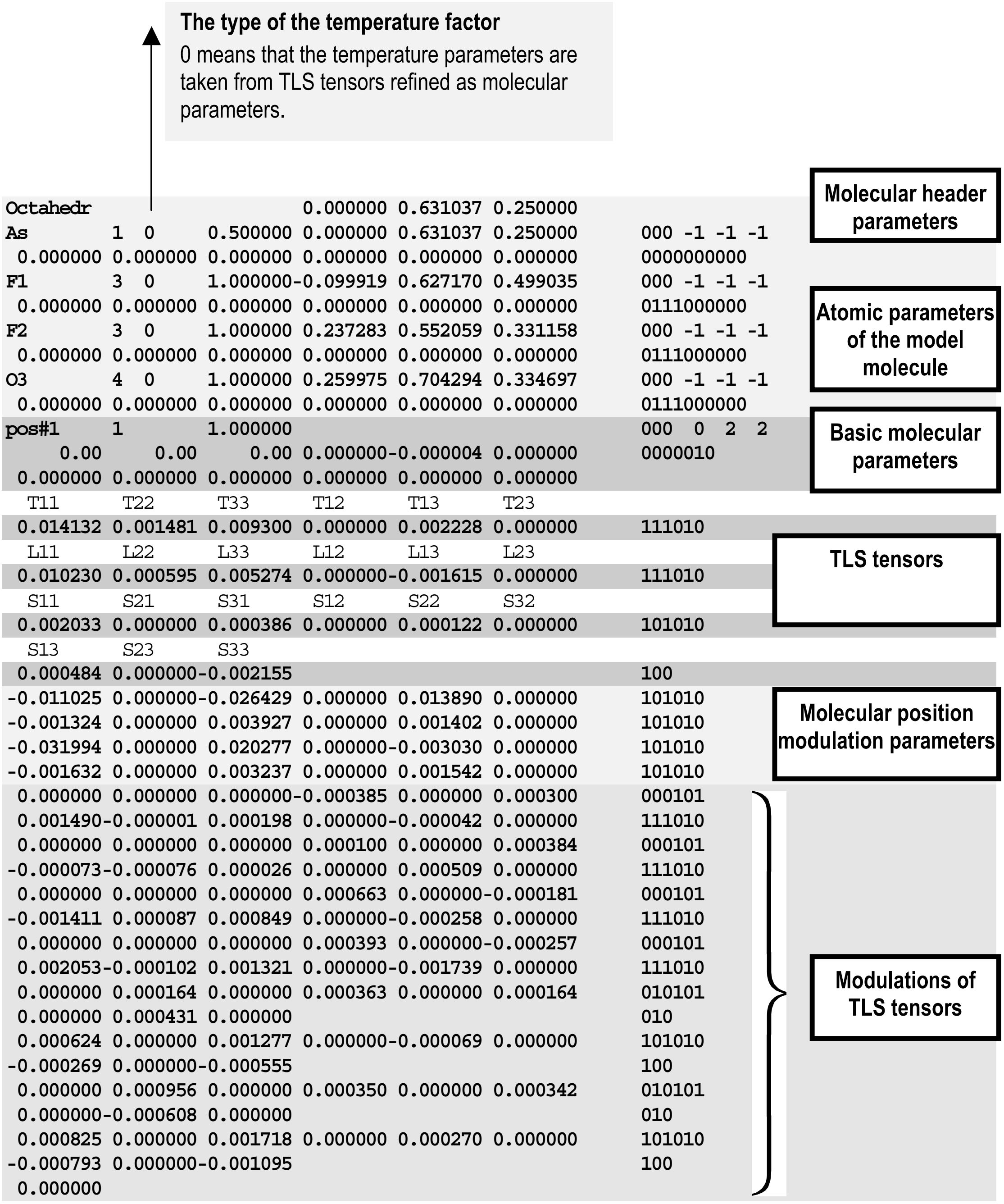
where $\mathbf{T} \equiv \left\langle \mathbf{t}\mathbf{t}^{\mathrm{T}} \right\rangle$, $\mathbf{L} \equiv \left\langle \boldsymbol{\lambda}\boldsymbol{\lambda}^{\mathrm{T}} \right\rangle$ and $\mathbf{S} \equiv \left\langle \boldsymbol{\lambda}\mathbf{t}^{\mathrm{T}} \right\rangle$.

In this context, the cross product is used as follows: $\mathbf{L} \times \mathbf{r}^{\mathrm{T}}$ yields a matrix whose *i*th row is the cross product of the *i*th row of **L** and **r**.
Equation E 50 gives the mean square displacement of a point **r** in a rigid body in terms of three tensors **T**, **L** and **S**. Considering in particular the set of points {**r**} corresponding to the rest positions of atoms in a single rigid body, **U** is the mean square displacement of each such atom, and can be identified as the anisotropic displacement parameter that occurs in the Debye-Waller factor in the expression for the structure factor. **T** and **L** are symmetric tensors, while **S** is in general asymmetric. Expanding equation E 50 out fully shows that the trace of **S** is not fixed by **U**. Hence, there are a total of 20 refinable parameters (6 from **T**, 6 from **L** and 8 from **S**).

Setting or deleting TLS tensors

The TLS tensors can be set in *EDITM40* - see page 165.

Figure 80 *Example of* `m40` *with TLS Tensors*



The type of the temperature factor
0 means that the temperature parameters are taken from TLS tensors refined as molecular parameters.

```
Octahedr                    0.000000 0.631037 0.250000
As        1  0     0.500000 0.000000 0.631037 0.250000      000 -1 -1 -1
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1        3  0     1.000000-0.099919 0.627170 0.499035      000 -1 -1 -1
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F2        3  0     1.000000 0.237283 0.552059 0.331158      000 -1 -1 -1
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
O3        4  0     1.000000 0.259975 0.704294 0.334697      000 -1 -1 -1
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
pos#1     1        1.000000                                 000  0  2  2
     0.00      0.00      0.00 0.000000-0.000004 0.000000    0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
  T11      T22      T33      T12      T13      T23
 0.014132 0.001481 0.009300 0.000000 0.002228 0.000000      111010
  L11      L22      L33      L12      L13      L23
 0.010230 0.000595 0.005274 0.000000-0.001615 0.000000      111010
  S11      S21      S31      S12      S22      S32
 0.002033 0.000000 0.000386 0.000000 0.000122 0.000000      101010
  S13      S23      S33
 0.000484 0.000000-0.002155                                 100
-0.011025 0.000000-0.026429 0.000000 0.013890 0.000000      101010
-0.001324 0.000000 0.003927 0.000000 0.001402 0.000000      101010
-0.031994 0.000000 0.020277 0.000000-0.003030 0.000000      101010
-0.001632 0.000000 0.003237 0.000000 0.001542 0.000000      101010
 0.000000 0.000000 0.000000-0.000385 0.000000 0.000300      000101
 0.001490-0.000001 0.000198 0.000000-0.000042 0.000000      111010
 0.000000 0.000000 0.000000 0.000100 0.000000 0.000384      000101
-0.000073-0.000076 0.000026 0.000000 0.000509 0.000000      111010
 0.000000 0.000000 0.000000 0.000663 0.000000-0.000181      000101
-0.001411 0.000087 0.000849 0.000000-0.000258 0.000000      111010
 0.000000 0.000000 0.000000 0.000393 0.000000-0.000257      000101
 0.002053-0.000102 0.001321 0.000000-0.001739 0.000000      111010
 0.000000 0.000164 0.000000 0.000363 0.000000 0.000164      010101
 0.000000 0.000431 0.000000                                 010
 0.000624 0.000000 0.001277 0.000000-0.000069 0.000000      101010
-0.000269 0.000000-0.000555                                 100
 0.000000 0.000956 0.000000 0.000350 0.000000 0.000342      010101
 0.000000-0.000608 0.000000                                 010
 0.000825 0.000000 0.001718 0.000000 0.000270 0.000000      101010
-0.000793 0.000000-0.001095                                 100
 0.000000
```

Molecular header parameters

Atomic parameters of the model molecule

Basic molecular parameters

TLS tensors

Molecular position modulation parameters

Modulations of TLS tensors

3.1.21 Molecular position modulation

Molecular position modulation parameters *(This is part of table in page 98)*						
xtsin1	ytsin1	ztsin1	xtcos1	ytcos1	ztcos1	kkkkkk
…	…	…	…	…	…	...
xtsin16	ytsin16	ztsin16	xtcos16	ytcos16	ztcos16	kkkkkk
xrsin1	yrsin1	zrsin1	xrcos1	yrcos1	zrcos1	kkkkkk
…	…	…	…	…	…	...
xrsin16	yrsin16	zrsin16	xrcos16	yrcos16	zrcos16	kkkkkk

The number of molecular position modulation waves is defined by wm$_p$ (see the basic molecular parameters, page 142). The position modulation of each atom is defined by the molecular position modulation parameters. The atoms having w$_p$>0 have an additional "internal" modulation which generally allows to break down the rigidity of the molecule during modulation[1].

Meaning of parameters

xtsin1,...,ztcos1	The translation components of the 1st position modulation wave (see equation E 53, page 149)
xrsin1,...,zrcos1	The rotation components of the 1st position modulation wave (see equation E 54, page 149)

Theoretical

Let us consider a displacively modulated crystal in which the νth atom in the unit cell defined by n is located at:

$$\mathbf{r}_{\mathbf{n}\nu} = \mathbf{r}_\nu^0 + \mathbf{n} + \mathbf{u}_\nu\{\mathbf{q}_1\cdot(\mathbf{g}_\nu+\mathbf{n}),....,\mathbf{q}_d\cdot(\mathbf{g}_\nu+\mathbf{n})\}, \qquad (E\ 51)$$

where $\mathbf{r}_\nu^0$ is the average position of atom ν, $\mathbf{n}$ represents the lattice translations, $\mathbf{u}_\nu$ is the d-dimensional periodic vector field $\mathbf{u}_\nu(x_1,....,x_d) = \mathbf{u}_\nu(x_1+n_1,....,x_d+n_d)$, $n_1, ..., n_d$ being integers, $\mathbf{q}_1,....,\mathbf{q}_d$ are incommensurate modulation vectors, which are rationally independent in the basis $\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*$; $\mathbf{g}_\nu$ is the phase reference point.

In the molecular displacement model all atoms in the molecule share the same phase reference point **g**. The model assumes a molecule to be displaced as a rigid unit, i.e. the displacements are limited to translation and libration displacement coordinates. For all atoms in the rigid body

$$\mathbf{u}_\nu = \mathbf{u}_m^t\{\mathbf{q}_1\cdot(\mathbf{g}+\mathbf{n}),....,\mathbf{q}_d\cdot(\mathbf{g}+\mathbf{n})\} + \mathbf{u}_m^r\{\mathbf{q}_1\cdot(\mathbf{g}+\mathbf{n}),....,\mathbf{q}_d\cdot(\mathbf{g}+\mathbf{n})\}\times(\mathbf{r}_\nu^{\mathbf{0}}-\mathbf{g}), \qquad (E\ 52)$$

where $\mathbf{u}^{\mathbf{t}}$ and $\mathbf{u}^r$ are respectively the displacement vector field and the rotational displacive vector field, m is number of a molecular position. Assuming that both

---

[1] But most of the molecule must be refined with wp ≤ 0.

vector fields are harmonic functions they can be expanded into a truncated Fourier series

$$\mathbf{u}_\nu^t = \sum_{i=1}^{l} \mathbf{U}_m^{t,s}(i)\sin[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n}+\mathbf{g}_\nu)] + \sum_{i=1}^{l} \mathbf{U}_m^{t,c}(i)\cos[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n}+\mathbf{g}_\nu)] \qquad (E\ 53)$$

and

$$\mathbf{u}_\nu^r = \sum_{i=1}^{l} \mathbf{U}_m^{r,s}(i)\sin[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n}+\mathbf{g}_\nu)] + \sum_{i=1}^{l} \mathbf{U}_m^{r,c}(i)\cos[2\pi\overline{\mathbf{q}}_i \cdot (\mathbf{n}+\mathbf{g}_\nu)], \qquad (E\ 54)$$

where $\overline{\mathbf{q}}_i$ is defined according to equation E 21, page 123.

Setting and deleting of molecular position modulation

This is described in *EDITM40*, page 168 and 191.

3.1.22 Molecular occupation modulation

<table>
<tr><td colspan="7"><i>Molecular occupation modulation parameters, harmonic functions</i> (see page 149)
<i>(This is part of table in page 98)</i></td></tr>
<tr><td>om</td><td></td><td></td><td></td><td></td><td></td><td>k</td></tr>
<tr><td>omsin1</td><td>omcos1</td><td></td><td></td><td></td><td></td><td>kk</td></tr>
<tr><td>...</td><td>...</td><td></td><td></td><td></td><td></td><td>...</td></tr>
<tr><td>omsin16</td><td>omcos16</td><td></td><td></td><td></td><td></td><td>kk</td></tr>
<tr><td>...</td><td>...</td><td></td><td></td><td></td><td></td><td>...</td></tr>
<tr><td colspan="7"><i>Molecular occupation modulation parameters, crenel function</i> (see page 149)</td></tr>
<tr><td>om</td><td></td><td></td><td></td><td></td><td></td><td>k</td></tr>
<tr><td>omsin1</td><td>omcos1</td><td></td><td></td><td></td><td></td><td>kk</td></tr>
</table>

The molecular occupation modulation is defined for positions with $\mathtt{wm}_0 > 0$ (see page 142). It is calculated in analogy with occupation modulation for free atoms - see page 127. Harmonic occupation waves can be set in *EDITM40*, see page 168 and 191. Setting of a crenel occupation function is described in page 286.

3.1.23 Molecular temperature modulation

Modulation of TLS tensors *(This is part of table in page 98)*						
T11sin1	T22sin1	T33sin1	T12sin1	T13sin1	T23sin1	kkkkkk
T11cos1	T22cos1	T33cos1	T12cos1	T13cos1	T23cos1	kkkkkk
…	…	…	…	…	…	...
T11sin16	T22sin16	T33sin16	T12sin16	T13sin16	T23sin16	kkkkkk
T11cos16	T22cos16	T33cos16	T12cos16	T13cos16	T23cos16	kkkkkk
L11sin1	L22sin1	L33sin1	L12sin1	L13sin1	L23sin1	kkkkkk
L11cos1	L22cos1	L33cos1	L12cos1	L13cos1	L23cos1	kkkkkk
…	…	…	…	…	…	...
L11sin16	L22sin16	L33sin16	L12sin16	L13sin16	L23sin16	kkkkkk
L11cos16	L22cos16	L33cos16	L12cos16	L13cos16	L23cos16	kkkkkk
S11sin1	S21sin1	S31sin1	S12sin1	S22sin1	S32sin1	kkkkkk
S13sin1	S23sin1	S33sin1				kkk
S11cos1	S21cos1	S31cos1	S12cos1	S22cos1	S32cos1	kkkkkk
S13cos1	S23cos1	S33cos1				kkk
…	…	…	…	…	…	...
…	…	…				...
S11sin16	S21sin16	S31sin16	S12sin16	S22sin16	S32sin16	kkkkkk
S13sin16	S23sin16	S33sin16				kkk
S11cos16	S21cos16	S31cos16	S12cos16	S22cos16	S32cos16	kkkkkk
S13cos16	S23cos16	S33cos16				kkk

The TLS parameters for rigid body description of temperature parameters are explained in page 145. For (3+1) modulated structures the non-harmonic ADP parameters can be modulated in similar way as occupancy, position or temperature parameters. The number of TLS modulation waves is given by the parameter wm$_t$ (see page 142).

Any component of a TLS tensor is then a function of the internal coordinate:

$$p_{\mathbf{n}v} = p_v^0 + p_v\{\mathbf{q}(\mathbf{g}_v + \mathbf{n})\}, \qquad (E\ 55)$$

where p_v^0 is the average value of the relevant component of the TLS tensor for atom v, $\mathbf{n}$ represents the lattice translations, p_v is the periodic function $p_v(x) = p_v(x+n)$, n being integer, $\mathbf{q}$ is the incommensurate modulation vector, $\mathbf{g}_v$ determines the phase reference point of the displaced entity.

The function p_v is the general modulation function of the atomic position. It can be expanded into a Fourier series:

$$p_\nu = \sum_{i=1}^{l} P_\nu^s(i)\sin[2\pi\bar{\mathbf{q}}_i \cdot (\mathbf{n}+\mathbf{g}_\nu)] + \sum_{i=1}^{l} P_\nu^c(i)\cos[2\pi\bar{\mathbf{q}}_i \cdot (\mathbf{n}+\mathbf{g}_\nu)], \qquad (E\ 56)$$

Meaning of parameters

`T11sin1, ...., T23cos1`	Components of the first modulation wave of the tensor T (see equation E 50).
`L11sin1, ...., L23cos1`	Components of the first modulation wave of the tensor L.
`S11sin1, ...., S33cos1`	Components of the first modulation wave of the tensor S.
kkkkkk	The refinement keys related to parameters present in the same line of `m40`

.

Setting and deleting of molecular temperature modulation

Molecular temperature modulation waves can be set in *EDITM40*, see page 168 and 191.

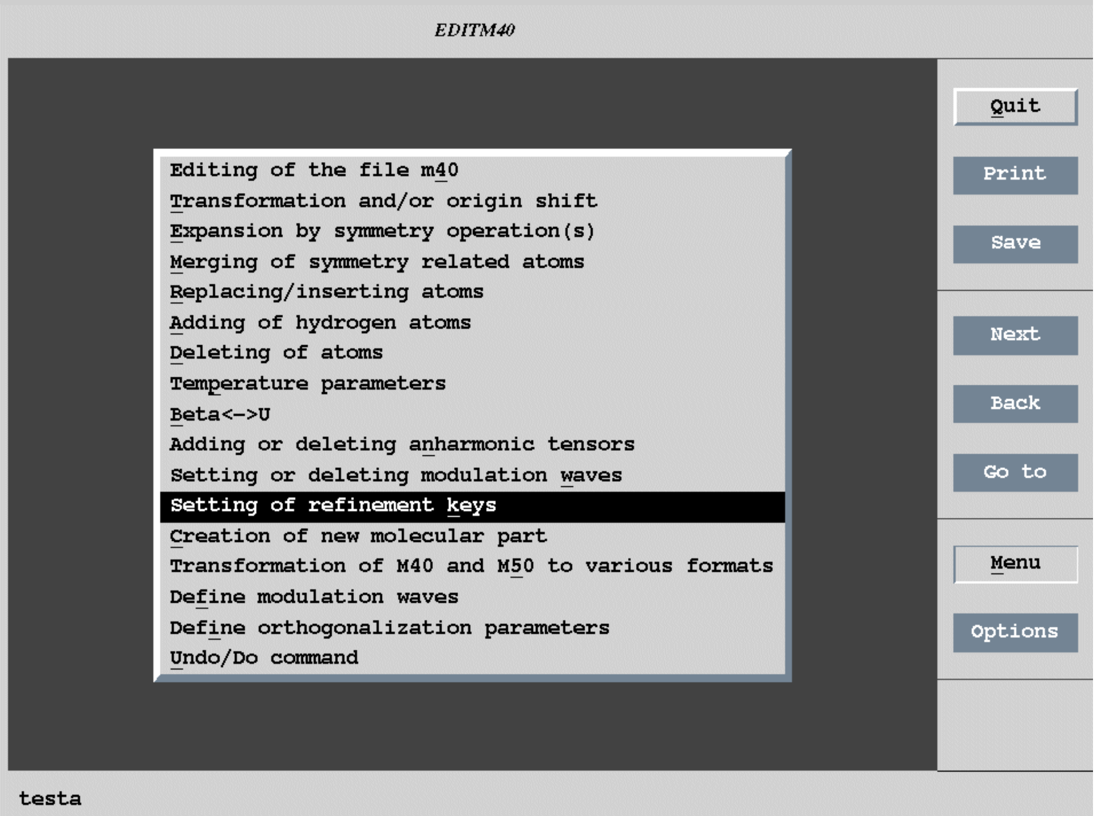
3.1.24 Phason for molecular positions

The phason for molecular positions is defined analogically to the one for free atoms (see page 139).

3.2 Program *EDITM40*

Input: `m50`,[`m40`],[`m48`],[`m45`]
Output: `m40`
M50 control keywords: none

Figure 81 *The Basic Window of EDITM40* [1]



EDITM40

Editing of the file m40
Transformation and/or origin shift
Expansion by symmetry operation(s)
Merging of symmetry related atoms
Replacing/inserting atoms
Adding of hydrogen atoms
Deleting of atoms
Temperature parameters
Beta<->U
Adding or deleting anharmonic tensors
Setting or deleting modulation waves
Setting of refinement keys
Creation of new molecular part
Transformation of M40 and M50 to various formats
Define modulation waves
Define orthogonalization parameters
Undo/Do command

3.2.1 Description of *EDITM40*

EDITM40 is an interactive program for changing the *basic refinement parameter file* `m40`. It is rather set of tools than true editor and it is focused for making changes in `m40` which cannot be simply done "by hand".
Before reading this chapter the user should have a preliminary knowledge about structure parameters and the structure parameters file `m40` described in § 3.1 page 93.

The usage of *EDITM40* is closely connected with *REFINE* (see page 219).

---

[1] The list of tools in the basic window of *EDITM40* changes according to the type of the structure. For instance the item "Setting or deleting modulation waves" is missing for standard structures. The item "Undo/Do command" is only present if some changes have been already done.

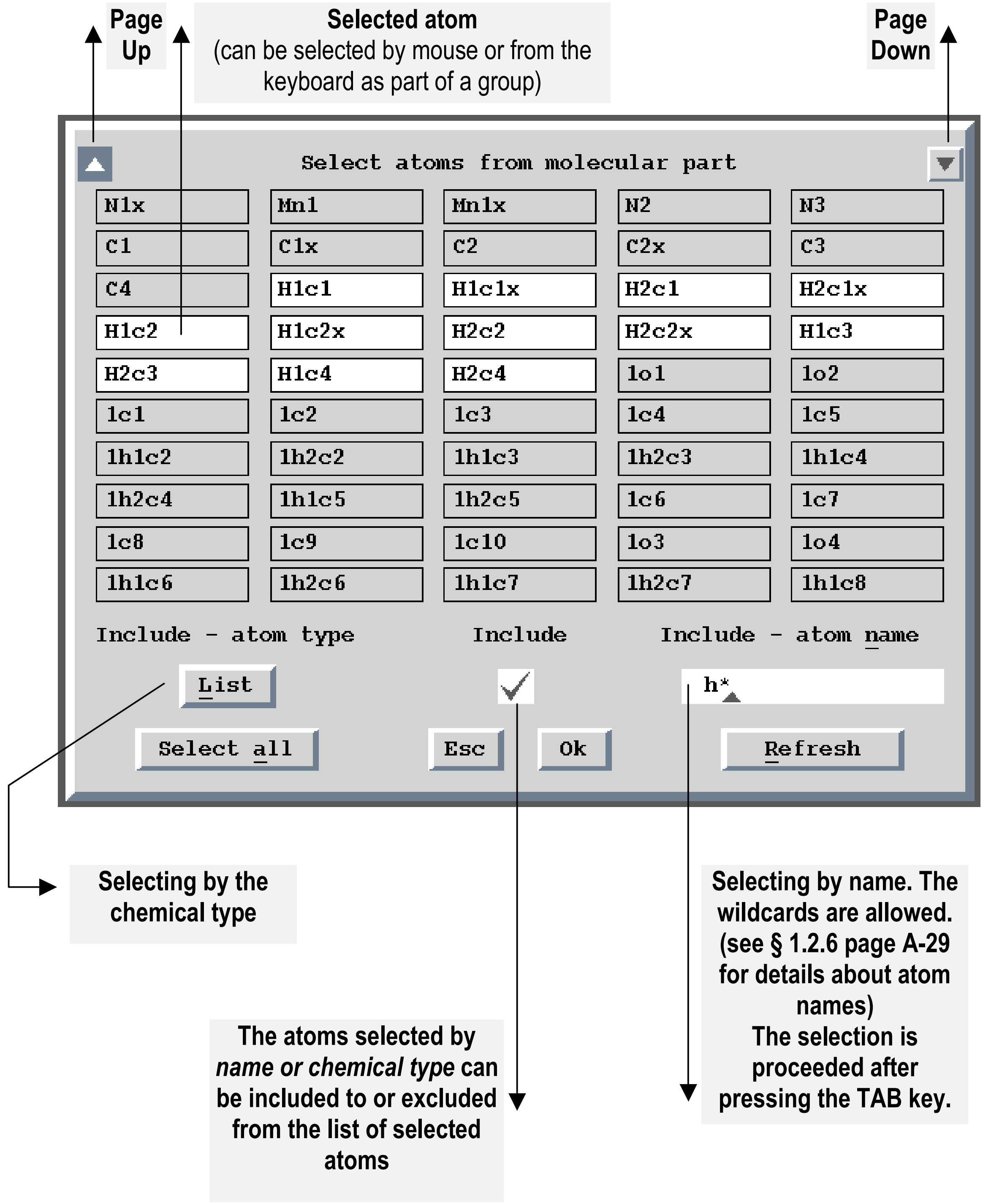
Rules for making temporary and permanent changes

All changes made in *EDITM40* are temporary until the program is closed by Quit button and the changes are confirmed by the user. This rule has no exceptions, for instance holds true also for the tool Editing of the file `m40`.
After quitting *EDITM40* and confirming the changes there is no way to get back the original `m40` except from the backup copy made by *REFINE* (see page 227).

Selecting atoms

Many *EDITM40* procedures uses the Select Atoms form to select a group of atoms for some action. In the upper part of the form there are listed atoms from `m40`. They can be selected by clicking the left mouse button or from the keyboard by typing the atom name (or a group including wildcards) in the textbox in the lower part of the form. The selected atoms have white background. In the case of a long list spread over more than one page the selection made from the keyboard influences the whole list. If the structure contains molecules, the Select atoms form is opened separately for the atomic and molecular part.

Figure 82 *The Select Atoms Form*



Select atoms from molecular part

N1x	Mn1	Mn1x	N2	N3
C1	C1x	C2	C2x	C3
C4	H1c1	H1c1x	H2c1	H2c1x
H1c2	H1c2x	H2c2	H2c2x	H1c3
H2c3	H1c4	H2c4	1o1	1o2
1c1	1c2	1c3	1c4	1c5
1h1c2	1h2c2	1h1c3	1h2c3	1h1c4
1h2c4	1h1c5	1h2c5	1c6	1c7
1c8	1c9	1c10	1o3	1o4
1h1c6	1h2c6	1h1c7	1h2c7	1h1c8

Include ☑

Include - atom name: h*

Selected atom
(can be selected by mouse or from the keyboard as part of a group)

Selecting by the chemical type

The atoms selected by *name or chemical type* can be included to or excluded from the list of selected atoms

**Selecting by name. The wildcards are allowed. (see § 1.2.6 page A-29 for details about atom names)
The selection is proceeded after pressing the TAB key.**

The examples given later in this chapter are mostly based on the molecular structure **testm** coming with the package as a testing example.

Figure 83 *Testm.m40*

```
    7    1    1    1
    4    1
 2.575509 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 1.234301 0.000000 0.000000 0.000000 0.000000 0.000000      100000
Fe        1  2     0.250000 0.250000 0.250000 0.250000      000  0  1  1
 0.001540 0.002149 0.012272 0.000000 0.000000-0.001499      0000111001
 0.002502 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000185-0.000253 0.000000      000110
 0.000000                                                   0
O1        3  2     0.125000 0.250000 0.307577 0.000000      000  0  1  1
 0.002504 0.002569 0.005933 0.000000 0.000000 0.000000      0010111000
 0.000000 0.000000 0.000000 0.006269 0.000000 0.000000      000100
 0.000000 0.000000 0.000000 0.000000-0.000062 0.000000      000010
 0.000000 0.000000 0.000000 0.000035 0.000000 0.000000      000100
 0.000000                                                   0
O1f       5  2     0.125000 0.250000 0.307577 0.000000      000  0  1  1
 0.002504 0.002569 0.005933 0.000000 0.000000 0.000000      0000000000
 0.000000 0.000000 0.000000 0.006269 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000-0.000062 0.000000      000000
 0.000000 0.000000 0.000000 0.000035 0.000000 0.000000      000000
 0.000000                                                   0
Na1       4  2     0.250000 0.250000 0.000000 0.250000      000  0  1  1
 0.003767 0.007208 0.017782 0.000000 0.000000 0.005009      0000111001
 0.000000 0.000000 0.000000 0.009265 0.000000 0.000000      000100
 0.000000 0.000000 0.000000 0.000308 0.000096 0.000000      000110
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000                                                   0
Na2       4  2     0.250000 0.000000 0.250000 0.250000      000  0  1  1
 0.006046 0.004531 0.015942 0.000000 0.000000 0.000000      0000111000
-0.003914 0.000000 0.000000 0.000000-0.011934 0.000000      100010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000280      000001
 0.000000 0.000000 0.000000 0.000000 0.002393 0.000000      000010
 0.000000                                                   0
Na3       4  2     0.500000 0.118296 0.126419 0.500000      000  1  1  1
 0.002508 0.004740 0.022151-0.000079 0.000000 0.000000      0110111100
 0.867840                                                   1
 0.000000-0.022042                                          01
 0.000000 0.000000-0.001981 0.000365-0.009662 0.000000      001110
 0.000000 0.000000 0.000000 0.000000 0.000606 0.000496      000011
-0.000015 0.000743 0.005176 0.000289 0.000000 0.000000      111100
 0.000000                                                   0
Na4       4  2     0.250000 0.000000 0.000000 0.250000      000  1  1  1
 0.004886 0.004643 0.017275 0.000000 0.000000 0.000000      0000111000
 0.669567                                                   1
 0.000000-0.147027                                          01
 0.000000 0.000000 0.003001 0.000000 0.000000 0.000000      001000
 0.000000 0.000000 0.000000-0.000033 0.000000 0.000000      000100
 0.002327-0.001012 0.001998 0.000000 0.000000 0.000000      111000
 0.000000                                                   0
Po4       0                 0.119955 0.121983 0.000000
P         2  2     0.500000 0.119801 0.121386 0.000000      000  0 -1  1
 0.001488 0.001991 0.007169-0.000368 0.000000 0.000000      0000111100
 0.000000 0.000000 0.000000 0.000000 0.000171-0.000181      000011
-0.000028 0.000192 0.000268 0.000051 0.000000 0.000000      111100
 0.000000                                                   0
```

Figure 83 *(Continued)*

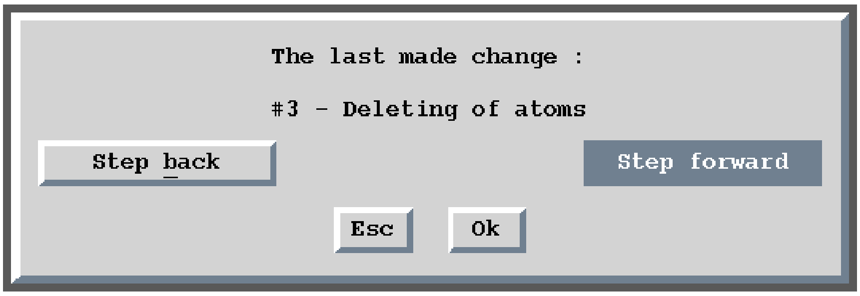
```
O2        3  2     1.000000 0.157912 0.163473 0.175969     000  0 -1  1
 0.003871 0.005279 0.008810-0.002440-0.000327-0.000840     0111111111
 0.000364-0.000601-0.000221-0.000021-0.001290 0.001471     111111
-0.000297 0.001116 0.000691-0.000255 0.000603-0.001057     111111
 0.000000                                                  0
O4        3  2     0.500000 0.138480 0.023283 0.000000     000  0 -1  1
 0.005204 0.002468 0.024080 0.000089 0.000000 0.000000     0110111100
 0.000000 0.000000 0.000000 0.000000-0.000818-0.000854     000011
-0.000172 0.000495 0.003761 0.001313 0.000000 0.000000     111100
 0.000000                                                  0
O5        3  2     0.500000 0.025914 0.139070 0.000000     000  0 -1  1
 0.001766 0.006197 0.022536 0.000413 0.000000 0.000000     0110111100
 0.000000 0.000000 0.000000 0.000000 0.000981-0.000219     000011
-0.000273-0.001402-0.000084-0.001155 0.000000 0.000000     111100
 0.000000                                                  0
pos#1     1        1.000000                                000  0  1  0
    0.000    0.000    0.000-0.000003 0.000000 0.000000     0000110
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.000000 0.000000 0.002955 0.000477 0.001805 0.000000     001110
 0.003709 0.005367 0.000000 0.000000 0.000000 0.032134     110001
 0.000000                                                  0
Fe                 0.000000 0.000000 0.000000 0.000000
 0.000014 0.000017 0.000082 0.000000 0.000000 0.000027
 0.000037 0.000000 0.000000 0.000000 0.000000 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.000000 0.000000 0.000000 0.000022 0.000044 0.000000
 0.000000
                       ............
```

3.2.2 Service tools

Do/Undo command

EDITM40 saves every change of `m40` in a temporary backup file. The Do/Undo interface enables to step back and forward in the list of the backup copies. The selected copy becomes the current temporary[1] `m40` file. ***The backup files are deleted after quitting EDITM40.***

Figure 84 *The Undo/Do Form*



The last made change :

#3 - Deleting of atoms

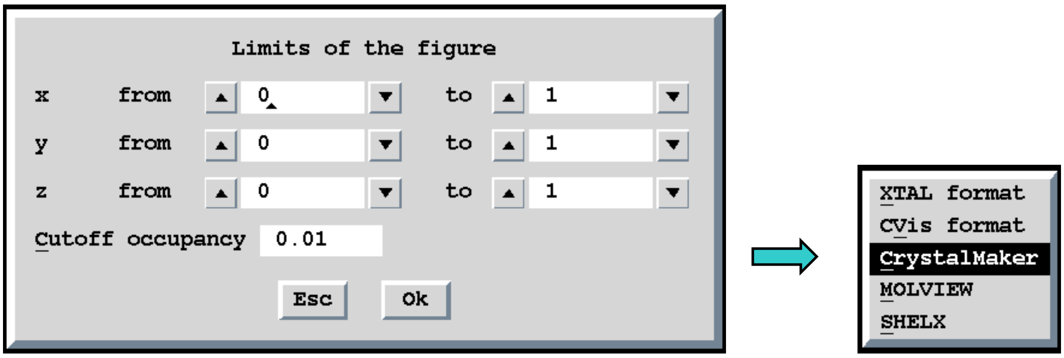
Editing of the file `m40`

The first item of the *EDITM40* main menu is Editing of `m40`. However, the user does not edit original `m40` but only a copy containing the current stage of temporary `m40`. The changes made by the user in the editing mode become permanent after quitting *EDITM40* and confirmation by the user.

Conversion of `m40` and `m50` to various formats

This tool converts a structure to formats of various crystallographic plotting programs. The result of the conversion of the modulated structure is a 3 dimensional structure expanded to a desired volume. The symmetry is reduced to *P1*.

Figure 85 *Conversion of `m40` and `m50` to various formats*



Limits of the figure

x from 0 to 1

y from 0 to 1

z from 0 to 1

Cutoff occupancy 0.01

---

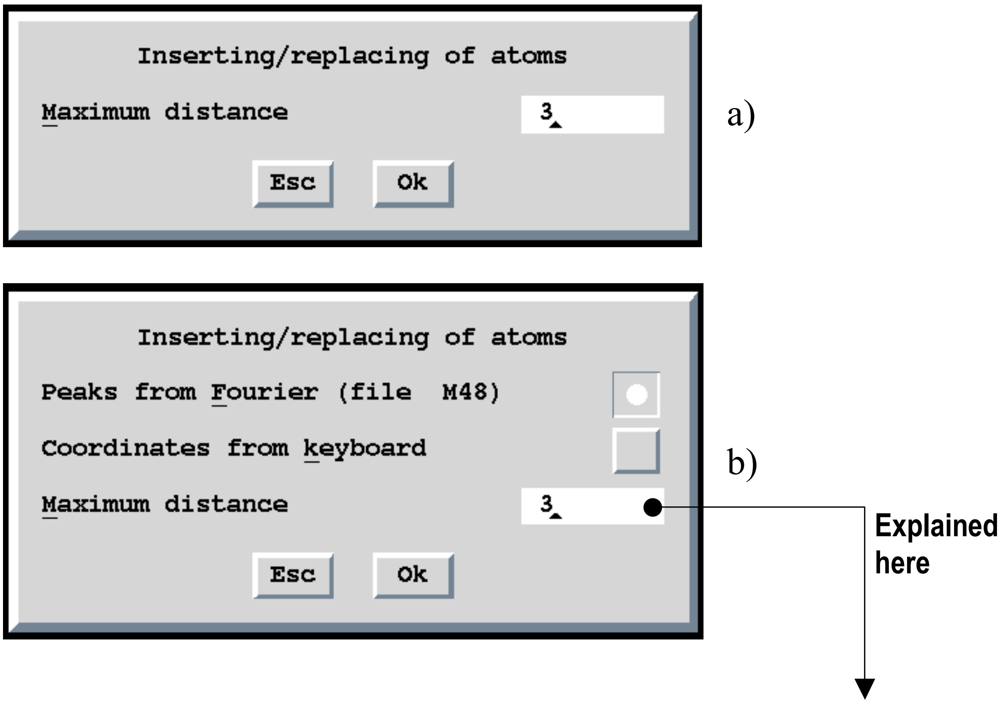
[1] The change is permanent after quitting *EDITM40* and confirmation by the user.

3.2.3 Procedures for deleting or adding atoms

Replacing/inserting atoms

The Replacing/inserting tool can be used for inserting new atoms from the keyboard or for inserting peaks previously calculated by *FOURIER*. The initial form varies dependent whether the list of peaks exists (Figure 86b) or does not exist (Figure 86a). This tool is usually used for adding small number of new atoms or for adding the strongest maxima from Fourier maps. For importing SHELX files there is a more efficient way through Tools->Transfer files from SHELX (see page 43).

Figure 86 *Replacing/inserting atoms: the initial form*



Inserting/replacing of atoms

Maximum distance 3

Inserting/replacing of atoms

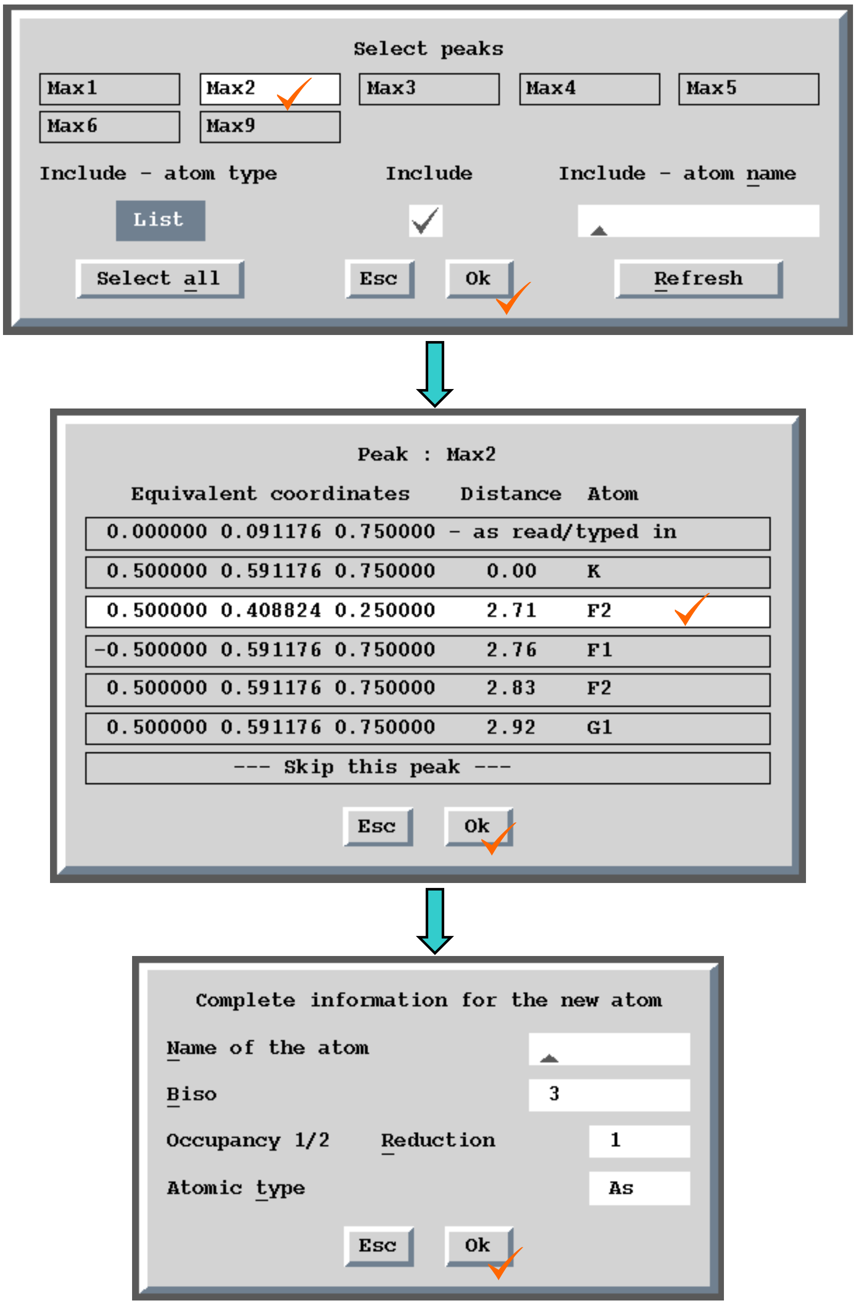
Peaks from Fourier (file M48) ●

Coordinates from keyboard ○

Maximum distance 3

shows the typical dialogue during adding of new maxima from their list saved by *FOURIER* in `m48`. The user first selects the maxima to be added to `m40`. Then *EDITM40* displays for selected maximum list of the equivalent positions and their distances to atoms already present in `m40` so that the user can select a position within an existing molecule or molecular fragment in `m40`. The limit for calculation of these distances is set in the form shown in Figure 86. Finally the information about atom name, initial isotropic temperature parameter, occupancy and chemical type is required. The reduction of occupancy is used if the position is less occupied than it follows from the site symmetry.

Figure 87 *Adding maxima from m48.*



Include ☑

Peak : Max2

Equivalent coordinates	Distance	Atom
0.000000 0.091176 0.750000 - as read/typed in		
0.500000 0.591176 0.750000	0.00	K
0.500000 0.408824 0.250000	2.71	F2
-0.500000 0.591176 0.750000	2.76	F1
0.500000 0.591176 0.750000	2.83	F2
0.500000 0.591176 0.750000	2.92	G1
--- Skip this peak ---		

Complete information for the new atom

Name of the atom

Biso 3

Occupancy 1/2 Reduction 1

Atomic type As

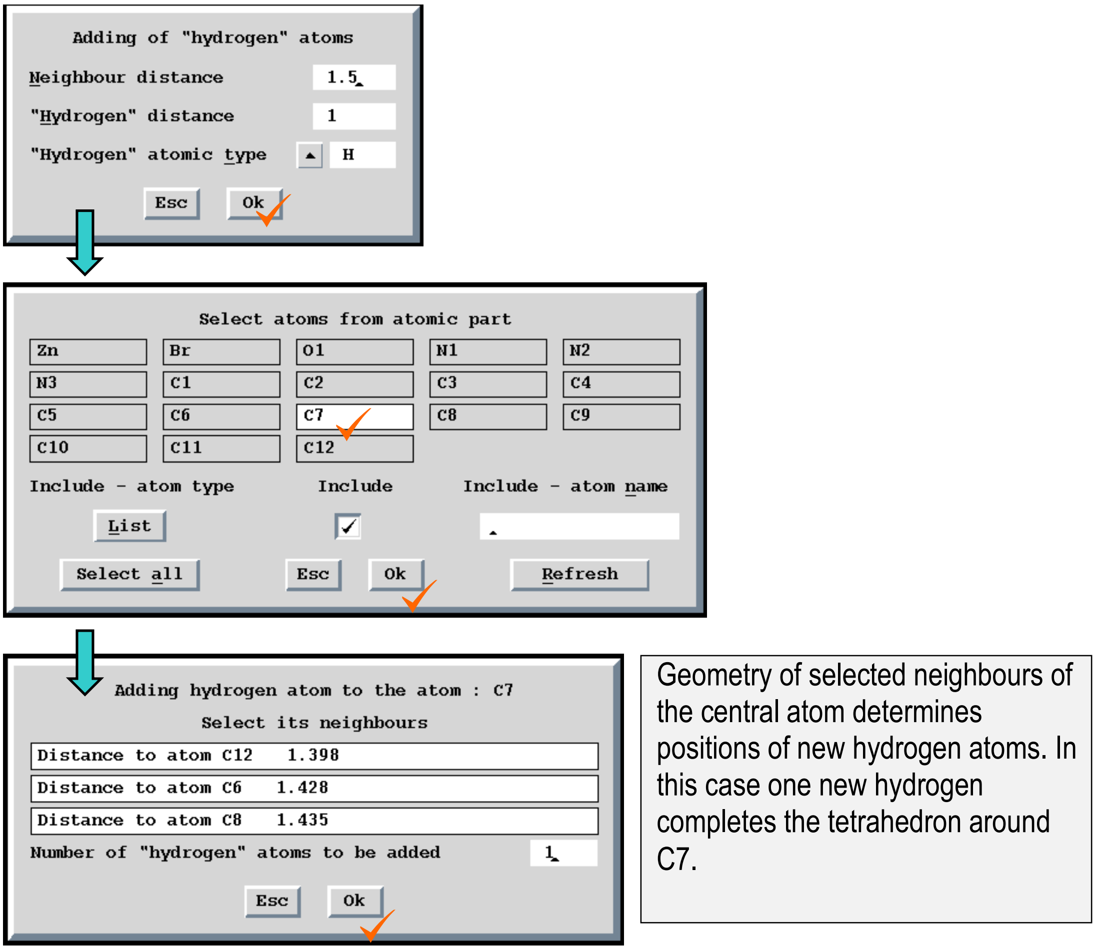
Deleting of atoms

The atoms to be deleted can be selected through the Select atoms form (see page 155). In case when molecules are present the form opens separately for the atomic part and for the model molecule. If the model molecule is completely deleted the program automatically removes also the molecular header and all parameters related to molecular positions.

Adding of hydrogen atoms

Figure 88 shows a typical dialogue during adding of new hydrogen atoms. *EDITM40* calculates new hydrogen positions to be in the vertices of a tetrahedron, triangle or abscissa with the center at the selected atom. In the first form the user specifies the distance between the central atom and new hydrogens ("hydrogen distance") and the distance limit for searching of the vertices which are already occupied by existing atoms ("neighbour distance"). Then the Select atoms form is opened in order to select the central atoms around which the hydrogens are to be calculated. For each central atom the last form is opened to specify number of hydrogens defining the shape of the body (tetrahedron, triangle or abscissa). The new hydrogen atoms are added by the program to `m40` with default names.

Figure 88 *Adding of Hydrogens*



Adding of "hydrogen" atoms

Neighbour distance 1.5

"Hydrogen" distance 1

"Hydrogen" atomic type H

Select atoms from atomic part

Zn	Br	O1	N1	N2
N3	C1	C2	C3	C4
C5	C6	C7	C8	C9
C10	C11	C12		

Include - atom type

Include ☑

Include - atom name

Adding hydrogen atom to the atom : C7

Select its neighbours

Distance to atom C12 1.398

Distance to atom C6 1.428

Distance to atom C8 1.435

Number of "hydrogen" atoms to be added 1

Geometry of selected neighbours of the central atom determines positions of new hydrogen atoms. In this case one new hydrogen completes the tetrahedron around C7.

Merging of symmetry related atoms

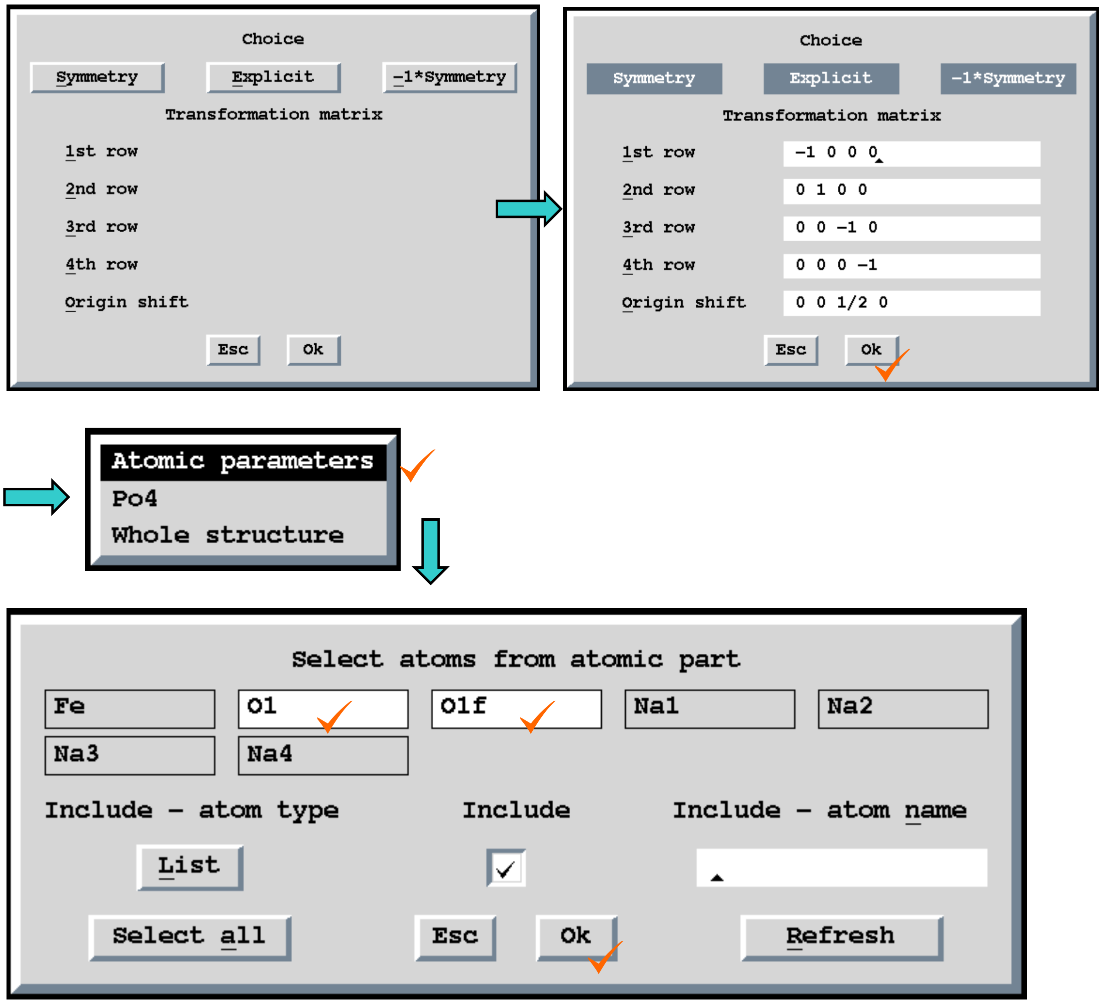
The program merges atoms within a given distance limit.

3.2.4 Transformation *procedures*

Transformation and/or origin shift

Figure 89 shows the tool for transformation of a selected part of `m40`. In the first form the user selects the way of entering the transformation matrix. The next form already contains the matrix. Then a menu follows showing available parts of `m40` (in the given example `m40` contains a molecule named "PO4"). Finally the Select atoms form is used for the definition which atoms from the chosen part of `m40` will be transformed.

Figure 89 *Transformation and origin shift*



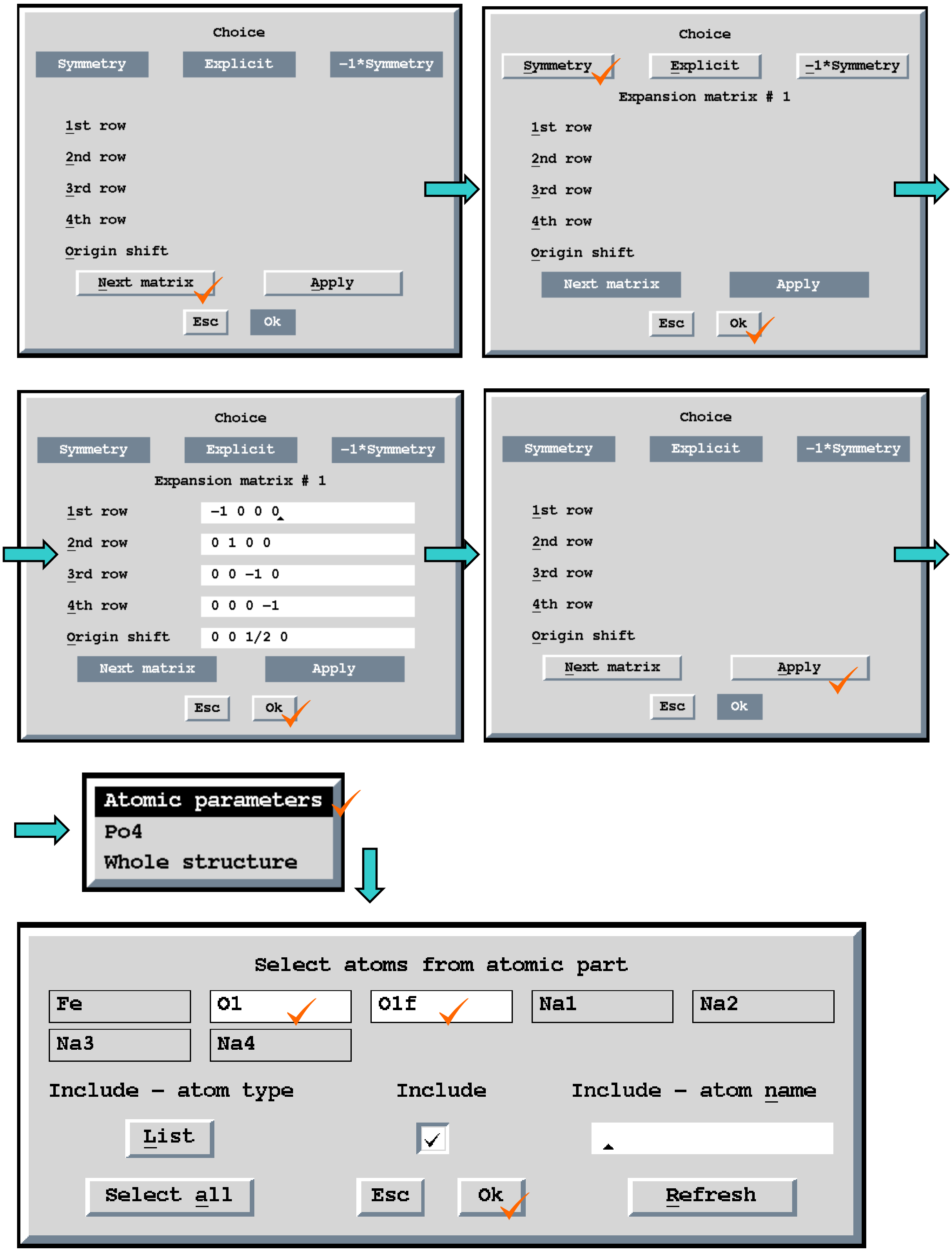
1st row: -1 0 0 0
2nd row: 0 1 0 0
3rd row: 0 0 -1 0
4th row: 0 0 0 -1
Origin shift: 0 0 1/2 0

Include: ☑

Expansion by symmetry operation(s)

This tool generates a set of new atoms from atoms already existing in `m40` using one or more symmetry matrices. The interface is similar to the one for the transformation (see page 162) with the exception that more than one transformation matrices can be used. The new atoms are added to `m40` with automatically generated names.

Figure 90 *expansion of* `m40` *by symmetry operations*



1st row -1 0 0 0
2nd row 0 1 0 0
3rd row 0 0 -1 0
4th row 0 0 0 -1
Origin shift 0 0 1/2 0

Include ☑

Transforming m40 to supercell

This tool transforms a commensurate structure a superstructure. The new 3-dimensional structure has a new name given by the user so that the parent commensurate structure is not changed. The procedure does not require any additional information as the superstructure is sufficiently defined by the q-vector and the commensurate option parameters (see page 76). The transformation is a complete one, i.e. the new structure contains all necessary files (`m40`, `m50` and `m91`) for the refinement.

3.2.5 Procedures handling temperature parameters and ADP

JANA98 works with the following types of displacement parameters:

- Isotropic or anisotropic temperature parameters of an individual atom
- TLS tensors for refining of molecular temperature parameters
- Anharmonic displacement parameters (ADP)

All types are available for both standard and modulated structures. However, ADP parameters cannot be modulated for 3+2 and 3+3 dimensional structures.

Temperature parameters

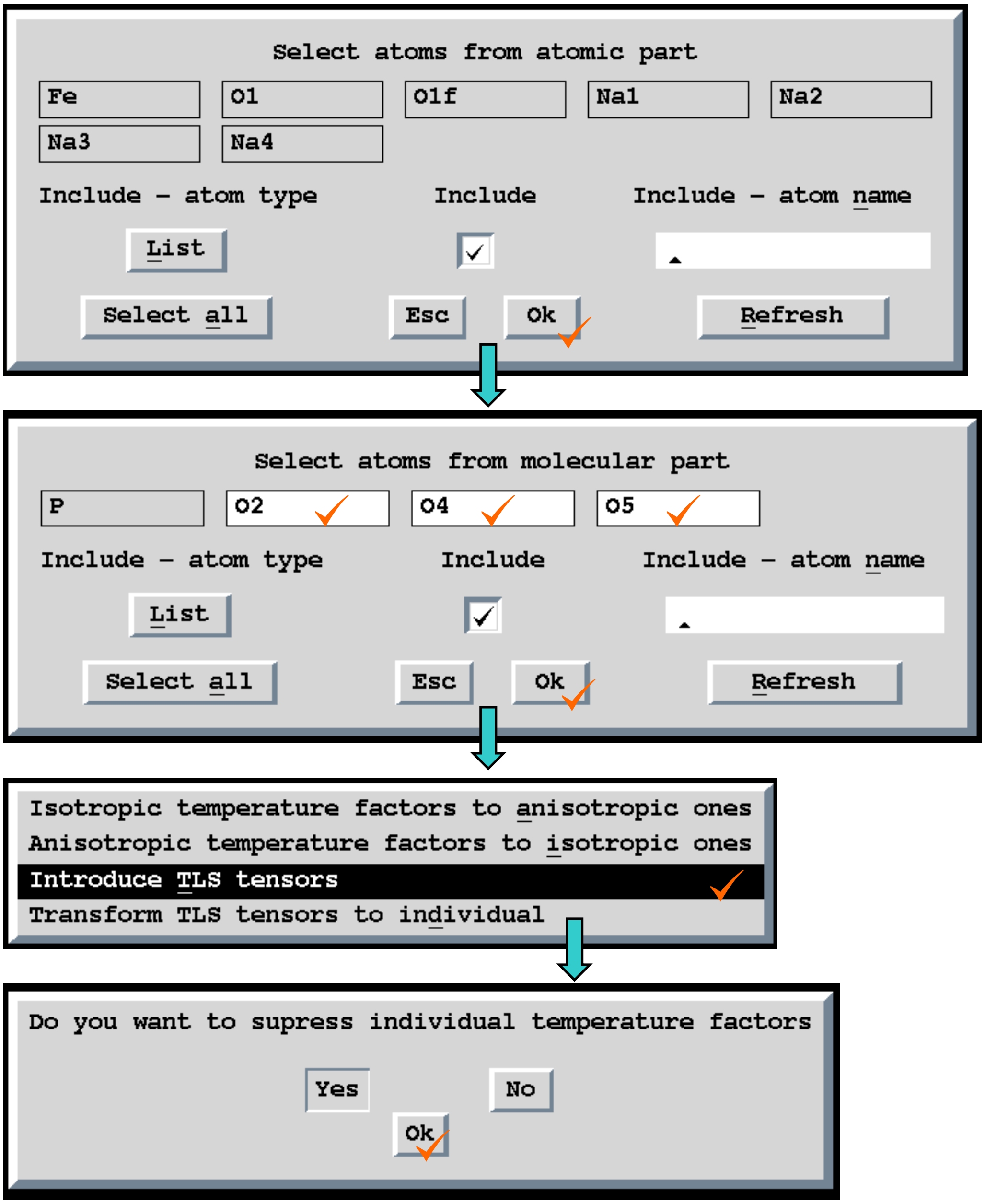
This tool converts between isotropic and anisotropic temperature parameters. In case of molecules it can also convert individual temperature parameters of a model molecule (i.e. isotropic and anisotropic temperature parameters) to molecular temperature parameters (i.e. TLS tensors). See page 140 for more information about molecules, page 120 and 131 for more information about individual temperature parameters, page 145 and 150 for more information about TLS tensors.

Figure 91 shows usage of this tool for conversion between individual and molecular temperature parameters.

Beta<->U

This tool converts between beta and U form of individual temperature parameters. This change can only be done for all atoms in `m40`. See page 120 for more information.

Figure 91 *Conversion of individual temperature parameters to TLS*

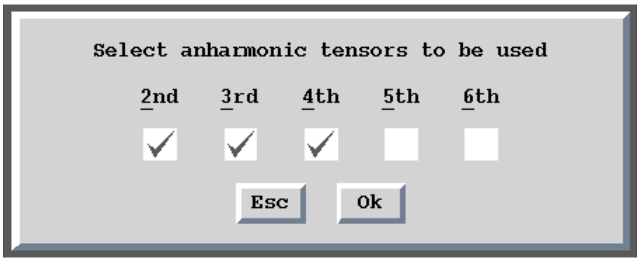


Do you want to supress individual temperature factors

Setting or deleting anharmonic tensors

The anharmonic displacement parameters (see page 121 and 137.) are available up to the 6th order and can be set or deleted through a simple interface shown in Figure 92. The second order corresponds to an individual anisotropic temperature factor. The 5th and 6th order tensors should be used only for a very precise data.

Figure 92 *Setting anharmonic tensors*



Select anharmonic tensors to be used

2nd ☑ 3rd ☑ 4th ☑ 5th ☐ 6th ☐

3.2.6 Setting modulation waves and refinement keys

Setting of refinement keys

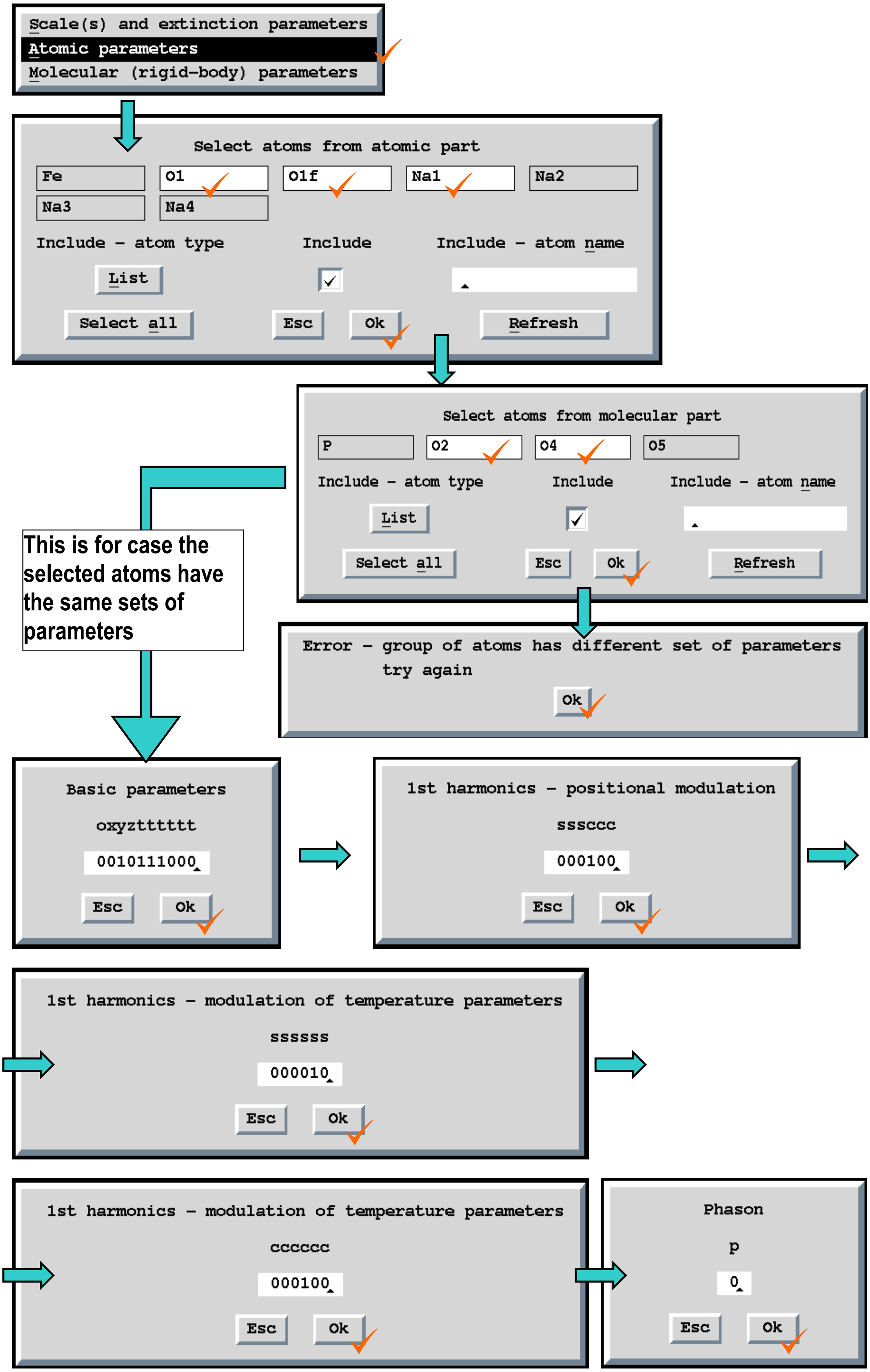
The refinement keys are explained in pages 93 and 223. This tool can be used for setting keys which cannot be set automatically (see Table 13, page 226) or if the automatic setting of refinement keys is off[1]. In case when automatic setting is on the keys set here will be overwritten by *REFINE* expect those which are not handled automatically.

The refinement keys can only be set for groups of atoms having the same sets of parameters. This is for instance group of O1, O1f and Na1 in Figure 83 but not atoms O1 and O2 or atoms Na2 and Na3.

---

[1] We recommend work without automatic refinement keys only in well-founded cases.

Figure 93 *Example of setting refinement keys*
This example is based on testm.m40 - see Figure 83, page 156.



This is for case the selected atoms have the same sets of parameters

Error - group of atoms has different set of parameters
try again

Basic parameters
oxyzttttttt
0010111000

1st harmonics - positional modulation
ssscccc
000100

1st harmonics - modulation of temperature parameters
ssssss
000010

1st harmonics - modulation of temperature parameters
cccccc
000100

Phason
p
0

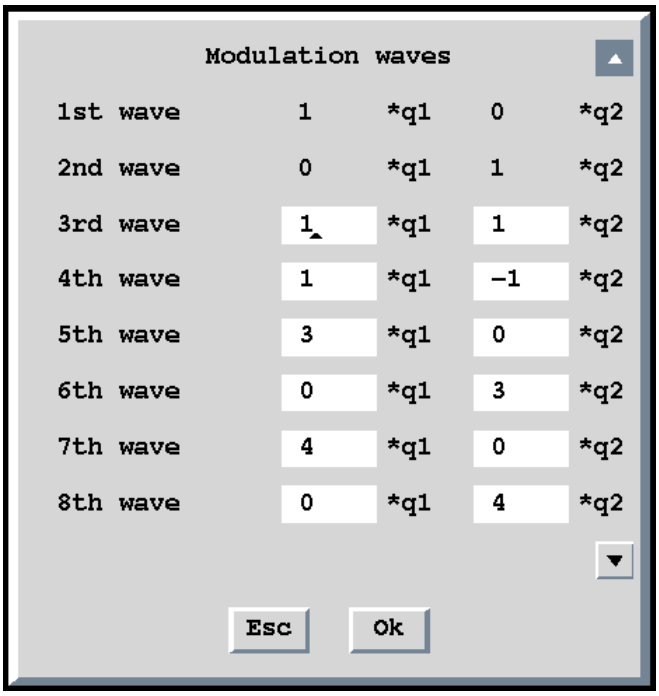
Setting or deleting modulation waves

This tool initialises or deletes modulation parameters for atoms in atomic part of `m40`, for atoms from model molecules and for molecular positions. The modulation parameters are added with default initial values (small positive numbers) and with refinement keys set to "1". In the automatic mode (see page 223) the refinement keys are changed in *REFINE* by application of the symmetry restriction rules. An example is given in Figure 95.

Define modulation waves

This tool is used for definition of wave vectors (see page 105 for more information). In the case of four-dimensional structures the default setting is usually the optimal one. The example shown in **Figure 94** is for a 5-dimensional structure with q-vectors $\mathbf{q}_1$ and $\mathbf{q}_2$. The first two waves cannot be changed and are equal to the first and the second vector, respectively. The 3rd and 4th waves are respectively defined by the user as $\mathbf{q}_1 + \mathbf{q}_2$ and $\mathbf{q}_1 - \mathbf{q}_2$. The remaining waves are left with the default parameters.

Figure 94 *definition of wave vectors for a 5-dimensional case*



Modulation waves

1st wave 1 *q1 0 *q2

2nd wave 0 *q1 1 *q2

3rd wave 1 *q1 1 *q2

4th wave 1 *q1 -1 *q2

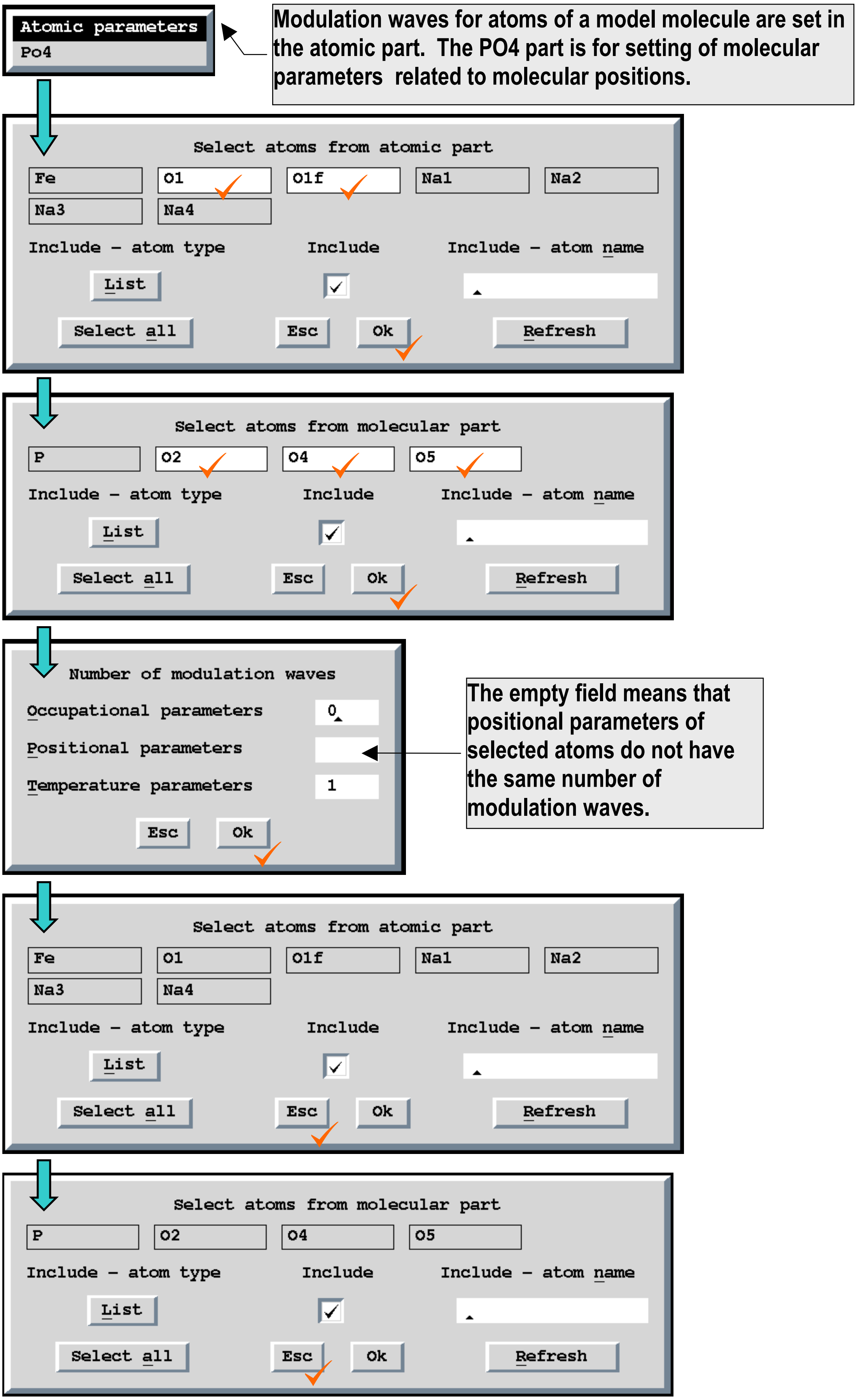
5th wave 3 *q1 0 *q2

6th wave 0 *q1 3 *q2

7th wave 4 *q1 0 *q2

8th wave 0 *q1 4 *q2

Figure 95 *Example of setting modulation waves*



Modulation waves for atoms of a model molecule are set in the atomic part. The PO4 part is for setting of molecular parameters related to molecular positions.

Number of modulation waves

Occupational parameters 0

Positional parameters

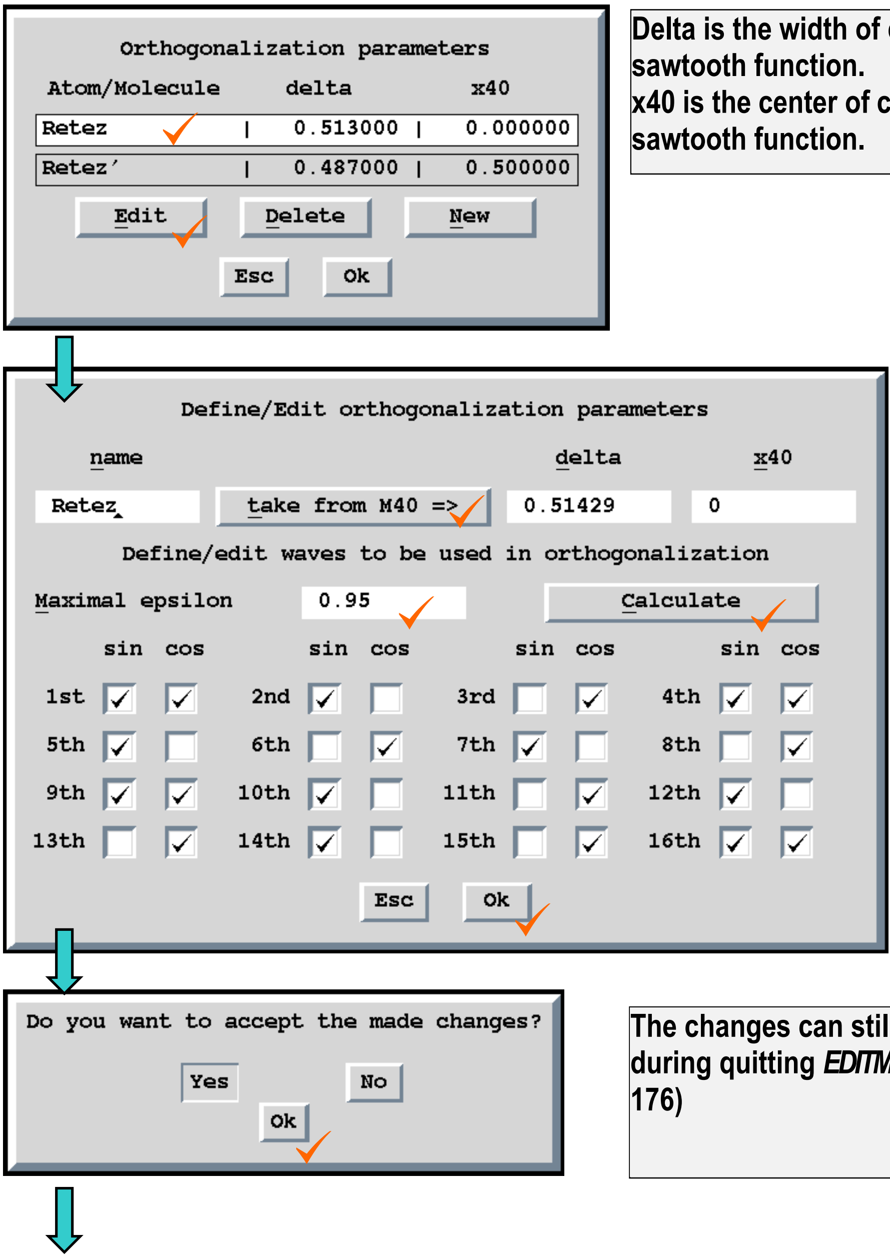
Temperature parameters 1

The empty field means that positional parameters of selected atoms do not have the same number of modulation waves.

Setting orthogonalization parameters

The orthogonalization is needed in cases when modulation of position, temperature or ADP parameters is combined with a crenel or sawtooth function not defined on a full interval of x_4. The underlying theory is given in the chapter "
Special and harmonic functions applied together" in page 133. The details about **ortho** command are given in page 106. The orthogonalization can be made for individual atoms as well as for molecules.

Figure 96 *Example of the orthogonalization tool*



Orthogonalization parameters

Atom/Molecule	delta	x40
Retez	0.513000	0.000000
Retez'	0.487000	0.500000

Define/Edit orthogonalization parameters

name		delta	x40
Retez	take from M40 =>	0.51429	0

Define/edit waves to be used in orthogonalization

Maximal epsilon 0.95

	sin	cos		sin	cos		sin	cos		sin	cos
1st	☑	☑	2nd	☑	☐	3rd	☐	☑	4th	☑	☑
5th	☑	☐	6th	☐	☑	7th	☑	☐	8th	☐	☑
9th	☑	☑	10th	☑	☐	11th	☐	☑	12th	☑	☐
13th	☐	☑	14th	☑	☐	15th	☐	☑	16th	☑	☑

Do you want to accept the made changes?

Delta is the width of crenel or sawtooth function.
x40 is the center of crenel or sawtooth function.

The changes can still be avoided during quitting *EDITM40* (see page 176)

The result is saved in the header of m40:

```
commands
ortho Retez     0.514290  0.000000  1.000000
11100111100110011110011001100111    0.950
ortho Retez'    0.487000  0.500000  1.000000
11100111100110010000000000000000   -1.000
end
```

The ortho command consists of two lines. The first line tells the program that modulation functions for selected atom or molecular position will be orthogonalized. The second (optional) line tells which functions will be included in the orthogonalization. The missing second line means all functions are included. If we set epsilon equal to 1 and press "Calculate" all checkboxes in the form will be checked, i.e. all functions will be included.

3.2.7 Procedures handling molecules

The concept of molecules in *JANA98* is described in page 140. The chapters starting in page 141 explain molecular parameters and underlying theory. Here we shall proceed by description of the basic tools in *EDITM40* for work with molecules.

Creation of new molecular part

The example given in Figure 97, Figure 98 and Figure 99 shows creation of a molecule from the atomic part of `m40`. Another way is creation of a new molecule from a model molecule in `m45` - see page 177.

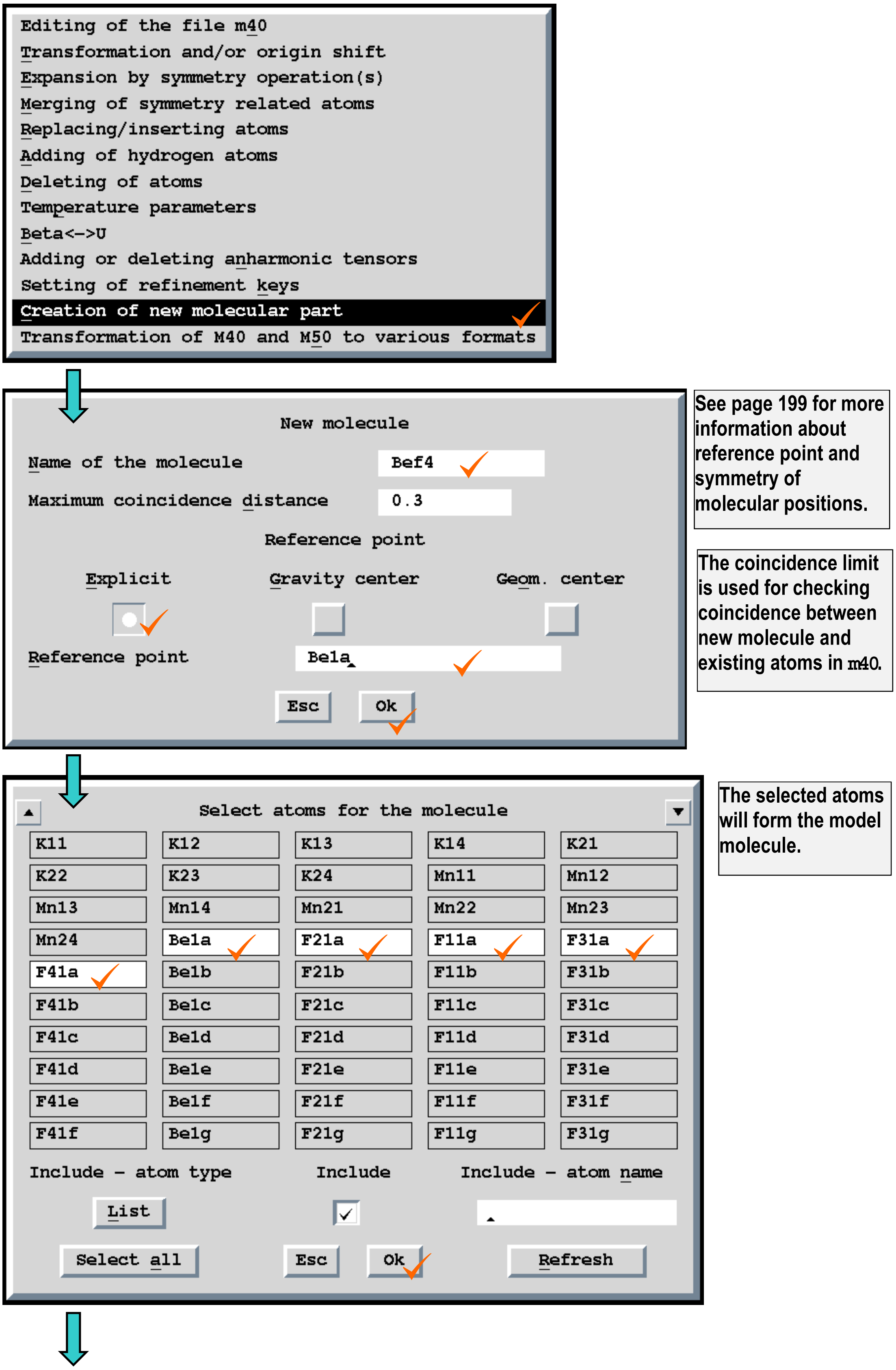
Figure 97 *Atomic* `m40`

(See Figure 98 for creating molecules from this input)

```
   76    0    0    1
 0.349776 0.134152 0.151879 0.211076 0.141322 0.156513      111111
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K11       1  2     1.000000 0.148290 0.560673 0.581993
 0.009251 0.032521 0.022902-0.000130 0.003376-0.016116      0111111111
K12       1  2     1.000000 0.656510 0.567390 0.557002
 0.009251 0.032521 0.022902-0.000130 0.003376-0.016116      0111000000
. . . . . . . . . . . . .
Mn23      2  1     1.000000 0.791403 0.142776 0.648432
 0.006213 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
Mn24      2  1     1.000000 0.295958 0.151763 0.662833
 0.006213 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
Be1a      3  2     1.000000 0.246889 0.870496 0.470083
 0.008280 0.017171 0.011088 0.005632-0.002418-0.000267      0111111111
F21a      4  2     1.000000 0.257669 0.925575 0.330705
 0.022625 0.029282 0.008272-0.004914-0.003285 0.000824      0111111111
F11a      4  2     1.000000 0.277380 0.963096 0.575194
 0.040734 0.011777 0.019152-0.010180-0.000190-0.005892      0111111111
F31a      4  2     1.000000 0.280730 0.735286 0.488883
 0.033603 0.025845 0.048385 0.023503-0.011240-0.003297      0111111111
F41a      4  2     1.000000 0.171727 0.850744 0.498849
 0.016242 0.036662 0.049694-0.003854 0.017564-0.001100      0111111111
Be1b      3  2     1.000000 0.742221 0.864892 0.481135
 0.007908 0.017008 0.011623 0.005869-0.001552-0.000715      0111111111
F21b      4  2     1.000000 0.743201 0.928876 0.343924
 0.021115 0.029213 0.009851-0.004193-0.005490 0.002937      0111111111
F11b      4  2     1.000000 0.781307 0.949327 0.581643
 0.039648 0.012903 0.019112-0.011380-0.004197-0.004666      0111111111
F31b      4  2     1.000000 0.775415 0.727747 0.480868
 0.031739 0.025680 0.050415 0.023121-0.007266-0.008192      0111111111
F41b      4  2     1.000000 0.669810 0.845461 0.530775
 0.021956 0.037011 0.043631-0.004998 0.021408-0.001483      0111111111
. . . . . . . . . . . . .
Be1k      3  2     1.000000 0.068362 0.047840 0.261548
 0.019697 0.011907 0.004935 0.001630-0.001047 0.000038      0111111111
F21k      4  2     1.000000 0.096999 0.180200 0.307647
 0.025429 0.012521 0.022229-0.001520 0.005786-0.008363      0111111111
F11k      4  2     1.000000 0.122882-0.040546 0.201213
 0.008452 0.021032 0.042179 0.005572 0.000699-0.006543      0111111111
F31k      4  2     1.000000 0.016095 0.066117 0.152211
 0.041347 0.051257 0.015229 0.013392-0.017012 0.002357      0111111111
F41k      4  2     1.000000 0.034112-0.026195 0.378345
 0.032080 0.035278 0.035241-0.007909 0.006918 0.022251      0111111111
Be1l      3  2     1.000000 0.571119 0.042951 0.266168
 0.018394 0.011366 0.006778 0.002253-0.002807-0.003333      0111111111
F21l      4  2     1.000000 0.584092 0.190777 0.246193
 0.026014 0.008948 0.025217 0.004418 0.006161-0.000822      0111111111
F11l      4  2     1.000000 0.633315-0.039133 0.232543
 0.006830 0.021530 0.043303 0.003176 0.002571 0.003374      0111111111
F31l      4  2     1.000000 0.515994-0.008338 0.174351
 0.034285 0.047889 0.025660 0.000959-0.020207-0.016908      0111111111
F41l      4  2     1.000000 0.549079 0.013876 0.411173
 0.036036 0.050303 0.016259-0.005554 0.004154 0.016386      0111111111
```

Figure 98 *creation of molecules from the atomic part of* `m40`

(The input `m40` *file is shown in* ***Figure 97****)*



New molecule

Name of the molecule: Bef4 ✓

Maximum coincidence distance: 0.3

Reference point

● Explicit ✓ ☐ Gravity center ☐ Geom. center

Reference point: Be1a ✓

See page 199 for more information about reference point and symmetry of molecular positions.

The coincidence limit is used for checking coincidence between new molecule and existing atoms in `m40`.

Select atoms for the molecule

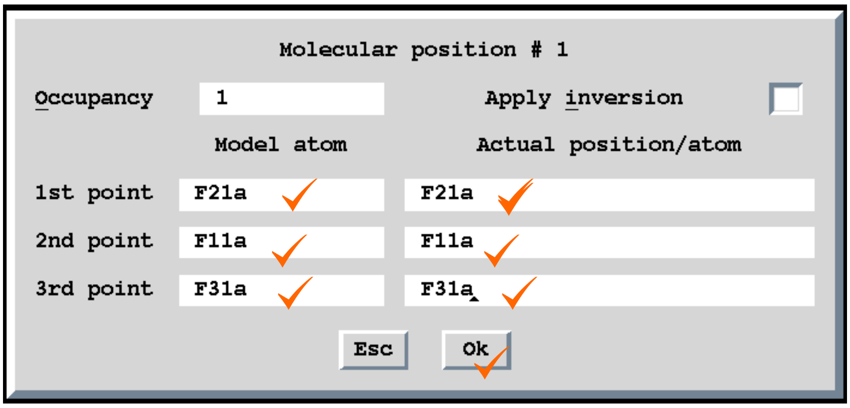
K11	K12	K13	K14	K21
K22	K23	K24	Mn11	Mn12
Mn13	Mn14	Mn21	Mn22	Mn23
Mn24	Be1a ✓	F21a ✓	F11a ✓	F31a ✓
F41a ✓	Be1b	F21b	F11b	F31b
F41b	Be1c	F21c	F11c	F31c
F41c	Be1d	F21d	F11d	F31d
F41d	Be1e	F21e	F11e	F31e
F41e	Be1f	F21f	F11f	F31f
F41f	Be1g	F21g	F11g	F31g

Include - atom type: List

☑ Include

Include - atom name:

The selected atoms will form the model molecule.



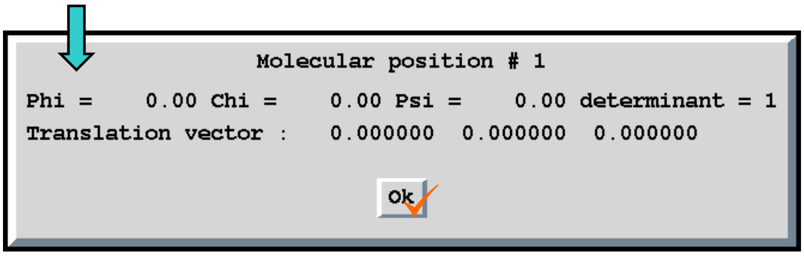
Molecular position # 1

Occupancy 1 Apply inversion ☐

	Model atom	Actual position/atom
1st point	F21a ✓	F21a ✓
2nd point	F11a ✓	F11a ✓
3rd point	F31a ✓	F31a ✓

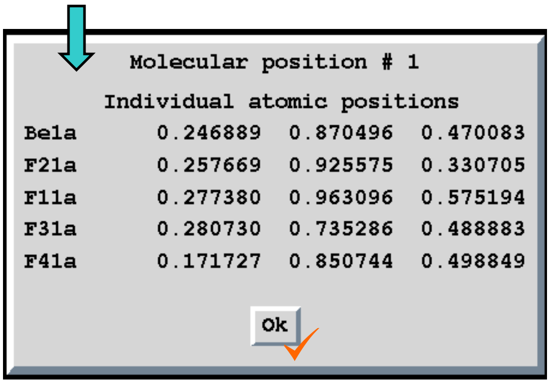
The inversion is explained with equation E 42, page 168.

The 1st molecular position is defined by rotations and translations bringing into coincidence three selected atoms in the model molecule and three actual positions. In this case the model molecule coincides with the actual position. The actual positions can also be given as coordinates. The relationship between F21a, F11a, F31a and three points in the actual position is explained in page 195.



Molecular position # 1

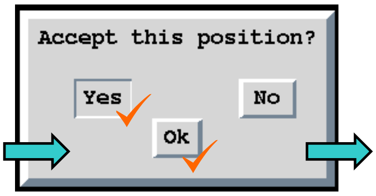
```
Phi =     0.00 Chi =     0.00 Psi =     0.00 determinant = 1
Translation vector :   0.000000  0.000000  0.000000
```



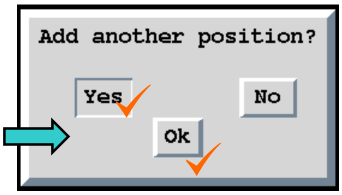
Molecular position # 1

Individual atomic positions

```
Be1a      0.246889  0.870496  0.470083
F21a      0.257669  0.925575  0.330705
F11a      0.277380  0.963096  0.575194
F31a      0.280730  0.735286  0.488883
F41a      0.171727  0.850744  0.498849
```

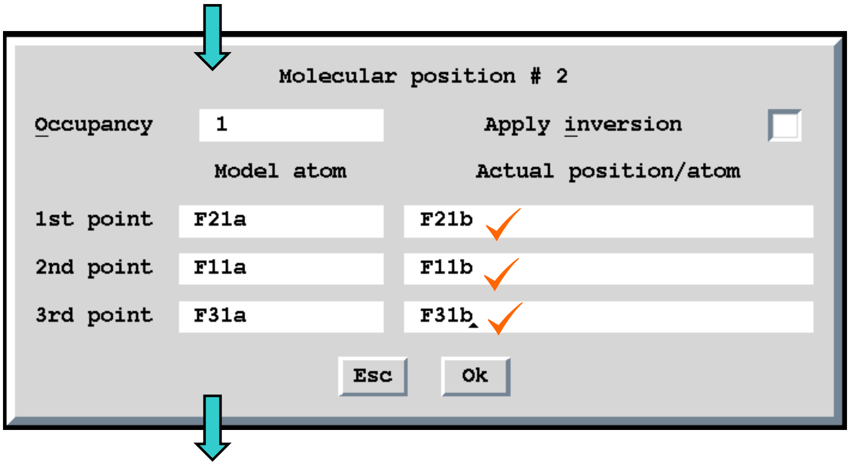


Accept this position?



Add another position?

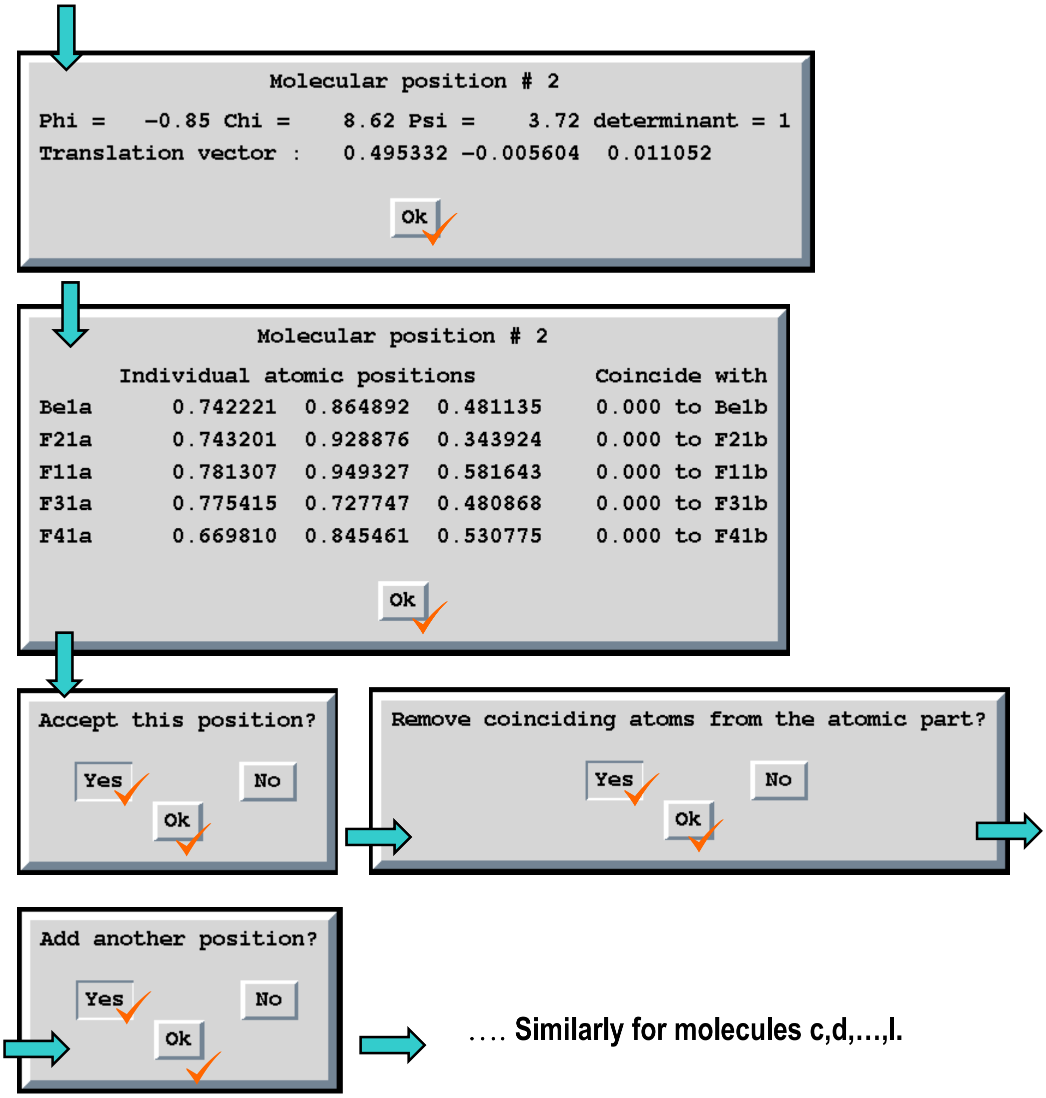
The atoms Be1a, F21a, F11a, F31a and F41a are moved from the atomic part to the model molecule. The position #1 corresponds to their former position in the atomic part. Now we shall create another position coincident with atoms Be1b, F21b, F11b, F31b and F41b.



Molecular position # 2

Occupancy 1 Apply inversion ☐

	Model atom	Actual position/atom
1st point	F21a	F21b ✓
2nd point	F11a	F11b ✓
3rd point	F31a	F31b ✓



```
                Molecular position # 2
Phi =   -0.85 Chi =    8.62 Psi =    3.72 determinant = 1
Translation vector :    0.495332 -0.005604  0.011052
```

```
                Molecular position # 2
      Individual atomic positions           Coincide with
Be1a        0.742221  0.864892  0.481135    0.000 to Be1b
F21a        0.743201  0.928876  0.343924    0.000 to F21b
F11a        0.781307  0.949327  0.581643    0.000 to F11b
F31a        0.775415  0.727747  0.480868    0.000 to F31b
F41a        0.669810  0.845461  0.530775    0.000 to F41b
```

```
Accept this position?
```

```
Remove coinciding atoms from the atomic part?
```

```
Add another position?
```

…. **Similarly for molecules c,d,…,l.**

The result of the process shown in Figure 98 is `m40` with one molecule BeF_4 repeated in 12 positions. The reverse process can be done with the tool "Move atoms from molecule to atomic part".

Figure 99 *`M40` from Figure 97 after creating molecules*

```
   16    1    0    1
    5   12
 0.349776 0.134152 0.151879 0.211076 0.141322 0.156513      111111
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K11       1  2     1.000000 0.148290 0.560673 0.581993
 0.009251 0.032521 0.022902-0.000130 0.003376-0.016116      0111111111
. . . . . . . . . . . .
Mn23      2  1     1.000000 0.791403 0.142776 0.648432
 0.006213 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
Mn24      2  1     1.000000 0.295958 0.151763 0.662833
 0.006213 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
Bef4      0                 Be1a
Be1a      3  2     1.000000 0.246889 0.870496 0.470083
 0.008280 0.017171 0.011088 0.005632-0.002418-0.000267      0000111111
F21a      4  2     1.000000 0.257669 0.925575 0.330705
 0.022625 0.029282 0.008272-0.004914-0.003285 0.000824      0111111111
F11a      4  2     1.000000 0.277380 0.963096 0.575194
 0.040734 0.011777 0.019152-0.010180-0.000190-0.005892      0111111111
F31a      4  2     1.000000 0.280730 0.735286 0.488883
 0.033603 0.025845 0.048385 0.023503-0.011240-0.003297      0111111111
F41a      4  2     1.000000 0.171727 0.850744 0.498849
 0.016242 0.036662 0.049694-0.003854 0.017564-0.001100      0111111111
pos#1     1        1.000000                                 000  0  0  0
    0.000    0.000    0.000 0.000000 0.000000 0.000000      0000111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#2     1        1.000000                                 000  0  0  0
   -0.850    8.618    3.720 0.495332-0.005604 0.011052      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#3     1        1.000000                                 000  0  0  0
  -20.359    6.838  165.405 0.742645-0.247409 0.069336      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#4     1        1.000000                                 000  0  0  0
    6.400  -10.099 -176.091 0.240299-0.238364 0.053167      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#5     1        1.000000                                 000  0  0  0
   91.206    5.801   93.885-0.133621-0.122569 0.402071      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#6     1        1.000000                                 000  0  0  0
  112.014  -17.302   91.133 0.365095-0.131409 0.409956      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#7     1        1.000000                                 000  0  0  0
 -102.659   -2.041  -93.460 0.617538-0.097104-0.336053      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#8     1        1.000000                                 000  0  0  0
 -107.375   16.955  -83.677 0.109664-0.108406-0.351159      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#9     1        1.000000                                 000  0  0  0
  -46.987  -76.955  -60.251 0.573501-0.412600 0.282860      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#10    1        1.000000                                 000  0  0  0
  145.021  -86.839  122.420 0.059928-0.407833 0.268403      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#11    1        1.000000                                 000  0  0  0
   44.286   62.153  137.005-0.178527-0.822657-0.208536      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#12    1        1.000000                                 000  0  0  0
   -6.281   77.758   70.412 0.324230-0.827545-0.203915      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
```

The atomic part

The molecular header (see page 165) and atomic parameters of the model mlecule.

Parameters of the 1st molecular position (see page 167)

Parameters of the 2nd molecular position

Parameters of the 3rd molecular position

Using the model molecule file `m45`

Another possibility is to create a molecule from atoms given in `m45`. This file contains a user-defined model molecule in arbitrary crystal coordinate system. *EDITM40* reads the molecule, verifies the chemical types of atoms and offers creating of molecular positions (see **Figure 101**).

Figure 100 *An Example of the `m45` File. It is used in Figure 101.*

Cell parameters in a free format. They can be different from the cell parameters in `m50`.

```
1 1 1 90 90 90
Ac1       2  1     0.083333 0.433000 0.000000 0.000000
Ah11      3  1     0.166667 1.010000 0.816000 0.000000
Ac2       2  1     0.166667-0.433000 0.000000 1.225000
Ah21      3  1     0.166667-1.010000 0.816000 1.225000
Ah22      3  1     0.166667-1.010000-0.816000 1.225000
Ac3       2  1     0.166667 0.433000 0.000000 2.450000
Ah31      3  1     0.166667 1.010000 0.816000 2.450000
Ah32      3  1     0.166667 1.010000-0.816000 2.450000
Ac4       2  1     0.166667-0.433000 0.000000 3.675000
Ah41      3  1     0.166667-1.010000 0.816000 3.675000
Ah42      3  1     0.166667-1.010000-0.816000 3.675000
Ac5       2  1     0.166667 0.433000 0.000000 4.900000
Ah51      3  1     0.166667 1.010000 0.816000 4.900000
Ah52      3  1     0.166667 1.010000-0.816000 4.900000
Ac6       2  1     0.166667-0.433000 0.000000 6.125000
Ah61      3  1     0.166667-1.010000 0.816000 6.125000
Ah62      3  1     0.166667-1.010000-0.816000 6.125000
Ac7       2  1     0.166667 0.433000 0.000000 7.350000
Ah71      3  1     0.166667 1.010000 0.816000 7.350000
Ah72      3  1     0.166667 1.010000-0.816000 7.350000
Ac8       2  1     0.166667-0.433000 0.000000 8.575000
Ah81      3  1     0.166667-1.010000 0.816000 8.575000
Ah82      3  1     0.166667-1.010000-0.816000 8.575000
Ac9       2  1     0.166667 0.433000 0.000000 9.800000
Ah91      3  1     0.166667 1.010000 0.816000 9.800000
Ah92      3  1     0.166667 1.010000-0.816000 9.800000
Ac10      2  1     0.166667-0.433000 0.00000011.025000
Ah101     3  1     0.166667-1.010000 0.81600011.025000
Ah102     3  1     0.166667-1.010000-0.81600011.025000
```

Atom name

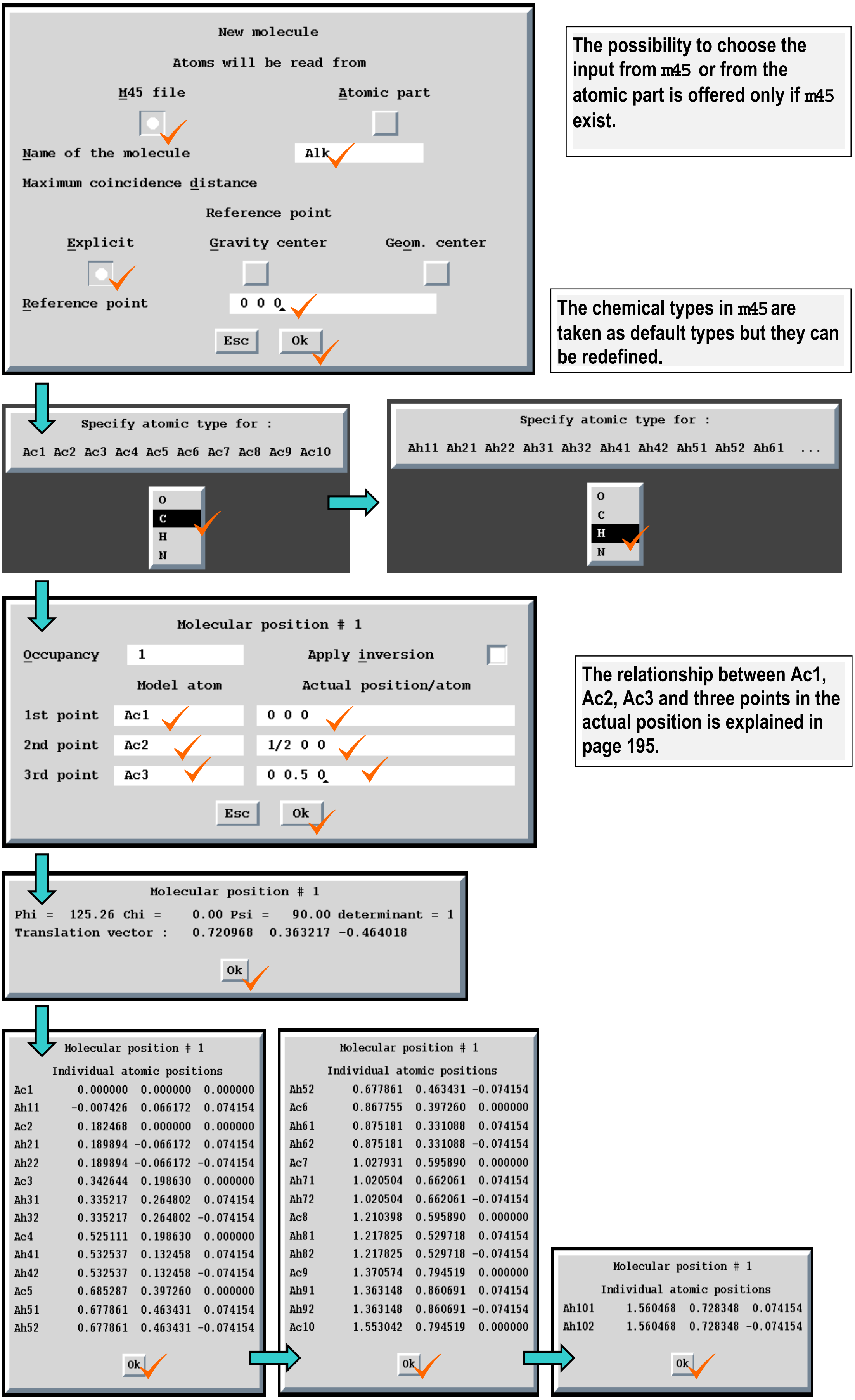
Occupancy

Temperature parameter type (Not relevant)

coordinates of model atoms

Chemical type (see Chtype, page 134)

Figure 101 *Inserting a molecule from* `m45` *(see Figure 100)*



```
New molecule

Atoms will be read from
M45 file ●          Atomic part ☐
Name of the molecule            Alk
Maximum coincidence distance

Reference point
Explicit ●     Gravity center ☐     Geom. center ☐
Reference point                 0 0 0
```

The possibility to choose the input from `m45` or from the atomic part is offered only if `m45` exist.

```
Specify atomic type for :
Ac1 Ac2 Ac3 Ac4 Ac5 Ac6 Ac7 Ac8 Ac9 Ac10
```

```
Specify atomic type for :
Ah11 Ah21 Ah22 Ah31 Ah32 Ah41 Ah42 Ah51 Ah52 Ah61  ...
```

The chemical types in `m45` are taken as default types but they can be redefined.

```
Molecular position # 1
Occupancy   1                 Apply inversion ☐
            Model atom        Actual position/atom
1st point   Ac1               0 0 0
2nd point   Ac2               1/2 0 0
3rd point   Ac3               0 0.5 0
```

The relationship between Ac1, Ac2, Ac3 and three points in the actual position is explained in page 195.

```
Molecular position # 1
Phi =   125.26 Chi =     0.00 Psi =    90.00 determinant = 1
Translation vector :     0.720968   0.363217  -0.464018
```

```
Molecular position # 1
Individual atomic positions
Ac1      0.000000  0.000000  0.000000
Ah11    -0.007426  0.066172  0.074154
Ac2      0.182468  0.000000  0.000000
Ah21     0.189894 -0.066172  0.074154
Ah22     0.189894 -0.066172 -0.074154
Ac3      0.342644  0.198630  0.000000
Ah31     0.335217  0.264802  0.074154
Ah32     0.335217  0.264802 -0.074154
Ac4      0.525111  0.198630  0.000000
Ah41     0.532537  0.132458  0.074154
Ah42     0.532537  0.132458 -0.074154
Ac5      0.685287  0.397260  0.000000
Ah51     0.677861  0.463431  0.074154
Ah52     0.677861  0.463431 -0.074154
```

```
Molecular position # 1
Individual atomic positions
Ah52     0.677861  0.463431 -0.074154
Ac6      0.867755  0.397260  0.000000
Ah61     0.875181  0.331088  0.074154
Ah62     0.875181  0.331088 -0.074154
Ac7      1.027931  0.595890  0.000000
Ah71     1.020504  0.662061  0.074154
Ah72     1.020504  0.662061 -0.074154
Ac8      1.210398  0.595890  0.000000
Ah81     1.217825  0.529718  0.074154
Ah82     1.217825  0.529718 -0.074154
Ac9      1.370574  0.794519  0.000000
Ah91     1.363148  0.860691  0.074154
Ah92     1.363148  0.860691 -0.074154
Ac10     1.553042  0.794519  0.000000
```

```
Molecular position # 1
Individual atomic positions
Ah101    1.560468  0.728348  0.074154
Ah102    1.560468  0.728348 -0.074154
```

Figure 102 *`M40` with a molecule added from `m45` (see Figure 101)*

```
    0    1    0    1
   29    1
 1.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
Alk       1              0.000000 0.000000 0.000000
Ac1       2  1     0.083333 0.052666 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah11      3  1     0.166667 0.180147 0.114604 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac2       2  1     0.166667-0.052666 0.000000 0.111322
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah21      3  1     0.166667-0.065544 0.114604 0.111322
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah22      3  1     0.166667-0.180147-0.114604 0.111322
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac3       2  1     0.166667 0.052665 0.000000 0.222644
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah31      3  1     0.166667 0.180147 0.114603 0.222644
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah32      3  1     0.166667 0.065544-0.114604 0.222644
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac4       2  1     0.166667-0.052666 0.000000 0.333966
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah41      3  1     0.166667-0.065544 0.114603 0.333966
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah42      3  1     0.166667-0.180147-0.114604 0.333966
 0. Ac5       2  1     0.166667 0.052665 0.000000 0.445289
```

. . . .

```
037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac7       2  1     0.166667 0.052665 0.000000 0.667933
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah71      3  1     0.166667 0.180147 0.114603 0.667933
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah72      3  1     0.166667 0.065544-0.114604 0.667933
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac8       2  1     0.166667-0.052666 0.000000 0.779255
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah81      3  1     0.166667-0.065544 0.114603 0.779255
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah82      3  1     0.166667-0.180147-0.114604 0.779255
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac9       2  1     0.166667 0.052665 0.000000 0.890577
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah91      3  1     0.166667 0.180147 0.114603 0.890577
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah92      3  1     0.166667 0.065544-0.114604 0.890577
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ac10      2  1     0.166667-0.052666 0.000000 1.001899
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah101     3  1     0.166667-0.065544 0.114603 1.001899
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
Ah102     3  1     0.166667-0.180147-0.114604 1.001899
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
pos#1     1        1.000000                                 000  0  0  0
  125.258    0.000   90.000 0.720968 0.363217-0.464018      0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
```

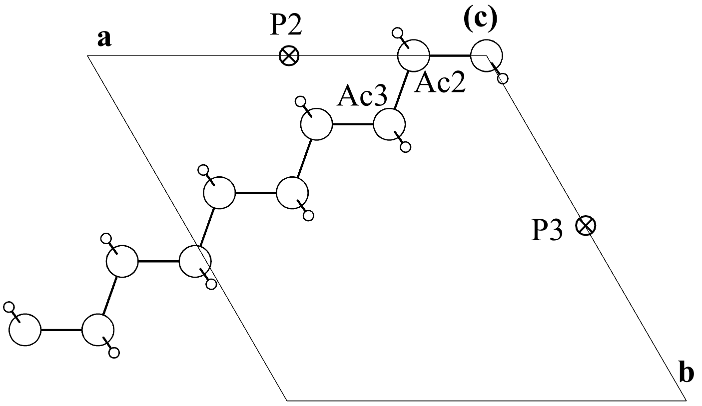
Placing the model molecule

The molecular position is defined by the relationship between three atoms A1,A2 and A3 of the model molecule and corresponding three points P1,P2 and P3 in actual positions. If the triangles defined by the model atoms and the three points are the same (i.e. with the same distances and angles) the three model atoms are simply transformed to the new positions. If the triangles are different the program places the three model atoms by the following rules:
- A1 coincides with P1
- The vector A1A2 has the same direction as P1P2
- The plane A1A2A3 coincides with P1P2P3

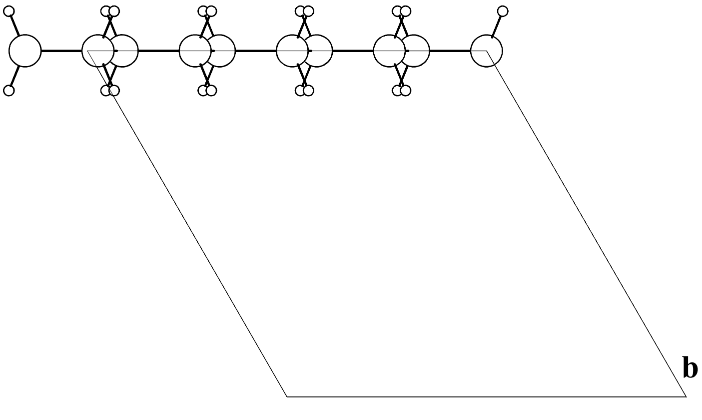
See examples in Figure 103.

Figure 103 *Placing a model molecule according to Figure 101*

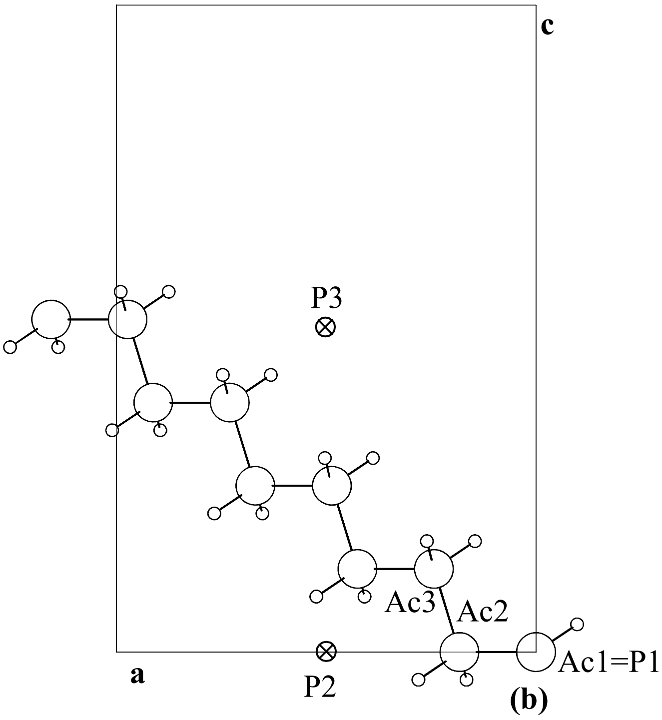
(a) *The actual positions of three atoms Ac1, Ac2 and Ac3 of the model molecule are defined by three points P1=(0,0,0), P2=(½,0,0) and P3=(0,½,0). The plane of the alkane string in the actual position is parallel with the* ***ab*** *plane.*



(b) *The actual positions of three atoms Ac1, Ac2 and Ac3 of the model molecule are defined by three points P1=(0,0,0), P2=(½,0,0) and P3=(½,0,½). The plane of the alkane string in the actual position is parallel with the* ***ac*** *plane.*



***(c)** Figure 103b viewed along the **b** axis*



Setting the rotation axis

The basic molecular parameters (see page 142) contain also the definition of the rotation axis in the model molecule and in the molecular position. Usually the parameters are set to zeroes meaning that they are not used. If the vectors are defined, the program refines rotations around them. The details about this procedure are in page 144.

The rotation axis can be defined through the tool "Transformation of molecular parameters". The following example illustrates the usage of a predefined rotation axis for refinement of terminal CH_3 groups.

The ***model molecule*** (see Figure 105 page 182) consists of three hydrogen atoms and two carbon atoms. The occupation of carbons is set to zero so that they don't contribute to structure factors. The rotation axis of the model molecule is defined by vector C1m-C2m. The z axis in the actual position is defined respectively by the center to which the hydrogen atoms are bound and the atom to which the methyl group is attached.

The rotational z axis can be set with help of the "Molecular transformation" form. The vector defining the direction of the axis can be entered by coordinates or by pair of atom names from the atomic part of the m40 file. In the latter case the vector coordinates are calculated as the difference of the second and the first atom position. In Figure 104 - Figure 111 the whole process is illustrated for a molecule with two CH_3 groups (the molecule is shown in Figure 107, page 185).

Figure 104 `M40` *before introducing CH_3 molecules*

Atoms C11,O11,....,H113 form the molecule shown in Figure 107.The methyl hydrogens bounded to C14 and C15 are not yet present (they will be added with the CH3 molecule from `m45`*)*

```
   90    0    0    1
 1.803017 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 1.141678 0.000000 0.000000 0.000000 0.000000 0.000000      100000
                                             . . .
C11       1  2     1.000000 0.869038 0.451124 0.212041
 0.016098 0.017315 0.015663-0.002421 0.007412-0.001486      0111111111
O11       4  2     1.000000 0.727352 0.471339 0.188297
 0.018301 0.041113 0.026374 0.007646 0.015376 0.010597      0111111111
O12       4  2     1.000000 0.956563 0.418208 0.311352
 0.022078 0.024367 0.015739-0.000031 0.007045 0.004846      0111111111
C12       1  2     1.000000 0.911684 0.477184 0.098719
 0.009718 0.020383 0.012069 0.001083 0.003577 0.000915      0111111111
N11       3  2     1.000000 1.031733 0.428122 0.089096
 0.012458 0.017874 0.012092-0.000285 0.003915 0.000004      0111111111
C13       1  2     1.000000 0.958955 0.558687 0.118413
 0.016140 0.016664 0.017376 0.002629 0.007401 0.002030      0111111111
C14       1  2     1.000000 0.963531 0.591615-0.010491
 0.034990 0.021687 0.024710-0.000779 0.013778 0.007109      0111111111
C15       1  2     1.000000 1.109120 0.572727 0.242523
 0.024076 0.018183 0.025583-0.003187 0.005030-0.001383      0111111111
H11       2  1     1.000000 0.686709 0.460450 0.243979
 0.033046 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H12       2  1     1.000000 0.824757 0.467027 0.021842
 0.017337 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H13       2  1     1.000000 0.877679 0.582610 0.137760
 0.007300 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H111      2  1     1.000000 0.996344 0.382553 0.079640
 0.022602 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H112      2  1     1.000000 1.059544 0.438538 0.019206
 0.031130 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H113      2  1     1.000000 1.119485 0.429011 0.163605
 0.036050 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
                                             . . .
H452      2  1     1.000000 1.608403 0.085468 0.939454
 0.054029 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H453      2  1     1.000000 1.521280 0.132241 0.810674
 0.072232 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
```

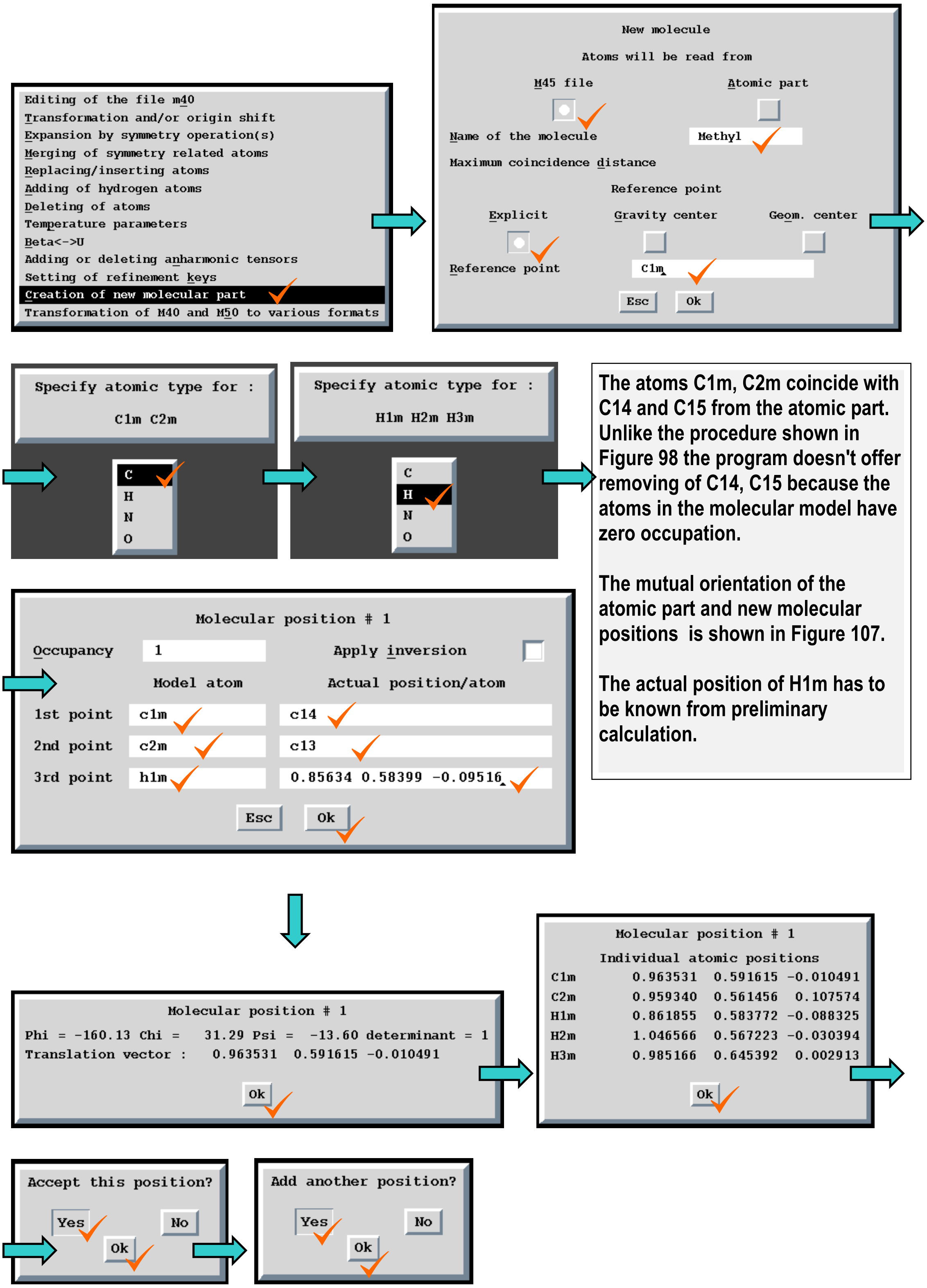
Figure 105 `M45` *with a CH_3 molecule*

Note that C1m and C2m have zero occupation

```
 1.000 1.000 1.000 90. 90. 120.
C1m       1  1     0.000000 0.000000 0.000000 0.000000
C2m       1  1     0.000000 0.000000 0.000000 1.400000
H1m       2  1     1.000000 0.942804 0.000000-0.333333
H2m       2  1     1.000000 0.000000 0.942804-0.333333
H3m       2  1     1.000000-0.942804-0.942804-0.333333
```

Figure 106 *Adding CH₃ molecule from m45 and defining its molecular positions*

This procedure is commented in Figure 101. The m45 with CH3 molecules is shown in Figure 105.



Atoms will be read from

M45 file ● Atomic part ☐

Name of the molecule: Methyl

Maximum coincidence distance

Reference point: Explicit ● Gravity center ☐ Geom. center ☐

Reference point: C1m

Specify atomic type for : C1m C2m — C

Specify atomic type for : H1m H2m H3m — H

Molecular position # 1

Occupancy 1 Apply inversion ☐

	Model atom	Actual position/atom
1st point	c1m	c14
2nd point	c2m	c13
3rd point	h1m	0.85634 0.58399 -0.09516

The atoms C1m, C2m coincide with C14 and C15 from the atomic part. Unlike the procedure shown in Figure 98 the program doesn't offer removing of C14, C15 because the atoms in the molecular model have zero occupation.

The mutual orientation of the atomic part and new molecular positions is shown in Figure 107.

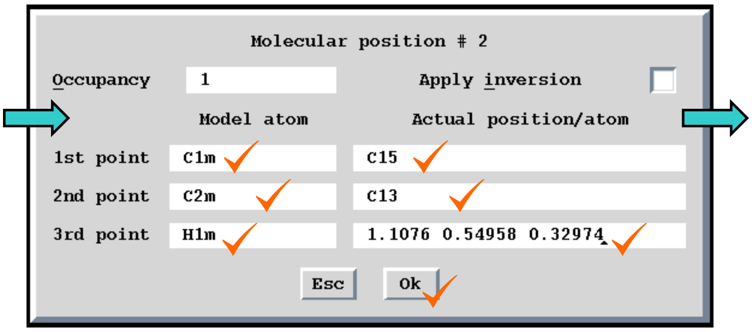
The actual position of H1m has to be known from preliminary calculation.

```
Molecular position # 1
Phi = -160.13 Chi =   31.29 Psi =  -13.60 determinant = 1
Translation vector :   0.963531  0.591615 -0.010491
```

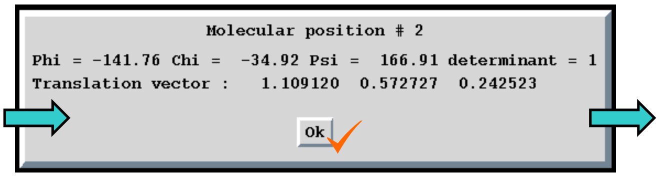
```
Molecular position # 1
Individual atomic positions
C1m       0.963531  0.591615 -0.010491
C2m       0.959340  0.561456  0.107574
H1m       0.861855  0.583772 -0.088325
H2m       1.046566  0.567223 -0.030394
H3m       0.985166  0.645392  0.002913
```

Accept this position?

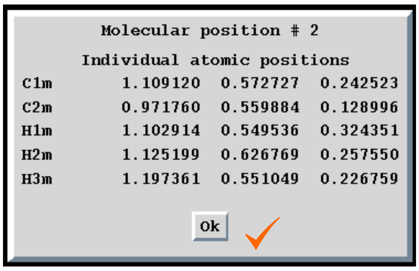
Add another position?



```
Molecular position # 2
Occupancy     1              Apply inversion  ☐
              Model atom     Actual position/atom
1st point     C1m            C15
2nd point     C2m            C13
3rd point     H1m            1.1076 0.54958 0.32974
```



```
Molecular position # 2
Phi = -141.76 Chi =  -34.92 Psi =  166.91 determinant = 1
Translation vector :   1.109120  0.572727  0.242523
```



```
Molecular position # 2
Individual atomic positions
C1m        1.109120  0.572727  0.242523
C2m        0.971760  0.559884  0.128996
H1m        1.102914  0.549536  0.324351
H2m        1.125199  0.626769  0.257550
H3m        1.197361  0.551049  0.226759
```

As the occupation of carbons in the model molecule is zero their superposition with the ones in the atomic structure is possible. The resulting structure is shown in Figure 107. Figure 108 shows the corresponding `m40`.

The new `m40` still contains molecular positions with default rotation axes. The next step, i.e. setting the rotation axes along vectors C13-C14 and C13-C15, is shown in Figure 109. Figure 110 shows m40 file with new definition of rotation axis. In Figure 111 a part of the listing of *REFINE* is presented showing refinement of the rotation angle along these rotation axes.

Figure 107 *The mutual orientation of the atomic part and new molecular positions*

This figure illustrates the procedure shown in Figure 106.

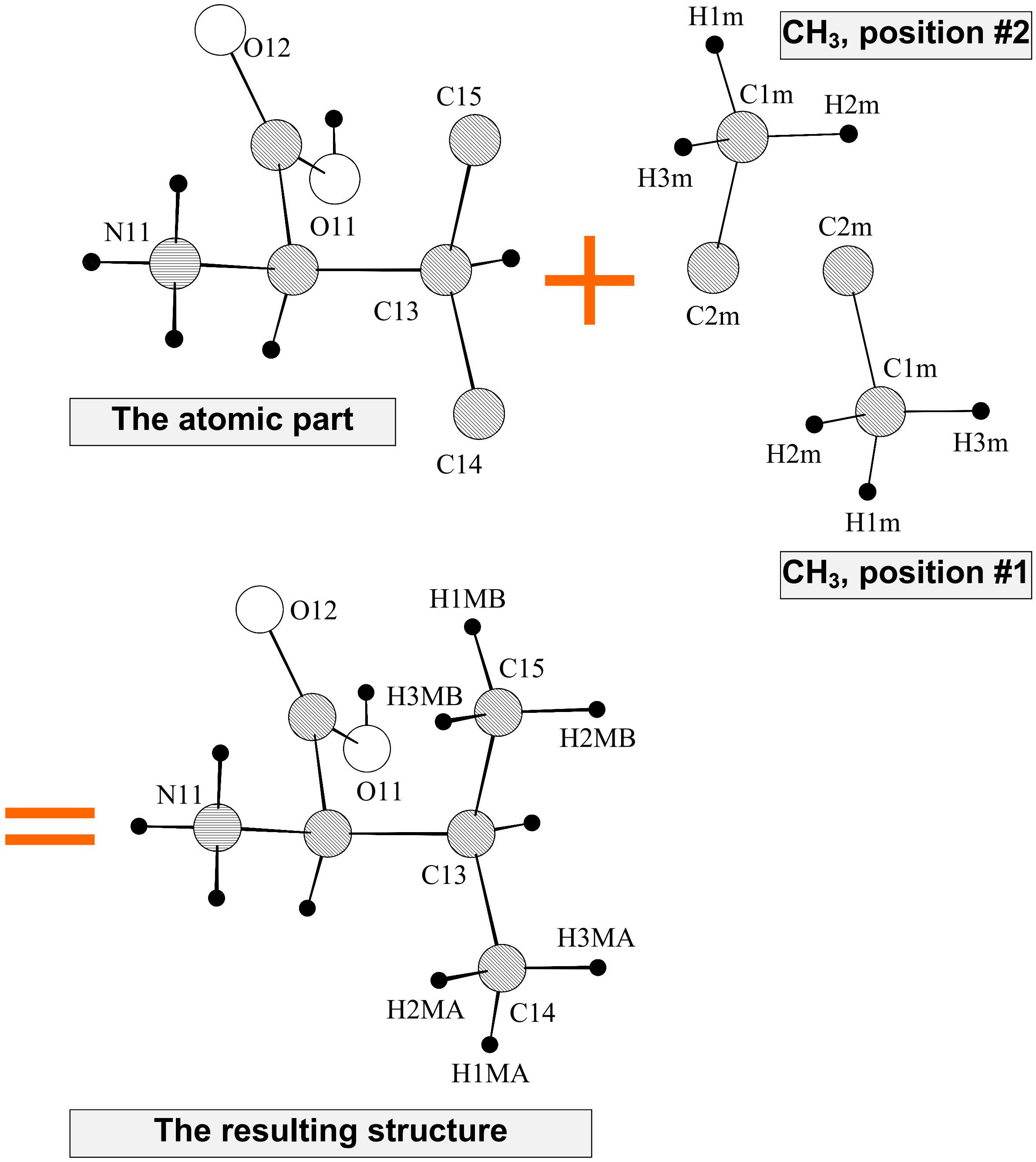


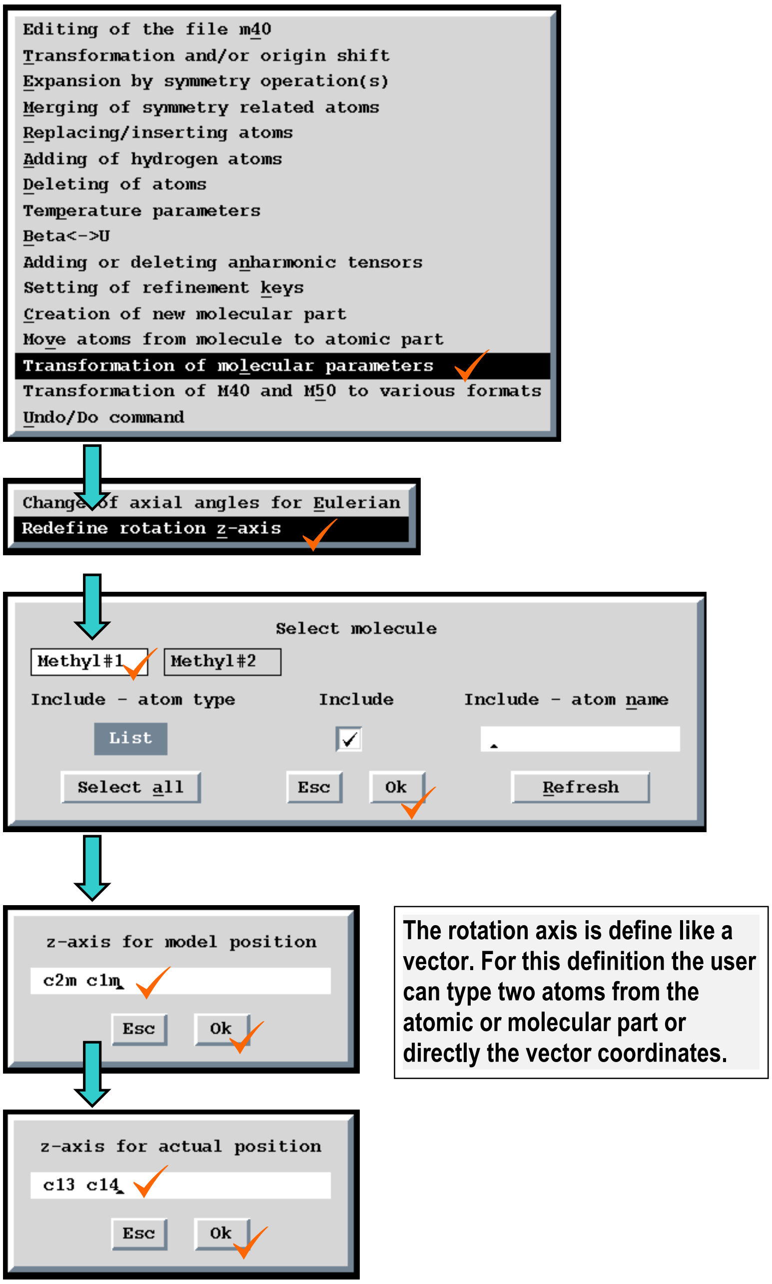
Figure 108 *`M40` after adding CH_3 molecule*

This file is the result of a procedure shown in Figure 106, page 183 applied to `m40` from Figure 104, page 182.

```
   90    1    0    1
    5    2
 1.803017 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 1.141678 0.000000 0.000000 0.000000 0.000000 0.000000      100000
. . .
C11       1  2     1.000000 0.869038 0.451124 0.212041
 0.016098 0.017315 0.015663-0.002421 0.007412-0.001486      0111111111
O11       4  2     1.000000 0.727352 0.471339 0.188297
 0.018301 0.041113 0.026374 0.007646 0.015376 0.010597      0111111111
O12       4  2     1.000000 0.956563 0.418208 0.311352
 0.022078 0.024367 0.015739-0.000031 0.007045 0.004846      0111111111
C12       1  2     1.000000 0.911684 0.477184 0.098719
 0.009718 0.020383 0.012069 0.001083 0.003577 0.000915      0111111111
N11       3  2     1.000000 1.031733 0.428122 0.089096
 0.012458 0.017874 0.012092-0.000285 0.003915 0.000004      0111111111
C13       1  2     1.000000 0.958955 0.558687 0.118413
 0.016140 0.016664 0.017376 0.002629 0.007401 0.002030      0111111111
C14       1  2     1.000000 0.963531 0.591615-0.010491
 0.034990 0.021687 0.024710-0.000779 0.013778 0.007109      0111111111
C15       1  2     1.000000 1.109120 0.572727 0.242523
 0.024076 0.018183 0.025583-0.003187 0.005030-0.001383      0111111111
H11       2  1     1.000000 0.686709 0.460450 0.243979
 0.033046 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H12       2  1     1.000000 0.824757 0.467027 0.021842
 0.017337 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H13       2  1     1.000000 0.877679 0.582610 0.137760
 0.007300 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H111      2  1     1.000000 0.996344 0.382553 0.079640
 0.022602 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H112      2  1     1.000000 1.059544 0.438538 0.019206
 0.031130 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H113      2  1     1.000000 1.119485 0.429011 0.163605
 0.036050 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
. . .
H452      2  1     1.000000 1.608403 0.085468 0.939454
 0.054029 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H453      2  1     1.000000 1.521280 0.132241 0.810674
 0.072232 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
Methyl    0                 C1m
C1m       1  1     0.000000 0.000000 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
C2m       1  1     0.000000 0.065138 0.000000 0.142150
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
H1m       2  1     1.000000 0.083901 0.000000-0.033845
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
H2m       2  1     1.000000-0.065214 0.044791-0.033845
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
H3m       2  1     1.000000-0.065214-0.044791-0.033845
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
pos#1     1        1.000000                                 000  0  0  0
 -160.127   31.293  -13.596 0.963531 0.591615-0.010491      0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#2     1        1.000000                                 000  0  0  0
 -141.760  -34.920  166.910 1.109120 0.572727 0.242523      0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
```

Figure 109 *Definition of a rotation axis for a molecule*

This procedure defines rotation axes for CH_3 groups. The groups were previously set through a procedure shown in Figure 106, page 183.



z-axis for model position: c2m c1m

z-axis for actual position: c13 c14

The rotation axis is define like a vector. For this definition the user can type two atoms from the atomic or molecular part or directly the vector coordinates.

. . . . the same for Methyl#2.

Figure 110 *M40 after redefinition of rotation axes for CH_3 groups*

This file is the result of a procedure shown in Figure 109 page 187 applied to `m40` *from Figure 108 page 186.*

```
   90    1    0    1
    5    2
 1.803017 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
 1.141678 0.000000 0.000000 0.000000 0.000000 0.000000      100000
. . .
C11       1  2     1.000000 0.869038 0.451124 0.212041
 0.016098 0.017315 0.015663-0.002421 0.007412-0.001486      0111111111
O11       4  2     1.000000 0.727352 0.471339 0.188297
 0.018301 0.041113 0.026374 0.007646 0.015376 0.010597      0111111111
O12       4  2     1.000000 0.956563 0.418208 0.311352
 0.022078 0.024367 0.015739-0.000031 0.007045 0.004846      0111111111
C12       1  2     1.000000 0.911684 0.477184 0.098719
 0.009718 0.020383 0.012069 0.001083 0.003577 0.000915      0111111111
N11       3  2     1.000000 1.031733 0.428122 0.089096
 0.012458 0.017874 0.012092-0.000285 0.003915 0.000004      0111111111
C13       1  2     1.000000 0.958955 0.558687 0.118413
 0.016140 0.016664 0.017376 0.002629 0.007401 0.002030      0111111111
C14       1  2     1.000000 0.963531 0.591615-0.010491
 0.034990 0.021687 0.024710-0.000779 0.013778 0.007109      0111111111
C15       1  2     1.000000 1.109120 0.572727 0.242523
 0.024076 0.018183 0.025583-0.003187 0.005030-0.001383      0111111111
H11       2  1     1.000000 0.686709 0.460450 0.243979
 0.033046 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H12       2  1     1.000000 0.824757 0.467027 0.021842
 0.017337 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H13       2  1     1.000000 0.877679 0.582610 0.137760
 0.007300 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H111      2  1     1.000000 0.996344 0.382553 0.079640
 0.022602 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H112      2  1     1.000000 1.059544 0.438538 0.019206
 0.031130 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H113      2  1     1.000000 1.119485 0.429011 0.163605
 0.036050 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
. . .
H452      2  1     1.000000 1.608403 0.085468 0.939454
 0.054029 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
H453      2  1     1.000000 1.521280 0.132241 0.810674
 0.072232 0.000000 0.000000 0.000000 0.000000 0.000000      0111100000
Methyl    0                 C1m
C1m       1  1     0.000000 0.000000 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
C2m       1  1     0.000000 0.065138 0.000000 0.142150
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
H1m       2  1     1.000000 0.083901 0.000000-0.033845
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
H2m       2  1     1.000000-0.065214 0.044791-0.033845
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
H3m       2  1     1.000000-0.065214-0.044791-0.033845
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
pos#1     1        1.000000                                 000  0  0  0
 -156.303    0.000    0.000 0.963531 0.591615-0.010491      0000000
 C2m C1m                    C13 C14
pos#2     1        1.000000                                 000  0  0  0
   -1.841    0.000    0.000 1.109120 0.572727 0.242523      0000000
 C2m C1m                    C13 C15
```

Figure 111 *The refinement listing showing refinement of CH_3 groups*
The angles chi and psi are not refined because of redefinition of rotation axes.

```
=========================================================================================
Methyl#1      ai       phi       chi        psi      x-trans   y-trans   z-trans  ch/esd
 0         1.000000 -156.3030  0.000000  0.000000  0.963531  0.591615 -0.010491* -6.01
 1         1.000000 -157.1601  0.000000  0.000000  0.959992  0.589178 -0.015414*  0.14
 2         1.000000 -157.2108  0.000000  0.000000  0.959964  0.589132 -0.015307* -0.04
 3         1.000000 -157.2137  0.000000  0.000000  0.959955  0.589119 -0.015340
esd        0.000000    0.4850  0.000000  0.000000  0.000914  0.000461  0.000769
=========================================================================================
Methyl#2      ai       phi       chi        psi      x-trans   y-trans   z-trans  ch/esd
 0         1.000000 -1.841000  0.000000  0.000000  1.109120  0.572727* 0.242523   5.17
 1         1.000000 -3.379539  0.000000  0.000000  1.111795  0.575330* 0.242049  -0.31
 2         1.000000 -3.473298* 0.000000  0.000000  1.111667  0.575181  0.242014  -0.07
 3         1.000000 -3.505554  0.000000  0.000000  1.111650  0.575211  0.242032
esd        0.000000  0.481423  0.000000  0.000000  0.000819  0.000478  0.000792
=========================================================================================
```

Reference point, symmetry restrictions and refinement keys

The basic information about the reference point is given in page 141.

Symmetry restrictions of molecular parameters (i.e. setting of the refinement keys, see page 223 for details) are calculated from the site symmetry of the reference point in the first molecular position. For example in Figure 114, page197, the molecular position modulation parameters are restricted because the reference point (0,y,¼) (equal to the position of As in the model molecule) falls into a special position when transformed to the first molecular position. On the other hand the TLS parameters of the molecule shown in **Figure 120**(c), page 214 are not restricted because the reference point (0,0,0) is transformed to the general position in the first molecular position.

Symmetry restrictions of atomic parameters of the model molecule are derived from the site symmetry of the points to which the model atoms are transformed for the first molecular position. Therefore, in Figure 114, page197, the atomic temperature modulation parameters are restricted for atom As but not for the remaining atoms of the model molecule. The exception is the case when a local symmetry is applied to the model molecule. Then the symmetry restrictions of atomic model parameters result also from the given point group symmetry.

The basic assumption is that *all molecular positions have the same site symmetry*.

In the refinement using the automatic mode for setting of refinement keys (see page 223) the position of the first atom of the model molecule is always fixed in order to prevent shifts of the whole molecule during the refinement. The shifts are refined for each molecular position through the translation parameters `x-trans`, `y-trans` and `z-trans` (see page 142).

The rotation parameters `phi`, `chi` and `psi` of the first molecular position are also automatically fixed because refinement of coordinates of the model atoms can cause rotation of the model molecule which may conflict with the rotation of the first molecular position. This automatic fixation may cause difficulties in case when rotation of the molecule through the refinement of coordinates is impossible - see footnote in page 214.

Transferring of atoms of molecule to the atomic part

The tool "Move atoms from molecule to atomic part" can be used for moving part of atoms or all atoms of a model molecule. Each molecular position originates a new atom in the atomic part with a name combined from its name in the model molecule and "a", "b", "c", ... extension for the 1st, 2nd, 3rd, ... position. If all toms of the model molecule are transformed to the atomic part of `m40`, the basic molecular parameters concerning molecular positions and also the molecular header are automatically removed from the file. A useful test of a successful transferring of atoms is that the R value resulting from the zero cycle of the refinement carried out after the transformation should be the same as the one before the transformation.

Transformation of molecular parameters

This tool has been already presented in Figure 109 in page 187 where it is used for redefinition of the molecular rotation axis. Another option is "Change the axial angles to Eulerian". This changes the parameter `Irot` in the header of `m40` (see page 107) defining the type of molecular rotations. See page 143 for underlying theory.

If in the Eulerian setting the angle $\chi \cong 0$, the remaining angles ϕ and ψ are strongly correlated and the refinement can crash on the singularity. This is the case when the Eulerian angles should be transformed to the axial ones. On the other hand the refinement of the axial rotations can fail for some angles, too.

3.2.8 Choosing between molecular and atomic model parameters

Each atom of a model molecule has refinable atomic parameters. They define the shape of the model molecule, its temperature and modulation parameters. These parameters are transformed to all molecular positions.

A common question when introducing a molecule is whether some atomic parameters of the model molecule have rigid body behaviour, i.e. if they can be refined for all model atoms together and for each molecular position independently. The relationship between atomic and molecular parameters is shown in Table 12.

Table 12 *Atomic and corresponding molecular parameters*

Atomic parameters	Corresponding molecular parameters
position modulation (page 123)	molecular position modulation (page 148)
temperature modulation (page 131)	modulation of TLS tensors (page 150)

A structure having a molecule with only one position and with all parameters refined as atomic parameters is equivalent to a structure without molecule. A maximum rigidity exhibits a molecule with rigid body temperature and modulation parameters having more than one position. An intermediate case often occurs in practice.

In case of modulations *JANA98* combines atomic and molecular contributions to the modulation function. Therefore for pure atomic modulations the number of modulation waves in the atomic header has to be positive (see page 116, parameters w_i) and the corresponding number in the basic molecular parameters (see page 142, parameters wm_i) has to be less or equal to zero. If the number of modulation waves in the atomic header is less or equal to zero and the corresponding number in the basic molecular parameters is positive the modulation is purely molecular (rigid body). With both numbers positive the contributions are combined.

This feature can be used for instance for testing if modulations of some atom behave within the rigid body approximation given by some existing (sufficiently large) molecule. The modulations of all atoms of the molecule except the one in question are refined in the molecular part. The modulations of the atom therefore consist of the contribution from the molecule and from its own atomic contributions. If the latter are below the 3σ limit the modulations of this atom can be refined in the rigid body approximation.

The modulation waves for atoms of the model molecule and for molecular positions are added or deleted with the same tool (Setting or deleting modulation waves) like for atoms in the atomic part - see page 168.

The transformation between atomic and molecular modulation parameters is not possible[1]. The individual modulations are usually changed to molecular ones by deleting individual modulation parameters (by setting number of waves to 0), setting initial molecular waves and refinement. The same procedure is used for changing molecular modulations to atomic ones. Refinement of molecular and individual

---

[1] *EDITM40* can only transform atoms from a molecule to the atomic part of `m40`. This tool transforms also modulations and makes the transformations separately for each molecular position.

position parameters together is also possible but only for a small fragment of the molecule (see Figure 117, page 205).

The change of atomic temperature parameters to TLS tensors can be done with a tool called "Temperature parameters" (see page 164). By this transformation the Ttype parameter (see page 116) is set to zero, the individual temperature parameters are deleted and the TLS parameters are set to **initial** values. The transformation of TLS tensors back to individual temperature parameters transforms TLS to individual temperature parameters (i.e. the temperature parameters are not lost).

The following examples show the basic type of changes between atomic and molecular parameters:

- Change from individual to molecular position modulation (Figure 113 page 194)
- Change from molecular to individual position modulation
- Change from individual temperature parameters to TLS tensors
- Introducing TLS modulation
- Change from TLS to individual temperature modulation
- Refinement of individual and molecular modulation together

Figure 112 *File `m50` used for examples in Figure 113 - Figure 117.*

The keywords in `m50` are explained in §0, page 77.

```
title iris
cell 4.818 16.001 6.374 90 99.36 90
lambda 0.56087
ndim 4
ncomp 1
qi 0.8607 0 0.5585
qr 0 0 0
spgroup C2/c(a0g)   0
lattice C
centro
symmetry      x1     x2     x3     x4
symmetry     -x1     x2 1/2-x3    -x4
unitsnumb 4
atom As
atweight 74.922 dmax 3 formula 1
f' 0.276 f" 1.331
formtab 32
   32.983   32.278   30.491   28.301   26.218   24.386   22.739   21.196
   19.722   18.313   16.976   15.717   14.540   13.451   12.454   11.552
   10.744   10.030    9.403    8.858    8.386    7.978    7.626    7.320
    7.053    6.817    6.606    6.415    6.239    6.076    5.922    5.774
atom K
atweight 39.098 dmax 0 formula 1
f' 0.14 f" 0.156
formtab 32
   18.999   18.206   16.732   15.244   13.726   12.269   10.980    9.909
    9.057    8.398    7.888    7.480    7.134    6.823    6.528    6.241
    5.956    5.674    5.395    5.120    4.851    4.589    4.336    4.093
    3.861    3.640    3.431    3.235    3.052    2.882    2.724    2.579
atom F
atweight 18.998 dmax 0 formula 4
f' 0.01 f" 0.006
formtab 32
    8.999    8.815    8.303    7.561    6.709    5.851    5.054    4.353
    3.760    3.270    2.873    2.558    2.309    2.112    1.957    1.834
    1.735    1.654    1.587    1.531    1.482    1.438    1.398    1.360
    1.324    1.289    1.255    1.221    1.187    1.153    1.120    1.087
atom O
atweight 15.999 dmax 0 formula 2
f' 0.006 f" 0.004
formtab 32
    7.999    7.798    7.246    6.472    5.623    4.808    4.089    3.489
    3.006    2.628    2.337    2.115    1.946    1.816    1.715    1.634
    1.568    1.512    1.463    1.419    1.377    1.337    1.298    1.260
    1.221    1.183    1.145    1.108    1.070    1.033    0.997    0.961
atom H
atweight 1.008 dmax 0 formula 2
f' 0 f" 0
formtab 32
    1.000    0.960    0.854    0.713    0.568    0.438    0.331    0.248
    0.184    0.138    0.103    0.078    0.060    0.046    0.036    0.028
    0.022    0.018    0.014    0.011    0.009    0.007    0.006    0.005
    0.005    0.004    0.004    0.003    0.003    0.003    0.003    0.003
noofref 4351
slimits 0.362376 0.453337 0.520919 0.575227 0.622421 0.665302 0.71257 0.91344
flimits 41 65.8 112.2 177.7 294.3 515.7 1068.1 53554
end
```

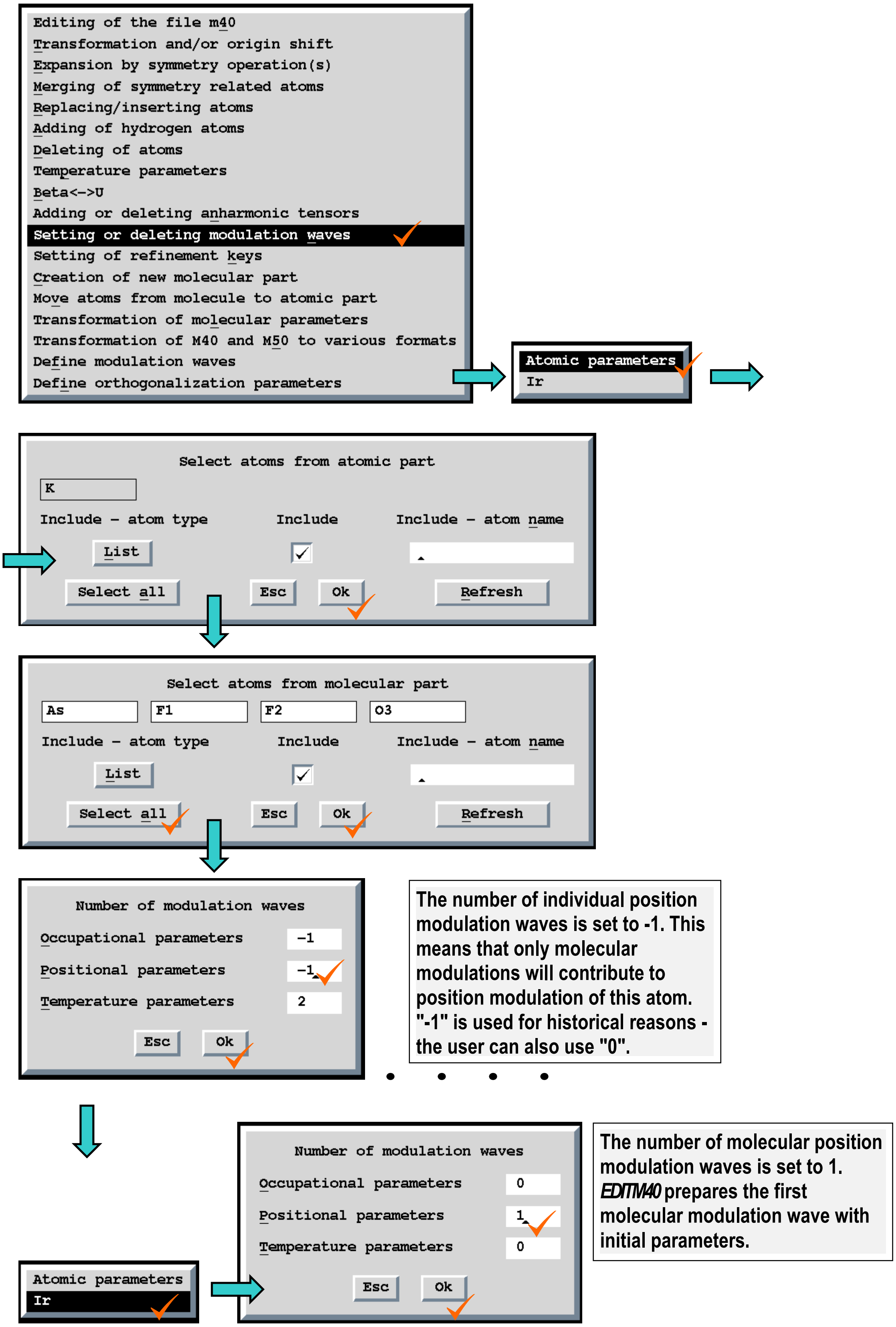
Figure 113 *Change from individual to molecular position modulation.*

(a) *The input* `m40` *with molecule "Ir". All atoms of the molecule have individual temperature parameters, position and temperature modulation. The molecule has only one position, which is almost identical with location of the model molecule.*

```
    1    1    1    1
    4    1
 1.233500 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.003581 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2     0.500000 0.500000 0.590911 0.750000      000  0  2  2
 0.029016 0.002668 0.013575 0.000000 0.004220 0.000000      0010111010
 0.008186 0.000000-0.013280 0.000000 0.017287 0.000000      101010
-0.012223 0.000000-0.007032 0.000000-0.003230 0.000000      101010
 0.000000 0.000000 0.000000-0.000954 0.000000 0.000345      000101
-0.000173-0.000513 0.002312 0.000000 0.000230 0.000000      111010
 0.000000 0.000000 0.000000-0.000181 0.000000 0.000177      000101
 0.000704-0.000519 0.001394 0.000000 0.001072 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  2     0.500000 0.000000 0.631034 0.250000      000 -1  2  2
 0.014251 0.001482 0.009415 0.000000 0.002167 0.000000      0000111010
-0.011066 0.000000-0.026381 0.000000 0.013900 0.000000      101010
-0.001408 0.000000 0.003936 0.000000 0.001399 0.000000      101010
 0.000000 0.000000 0.000000-0.000377 0.000000 0.000315      000101
 0.001454 0.000006 0.000200 0.000000-0.000069 0.000000      111010
 0.000000 0.000000 0.000000 0.000072 0.000000 0.000396      000101
 0.000324-0.000073-0.000110 0.000000 0.000559 0.000000      111010
 0.000000                                                   0
F1        3  2     1.000000-0.099442 0.627230 0.499285      000 -1  2  2
 0.032724 0.003663 0.011853-0.000583 0.008286 0.000649      0111111111
-0.008156 0.010790-0.024354-0.016899 0.014180-0.005781      111111
-0.001976-0.000100 0.003576 0.009636 0.001480 0.004834      111111
 0.003985-0.000299-0.000034-0.001854 0.001382-0.000983      111111
 0.003638-0.000413 0.001057-0.000696 0.001299-0.000550      111111
 0.003214-0.000586 0.000844-0.000326 0.002304-0.000025      111111
-0.004434-0.000095-0.000701 0.001984-0.001166-0.000102      111111
 0.000000                                                   0
F2        3  2     1.000000 0.237615 0.551939 0.331419      000 -1  2  2
 0.033182 0.002964 0.022325 0.004083 0.001742 0.001384      0111111111
 0.029160 0.013699 0.008396-0.004471 0.013626 0.008757      111111
 0.004339 0.001515 0.006794 0.002461 0.000564-0.003003      111111
-0.004358 0.000807 0.001764 0.000156-0.002772 0.001099      111111
 0.004291 0.000048-0.003561 0.000222-0.001914-0.000122      111111
 0.000010-0.000582-0.000682-0.001011 0.000806-0.000342      111111
 0.000869 0.000063 0.000758 0.001514 0.000612-0.000221      111111
 0.000000                                                   0
O3        4  2     1.000000 0.259402 0.704245 0.334580      000 -1  2  2
 0.029668 0.003528 0.025195-0.004808 0.012403-0.004559      0111111111
-0.048999 0.016093-0.062066-0.004630 0.014305 0.006973      111111
-0.007971 0.001982 0.000713 0.002482 0.003071-0.003983      111111
 0.007049-0.000319-0.002709-0.001257 0.001678 0.001388      111111
 0.002403 0.000256 0.002174-0.000508 0.000980-0.000301      111111
 0.001099-0.001438-0.003881 0.002401-0.001474 0.003273      111111
 0.004224 0.000760 0.008087-0.003033 0.009431-0.002921      111111
 0.000000                                                   0
pos#1     1        1.000000                                 000  0  0  0
    0.000    0.000    0.000 0.000000 0.000000 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
```

This is the model molecule. This structure model is equivalent to a structure without molecule as the molecule has only one position and all model parameters are refined

Figure 113(b) *The procedure to change individual position parameters to molecular ones.*



Select atoms from atomic part: K

Include ☑

Select atoms from molecular part: As, F1, F2, O3

Include ☑

Number of modulation waves

Occupational parameters -1

Positional parameters -1

Temperature parameters 2

The number of individual position modulation waves is set to -1. This means that only molecular modulations will contribute to position modulation of this atom. "-1" is used for historical reasons - the user can also use "0".

• • • •

Number of modulation waves

Occupational parameters 0

Positional parameters 1

Temperature parameters 0

The number of molecular position modulation waves is set to 1. *EDITM40* prepares the first molecular modulation wave with initial parameters.

Figure 113 (c) *The resulting* `m40` *with initial parameters set for the first molecular modulation wave. The file with two molecular position modulation waves refined is used as the input file in the next example.*

```
    1    1    1    1
    4    1
 1.233500 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.003581 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2     0.500000 0.500000 0.590911 0.750000      000  0  2  2
 0.029016 0.002668 0.013575 0.000000 0.004220 0.000000      0010111010
 0.008186 0.000000-0.013280 0.000000 0.017287 0.000000      101010
-0.012223 0.000000-0.007032 0.000000-0.003230 0.000000      101010
 0.000000 0.000000 0.000000-0.000954 0.000000 0.000345      000101
-0.000173-0.000513 0.002312 0.000000 0.000230 0.000000      111010
 0.000000 0.000000 0.000000-0.000181 0.000000 0.000177      000101
 0.000704-0.000519 0.001394 0.000000 0.001072 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  2     0.500000 0.000000 0.631034 0.250000      000 -1  0  2
 0.014251 0.001482 0.009415 0.000000 0.002167 0.000000      0000111010
 0.000000 0.000000 0.000000-0.000377 0.000000 0.000315      000101
 0.001454 0.000006 0.000200 0.000000-0.000069 0.000000      111010
 0.000000 0.000000 0.000000 0.000072 0.000000 0.000396      000101
 0.000324-0.000073-0.000110 0.000000 0.000559 0.000000      111010
 0.000000                                                   0
F1        3  2     1.000000-0.099442 0.627230 0.499285      000 -1  0  2
 0.032724 0.003663 0.011853-0.000583 0.008286 0.000649      0111111111
 0.003985-0.000299-0.000034-0.001854 0.001382-0.000983      111111
 0.003638-0.000413 0.001057-0.000696 0.001299-0.000550      111111
 0.003214-0.000586 0.000844-0.000326 0.002304-0.000025      111111
-0.004434-0.000095-0.000701 0.001984-0.001166-0.000102      111111
 0.000000                                                   0
F2        3  2     1.000000 0.237615 0.551939 0.331419      000 -1  0  2
 0.033182 0.002964 0.022325 0.004083 0.001742 0.001384      0111111111
-0.004358 0.000807 0.001764 0.000156-0.002772 0.001099      111111
 0.004291 0.000048-0.003561 0.000222-0.001914-0.000122      111111
 0.000010-0.000582-0.000682-0.001011 0.000806-0.000342      111111
 0.000869 0.000063 0.000758 0.001514 0.000612-0.000221      111111
 0.000000                                                   0
O3        4  2     1.000000 0.259402 0.704245 0.334580      000 -1  0  2
 0.029668 0.003528 0.025195-0.004808 0.012403-0.004559      0111111111
 0.007049-0.000319-0.002709-0.001257 0.001678 0.001388      111111
 0.002403 0.000256 0.002174-0.000508 0.000980-0.000301      111111
 0.001099-0.001438-0.003881 0.002401-0.001474 0.003273      111111
 0.004224 0.000760 0.008087-0.003033 0.009431-0.002921      111111
 0.000000                                                   0
pos#1     1        1.000000                                 000  0  1  0
    0.000    0.000    0.000 0.000000 0.000000 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.000100 0.000100 0.000100 0.000100 0.000100 0.000100      111111
 0.000100 0.000100 0.000100 0.000100 0.000100 0.000100      111111
 0.000000
```

Number of position modulation waves for atoms of the model molecule is 0 meaning that the modulations are calculated in molecular part within the rigid body approximation

This is the 1st molecular position modulation wave with initial parameters and refinement keys.

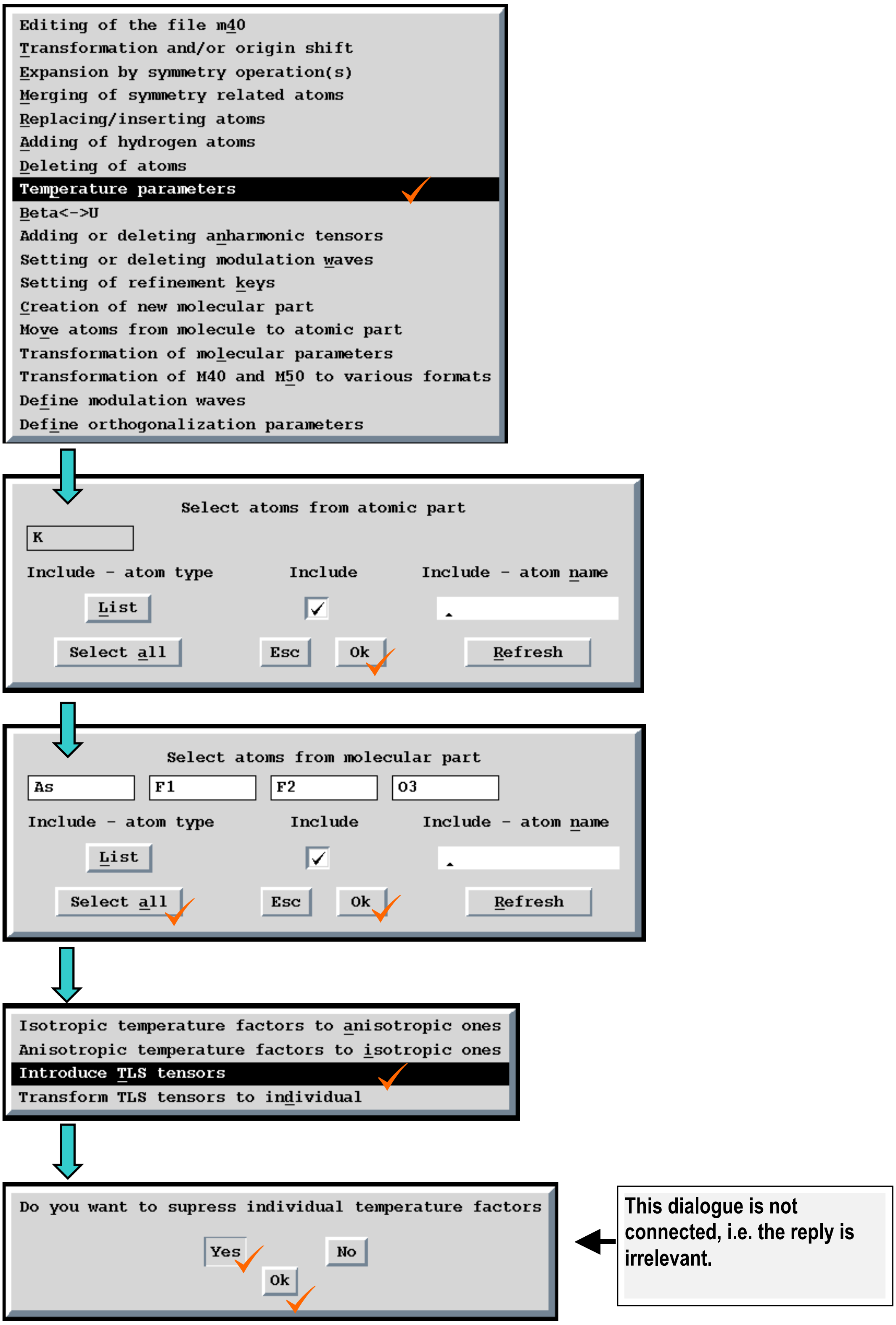
Figure 114 *Change from individual to TLS temperature parameters.*

(a) *The input* `m40` *with molecule "Ir". Atoms of the molecule have individual temperature parameters and their modulations. The position modulation parameters are refined in the molecular part. The file results from the procedure shown in Figure 113, followed by adding of another position modulation wave and refinement.*

```
    1    1    1    1
    4    1
 1.233566 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
-0.001334 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2     0.500000 0.500000 0.590934 0.750000      000  0  2  2
 0.028994 0.002669 0.013609 0.000000 0.004210 0.000000      0010111010
 0.008041 0.000000-0.013254 0.000000 0.017274 0.000000      101010
-0.012682 0.000000-0.006921 0.000000-0.003292 0.000000      101010
 0.000000 0.000000 0.000000-0.000918 0.000000 0.000315      000101
-0.000070-0.000510 0.002317 0.000000 0.000158 0.000000      111010
 0.000000 0.000000 0.000000-0.000250 0.000000 0.000203      000101
 0.000460-0.000517 0.001334 0.000000 0.000804 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  2     0.500000 0.000000 0.631034 0.250000      000 -1  0  2
 0.014290 0.001484 0.009401 0.000000 0.002176 0.000000      0000111010
 0.000000 0.000000 0.000000-0.000397 0.000000 0.000311      000101
 0.001495 0.000006 0.000184 0.000000-0.000056 0.000000      111010
 0.000000 0.000000 0.000000 0.000075 0.000000 0.000380      000101
 0.000304-0.000075-0.000129 0.000000 0.000586 0.000000      111010
 0.000000                                                   0
F1        3  2     1.000000-0.099958 0.627151 0.499018      000 -1  0  2
 0.033016 0.003687 0.011917-0.000542 0.008448 0.000632      0111111111
 0.004425-0.000378 0.000617-0.002000 0.001744-0.001087      111111
 0.003334-0.000347 0.000943-0.000668 0.001176-0.000463      111111
 0.003765-0.000670 0.001541-0.000568 0.002360 0.000007      111111
-0.005254-0.000199-0.000573 0.001926-0.001478-0.000148      111111
 0.000000                                                   0
F2        3  2     1.000000 0.237807 0.552021 0.331383      000 -1  0  2
 0.033284 0.002918 0.022232 0.004092 0.001943 0.001228      0111111111
-0.004573 0.000856 0.001148 0.000105-0.003033 0.000978      111111
 0.003627 0.000006-0.003438 0.000156-0.001996-0.000212      111111
 0.000784-0.000442-0.000062-0.000935 0.000625 0.000062      111111
 0.000568 0.000061 0.001199 0.001439 0.000748-0.000172      111111
 0.000000                                                   0
O3        4  2     1.000000 0.259747 0.704321 0.334748      000 -1  0  2
 0.029854 0.003628 0.025478-0.004927 0.012509-0.004698      0111111111
 0.007986-0.000453-0.002356-0.001169 0.002582 0.001678      111111
 0.003601 0.000404 0.003345-0.000755 0.001754-0.000521      111111
-0.000017-0.001508-0.003817 0.002362-0.001954 0.003239      111111
 0.004407 0.000668 0.008726-0.002768 0.008894-0.003002      111111
 0.000000                                                   0
pos#1     1        1.000000                                 000  0  2  0
    0.000    0.000    0.000 0.000000-0.000003 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
-0.011060 0.000000-0.026436 0.000000 0.013900 0.000000      101010
-0.001300 0.000000 0.003941 0.000000 0.001395 0.000000      101010
-0.032081 0.000000 0.020306 0.000000-0.003041 0.000000      101010
-0.001761 0.000000 0.003223 0.000000 0.001533 0.000000      101010
 0.0000000
```

This is the model molecule.

Figure 114 (b) *The procedure to change individual temperature parameters to TLS tensors*



Select atoms from atomic part

K

Select atoms from molecular part

As | F1 | F2 | O3

Do you want to supress individual temperature factors

This dialogue is not connected, i.e. the reply is irrelevant.

Figure 114(c) *The resulting* `m40` *with initial parameters set for the TLS tensors refinement.*

```
    1    1    1    1
    4    1
 1.233566 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
-0.001334 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2     0.500000 0.500000 0.590934 0.750000      000  0  2  2
 0.028994 0.002669 0.013609 0.000000 0.004210 0.000000      0010111010
 0.008041 0.000000-0.013254 0.000000 0.017274 0.000000      101010
-0.012682 0.000000-0.006921 0.000000-0.003292 0.000000      101010
 0.000000 0.000000 0.000000-0.000918 0.000000 0.000315      000101
-0.000070-0.000510 0.002317 0.000000 0.000158 0.000000      111010
 0.000000 0.000000 0.000000-0.000250 0.000000 0.000203      000101
 0.000460-0.000517 0.001334 0.000000 0.000804 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  0     0.500000 0.000000 0.631034 0.250000      000 -1  0  2
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000111010
 0.000000 0.000000 0.000000-0.000397 0.000000 0.000311      000101
 0.001495 0.000006 0.000184 0.000000-0.000056 0.000000      111010
 0.000000 0.000000 0.000000 0.000075 0.000000 0.000380      000101
 0.000304-0.000075-0.000129 0.000000 0.000586 0.000000      111010
 0.000000                                                   0
F1        3  0     1.000000-0.099958 0.627151 0.499018      000 -1  0  2
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111111111
 0.004425-0.000378 0.000617-0.002000 0.001744-0.001087      111111
 0.003334-0.000347 0.000943-0.000668 0.001176-0.000463      111111
 0.003765-0.000670 0.001541-0.000568 0.002360 0.000007      111111
-0.005254-0.000199-0.000573 0.001926-0.001478-0.000148      111111
 0.000000                                                   0
F2        3  0     1.000000 0.237807 0.552021 0.331383      000 -1  0  2
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111111111
-0.004573 0.000856 0.001148 0.000105-0.003033 0.000978      111111
 0.003627 0.000006-0.003438 0.000156-0.001996-0.000212      111111
 0.000784-0.000442-0.000062-0.000935 0.000625 0.000062      111111
 0.000568 0.000061 0.001199 0.001439 0.000748-0.000172      111111
 0.000000                                                   0
O3        4  0     1.000000 0.259747 0.704321 0.334748      000 -1  0  2
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111111111
 0.007986-0.000453-0.002356-0.001169 0.002582 0.001678      111111
 0.003601 0.000404 0.003345-0.000755 0.001754-0.000521      111111
-0.000017-0.001508-0.003817 0.002362-0.001954 0.003239      111111
 0.004407 0.000668 0.008726-0.002768 0.008894-0.003002      111111
 0.000000                                                   0
pos#1     1        1.000000                                 000  0  2  0
    0.000    0.000    0.000 0.000000-0.000003 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.010000 0.010000 0.010000 0.000000 0.000000 0.000000      111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      111111
 0.000000 0.000000 0.000000                                 110
-0.011060 0.000000-0.026436 0.000000 0.013900 0.000000      101010
-0.001300 0.000000 0.003941 0.000000 0.001395 0.000000      101010
-0.032081 0.000000 0.020306 0.000000-0.003041 0.000000      101010
-0.001761 0.000000 0.003223 0.000000 0.001533 0.000000      101010
 0.000000
```

The parameter Ttype **(see page 134) is set to zero meaning that the temperature parameters are refined in the molecular part like TLS tensors. The individual temperature parameters U_{ij} are set to zero, too.**

TLS tensors: initial values and refinement keys.

Figure 115 *Introducing TLS modulation*

(a) *The input* `m40` *with molecule "Ir" . The temperature parameters are refined like TLS tensors but modulations of temperature parameters are still individual. The position modulation parameters are refined in the molecular part. The file results from the procedure shown in Figure 114, followed by refinement of the initial TLS parameters*[1]*.*

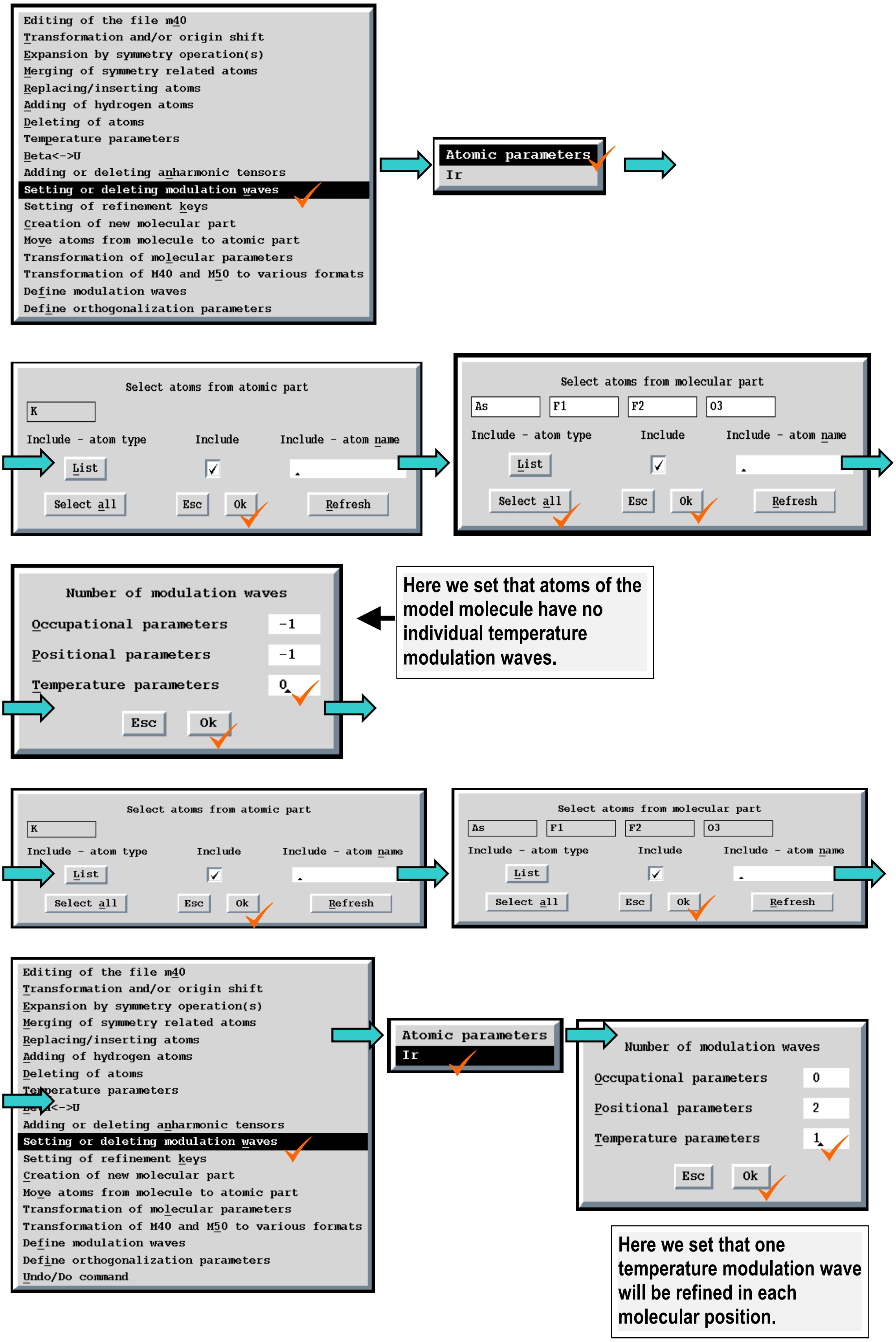
```
    1    1    1    1
    4    1
  1.234794 0.000000 0.000000 0.000000 0.000000 0.000000      100000
  0.000000
  0.017930 0.000000 0.000000 0.000000 0.000000 0.000000      100000
  0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K          2  2     0.500000 0.500000 0.590943 0.750000      000  0  2  2
  0.029000 0.002672 0.013612 0.000000 0.004203 0.000000      0010111010
  0.008053 0.000000-0.013250 0.000000 0.017274 0.000000      101010
 -0.012674 0.000000-0.006933 0.000000-0.003295 0.000000      101010
  0.000000 0.000000 0.000000-0.000945 0.000000 0.000304      000101
 -0.000146-0.000510 0.002324 0.000000 0.000154 0.000000      111010
  0.000000 0.000000 0.000000-0.000257 0.000000 0.000209      000101
  0.000435-0.000516 0.001311 0.000000 0.000777 0.000000      111010
  0.000000                                                   0
Ir         1                 As
As         1  0     0.500000 0.000000 0.631034 0.250000      000 -1  0  2
  0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
  0.000000 0.000000 0.000000-0.000397 0.000000 0.000311      000000
  0.001495 0.000006 0.000184 0.000000-0.000056 0.000000      000000
  0.000000 0.000000 0.000000 0.000075 0.000000 0.000380      000000
  0.000304-0.000075-0.000129 0.000000 0.000586 0.000000      000000
  0.000000                                                   0
F1         3  0     1.000000-0.099982 0.627143 0.498994      000 -1  0  2
  0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
  0.004425-0.000378 0.000617-0.002000 0.001744-0.001087      000000
  0.003334-0.000347 0.000943-0.000668 0.001176-0.000463      000000
  0.003765-0.000670 0.001541-0.000568 0.002360 0.000007      000000
 -0.005254-0.000199-0.000573 0.001926-0.001478-0.000148      000000
  0.000000                                                   0
. . . .

O3         4  0     1.000000 0.259792 0.704290 0.334738      000 -1  0  2
  0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
  0.007986-0.000453-0.002356-0.001169 0.002582 0.001678      000000
  0.003601 0.000404 0.003345-0.000755 0.001754-0.000521      000000
 -0.000017-0.001508-0.003817 0.002362-0.001954 0.003239      000000
  0.004407 0.000668 0.008726-0.002768 0.008894-0.003002      000000
  0.000000                                                   0
pos#1      1        1.000000                                 000  0  2  0
     0.000    0.000    0.000 0.000000 0.000001 0.000000      0000010
  0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
  0.014375 0.001490 0.009421 0.000000 0.002205 0.000000      111010
  0.010616 0.000608 0.005178 0.000000-0.002004 0.000000      111010
  0.002297 0.000000 0.000650 0.000000 0.000123 0.000000      101010
  0.000298 0.000000-0.002420                                 100
 -0.011055 0.000000-0.026441 0.000000 0.013895 0.000000      101010
 -0.001313 0.000000 0.003937 0.000000 0.001393 0.000000      101010
 -0.032074 0.000000 0.020323 0.000000-0.003045 0.000000      101010
 -0.001787 0.000000 0.003258 0.000000 0.001527 0.000000      101010
  0.000000                                                   0
```

TLS tensors: refined values

Molecular position modulation parameters

---

[1] It should be noted that combination of TLS tensors with atomic temperature modulation is an artificial case used only for the purpose of this manual. The refinement of individual temperature modulation parameters together with TLS tensors cannot work well. For this reason the relevant refinement keys are set to zero.



Number of modulation waves

Occupational parameters -1

Positional parameters -1

Temperature parameters 0

Here we set that atoms of the model molecule have no individual temperature modulation waves.

Number of modulation waves

Occupational parameters 0

Positional parameters 2

Temperature parameters 1

Here we set that one temperature modulation wave will be refined in each molecular position.

Figure 115 (b) *The procedure to change individual temperature modulation parameters to modulation of TLS tensors*

Figure 115(c) *The resulting* `m40` *with initial parameters prepared for the refinement of TLS tensors modulations.*

```
    1    1    1    1
    4    1
 1.234794 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.017930 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2     0.500000 0.500000 0.590943 0.750000      000  0  2  2
 0.029000 0.002672 0.013612 0.000000 0.004203 0.000000      0010111010
 0.008053 0.000000-0.013250 0.000000 0.017274 0.000000      101010
-0.012674 0.000000-0.006933 0.000000-0.003295 0.000000      101010
 0.000000 0.000000 0.000000-0.000945 0.000000 0.000304      000101
-0.000146-0.000510 0.002324 0.000000 0.000154 0.000000      111010
 0.000000 0.000000 0.000000-0.000257 0.000000 0.000209      000101
 0.000435-0.000516 0.001311 0.000000 0.000777 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  0     0.500000 0.000000 0.631034 0.250000      000 -1  0  0
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1        3  0     1.000000-0.099982 0.627143 0.498994      000 -1  0  0
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F2        3  0     1.000000 0.237892 0.552001 0.331404      000 -1  0  0
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
O3        4  0     1.000000 0.259792 0.704290 0.334738      000 -1  0  0
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
pos#1     1        1.000000                                 000  0  2  1
    0.000    0.000    0.000 0.000000 0.000001 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.014375 0.001490 0.009421 0.000000 0.002205 0.000000      111010
 0.010616 0.000608 0.005178 0.000000-0.002004 0.000000      111010
 0.002297 0.000000 0.000650 0.000000 0.000123 0.000000      101010
 0.000298 0.000000-0.002420                                 100
-0.011055 0.000000-0.026441 0.000000 0.013895 0.000000      101010
-0.001313 0.000000 0.003937 0.000000 0.001393 0.000000      101010
-0.032074 0.000000 0.020323 0.000000-0.003045 0.000000      101010
-0.001787 0.000000 0.003258 0.000000 0.001527 0.000000      101010
 0.000010 0.000010 0.000010 0.000010 0.000010 0.000010      111111
 0.000010 0.000010 0.000010 0.000010 0.000010 0.000010      111111
 0.000010 0.000010 0.000010 0.000010 0.000010 0.000010      111111
 0.000010 0.000010 0.000010 0.000010 0.000010 0.000010      111111
 0.000010 0.000010 0.000010 0.000010 0.000010 0.000010      111111
 0.000010 0.000010-0.000020                                 110
 0.000010 0.000010 0.000010 0.000010 0.000010 0.000010      111111
 0.000010 0.000010-0.000020                                 110
 0.000000                                                   0
```

TLS modulation parameters (the 1st modulation wave) with initial values and refinement keys.

Figure 116 *Change from TLS to individual temperature modulation*

(a) *The input* `m40` *with molecule "Ir" is shown in Figure 115(a), page 200. The temperature parameters of the model molecule are refined like TLS tensors.*

(b) *The procedure to transform TLS tensors to individual temperature modulation.*

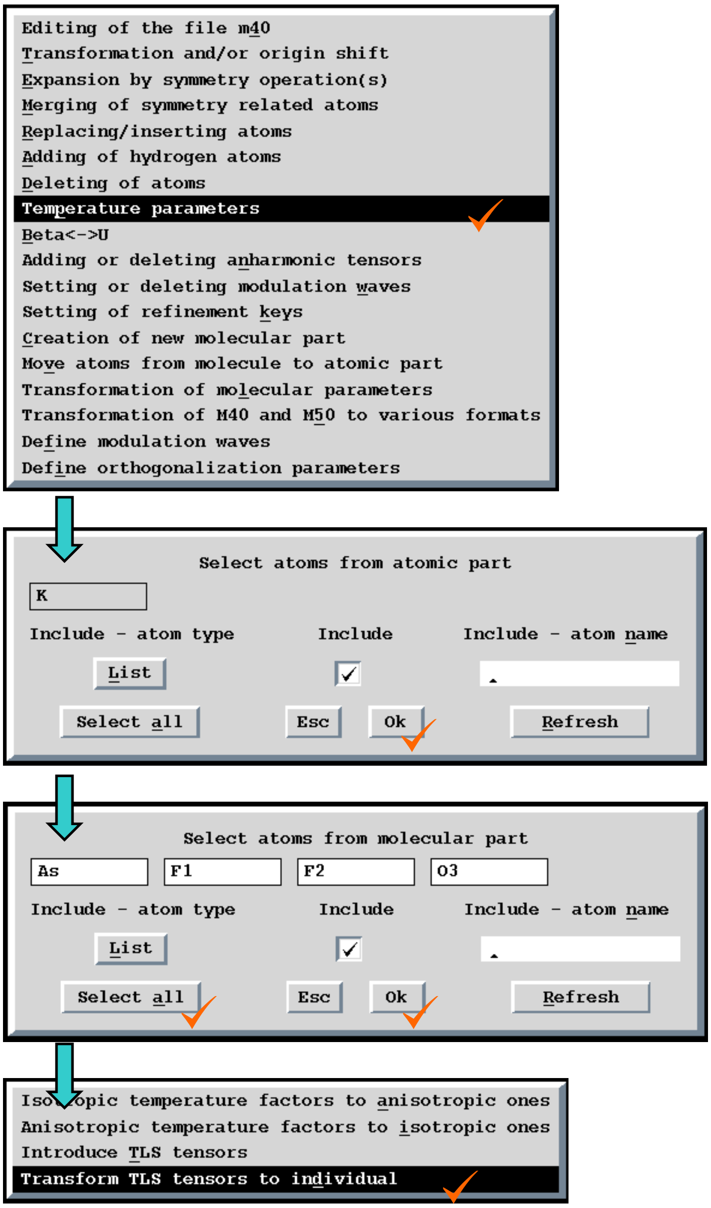


Figure 116(c) *The resulting* `m40` *with TLS tensors transformed to atomic temperature parameters.*

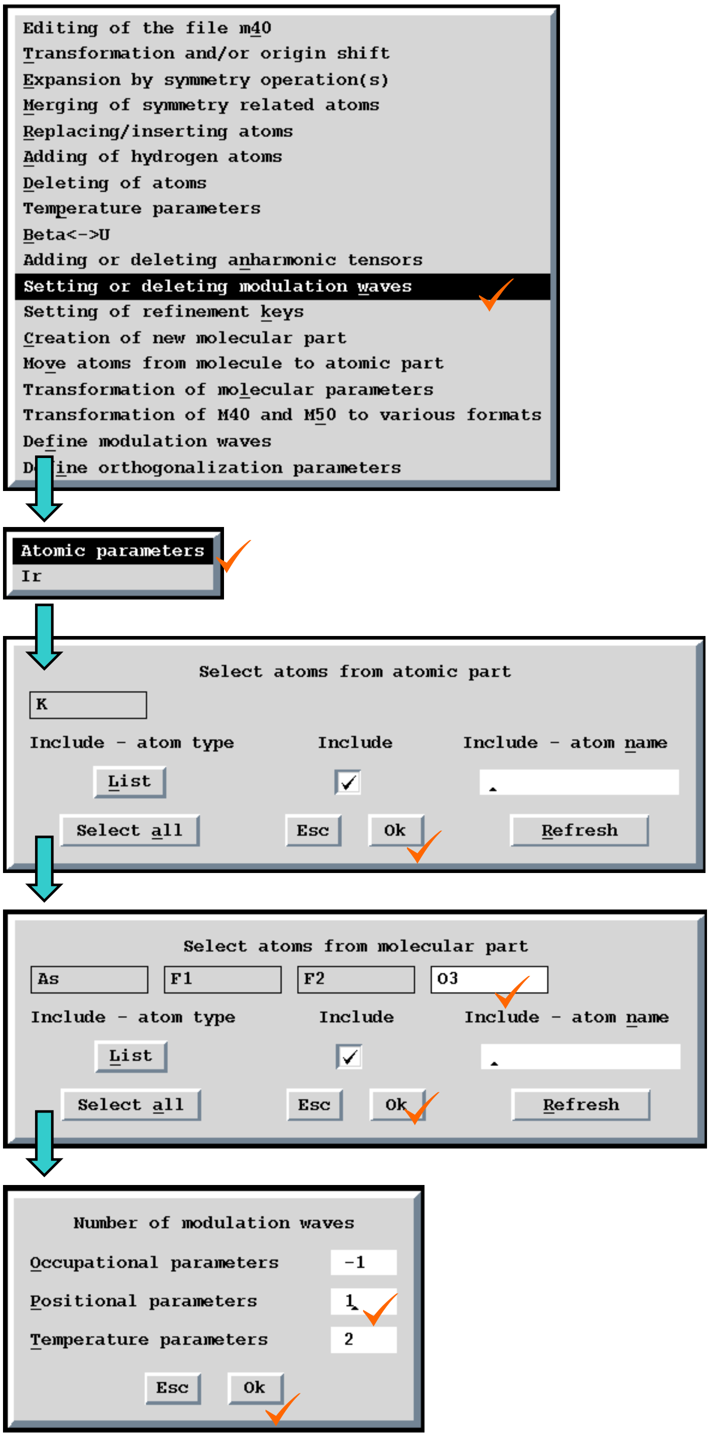
```
    1    1    1    1
    4    1
 1.234794 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.017930 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2      0.500000 0.500000 0.590943 0.750000      000  0  2  2
 0.029000 0.002672 0.013612 0.000000 0.004203 0.000000      0010111010
 0.008053 0.000000-0.013250 0.000000 0.017274 0.000000      101010
-0.012674 0.000000-0.006933 0.000000-0.003295 0.000000      101010
 0.000000 0.000000 0.000000-0.000945 0.000000 0.000304      000101
-0.000146-0.000510 0.002324 0.000000 0.000154 0.000000      111010
 0.000000 0.000000 0.000000-0.000257 0.000000 0.000209      000101
 0.000435-0.000516 0.001311 0.000000 0.000777 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  2      0.500000 0.000000 0.631034 0.250000      000 -1  0  2
 0.014375 0.001490 0.009421 0.000000 0.002205 0.000000      0000000000
 0.000000 0.000000 0.000000-0.000397 0.000000 0.000311      000000
 0.001495 0.000006 0.000184 0.000000-0.000056 0.000000      000000
 0.000000 0.000000 0.000000 0.000075 0.000000 0.000380      000000
 0.000304-0.000075-0.000129 0.000000 0.000586 0.000000      000000
 0.000000                                                   0
F1        3  2      1.000000-0.099982 0.627143 0.498994      000 -1  0  2
 0.033524 0.003678 0.011490-0.000405 0.008323 0.000604      0111000000
 0.004425-0.000378 0.000617-0.002000 0.001744-0.001087      000000
 0.003334-0.000347 0.000943-0.000668 0.001176-0.000463      000000
 0.003765-0.000670 0.001541-0.000568 0.002360 0.000007      000000
-0.005254-0.000199-0.000573 0.001926-0.001478-0.000148      000000
 0.000000                                                   0
F2        3  2      1.000000 0.237892 0.552001 0.331404      000 -1  0  2
 0.031954 0.003071 0.022588 0.004926 0.001318 0.001488      0111000000
-0.004573 0.000856 0.001148 0.000105-0.003033 0.000978      000000
 0.003627 0.000006-0.003438 0.000156-0.001996-0.000212      000000
 0.000784-0.000442-0.000062-0.000935 0.000625 0.000062      000000
 0.000568 0.000061 0.001199 0.001439 0.000748-0.000172      000000
 0.000000                                                   0
O3        4  2      1.000000 0.259792 0.704290 0.334738      000 -1  0  2
 0.029510 0.003333 0.026303-0.005111 0.012417-0.004628      0111000000
 0.007986-0.000453-0.002356-0.001169 0.002582 0.001678      000000
 0.003601 0.000404 0.003345-0.000755 0.001754-0.000521      000000
-0.000017-0.001508-0.003817 0.002362-0.001954 0.003239      000000
 0.004407 0.000668 0.008726-0.002768 0.008894-0.003002      000000
 0.000000                                                   0
pos#1     1         1.000000                                 000  0  2  0
     0.000    0.000   0.000 0.000000 0.000001 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
-0.011055 0.000000-0.026441 0.000000 0.013895 0.000000      101010
-0.001313 0.000000 0.003937 0.000000 0.001393 0.000000      101010
-0.032074 0.000000 0.020323 0.000000-0.003045 0.000000      101010
-0.001787 0.000000 0.003258 0.000000 0.001527 0.000000      101010
 0.000000
```

The dark shaded lines contain the transformed individual temperature parameters.

Figure 117 *Refinement of individual and molecular modulation together*

(a) *The input* `m40` *with molecule "Ir" is shown in Figure 114(a), page 197. The position modulations of the model molecule are refined in the molecular part.*

(b) *The procedure to add one individual position modulation wave to atom O3 of the model molecule.*



Select atoms from atomic part: K

Include ☑

Select atoms from molecular part: As F1 F2 O3

Include ☑

Occupational parameters -1

Positional parameters 1

Temperature parameters 2

Selection of all atoms of the model molecule is not possible. The major part of the model molecule must have only molecular modulation.

Figure 117(c) *The resulting* `m40` *with position modulation parameters of atom O3 calculated like combination of molecular (2 waves) and individual atomic (1 wave) contributions. The comparison of the individual position modulation parameters with their estimated standard deviations can be used as a measure how far is the position modulation of O3 from the rigid body approximation.*

```
    1    1    1    1
    4    1
 1.233062 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
-0.003372 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
K         2  2     0.500000 0.500000 0.590927 0.750000      000  0  2  2
 0.028974 0.002661 0.013591 0.000000 0.004226 0.000000      0010111010
 0.008163 0.000000-0.013260 0.000000 0.017298 0.000000      101010
-0.012632 0.000000-0.006926 0.000000-0.003274 0.000000      101010
 0.000000 0.000000 0.000000-0.000923 0.000000 0.000311      000101
-0.000144-0.000511 0.002318 0.000000 0.000166 0.000000      111010
 0.000000 0.000000 0.000000-0.000233 0.000000 0.000191      000101
 0.000315-0.000491 0.001401 0.000000 0.000897 0.000000      111010
 0.000000                                                   0
Ir        1                 As
As        1  2     0.500000 0.000000 0.631034 0.250000      000 -1  0  2
 0.014260 0.001484 0.009398 0.000000 0.002167 0.000000      0000111010
 0.000000 0.000000 0.000000-0.000392 0.000000 0.000311      000101
 0.001473 0.000007 0.000200 0.000000-0.000042 0.000000      111010
 0.000000 0.000000 0.000000 0.000082 0.000000 0.000395      000101
 0.000317-0.000080-0.000096 0.000000 0.000600 0.000000      111010
 0.000000                                                   0
```

. . . .

```
F2        3  2     1.000000 0.237793 0.551952 0.331451      000 -1  0  2
 0.033107 0.002941 0.022301 0.004062 0.001804 0.001302      0111111111
-0.004765 0.000856 0.001537 0.000078-0.003154 0.001045      111111
 0.004140 0.000040-0.003533 0.000282-0.001855-0.000111      111111
 0.000417-0.000498-0.000275-0.000943 0.000839-0.000108      111111
 0.001132 0.000075 0.001248 0.001467 0.000710-0.000163      111111
 0.000000                                                   0
O3        4  2     1.000000 0.259432 0.704225 0.334598      000 -1  1  2
 0.029636 0.003572 0.024700-0.004798 0.011995-0.004461      0111111111
-0.001022 0.001363-0.004077-0.001334 0.000398-0.002854      111111
 0.007401-0.000459-0.002404-0.001127 0.002128 0.001734      111111
 0.003866 0.000433 0.002873-0.000778 0.001948-0.000513      111111
 0.001029-0.001433-0.003246 0.002369-0.001507 0.002928      111111
 0.004260 0.000676 0.007157-0.002878 0.008940-0.002886      111111
 0.000000                                                   0
pos#1     1        1.000000                                 000  0  2  0
    0.000    0.000    0.000 0.000000 0.000003 0.000000      0000010
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
-0.011043 0.000000-0.026361 0.000000 0.013892 0.000000      101010
-0.001271 0.000000 0.003932 0.000000 0.001400 0.000000      101010
-0.031089 0.000000 0.019840 0.000000-0.003164 0.000000      101010
-0.001732 0.000000 0.003092 0.000000 0.001535 0.000000      101010
 0.000000                                                  0
```

The individual position modulation parameters of O3

The molecular position modulation parameters.

. . . .

```
O3                 0.000000 0.000430 0.000131 0.000332
 0.000934 0.000096 0.000677 0.000245 0.000655 0.000210
 0.000683 0.000210 0.000534 0.000581 0.000200 0.000543
 0.001184 0.000115 0.000785 0.000296 0.000732 0.000233
 0.001348 0.000130 0.000991 0.000346 0.000918 0.000288
 0.001651 0.000157 0.001036 0.000405 0.001035 0.000333
 0.001732 0.000157 0.001098 0.000420 0.001093 0.000327
 0.000000
```

The e.s.d's of the individual position modulation parameters of O3.

3.2.9 Using local symmetry for molecules

In *JANA98* symmetry of the model molecule can be defined through a point group symbol (see page 141). Both Hermann-Mauguin and international symbols are allowed.

The local symmetry is defined with respect to the Cartesian axes related to the current crystal axis by matrix given in equation E 43, page 143. If the Cartesian system is redefined with help of the redefinition of the rotation z axis (see page 144) the local symmetry is based on the new coordinate system.

The examples that follow concern a structure containing three symmetry independent AsF_6 octahedrons. Changing from free atomic model to molecular models with various local symmetry enables to check rigidity and regularity of the octahedrons.

In the given example the symmetry considerations are simplified by the fact that molecular positions have general symmetry. If they would have its own local symmetry (for instance a mirror plane) the local symmetry of the model molecule would combine with a local symmetry of the molecular position.

Figure 118 *`M50` for examples concerning the local symmetry*

The keywords in `m50` are explained in page 77.

```
title JAJA in the supercell
cell 9.391 11.336 28.173 90 90 90
lambda 0.5609
spgroup Pnab  60
lattice P
centro
symmetry      x     y     z
symmetry  1/2-x 1/2+y 1/2+z
symmetry      x 1/2+y 1/2-z
symmetry  1/2-x     y    -z
unitsnumb 24
atom As
atweight 74.922 dmax 3 formula 1
f' 0.196 f" 1.332
formtab 32
   32.983   32.278   30.491   28.301   26.218   24.386   22.739   21.196
   19.722   18.313   16.976   15.717   14.540   13.451   12.454   11.552
   10.744   10.030    9.403    8.858    8.386    7.978    7.626    7.320
    7.053    6.817    6.606    0.000    0.000    0.000    0.000    0.000
atom K
atweight 39.098 dmax 0 formula 1
f' 0.118 f" 0.156
formtab 32
   18.999   18.206   16.732   15.244   13.726   12.269   10.980    9.909
    9.057    8.398    7.888    7.480    7.134    6.823    6.528    6.241
    5.956    5.674    5.395    5.120    4.851    4.589    4.336    4.093
    3.861    3.640    3.431    0.000    0.000    0.000    0.000    0.000
atom F
atweight 18.998 dmax 0 formula 5
f' 0.006 f" 0.006
formtab 32
    8.999    8.815    8.303    7.561    6.709    5.851    5.054    4.353
    3.760    3.270    2.873    2.558    2.309    2.112    1.957    1.834
    1.735    1.654    1.587    1.531    1.482    1.438    1.398    1.360
    1.324    1.289    1.255    0.000    0.000    0.000    0.000    0.000
atom O
atweight 15.999 dmax 0 formula 1
f' 0.003 f" 0.004
formtab 32
    7.999    7.798    7.246    6.472    5.623    4.808    4.089    3.489
    3.006    2.628    2.337    2.115    1.946    1.816    1.715    1.634
    1.568    1.512    1.463    1.419    1.377    1.337    1.298    1.260
    1.221    1.183    1.145    0.000    0.000    0.000    0.000    0.000
atom H
atweight 1.008 dmax 0 formula 1
f' 0 f" 0
formtab 32
    1.000    0.960    0.854    0.713    0.568    0.438    0.331    0.248
    0.184    0.138    0.103    0.078    0.060    0.046    0.036    0.028
    0.022    0.018    0.014    0.011    0.009    0.007    0.006    0.005
    0.005    0.004    0.004    0.000    0.000    0.000    0.000    0.000
noofref 5375
slimits 0.347273 0.442088 0.514002 0.572307 0.622045 0.667085 0.711857 0.753508
flimits 66.6 116.7 201.5 349 584.2 1141.4 2586.7 197781.8
end
```

Figure 119 *Setting a molecular model without local symmetry*

(a) *Input* `m40` *containing atomic model of the structure. The R value from the refinement of this structure was 4.91% for observed reflections.*

```
   25   0    0   0
 0.553924 0.000000 0.000000 0.000000 0.000000 0.000000     100000
 0.000000
 0.421884 0.000000 0.000000 0.000000 0.000000 0.000000     100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000     000000
Ka        2  2     0.500000 0.250000 0.341885 0.000000
 0.036739 0.039739 0.041075 0.000000 0.007541 0.000000     0010111010
Kb        2  2     1.000000 0.230410 0.338262 0.165883
 0.030894 0.036062 0.030158-0.003391-0.001207 0.001991     0111111111
Kc        2  2     1.000000 0.251236 0.322547 0.331665
 0.031621 0.030560 0.031993 0.002891 0.001232 0.001123     0111111111
Kd        2  2     0.500000 0.250000 0.318335 0.500000
 0.031541 0.028024 0.030752 0.000000 0.001138 0.000000     0010111010
Asa       1  2     1.000000 0.076569 0.104506 0.080828
 0.018308 0.017954 0.025836-0.000324-0.000520-0.000353     0111111111
F1a       3  2     1.000000-0.096404 0.049983 0.080027
 0.024560 0.040997 0.075323-0.012229-0.001312 0.002634     0111111111
F2a       3  2     1.000000 0.244947 0.168418 0.081473
 0.028301 0.041054 0.075391-0.004800 0.000596 0.000527     0111111111
F3a       3  2     1.000000 0.017405 0.203467 0.122918
 0.039527 0.043393 0.053351 0.003039 0.010724-0.022492     0111111111
F4a       3  2     1.000000 0.116884 0.003183 0.124304
 0.047890 0.046066 0.055105 0.002702-0.002904 0.029093     0111111111
F5a       3  2     1.000000 0.022328 0.203062 0.037616
 0.035212 0.044062 0.048320 0.001257-0.008604 0.017990     0111111111
F6a       3  2     1.000000 0.120183 0.003191 0.038150
 0.051513 0.045645 0.053383 0.007023 0.000484-0.027526     0111111111
Asb       1  2     1.000000 0.106961 0.093846 0.415836
 0.026233 0.019125 0.024413-0.001530 0.000201-0.001199     0111111111
F1b       3  2     1.000000 0.128467 0.245043 0.413639
 0.098775 0.021216 0.049971-0.010923 0.015824-0.001388     0111111111
F2b       3  2     1.000000 0.092824-0.059064 0.418986
 0.073228 0.029393 0.080465-0.001935 0.009063 0.002165     0111111111
F3b       3  2     1.000000 0.187436 0.085393 0.361604
 0.123808 0.045358 0.043183-0.005468 0.038849-0.002239     0111111111
F4b       3  2     1.000000-0.055691 0.110869 0.390266
 0.071723 0.070229 0.115648 0.015041-0.057738 0.000030     0111111111
F5b       3  2     1.000000 0.031585 0.115491 0.470551
 0.088945 0.047566 0.052497 0.003379 0.031311-0.004997     0111111111
F6b       3  2     1.000000 0.271597 0.086981 0.443235
 0.038769 0.082243 0.090329-0.000936-0.026437-0.014413     0111111111
Asc       1  2     1.000000 0.082771 0.107473 0.757335
 0.018084 0.021286 0.027039-0.001343 0.001200 0.000183     0111111111
F1c       3  2     1.000000-0.092688 0.057732 0.759366
 0.023728 0.050100 0.073426-0.013932 0.010129-0.011077     0111111111
F2c       3  2     1.000000 0.252783 0.166664 0.755343
 0.028795 0.043461 0.079426-0.002630 0.001123 0.001600     0111111111
F3c       3  2     1.000000 0.029290 0.214849 0.796523
 0.035401 0.056236 0.091378-0.003754 0.009211-0.041658     0111111111
F4c       3  2     1.000000 0.121184 0.015781 0.803403
 0.051938 0.095588 0.098611 0.017959 0.007636 0.071023     0111111111
F5c       3  2     1.000000 0.030651 0.196444 0.711747
 0.037782 0.129176 0.068125-0.000332-0.004932 0.059633     0111111111
F6c       3  2     1.000000 0.121874-0.005289 0.719188
 0.050470 0.087921 0.131062-0.017662 0.033382-0.080175     0111111111
```

The first AsF_6 octahedron

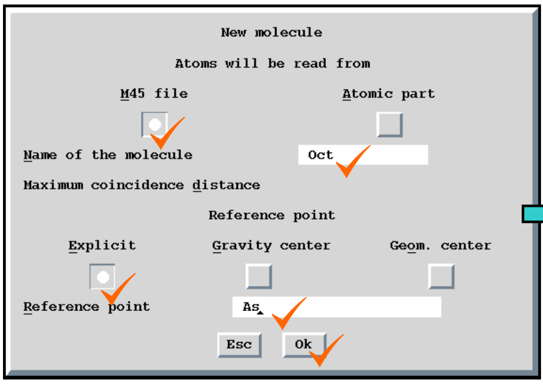
The second AsF_6 octahedron

The third AsF_6 octahedron

Figure 119(b) *Procedure to create a molecular model without specifying local symmetry. From two possible ways - creating from atoms already present in the atomic part (see Figure 98, page 173) and creating from m45 (see page 177)- the second one is used in order to have the model molecule in a coordinate system useful for later application of the local symmetry. Only important windows are shown - adding of a molecule from m45 is fully described in Figure 101, page 178.*

```
1 1 1 90 90 90
As         1  2    1.000000 0.000000 0.000000 0.000000
F1         3  2    1.000000 0.000000 0.000000 1.738000
F2         3  2    1.000000 0.000000 0.000000-1.738000
F3         3  2    1.000000 1.720000 0.000000 0.000000
F4         3  2    1.000000-1.720000 0.000000 0.000000
F5         3  2    1.000000 0.000000 1.720000 0.000000
F6         3  2    1.000000 0.000000-1.720000 0.000000
```

The m45 containing AsF_6 octahedron defined in Cartesian system. The non-zero coordinates are the As-F distances calculated from the atomic model.


New molecule

Atoms will be read from

M45 file ● Atomic part ☐

Name of the molecule: Oct

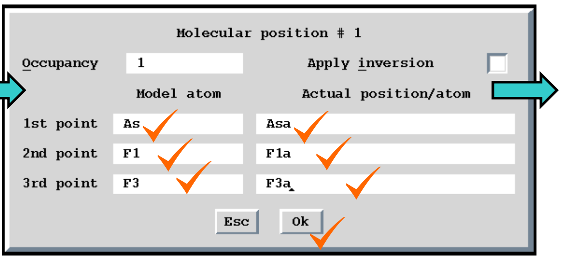
Maximum coincidence distance

Reference point

Explicit ● Gravity center ☐ Geom. center ☐

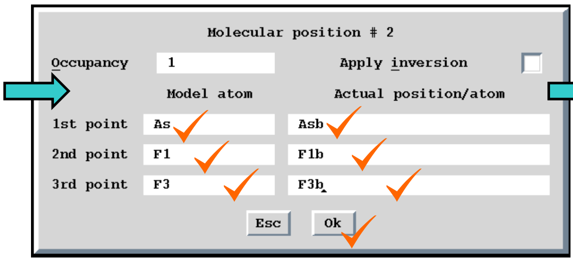
Reference point: As

The positions of the model octahedron are defined as coincident with the existing octahedrons in the atomic model.


Molecular position # 1

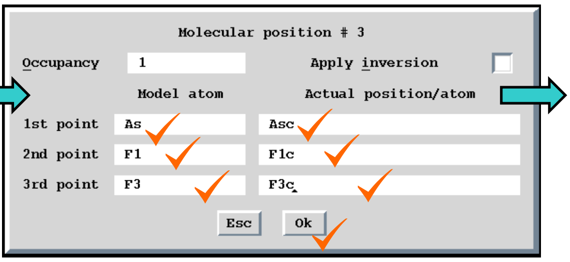
Occupancy: 1 Apply inversion ☐

	Model atom	Actual position/atom
1st point	As	Asa
2nd point	F1	F1a
3rd point	F3	F3a


Molecular position # 2

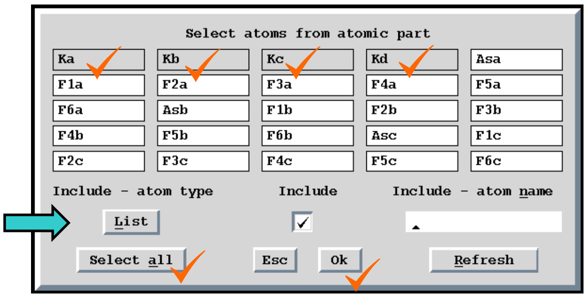
Occupancy: 1 Apply inversion ☐

	Model atom	Actual position/atom
1st point	As	Asb
2nd point	F1	F1b
3rd point	F3	F3b


Molecular position # 3

Occupancy: 1 Apply inversion ☐

	Model atom	Actual position/atom
1st point	As	Asc
2nd point	F1	F1c
3rd point	F3	F3c


Select atoms from atomic part

Ka	Kb	Kc	Kd	Asa
F1a	F2a	F3a	F4a	F5a
F6a	Asb	F1b	F2b	F3b
F4b	F5b	F6b	Asc	F1c
F2c	F3c	F4c	F5c	F6c

Include - atom type: List Include ☑ Include - atom name

The atoms to be deleted in the atomic part.

Unlike creating of the molecule from existing atoms in the atomic part the coincidence of new molecular position with atoms already existing in the atomic part is not checked. The atoms have to be deleted through the tool Deleting of atoms.

Figure 119(c) *The resulting* `m40` *with a molecule "Oct" in three positions. The model octahedron is oriented along the* ***c*** *axis.*

```
    4   1    0    1
    7   3
 0.553924 0.000000 0.000000 0.000000 0.000000 0.000000     100000
 0.000000
 0.421884 0.000000 0.000000 0.000000 0.000000 0.000000     100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000     000000
Ka       2  2    0.500000 0.250000 0.341885 0.000000
 0.036739 0.039739 0.041075 0.000000 0.007541 0.000000     0010111010
Kb       2  2    1.000000 0.230410 0.338262 0.165883
 0.030894 0.036062 0.030158-0.003391-0.001207 0.001991     0111111111
Kc       2  2    1.000000 0.251236 0.322547 0.331665
 0.031621 0.030560 0.031993 0.002891 0.001232 0.001123     0111111111
Kd       2  2    0.500000 0.250000 0.318335 0.500000
 0.031541 0.028024 0.030752 0.000000 0.001138 0.000000     0010111010
Oct      0                0As
As       1  1    1.000000 0.000000 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
F1       3  1    1.000000 0.000000 0.000000 0.061690
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
F2       3  1    1.000000 0.000000 0.000000-0.061690
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
F3       3  1    1.000000 0.183154 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
F4       3  1    1.000000-0.183154 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
F5       3  1    1.000000 0.000000 0.151729 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
F6       3  1    1.000000 0.000000-0.151729 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000     0000000000
pos#1    1       1.000000                                  000  0  0  0
  111.540  -43.611  -91.027 0.076569 0.104506 0.080828     0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#2    1       1.000000                                  000  0  0  0
  -10.830   63.416  -94.594 0.106961 0.093846 0.415836     0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#3    1       1.000000                                  000  0  0  0
  107.316  -39.923  -87.546 0.082771 0.107473 0.757335     0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
```

The model molecule

Three positions of the model molecule correspond to positions of octahedrons in the free atomic model.

Figure 119(d) *The structure from Figure 119(c)refined with TLS tensors*[1].

```
    4    1    0    1
    7    3
 0.548310 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.247648 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
Ka        2  2     0.500000 0.250000 0.341796 0.000000
 0.035935 0.040149 0.041116 0.000000 0.007367 0.000000      0010111010
Kb        2  2     1.000000 0.230401 0.338221 0.165874
 0.030349 0.035855 0.029715-0.003354-0.001479 0.001819      0111111111
Kc        2  2     1.000000 0.251256 0.322471 0.331663
 0.031699 0.029773 0.031780 0.002807 0.001166 0.001108      0111111111
Kd        2  2     0.500000 0.250000 0.318375 0.500000
 0.031445 0.027776 0.030263 0.000000 0.001246 0.000000      0010111010
Oct       0                 As
As        1  0     1.000000 0.000000 0.000000 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1        3  0     1.000000-0.001816-0.001824 0.061663
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F2        3  0     1.000000 0.007185 0.004706-0.061636
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F3        3  0     1.000000 0.182359-0.001421 0.003652
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F4        3  0     1.000000-0.182270 0.001312 0.001710
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F5        3  0     1.000000 0.003928 0.150906 0.003380
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
F6        3  0     1.000000-0.003286-0.151227 0.001236
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0111000000
pos#1     1        1.000000                                 000  0  0  0
  111.540  -43.611  -91.027 0.076523 0.104605 0.080819      0000111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.004777 0.003445 0.000440-0.000714 0.000044-0.000002      111111
 0.002448 0.001925 0.000401 0.000650 0.000145 0.000113      111111
 0.000089-0.000022-0.000110 0.000105-0.000094 0.000103      111111
 0.000058-0.000004 0.000005                                 110
pos#2     1        1.000000                                 000  0  0  0
  -14.739   64.584 -100.240 0.107172 0.093833 0.415891      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.005414 0.004000 0.000472-0.000137 0.000095 0.000090      111111
 0.005132 0.001751 0.000819-0.000091-0.000350 0.000045      111111
 0.000330-0.000706-0.000075-0.000393-0.000386 0.000023      111111
-0.000436 0.000279 0.000056                                 110
pos#3     1        1.000000                                 000  0  0  0
  107.712  -39.297  -87.760 0.082771 0.107513 0.757349      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.005251 0.003844 0.000432-0.000467-0.000044 0.000043      111111
 0.003392 0.001512 0.001107 0.000575 0.000889 0.000110      111111
-0.000178-0.000384-0.000193-0.000285 0.000233 0.000049      111111
-0.000168-0.000599-0.000055                                 110
```

The model molecule. Note that the refined coordinates are not far from the input coordinated from Figure 119(c).

Molecular positions with TLS parameters.

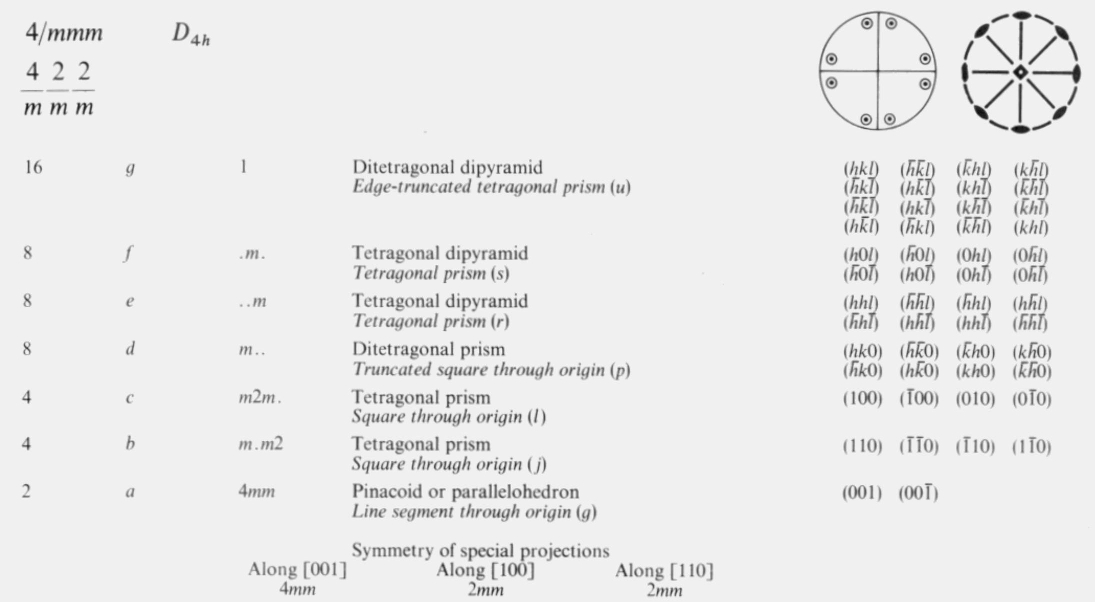
---

[1] The R value was 5.59 % (compare with 4.91% of the free atomic model). The refinement with anisotropic temperature parameters (i.e. without TLS tensors) converged with R=7.45%.

Figure 120 *Setting the local symmetry D_{4h}*

***(a)** Input `m40` is in Figure 119(c). The refined `m40` from Figure 119(d) cannot be used because the model coordinates already do not have the D_{4h} symmetry.*

***(b)** Procedure to define the local symmetry D_{4h}. According to the International tables, Vol A, atoms F1 and F2 are in the symmetry position a, F3, F4, F5 and F6 are in the symmetry position c and As is not expanded.*



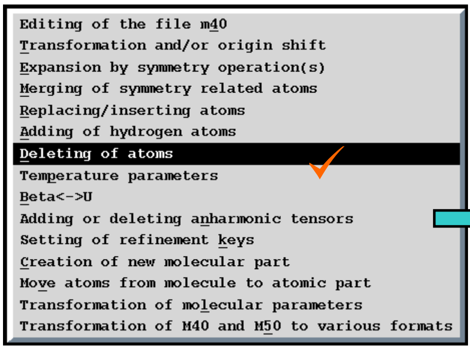
$4/mmm$ D_{4h}

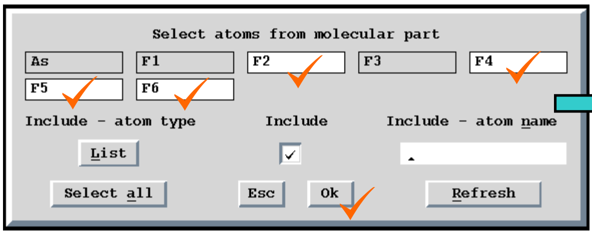
$4\ 2\ 2$ / $m\ m\ m$

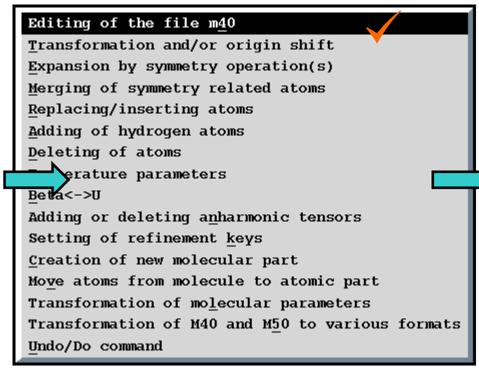
16	*g*	1	Ditetragonal dipyramid *Edge-truncated tetragonal prism (u)*	(hkl) $(\bar{h}k\bar{l})$ $(\bar{h}\bar{k}\bar{l})$ $(h\bar{k}l)$	$(\bar{h}\bar{k}l)$ $(h\bar{k}\bar{l})$ $(hk\bar{l})$ $(\bar{h}kl)$	$(\bar{k}hl)$ $(kh\bar{l})$ $(k\bar{h}\bar{l})$ $(\bar{k}\bar{h}l)$	$(k\bar{h}l)$ $(\bar{k}\bar{h}\bar{l})$ $(\bar{k}h\bar{l})$ (khl)
8	*f*	$.m.$	Tetragonal dipyramid *Tetragonal prism (s)*	$(h0l)$ $(\bar{h}0\bar{l})$	$(\bar{h}0l)$ $(h0\bar{l})$	$(0hl)$ $(0h\bar{l})$	$(0\bar{h}l)$ $(0\bar{h}\bar{l})$
8	*e*	$..m$	Tetragonal dipyramid *Tetragonal prism (r)*	(hhl) $(\bar{h}h\bar{l})$	$(\bar{h}\bar{h}l)$ $(h\bar{h}\bar{l})$	$(\bar{h}hl)$ $(hh\bar{l})$	$(h\bar{h}l)$ $(\bar{h}\bar{h}\bar{l})$
8	*d*	$m..$	Ditetragonal prism *Truncated square through origin (p)*	$(hk0)$ $(\bar{h}k0)$	$(\bar{h}\bar{k}0)$ $(h\bar{k}0)$	$(\bar{k}h0)$ $(kh0)$	$(k\bar{h}0)$ $(\bar{k}\bar{h}0)$
4	*c*	$m2m.$	Tetragonal prism *Square through origin (l)*	(100)	$(\bar{1}00)$	(010)	$(0\bar{1}0)$
4	*b*	$m.m2$	Tetragonal prism *Square through origin (j)*	(110)	$(\bar{1}\bar{1}0)$	$(\bar{1}10)$	$(1\bar{1}0)$
2	*a*	$4mm$	Pinacoid or parallelohedron *Line segment through origin (g)*	(001)	$(00\bar{1})$		

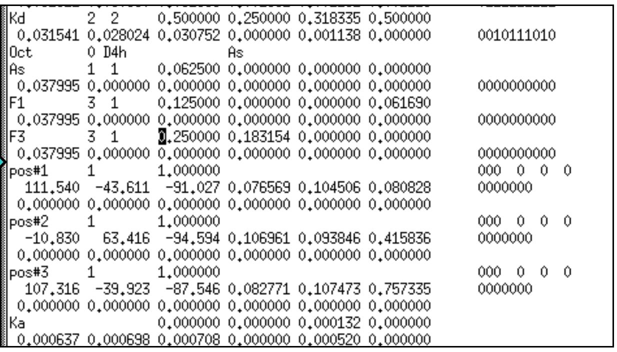
Symmetry of special projections

Along [001]	Along [100]	Along [110]
$4mm$	$2mm$	$2mm$









```
Kd          2  2      0.500000 0.250000 0.318335 0.500000
 0.031541 0.028024 0.030752 0.000000 0.001138 0.000000      0010111010
Oct         0  D4h             As
As          1  1      0.062500 0.000000 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1          3  1      0.125000 0.000000 0.000000 0.061690
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F3          3  1      0.250000 0.183154 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
pos#1       1         1.000000                              000  0  0  0
  111.540  -43.611   -91.027 0.076569 0.104506 0.080828     0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#2       1         1.000000                              000  0  0  0
  -10.830   63.416   -94.594 0.106961 0.093846 0.415836     0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#3       1         1.000000                              000  0  0  0
  107.316  -39.923   -87.546 0.082771 0.107473 0.757335     0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
Ka                    0.000000 0.000000 0.000132 0.000000
 0.000637 0.000698 0.000708 0.000000 0.000520 0.000000
```

Here the user should enter the point group symbol and the site occupation factors for the model atoms. *JANA98* does not offer a user interface for these operations.

Figure 120(c) *Resulting* `m40` *after refinement of TLS parameters*[1].

```
    4    1    0    1
    3    3
 0.547455 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.220582 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
Ka        2  2     0.500000 0.250000 0.341929 0.000000
 0.036498 0.040330 0.040841 0.000000 0.007473 0.000000      0010111010
Kb        2  2     1.000000 0.230231 0.338285 0.165838
 0.030771 0.036116 0.029813-0.003109-0.001382 0.001712      0111111111
Kc        2  2     1.000000 0.251189 0.322495 0.331615
 0.032286 0.029363 0.032024 0.002423 0.001334 0.000639      0111111111
Kd        2  2     0.500000 0.250000 0.318449 0.500000
 0.032787 0.028146 0.029984 0.000000 0.001005 0.000000      0010111010
Oct       0 D4h             As
As        1  0     0.062500 0.000000 0.000000 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1        3  0     0.125000 0.000000 0.000000 0.061602
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0001000000
F3        3  0     0.250000 0.182469 0.000000 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0100000000
pos#1     1        1.000000                                 000  0  0  0
  113.607  -44.370  -91.078 0.076281 0.104629 0.080825      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.004824 0.003379 0.000439-0.000724 0.000044-0.000003      111111
 0.002619 0.002190 0.000410 0.000559 0.000088 0.000127      111111
 0.000079 0.000001-0.000154 0.000242-0.000101 0.000140      111111
 0.000051 0.000001 0.000022                                 110
pos#2     1        1.000000                                 000  0  0  0
  -11.950   63.764  -96.713 0.107186 0.094014 0.415867      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.005402 0.004019 0.000469-0.000143 0.000095 0.000071      111111
 0.005245 0.001936 0.000831 0.000114-0.000234 0.000110      111111
 0.000167-0.000640 0.000000-0.000368-0.000238 0.000037      111111
-0.000389 0.000175 0.000070                                 110
pos#3     1        1.000000                                 000  0  0  0
  109.615  -40.498  -87.568 0.082541 0.107527 0.757354      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.005220 0.003850 0.000433-0.000441-0.000051 0.000041      111111
 0.003563 0.001680 0.001071 0.000618 0.000774 0.000213      111111
-0.000230-0.000450-0.000179-0.000277 0.000215 0.000071      111111
-0.000156-0.000623 0.000016                                 110
```

Refinement keys of the first molecular position - see footnote.

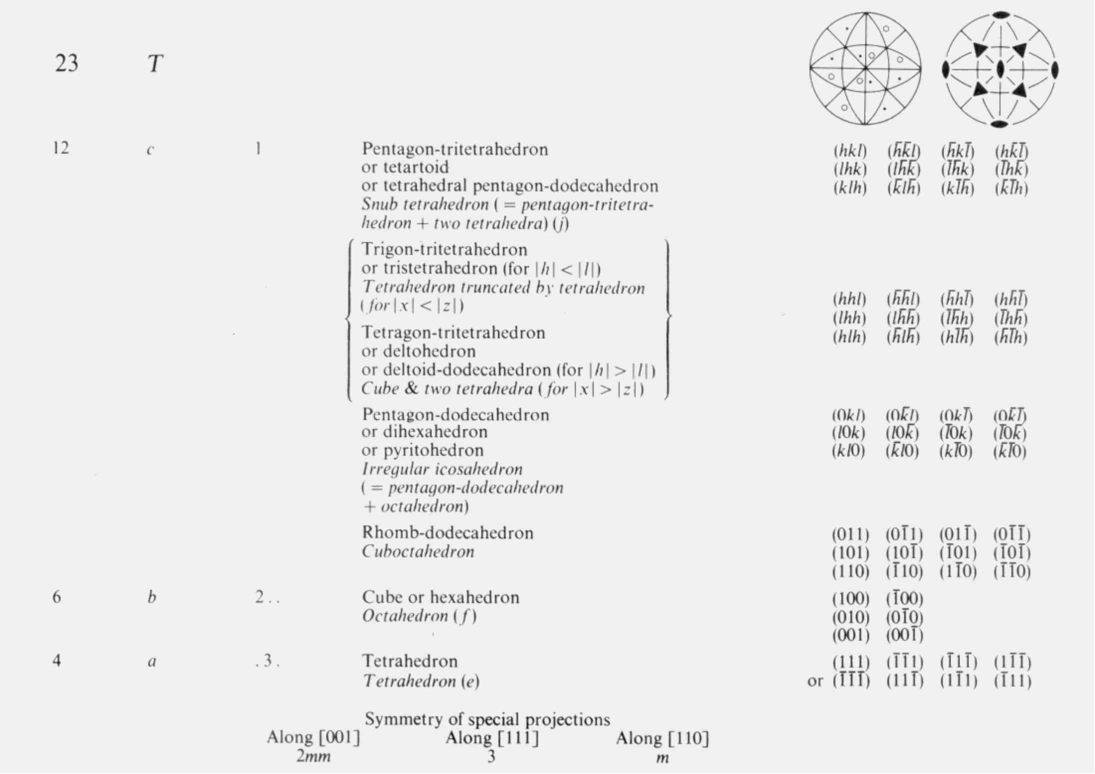
---

[1] The R value was 6.84 % (compare with 5.59% of the molecular model without applying of the local symmetry). The symmetry restrictions of the model molecule coordinates are calculated from the point group D_{4h}. Refinement of restricted coordinates can influence the size of the molecule but not rotation. Therefore the rotation angles of the first molecular position cannot be fixed in order to allow proper orienting of the first molecular position. For this the automatic refinement mode has to be disabled because in the automatic mode the rotation parameters of the first molecular position are always fixed for the reasons explained in page 189.

Figure 121 *Setting the local symmetry T*

(a) *Input* `m40` *is in Figure 119(c). The refined* `m40` *from Figure 119(d) cannot be used because the model coordinates already do not have the* D_{4h} *symmetry.*

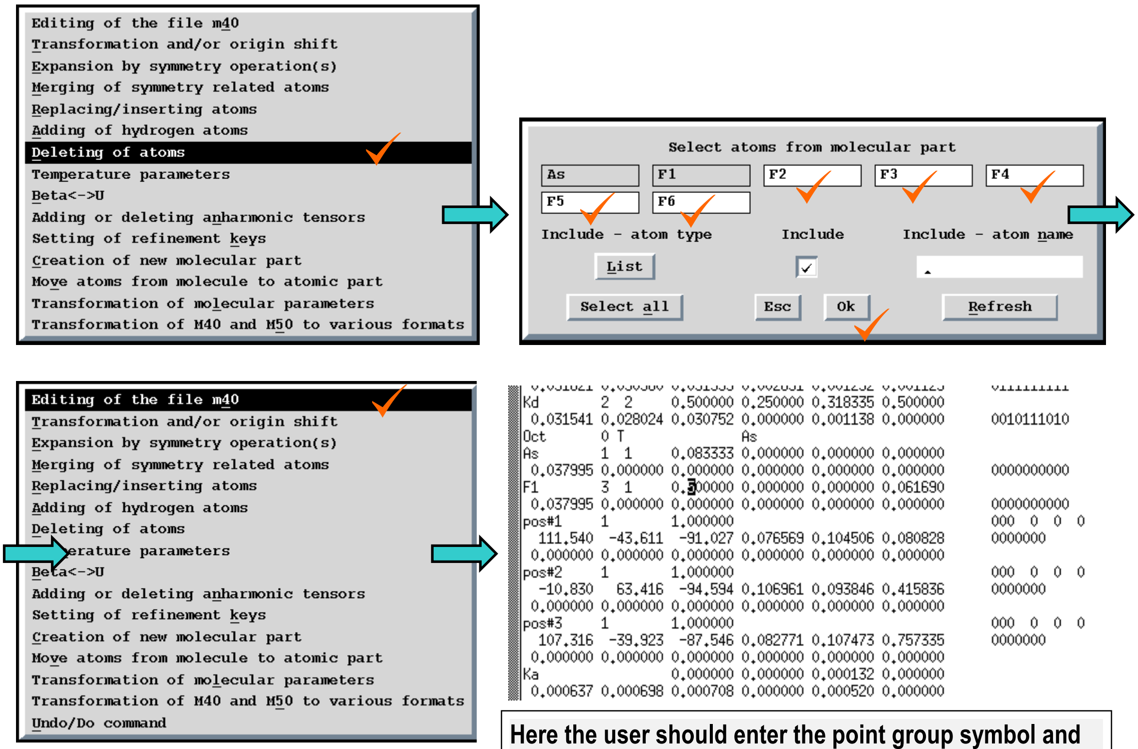
(b) *Procedure to define the local symmetry* D_{4h}*. In the point group T all atoms F are equivalent.*



23	*T*			
12	*c*	1	Pentagon-tritetrahedron or tetartoid or tetrahedral pentagon-dodecahedron *Snub tetrahedron (= pentagon-tritetrahedron + two tetrahedra) (j)*	(hkl) $(\bar{h}\bar{k}l)$ $(\bar{h}k\bar{l})$ $(h\bar{k}\bar{l})$ (lhk) $(l\bar{h}\bar{k})$ $(\bar{l}\bar{h}k)$ $(\bar{l}h\bar{k})$ (klh) $(\bar{k}l\bar{h})$ $(k\bar{l}\bar{h})$ $(\bar{k}\bar{l}h)$
			Trigon-tritetrahedron or tristetrahedron (for $\lvert h\rvert < \lvert l\rvert$) *Tetrahedron truncated by tetrahedron (for* $\lvert x\rvert < \lvert z\rvert$*)* Tetragon-tritetrahedron or deltohedron or deltoid-dodecahedron (for $\lvert h\rvert > \lvert l\rvert$) *Cube & two tetrahedra (for* $\lvert x\rvert > \lvert z\rvert$*)*	(hhl) $(\bar{h}\bar{h}l)$ $(\bar{h}h\bar{l})$ $(h\bar{h}\bar{l})$ (lhh) $(l\bar{h}\bar{h})$ $(\bar{l}\bar{h}h)$ $(\bar{l}h\bar{h})$ (hlh) $(\bar{h}l\bar{h})$ $(h\bar{l}\bar{h})$ $(\bar{h}\bar{l}h)$
			Pentagon-dodecahedron or dihexahedron or pyritohedron *Irregular icosahedron (= pentagon-dodecahedron + octahedron)*	$(0kl)$ $(0\bar{k}l)$ $(0k\bar{l})$ $(0\bar{k}\bar{l})$ $(l0k)$ $(l0\bar{k})$ $(\bar{l}0k)$ $(\bar{l}0\bar{k})$ $(kl0)$ $(\bar{k}l0)$ $(k\bar{l}0)$ $(\bar{k}\bar{l}0)$
			Rhomb-dodecahedron *Cuboctahedron*	(011) $(0\bar{1}1)$ $(01\bar{1})$ $(0\bar{1}\bar{1})$ (101) $(10\bar{1})$ $(\bar{1}01)$ $(\bar{1}0\bar{1})$ (110) $(\bar{1}10)$ $(1\bar{1}0)$ $(\bar{1}\bar{1}0)$
6	*b*	2 . .	Cube or hexahedron *Octahedron (f)*	(100) $(\bar{1}00)$ (010) $(0\bar{1}0)$ (001) $(00\bar{1})$
4	*a*	. 3 .	Tetrahedron *Tetrahedron (e)*	(111) $(\bar{1}\bar{1}1)$ $(\bar{1}1\bar{1})$ $(1\bar{1}\bar{1})$ or $(\bar{1}\bar{1}\bar{1})$ $(11\bar{1})$ $(1\bar{1}1)$ $(\bar{1}11)$

Symmetry of special projections

Along [001]	Along [111]	Along [110]
2mm	3	*m*



```
Kd          2  2     0.500000 0.250000 0.318335 0.500000
 0.031541 0.028024 0.030752 0.000000 0.001138 0.000000      0010111010
Oct         0  T              As
As          1  1     0.083333 0.000000 0.000000 0.000000
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1          3  1     0.500000 0.000000 0.000000 0.061690
 0.037995 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
pos#1       1        1.000000                               000  0  0  0
  111.540  -43.611  -91.027 0.076569 0.104506 0.080828      0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#2       1        1.000000                               000  0  0  0
  -10.830   63.416  -94.594 0.106961 0.093846 0.415836      0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
pos#3       1        1.000000                               000  0  0  0
  107.316  -39.923  -87.546 0.082771 0.107473 0.757335      0000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
Ka                   0.000000 0.000000 0.000132 0.000000
 0.000637 0.000698 0.000708 0.000000 0.000520 0.000000
```

Here the user should enter the point group symbol and the site occupation factors for the model atoms

Figure 121(c) *The resulting* `m40` *after refinement of TLS parameters*[1]

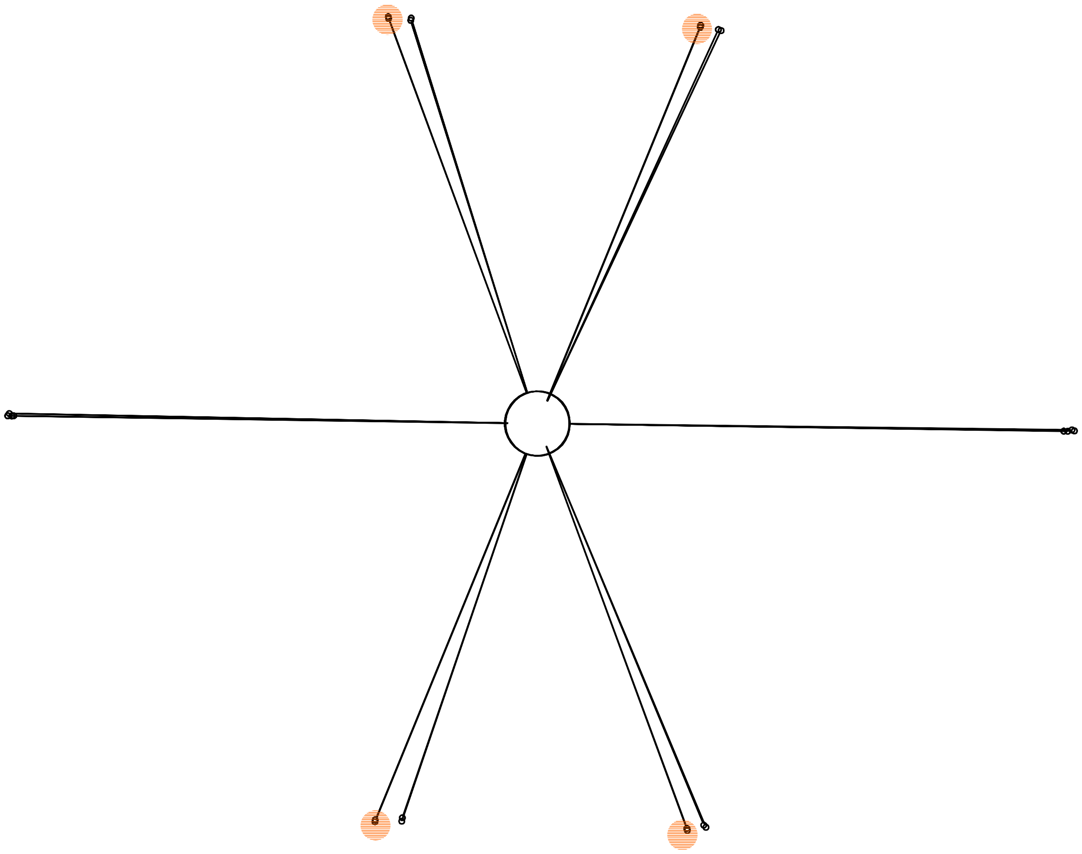
```
    4    1    0    1
    2    3
 0.546634 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000
 0.197213 0.000000 0.000000 0.000000 0.000000 0.000000      100000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      000000
Ka        2  2     0.500000 0.250000 0.341944 0.000000
 0.036588 0.040202 0.040786 0.000000 0.007372 0.000000      0010111010
Kb        2  2     1.000000 0.230215 0.338273 0.165834
 0.030899 0.035915 0.029662-0.003056-0.001376 0.001772      0111111111
Kc        2  2     1.000000 0.251195 0.322492 0.331625
 0.032421 0.029257 0.031823 0.002401 0.001330 0.000610      0111111111
Kd        2  2     0.500000 0.250000 0.318460 0.500000
 0.032805 0.028173 0.029868 0.000000 0.000881 0.000000      0010111010
Oct       0 T               As
As        1  0     0.083333 0.000000 0.000000 0.000000
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0000000000
F1        3  0     0.500000 0.000000 0.000000 0.061109
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000      0001000000
pos#1     1        1.000000                                 000  0  0  0
  113.539  -44.382  -91.068 0.076274 0.104628 0.080821      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.004813 0.003370 0.000440-0.000716 0.000044-0.000003      111111
 0.002620 0.002209 0.000405 0.000566 0.000089 0.000128      111111
 0.000087 0.000007-0.000158 0.000240-0.000108 0.000140      111111
 0.000035 0.000005 0.000022                                 110
pos#2     1        1.000000                                 000  0  0  0
  -11.863   63.719  -96.690 0.107210 0.093985 0.415869      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.005388 0.004010 0.000470-0.000145 0.000094 0.000071      111111
 0.005246 0.001930 0.000816 0.000112-0.000200 0.000105      111111
 0.000214-0.000672 0.000006-0.000336-0.000271 0.000035      111111
-0.000387 0.000176 0.000056                                 110
pos#3     1        1.000000                                 000  0  0  0
  109.554  -40.526  -87.535 0.082537 0.107516 0.757358      0111111
 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
 0.005202 0.003839 0.000433-0.000437-0.000048 0.000043      111111
 0.003613 0.001674 0.001056 0.000629 0.000762 0.000198      111111
-0.000230-0.000440-0.000180-0.000263 0.000209 0.000077      111111
-0.000160-0.000596 0.000021                                 110
```

---

[1] The R value was 6.92% (compare with 6.84% for the local symmetry D_{4h}). The rotation angles of the first molecular position were not be fixed - see footnote on page 214 for explanation.

Figure 122 *The shape of AsF_6 octahedron.*

This figure contains AsF_6 octahedrons refined in a free atomic model, in a molecular model without applying a local symmetry (see Figure 119, page 209), in a molecular model with local symmetry D4h (see Figure 120, page 213)and in a molecular model with local symmetry T (see Figure 121, page 215). The octahedrons are overlayed and viewed along ***b*** *axis. The red circles denote superimposed positions with the local symmetry T and D_{4h}.*

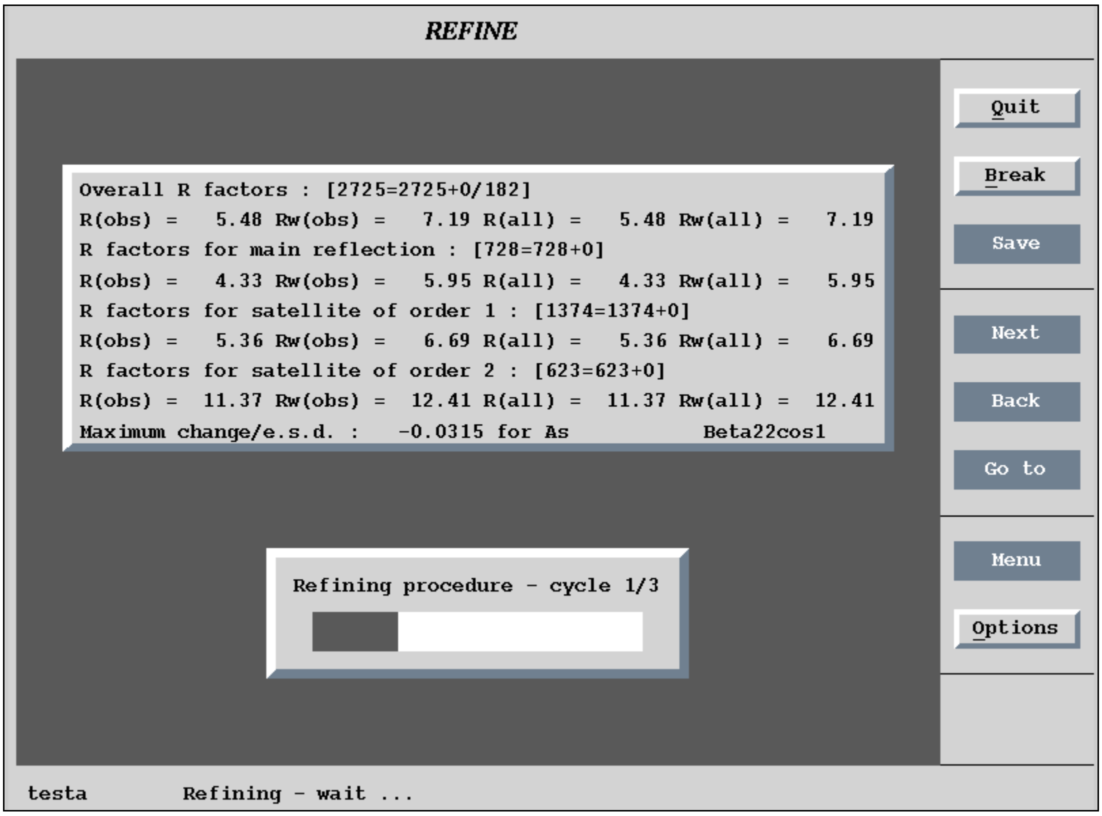


3.3 Program *REFINE*

Input: `m50,m91,[m40]`
Output: `m40,m80,ref`
M50 control keywords: the section between the keywords `refine` and `end`

3.3.1 Description of *REFINE*

Figure 123 *The Basic Window of REFINE*



```
Overall R factors : [2725=2725+0/182]
R(obs) =    5.48 Rw(obs) =    7.19 R(all) =    5.48 Rw(all) =    7.19
R factors for main reflection : [728=728+0]
R(obs) =    4.33 Rw(obs) =    5.95 R(all) =    4.33 Rw(all) =    5.95
R factors for satellite of order 1 : [1374=1374+0]
R(obs) =    5.36 Rw(obs) =    6.69 R(all) =    5.36 Rw(all) =    6.69
R factors for satellite of order 2 : [623=623+0]
R(obs) =   11.37 Rw(obs) =   12.41 R(all) =   11.37 Rw(all) =   12.41
Maximum change/e.s.d. :   -0.0315 for As           Beta22cos1
```

```
Refining procedure - cycle 1/3
```

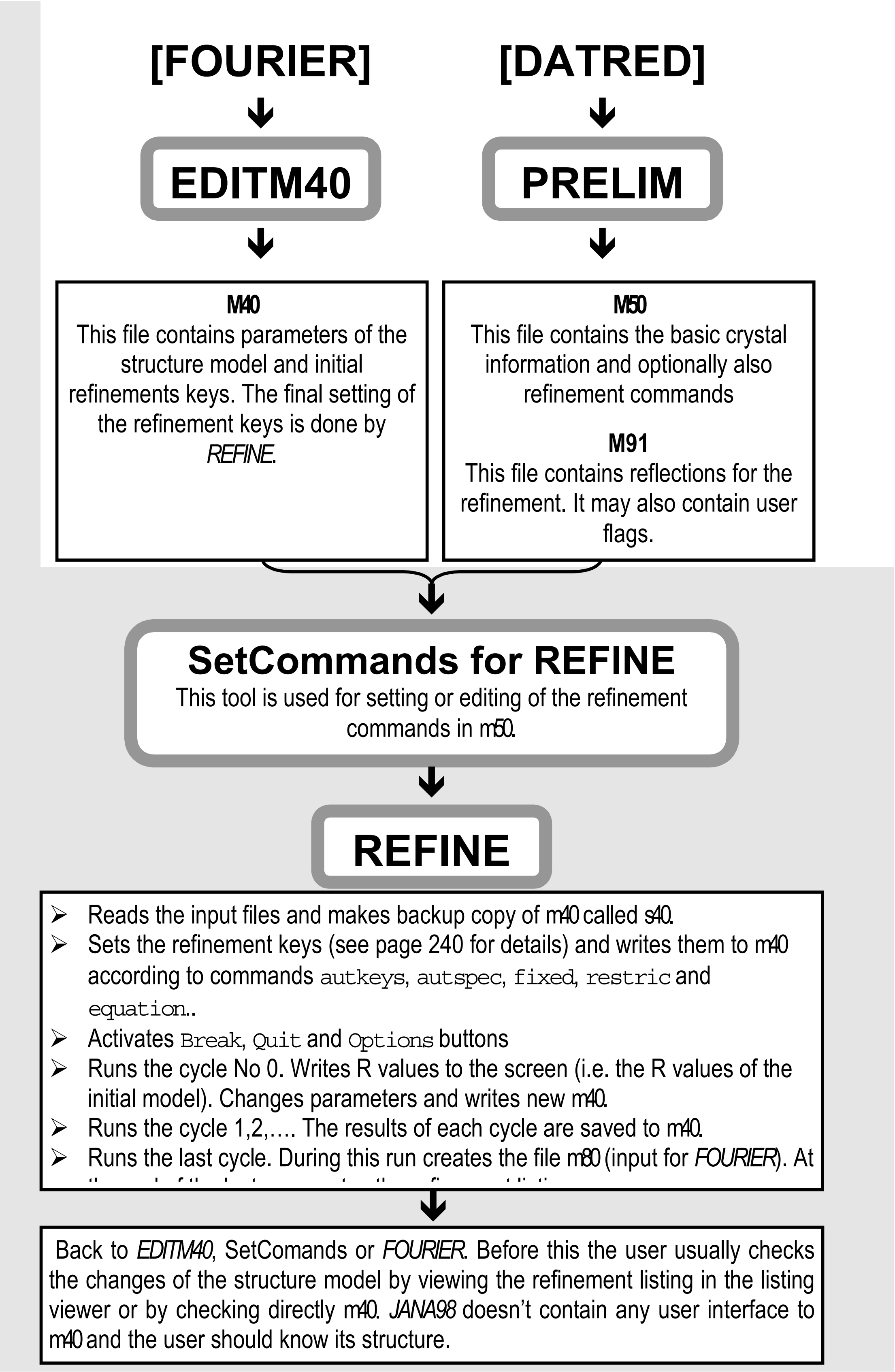
```
testa         Refining - wait ...
```

REFINE is a program for refinement of structure models. It needs as an input three files:

- m40 with parameters of the structure model and refinement keys, indicating parameters to be refined
- m50 with basic crystal information and with refinement commands (number of refinement cycles etc)
- m91 with reflections for the refinement

The scheme in the next page shows *REFINE* in the framework of *JANA98.*

Figure 124 *The Scheme of the Refinement Run*



M40

This file contains parameters of the structure model and initial refinements keys. The final setting of the refinement keys is done by *REFINE*.

M50

This file contains the basic crystal information and optionally also refinement commands

M91

This file contains reflections for the refinement. It may also contain user flags.

SetCommands for REFINE

This tool is used for setting or editing of the refinement commands in m50.

REFINE

- Reads the input files and makes backup copy of m40 called s40.
- Sets the refinement keys (see page 240 for details) and writes them to m40 according to commands `autkeys`, `autspec`, `fixed`, `restric` and `equation`..
- Activates `Break`, `Quit` and `Options` buttons
- Runs the cycle No 0. Writes R values to the screen (i.e. the R values of the initial model). Changes parameters and writes new m40.
- Runs the cycle 1,2,…. The results of each cycle are saved to m40.
- Runs the last cycle. During this run creates the file m80 (input for *FOURIER*). At

Back to *EDITM40*, SetComands or *FOURIER*. Before this the user usually checks the changes of the structure model by viewing the refinement listing in the listing viewer or by checking directly m40. *JANA98* doesn't contain any user interface to m40 and the user should know its structure.

The weighting scheme, minimised function and statistical quantities

The reflection file contains for each independent reflection the intensity I and its estimated standard deviation σ(I). The sigma's are strictly taken as a measure of accuracy of the corresponding intensity. The weight of a reflection is based by default on this value and the program doesn't make any optimisation of the weighting scheme.

For the refinement based of F and for the default weighting scheme (see §3.3.7, page 241 for details) the weight is defined by expression

$$w = \frac{1}{\sigma^2(|F_o|) + (uF_o)^2} \qquad E57$$

and the minimised function is

$$P = \sum w(|F_c| - |F_o|)^2 . \qquad E58$$

The coefficient *u* in the equation *E57* is the instability factor, which can be changed by the user but it is not further changed by the program. The proper value of the instability factor should be adjusted for a given diffractometer and should not be changed for different structures measured under the same conditions. This approach make easier a comparison of different structures - for instance comparison of their goodness of fit, see later in this paragraph. The typical value of *u* is about 0.01.

For the refinement based on F^2 the weight is defined by expression

$$w' = \frac{w}{4F_o^{\,2}} , \qquad E59$$

where *w* is the weight from equation *E57*. The minimised function is

$$P = w'(F_o^2 - F_c^2)^2 . \qquad E60$$

Goodness of fit is defined as

$$S = \sqrt{\frac{\sum w(F_o - F_c)^2}{m-n}}, \qquad E61$$

where m is number of reflections and n is number of parameters refined. If the refinement is based on F^2 the goodness of fit is defined by expression

$$S = \sqrt{\frac{\sum w(F_o^2 - F_c^2)^2}{m-n}}\ . \qquad E62$$

Goodness of fit can be understood as a measure of the fit between the distribution expected from the weights used in the refinement and the distribution of $|F_c|$- $|F_o|$. If the weights are correctly estimated, errors in the data are random and the structure model is statistically correct, i.e. the value of S is 1.

JANA98 does not make any adjustement of the weighting scheme to force S to 1. S between 2-4 does not necessarily mean that the structure model is wrong or that there are systematic errors in the data set. For the data measured with a high accuracy and to a high θ angle large S can mean that the model does not describe all details of the structure like anharmonic displacements, lone-pair and bonding electrons etc. On the other hand S less than 1 indicates that the accuracy of data is not high enough to prove the model.

The estimated standard deviations of refined parameters are calculated from diagonal elements of the normal matrix and normalised to ideal goodness of fit S=1. The differences about 5 e.s.d. are already visible in the difference Fourier.

Related chapters

This chapter focuses only on the topics related immediately to the refinement program. Here are another sources of information:

- § 3.1, page 93 describes structure parameters and the structure of the refinement parameter file `m40`.
- § 3.2, page 153 describes *EDITM40*, the program which is used for building and modifications of the structure model.

3.3.2 Structure parameters

The structure parameters are listed in Table 10, page 98. Their names can be used in the refinement control commands (see later).

3.3.3 Refinement keys

Each refinable parameter of a structure model has the corresponding refinement key. It key indicates whether the parameter will be refined. Only the parameters with positive refinement keys are refined.

The automatic and special refinement keys.

The structure parameters can be divided to two groups with respect to the refinement keys:

- ***Parameters with automatic refinement keys***. The refinement keys of these parameters can be automatically set up by *REFINE*. Optionally *REFINE* can also derive the symmetry restrictions of these parameters and make corresponding changes of their initial values.
- ***Parameters with special refinement keys***. The setting of their refinement keys is not fully automatic and need assistance of the user. Only several parameters have special refinement keys. They are listed in Table 13, page 226.

The Manual and Automatic mode

The user can run the refinement program in manual or automatic mode for setting of refinement keys.

- In the ***manual mode*** the setting of refinement keys is done by the user. *REFINE* does not check the correctness of these keys. The keys read from the input `m40` are not changed by *REFINE* except there are user constraints defined by refinement commands.
- In the ***automatic mode*** *REFINE* resets part of or all automatic refinement keys read from the input `m40`. The level of automatization depends on `autkeys` and `autspec` commands. It should be noted that the special refinement keys (see Table 13) are not set by *REFINE* even in the automatic mode or they are changed only under special conditions.

Autkeys and Autspec commands

These two commands are part of the Basic commands (page 231). They are applicable to parameters with automatic refinement keys. The combination of both commands influences which keys are set automatically and which are left under the user control. ***The authors recommend to use the highest (default) automatic mode whenever it is possible.***

- ***Autkeys command*** turns on/off the automatic initialization of the refinement keys. If the `autkeys` mode is activated *REFINE* sets at the beginning of the run all refinable parameters to 1. All refinement keys in input `m40` except the special ones are ignored. With autkeys *REFINE* also sets refinement keys in response to settings in the Modulation commands (see page 253). For instance if we select only main reflection for the refinement the refinement keys of modulation parameters are automatically set to "0". It also works for refinement of composites: if we select only reflections of one subsystem the parameters pertaining to atoms of other subsystems are fixed. In Autkeys mode *REFINE* also make fixation of the origin where necessary[1].
- ***Autspec command*** turns on/off automatic symmetry restrictions following from the site symmetry. In case when the restriction means that a certain parameters has to have a fixed value the program makes an initialization and fixes its values by setting of the relevant refinement key to 0. In case when the restriction means some relationship between more than one parameter the program generates an equation.

User constraints

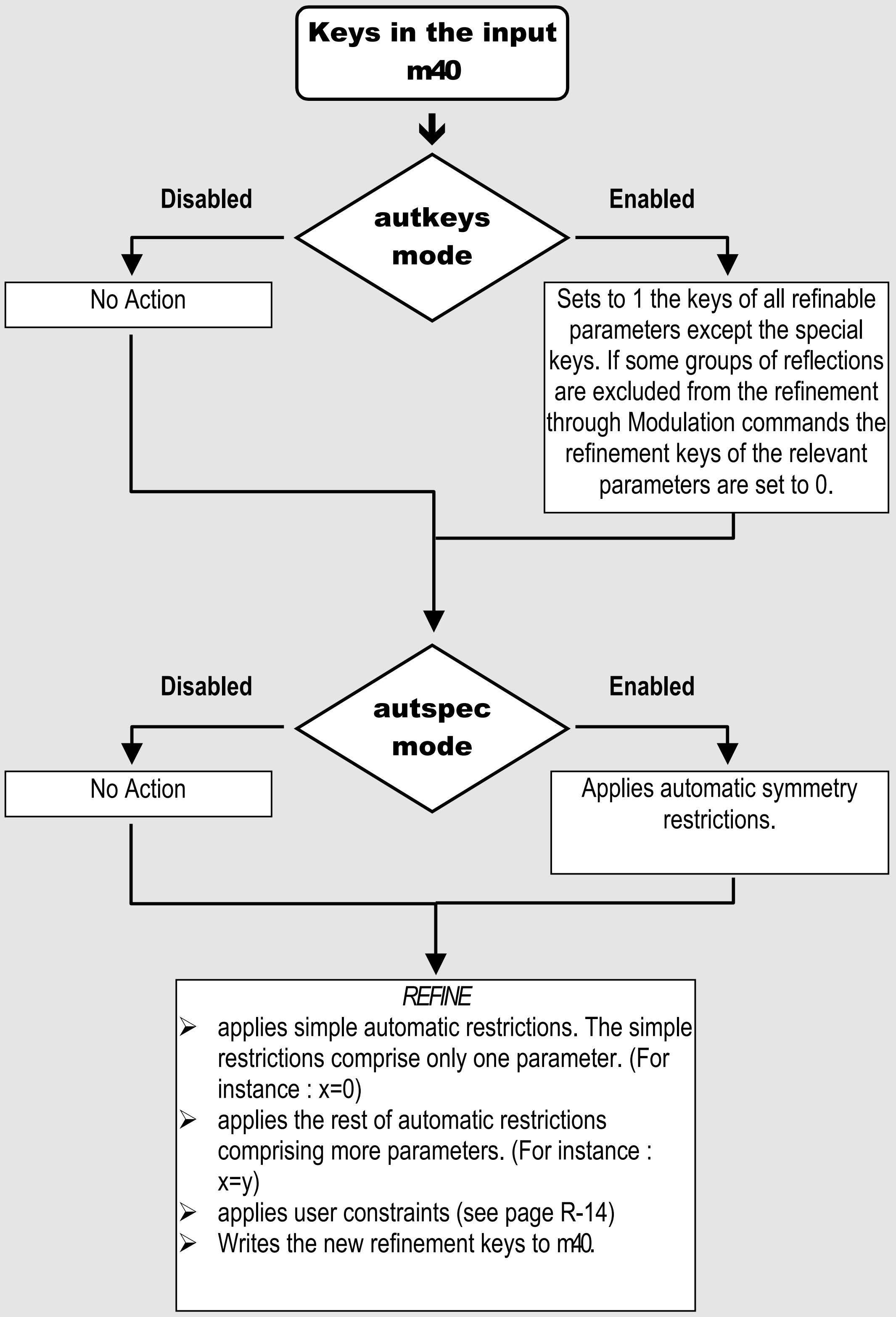
Refinement of some parameters in automatic mode and the others in manual mode is not possible. However, the user can define user constraints by `equation`, `restric` and `fixed` commands - see page 224 for details. These commands are used for making various constraints for user-defined groups of parameters and/or modify settings done by `autkeys` and `autspec` commands.

---

[1] In three dimensions only. The remaining axes must be fixed by the user - see page 245.

The setting order for refinement keys

Figure 125 The setting order of refinement keys



Sets to 1 the keys of all refinable parameters except the special keys. If some groups of reflections are excluded from the refinement through Modulation commands the refinement keys of the relevant parameters are set to 0.

Applies automatic symmetry restrictions.

REFINE

- applies simple automatic restrictions. The simple restrictions comprise only one parameter. (For instance : x=0)
- applies the rest of automatic restrictions comprising more parameters. (For instance : x=y)
- applies user constraints (see page R-14)
- Writes the new refinement keys to m40.

Usage of the button Break

The refinement can be interrupted by button Break. At the moment the button Break is pushed the actual parameters and its refinement keys are saved in `m40`. This can be used for the checking of the refinement keys before finishing of the zero refinement cycle. Another possible usage of Break is to start *REFINE*, break it immediately, turn off the `autkeys` or `autspec` mode, set manually some keys and restart the refinement.

Initial values of modulation parameters

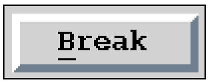
Modulation parameters of a structure model usually cannot be refined from zero amplitudes as the normal matrix is becoming singular. Therefore *EDITM40* sets new modulation parameters to small positive values. *REFINE* changes the initial values only if it is necessary during application of symmetry restrictions and user constraints. This means for example that if the initial value of some parameter is not zero but this parameter should be zero because of symmetry restriction or user constraints *REFINE* sets its value to zero.

EDITM40 doesn't check the symmetry restrictions and sets the initial values to all new parameters. In the case the `autspec` mode is disabled the user should set not only refinement keys but also their initial values.
In the case of manual parameters (see Table 13) the non-zero initial value sometimes causes that the parameter will be refined.

Table 13 *The parameters without automatic refinement keys*

Symbol and name	**Initial value**	**Refinement key if the autkey mode is turned on**
`ai` site occupation	usually set by *EDITM40*	set by user
`o` constant term of occupation wave	usually set by *EDITM40*	set by user
`o,osin1` width and center of crenel function (see page 128)	set by user	set by user
`scale6, scale5` etc. twin domain scales (see page 108)	set by user	set by user
`xsin$, ysin$, zsin$, xcos$` and `ycos$` slope (the 1st three parameters), center and width of sawtooth function (see page 124)	set by user	set by user
`scale 1` scale factor of the first data set (see page 108)	usually set by *EDITM40*	1
`scale2, scale3` etc. scale factor of the 2nd, 3rd etc. data sets (see page 108)	set by user	1 if the initial value is not 0
`phason, phasonm` (see page 139 and 151)	set by user	1 if the initial value is not 0

Interactive commands

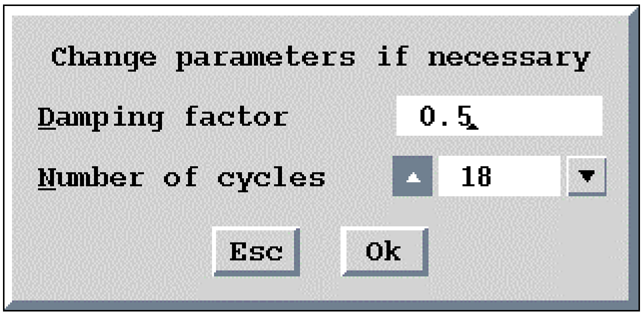


This button cancels immediately the refinement. After the break the refinement listing is incomplete and the file `m80` is not created or it is incomplete. `Break` can be used for checking of refinement keys (see page 226).

This button causes *REFINE* finishes the current cycle, then calculates the last cycle, creates `m80`, completes the listing from scratch files and exits.

This button can be used for changing of the number of the cycles or of the damping factor during the refinement run. It is often used if the results printed to the screen during the refinement indicate that the refinement needs to be damped, the number of cycles needs to be enlarged etc. If *REFINE* ends regularly, i.e. if it is not cancelled by `Break` button, the changes are written to `m50` after the last cycle is finished. If the refinement runs with the refinement repeat (see page 231 for details) the changes are written to `m50` after the last cycle of each refinement repeat.

Figure 126 *The Refinement Options*


Change parameters if necessary

Damping factor 0.5

Number of cycles 18

Backup copy of `m40`

REFINE saves the input `m40` as `s40`. This can be recovered by `Recover m40 file` of the Tools menu, which is accessible from the basic window of *JANA98*. If the refinement runs with the refinement repeat (see page 231 for details) `s40` is created at the beginning of each repeat. The initial `m40` is therefore definitively lost at the beginning of the second refinement repeat.

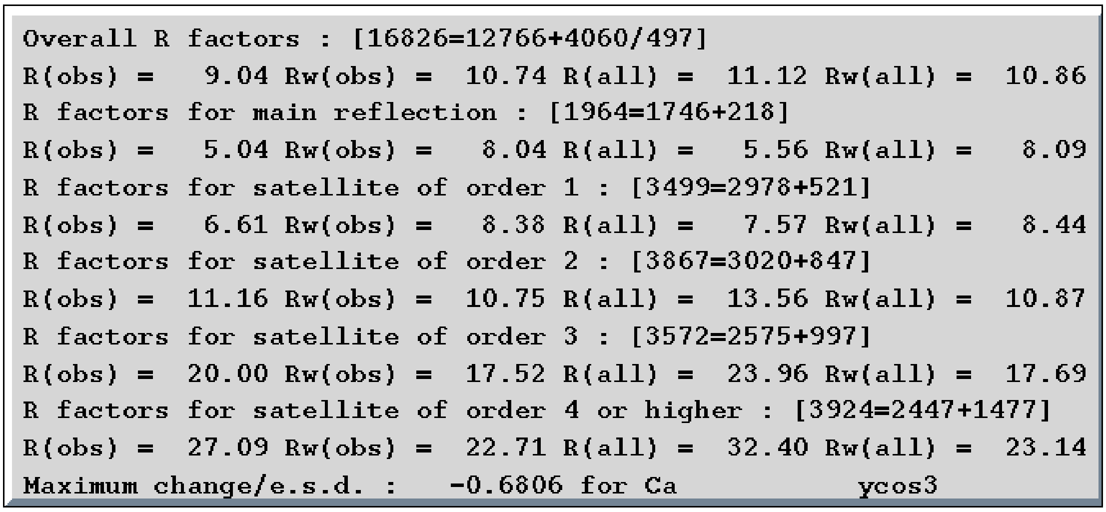
The screen output of the refinement

The following figure shows the output *REFINE* writes to the screen after each refinement cycle. The expression
`[16826=12766+4060/497]`
means that the total number of reflections used for the refinement if 16826, from which 12766 is observed and 4060 is unobserved. The number of refined parameters is 497. Reflection is considered observed if its intensity is larger than $k\sigma(I)$. The value k and the fact if the unobserved reflections will or will not be used in the refinement can be defined by the user - see page 237.

The expression
`[1964=1746+218]`
has the same meaning main reflections, another one is for the 1st order satellites etc.

The most important value of the R factors for checking the convergence of the refinement is `Rw(all)`, because the minimised function is the nominator of `Rw(all)`. The `Rw(obs)` and `Rw(all)` are calculated from observed reflections and all reflections, respectively.

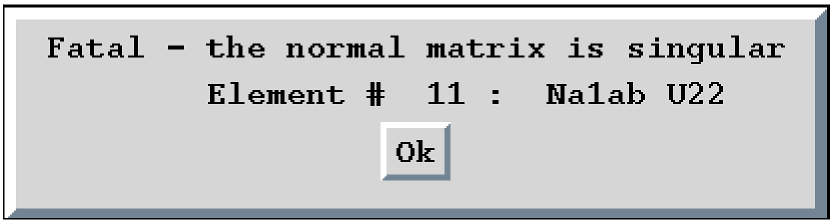
Figure 127 *The Screen Output of REFINE*



```
Overall R factors : [16826=12766+4060/497]
R(obs) =   9.04 Rw(obs) =  10.74 R(all) =  11.12 Rw(all) =  10.86
R factors for main reflection : [1964=1746+218]
R(obs) =   5.04 Rw(obs) =   8.04 R(all) =   5.56 Rw(all) =   8.09
R factors for satellite of order 1 : [3499=2978+521]
R(obs) =   6.61 Rw(obs) =   8.38 R(all) =   7.57 Rw(all) =   8.44
R factors for satellite of order 2 : [3867=3020+847]
R(obs) =  11.16 Rw(obs) =  10.75 R(all) =  13.56 Rw(all) =  10.87
R factors for satellite of order 3 : [3572=2575+997]
R(obs) =  20.00 Rw(obs) =  17.52 R(all) =  23.96 Rw(all) =  17.69
R factors for satellite of order 4 or higher : [3924=2447+1477]
R(obs) =  27.09 Rw(obs) =  22.71 R(all) =  32.40 Rw(all) =  23.14
Maximum change/e.s.d. :   -0.6806 for Ca          ycos3
```

Singular Refinement

In case the normal matrix cannot be inverted because its determinant is zero *REFINE* exits with message



```
Fatal - the normal matrix is singular
        Element #  11 :  Na1ab U22
```

In the case showed in this example the parameter responsible for singularity is `U22` of atom Na1ab (alone or in interaction with other parameters).

Singularity is often caused by some general problem in the structure model. The following list shows several possible reasons:

- Some atom is too close to a special position (either by user mistake or due to existence of a split position).
- Some atom is too close to a special position of the former/higher space group after transforming to a subgroup structure. Such models cannot be often refined without atom being moved from former special positions (by a text editor in `m40` file).
- Incorrect or incomplete symmetry restrictions of atoms or molecules located at special positions. This can happened only if the automatic procedure for setting them is switched off .
- The space group origin is incorrectly or incompletely fixed. This can happened only if the automatic procedure for setting of refinement keys is switched off.
- Large change of orthogonalization parameters for existing orthogonalized waves.
- Zero or too small initial values of phason, twin scales etc. - see Table 13.
- Non-zero refinement key corresponding to a non-existing scale. It may happen for instance if the twinning option has been removed in *PRELIM* but the twin scales still exist in `m40`.
- Improper parameter used for fixing of origin in the superspace (see Fixed command - option "x4 axis", page 245). The simplest case is if the chosen parameter is fixed to 0 by symmetry or if its modulation is very small.
- Inappropriate shape (planar, linear molecule) of a molecule with TLS description of the thermal motion. In this case the TLS components are to be restricted to account for the special shape by using equation commands (page 247).

Checking of the Results

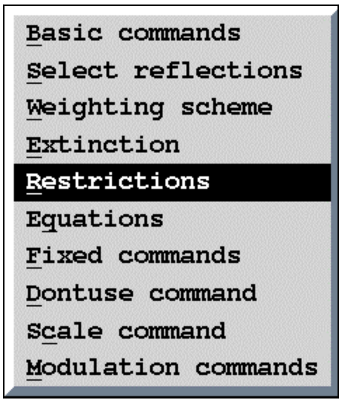
This is list of the most important results the user should check during and after the refinement:

- ***Convergence***. The `Rw` values in the screen output of the refinement show whether the refinement is convergent. The `change/e.s.d` value shows if the refinement is already converged - is is typically less then 0.1 in this case. See the example in page 228.
- ***Temperature parameters***. Their values should not be too large (`m40`) and they must positive definite (see the end of the refinement listing).
- ***Correlations***. See the end of the refinement listing.
- ***R-statistics*** (in the listing).
- ***Refined values*** in `m40`.
- ***Ratio between values and e.s.d's***. for refined parameters (`m40`, but the e.s.d.'s are not listed together with parameters, or the listing). In the case of modulation waves it can be an indication if contribution of some wave is significant.

3.3.4 The SetCommands tool for *REFINE*

The description of the SetCommands interface is in page39. The following menu can be opened also by clicking of the right mouse button on the icon of *REFINE*.

Figure 128 *The Main Menu of the Refinement Commands*



Commands entered through the SetCommands interface are save in `m50`. Their syntax and meaning is explained in the tables following the description of single forms of the SetCommands tool. In Table 14 the commands are listed alphabetically.

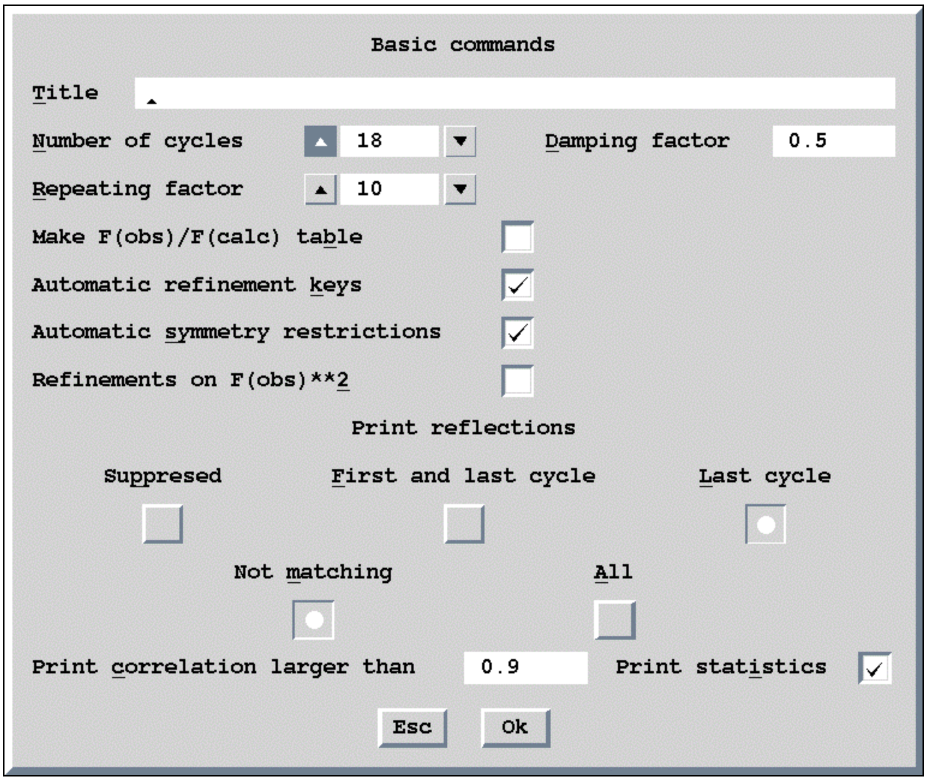
Table 14 *The Index of the Refinement Control Commands*

Autkeys......236	Diff.............254	Itype...........244	Selcomp 254	Unstab........242
Autspec 236	Dontuse 240	Method.......254	Selsat 254	Useunobs ...239
Badref........239	Equation.....252	Omdif 259	Siglevel...... 239	Weight........242
Checkran....259	Fixed..........250	Overlap......254	Skipbad 239	Yomax........242
Chidif..........259	Fofc............236	Print...........236	Skipflag...... 240	Yomin.........242
Corr............237	Fsquare......236	Radius 244	Snlmn 240	
Cycles........236	Grid 254	Restric 251	Snlmx 240	
Damp 236	Idistr...........244	Repeat.......236	Stat............ 237	
Dfoftw.........259	Iext.............244	Scale..........240	Thdif 259	

3.3.5 The Basic Commands

The Basic commands form is used for fundamental settings like number of refinement cycles and also for basic options of the refinement listing.

Figure 129 *The Basic Commands for REFINE*



Basic commands

Title

Number of cycles 18

Damping factor 0.5

Repeating factor 10

Make F(obs)/F(calc) table ☐

Automatic refinement keys ☑

Automatic symmetry restrictions ☑

Refinements on F(obs)**2 ☐

Print reflections

Suppresed ☐

First and last cycle ☐

Last cycle ●

Not matching ●

All ☐

Print correlation larger than 0.9

Print statistics ☑

The number of cycles, F_oF_c table and main features of the listing

The maximal number of refinement cycles is 18. Before the first cycle and after the last cycle *REFINE* can print to the listing either a list of all reflections or non-matching reflections. The criterion for "not matching" reflections can be set in the Select reflections form (see page 237). During the last cycle *REFINE* can create list of F_o and F_c (`m83`).

The number of refinement cycles is limited to 18 because the number of temporary files used for creation of the refinement listing depends on the number of refinement cycles. However, the refinement run can be repeated several times as defined by the repeating factor. The repeating factor 0 or 1 means that there is no refinement repeat and that refinement ends after the last cycle. Otherwise the whole process repeated and the output files such as s40, m80 and ref. are overwritten.

Refinement keys and symmetry restrictions

The automatic refinement keys and automatic symmetry restrictions are explained in page 223.

The minimised function

The minimised functions for the refinement based on F_{obs} and F_{obs}^2 are given in page 221.

The damping factor

REFINE calculates and applies the changes of refined parameters after each refinement cycle. The damping factor multiplies the changes by a factor given by the user before their application.

If the refinement converges well the damping factor should be 1. In case of oscillating refinement it should be ½ or less. The oscillating refinement can be usually recognised from the last line of the screen output (see page 228): if the values of change/e.s.d. of the same parameter and the same atom are positive and negative in successive refinement cycles and their absolute value don't fall down. The values maximum and average of change/e.s.d. are not reduced by the damping factor and therefore you cannot reach satisfactory values only by selecting of "proper" damping factor.

Another example where the damping factor should be used is transformation of the space group to a lower one. At the beginning stage the parameters of the expanded structure are too close to the former higher symmetric ones. Therefore it is necessary to break down the symmetry by changing of some parameters (usually coordinates) but nevertheless the initial changes may be unrealistic due to strong non-linear character of such refinement and the reasonable damping factor for several first cycles is about 0.1 or less.

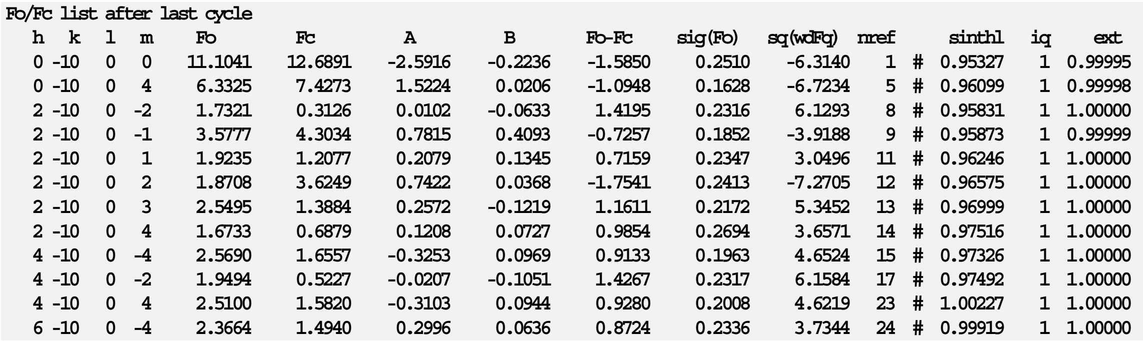
List of reflections in the refinement listing

The user can choose if the refinement listing will contain list of reflections. The list can be printed after the first or the last cycle and it can be the full list or only list of non-matching reflections. A non-matching reflection fulfils the equation $\sqrt{w_F \left|F_o - F_c\right|} > u$ or $\sqrt{w_{F^2} \left|F_o^2 - F_c^2\right|} > u$ for the refinement based on F^2, where *u* is defined by the user. The user limit can be changed *via* the Select reflections form (page 238).

The list of reflections contains

- indices, F_o, F_c, real(A) and the imaginary(B) part of the structure factor or indices , F_o, F_c, F_{c1}, F_{c2} for twinned structures. The last two items are cotributions of the first and second twin domain to the structure factor.
- the difference F_o-F_c and $\sigma(F_o)$
- the weighted difference, which is defined as $\sqrt{w_F \left|F_o - F_c\right|}$ for the refinement based on F and $\sqrt{w_{F^2} \left|F_o^2 - F_c^2\right|}$ for the refinement based on F^2.
- number of reflection (nref) in `m91`
- $\sin\frac{\theta}{\lambda}$
- The flag (#) indicating non-matching reflections
- The flag (*) indicating unobserved reflections
- Number of the scale factor (iq). The scale factors can be assigned for different data sets (see page 74), for twin domains (see page 75) or for groups of reflections (see the Dontuse command, page 238) .
- Extinction correction

Figure 130 *FoFc List in the Refinement Listing*



```
Fo/Fc list after last cycle
  h   k   l   m     Fo        Fc         A         B       Fo-Fc    sig(Fo)   sq(wdFq)  nref      sinthl   iq    ext
  0 -10   0   0   11.1041   12.6891   -2.5916   -0.2236   -1.5850    0.2510   -6.3140    1  #  0.95327   1  0.99995
  0 -10   0   4    6.3325    7.4273    1.5224    0.0206   -1.0948    0.1628   -6.7234    5  #  0.96099   1  0.99998
  2 -10   0  -2    1.7321    0.3126    0.0102   -0.0633    1.4195    0.2316    6.1293    8  #  0.95831   1  1.00000
  2 -10   0  -1    3.5777    4.3034    0.7815    0.4093   -0.7257    0.1852   -3.9188    9  #  0.95873   1  0.99999
  2 -10   0   1    1.9235    1.2077    0.2079    0.1345    0.7159    0.2347    3.0496   11  #  0.96246   1  1.00000
  2 -10   0   2    1.8708    3.6249    0.7422    0.0368   -1.7541    0.2413   -7.2705   12  #  0.96575   1  1.00000
  2 -10   0   3    2.5495    1.3884    0.2572   -0.1219    1.1611    0.2172    5.3452   13  #  0.96999   1  1.00000
  2 -10   0   4    1.6733    0.6879    0.1208    0.0727    0.9854    0.2694    3.6571   14  #  0.97516   1  1.00000
  4 -10   0  -4    2.5690    1.6557   -0.3253    0.0969    0.9133    0.1963    4.6524   15  #  0.97326   1  1.00000
  4 -10   0  -2    1.9494    0.5227   -0.0207   -0.1051    1.4267    0.2317    6.1584   17  #  0.97492   1  1.00000
  4 -10   0   4    2.5100    1.5820   -0.3103    0.0944    0.9280    0.2008    4.6219   23  #  1.00227   1  1.00000
  6 -10   0  -4    2.3664    1.4940    0.2996    0.0636    0.8724    0.2336    3.7344   24  #  0.99919   1  1.00000
```

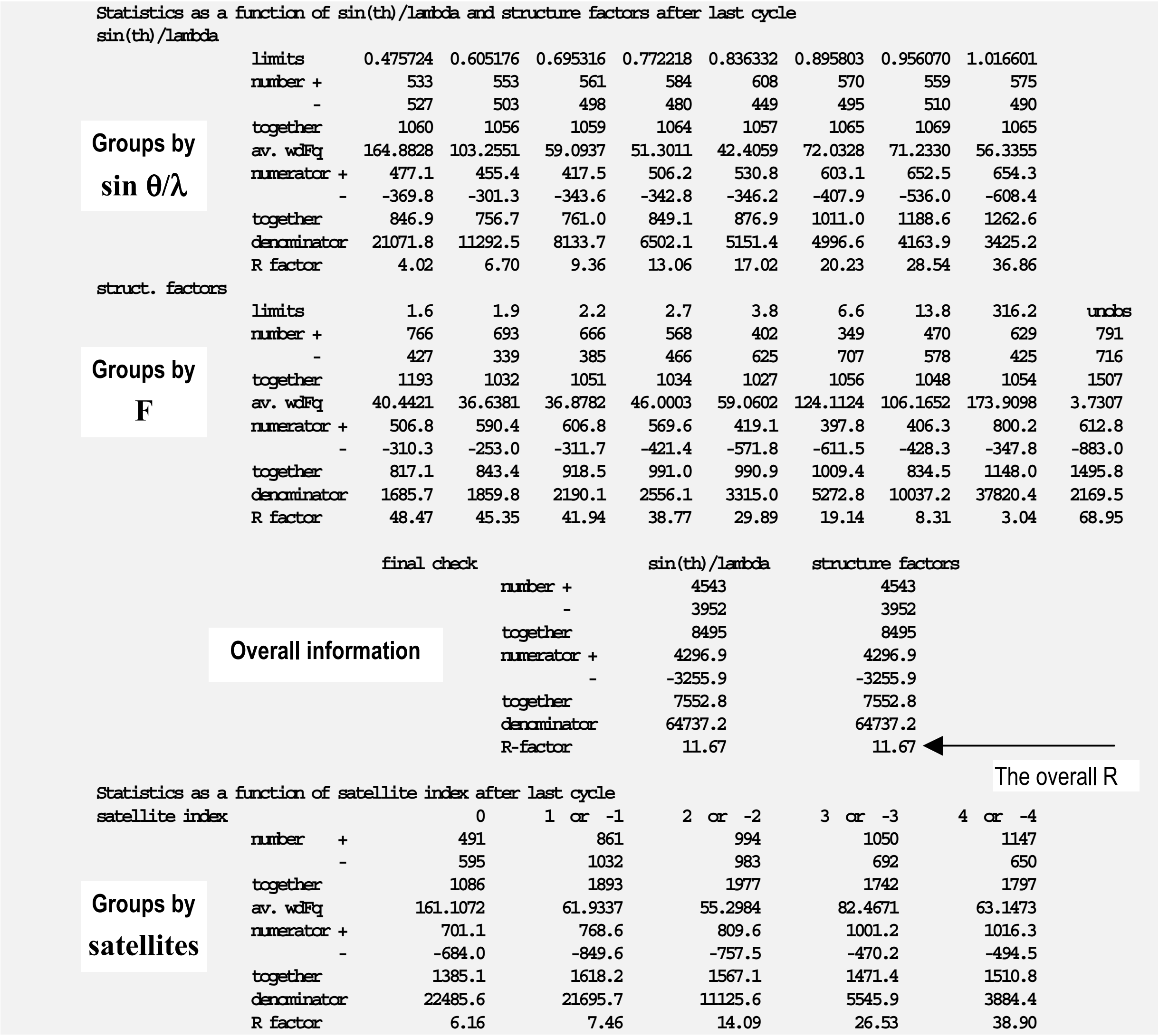
R statistics

The R statistics shows the R factors for reflections distributed to approximately equally populated groups of sinθ/λ or F. The intervals of sinθ/λ and F for the groups are calculated by *PRELIM* during Creation of the refinement reflection file.

Information for each group of reflections in the listing:

• number +	number of reflections with positive F_o-F_c
• number -	number of reflections with negative F_o-F_c
• together	the total number of reflections in a group
• average wdFq	average value of $w_F\left\|F_o - F_c\right\|$ or $w_{F^2}\left\|F_o^2 - F_c^2\right\|$. It is closely related to goodness of fit (see equation *E62* page 222).
• numerator +	sum of the positive differences $\|F_o\|$-$\|F_c\|$
• numerator -	sum of the negative differences $\|F_o\|$-$\|F_c\|$
• together	sum of the differences $\| F_o$-$F_c \|$
• denominator	sum of $\|F_o\|$
• R factor	R factor $\frac{\sum_{hkl}\left\|\left\|F_o\right\|-\left\|F_c\right\|\right\|}{\sum_{hkl}\left\|F_o\right\|}$ for the group. It is always calculated from F_o and F_c regardless if the refinement is based on F or F^2

Figure 131 *R Statistics in the Refinement Listing*



```
Statistics as a function of sin(th)/lambda and structure factors after last cycle
sin(th)/lambda
                limits          0.475724  0.605176  0.695316  0.772218  0.836332  0.895803  0.956070  1.016601
                number +             533       553       561       584       608       570       559       575
                       -             527       503       498       480       449       495       510       490
                together            1060      1056      1059      1064      1057      1065      1069      1065
                av. wdFq        164.8828  103.2551   59.0937   51.3011   42.4059   72.0328   71.2330   56.3355
                numerator +        477.1     455.4     417.5     506.2     530.8     603.1     652.5     654.3
                          -       -369.8    -301.3    -343.6    -342.8    -346.2    -407.9    -536.0    -608.4
                together           846.9     756.7     761.0     849.1     876.9    1011.0    1188.6    1262.6
                denominator      21071.8   11292.5    8133.7    6502.1    5151.4    4996.6    4163.9    3425.2
                R factor            4.02      6.70      9.36     13.06     17.02     20.23     28.54     36.86
struct. factors
                limits               1.6       1.9       2.2       2.7       3.8       6.6      13.8     316.2      unobs
                number +             766       693       666       568       402       349       470       629        791
                       -             427       339       385       466       625       707       578       425        716
                together            1193      1032      1051      1034      1027      1056      1048      1054       1507
                av. wdFq         40.4421   36.6381   36.8782   46.0003   59.0602  124.1124  106.1652  173.9098     3.7307
                numerator +        506.8     590.4     606.8     569.6     419.1     397.8     406.3     800.2      612.8
                          -       -310.3    -253.0    -311.7    -421.4    -571.8    -611.5    -428.3    -347.8     -883.0
                together           817.1     843.4     918.5     991.0     990.9    1009.4     834.5    1148.0     1495.8
                denominator       1685.7    1859.8    2190.1    2556.1    3315.0    5272.8   10037.2   37820.4     2169.5
                R factor           48.47     45.35     41.94     38.77     29.89     19.14      8.31      3.04      68.95

                             final check             sin(th)/lambda    structure factors
                                         number +          4543              4543
                                                -          3952              3952
                                         together          8495              8495
                                         numerator +     4296.9            4296.9
                                                   -    -3255.9           -3255.9
                                         together        7552.8            7552.8
                                         denominator    64737.2           64737.2
                                         R-factor         11.67             11.67

Statistics as a function of satellite index after last cycle
satellite index                          0     1  or  -1     2  or  -2     3  or  -3     4  or  -4
                number    +            491           861           994          1050          1147
                          -            595          1032           983           692           650
                together              1086          1893          1977          1742          1797
                av. wdFq          161.1072       61.9337       55.2984       82.4671       63.1473
                numerator +          701.1         768.6         809.6        1001.2        1016.3
                          -         -684.0        -849.6        -757.5        -470.2        -494.5
                together            1385.1        1618.2        1567.1        1471.4        1510.8
                denominator        22485.6       21695.7       11125.6        5545.9        3884.4
                R factor              6.16          7.46         14.09         26.53         38.90
```

Correlations

The elements of the inverse matrix can be used as a measure of the interdependence of refined parameters. The quantity called *correlation coefficient* is defined by the following equation:

$$\delta_{ij} = \frac{b_{ij}}{\sqrt{b_{ii}}\sqrt{b_{jj}}},$$

where b_{ij} are elements of the inverse normal matrix.

The correlation coefficients can range from 0 to 1, the latter indicating completely dependent parameters. Correlation coefficients of standard structures are usually less than 0.2, but they can be much larger if, for example, some atomic positions are near to a special symmetric position (disordered structures) or if the structure has symmetry which is close to some supergroup.. The modulated structures can be understand as a perturbation of the regular structure and therefore it has necessarily larger correlation coefficients than standard structures. By our experience the values less than 0.9 are still acceptable for modulated structures.

Large correlations slow down the refinement and may cause an oscillation. For oscillating refinement the damping factor about 0.5 or less is necessary. The following example shows the typical output of a strongly correlated refinement. This output is located at the very end of the refinement listing.

```
There were  17 correlations larger than  0.900 in last refinement cycle
 0.946 correlation  : x Na1ba/z Na1ba                0.908 correlation  : y Na2aa/y O13ab

 0.945 correlation  : y Na1ba/y Na2aa                0.907 correlation  : y Na2aa/y O13aa

 0.939 correlation  : x Na2aa/z Na2aa                0.905 correlation  : y Na1ba/y O13ab

 0.925 correlation  : y Na2aa/y Na2ba                0.905 correlation  : y O13aa/y O13cb

-0.917 correlation  : xsin2 Na1aa/xsin2 Na1ba        0.904 correlation  : y Na1ba/y O13aa

 0.916 correlation  : y O13aa/y O13ab                0.903 correlation  : y O13ab/y O13bb

 0.915 correlation  : y Na1ba/y Na2ba               -0.903 correlation  : ysin1 Na1aa/ysin1 Na1ba

-0.914 correlation  : xsin2 Na2aa/xsin2 Na2ba        0.902 correlation  : y Na2aa/y O13bb

 0.910 correlation  : y O13aa/y O13bb
```

Table 15 *The Basic Commands of REFINE in the `m50` File*

Name	Syntax and Description
cycles	**cycles** `number` **Default value**: cycle 1 Number of the refinement cycles. Can be changed interactively during the refinement (see page 227). If `number` = 0, *REFINE* calculates directly the last cycle without any change of refined parameters.
damp	**damp** `number` **Default value**: **damp** 1 The damping factor multiplies the changes of refined parameters before their application after the refinement cycle. It can be changed interactively during the refinement (see page 227).
repeat	**repeat** `number` **Default value:** repeat 1 The refinement run will be performed `number` times.
fofc	**fofc** `flag` **Default value**: `fofc 0` Key for creating of the F_o-F_c table for publication. The table is written to `m93`. `flag` = 0 off `flag` = 1 on
autkeys	**autkeys** `flag` **Default value**: **autkeys** `1` Key for the automatic initialization of all refinement keys. See §3.3.3, page 223 for details. `flag` = 0 off `flag` = 1 on
autspec	**autspec** `flag` **Default value**: **autspec** 1 Key for automatic application of the symmetry restrictions to the refinement keys and initial values of refined parameters. See §3.3.3, page 223 for details. `flag` = 0 off `flag` = 1 on
fsquare	**fsquare** `number` **Default value**: **fsquare** 0 `flag` = 0 the refinement will be carried out on F_o `flag` = 1 the refinement on F_o^2 See §3.3.1, page 221 for details.
print	**print** `flag` **Default value**: **print** -2 Selects which reflections will be printed to the listing and when they will be printed. The selection is based on the limit for “non-matching reflections” (`limit`) and on the reflection weight (`w`). `Limit` can be modified by command **badref** - see §3.3.6, page 237, weight can be modified by command **weight** - see §3.3.7, page 241.

	`flag` = -2 only structure factors with $\lvert F_o-F_c\rvert$ > `limit` will be printed in the last cycle of the refinement. `flag` = -1 only structure factors with $\lvert F_o-F_c\rvert$ > `limit` will be printed in the first and in the last cycle of the refinement `flag` = 0 no reflections will be printed `flag` = 1 all structure factors used in the refinement will be printed in the first and the last cycle of the refinement `flag` = 2 all structure factors used in the refinement will be printed in the last cycle of the refinement
corr	**corr** `number` **Default value**: **corr** 0.9 The key for the printing of the correlations to the refinement listing. All correlations larger than |`number`| will be printed
stat	**stat** `flag` **Default value**: **stat** 1 key for the printing the R statistics to the refinement listing (See page 233 for details). `flag` = 0 off `flag` = 1 on

3.3.6 Modifying of the input reflection set

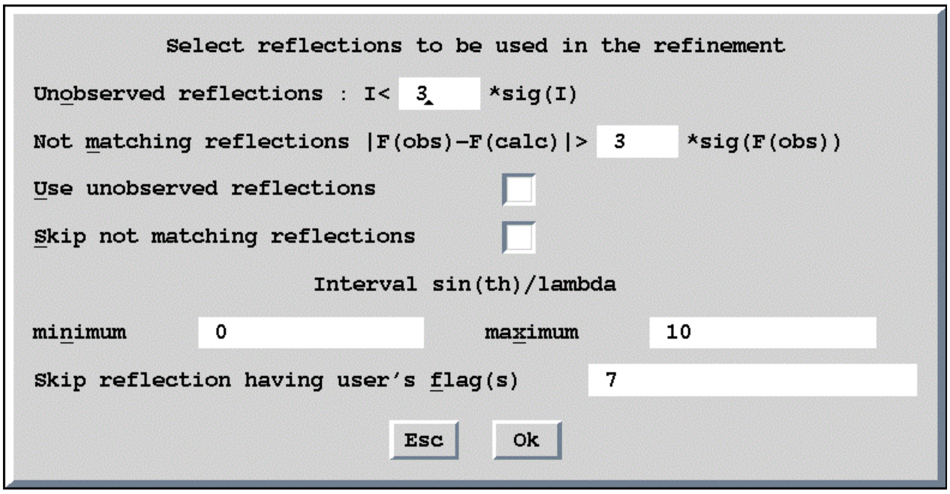
In this part the user makes decision which reflections will be used in the refinement, assigns scale factors for groups of reflections and sets the "non-matching" criterion. All these settings work both for standard and modulated structures. The selection of reflections based on satellite indices or number of composite parts is special for modulated structures and is described in page 253.

Selecting reflections

The decision about reflections used for the refinement can be made by several ways. The exclusion of unobserved reflections suppresses influence of weak reflections. The exclusion of non-matching reflections suppresses influence of the reflections that are not properly described by the structure model. This option should be used only in well-founded cases or as a tool for finding some regularities between not-matching reflections. The exclusion of high angle reflections by setting of the upper $\sin(\theta)/\lambda$ limit suppresses anharmonic effects and can be useful in initial stages of the refinement. The exclusion of the reflection *via* the user flag in `m91` is useful if only several reflections are to be excluded from the refinement. These are usually reflections having improper profiles or other indication that their intensity may be wrong. The last possibility is to exclude from the refinement a group defined by a condition on indices – see Dontuse command.

The limit for non-matching reflections influences also the refinement listing – see page 233.

Figure 132 *The Select Reflections Form*



Select reflections to be used in the refinement

Unobserved reflections : I< 3 *sig(I)

Not matching reflections |F(obs)-F(calc)|> 3 *sig(F(obs))

Use unobserved reflections ☐

Skip not matching reflections ☐

Interval sin(th)/lambda

minimum 0 maximum 10

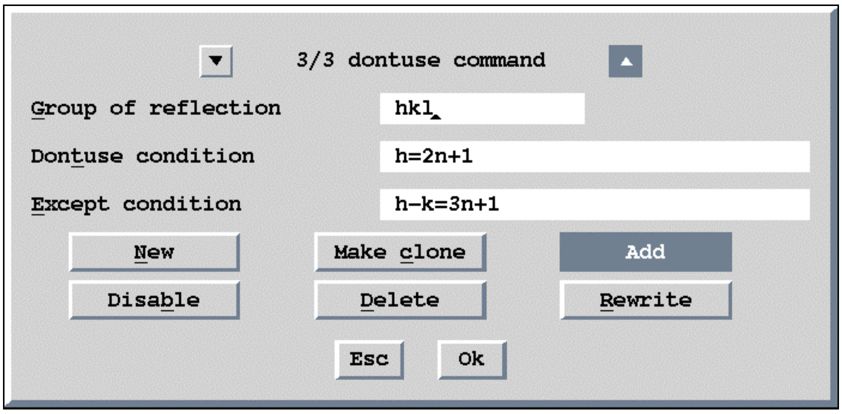
Skip reflection having user's flag(s) 7

The Dontuse command

The Dontuse command selects a group of the reflections, which will not be used in the refinement, on the basis of the Dontuse and Except conditions for the indices of the group. Here are the syntax rules for groups and conditions:

- The group is defined by hkl[mnp] letters. They can be replaced by zero but this is the only allowed change (i.e. h00 is allowed but hh0 causes the syntactic error).
- The left side of the Dontuse and Except conditions contains indices hkl[mnp] and integer numbers.
- The right side of the Dontuse and Except conditions contains integer numbers and the letter n as a symbol for arbitrary integer number. The indices cannot be in the right side.

Figure 133 *The Dontuse Command Form*



3/3 dontuse command

Group of reflection hkl

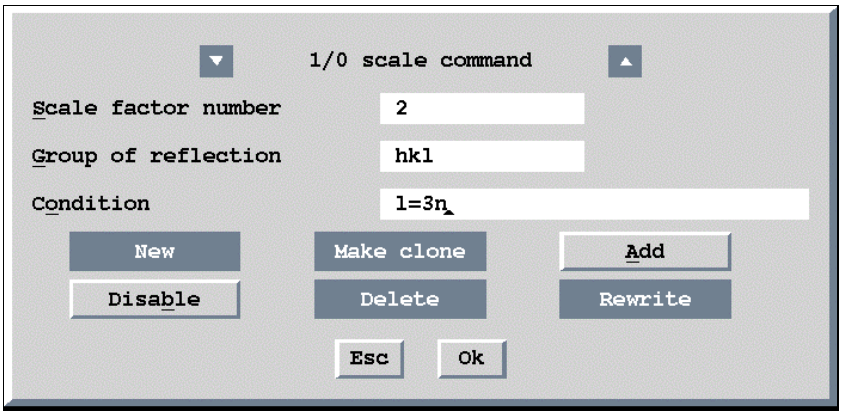
Dontuse condition h=2n+1

Except condition h-k=3n+1

The Scale command

The Scale command assigns a scale factor to a specified group of reflections. Together with this assignment the user should add the corresponding non-zero scale factor to `m40` (see page 108 for details about scale parameters). The rules for the definition of groups and conditions are the same like with the Dontuse command.

Figure 134 *The Scale Command Form*



Scale factor number: 2

Group of reflection: hkl

Condition: l=3n

Table 16 *The Select Reflections Commands of REFINE in the* `m50` *File*

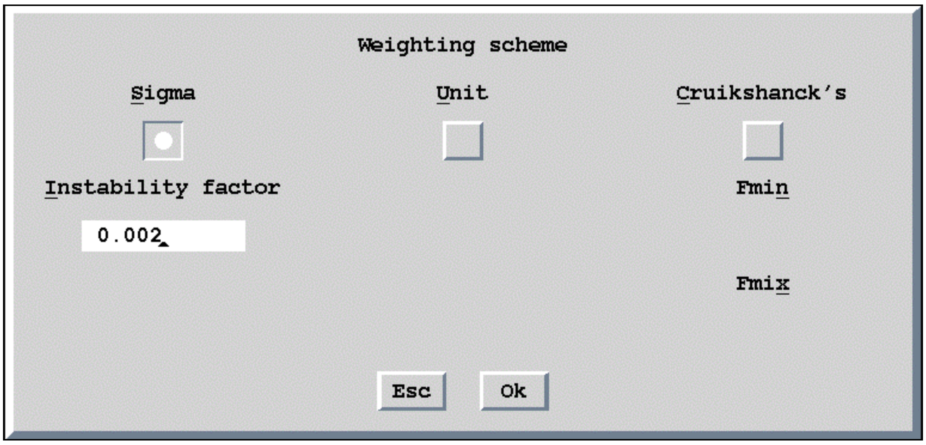
Name	Syntax and Description
siglevel	**siglevel** `number` **Default value**: **siglevel** 3 The limit for observed reflections I/σ(I).
badref	**badref** `number` **Default value**: **badref** 3 Sets the limit for not-matching reflections. The reflections with `|Fo-Fc|` > `number` will be marked in the listing by #. (A proper combination of **badref** and **print** makes possible to limit the output of the not-matching reflections.)
useunobs	**useunobs** `flag` **Default value**: **useunobs** 1 Key for using of the unobserved reflections in the refinement. The selection which reflections are considered observed can be made by **siglevel** command. `flag = 0` the unobserved reflections will be used `flag = 1` the unobserved reflections will not be used
skipbad	**skipbad** `flag` **Default value**: **skipbad** 0 Key for deleting of the non-matching reflections from the refinement. The reflections with `|Fo-Fc|` > `limit` will be deleted. The predefined value of limit is 3 and can be changed by the **badref** command. `flag = 0` the non-matching reflections will not be skipped `flag = 1` the non-matching reflections will be skipped In case that the flag is equal 1 the not matching reflections will be still present in the listing to allow some analysis.

***Table 16** (Continued)*

snlmn	**snlmn** `number` **Default value**: **snlmn** 0 Minimal value of sinϑ/λ for acceptance of the reflection into the refinement
snlmx	**Syntax**: **snlmx** `number` **Default value**: **snlmx** 10 Maximal value of sinϑ/λ for acceptance of the reflection into the refinement
skipflag	**skipflag** `flag1 flag2 . . .` **Default value**: none The reflections with the user key = `flag` will be skipped. See §2.2.10, page 87 for details about the user flag.
dontuse	**dontuse** *group* : *condition* `except` *condition* (The colon must be separated by spaces) **Default value**: none Defines the group of reflections which will not be used in the refinement. *Group* denotes a class of reflections for which `condition` will be tested. The number of indices in the `group` must be the same as the dimension of the superspace (hkl, hklm, hklmn, hklmnp). Some of the indices in *group* can be zeros. No other changes of *group* are permitted (i.e. h0l is allowed but hhl is forbidden). The syntax of `condition` is explained in the following examples. *Examples:* dontuse hklm : h-k+2l-m=5n+2 dontuse hklm : h-k+2l-m=5n+2 except h=2n dontuse 00l : l=2n dontuse hhl : h=2n *syntax error*
scale	**scale** *number* `for` *group* : *condition* (The colon must be separated by spaces) **Default value**: none Sets individual scale factor for a group of reflections fulfilling the *condition*. *Number* is used for the scale flag in the file `m91` (see §2.2.10, page 87) The syntax for the *group* and *condition* is the same as for the **dontuse** key. *Example:* scale 5 for hklm : h=2n

3.3.7 Weighting scheme

Figure 135 *The Weighting Scheme Form*



Weighting scheme

Sigma ●

Unit ☐

Cruikshanck's ☐

Instability factor

0.002

REFINE uses three types of weighting schemes. In the first one the ***weight is based on sigma*** - see equation E57, page 221. The coefficient *u* in the equation *E57* is the the instability factor, which can be set by the user. The instability factor should be used as instrument constant and its value should not be changed for structures measured on the same diffractometer under the same conditions. The typical value of *u* is about 0.01. The coefficient is multiplied by 100 before it is saved to `m50`.

Another weighting scheme uses ***unit weights***. It is useful in cases when the sigmas and not known or wrong.

The last one – the **Cruickshank's weight**, see equation E63, had been used for photographic data but it can be also used in cases when the sigmas and not known or wrong.

$$w = \left(2F_{o,\min} + F_o + \frac{2F_o^2}{F_{o,\max}} \right)^{-1} \qquad E63$$

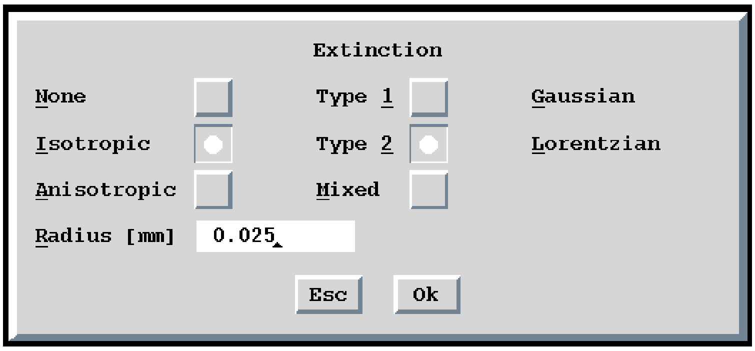
Table 17 *The Weighting Scheme Commands of REFINE in the* `m50` *File*

weight	**weight** `flag` **Default value**: **weight** 0 Sets the weighting scheme `flag = 0` The weight based on sigma (see equation E57, page 221). This type of weight depends on **unstab** command. `flag =1` unit weight `flag =2` Cruickshank's weight , see equation E63, page 241. This type of weight depends on **yomin** and **yomax** commands.
unstab	**unstab** `number` **Default value**: **unstab** 0 Coefficient of unstability used for the calculation of weight (, page 221). In `m50` its value is multiplied by 100 so that the coefficient used in equation E57 is `number`*0.01.
yomin	**yomin** `number` **Default value**: **yomin** 5 Minimum value of F_o used for the calculation of Cruickshank's weight
yomax	**yomax** `number` **Default value**: **yomax** 200 Maximum value of F_o used for the calculation of Cruickshank's weight

3.3.8 Extinction correction

The underlying theory for extinction correction and explanation of extinction parameters in `m40` is given in page 111. The Extinction form is used for definition of the extinction type. The corresponding changes of `m40` (i.e. setting of refinement keys and initial values[1]) is done automatically by *REFINE* before starting the refinement.

Figure 136 *The Extinction Form*



Extinction

None ☐ Type 1 ☐ Gaussian

Isotropic ● Type 2 ● Lorentzian

Anisotropic ☐ Mixed ☐

Radius [mm] 0.025

The radius is used for only for the case when $\bar{t}$ is not recorded in the m91 file and when the equation *E13* is to be used.

JANA98 works with the following types of the secondary extinction:

- Isotropic Type I, Gaussian distribution.
- Isotropic Type I, Lorentzian distribution.
- Isotropic Type II
- Isotropic mixed types, Gaussian distribution.
- Isotropic mixed types, Lorentzian distribution.
- Anisotropic Type I, Gaussian distribution.
- Anisotropic Type I, Lorentzian distribution.
- Anisotropic Type II

The anisotropic extinction has not been completely finished and its use should be first consulted with the authors.

---

[1] The extinction parameters of type 1 and type 2 are written in different lines of `m40`. Therefore, after change of the distribution type, the old parameters in `m40` must be deleted and replaced by zeroes. In *JANA98* this is done automatically in the autkeys mode. Without autkeys mode the parameters are changed but the refinement keys are not.

Table 18 *The Extinction Commands of REFINE in the `m50` File*

iext	**iext** `flag` **Default value**: **iext** 0 Key of the extinction correction `flag = 0` no extinction correction `flag = 1` isotropic extinction correction `flag = 2` anisotropic extinction correction
itype	**itype** `flag` **Default value**: **itype** 1 The type of the extinction correction `flag = 1` extinction type I `flag = 2` extinction type II `flag = 3` general case (simultaneous refinement of both I and II)
idistr	**idistr** `flag` **Default value**: **idistr** 1 Key of the statistical distribution of the mosaic domains `flag = 1` Gaussian distribution `flag = 2` Lorentzian distribution
radius	**radius** `number` **Default value**: **radius** 0.01 The radius of a spherical sample in cm

3.3.9 User constraints

JANA98 uses three types of user constraints:

- ***Fixed command*** fixes some parameters to the values they currently have in `m40`.
- ***Restriction command*** defines that some parameters of a given group of atoms are identical or complementary.
- ***Equation command*** is used for setting of linear equations between parameters.

The user constraints are set in the following order [1]:

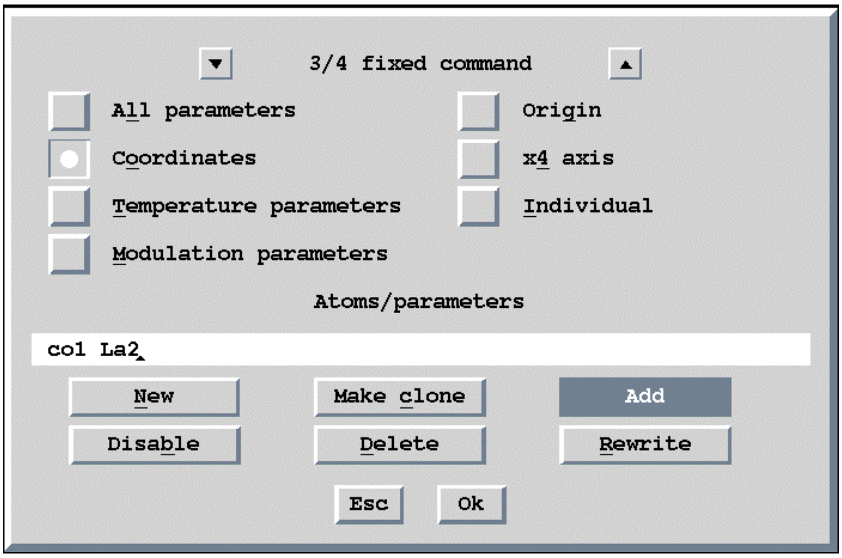
1. Equations defined by user
2. Equations defined following from the site symmetry
3. Restrictions

The Fixed commands are used just once in every refinement run during automatic set key procedure. In case that the autkyes is set to zero the fixed commands are ineffective.

The Fixed command

The Fixed command fixes parameters of one or more atoms to values they currently have in `m40`. The following figure shows its user interface. Very simple example of the Fixed command is fixing the whole structure except scale parameters - this is done by choosing "All parameters" and pressing "*" in the text box of the Fixed command form.

Figure 137 The Fixed Commands Form



3/4 fixed command

☐ All parameters
● Coordinates
☐ Temperature parameters
☐ Modulation parameters
☐ Origin
☐ x4 axis
☐ Individual

Atoms/parameters: col La2

---

[1] The Restriction command generates in `m50` one or more commads with the keyword **restric**. Similarly the Equation command generates lines with the keyword **equation**. The order of these lines in `m50` is not important for the setting order of refinement keys, i.e. the Equation command has higher priority regardless of its position in `m50`.

The options ***All parameters, Coordinates, Temperature parameters and Modulation parameters*** are used for a group of atoms. The group has to be defined in the textbox by list of names separated by one or more spaces. Wildcards are permitted.

The options ***Origin*** is used for fixing of the three dimensional origin in case when needed. Usually there is no need to use this option because *REFINE* makes it automatically by fixing the relevant coordinates of the heaviest atom in the structure. Sometimes (for instance if the position of the heaviest atom is not fully occupied) the atom used for the origin fixing should be defined by fixed command. The textbox should contain the name of the atom used for the origin fixing.

The options ***X4 axis*** is used for fixing of the forth coordinate of the origin in case when needed. The fixing is necessary if the sign of `x4` is positive for all symmetry operators it is done by fixing one modulation parameter of some atom. The textbox should therefore contain the name of this parameter followed by the name of the relevant atom in the square brackets, for example xsin1[C12].

REFINE never fixes `x4` automatically because the selection may be ambiguous, but it tests whether `x4` needs to be fixed and warns the user if it is necessary. The fixed parameter has to be sufficiently modulated but it can be hard to say which one it is at the beginning stage of the refinement. The possible strategy is to fix some parameter, run several cycles of refinement and look if there is some better choice. The usual way is to fix by this command `xcos1 (`or `ycos1,` or `zcos1)` of one atom and set its value to zero.

For ***five and six dimensional structures*** the origin fixing in `x5` and x6 direction has to be done by Equation command.

Individual is an option for fixing single parameters. The textbox should contain a list of parameter names with relevant atom names in the square brackets. Wildcards for atom names are permitted but they are forbidden for parameter names. For example `xsin1[na*]` means that the parameter `xsin1` will be fixed for all atom names starting “`na`”, while `xsin*[na]` is not allowed.

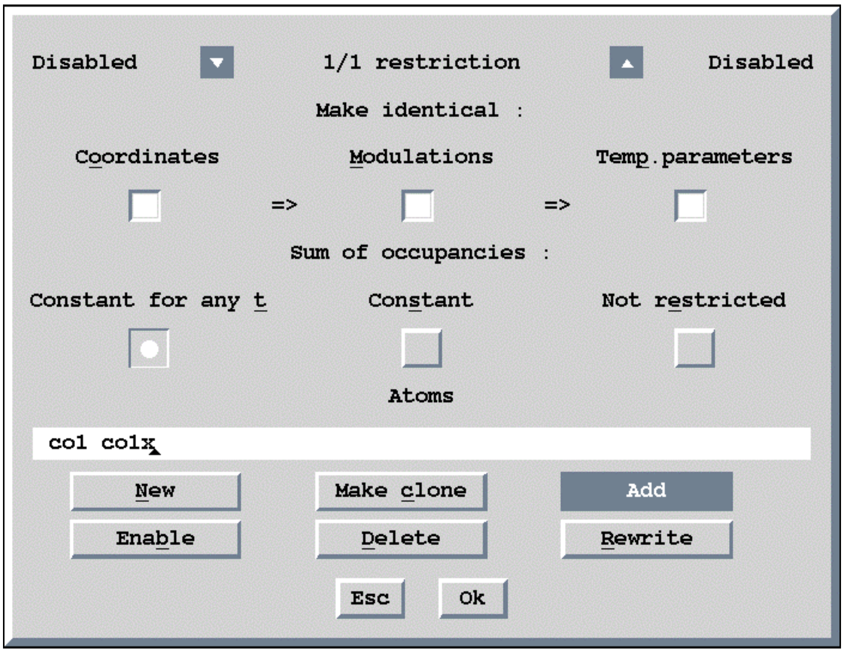
The Restriction command

The Restriction command defines that some parameters of a given group of atoms are identical or complementary. The priority of the Restriction command is lower than the one of the Equation command[1].

---

[1] The Restriction command generates in `m50` one or more commads with the keyword **restric**. Similarly the Equation command generates lines with the keyword **equation**. The order of these lines in `m50` is not important for the setting order of refinement keys, i.e. the Equation command has higher priority regardless of its position in `m50`.

Figure 138 *The Restriction Form*



☐ Coordinates ☐ Modulations ☐ Temp.parameters

● Constant for any t ☐ Constant ☐ Not restricted

Atoms: col colx

The restrictions selected in the form are defined for one or more atoms given in the textbox. The first atom of the group is used as a representative one for refining of equal or complementary parameters and therefore the relevant refinement keys of all other atoms are set to zero. The wildcards in atom names are permitted.
The parameters describing occupancy modulation of atoms can be set to have constant occupancy for each t. This allows so called substitutional modulation.

The Equation command

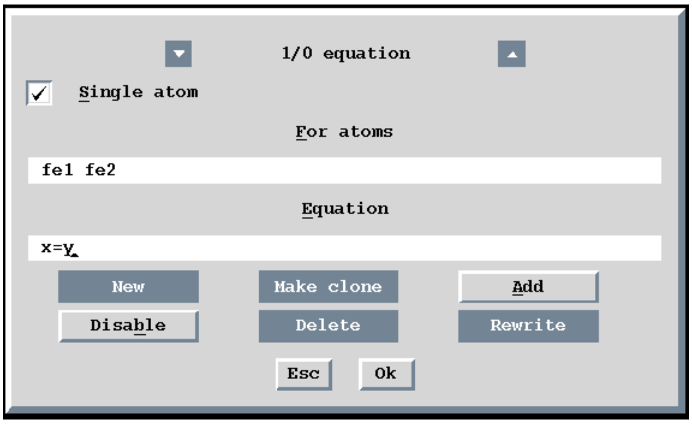
The Equation command is used for setting of linear equations between parameters[1].

The user can define two types of equations:
Internal equations define relations between parameters of one atom or molecule. In the example given in Figure 139 the equation "x = y" is applied first for coordinates of atom Fe1 and then for coordinates of atom Fe2. The Equation textbox should contain a linear equation for parameters without atom reference, i.e. *without* atom names in the square brackets.
Inter atomic/molecular equations can be used for relations between parameters of different atoms/molecules and/or other parameters. They are activated if the Single atom checkbox is cleared. In case of atomic or molecular parameters the Equation textbox should contain a linear equation for parameters *with* atom/molecule reference, i.e. with atom/molecule name in square brackets. The wildcards in atom names are *not* possible. In the example given in Figure 140 the x coordinate of atom Fe1 and Fe2 are equal.

---

[1] Parameters used in an equation can be part of a restriction or fixed command.

Figure 139 *The Equation Form with an internal equation*

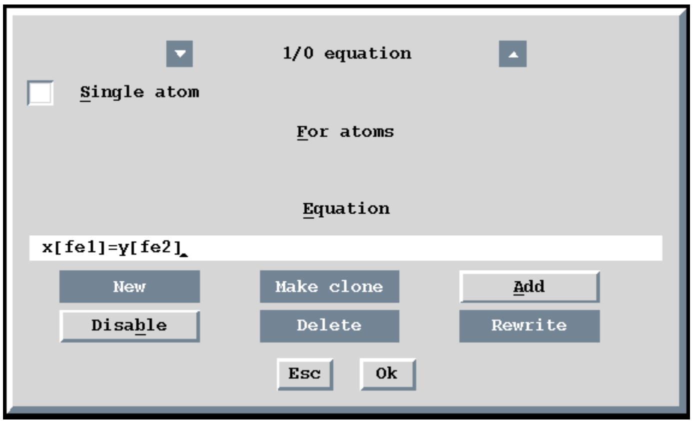


☑ Single atom

For atoms: fe1 fe2

Equation: x=y

Figure 140 *The Equation form with an inter atomic/molecular equation.*



☐ Single atom

For atoms:

Equation: x[fe1]=y[fe2]

Syntax rules for structure parameters

The basic element of expressions used in user constraints is `p[A]` (meaning: parameter `p` of atom `A`), where `p` is one from parameter names from Table 10, page 98, and `A` is the relevant atom. The name of atom can contain an identifier of molecular or symmetry position (see page 27). The wildcards in atom names are usually allowed (except logical cases, like origin fixing by the Fixed command, and except the interatomic equations).

Instead of `A` we can sometimes use `M#n`, where `M` is name of a molecule and `n` is the sequence number of the molecular position in `m40`. `M#n` therefore define a group consisting of all atoms of the model molecule in the `n`-th molecular position.

Tip: if you are not sure with influence of a user constraint you can set the constraint, start *REFINE*, break it immediately by Break button and check the refinement keys in `m40` and the refine listing where all equations are listed. In case the equation was not fulfilled the listing contains a warning.

The following table shows several examples of user constraints. The are given for a hypothetical structure consisting of
A1, A2, A3 individual atoms,
B1, B2, B3, B4 atoms in the model molecule M1 and
C1, C2, C3, C4 atoms in the model molecule M2.
Each molecule has several positions.

Table 19 *Examples of User Constraints*

Command	Options	Textbox	Notes
fixed	origin	A2	The origin in 3 dimensions will be fixed by fixing atom A2. Refine automatically fixes only the coordinates of A2 which must be fixed.
fixed	x4 axis	xcos1[A2]	xcos component of the first positional modulation wave of A2 will be used for fixing of the origin in the superspace.
fixed	coordinates	B*	Fixes coordinates of model molecule M1.
fixed	modulation parameters	M1	Fixes molecular modulation parameters calculated for the first position of molecule M1.
fixed	modulation parameters	M1#1	The same as previous example.
fixed	modulation parameters	M1#2 M1#3	Fixes molecular modulation parameters calculated for the 2nd and 3rd position of molecule M1.
fixed	modulation parameters		
equation	for atoms M1#1 M2#1	S23 = 0	Fixes to zero the S23 component of the TLS tensor of the 1st position of molecules M1, M2.

Table 20 *The Constraints Commands of REFINE in the* `m50` *File*

fixed	**fixed** `flag at1 at2 . . .` **fixed** `parameter[at1] parameter[at2] . . .` **Default value**: none Key for fixing groups of parameters for atoms `at1, at2` …. The corresponding refinement keys of these atoms are set to zero which causes the selected parameters not to be refined. This command doesn't work if the automatic setting of the refinement keys is disabled by **autkeys** 0 command. The wildcards in *atom names* are permitted. `flag = xyz` positional parameters `flag = all` all parameters `flag = beta`[1] thermal parameters `flag = u` thermal parameters `flag = mod` modulation parameters `flag = pol` the origin will be fixed by atom `at1` (Example: **fixed** pol C12) `flag = x4` fixing of the origin in the x4 direction. The name of the atom is followed by the name of the fixed parameter. (Example: **fixed** x4 C12 xsin1 … the origin in the x4 direction will be fixed by means of the xsin1 parameter of atom C12) `parameter[at]` is a name of a parameter [of atom `at`] *Examples:* **fixed** `mod C1 c2 b*` fixes modulation of atoms C1,C2 and all atoms with names from B **fixed** `all *` fixes all parameters for all atoms **fixed** `scale6 x[Sn1] U12[Sn3]` fixes the 6th scale factor, x coordinate of the atom Sn1 and the U12 temperature parameter of the atom Sn3.

---

[1] The names `Beta` or `U` can be used regardless of the temperature parameters type currently used in `m40`.

Table 20 *(Continued)*

restric	**restric** `at1 flag  at2 at3 . . .`
	Default value: none
	Sets restrictions on coordinates, thermal, modulation and occupancy parameters for a group of atoms from `m40`. The type of restriction is defined by `flag`. The restricted parameters are refined only for atom `at1` and kept the same for `at2`, `at3` .... If `at1` is missing, the restricted parameters are refined for the first atom from the list on the right.
	
	The wildcards in *atom names* are permitted.
	
	In some cases `flag` can be negative, which means that the restriction is defined for two substitutionally modulated atoms. The occupation modulation waves of such atoms are complementary and the sum of occupancies at each t is constant.
	
	`flag = 4` ***occupational sum***
	The sum of occupancies in the group is constant.
	`flag = -4` ***complementary occupational waves*** (only for atom pairs)
	`o[at1] + o[at2] is constant`
	`osin1[at2] = -osin1[at1]`
	`. . . . . .`
	`osin16[at2] = -osin16[at1]`
	`flag = 13` ***thermal parameters***
	The thermal parameters in the group are equal.
	`flag = 3` ***occupational sum + thermal parameters***
	The sum of occupancies in the group is constant. The thermal parameters in the group are equal.
	`flag = -3` ***complementary occupational waves + thermal parameters*** (only for pair of atoms)
	Two atoms have complementary modulation waves and equal thermal parameters.
	`flag = 12` ***thermal and modulation parameters***
	The thermal and modulation parameters in the group are equal.
	`flag =  2` ***occupational sum + thermal and modulation parameters***
	Sum of occupancies in the group is constant; thermal and modulation parameters are equal.
	`flag =  -2` ***complementary occupational waves + thermal and modulation parameters***
	(only for pairs of atoms)
	Two atoms have complementary modulation waves and equal thermal and modulation parameters.
	`flag = 11` ***positional, thermal and modulation parameters***
	Positional, thermal and modulation parameters in the group are equal.
	`flag = 1` ***occupational sum + positional, thermal and modulation parameters***
	Sum of the occupancies in the group is constant; positional, thermal and modulation parameters in the group are equal.

`flag = -1` ***complementary occupational waves + positional, thermal and modulation parameters***
(only for pairs of atoms)
Two atoms have complementary modulation waves and equal positional, thermal and modulation `parameters`.

Examples:
restric `Cu1 11 Ag1 al* C?`
restric `h* 13` In this example the restricted parameters are refined for the first atom of the list `h*`.

equation

equation for atom `at1 . . atn : parameter = expression`
equation `: parameter[at] = expression`
(The colon must be separated by spaces)
Default value: none

Setting of linear constraints to refined parameters.

`at1 . . . atn`	list of atoms from `m40` for which the restriction is to be applied. The names of atoms are case insensitive. **Wildcards are not permitted**!
`parameter`	name of the constrained parameter
`parameter[at]`	name of the constrained parameter of atom `at`
`expression`	linear expression composed from numbers, arithmetic signs “+”, “-“ ,“*” and names of the constrained parameters. Note that a parameter with direct reference to atom (`parameter[at]`) is only allowed in the second type of the equation command.

Examples:
equation for atoms `cu1 cu2 Al3 : y = 0.2*x + 0.15`
equation : `ocos1[co2] = 0.0423 - 0.7*osin1[fe3]`

equation : `ocos1[co*] = 0.0423 - 0.7*osin1[fe3]`
syntax error (wildcards are not permitted)

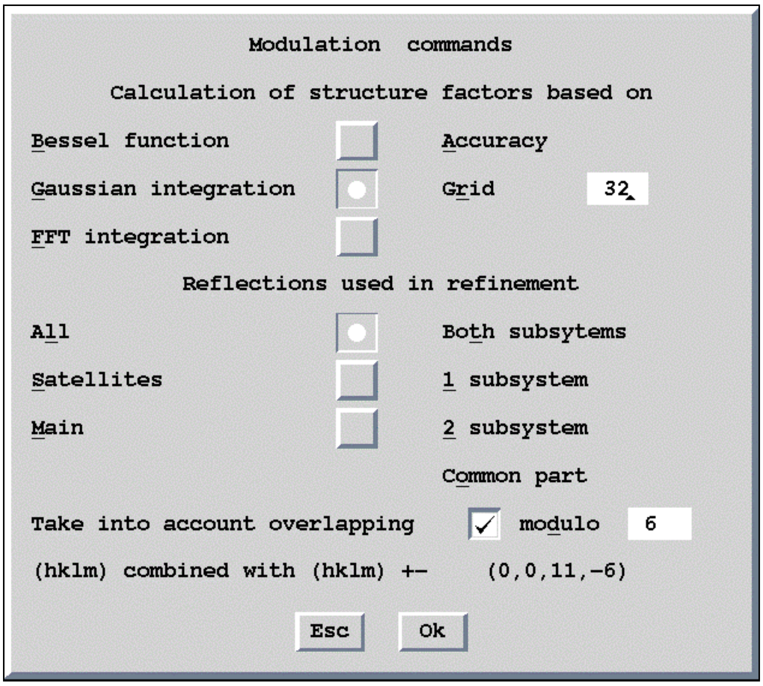
equation `: ocos1[co2] = 0.0423 - 0.7*osin1`
syntax error (mixed `parameter` and `parameter[at]`)

equation for atoms `cu1 cu2 Al3 : y = 0.2*x[c25] + 0.15`
syntax error (mixed `parameter` and `parameter[at]`)

3.3.10 Modulation commands

The Modulation commands are used for user options applicable only to modulated or composite structures. The following figure shows the user interface.

Figure 141 *The Modulation Commands Form*



Modulation commands

Calculation of structure factors based on

Bessel function ☐ Accuracy

Gaussian integration ● Grid 32

FFT integration ☐

Reflections used in refinement

All ● Both subsytems

Satellites ☐ 1 subsystem

Main ☐ 2 subsystem

Common part

Take into account overlapping ☑ modulo 6

(hklm) combined with (hklm) +- (0,0,11,-6)

The first part of the form is used for choosing the calculation method for structure factors. The numerical Gaussian and FFT method work well in most cases and with the default grid 32 combine good accuracy with reasonable speed of the refinement. The analytical Bessel method is fast and accurate for one or two harmonic waves but the calculation time grows rapidly for more complex modulations. However for 5 and 6 dimensional structures the numerical methods are not implemented and the Bessel functions are the only possibility[1].

In the second part of the form there are selection rules for reflections that will be used in the refinement. The options for the composites are activated only for composite structures. In the autkeys mode *REFINE* sets refinement keys according to selected reflections - see page 224 for details.

If we select for the refinement all reflections including satellites but we do not refine any modulation parameters the contributions of calculated structure factors to satellite reflections will be zero and the relevant R values for satellites will be 100%. The refinement runs well, but selecting of Main instead of All will speed up the calculation. This is the way how to refine the basic and modulated structure with the same reflection file. For large data sets, however, the skipping of satellites may be time consuming.

---

[1] For this reason modulation of ADP parameters is not available for 5 and 6 dimensional structures.

The last point in the form concerns the overlapping of satellite reflections for cases when the structure is almost commensurate. In such cases the program combines intensities of closed satellites as for twinned structure.

Table 21 *The Modulation Commands of REFINE in the* `m50` *File*

method	**method** `flag` **Default value**: **method** 1 Key for selection of the structure factors calculation method `flag = 0` Bessel functions `flag = 1` Gaussian method `flag = 2` Fast Fourier transform method
grid	**grid** `number` **Default value**: **grid** 32 The grid for the Gaussian method (see the command **method**)
diff	**diff** `number` **Default value**: **diff** 0.00001 The accuracy of calculation of Bessel functions (see the command **method**)
selsat	**selsat** `flag` **Default value**: **selsat** -2 Selection key for including satellite reflections into the refinement. `flag = -2` all reflections will be included `flag = -1` only satellite reflections will be included `flag = 0` only main reflections will be included `flag = n` only reflections with the satellite index |m|=n will be included
selcomp	**selcomp** `flag` **Default value**: **selcomp** 0 Key for the selection of reflections for composite structures `flag = 0` selects all reflections `flag = 1` selects reflections of the 1st composite `flag = 2` selects reflections of the 2nd composite `flag = 3` selects reflections of the 3rd composite `flag = 4` selects reflections common to the 1st and 2nd composite `flag = 5` selects reflections common to the 1st and 3rd composite `flag = 6` selects reflections common to the 2nd and 3rd composite `flag = 7` selects reflections common to all three composite parts
overlap	**overlap** `flag` **Default value**: none Key for testing of overlaps in the “almost” commensurate structures. (see page 258 for the explanation)

3.3.11 Twins and overlaps

This paragraph describes the way *REFINE* treats twinning. Another information concerning twins can be found in the chapter about *PRELIM* (page 67).

The simplest case are completely overlapped twins which can be refined using standard tools. If the twins are partially overlapped their refinement requires usage of special commands developed only for data from diffractometers with a classical point detector. The commands are rarely used and are not included in the SetCommands interface. They have to be written directly to `m50` by a text editor.

Main assumptions

A twinned crystal fulfils the following rules:

- it is composed from several identical domains
- each domain is a 3 dimensional crystal
- the domains are diffracting independently and their diffracted intensities are additive
- effects of boundaries are negligible

Overlapping of twin lattices

The lattice vectors of the domains forming a twinned crystal are related by a set of twinning operators $\{\hat{E} \equiv \hat{T}_1, \hat{T}_2, \cdots \hat{T}_n\}$:

$$\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\} \xrightarrow{\hat{E}} \{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\}$$

$$\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\} \xrightarrow{\hat{T}_2} \{\hat{T}_2\mathbf{a}_1^*, \hat{T}_2\mathbf{a}_2^*, \hat{T}_2\mathbf{a}_3^*\}$$

. . . .

$$\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\} \xrightarrow{\hat{T}_n} \{\hat{T}_n\mathbf{a}_1^*, \hat{T}_n\mathbf{a}_2^*, \hat{T}_n\mathbf{a}_3^*\}$$

The number *m* of independent vectors resulting from the transformations is important for classification of twins. The independent vectors define a common twin lattice. The twins can be divided to three basic groups:

- Completely overlapped twins having m=3
- Twins without overlapping[1] having m=3n
- Twins with partial overlapping having m between 3 and 3n.

complete overlapping ←$3 \le m \le 3n$→ *no overlapping*

---

[1] This case can be refined as a standard single crystal

Classification of twins based on their twinning matrices

The representation of a twin operator is a twinning matrix[1]. Its type is important for the refinement as it implicitly defines which reflections of single twin domains overlaps. The twins can have three possible types of the twin matrices:

- Twins with integer twin matrix
- Twins with rational twin matrix[2]
- Twins with irrational twin matrix

In the case of integer twin matrices the reflections of single twin domains fully overlaps and cannot be distinguished. The twins with rational twin matrices usually contain groups of fully overlapped and fully separated reflections. Partially overlapped reflections are also possible if the denominators of some numbers in the matrices are large. The twins with irrational twin matrices contain with high probability partially overlapped reflections.

Refinement of completely overlapped twins

Refinement of completely overlapped twins requires only the information about number of twin domains and twin matrices which are entered by *PRELIM* during creation of the basic reflection file `m50`. The structure factor in the case of the complete overlapping is calculated as

$$F^2(\mathbf{H}) = v_1 F^2(\mathbf{HT}_1) + v_2 F^2(\mathbf{HT}_2) + \ldots + v_n F^2(\mathbf{HT}_n)$$

where ν_i is a volume fraction of i-th domain and T_i is matrix representation of i-th twinning operator.

Refinement of partially overlapped twins

The reflections in the case of partially overlapped twins can be
- fully separated
- fully overlapped
- partially overlapped.

The fully overlapped reflections of all domains can be indexed on the base of reciprocal axis of one of the domains. Separated reflections need special flag to specify to which domain they belong - the flag is entered in the Import tool of *PRELIM*.

---

[1] The twinning matrices in *JANA98* are defined with respect to the **row** indices.

[2] i.e. having diffraction pattern which can be described in a supercell

Refinement of twins having only fully separated or fully overlapping reflections

The basic assumption is that the twin does not contain any partially overlapped reflections. The flag of the `checkran` command (see Table 22) should be set to 0 in order to disable the test of random overlapping. For each reflection used in the refinement the expression

$\left|h_{i1} - h_{ij}\right|$,

where h_{i1} is the i'th index of the reflection in the first twin domain and h_{ij} , j >1, is the index of the same reflection transformed to the basis of the j'th domain, is evaluated to distinguish the two following cases:

- If $\left|h_{i1} - h_{ij}\right| < l_i$, the reflection fully overlaps with the j'th one
- If $\left|h_{i1} - h_{ij}\right| > l_i$, the reflections are fully separated.

The limiting value l_i is predefined as 0.01 and can be changed by the `dfoftw` command (see Table 22).

Note: by defining the l_i limits the decision can be made whether the reflections of a twin with non-integer twinning matrices will be considered to be fully or partially overlapped. If the l_i limits are set to such values that $\left|h_{i1} - h_{ij}\right|$ is always less than l_i all reflections of the twin are treated as fully overlapped.

Refinement of twins having partially overlapped reflections

The twins with random overlaps are refined with the `checkran` flag set to 1. In this case the twinning matrices defined in *PRELIM* cannot be used for the testing of the overlaps. The user should enter the orientation matrices[1] of the single twin domains to `m50` file (see the `checkran` command, Table 22). For each the following steps are performed:

- the setting angles ϕ_1,χ_1,σ_1 and θ_1 are calculated from the corresponding orientation matrix
- the indices of the reflection are transformed to the basis of other twin domain through the twinning matrix and **rounded to the closest integer indices**.
- the setting angles $\phi_2 = \phi_1,\chi_2,\sigma_2$ and θ_2 are calculated for the transformed indices from the orientation matrix of the twin domain in question
- The differences $|\chi_1 - \chi_2|$, $|\sigma_1 - \sigma_2|$, $|\theta_1 - \theta_2|$ are calculated and used for the test:

---

[1] The orientation matrices should correspond to the reciprocal indices defined as **column** vectors. This is a difference from the definition of twinning matrices in *PRELIM* which are defined with respect to the **row** indices.

Overlapped reflection:
$|\chi_1 - \chi_2| <$ `chidif1` and $|\sigma_1 - \sigma_2| <$ `omdif1` and $|\theta_1 - \theta_2| <$ `thdif1`

Discarded reflection:
$|\chi_1 - \chi_2| \in$ <`chidif1`,`chidif2`> or $|\sigma_1 - \sigma_2| \in$ < `omdif1`,`omdif2` > or
$|\theta_1 - \theta_2| \in$ < `thdif1`,`thdif2` >

Separated reflection:
$|\chi_1 - \chi_2| >$ `chidif1` and $|\sigma_1 - \sigma_2| >$ and $|\theta_1 - \theta_2| >$ `thdif1`

The predefined limiting values in degrees
`chidif1` = 0.1 `chidif2` = 5.0
`thdif1` = 0.1 `thdif1` = 0.5
omdif1 = 0.1 `omdif1` = 0.5
can be changed by means of the command `chidif`, `thdif` and `omdif`. The values for `chidif` correspond to $\theta = 10^\circ$. The testing for other values of θ takes into account the broadening of χ profiles.

Note: **the testing for random overlaps can be only used for data measured by the equi-inclination method**. In other case you should contact the authors.

Refinement of “almost” commensurate structures

The twinning is not the only source of overlaps. Another type of overlapping occurs in incommensurate structures with **q** vector near to the commensurate one. If the satellites have significant intensity up to high order there is probability of overlapping between the satellites of two neighbouring main reflections or between the satellites and main reflections.

The overlapping command defines the denominator of the closed fraction. For example if the modulation vector is (0.123,0.252) the overlap command can have the following form:
overlap 8
causing the program will combine the intensity of the reflection (h,k,l,m) with the intensity of one reflections (h-1,k,l-2,m+8) or (h+1,k,l+2,m-8) having lower satellite index.

Table 22 *The Overlap Commands of REFINE in the* `m50` *File*

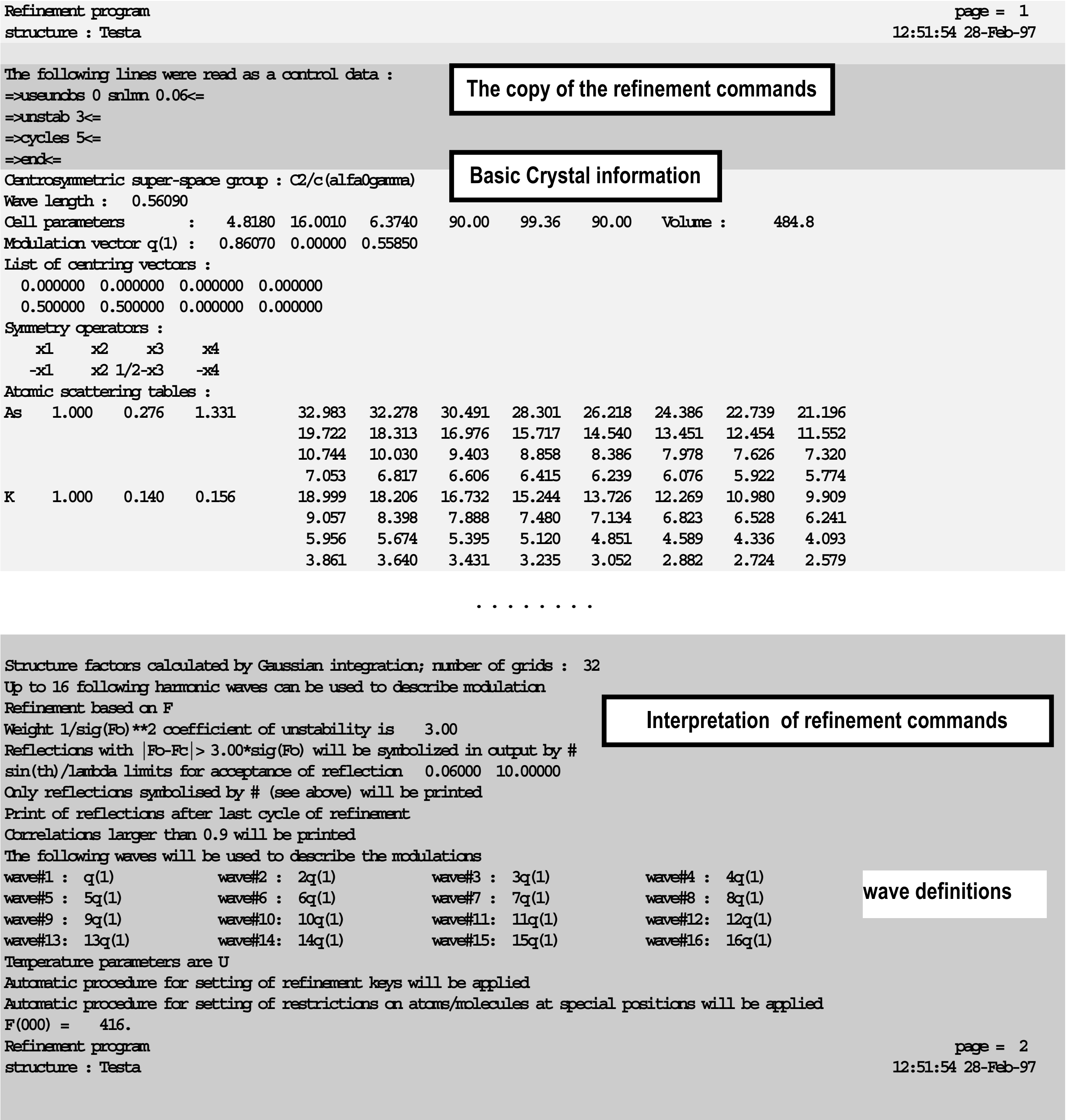
checkran	**checkran** `number` (`number` of orientation matrices)
	`r11 r12 r13`
	`r21 r22 r23`
	`r31 r32 r33`
	....
	Default value: **checkran** 0
	Command for the checking of overlaps.

dfoftw	**dfoftw** `number` (Ndim values) **dfoftw** `number` …. **dfoftw** 0.01 (Ndim values) **dfoftw** 0.01 …. Key for testing of the overlaps for meroedric or rational twins.
omdif **thdif** **chidif**	**omdif** `number1 number2` **thdif** `number1 number2` **chidif** `number1 number2` **Default value**: **omdif** 0.1 0.5 **thdif** 0.1 0.5 **chidif** 0.5 5.0 The limits in degrees for the testing of random overlaps.

3.3.12 Refinement listing

REFINE creates listing `ref` containing information about refinement. It can be viewed and printed by Listing viewer (see page 41). The listing is built at the end of the refinement from temporary files. If the refinement is interrupted by Break button the listing is incomplete.

Figure 142 *The refinement listing*



```
Refinement program                                                                              page =  1
structure : Testa                                                                      12:51:54 28-Feb-97

The following lines were read as a control data :
=>useunobs 0 snlmn 0.06<=
=>unstab 3<=
=>cycles 5<=
=>end<=
Centrosymmetric super-space group : C2/c(alfa0gamma)
Wave length :   0.56090
Cell parameters        :    4.8180  16.0010   6.3740    90.00    99.36    90.00    Volume :      484.8
Modulation vector q(1) :   0.86070  0.00000  0.55850
List of centring vectors :
  0.000000  0.000000  0.000000  0.000000
  0.500000  0.500000  0.000000  0.000000
Symmetry operators :
    x1     x2     x3     x4
   -x1     x2 1/2-x3    -x4
Atomic scattering tables :
As    1.000    0.276    1.331        32.983   32.278   30.491   28.301   26.218   24.386   22.739   21.196
                                     19.722   18.313   16.976   15.717   14.540   13.451   12.454   11.552
                                     10.744   10.030    9.403    8.858    8.386    7.978    7.626    7.320
                                      7.053    6.817    6.606    6.415    6.239    6.076    5.922    5.774
K     1.000    0.140    0.156        18.999   18.206   16.732   15.244   13.726   12.269   10.980    9.909
                                      9.057    8.398    7.888    7.480    7.134    6.823    6.528    6.241
                                      5.956    5.674    5.395    5.120    4.851    4.589    4.336    4.093
                                      3.861    3.640    3.431    3.235    3.052    2.882    2.724    2.579
```

. . . .

```
Structure factors calculated by Gaussian integration; number of grids :  32
Up to 16 following harmonic waves can be used to describe modulation
Refinement based on F
Weight 1/sig(Fo)**2 coefficient of unstability is    3.00
Reflections with |Fo-Fc|> 3.00*sig(Fo) will be symbolized in output by #
sin(th)/lambda limits for acceptance of reflection   0.06000  10.00000
Only reflections symbolised by # (see above) will be printed
Print of reflections after last cycle of refinement
Correlations larger than 0.9 will be printed
The following waves will be used to describe the modulations
wave#1 :  q(1)            wave#2 :  2q(1)           wave#3 :  3q(1)           wave#4 :  4q(1)
wave#5 :  5q(1)           wave#6 :  6q(1)           wave#7 :  7q(1)           wave#8 :  8q(1)
wave#9 :  9q(1)           wave#10:  10q(1)          wave#11:  11q(1)          wave#12:  12q(1)
wave#13:  13q(1)          wave#14:  14q(1)          wave#15:  15q(1)          wave#16:  16q(1)
Temperature parameters are U
Automatic procedure for setting of refinement keys will be applied
Automatic procedure for setting of restrictions on atoms/molecules at special positions will be applied
F(000) =     416.
Refinement program                                                                              page =  2
structure : Testa                                                                      12:51:54 28-Feb-97
```

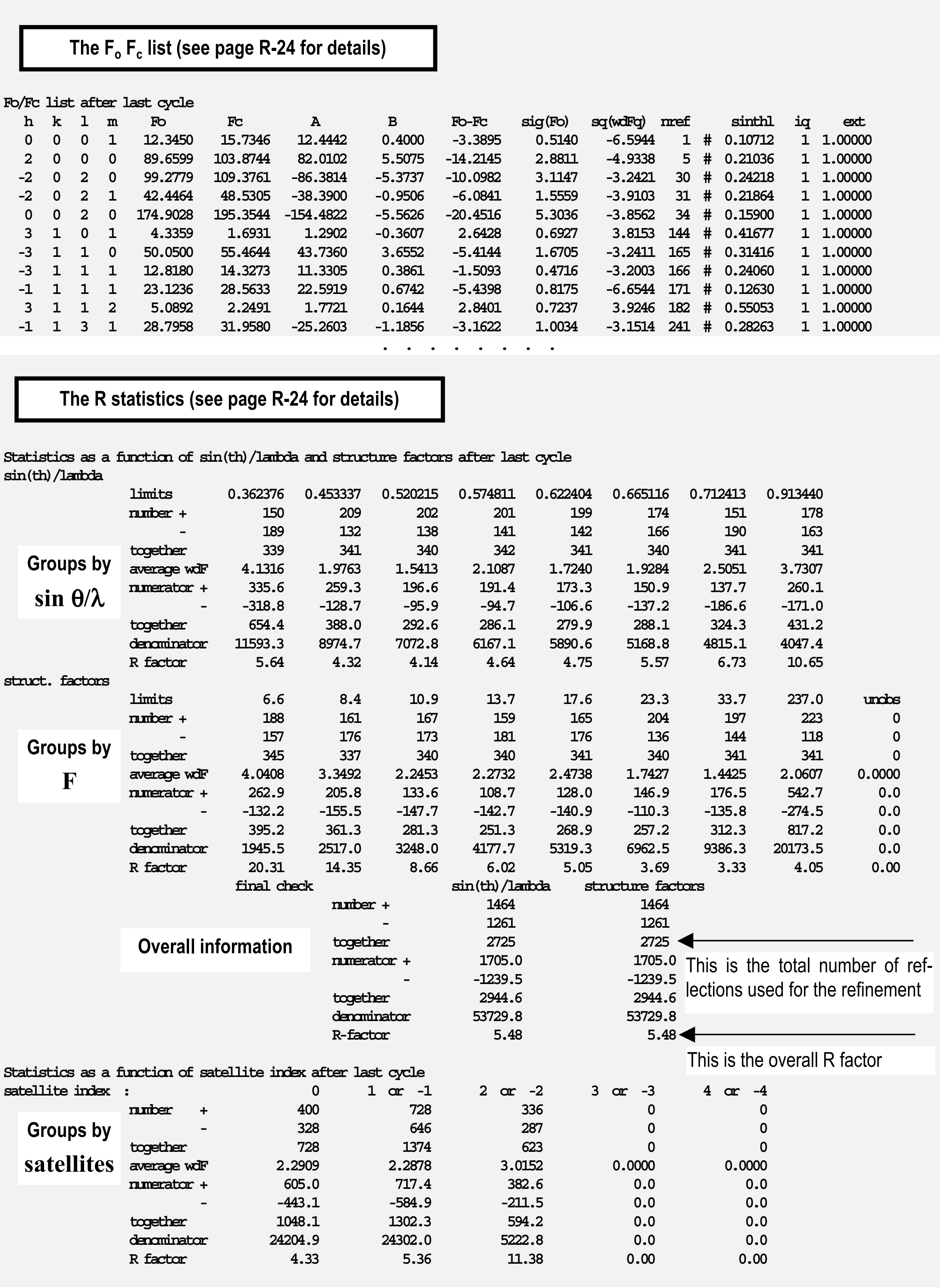
The copy of the refinement commands

Basic Crystal information

Interpretation of refinement commands

wave definitions

Figure 142 *The refinement listing (Continued)*



The F_o F_c list (see page R-24 for details)

```
Fo/Fc list after last cycle
  h   k   l   m       Fo         Fc         A          B        Fo-Fc    sig(Fo)  sq(wdFq)  nref      sinthl   iq     ext
  0   0   0   1    12.3450    15.7346    12.4442     0.4000    -3.3895    0.5140   -6.5944    1  #  0.10712   1  1.00000
  2   0   0   0    89.6599   103.8744    82.0102     5.5075   -14.2145    2.8811   -4.9338    5  #  0.21036   1  1.00000
 -2   0   2   0    99.2779   109.3761   -86.3814    -5.3737   -10.0982    3.1147   -3.2421   30  #  0.24218   1  1.00000
 -2   0   2   1    42.4464    48.5305   -38.3900    -0.9506    -6.0841    1.5559   -3.9103   31  #  0.21864   1  1.00000
  0   0   2   0   174.9028   195.3544  -154.4822    -5.5626   -20.4516    5.3036   -3.8562   34  #  0.15900   1  1.00000
  3   1   0   1     4.3359     1.6931     1.2902    -0.3607     2.6428    0.6927    3.8153  144  #  0.41677   1  1.00000
 -3   1   1   0    50.0500    55.4644    43.7360     3.6552    -5.4144    1.6705   -3.2411  165  #  0.31416   1  1.00000
 -3   1   1   1    12.8180    14.3273    11.3305     0.3861    -1.5093    0.4716   -3.2003  166  #  0.24060   1  1.00000
 -1   1   1   1    23.1236    28.5633    22.5919     0.6742    -5.4398    0.8175   -6.6544  171  #  0.12630   1  1.00000
  3   1   1   2     5.0892     2.2491     1.7721     0.1644     2.8401    0.7237    3.9246  182  #  0.55053   1  1.00000
 -1   1   3   1    28.7958    31.9580   -25.2603    -1.1856    -3.1622    1.0034   -3.1514  241  #  0.28263   1  1.00000
```

. . . .

The R statistics (see page R-24 for details)

```
Statistics as a function of sin(th)/lambda and structure factors after last cycle
sin(th)/lambda
                limits        0.362376  0.453337  0.520215  0.574811  0.622404  0.665116  0.712413  0.913440
                number +           150       209       202       201       199       174       151       178
                       -           189       132       138       141       142       166       190       163
                together           339       341       340       342       341       340       341       341
                average wdF     4.1316    1.9763    1.5413    2.1087    1.7240    1.9284    2.5051    3.7307
                numerator +      335.6     259.3     196.6     191.4     173.3     150.9     137.7     260.1
                          -     -318.8    -128.7     -95.9     -94.7    -106.6    -137.2    -186.6    -171.0
                together         654.4     388.0     292.6     286.1     279.9     288.1     324.3     431.2
                denominator    11593.3    8974.7    7072.8    6167.1    5890.6    5168.8    4815.1    4047.4
                R factor          5.64      4.32      4.14      4.64      4.75      5.57      6.73     10.65
struct. factors
                limits             6.6       8.4      10.9      13.7      17.6      23.3      33.7     237.0     unobs
                number +           188       161       167       159       165       204       197       223         0
                       -           157       176       173       181       176       136       144       118         0
                together           345       337       340       340       341       340       341       341         0
                average wdF     4.0408    3.3492    2.2453    2.2732    2.4738    1.7427    1.4425    2.0607    0.0000
                numerator +      262.9     205.8     133.6     108.7     128.0     146.9     176.5     542.7       0.0
                          -     -132.2    -155.5    -147.7    -142.7    -140.9    -110.3    -135.8    -274.5       0.0
                together         395.2     361.3     281.3     251.3     268.9     257.2     312.3     817.2       0.0
                denominator     1945.5    2517.0    3248.0    4177.7    5319.3    6962.5    9386.3   20173.5       0.0
                R factor         20.31     14.35      8.66      6.02      5.05      3.69      3.33      4.05      0.00
                         final check               sin(th)/lambda    structure factors
                                  number +                1464              1464
                                         -                1261              1261
                                  together                2725              2725
                                  numerator +           1705.0            1705.0
                                            -          -1239.5           -1239.5
                                  together              2944.6            2944.6
                                  denominator          53729.8           53729.8
                                  R-factor                5.48              5.48

Statistics as a function of satellite index after last cycle
satellite index  :                    0      1  or  -1      2  or  -2      3  or  -3      4  or  -4
                number     +        400            728            336              0              0
                           -        328            646            287              0              0
                together            728           1374            623              0              0
                average wdF      2.2909         2.2878         3.0152         0.0000         0.0000
                numerator +       605.0          717.4          382.6            0.0            0.0
                          -      -443.1         -584.9         -211.5            0.0            0.0
                together         1048.1         1302.3          594.2            0.0            0.0
                denominator     24204.9        24302.0         5222.8            0.0            0.0
                R factor           4.33           5.36          11.38           0.00           0.00
```

Groups by sin θ/λ

Groups by F

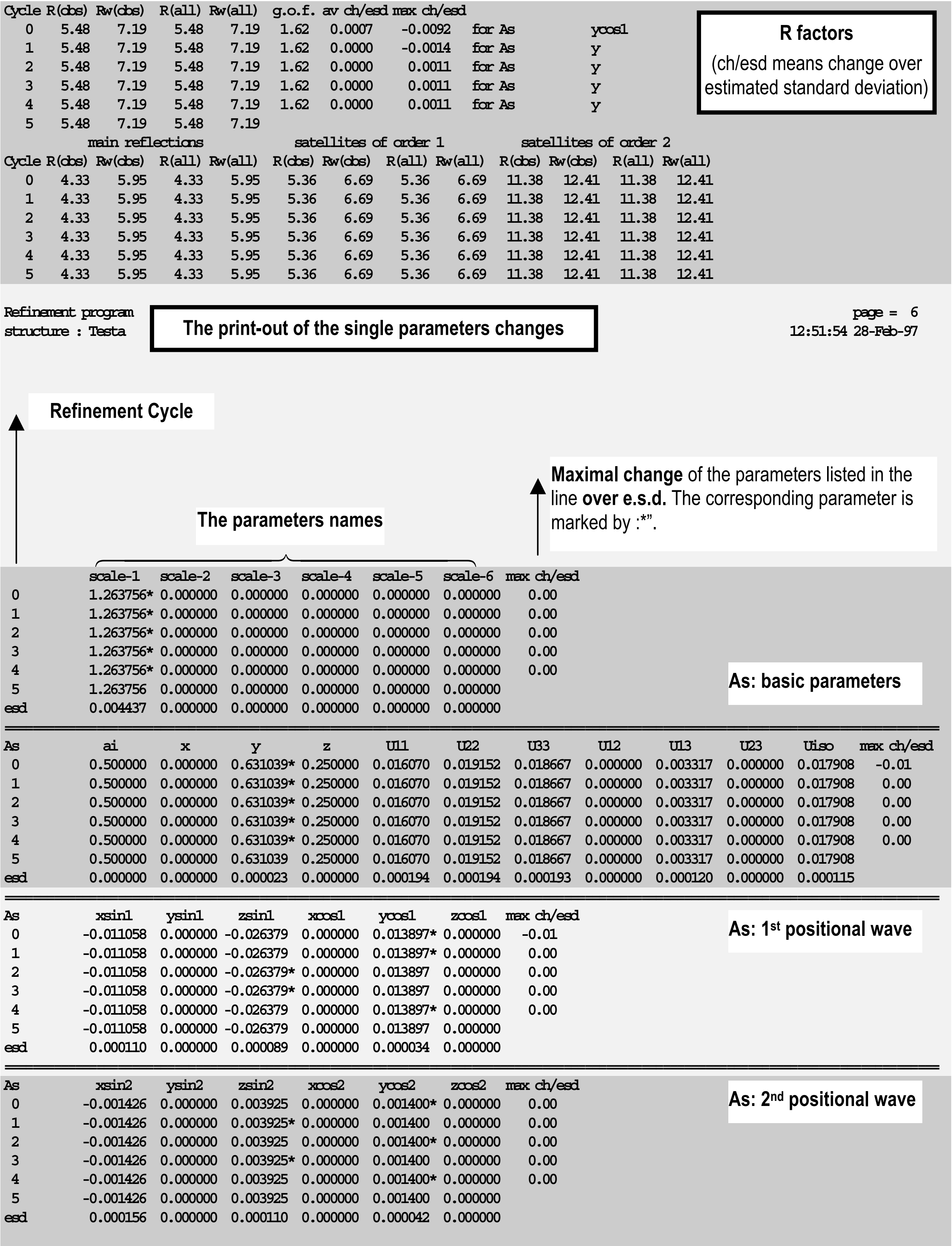
Overall information

This is the total number of reflections used for the refinement

This is the overall R factor

Groups by satellites

Figure 142 *The refinement listing (Continued)*



```
Cycle R(obs) Rw(obs)  R(all) Rw(all)  g.o.f. av ch/esd max ch/esd
  0   5.48    7.19    5.48    7.19   1.62   0.0007    -0.0092   for As          ycos1
  1   5.48    7.19    5.48    7.19   1.62   0.0000    -0.0014   for As          y
  2   5.48    7.19    5.48    7.19   1.62   0.0000     0.0011   for As          y
  3   5.48    7.19    5.48    7.19   1.62   0.0000     0.0011   for As          y
  4   5.48    7.19    5.48    7.19   1.62   0.0000     0.0011   for As          y
  5   5.48    7.19    5.48    7.19
           main reflections             satellites of order 1           satellites of order 2
Cycle R(obs) Rw(obs)  R(all) Rw(all)  R(obs) Rw(obs)  R(all) Rw(all)  R(obs) Rw(obs)  R(all) Rw(all)
  0   4.33    5.95    4.33    5.95     5.36    6.69    5.36    6.69   11.38   12.41   11.38   12.41
  1   4.33    5.95    4.33    5.95     5.36    6.69    5.36    6.69   11.38   12.41   11.38   12.41
  2   4.33    5.95    4.33    5.95     5.36    6.69    5.36    6.69   11.38   12.41   11.38   12.41
  3   4.33    5.95    4.33    5.95     5.36    6.69    5.36    6.69   11.38   12.41   11.38   12.41
  4   4.33    5.95    4.33    5.95     5.36    6.69    5.36    6.69   11.38   12.41   11.38   12.41
  5   4.33    5.95    4.33    5.95     5.36    6.69    5.36    6.69   11.38   12.41   11.38   12.41
```

R factors

(ch/esd means change over estimated standard deviation)

```
Refinement program                                                                     page =  6
structure : Testa                                                                 12:51:54 28-Feb-97
```

The print-out of the single parameters changes

Refinement Cycle

Maximal change of the parameters listed in the line **over e.s.d.** The corresponding parameter is marked by :*".

The parameters names

```
           scale-1   scale-2   scale-3   scale-4   scale-5   scale-6  max ch/esd
 0         1.263756* 0.000000  0.000000  0.000000  0.000000  0.000000    0.00
 1         1.263756* 0.000000  0.000000  0.000000  0.000000  0.000000    0.00
 2         1.263756* 0.000000  0.000000  0.000000  0.000000  0.000000    0.00
 3         1.263756* 0.000000  0.000000  0.000000  0.000000  0.000000    0.00
 4         1.263756* 0.000000  0.000000  0.000000  0.000000  0.000000    0.00
 5         1.263756  0.000000  0.000000  0.000000  0.000000  0.000000
esd        0.004437  0.000000  0.000000  0.000000  0.000000  0.000000
=====================================================================================================================
As            ai        x         y         z        U11       U22       U33       U12       U13       U23       Uiso    max ch/esd
 0         0.500000  0.000000  0.631039* 0.250000  0.016070  0.019152  0.018667  0.000000  0.003317  0.000000  0.017908   -0.01
 1         0.500000  0.000000  0.631039* 0.250000  0.016070  0.019152  0.018667  0.000000  0.003317  0.000000  0.017908    0.00
 2         0.500000  0.000000  0.631039* 0.250000  0.016070  0.019152  0.018667  0.000000  0.003317  0.000000  0.017908    0.00
 3         0.500000  0.000000  0.631039* 0.250000  0.016070  0.019152  0.018667  0.000000  0.003317  0.000000  0.017908    0.00
 4         0.500000  0.000000  0.631039* 0.250000  0.016070  0.019152  0.018667  0.000000  0.003317  0.000000  0.017908    0.00
 5         0.500000  0.000000  0.631039  0.250000  0.016070  0.019152  0.018667  0.000000  0.003317  0.000000  0.017908
esd        0.000000  0.000000  0.000023  0.000000  0.000194  0.000194  0.000193  0.000000  0.000120  0.000000  0.000115
=====================================================================================================================
As          xsin1     ysin1     zsin1     xcos1     ycos1     zcos1   max ch/esd
 0        -0.011058  0.000000 -0.026379  0.000000  0.013897* 0.000000   -0.01
 1        -0.011058  0.000000 -0.026379  0.000000  0.013897* 0.000000    0.00
 2        -0.011058  0.000000 -0.026379* 0.000000  0.013897  0.000000    0.00
 3        -0.011058  0.000000 -0.026379* 0.000000  0.013897  0.000000    0.00
 4        -0.011058  0.000000 -0.026379  0.000000  0.013897* 0.000000    0.00
 5        -0.011058  0.000000 -0.026379  0.000000  0.013897  0.000000
esd        0.000110  0.000000  0.000089  0.000000  0.000034  0.000000
=====================================================================================================================
As          xsin2     ysin2     zsin2     xcos2     ycos2     zcos2   max ch/esd
 0        -0.001426  0.000000  0.003925  0.000000  0.001400* 0.000000    0.00
 1        -0.001426  0.000000  0.003925* 0.000000  0.001400  0.000000    0.00
 2        -0.001426  0.000000  0.003925  0.000000  0.001400* 0.000000    0.00
 3        -0.001426  0.000000  0.003925* 0.000000  0.001400  0.000000    0.00
 4        -0.001426  0.000000  0.003925  0.000000  0.001400* 0.000000    0.00
 5        -0.001426  0.000000  0.003925  0.000000  0.001400  0.000000
esd        0.000156  0.000000  0.000110  0.000000  0.000042  0.000000
```

As: basic parameters

As: 1st positional wave

As: 2nd positional wave

Figure 142 *The refinement listing (Continued)*

```
===================================================================================================
As          U11sin1   U22sin1   U33sin1   U12sin1   U13sin1   U23sin1  max ch/esd
 0          0.000000  0.000000  0.000000 -0.001399* 0.000000  0.001612     0.00
 1          0.000000  0.000000  0.000000 -0.001399  0.000000  0.001612*    0.00
 2          0.000000  0.000000  0.000000 -0.001399  0.000000  0.001612*    0.00
 3          0.000000  0.000000  0.000000 -0.001399* 0.000000  0.001612     0.00
 4          0.000000  0.000000  0.000000 -0.001399  0.000000  0.001612*    0.00
 5          0.000000  0.000000  0.000000 -0.001399  0.000000  0.001612
esd         0.000000  0.000000  0.000000  0.000172  0.000000  0.000175
```

As: 1st temperature wave

```
As          U11cos1   U22cos1   U33cos1   U12cos1   U13cos1   U23cos1  max ch/esd
 0          0.001689 -0.000003  0.000386  0.000000 -0.000072* 0.000000     0.00
 1          0.001689 -0.000003* 0.000386  0.000000 -0.000072  0.000000     0.00
 2          0.001689 -0.000003* 0.000386  0.000000 -0.000072  0.000000     0.00
 3          0.001689 -0.000003* 0.000386  0.000000 -0.000072  0.000000     0.00
 4          0.001689 -0.000003* 0.000386  0.000000 -0.000072  0.000000     0.00
 5          0.001689 -0.000003  0.000386  0.000000 -0.000072  0.000000
esd         0.000206  0.000238  0.000215  0.000000  0.000164  0.000000
```

As: 1st temperature wave

```
===================================================================================================
As          U11sin2   U22sin2   U33sin2   U12sin2   U13sin2   U23sin2  max ch/esd
 0          0.000000  0.000000  0.000000  0.000264* 0.000000  0.001977     0.00
 1          0.000000  0.000000  0.000000  0.000264* 0.000000  0.001977     0.00
 2          0.000000  0.000000  0.000000  0.000264  0.000000  0.001977*    0.00
 3          0.000000  0.000000  0.000000  0.000264  0.000000  0.001977*    0.00
 4          0.000000  0.000000  0.000000  0.000264  0.000000  0.001977*    0.00
 5          0.000000  0.000000  0.000000  0.000264  0.000000  0.001977
esd         0.000000  0.000000  0.000000  0.000245  0.000000  0.000217
```

As: 2nd temperature wave

```
===================================================================================================
Refinement program                                                                      page =  7
structure : Testa                                                                12:51:54 28-Feb-97

As          U11cos2   U22cos2   U33cos2   U12cos2   U13cos2   U23cos2   phason   max ch/esd
 0          0.000283 -0.000805 -0.000181  0.000000  0.000856* 0.000000  0.000000     0.00
 1          0.000283 -0.000805 -0.000181* 0.000000  0.000856  0.000000  0.000000     0.00
 2          0.000283 -0.000805*-0.000181  0.000000  0.000856  0.000000  0.000000     0.00
 3          0.000283 -0.000805 -0.000181  0.000000  0.000856* 0.000000  0.000000     0.00
 4          0.000283*-0.000805 -0.000181  0.000000  0.000856  0.000000  0.000000     0.00
 5          0.000283 -0.000805 -0.000181  0.000000  0.000856  0.000000  0.000000
esd         0.000343  0.000272  0.000294  0.000000  0.000236  0.000000  0.000000
===================================================================================================
```

As: 2nd temperature wave

. . . .

```
O3          U11cos2   U22cos2   U33cos2   U12cos2   U13cos2   U23cos2   phason   max ch/esd
 0         -0.003972 -0.014366 -0.017415  0.013567 -0.014126  0.018702* 0.000000     0.00
 1         -0.003972 -0.014366*-0.017415  0.013567 -0.014126  0.018703  0.000000     0.00
 2         -0.003972 -0.014366 -0.017415  0.013567 -0.014126  0.018702* 0.000000     0.00
 3         -0.003972 -0.014366 -0.017415* 0.013567 -0.014126  0.018702  0.000000     0.00
 4         -0.003972 -0.014366 -0.017415* 0.013567 -0.014126  0.018702  0.000000     0.00
 5         -0.003972 -0.014366 -0.017415  0.013566 -0.014126  0.018702  0.000000
esd         0.002550  0.002628  0.002897  0.002092  0.002125  0.002218  0.000000
===================================================================================================


There were no correlations larger than  0.900 in last refinement cycle
Program started at 12:51:58 ended   at 12:53:59 cpu time :      1.47
```

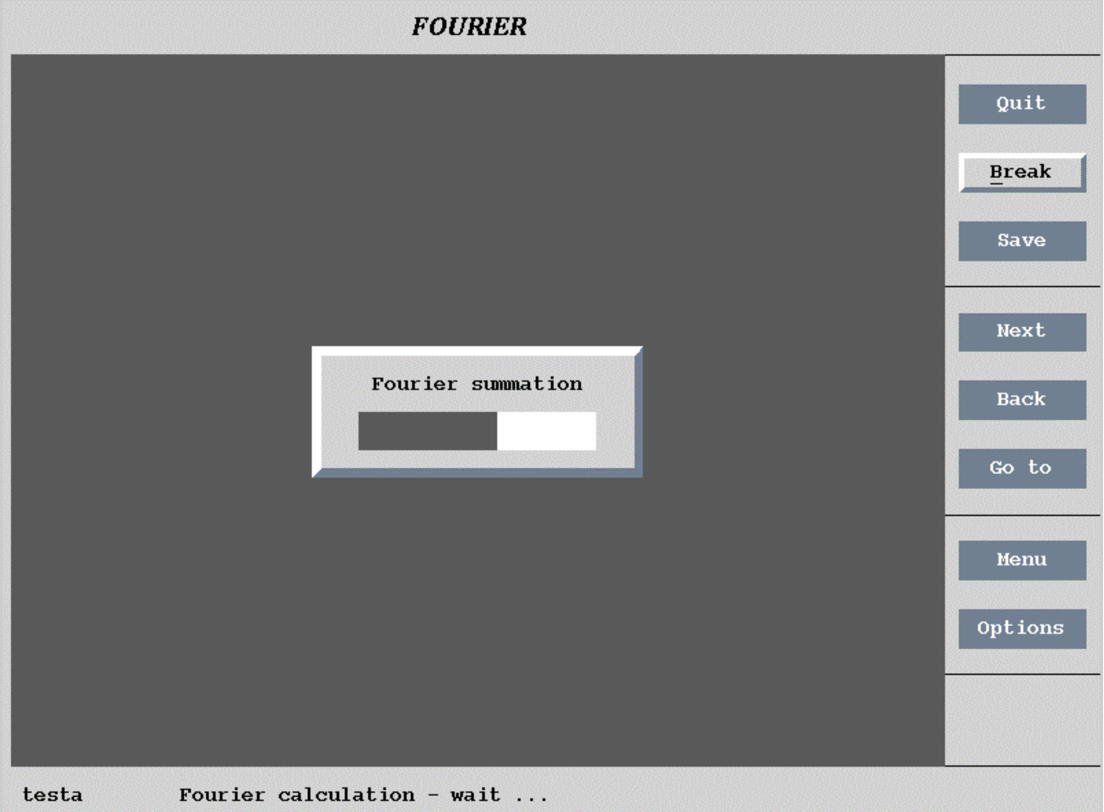
Final information

List of the correlations (see page R-26 for details)

3.4 Program *FOURIER*

Input: `m40,m50,m80, [m81]`
Output: `m81,m47,m48,fou`
M50 control keywords: the section between the keywords `fourier` and `end`

Figure 143 *The basic window of FOURIER*



```
testa         Fourier calculation - wait ...
```

3.4.1 Description of *FOURIER*

FOURIER performs a Fourier synthesis of coefficients defined at points of the (3+d) dimensional reciprocal lattice. These coefficients are complex numbers usually expressed as amplitudes and phases. The resulting map is a (3+d) dimensional periodic function in the direct space and its character depends on the used coefficients (Patterson, Fourier based on Fobs, etc).

The maps are stored as two-dimensional sections through the chosen (3+d) dimensional parallelepiped with edges along (3+d) basic vectors $\mathbf{A}_1, \mathbf{A}_2, \ldots, \mathbf{A}_{3+d}$. Its volume is defined by the scope parameters (see §0, page 273). The map orientation makes possible to define two-dimensional sections and the order in which they are stored. The two basic vectors defining the section will be called "section basic vectors" and the remaining ones "storing basic vectors". The corresponding coordinates will be called in analogy "section coordinates" and "storing coordinates". Each section is a function of 1+d storing coordinates. This choice does not have substantial meaning for regular structures but it plays important role in interpretation during the peak search and map visualisation.

FOURIER is usually used together with *CONTOUR*, which visualises the Fourier maps. Some examples of the *FOURIER* usage are therefore showed also in the chapter containing the description of the *CONTOUR* program (see §4.1,page 291).

The FOURIER run step by step

- *FOURIER* reads the input files `m40`, `m50` and `m80`. The *Fourier reflection file* `m80` contains the phase information so it is therefore necessary for every type of the map except the Patterson[1] map. It must be created[2] by *REFINE* before running *FOURIER*.

- Then the program interprets the control commands prepared by the user *via* the SetCommands tool. The two dimensional sections by 3+d dimensional map are stored in the `m81` file in the order defined by the orientation of the map.

- The calculated maps are searched for local minima and maxima. In the case of the modulated structure *FOURIER* tries to assign modulation waves to the maxima. The maxima (together with the modulation parameters) and minima are stored in the `m48` and `m47` files, respectively. Both `m47` and `m48` files are readable by *EDITM40* and *DIST* programs.

- *FOURIER* writes the listing `jobname.fou.`

---

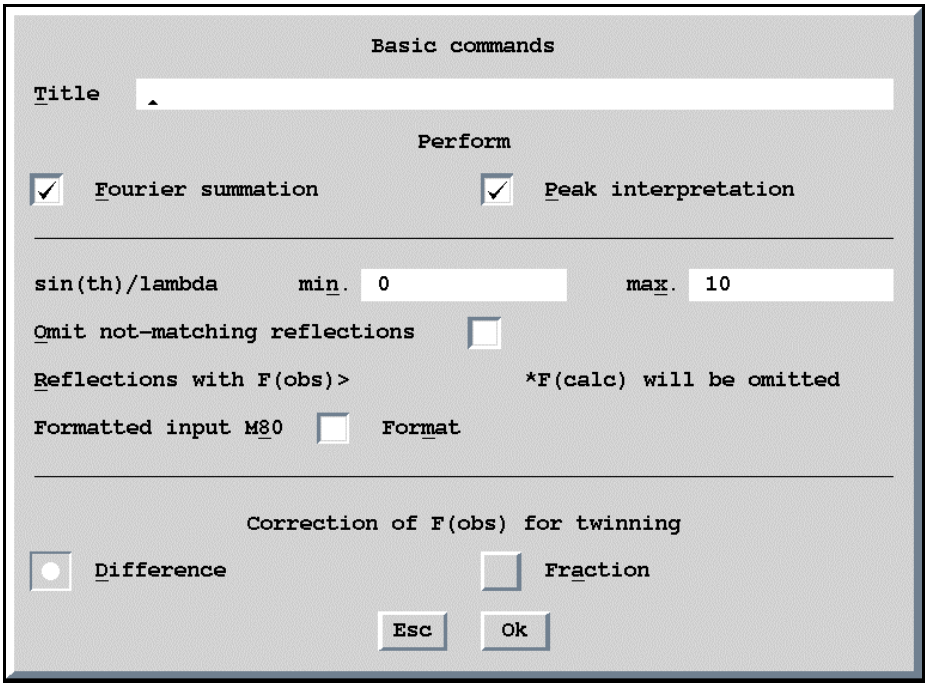
[1] In former versions of *JANA98* it was necessary to run zero cycles refinement with empty `m40` before calculation of the Patterson map. In this version it is no longer needed.

[2] *REFINE* creates `m80` during calculation of the last refinement cycle. Zero cycles is sufficient for the calculation of `m80`. If *REFINE* is interrupted by Break button the `m80` is not created.

3.4.2 Basic commands

The `Basic commands` form contains the input and output information . If the `Fourier summation` checkbox is ticked (and we don't want the Patterson type map) the input for *FOURIER* is `m80` with the phasing information. If the `Fourier summation` checkbox is free but the `Peak interpretation` checkbox is ticked *FOURIER* interprets already existing maps and the input is `m81`. The peak interpretation is usually fast and the user doesn't need to change the default setting, which turns on both the Fourier summation and the peak interpretation.

Figure 144 *FOURIER: The basic commands*



Basic commands

Title

Perform

☑ Fourier summation ☑ Peak interpretation

sin(th)/lambda min. 0 max. 10

Omit not-matching reflections ☐

Reflections with F(obs)> *F(calc) will be omitted

Formatted input M80 ☐ Format

Correction of F(obs) for twinning

● Difference ○ Fraction

The `m80` created by *REFINE* is a binary file. *FOURIER* can also read `m80` in the user defined ASCII form. This is meant like a support for those needing to interpret the results from electron microscopy or direct methods with *FOURIER*.

If the case of the composite structure the user selects which subsystem will be used for the Fourier calculation. All indices are then internally transformed to the chosen subsystem.

In the case of a twin the Fourier map is calculated for the first twin domain and therefore the observed structure factors must be corrected. *FOURIER* offers two method for doing it:

The difference method:

$$F_{obs} = \sqrt{I_{obs} - \sum_{i=2,n} v_i F_{c,i}^2}\,, \qquad E64$$

where n is number of twin domains and v_i is the volume fraction of the i^{th} domain.

The fraction method:

$$F_{obs}^{corr} = F_{obs} \frac{F_{calc1}}{F_{calc}} \qquad E65$$

There is no general way, however, to correct completely for twinning. According to our experience the first method has advantage in stage when the Fourier program is used to localise new atoms and the second one should be preferred in the final stages of the structure determination.

In the following table there is the list of commands that may be stored in `m50` after completing the SetCommands procedure. Only the commands different from the default setting are explicitly listed in `m50`.

Table 23 *The Basic Commands of FOURIER in the* `m50` *file*

Name	Syntax and Description
cutting	**cutting** `number` **Default value**: no cutting The reflections with $\lvert F_{obs}\rvert > \lvert F_{calc}\rvert$ * `number` will not be used for the calculation.
calc	**calc** `flag` **Default value**: **calc** 1 If the `flag` is set to zero, *FOURIER* doesn't calculate the map assuming that it was already calculated. It can be used for searching the map for the local extremes without its calculation.
method	**method** `flag` **Default value**: **method** 0 Selects the method for the calculation of the F_{obs} in the case of a twin. The Fourier map is calculated for the first twin domain. The observed structure factors must therefore corrected. Here are two ways how to do it: `flag` = 0 The difference method (equation *E64*, page 268). `flag` = 1 The fractional method (see equation *E65,* page 268)
peaks	**peaks** `flag` **Default value**: **peaks** 1 If the `flag` is set to zero, *FOURIER* doesn't search the map for the local extremes.
title	**title** `title_of_this_run`
snlmn	**snlmn** `min_value` **Default value**: **snlmn** 0 Sets the minimum value of $\sin\theta/\lambda$ for the acceptance of the reflections for the calculation.
snlmx	**snlmx** `max_value` **Default value**: **snlmn** 10 Sets the maximal value of $\sin\theta/\lambda$ for the acceptance of the reflections for the calculation.
subsys	**subsys** `number` **Default value**: **subsys** 1 Selects the composite subsystem for which the Fourier map will be calculated.

3.4.3 Type of the Map

The Fourier syntheses is calculated according to the following equation:

$$\sigma(\mathbf{r}) = \frac{1}{V}\sum_{\mathbf{H}} A(\mathbf{H})\exp(i\varphi(\mathbf{H}))\exp(-2\pi i\mathbf{H}\cdot\mathbf{r}),$$

where

$$\mathbf{H} = \sum_{i=1}^{3+d} h_i \mathbf{a}_i^*, \qquad E66$$

$A(\mathbf{H})$ are the coefficients of the syntheses and

$\varphi(\mathbf{H})$ are the phases.

The coefficients for various types of the Fourier synthesis are summarized in Table 24. The user interface is shown in Figure 145; the corresponding control commands written to m50 are explained in Table 24.

In the case of the Patterson/Checking Patterson/difference Patterson syntheses the coefficients are mutlplied by the factor:

$$\left(\frac{\sum_{i=1}^{n} Z_i}{\sum_{i=1}^{n} f_i\ (\sin\theta/\lambda)} \right)^2 \qquad E67$$

where Z_i is the atomic number and f_i is the atom form factor of the i-th atom,

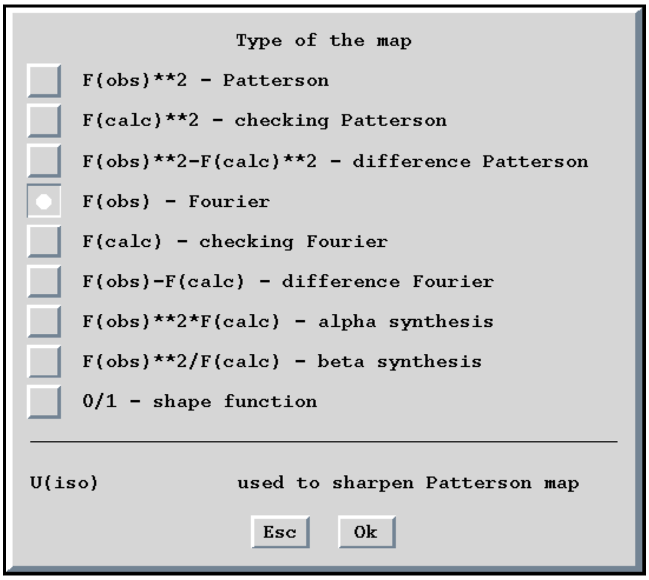
to sharpen the map. Note that it has no effect for neutron data where the atom form factor is constatnt. The additional sharpening by the overall temperature factor:

$$\exp\left(8\pi^2 U_{iso} \frac{\sin^2\theta}{\lambda^2} \right) \qquad or \qquad \exp\left(B_{iso} \frac{\sin^2\theta}{\lambda^2} \right) \qquad E68$$

is optional.

This procedure helps to reduce the overlap caused by peak broadening.

Figure 145 *FOURIER: The types of the maps*



Type of the map

☐ F(obs)**2 - Patterson
☐ F(calc)**2 - checking Patterson
☐ F(obs)**2-F(calc)**2 - difference Patterson
● F(obs) - Fourier
☐ F(calc) - checking Fourier
☐ F(obs)-F(calc) - difference Fourier
☐ F(obs)**2*F(calc) - alpha synthesis
☐ F(obs)**2/F(calc) - beta synthesis
☐ 0/1 - shape function

U(iso) used to sharpen Patterson map

Table 24 *The coefficients of the Fourier synthesis types available in FOURIER.*

Synthesis type	$A(\mathbf{H})$	$\varphi(\mathbf{H})$
Patterson	$F^2_{obs}(\mathbf{H})$	0
Checking Patterson	$F^2_{calc}(\mathbf{H})$	0
Difference Patterson	$F^2_{obs}(\mathbf{H}) - F^2_{calc}(\mathbf{H})$	0
Fourier	$\lvert F_{obs}(\mathbf{H})\rvert$	$\varphi_{calc}(\mathbf{H})$
Checking Fourier	$\lvert F_{calc}(\mathbf{H})\rvert$	$\varphi_{calc}(\mathbf{H})$
Difference Fourier	$\lVert F_{obs}(\mathbf{H})\rvert - \lvert F_{calc}(\mathbf{H})\rVert$	$\varphi_{calc}(\mathbf{H})$
Alpha synthesis	$F^2_{obs}(\mathbf{H})\lvert F_{calc}\rvert$	$\varphi_{calc}(\mathbf{H})$
Beta synthesis	$F^2_{obs}(\mathbf{H})/\lvert F_{calc}\rvert$	$\varphi_{calc}(\mathbf{H})$
Shape function	1 for all collected reflections	0

Table 25 *The Maptype Commands of FOURIER in the `m50` File*

Name	Syntax and Description
maptype	**maptype** `flag` **Default value**: **maptype** 4 Type of the Fourier syntheses `flag = 1` Patterson map (F^2_{obs} coefficients) `flag = 2` Checking Patterson (F^2_{calc} coefficients) `flag = 3` Difference Patterson (F^2_{obs} - F^2_{calc} coefficients) `flag = 4` Fourier map (F_{obs} coefficients) `flag = 5` Checking Fourier (F_{calc} coefficients) `flag = 6` Difference Fourier (F_{obs} - F_{calc} coefficients) `flag = 7` α-synthesis (F^2_{obs} F_{calc} coefficients) `flag = 8` β-synthesis (F^2_{obs}/F_{calc} coefficients) `flag = 9` Shape functions (The coefficients = 1)
uiso	**uiso** `number` **Default value**: **uiso** 0 The additional temperature factor in U form used for sharpening of the Patterson syntheses
biso	**biso** `number` **Default value**: **biso** 0 The additional temperature factor in B form used for sharpening of the Patterson syntheses

3.4.4 Scope and Orientation

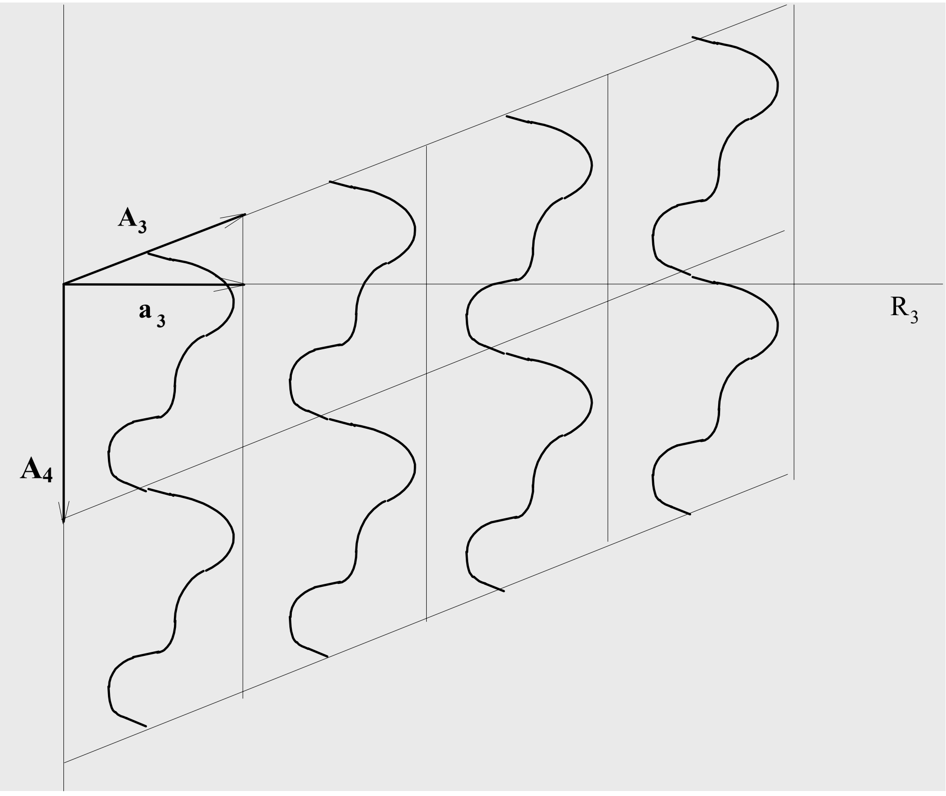
The section types in JANA98

This chapter is important only for modulated structures.

JANA98 distinguish three basic types of maps depending on way how the section basic vectors has been chosen. These section types allow visualising different aspects of the modulated structure. Two of them can be calculated directly by the FOURIER program and the remaining one can be extrapolated from the previous in the CONTOUR program. They can be understood from the following picture of the 3+d dimensional cell. Here $\mathbf{A}_3$ represents the first three (external) basic vectors of the 4-dimensional superspace, $\mathbf{A}_4$ are the internal basic vectors, $\mathbf{R}_3$ represents the real space.

In the following explanation we shall speak about 4-dimensional Fourier synthesis and the internal coordinate will be sometimes denoted as x4. The work with 5- and 6-dimensional maps is analogous.

Figure 146 *The Four Dimensional Elementary Cell*



- ***The $A_3 - A_4$ maps***

One of the section basic vector is from $\mathbf{A_3}$ and the second one is $\mathbf{A_4}$. Such maps can show the modulation of a selected atom in particular direction directly in one section.

- ***The $A_3 - A_3$ maps***

Both section basic vectors are from $\mathbf{A_3}$. The two dimensional sections of the map can show coordinations of atoms and their changes as a function of remaining two vectors including $\mathbf{A_4}$. The sections cannot show directly modulations as they are periodic. This is due the fact that they were calculated for constant internal coordinates. Moreover they are also deformed as $\mathbf{A}_1 \neq \mathbf{a}_1$. The real coordinations of atoms and their changes caused by modulation can be visualised by the sections of the real space R_3.

- ***The R_3 - R_3 maps***

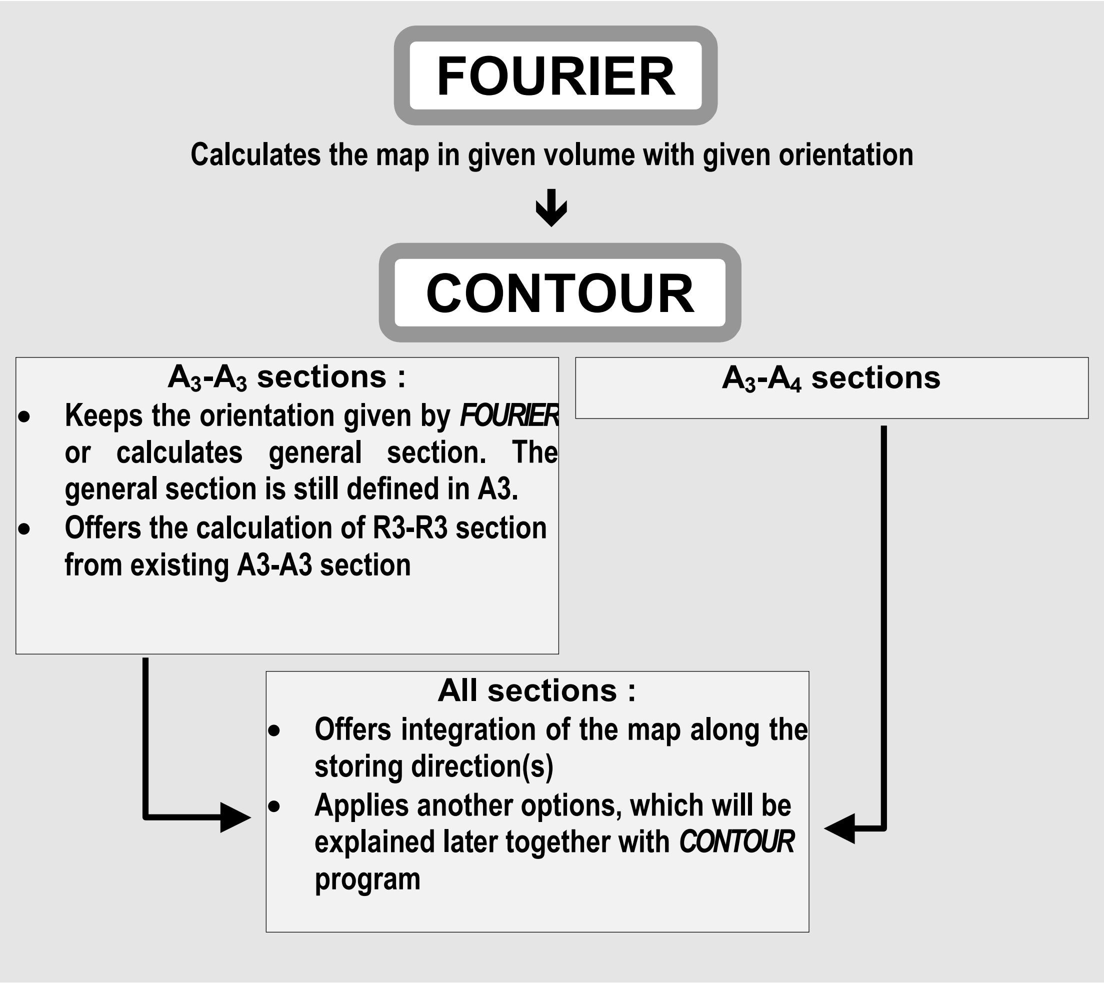
In order to have a picture without deformation the section through $\mathbf{R}_3$ has to be performed. These maps are no longer periodic and they cannot be calculated by standard Fourier summation technique. On the other hand they can be extrapolated from A_1-A_4 maps. The section then depends on the *internal coordinate t*, which is the projection of the forth coordinate to the $\mathbf{A}_4$ axis along $\mathbf{R}_3$ direction - $t = x_4 - \mathbf{q} \cdot \mathbf{r}$. Therefore this type of maps is also called *t-maps*.

There are two basic way how to use such maps. First we can calculate a section covering several cells and calculated for one selected *t*. Such map can shows directly and without deformation part of the modulated structure. The second possibility is to calculate these maps for more *t* values from the interval $\langle 0{,}1 \rangle$. Such maps will show behaviour of the selected part as a function of *t*. The main difference to the previous A_3 - A_3 maps is that these section are no more deformed, i.e. they show real geometry in 3d space, at the cost of losing the periodicity.

Communication between FOURIER and CONTOUR

If *FOURIER* is used only to localise maxima and estimate new modulation parameters the *CONTOUR* program is not needed. On other hand the *CONTOUR* program allows to visualise the sections of the map and draw conclusions necessary to find more details concerning different aspects of the modulation. The following scheme shows the sharing of the work between *FOURIER* and *CONTOUR*.

Figure 147 *Communication between FOURIER and CONTOUR*



Calculates the map in given volume with given orientation

A_3-A_3 sections :

- Keeps the orientation given by *FOURIER* or calculates general section. The general section is still defined in A3.
- Offers the calculation of R3-R3 section from existing A3-A3 section

All sections :

- Offers integration of the map along the storing direction(s)
- Applies another options, which will be explained later together with *CONTOUR* program

The Scope Form

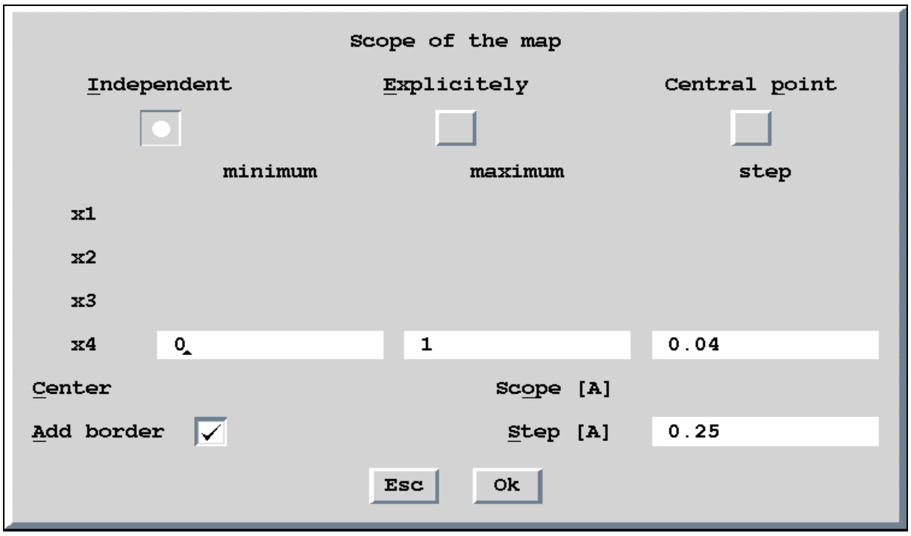
With the Scope form the user can define the following parameters:

- Volume of the map in coordinates $\mathbf{A}_3$
- The interval and the step for the internal coordinate
- The step for the calculation of the map

The Scope of the map should be defined with respect to the desired orientation of the map and later operations in *CONTOUR*.

The ***independent volume*** is optimal if we calculate the map for the reason of the peak search or if we will later user *CONTOUR* for plotting the sections through the whole cell. If the map is calculated in the independent volume Contour has enough information for calculating general sections of arbitrary area and width.

Figure 148 *The Fourier syntheses calculated in the independent volume*



Scope of the map

● Independent ☐ Explicitely ☐ Central point

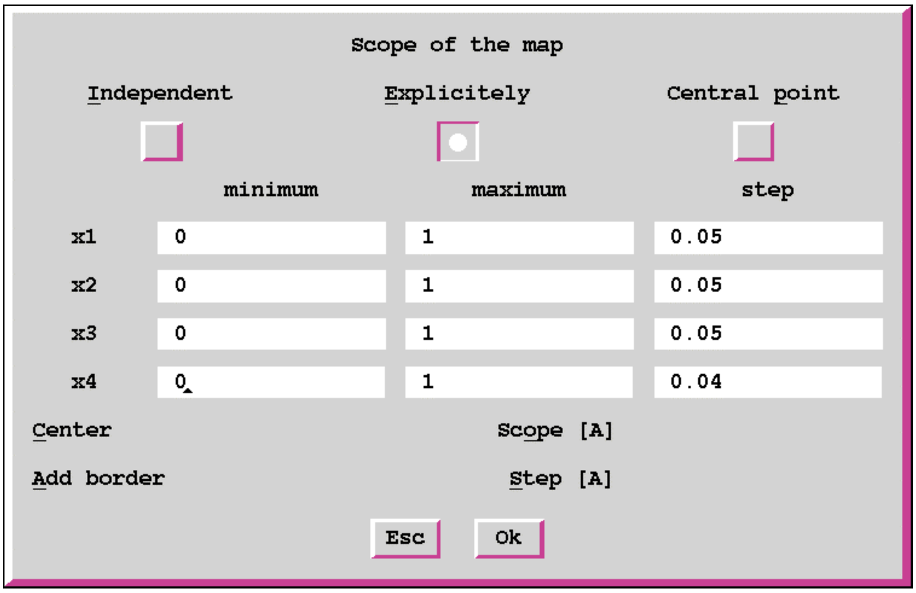
	minimum	maximum	step
x1			
x2			
x3			
x4	0	1	0.04

Add border ☑

Step [A] 0.25

The ***explicit setting*** of the map volume is useful in the cases when some part of the map has to be examined in details. It usually requires small steps and the calculation of the whole independent volume could be excessively long.
Another reason for the explicit choice is if we need a section comprising more elementary cells, for instance x1 ∈ <-0.25,0.25>; x2 ∈ <-0.25,0.25>. Such section can be calculated by *CONTOUR* from the map in the independent volume like general section. If we use, however, the explicit setting of the map volume the section can be plotted directly without calculation of the general section.
The last reason for the explicit setting is if we need different steps along the single coordinates.

Figure 149 *The Fourier synthesis calculated in explicitly defined volume*



Scope of the map

Independent ○ Explicitely ● Central point ○

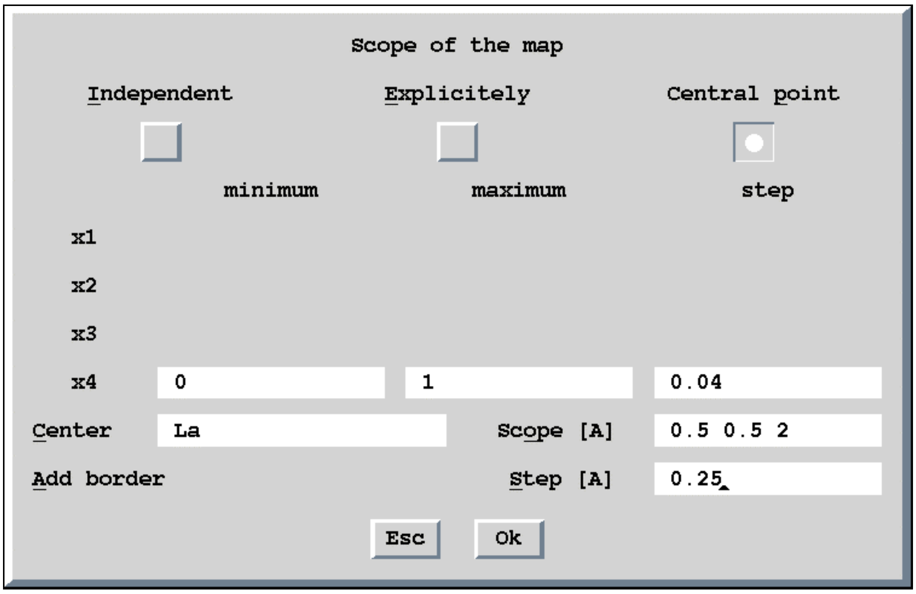
	minimum	maximum	step
x1	0	1	0.05
x2	0	1	0.05
x3	0	1	0.05
x4	0	1	0.04

Center
Scope [A]
Add border
Step [A]

The calculation of the map ***around the central point*** is usually used if we need a $\mathbf{A}_3$-$\mathbf{A}_4$ map showing the modulation of some atom. In the example below the La atom is chosen as the central point and the volume of the map is defined by ***Scope*** option along three coordinates in $\mathbf{A}_1$: x1 ∈ <x1(La)-0.5, x1(La)+0.5>; x2 ∈ <x2(La)-0.5, x2(La)+0.5>; x3 ∈ <x3(La)-2,x3(La)+2>. The section orientation will be defined later *via* the Orientation form like x3-x4. The directions along x1 and x2 will be the width of the section and the map could be integrated along these directions by *CONTOUR*.

The central point can be defined by atom name or by three coordinates. If the atom used for the definition of the central point is a part of a molecule its position should be specified according to the conventions in § 1.2.6 page 27).

Figure 150 *The Fourier synthesis calculated around a central point*



Scope of the map

Independent ○ Explicitely ○ Central point ●

	minimum	maximum	step
x1			
x2			
x3			
x4	0	1	0.04

Center La
Scope [A] 0.5 0.5 2
Add border
Step [A] 0.25

The ***length and the step of the additional coordinate*** should be also defined with respect to the orientation of the map. In the case of **A_3-A_4** maps the length setting influences if we will have one or more periods of the modulation function in the plot. The step controls the precision of the function shape. For **A_3-A_3** sections we don't need more than one period in x4 and the step controls the number and distance of the successive layers.

The ***step of the map in Å*** is the calculation step used by *FOURIER*. The fine step - for instance 0.1 Å - causes smooth contours in the plot but at the cost of long calculation and large files. The default step 0.25 Å is usually in agreement with the accuracy of the data. This means that we will not see more with finer step but it may be useful for publication reasons.

The Orientation Form

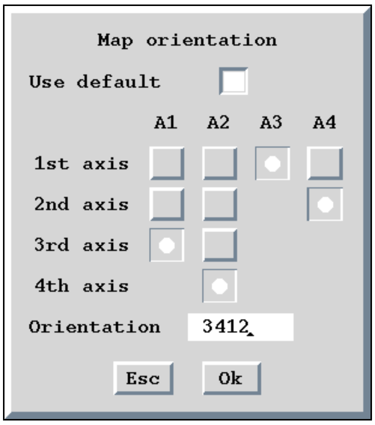
The first two axes in the Orientation form defines the section basic vectors and the remaining ones the storing basic vectors. In the CONTOUR program the map can be integrated along the storing basic vectors or the section can be plotted separately.

In the following example the section is defined by A_3 and A_4 (it is therefore the **A_3-A_4** map) and it depends on x1 and x2. The section will be stored and later plotted in the following order:

```
x3-x4(x11,x21), x3-x4(x12,x21), . . . . , x3-x4(x1n,x21),
x3-x4(x11,x22), x3-x4(x12,x22), . . . . , x3-x4(x1n,x22),
                      . . . .
x3-x4(x11,x2m), x3-x4(x12,x2m), . . . . , x3-x4(x1n,x2m),
```

where subscripts denotes the step numbers, `n` is number of steps in A_1 direction, `m` is number of steps in A_2 direction.

Figure 151 *Setting the map orientation*



Map orientation

Use default ☐

	A1	A2	A3	A4
1st axis	☐	☐	●	☐
2nd axis	☐	☐		●
3rd axis	●	☐		
4th axis		●		

Orientation 3412

Table 26 *The Scope and Orientation Commands of FOURIER in the `m50` File*

Name	Syntax and Description
addbord	**addbord** `flag` **Default value**: **addbord** 1 If `flag` is 1, one step is added to the volume of the map in each direction..
center	**center** `x1 x2 x3` **center** `atom_name` Defines the central point of the map. It is used together with the `xlim` keys and the scope key to define the volume of the map. The central point can be defined by fractional coordinates `x1`, `x2` and `x3` or by the name of some atom present in the `m40` file.
orient	**orient** `code` This key defines two dimensional sections by the electron density map and their order the `m81` file. It is also called the orientation of the map. The `code` consists of three (or more for 3+d structures) sequence numbers of the basic cell vectors. The first and the second basic vector define the section, which depends on the remaining coordinates. Example: `orient 1234` The plane of this section is defined by **a** and **b** cell vectors. The horizontal axis of the section runs along **a**. The sections are stored in `m81` file by ascending order of the *z* coordinate and x4 coordinate. See the Orientation form for more details.

Table 26 *(Continued)*

scope	**scope** `rx ry rz`
	This command is used together with the **center** key for definition of the map volume. The numbers define the size of the map along **a**, **b** and **c** axis, respectively. In the case of 3+d structures the information given by the `scope` command is extended by the `x4lim`, `x5lim` and `x6lim` commands.
	Example 1: `center As`
	`scope 2 2 1`
	`orient 123`
	The volume with the central point in the arsenic atom and the dimensions ±1Å along **a** axis, ±1Å along **b** axis and ±0.5Å along **c** axis is defined. The plane of the section is **ab**.
	Example 2: `center As`
	`scope 2 2 1 step 0.2`
	`orient 123`
	The same as the previous example but with changed calculation step.
	Example 3: `center As`
	`scope 0.5 0.5 2`
	`x4lim 0 1 0.1`
	`orient 3412`
	This example prepares the Fourier map for plotting of the z modulation function component of arsenic.

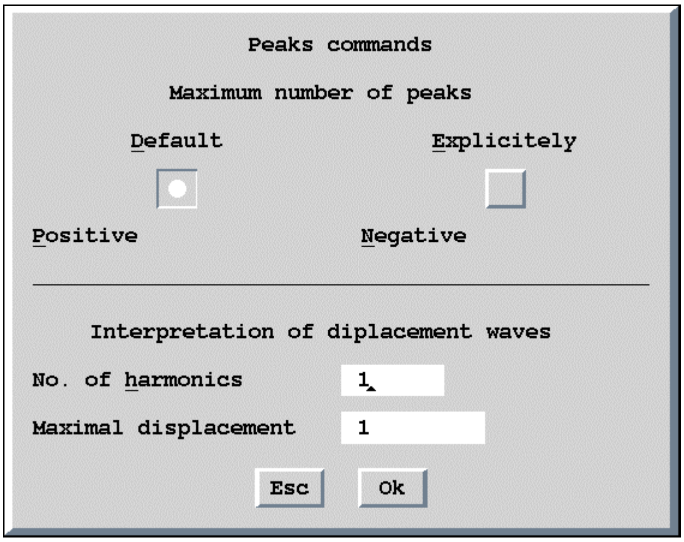
step	**step** `number` **Default value**: **step** 0.25 Sets step in Å for calculation of the maps. This step can also be changed separately for single axis by `xlim`, `ylim` etc. commands.

xlim
ylim
zlim
x1lim
x2lim
x3lim
x4lim
x5lim
x6lim
	xlim `min max step`
	The size of the map long the x axis. The analogous commands exist for **ylim**, **zlim**, **x1lim**, **x2lim**, **x3lim**, **x4lim**, **x5lim** and **x6lim**. **Xlim**, **ylim** and **zlim** mean the same like **x1lim**, **x2lim** and **x3lim**, respectively.
	Example: `xlim 0 0.5 0.1`
	`ylim 0 0.5 0.1`
	`zlim 0 0.5 0.1`
	Sets the volume of the map to one quarter of the unit cell. The step for the calculation is 0.1Å.

3.4.5 The Peaks Commands

For modulated structures the peak search is performed only if the internal basic vectors are used as last storing vectors. This means also that the fastest storing vector and both section basic vectors are the external ones. Then the program tries to assign modulation parameters to positive peaks.

The user settings for the peaks searching are entered in the Peaks Commands form.



Default ● Explicitely ☐

Positive Negative

No. of harmonics 1

Maximal displacement 1

Table 27 *The Peaks Commands of FOURIER in the* `m50` *File*

Name	Syntax and Description
positive	**positive** `number` The maximal number of positive peaks to be found in the map. The default value is `n`+5, where `n` depends on the map type[1].
negative	**negative** `number` **Default value**: **negative** 5 The maximal number of negative peaks to be found in the map.
dmax	**dmax** `number` The limit in Å for distinguishing two different maxima. The default value is 1 Å.
harmonic	**harmonic** `number` The number of harmonic waves used for the construction of the curve representing the position of the maximum. Using more than 2 harmonics is not recommended.

---

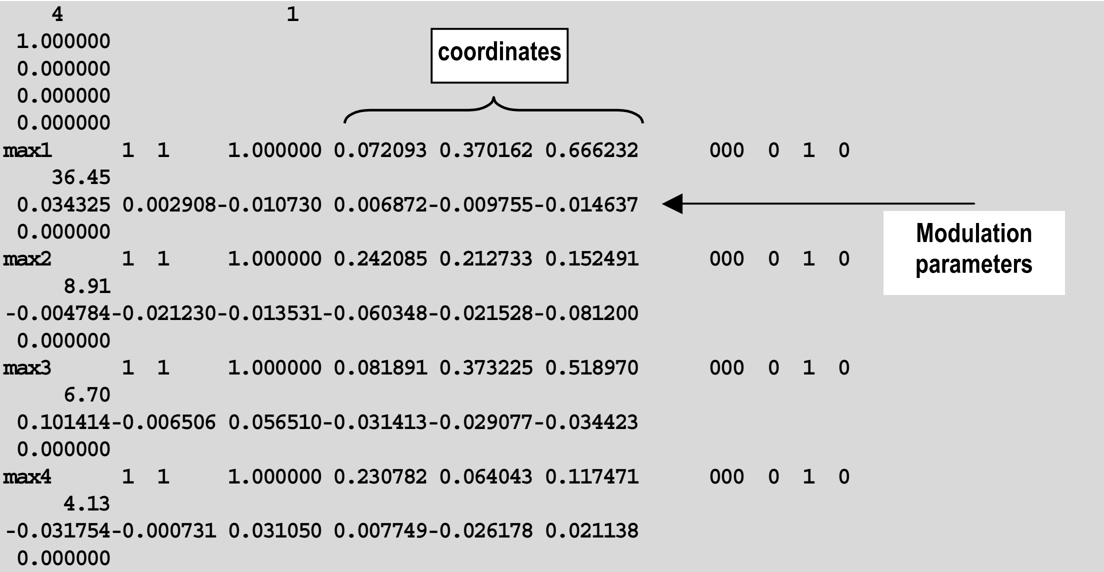
[1] In the case of the normal Fourier map (`maptype` 2) `n` is the number of atoms missing in the `m40` file. The total number of atoms which should be present in the `m40` is calculated from the chemical formula and the number of the chemical units in the elementary cell - this information is entered by *PRELIM* and saved in the basic crystal information part of the `m50` file. For other types of the maps `n` = 0.

3.4.6 The `m48` and `m47` Files

FOURIER stores local maxima and minima of the map to the `m48` and `m47` file. The maxima for modulated structures are saved together with modulation parameters calculated from the map.

The distances between the atoms existing in the structure and the peaks found in the map can be calculated by *DIST* program, which can directly read both `m47` and `m48` file. The positive maxima can be added to the `m40` file by *EDITM40*, which can read the `m48` file. In the case of modulated structures the modulation parameters are added together with the coordinates of the maxima so that the refinement can start with the starting point calculated from the map.

Figure 152 *The `m48` File for Modulated Structure*
(A file for an ordinary structure is analogical.)



```
    4                   1
 1.000000
 0.000000
 0.000000
 0.000000
max1      1  1     1.000000 0.072093 0.370162 0.666232      000  0  1  0
    36.45
 0.034325 0.002908-0.010730 0.006872-0.009755-0.014637
 0.000000
max2      1  1     1.000000 0.242085 0.212733 0.152491      000  0  1  0
     8.91
-0.004784-0.021230-0.013531-0.060348-0.021528-0.081200
 0.000000
max3      1  1     1.000000 0.081891 0.373225 0.518970      000  0  1  0
     6.70
 0.101414-0.006506 0.056510-0.031413-0.029077-0.034423
 0.000000
max4      1  1     1.000000 0.230782 0.064043 0.117471      000  0  1  0
     4.13
-0.031754-0.000731 0.031050 0.007749-0.026178 0.021138
 0.000000
```

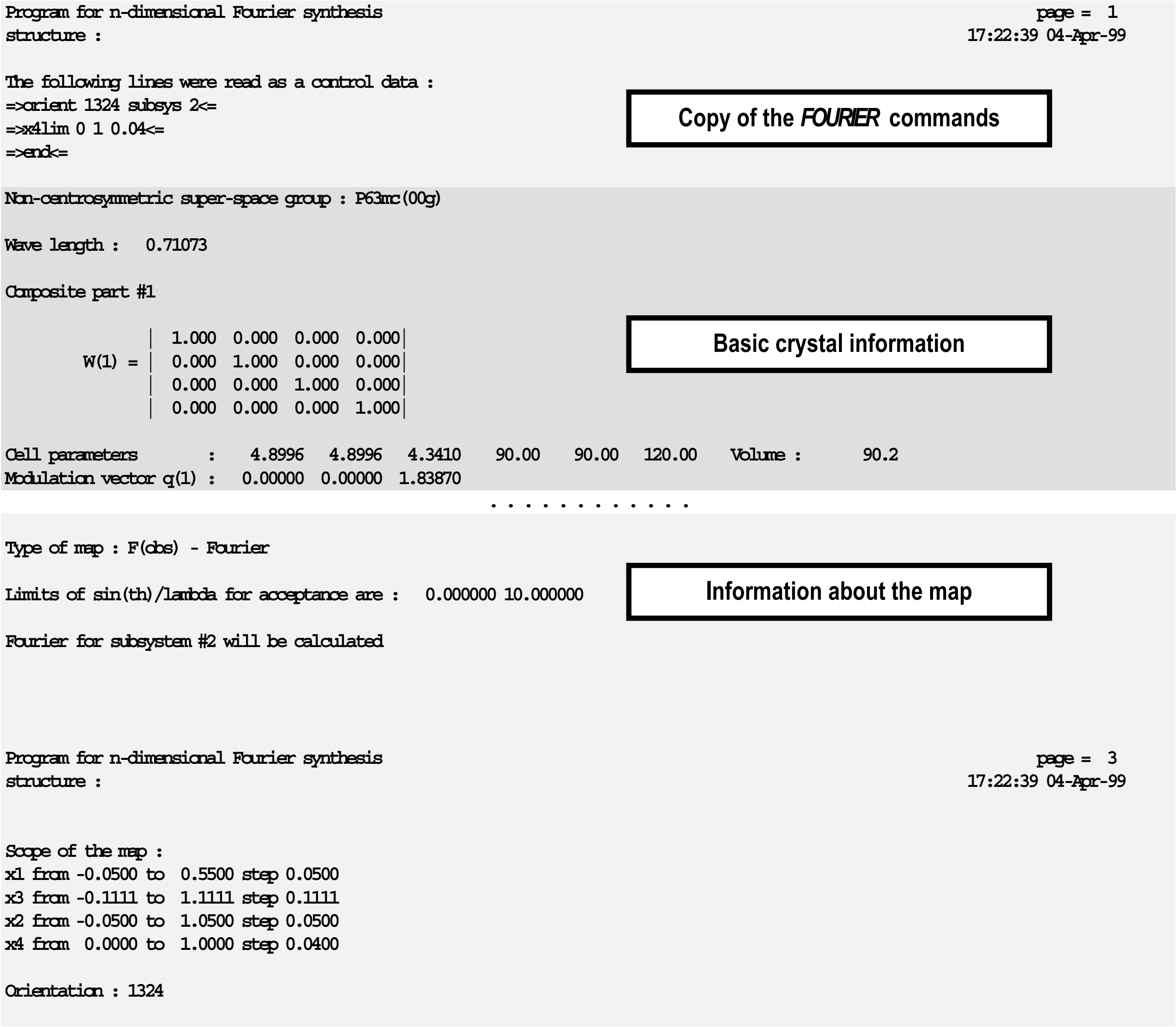
Figure 153 *The `m47` File for Ordinary or Modulated Structure*

```
    4                   1
 1.000000
 0.000000
 0.000000
 0.000000
min1      1  1     1.000000 0.041514 0.425487 0.748936      000  0  0  0
    19.02
min2      1  1     1.000000 0.004098 0.313358 0.716243      000  0  0  0
    10.99
min3      1  1     1.000000-0.000985 0.032555 0.749904      000  0  0  0
     3.55
min4      1  1     1.000000-0.003849 0.482830 0.750187      000  0  0  0
     2.01
 3.000000
```

3.4.7 Fourier Listing

FOURIER creates listing `jobname.fou`. It is wide listing and it is accessible from the listing viewer (see page 41).

Figure 154 *The Fourier Listing for Modulated Structure*
The listing for standard structures is similar but simplier becouse it doesn't contain the section concerning the assignment of modulation vawes.



```
Program for n-dimensional Fourier synthesis                                                          page =  1
structure :                                                                                  17:22:39 04-Apr-99

The following lines were read as a control data :
=>orient 1324 subsys 2<=
=>x4lim 0 1 0.04<=
=>end<=

Non-centrosymmetric super-space group : P63mc(00g)

Wave length :   0.71073

Composite part #1

         | 1.000  0.000  0.000  0.000|
 W(1) =  | 0.000  1.000  0.000  0.000|
         | 0.000  0.000  1.000  0.000|
         | 0.000  0.000  0.000  1.000|

Cell parameters       :    4.8996   4.8996   4.3410    90.00    90.00   120.00    Volume :      90.2
Modulation vector q(1) :  0.00000  0.00000  1.83870
                                             . . . . . . . . . . . .

Type of map : F(obs) - Fourier

Limits of sin(th)/lambda for acceptance are :   0.000000 10.000000

Fourier for subsystem #2 will be calculated




Program for n-dimensional Fourier synthesis                                                          page =  3
structure :                                                                                  17:22:39 04-Apr-99


Scope of the map :
x1 from -0.0500 to  0.5500 step 0.0500
x3 from -0.1111 to  1.1111 step 0.1111
x2 from -0.0500 to  1.0500 step 0.0500
x4 from  0.0000 to  1.0000 step 0.0400

Orientation : 1324
```

Figure 154 *(Continued)*

Positive peaks
are listed for each step in the x4 coordinate. In this case the step in x4 is 0.04.
`rho` is the absolute height of the maximum in e/A^3.
`rel` is relative height scaled together with the height of the negative peaks.

```
Searching of positive peaks - maximum number of peaks to be found :   5

             x4= 0.000                              x4= 0.040                              x4= 0.080
peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |
  1/  2  0.0000  0.0000 -0.0162     28.89|  1/  2  0.0000  0.0000 -0.0086     26.90|  1/  2  0.0000  0.0000 -0.0135     29.29|
  2/  4  0.5000  0.0005  0.0418      3.89|  2/  4  0.4930  0.5070  0.0604      4.30|  2/  1  0.3250  0.6500  0.6643      6.21|
  3/  3  0.1059  0.8941  0.3963      3.38|  3/  1  0.3250  0.6500  0.0616      2.54|  3/  4  0.4926  0.5074  0.0985      4.71|
  4/  1  0.3250  0.6500  0.8877      1.57|  4/  3  0.1000  0.1983  0.3993      2.13|  4/  3  0.2785  0.1500  0.4286      2.19|
  5/  5  0.2921  0.1500  0.3386      1.49|  5/  0  0.2683  0.7317  0.4868      1.94|  5/  5  0.2500  0.5130  0.4727      2.16|


             x4= 0.120                              x4= 0.160                              x4= 0.200
peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |
  1/  2  0.0000  0.0000  0.0065     30.68|  1/  1  0.3250  0.6500  0.4240     37.21|  1/  1  0.3250  0.6500  0.4528     92.83|
  2/  1  0.3250  0.6500  0.7110     12.16|  2/  2  0.0000  0.0000  0.0362     23.91|  2/  0  0.0000  0.0000  0.8951     13.64|
  3/  4  0.4897  0.5103  0.1518      4.67|  3/  3  0.3100  0.1500  0.4566      7.11|  3/  3  0.3180  0.1500  0.4785     13.14|
  4/  5  0.2141  0.5124  0.4560      3.28|  4/  4  0.0452  0.5250  0.0097      5.06|  4/  4  0.0576  0.5250  0.0393      8.90|
  5/  3  0.2952  0.1500  0.4538      3.14|  5/  5  0.4346  0.4682  0.2971      4.75|  5/  5  0.1134  0.7311  0.0040      7.14|
```

. . . .

```
             x4= 0.960                              x4= 1.000
peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |
  1/  2  0.0000  0.0000 -0.0422     30.71|  1/  2  0.0000  0.0000 -0.0162     28.89|
  2/  4  0.5074  0.4926  0.0106      3.84|  2/  4  0.5000  0.0005  0.0418      3.89|
  3/  0  0.4188  0.0347  0.3131      2.35|  3/  3  0.1059  0.8941  0.3963      3.38|
  4/  5  0.3483  0.1750  0.0957      1.57|  4/  1  0.3250  0.6500  0.8877      1.57|
  5/  0  0.3034  0.1500  0.3806      1.47|  5/  5  0.2921  0.1500  0.3386      1.49|
```

Successfully interpreted peaks
This is the list of peaks which have been interpreted with 1 harmonic wave. The rest of the peaks was discarded.
The 2nd peak corresponds to the position of Co atom.

```
The list of positive peaks written to the file m48

These peaks were successfully interpreted with 1 harmonic wave(s)
         x         y         z            rho   rel                       x         y         z            rho   rel

  1.  0.325616  0.650620  0.766937       36.84  287              4.  0.504202  0.008529  0.030857       5.59   44
  2.  0.000000  0.000000  0.956669       24.51  191 =Co1         5.  0.338993  0.141536  0.188239       4.06   32
  3.  0.211490  1.206709  0.459964        7.17   56
```

Figure 154 *(Continued)*

Negative peaks

are listed for each step in the x4 coordinate. In this case the step in x4 is 0.04.

`rho` is the absolute height of the maximum in e/A^3.

`rel` is relative height scaled together with the height of the negative peaks.

```
Searching of negative peaks - maximum number of peaks to be found :   5
Program for n-dimensional Fourier synthesis                                                            page =  5
structure :                                                                                   17:22:39 04-Apr-99


            x4= 0.000                              x4= 0.040                              x4= 0.080
peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |
  1/  0  0.0000  0.0000  0.4823    -13.42|  1/  0  0.0000  0.0000  0.4905     -7.37|  1/  0  0.5215  0.3322  0.3333     -4.66|
  2/  0  0.3305  0.5148  0.9423     -4.63|  2/  0  0.5144  0.3485  0.3678     -4.94|  2/  0  0.4368  0.5632  0.2723     -4.58|
  3/  0  0.5182  0.1513  0.3109     -4.23|  3/  0  0.4195  0.5387  0.1398     -4.10|  3/  0  0.0283  0.9717  0.4836     -4.12|
  4/  0  0.3250  0.6500  0.3881     -2.22|  4/  0  0.1690  0.4103  0.3692     -2.66|  4/  0  0.2075  0.7925  0.4213     -3.31|
  5/  0  0.2311  0.3749  0.3564     -1.84|  5/  0  0.3529  0.1750  0.7783     -2.45|  5/  0  0.2953  0.7047  0.1704     -3.30|
```

. . . .

```
            x4= 0.960                              x4= 1.000
peak/atom    x       y       z        rho |peak/atom    x       y       z        rho |
  1/  0  0.0000  0.0000  0.4565    -13.80|  1/  0  0.0000  0.0000  0.4823    -13.42|
  2/  0  0.1825  0.5159  0.8627     -5.11|  2/  0  0.3305  0.5148  0.9423     -4.63|
  3/  0  0.3250  0.6500  0.2174     -4.87|  3/  0  0.5182  0.1513  0.3109     -4.23|
  4/  0  0.5062  0.3477  0.0833     -3.17|  4/  0  0.3250  0.6500  0.3881     -2.22|
  5/  0  0.4441  0.2250  0.2000     -3.03|  5/  0  0.2311  0.3749  0.3564     -1.84|
```

List of negative peaks

This is the list of the strongest negative peaks. They are not necessarily present in all sections (i.e.for all tested values of x4).

```
The list of negative peaks written to the file m47

 which are the first  5 extremes peaks
       x         y         z            rho   rel                    x         y         z            rho   rel

  1.  0.324998  0.650001  0.194465     -19.02 -148 =La           4.  0.102622  0.550001  0.367333     -15.57 -121
  2.  0.324998  0.650000  0.930019     -16.35 -127               5. -0.000004  0.000002  0.469990     -14.58 -114
  3.  0.119941  0.880055  0.935496     -15.80 -123
```

3.5 Advanced Topics

3.5.1 Setting and deleting special modulation functions

Using a special modulation functions should be based on density maps indicating need of this kind of description. Usually the structure is first solved with harmonic modulations functions, which may fail for some atom. Investigation of A_3-A_4 sections[1] through an electron density map centerd in this atom can show a step-like behaviour which can be described either by a crenel or sawtooth function. Here are the steps how to set a special function in *JANA98*:

- Estimation of initial coefficients of the special function from an appropriate contour section. For a **crenel function** (see page 128) we need the center (`osin1`) and the width (`o`). For a **sawtooth function** (see page 124) we need the center(`xcos$`), the width(`ycos$`) and the A_3 coordinate (`xsin$`, `ysin$` or `zsin$`[2]). All of them can be easily found in a *CONTOUR* plot using the Locator.
- The initial parameters must be written to `m40` with a text editor. For a **crenel function** (see example in Figure 77, page 129) s_0 and w_0 should be 1, `o` and `osin1` have values read from the contour plot, `ocos1` is always 0. For a **sawtooth function** (see example in Figure 74, page 126) s_p and w_p should be 1, `xsin$`, `ysin$`, `zsin$`, `xcos$` and `ycos$` have values read from the contour plot and `zcos$` is always 0. In both cases the user should also set the refinement keys because they are not automatic.
- The initial function can be checked with *CONTOUR* with Options->Define atom to be indicated in map. It is especially useful when setting a sawtooth function.
- Refinement of the initial parameters.
- The special function can be further combined with harmonic position modulation functions. For a **crenel function** we simply add a harmonic position wave by *EDITM40*. For a **sawtooth function** the special function is always saved in `m40` like the last position modulation wave. Therefore to add (by *EDITM40*) the first harmonic wave we set the number of modulation waves to 2. If the special function is not defined in a full interval the harmonic functions need to be orthogonalized. This can be done through the *EDITM40* interface shown in page 170.
- Deleting of special function cane be done by *EDITM40* by setting number of relevant waves to 0.

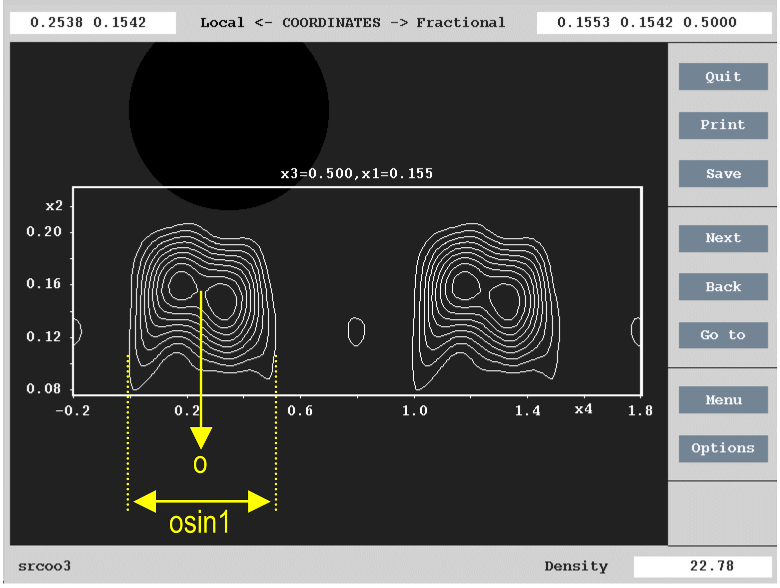
The figures below show example of setting a crenel and sawtooth modulation function.

---

[1] Special functions are only available for 3+1 dimensional structures.

[2] For each of them we need a different section definition (A_1-A_4, A_2-A_4 and A_3-A_4).

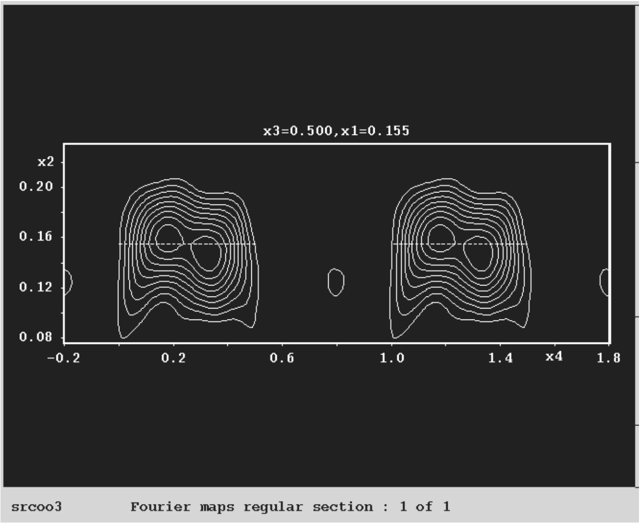
Figure 155 *Setting a crenel function*



0.2538 0.1542 Local <- COORDINATES -> Fractional 0.1553 0.1542 0.5000

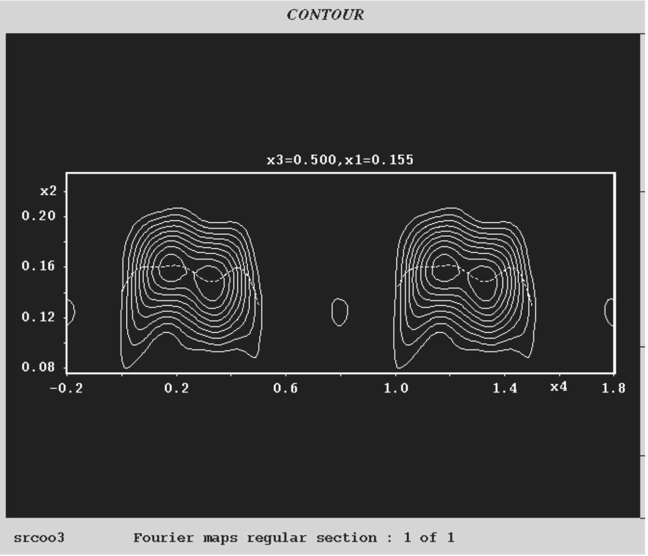
srcoo3 Density 22.78

(a) Usage of locator for reading initial parameters of a crenel function from a contour plot.



srcoo3 Fourier maps regular section : 1 of 1

(b) The contour plot with the initial crenel modulation function indicated by a dashed line.

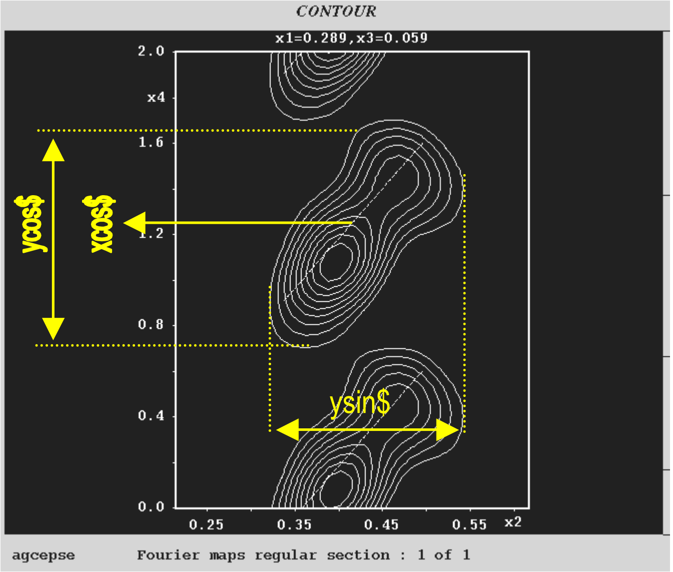


CONTOUR

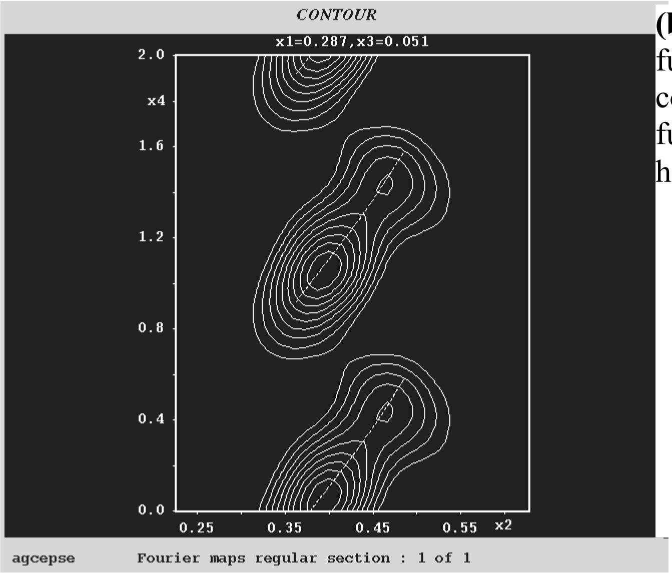
srcoo3 Fourier maps regular section : 1 of 1

(c) The position modulation function calculated as combination of the crenel function and three orthogonalized harmonic modulation waves.

Figure 156 *Setting a sawtooth function*



(a) Setting initial parameters of a sawtooth function from a contour plot.



(b) The position modulation function calculated as combination of the sawtooth function and one orthogonalized harmonic modulation wave.

3.5.2 Commensurate structures

This part is being prepared for the *JANA2000* manual.

4

Structure Interpretation

This chapter describes basic programs of JANA98, their input and output files and the control keys.

IN THIS CHAPTER:

Next : References, page 341.
Previous chapter: Structure Solution and Refinement, page 91

4

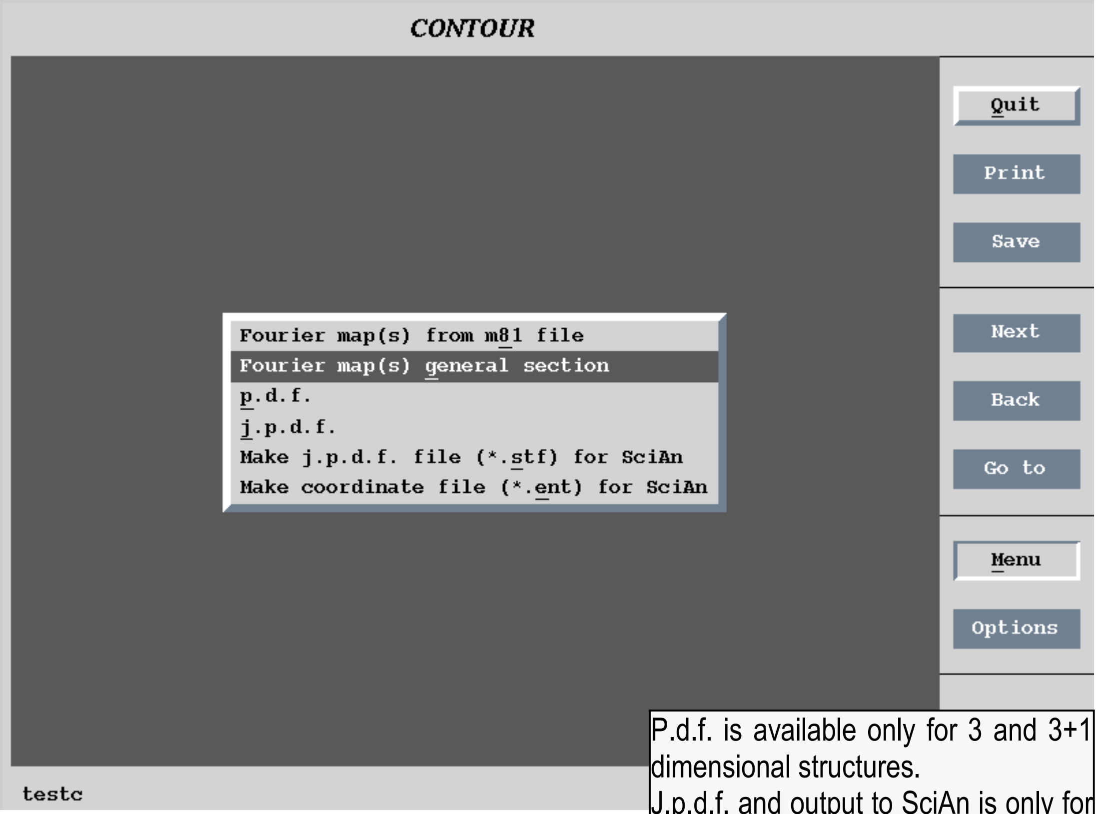
Structure Interpretation

This part describes structure interpretation programs of *JANA98*. *CONTOUR* is used for visualisation of electron density maps and probability density functions. *GRAPHT* plots positional, temperature and occupation parameters and derived entities as function of the `t` coordinate. *DIST* is used for calculation of distances, torsion angles and best planes.

4.1 Program *CONTOUR*

Input: [`m40`],`m50`,[`m81`]
Output: HPGL file, PostScript file, PCX file or SciAn file
M50 control keywords: none

Figure 157 *The basic Window of CONTOUR*



P.d.f. is available only for 3 and 3+1 dimensional structures.
J.p.d.f. and output to SciAn is only for 3 dimensional structures

4.1.1 Description of *CONTOUR*

CONTOUR produces contours plots of Fourier maps in the direct space for regular and modulated structures. For regular and four dimensional structures it can also make maps of probability density function (p.d.f.) and joint probability density function (j.p.d.f.) based on refined parameters including Gram-Charlier expansion terms up to 6th order. *CONTOUR* doesn't make 3d visualization of electron density, but it can generate input files for program SciAn.

CONTOUR is a fully interactive program and doesn't need any control commands in the `m50` file. Like in other programs of *JANA98* its functionality varies for ordinary and modulated structures.

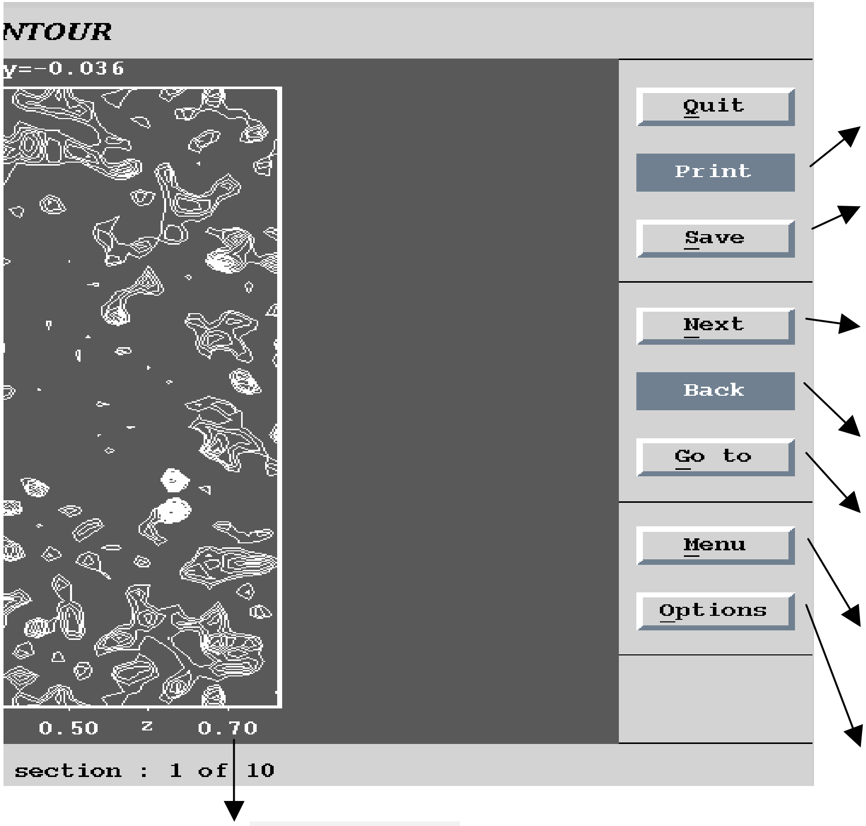
We recommend reading of *FOURIER* (page 265) before this chapter.

4.1.2 Drawing R_3-R_3 and A_3-A_3 sections stored in the `m81` File

The tool "Fourier maps from m81 file" allows plotting of two dimensional sections by an electron density map stored in the `m81` file by *FOURIER*. The sections orientation and the order they are stored in `m81` are defined by *FOURIER* control commands (see page 273).

After selecting “Fourier map(s) from m81 file” the program automatically plots the first section and activates operating buttons on the right of the basic window. Their functions are common for most types of *CONTOUR* tasks.

Figure 158 *The Operating Buttons of the CONTOUR Window*



Sends output to the printer specified in Preferences (see page 49)

Saves the picture in HPGL, PostScript, ASCII, PCX (PC only) or STF format

Goes to the next section

Goes to the previous section

Goes to a specific section

Goes back to the main menu

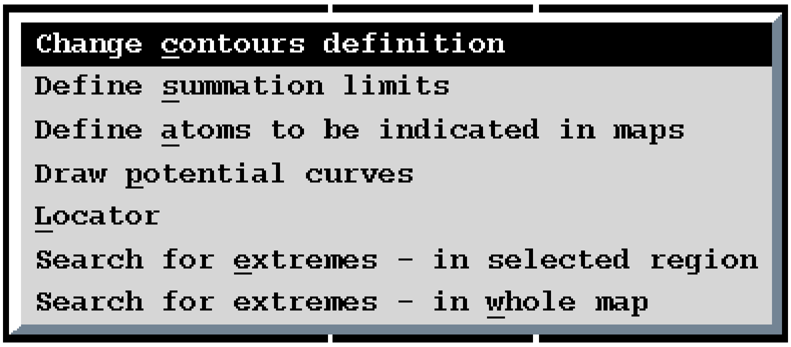
Goes to the Contour options menu (later in this chapter)

fractional coordinates

4.1.3 Contour options and Contour tools

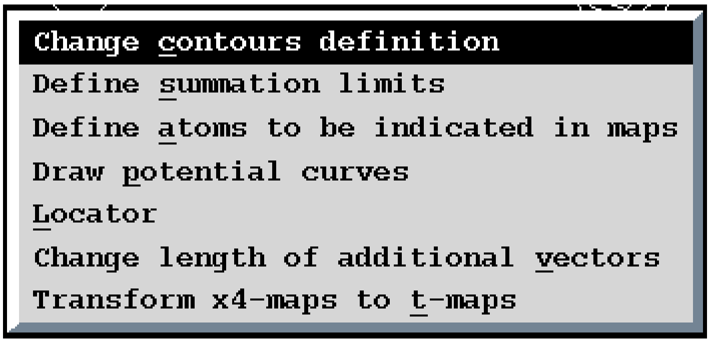
The Contour options are common for `m81` plots, general sections and probability density plots. The menu differs for ordinary and modulated structures.

Figure 159 *Contour options for ordinary structures*



- *Change contour definitions* sets the basic properties of the contour plot (see Figure **161**).
- *Define summation limits* is used for superposition of sections (see page 299).
- *Define atoms to be indicated in maps* is used for selection of atoms which will be marked in the plots by a cross.
- *Draw potential curves* plots electron density curves and potential curves defined by a section through selected maxima - see Figure 164 - Figure 166.
- *Locator* identifies atoms being near to the current pointer position within a user defined limit - see Figure 167.
- *Search for extremes - in selected regions* searches maxima and minima in a region selected by mouse in the current section - see Figure 163.
- *Search for extremes - in whole map* searches maxima and minima in the whole map (without defining an area by mouse).

Figure 160 *Contour options for modulated structures*

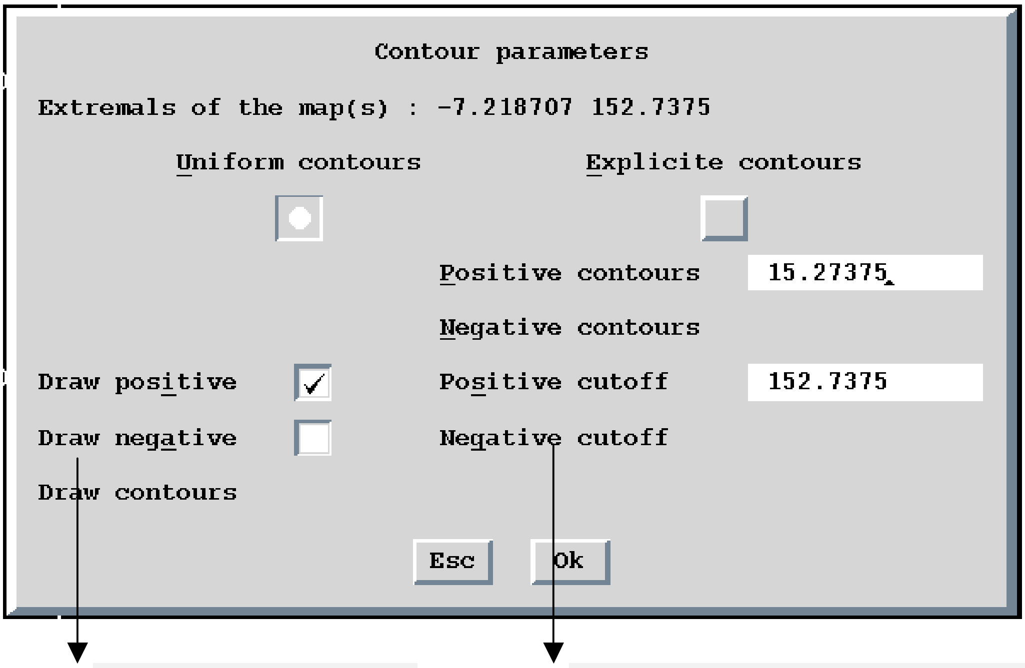


- *Change length of additional vector* is used for scaling sections containing x4 coordinate - see page 308.
- *Transform x4 map to t-maps*. The difference between x4 maps and t-maps is explained in page 273. Example of a t-section is in Figure 174.

Figure 161 *The Contour Definitions Menu*

(a) *With uniform contours*

The units of the contour intervals are e/Å^3*.*
The contour intervals are set automatically with respect to the highest maximum found in the map. The maxima from all layers are taken into account. If there is a strong maximum in some layer, the default contour interval can be too rough for the rest of the sections and needs to be changed.



Contour parameters

Extremals of the map(s) : -7.218707 152.7375

Uniform contours ● Explicite contours ☐

Positive contours 15.27375

Negative contours

Draw positive ☑ Positive cutoff 152.7375

Draw negative ☐ Negative cutoff

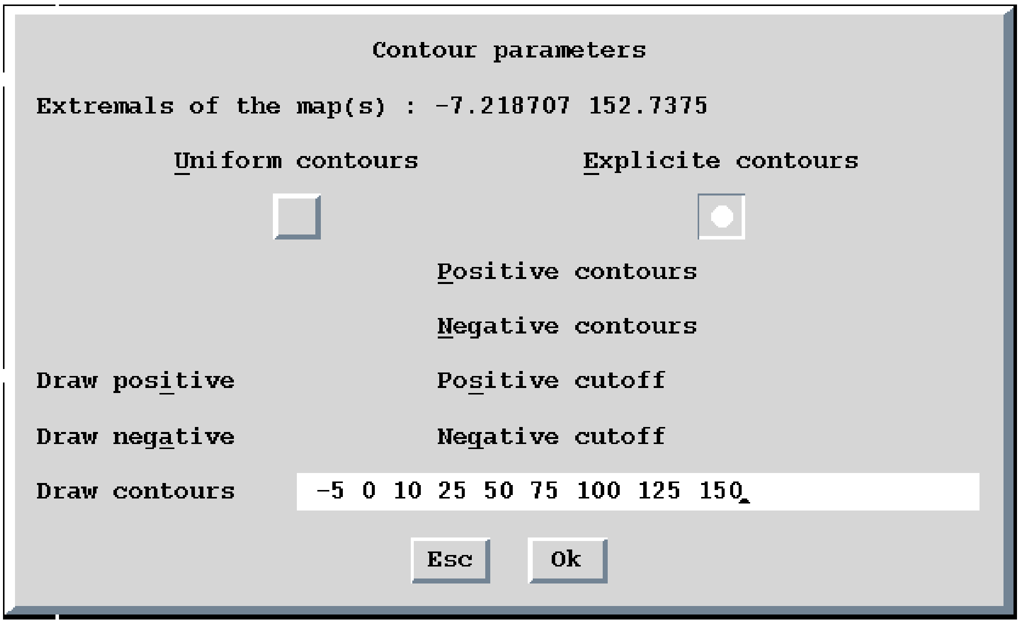
Draw contours

The negative maxima (When selected) are plotted by dashed lines

The cutoff interval can suppress plotting of very strong maxima in order to get better performance and shorter HPGL or PostScript files.

(b) *With explicit contours*

The contour to be plotted are explicitly listed in the text box.



Contour parameters

Extremals of the map(s) : -7.218707 152.7375

Uniform contours ☐ Explicite contours ●

Positive contours

Negative contours

Draw positive Positive cutoff

Draw negative Negative cutoff

Draw contours -5 0 10 25 50 75 100 125 150

Figure 162 *CONTOUR: A Strong Maximum Affected by the "Positive Cutoff" Limit.*

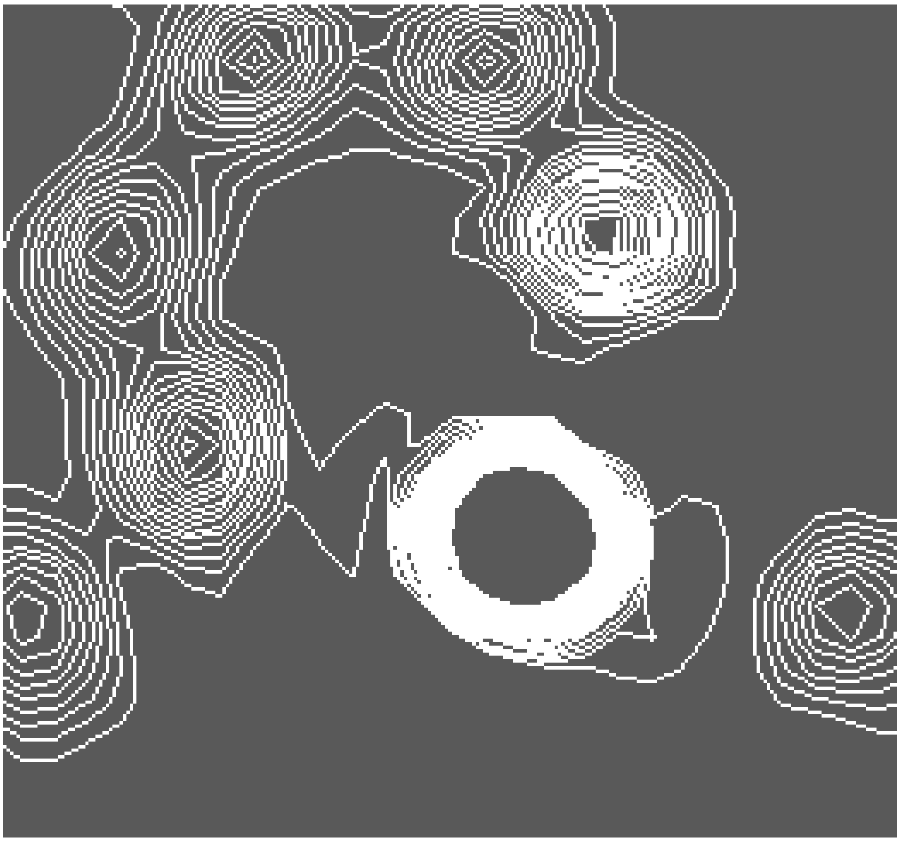


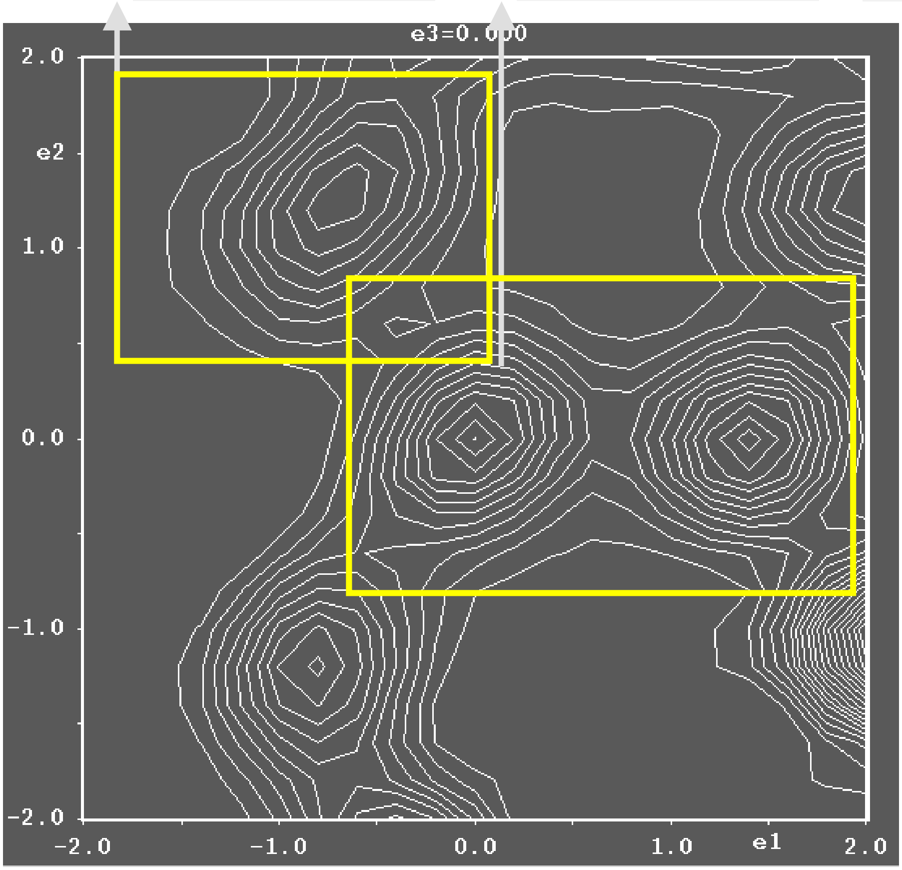
Figure 163 *Searching for extremes*

The area which the program searches for extremes is defined by conjunction of up to 5 rectangles.

1.
Click (i.e. press and release) the first mouse button to start dragging of a rectangle

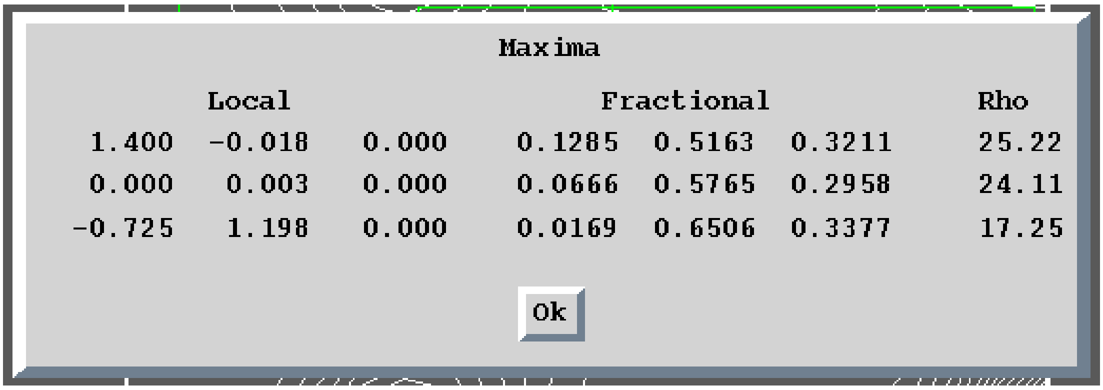
2.
Click the first mouse button again to finish the rectangle. The point 1 and 2 can be repeated.

3.
Click the second mouse button to start the searching procedure



The search results:

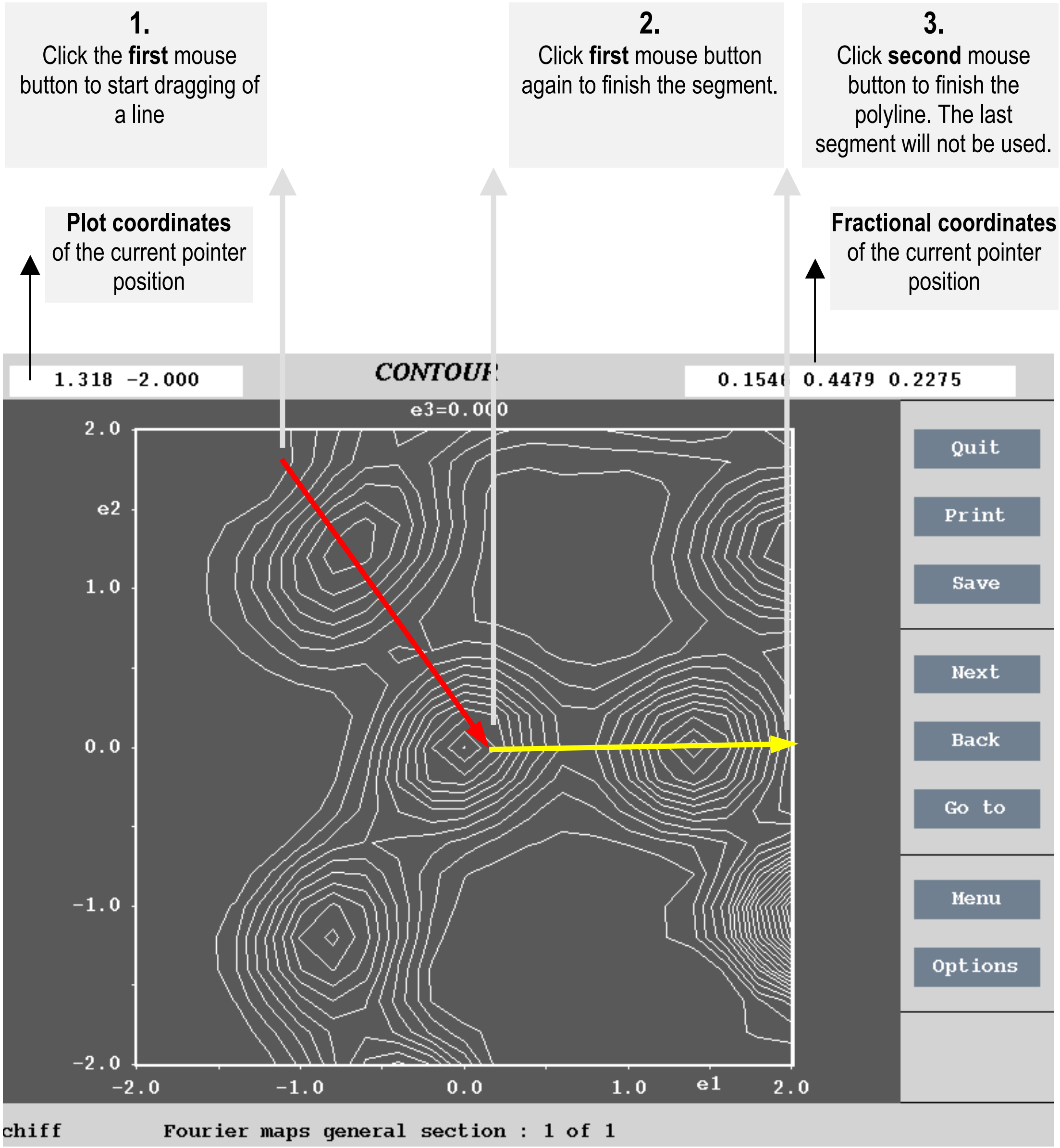
(Rho is the peak height in e/Å^3)



```
                              Maxima

            Local                            Fractional                 Rho
   1.400   -0.018    0.000      0.1285   0.5163   0.3211        25.22
   0.000    0.003    0.000      0.0666   0.5765   0.2958        24.11
  -0.725    1.198    0.000      0.0169   0.6506   0.3377        17.25
```

Figure 164 *Defining electron density and potential curves*

The section through the maxima is defined by a polyline drawn to the contour plot.



1.
Click the **first** mouse button to start dragging of a line

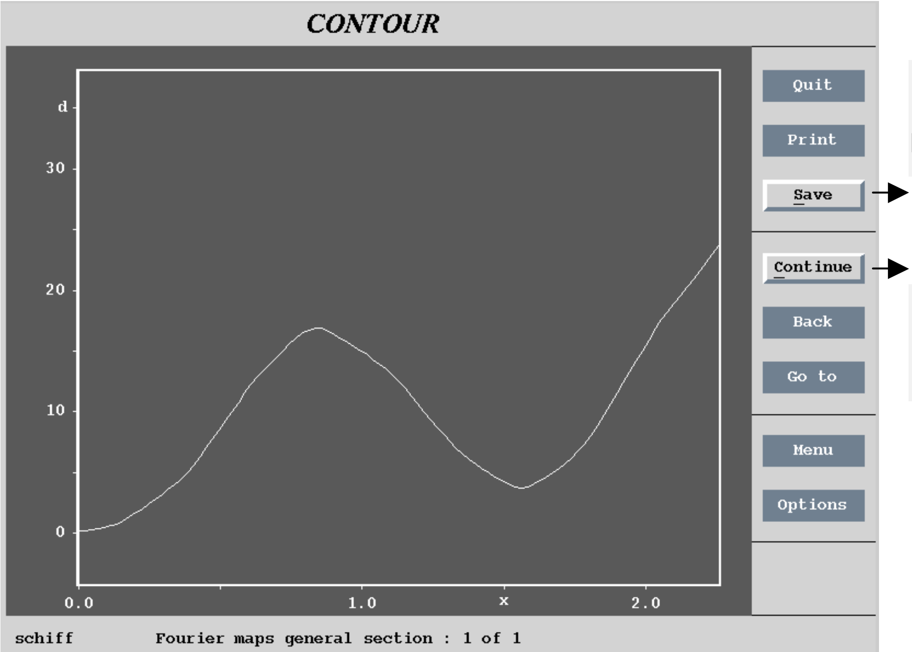
2.
Click **first** mouse button again to finish the segment.

3.
Click **second** mouse button to finish the polyline. The last segment will not be used.

Plot coordinates of the current pointer position

Fractional coordinates of the current pointer position

Figure 165 *Example of an electron density curve*

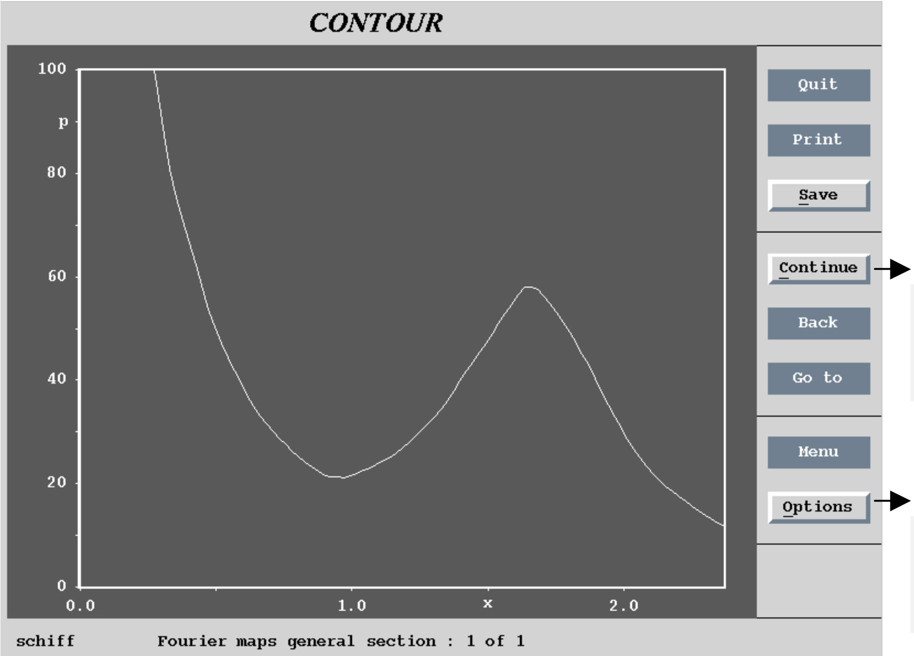


Saves the picture in HPGL or PostScript format

Goes to the potential curve plot

Figure 166 *Example of a potential curve*

These curves play an important role in a study of anharmonic effects in the crystal. The program can estimate errors either by extrapolating from the error map (if calculated in advance) or directly by applying the Monte Carlo method for the selected path.

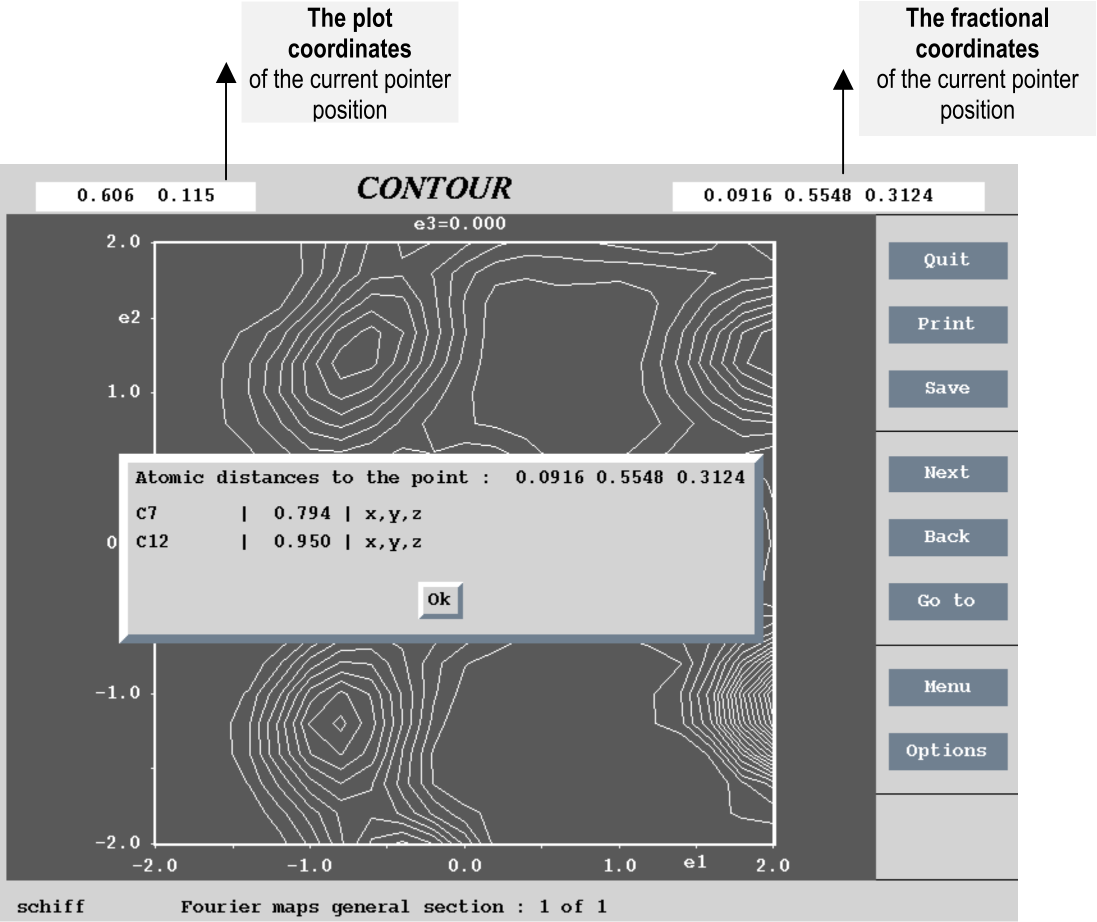


Goes to the basic contour plot

Options for the potential curve

Figure 167 *Usage of locator*

The locator mode is activated by choosing "Locator" from the Options menu and finished by pressing the second mouse button. The atoms found near to the locator position are listed together with their symmetry codes.



The plot coordinates of the current pointer position

The fractional coordinates of the current pointer position

```
0.606  0.115
```

```
0.0916 0.5548 0.3124
```

```
Atomic distances to the point :  0.0916 0.5548 0.3124
C7        |  0.794 | x,y,z
C12       |  0.950 | x,y,z
```

```
schiff        Fourier maps general section : 1 of 1
```

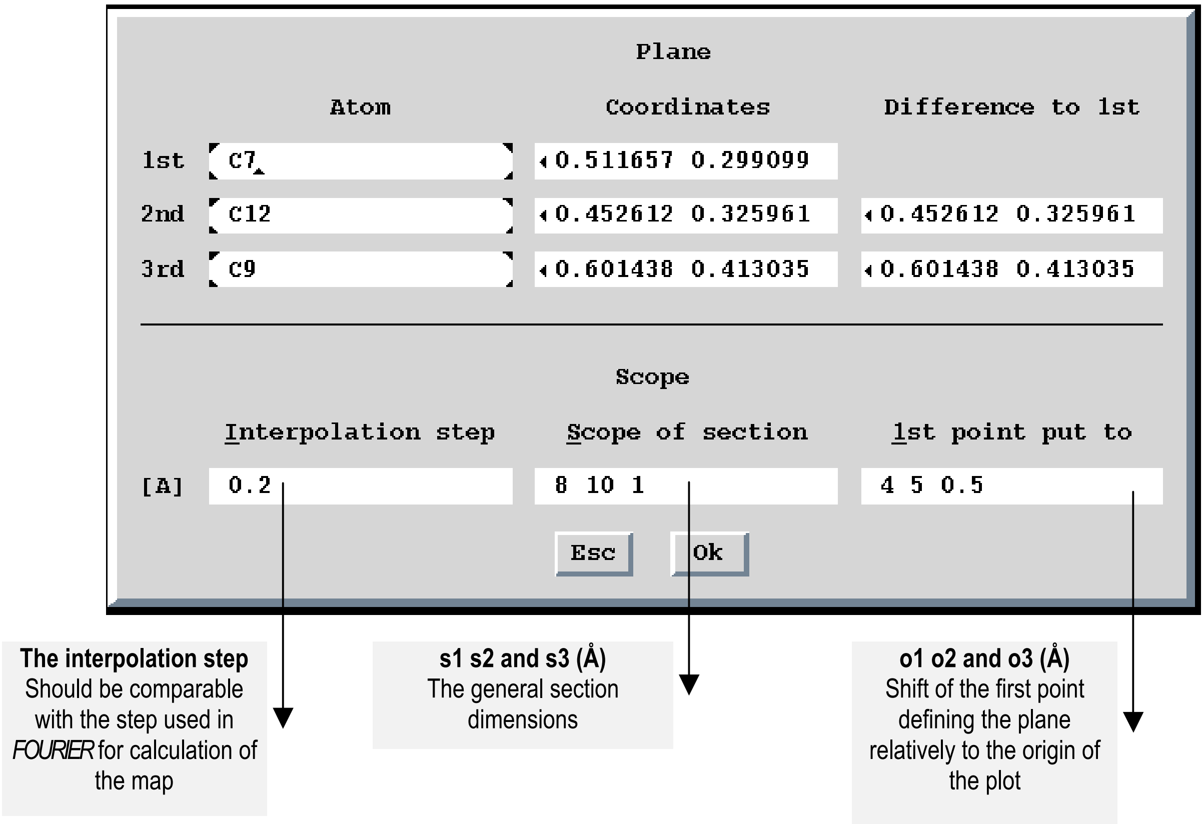
4.1.4 Drawing General sections

The `m81` file contains a set of two dimensional sections by electron density map. The sections are calculated by *FOURIER* according to the `orient` control command. The section orientation is not arbitrary because each plane has to be parallel with two basic cell vectors.

CONTOUR allows to recalculate the sections to be parallel with an arbitrary user defined plane. This plane is called the *general section plane* and can be defined with help of the *Plane form*. Calculation of general sections is possible for R_3-R_3 and A_3-A_3 section types.

Figure 168 *The Plane form of CONTOUR*

This form is used for the definition of a general section plane. The symbols s1,s2,s3,o1,o2,o3 will be used in the next figure.



Plane

	Atom	Coordinates	Difference to 1st
1st	C7	0.511657 0.299099	
2nd	C12	0.452612 0.325961	0.452612 0.325961
3rd	C9	0.601438 0.413035	0.601438 0.413035

Scope

	Interpolation step	Scope of section	1st point put to
[A]	0.2	8 10 1	4 5 0.5

The interpolation step
Should be comparable with the step used in *FOURIER* for calculation of the map

s1 s2 and s3 (Å)
The general section dimensions

o1 o2 and o3 (Å)
Shift of the first point defining the plane relatively to the origin of the plot

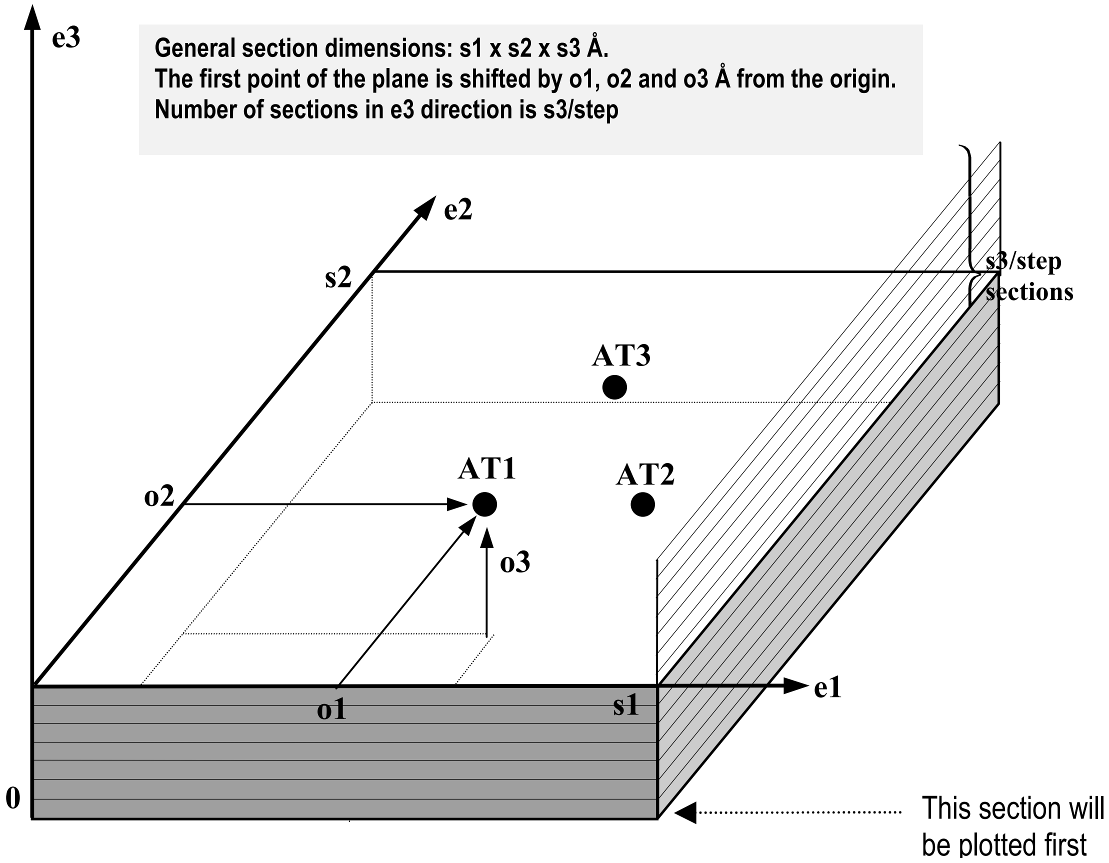
The plane is defined by three points which can be entered like atom names, by coordinates or as one point or atom and two vectors from that point. The atom names may contain molecular position indicators and internal symmetry codes (see § 1.2.6 page 27).

If the map is calculated by *FOURIER* in the whole independent volume of the 3+d space, the sections can be arbitrarily expanded or translated. The area of the general plane is called **scope** and is defined by the length (in Å) of *x* and *y* plot axes in the Scope text box. The third component defines the "width" of the section in Å along the *z* plot axis. The number of sections perpendicular to *z* is equal to the width divided by the interpolation step.

The option "1st point put to" defines where the first point defining the plane will appear in the contour plot. With (0,0,0) the first point is equivalent with the origin in the lower left corner. Otherwise it is translated according to Figure 169 and Figure 170.

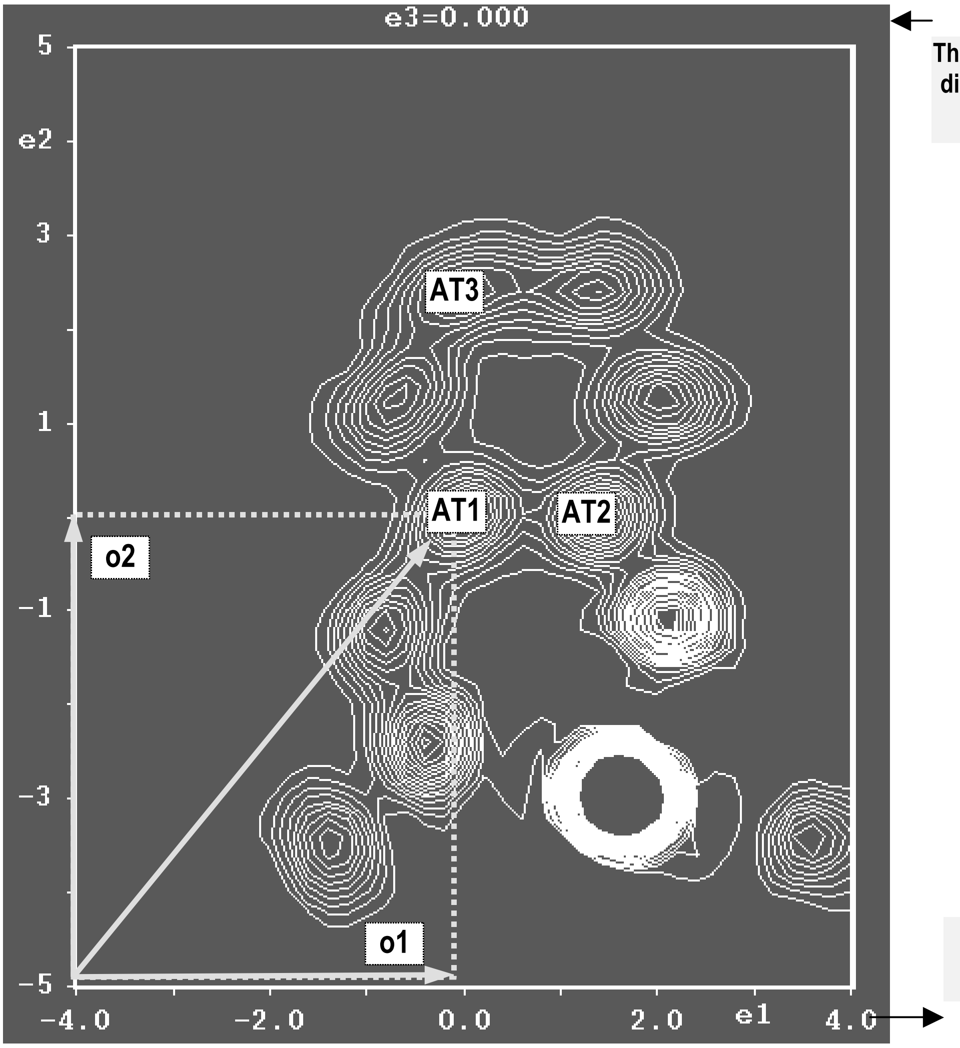
Figure 169 *The scope definition and contour plot orientation*

Cartesian axis used by CONTOUR for plotting are e1, e2 and e3. The measurement unit is Å.
The general section is defined by three points AT1, AT2 and AT3 entered in the Plane form. The line AT1 - AT2 is parallel to e1.
The section dimensions(Å) are s1, s2 and s3. The position of AT1 in the plotting axes is (o1,o2,o3). The first plotted section goes through AT1.



The next figure shows a general section defined by three points AT1, AT2 and AT3 calculated with scope s1=8Å, s2=10Å and s3=1Å. The position of AT1 is (4,5,0.5). Because the structure fragment is not exactly planar, all sections in the e3 direction are superimposed by means of the “Define summation limits” option of the Contour options menu. Plotting of the strongest maximum was suppressed by the “positive cutoff” limit entered in the Contour definitions menu.

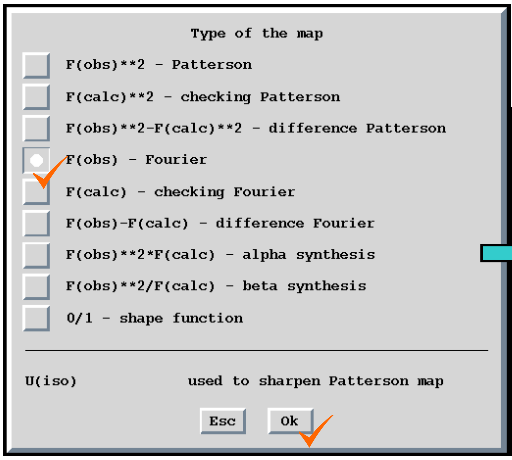
Figure 170 *An Example of the General Section for Ordinary Structure*



The summation direction is e3

Absolute units in Å

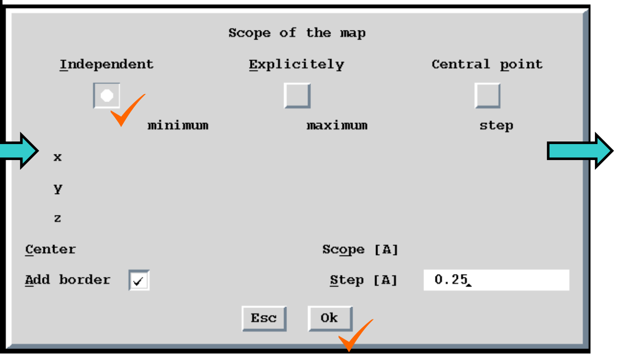
Figure 171 *The procedure to prepare the general section from Figure 170.*



Type of the map

☐ F(obs)**2 - Patterson
☐ F(calc)**2 - checking Patterson
☐ F(obs)**2-F(calc)**2 - difference Patterson
● F(obs) - Fourier ✓
☐ F(calc) - checking Fourier
☐ F(obs)-F(calc) - difference Fourier
☐ F(obs)**2*F(calc) - alpha synthesis
☐ F(obs)**2/F(calc) - beta synthesis
☐ 0/1 - shape function

U(iso) used to sharpen Patterson map



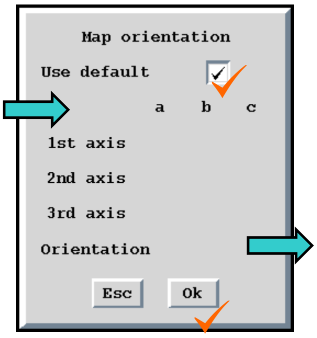
Scope of the map

Independent ● ✓ | Explicitely ☐ | Central point ☐

minimum maximum step

x
y
z

Center
Scope [A]
Add border ☑
Step [A] 0.25

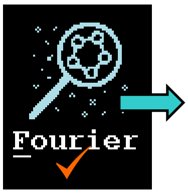


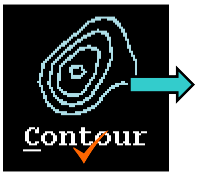
Map orientation

Use default ☑ ✓

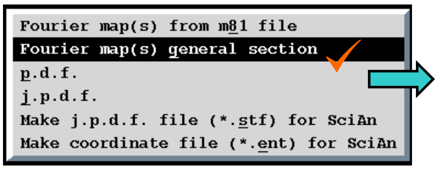
a b c

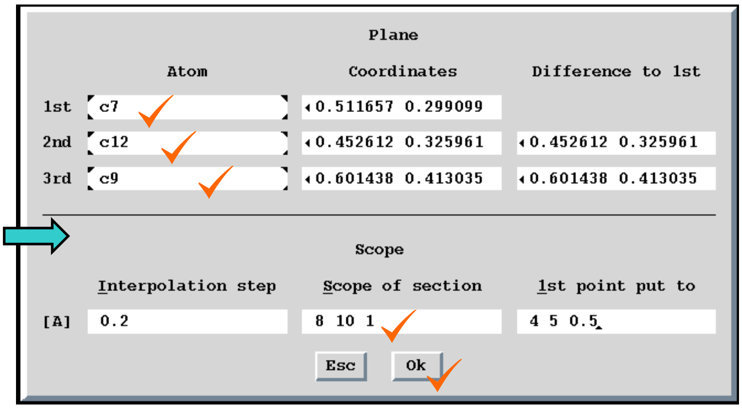
1st axis
2nd axis
3rd axis
Orientation





Step 2:
Calculation of General section



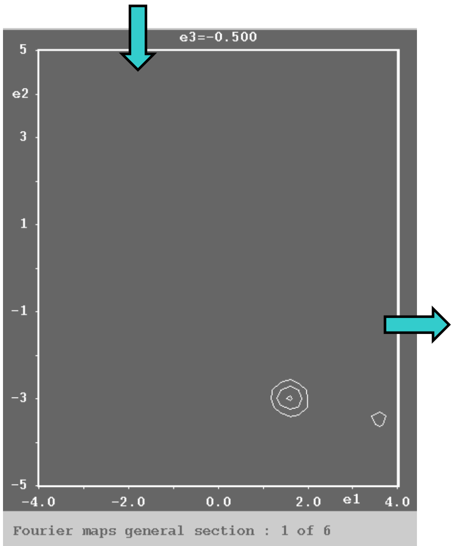


Plane

	Atom	Coordinates	Difference to 1st
1st	c7 ✓	0.511657 0.299099	
2nd	c12 ✓	0.452612 0.325961	0.452612 0.325961
3rd	c9 ✓	0.601438 0.413035	0.601438 0.413035

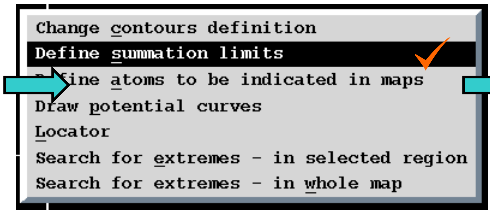
Scope

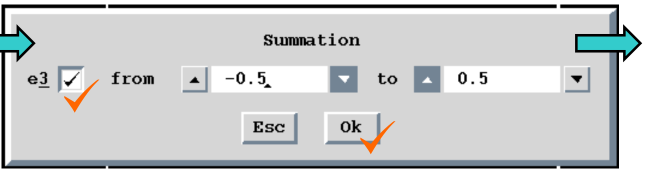
	Interpolation step	Scope of section	1st point put to
[A]	0.2	8 10 1 ✓	4 5 0.5



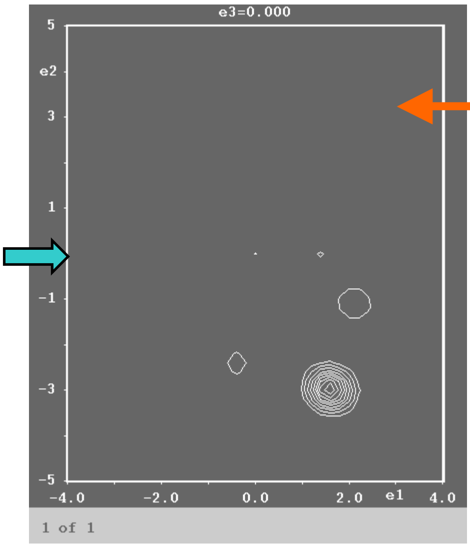
Fourier maps general section : 1 of 6

Step 1: the Fourier map is calculated in the independent volume so that the general section can be arbitrarily expanded.
Step 2: the general section is defined by three atoms from m40. The width of the section is set to 1Å so that (with the interpolation step 0.2Å) 6 parallel general sections is calculated. The last figure in this page shows the first section located 0.5 Å below the plane c7-c12-c9.

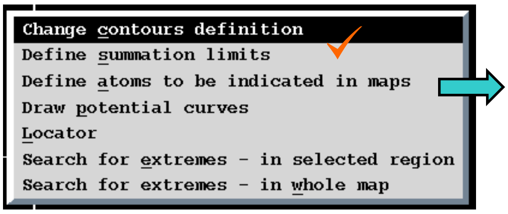


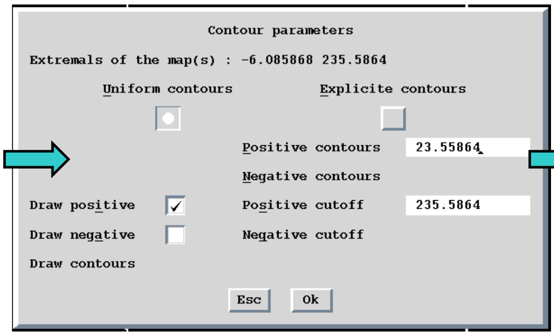


e3 ☑ from -0.5 to 0.5



This figure shows the superposition of the six sections parallel with the c7-c12-c9 plane. Very strong maximum in the lower right corner causes the other maxima are not visible with the default contour setting.





Extremals of the map(s) : -6.085868 235.5864

Uniform contours ● Explicite contours ☐

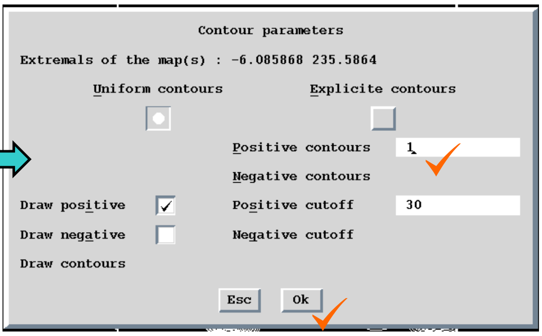
Positive contours 23.55864

Negative contours

Draw positive ☑ Positive cutoff 235.5864

Draw negative ☐ Negative cutoff

Draw contours



Extremals of the map(s) : -6.085868 235.5864

Uniform contours ● Explicite contours ☐

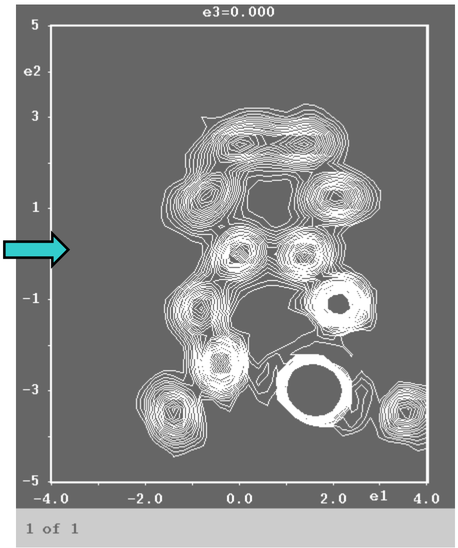
Positive contours 1

Negative contours

Draw positive ☑ Positive cutoff 30

Draw negative ☐ Negative cutoff

Draw contours

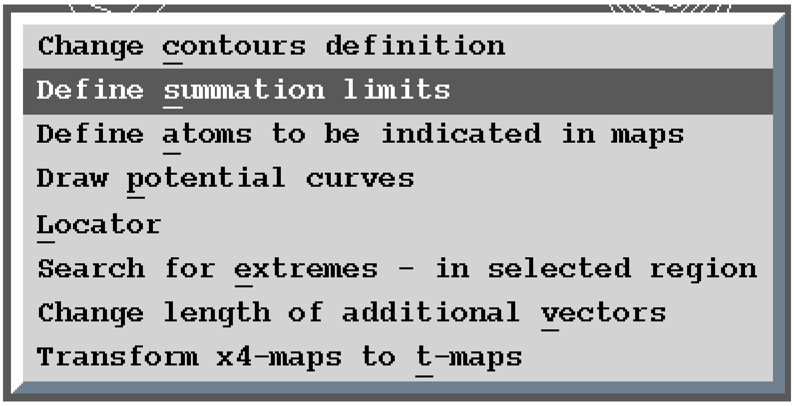


The last figure shows the section after editing the contour definitions. The contours of the largest maximum are truncated by setting the "Positive cutoff" to 30.

General section for modulated structures

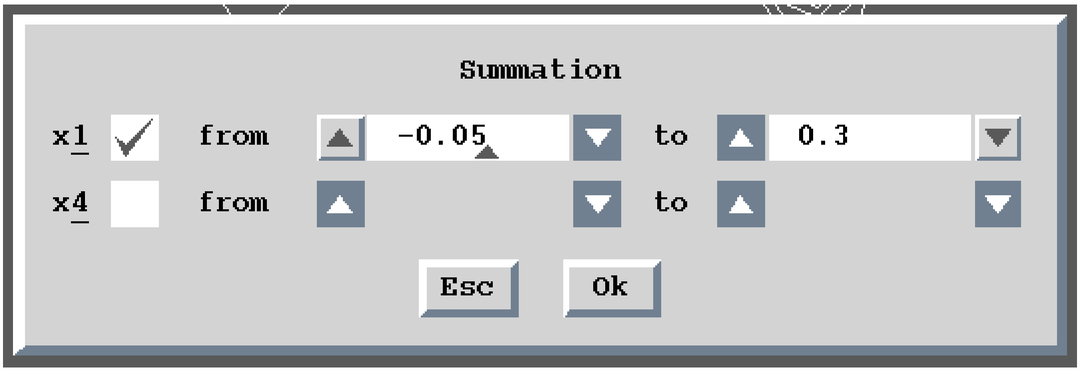
The way of a general plane definition is the same like for ordinary structures. Summation can be done not only for e3 axis but also for remaining directions (x4, x5, x6).

Figure 172 The Contour Options Menu for Modulated Structures



Change contours definition
Define summation limits
Define atoms to be indicated in maps
Draw potential curves
Locator
Search for extremes - in selected region
Change length of additional vectors
Transform x4-maps to t-maps

Figure 173 The Contour Summation Limits Form for Modulated Structures



Summation

x1	☑	from	-0.05	to	0.3
x4	☐	from		to	

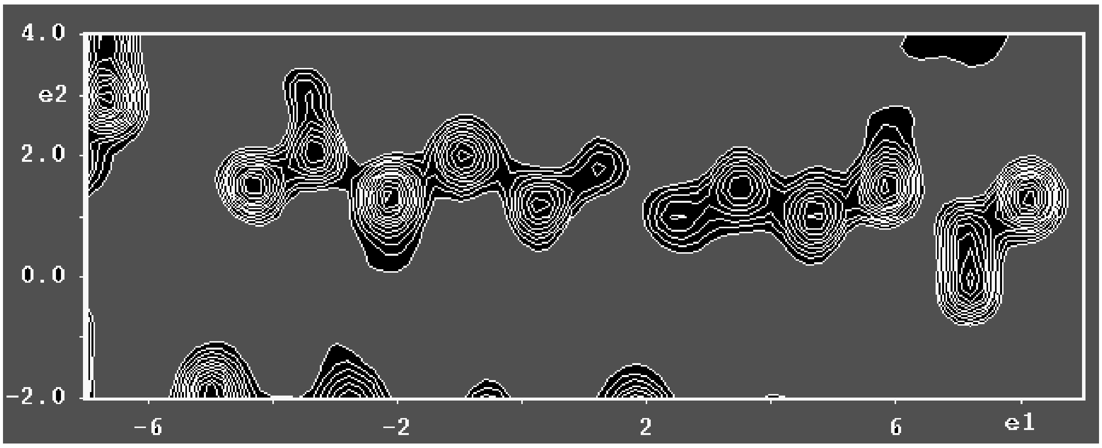
For modulated structures the x4 maps (The A_3-A_3 section type, see page 273) can also be transformed to t-maps (i.e. R_3-R_3 section type). The order of steps is important because

- Summed maps cannot be transformed to t-maps
- t-maps cannot be transformed back to x4-maps

With x4 maps the geometry of the structure may be deformed but the sections are periodic. With t-maps the geometry is the same like in the real space but the sections are no longer periodic. This is illustrated in the following figures.

Figure 174 *An Example of the General Section for Modulated Structure*

(a) The sections were transformed to t-maps and summed along the e3 direction. This is plot for t=0.



(b) The result of the additional summation along the x4 direction.

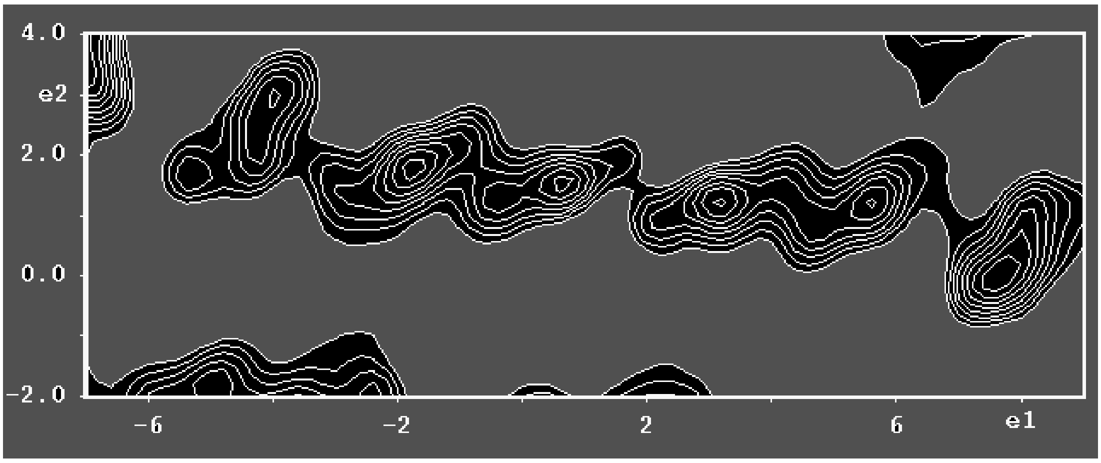


Figure 175 *Four cells of an A_3-A_3 section. The plot is periodic.*

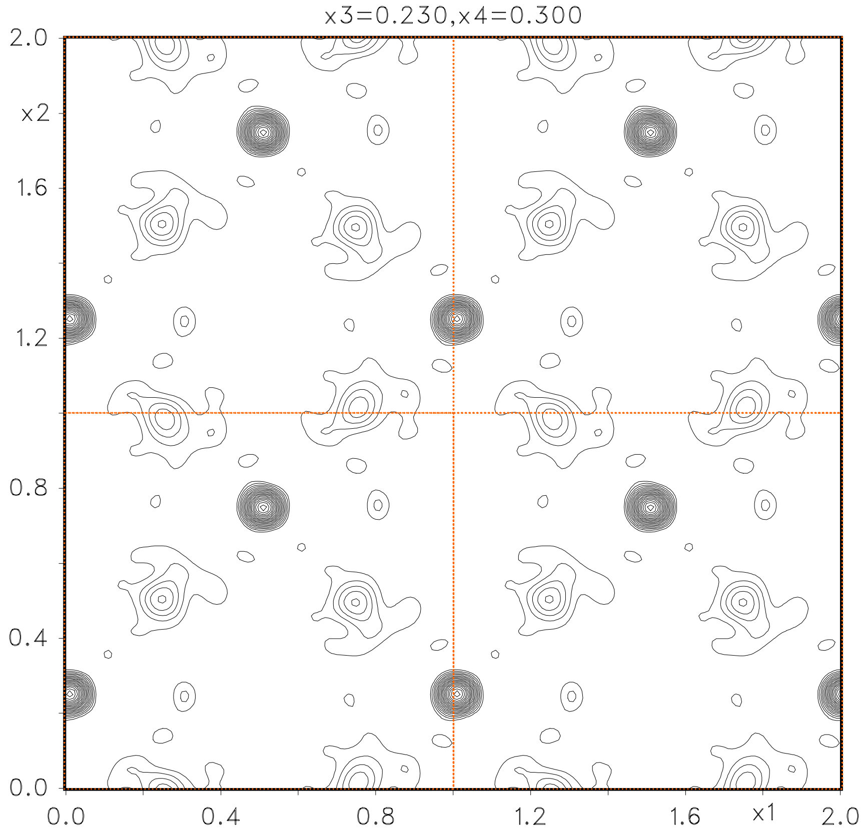
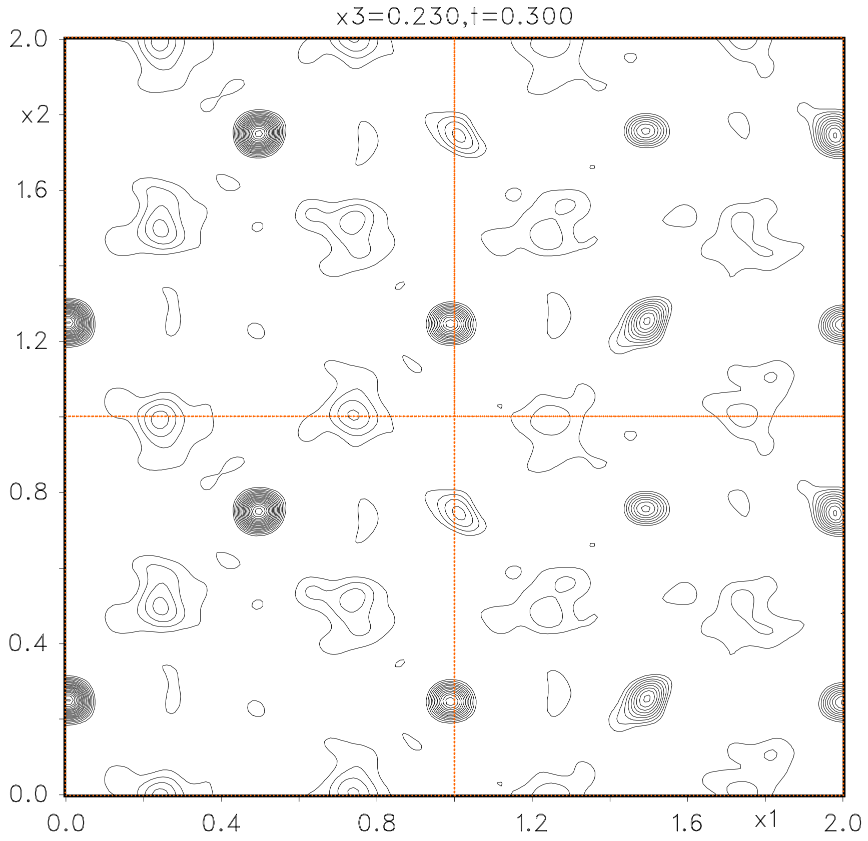


Figure 176 *Four cells of a R_3-R_3 section. The plot is not periodic.*



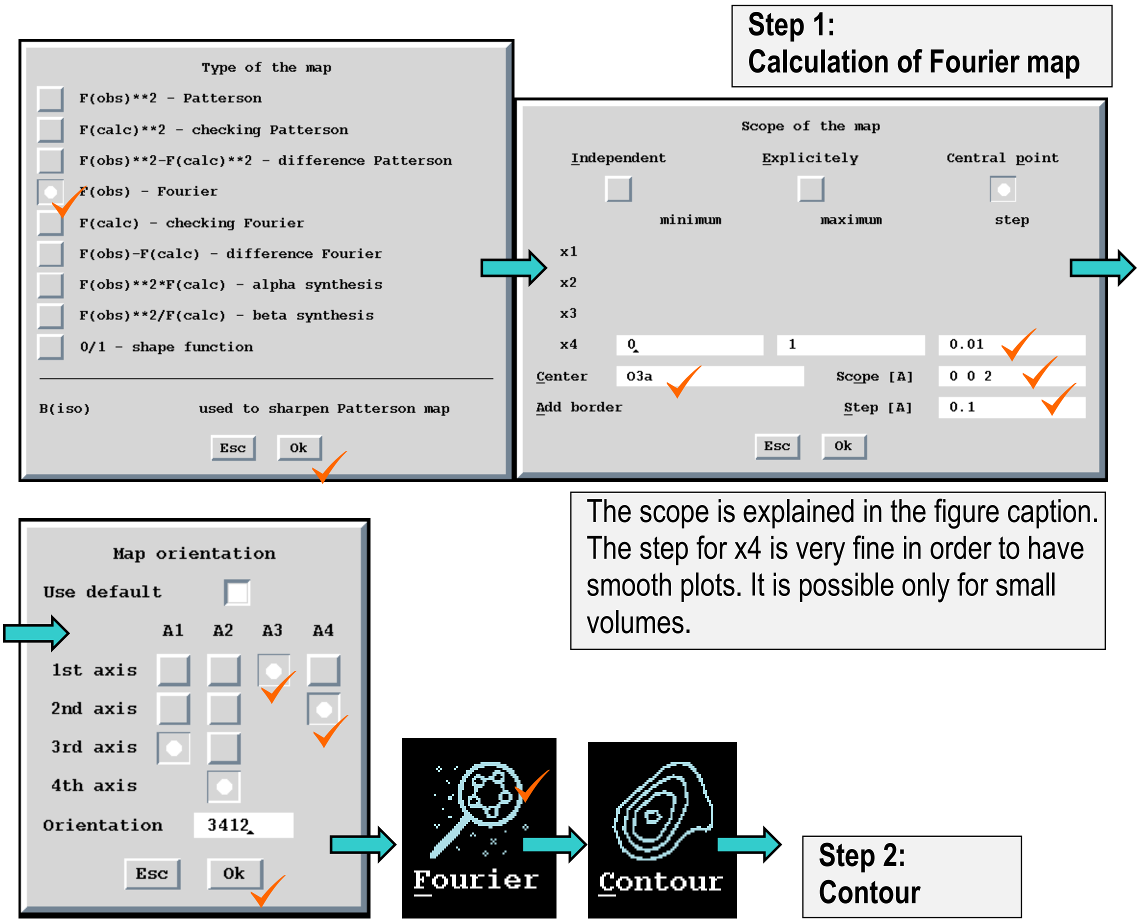
4.1.5 Drawing A$_3$-A$_4$ sections

If the 2-dimensional sections prepared by Fourier contain the x4 (x5,x6) axis *CONTOUR* does not open the main menu and plots immediately the first section. For this type of sections
CONTOUR allows to draw modulation functions for one or more atoms. The electron density map should be calculated in a volume centerd in the atom in question.

The next example illustrates usage of Contour for plotting of modulation functions.

Figure 177 *Plotting of A$_3$-A$_4$ sections*

In this example the Fourier map is calculated in the vicinity of atom O3a of a testing four dimensional structure. The scope 0 0 2 means that the calculated sections go exactly through x(O3a) and y(O3a) and extend ±1Å around z(O3a). The fourth coordinate varies from 0 to 1. The first two axes in the Map orientation form comprises the fourth coordinate which therefore appears in the plotted sections.



Step 1:
Calculation of Fourier map

Type of the map
☐ F(obs)**2 - Patterson
☐ F(calc)**2 - checking Patterson
☐ F(obs)**2-F(calc)**2 - difference Patterson
● F(obs) - Fourier
☐ F(calc) - checking Fourier
☐ F(obs)-F(calc) - difference Fourier
☐ F(obs)**2*F(calc) - alpha synthesis
☐ F(obs)**2/F(calc) - beta synthesis
☐ 0/1 - shape function
B(iso) used to sharpen Patterson map

Scope of the map
☐ Independent ☐ Explicitely ● Central point

	minimum	maximum	step
x1			
x2			
x3			
x4	0	1	0.01

Center: O3a
Scope [A]: 0 0 2
Add border
Step [A]: 0.1

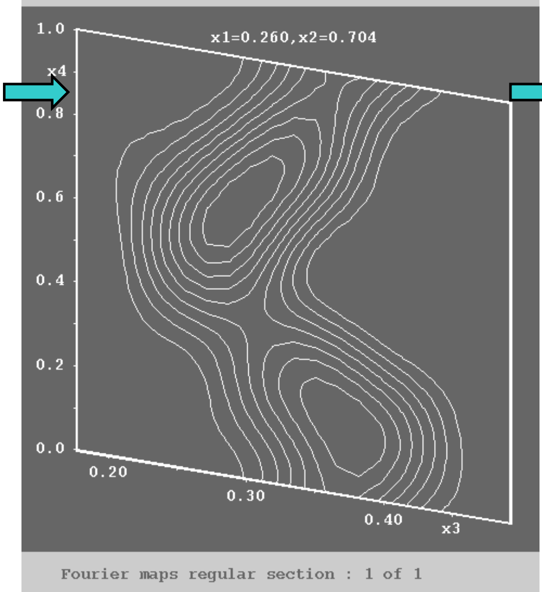
The scope is explained in the figure caption. The step for x4 is very fine in order to have smooth plots. It is possible only for small volumes.

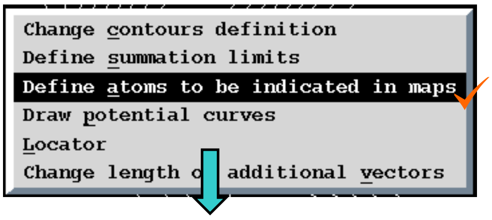
Map orientation
Use default ☐

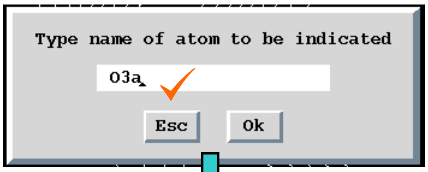
	A1	A2	A3	A4
1st axis	☐	☐	●	☐
2nd axis	☐	☐		●
3rd axis	●	☐		
4th axis		●		

Orientation: 3412

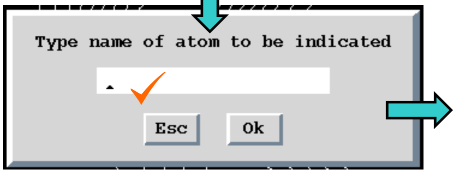
Step 2:
Contour

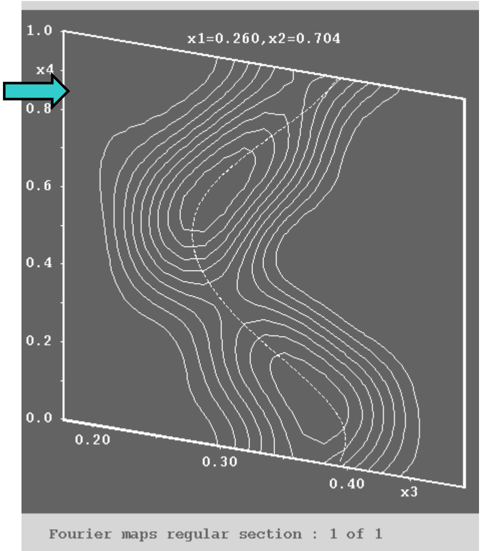


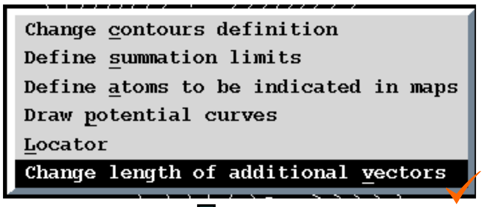


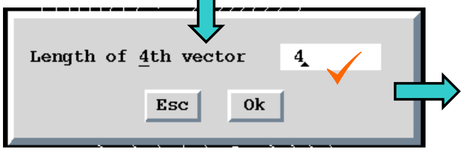


Type name of atom to be indicated: O3a



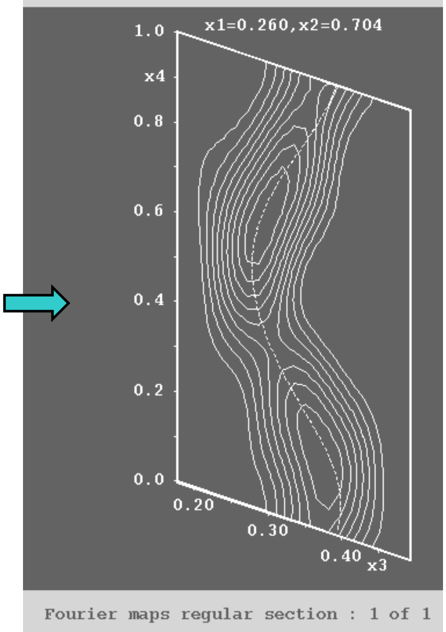






Length of 4th vector: 4

The change of the length of additional vector (the x4 coordinate in this case) is made only like an exercise. The default value 2 Å is usually convenient.



4.1.6 Drawing (joined) probability density functions

The program *CONTOUR* allows to plot and analyse the ***probability density function*** (*p.d.f.)* , which makes visualization of the refined "shape" of the selected atom. The

$$p.d.f.(\mathbf{u}) = p.d.f._{har}(\mathbf{u})\left[1 + (1/3!)C^{ijk}H_{ijk}(\mathbf{u}) + (1/4!)D^{ijkl}H_{ijkl}(\mathbf{u}) + (1/5!)E^{ijklm}H_{ijklm}(\mathbf{u})\ldots\right] \qquad (E\ 69)$$

where $H_{ij\ldots n}$ are Hermite polynomials, C,D,E,F are anharmonic displacement tensors (see page 122).

For more complex picture of the density the individual *p.d.f.'s* have to be combined. The resulting function is called ***joined probability density function*** (*j.p.d.f*).

CONTOUR makes also possible calculation and drawing of an error map that takes into account the covariation matrix. This map is calculated by a Monte Carlo method and may be time consuming.

JANA98 makes only 2-dimensional maps. However, *CONTOUR* allows saving a 3d map in the "stf" format, which can be used as an input to SciAn[1].

---

[1] See http://www.scri.fsu.edu/~lyons/scian/ for more information.

4.2 Program *DIST*

Input: `m40`, `m50`, [`m47`],[`m48`]
Output: `m61`,`dis`
M50 control keywords: the section between the keywords `dist` and `end`

4.2.1 Description of *DIST*

DIST calculates distances, angles, torsion angles and best planes for atoms present in the `m40` file. It can also use the coordinates of Fourier maxima and minima stored by *FOURIER* in the `m47` and `m48`. For modulated structures *DIST* calculates distances and angles as a function of the `t` coordinate.

The results are written to a wide listing `jobname.dis` in the newspaper columns form and also to `m61` in one column. The wide listing is accessible by Listing viewer (see page 41). The listing and `m61` are complementary (see page 330).

If *DIST* is started with default options it calculates the distances to each atom in `m40`. The minimum distance limit is 0 and the maximum distance is taken from `m50` according to the chemical type (see `dmax` key, page 77). With the default setting *DIST* does not list the full coordination of each atom because it calculates each distance only once. In the case of modulated structures the distances are listed for 10 values of `t` coordinate.

The following paragraphs contain description of the SetCommands interface for *DIST*. The options set by the user are transformed to commands and written to `m50`. The syntax of the resulting commands is also explained.

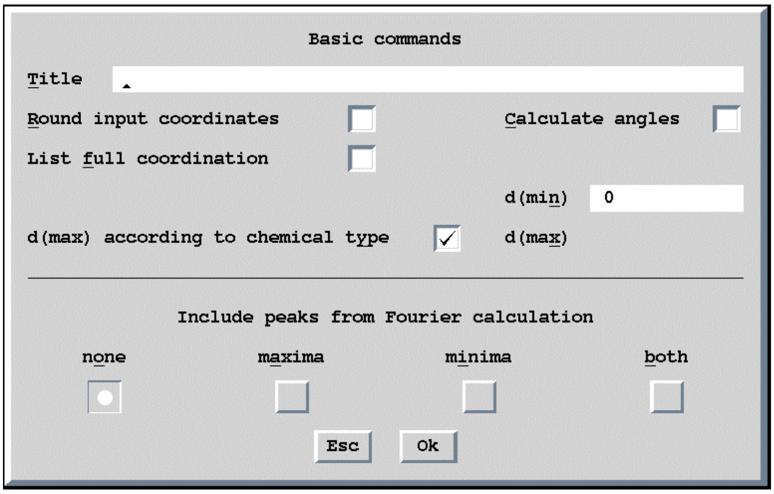
Figure 178 *The SetCommands menu for DIST*

4.2.2 Basic commands

In the Basic commands form the user chooses bond limits, calculation of angles, type of output and whether the Fourier peaks are to be included in the calculation. See Table 28 for details.

The lower bond limit is the same for all atoms. The upper bond limits can be overall or dependent on the chemical type of atoms. If two atoms have different upper bond limit the calculation is based on the average value of these limits.
If the upper bond limit is set according to chemical types the upper bond limit of Fourier peaks is based on the first chemical type listed in `m50`.

Figure 179 *The Basic Commands*



Title

Round input coordinates ☐

Calculate angles ☐

List full coordination ☐

d(min) 0

d(max) according to chemical type ☑

d(max)

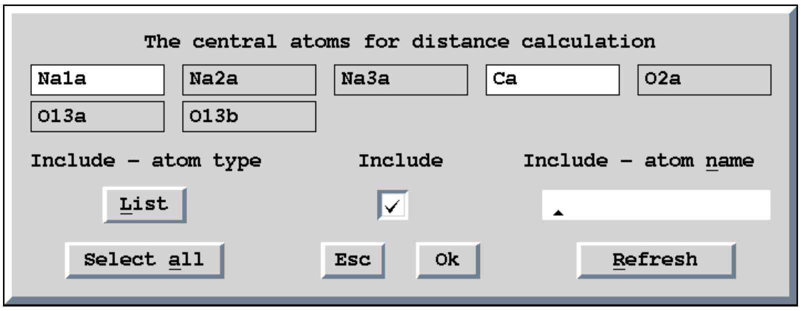
Include peaks from Fourier calculation

none ●
maxima ☐
minima ☐
both ☐

4.2.3 Selection commands

DIST calculates coordination for each atom flagged like a ***central atom***. In the listing the central atoms are surrounded by a box from asterisks. With the default setting all atoms from `m40` (and optionally `m47` and `m48`) are considered central atoms. This can be changed by Selection form. The Fourier peaks are not present in this form so that if they are included to the calculation they are all used as central atoms.

Figure 180 *Selection commands*



Na1a
Na2a
Na3a
Ca
O2a
O13a
O13b

Include – atom type

Include ☑

Include – atom name

Table 28 *Basic commands of DIST in the `m50` file*

Name	**Syntax and Description**
dmin **dmax**	**dmin** `number` **dmax** `number` **Default value**: **dmin** 0 The limits for calculation of distances in Å. Only distances and angles between `dmin` and `dmax` are calculated for each central atom. If `dmax` is omitted the upper distance limit is determined by the chemical type of the atom and taken from `m50` (see `dmax` key, page 77).
include	**include** `flag` **Default value**: **include** 0 Includes the Fourier peaks to the calculation `flag` = 0 the Fourier peaks are not used `flag` = 1 includes the `m48` file with **positive** Fourier peaks `flag` = 2 includes the `m47` file with **negative** Fourier peaks `flag` = 3 includes both `m47` and `m48` file
angles	**angles** `flag` **Default value**: **angles** 0 Turns on/off calculation of angles. `flag` = 0 The angles are not calculated `flag` =1 The angles are calculated
fullcoor	**fullcoor** `flag` **Default value**: **fullcoor** 0 `flag` = 0 Each distance will be calculated and printed only once. `flag` = 1 Full coordination will be calculated and printed for each central atom.
round	**round** `flag` **Default value**: **round** 0 `flag` = 0 Without rounding `flag` = 1 The distances and e.s.d.'s will be rounded to the first significant value.

Table 29 *Selection commands of DIST in the `m50` file*

Name	**Syntax and Description**
select	**select** `atoms` **Default value**: **select** * Select the central atoms for the calculation. The wildcards are permitted. If the select command is not present in `m50` all atoms are used as central ones.

4.2.4 Torsion angles and best planes

The calculation of torsion angles and best planes is intended for standard structures. In case of modulated structures only average positions of atoms are used.[1]

The angles and planes are defined by atom names from m40. The wild cards are not allowed but the internal symmetry codes (see page 27) are accepted.

Torsion angle is defined by four atoms `at1`, `at2`, `at3` and `at4` from `m40`. It is the angle between vectors `at2-at1` and `at3-at4` projected along `at2-at3`.

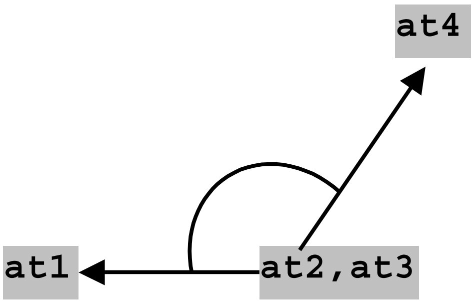
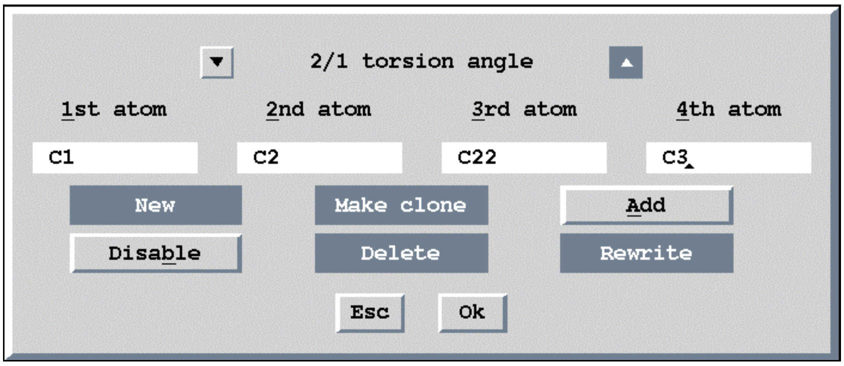


Figure 181 *The Torsion Angles Form*



2/1 torsion angle

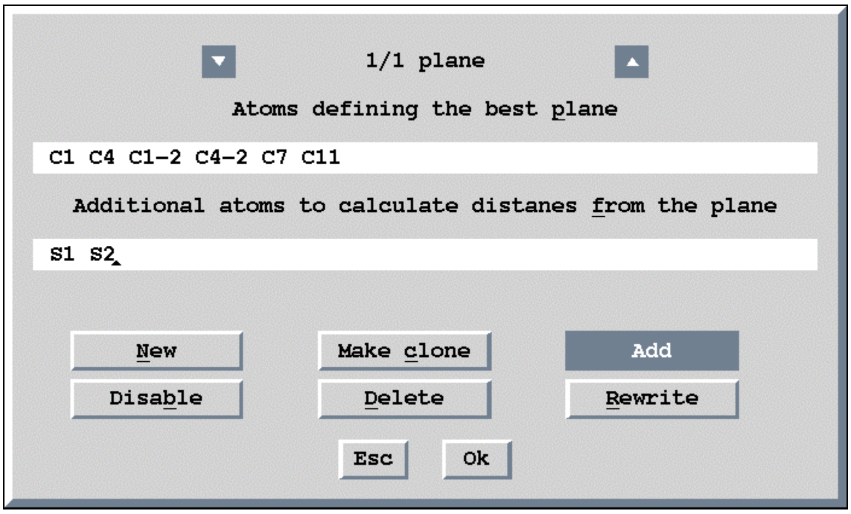
1st atom	2nd atom	3rd atom	4th atom
C1	C2	C22	C3

Best plane is defined by the atoms in the first line of the Best Planes form. The plane is calculated as the least square plane having minimal distances to these atoms. Each atom involved in the iteration process is taken with a weight derived from the e.s.d. of the distance between the atom and the plane. *DIST* then prints distances between the plane and the atoms listed in both lines of the form. *DIST* calculates also dihedral angles between all calculated best planes.

---

[1] We can expand the structure and save it in the SHELX format with "Transformation of m40 and m50 to various formats" of *EDITM40*, than import the expanded area back to *JANA98* by Tools->Transfer files from SHELX and calculate torsion angles and best planes for modulated positions.

Figure 182 *The best plane form*



1/1 plane

Atoms defining the best plane

C1 C4 C1-2 C4-2 C7 C11

Additional atoms to calculate distanes from the plane

S1 S2

Table 30 *Torsion angles and best planes commands of DIST in the* `m50` *file*

Name	Syntax and Description
torsion	**torsion** `at1 at2 at3 at4` Sets calculation of torsion angle. It is defined as the angle of vectors `at2-at1` and `at3-at4` projected along `at2-at3. At1, at2, at3` and `at4` are names of atoms from `m40`. The wild cards are not allowed but the internal symmetry codes (see page 27) are accepted. The command can be repeated to calculate several torsion angles.
plane	**plane** `n1 n2 at2 at2 …. atn2` Sets calculation of best plane. The plane is defined by names of atoms from `m40`. First `n1` atoms of the list are used for the definition of the plane. The distances from the plane are calculated for all atoms of the list (`n2` atoms). • The plane command must appear **after** the commands controlling distances and angles calculation. • The list of atoms can continue on arbitrary number of lines • The command can be repeated to calculate several best planes If there is more than one best plane defined *DIST* calculates angles between them.

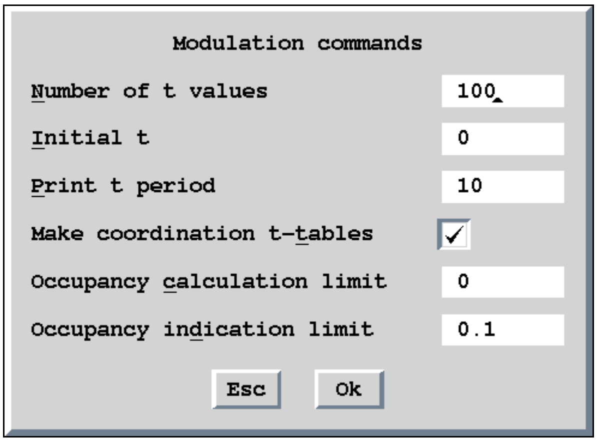
4.2.5 Modulation commands

The distances and angles for modulated structures are calculated as function of `t` coordinate. The function is calculated for "`Number of t values`" points and printed for each "`Print t period`". For commensurate structures the initial step is t_0 and the number of steps is given number of points where the function is defined.

In case of composite structure the coordination of central atoms can substantially vary with change of `t`. For this reason *DIST* allows to print comprehensive tables of the whole coordination as a function of `t`.

If some atoms in the structure are occupationally modulated the user may need to define "`Occupancy calculation limit`". The distances are calculated only for `t` values where occupancy is above the limit. The "`Occupancy indication limit`" means that if the occupancy of an atom for some value of `t` is below this limit the atom will be marked in the listing by “*”.

Figure 183 *Modulation commands*



Modulation commands

Number of t values	100
Initial t	0
Print t period	10
Make coordination t-tables	☑
Occupancy calculation limit	0
Occupancy indication limit	0.1

Table 31 *Modulation commands of DIST in the `m50` file*

Name	Syntax and Description
tzero	**tzero** `number` **Default value**: **tzero** 0 The initial value of `t` . The default value is 0. This command has no effect for commensurate structures.
nooft	**nooft** `number1` **Default value**: **nooft** 100 The number of equidistant `t` values used for calculation of distances and angles. The distances and angles are calculated for `number1` of `t` but only each `number2`th of them is printed to the listing. `Number2` is defined by **each** command. Implicitly `number1`=100, `number2`=10. This command has no effect for commensurate structures.
each	**each** `number2` **Default value**: **each** 10 The step for printing of the distances and angles as a function of `t`. The distances and angles are calculated for `number1` of `t` but only each `number2`th of them is printed to the listing. `Number1` is defined by **nooft** command. Implicitly `number1`=100, `number2`=10. This command has no effect for commensurate structures.
ttables	**ttables** `flag` **Default value**: **ttables** 0 Sets output of the comprehensive table of coordination as a function of the `t` coordinate. This command is especially useful for composites. `flag` = 0 off `flag` = 1 on
occut	**occut** `number` **Default value**: **occut** 0 The lower limit of occupation for which the distances will be calculated. This command is useful for structures with occupation modulation.
occind	**occind** `number` **Default value**: **occind** 0.1 The distances of atoms with occupation less than `number` will be indicated in the *DIST* listing by "`*`". This command is useful for structures with occupation modulation.

4.2.6 Dist listing of an ordinary structure

Figure 184 Simple DIST Listing for Ordinary Structure

```
Calculating of distances, angles, torsion angles and best planes                                         page =  1
structure : cro2h2o                                                                                09:03:54 07-Mar-97

Non-centrosymmetric space group : P63
Wave length :   0.71073
Cell parameters       :    5.5000   5.5000  14.1630    90.00    90.00   120.00    Volume :      371.0
List of centring vectors :
  0.000000  0.000000  0.000000
Symmetry operators :
    x     y     z
  x-y     x 1/2+z
   -y   x-y     z
   -x    -y 1/2+z
 -x+y    -x     z
    y  -x+y 1/2+z
Atomic scattering tables :
Fe    7.200    0.301    0.845        25.990   25.310   23.683   21.819   20.045   18.363   16.748   15.228
                                     13.838   12.596   11.506   10.563    9.758    9.079    8.510    8.036
                                      7.640    7.307    7.022    6.773    6.549    6.342    6.147    5.959
                                      5.776    5.596    5.418    0.000    0.000    0.000    0.000    0.000
Si    2.800    0.072    0.071        13.998   13.436   12.134   10.767    9.675    8.861    8.230    7.696
                                      7.201    6.718    6.240    5.770    5.314    4.879    4.472    4.095
                                      3.750    3.439    3.162    2.915    2.699    2.510    2.345    2.202
                                      2.077    1.969    1.875    0.000    0.000    0.000    0.000    0.000
O    18.000    0.008    0.006         7.999    7.798    7.246    6.472    5.623    4.808    4.089    3.489
                                      3.006    2.628    2.337    2.115    1.946    1.816    1.715    1.634
                                      1.568    1.512    1.463    1.419    1.377    1.337    1.298    1.260
                                      1.221    1.183    1.145    0.000    0.000    0.000    0.000    0.000
H     8.000    0.000    0.000         1.000    0.960    0.854    0.713    0.568    0.438    0.331    0.248
                                      0.184    0.138    0.103    0.078    0.060    0.046    0.036    0.028
                                      0.022    0.018    0.014    0.011    0.009    0.007    0.006    0.005
                                      0.005    0.004    0.004    0.000    0.000    0.000    0.000    0.000
```

Basic Crystal information

```
The following lines were read as a control data :
=>dmin 0 dmax 3<=
=>end<=
```

The *DIST* commands

```
The atom Si1fe    deleted from the distance  calculation as coincides from with : Si1
The atom Si2fe    deleted from the distance  calculation as coincides from with : Si2
```

List of atoms which will be used for the calculation.

Atoms with zero occupation or the ones coinciding with other atoms are not used.
Atoms of molecules (if present) are expanded to molecular positions and their names are extended by the discriminator characters (see § 1.2.2 page A-14) .

```
                                     fractional                                      cartesian
 no  atom        xf        yf        zf      sigxf     sigyf     sigzf       x         y         z       Uiso
  1  Fe1     +  0.332516  0.000384  0.000000  0.000000  0.000000  0.000000   1.8278    0.0018    0.0000   0.0051(  0)
  2  Si1     +  0.000000  0.000000  0.302204  0.000000  0.000000  0.000000   0.0000    0.0000    4.2801   0.0060(  0)
  4  Si2     +  0.333333  0.666667  0.300307  0.000000  0.000000  0.000000   0.0000    3.1754    4.2532   0.0054(  0)
  6  O1      +  0.336187  0.336928  0.072867  0.000000  0.000000  0.000000   0.9225    1.6048    1.0320   0.0096(  0)
  7  O2      +  0.000000  0.000000  0.426187  0.000000  0.000000  0.000000   0.0000    0.0000    6.0361   0.0081(  0)
  8  O3      +  0.333333  0.666667  0.426735  0.000000  0.000000  0.000000   0.0000    3.1754    6.0438   0.0095(  0)
  9  O4      +  0.333333 -0.333333 -0.072572  0.000000  0.000000  0.000000   2.7500   -1.5877   -1.0278   0.0105(  0)
 10  O5      + -0.328397 -0.212873  0.263519  0.000000  0.000000  0.000000  -1.2208   -1.0139    3.7322   0.0215(  0)
 11  O5'     + -0.327218 -0.080768  0.268809  0.000000  0.000000  0.000000  -1.5776   -0.3847    3.8071   0.0215(  0)
```

Figure 184 *(Continued)*

```
Full coordination of atoms will be printed
Distances will be calculated
for atom : Fe from  0.000 to  3.000
for atom : Si from  0.000 to  3.000
for atom : O  from  0.000 to  3.000
for atom : H  from  0.000 to  3.000
```

Interpretation of the control commands

1st page of distances printed in newspaper columns

```
Calculating of distances, angles, torsion angles and best planes                       page =  2
structure : cro2h2o                                                              09:03:54 07-Mar-97

                               O5........... 1.7144(   0)    O5'.......... 2.7577(   0)    * atom O5                 *
                               O5'.......... 1.7380(   0)    O5'.......... 2.7577(   0)    ***************************
                               O5'.......... 1.7380(   0)    *                             Si1.......... 1.6789(   0)
                               O5'.......... 1.7380(   0)    ***************************   Si2.......... 1.7144(   0)
***************************    *                             * atom O3                 *   O1........... 2.9512(   0)
* atom Fe1                *    ***************************   ***************************   O2........... 2.7975(   0)
***************************    * atom O1                 *   Fe1.......... 2.1077(   0)    O3........... 2.8304(   0)
O1........... 2.1105(   0)     ***************************   Fe1.......... 2.1077(   0)    O5........... 2.8290(   0)
O1........... 2.0932(   0)     Fe1.......... 2.1105(   0)    Fe1.......... 2.1077(   0)    O5........... 2.7487(   0)
O1........... 2.1080(   0)     Fe1.......... 2.1080(   0)    Si2.......... 1.7906(   0)    O5........... 2.8290(   0)
O2........... 2.1056(   0)     Fe1.......... 2.0932(   0)    O1........... 2.7611(   0)    O5........... 2.7487(   0)
O3........... 2.1077(   0)     O2........... 2.7825(   0)    O1........... 2.7611(   0)    O5'.......... 0.7272(   0)
O4........... 2.1056(   0)     O3........... 2.7611(   0)    O1........... 2.7611(   0)    O5'.......... 2.3494(   0)
*                              O4........... 2.7497(   0)    O5........... 2.8304(   0)    O5'.......... 2.4407(   0)
***************************    O5........... 2.9512(   0)    O5........... 2.8304(   0)    *
* atom Si1                *    O5'.......... 2.8145(   0)    O5........... 2.8304(   0)    ***************************
***************************    *                             O5'.......... 2.7973(   0)    * atom O5'                *
O2........... 1.7560(   0)     ***************************   O5'.......... 2.7973(   0)    ***************************
O5........... 1.6789(   0)     * atom O2                 *   O5'.......... 2.7973(   0)    Si1.......... 1.6913(   0)
O5........... 1.6789(   0)     ***************************   *                             Si2.......... 1.7380(   0)
O5........... 1.6789(   0)     Fe1.......... 2.1056(   0)    ***************************   O1........... 2.8145(   0)
O5'.......... 1.6913(   0)     Fe1.......... 2.1056(   0)    * atom O4                 *   O2........... 2.7577(   0)
O5'.......... 1.6913(   0)     Fe1.......... 2.1056(   0)    ***************************   O3........... 2.7973(   0)
O5'.......... 1.6913(   0)     Si1.......... 1.7560(   0)    Fe1.......... 2.1056(   0)    O5........... 0.7272(   0)
*                              O1........... 2.7825(   0)    Fe1.......... 2.1056(   0)    O5........... 2.4407(   0)
***************************    O1........... 2.7825(   0)    Fe1.......... 2.1056(   0)    O5........... 2.3494(   0)
* atom Si2                *    O1........... 2.7825(   0)    O1........... 2.7497(   0)    O5'.......... 2.9095(   0)
***************************    O5........... 2.7975(   0)    O1........... 2.7497(   0)    O5'.......... 2.8125(   0)
O3........... 1.7906(   0)     O5........... 2.7975(   0)    O1........... 2.7497(   0)    O5'.......... 2.9095(   0)
O5........... 1.7144(   0)     O5........... 2.7975(   0)    *                             O5'.......... 2.8125(   0)
O5........... 1.7144(   0)     O5'.......... 2.7577(   0)    ***************************   *
```

One column in detail:

```
***************************
* atom Fe1                *
***************************
O1........... 2.1105(   0)
O1........... 2.0932(   0)
O1........... 2.1080(   0)
O2........... 2.1056(   0)
O3........... 2.1077(   0)
O4........... 2.1056(   0)
*
```

central atom

The neighbouring atoms found between the distances limits (atom name , distance in Å, e.s.d if present in the m40 file)

Figure 185 DIST listing of an ordinary structure with angles

The beginning of the listing is similar like in the previous example.

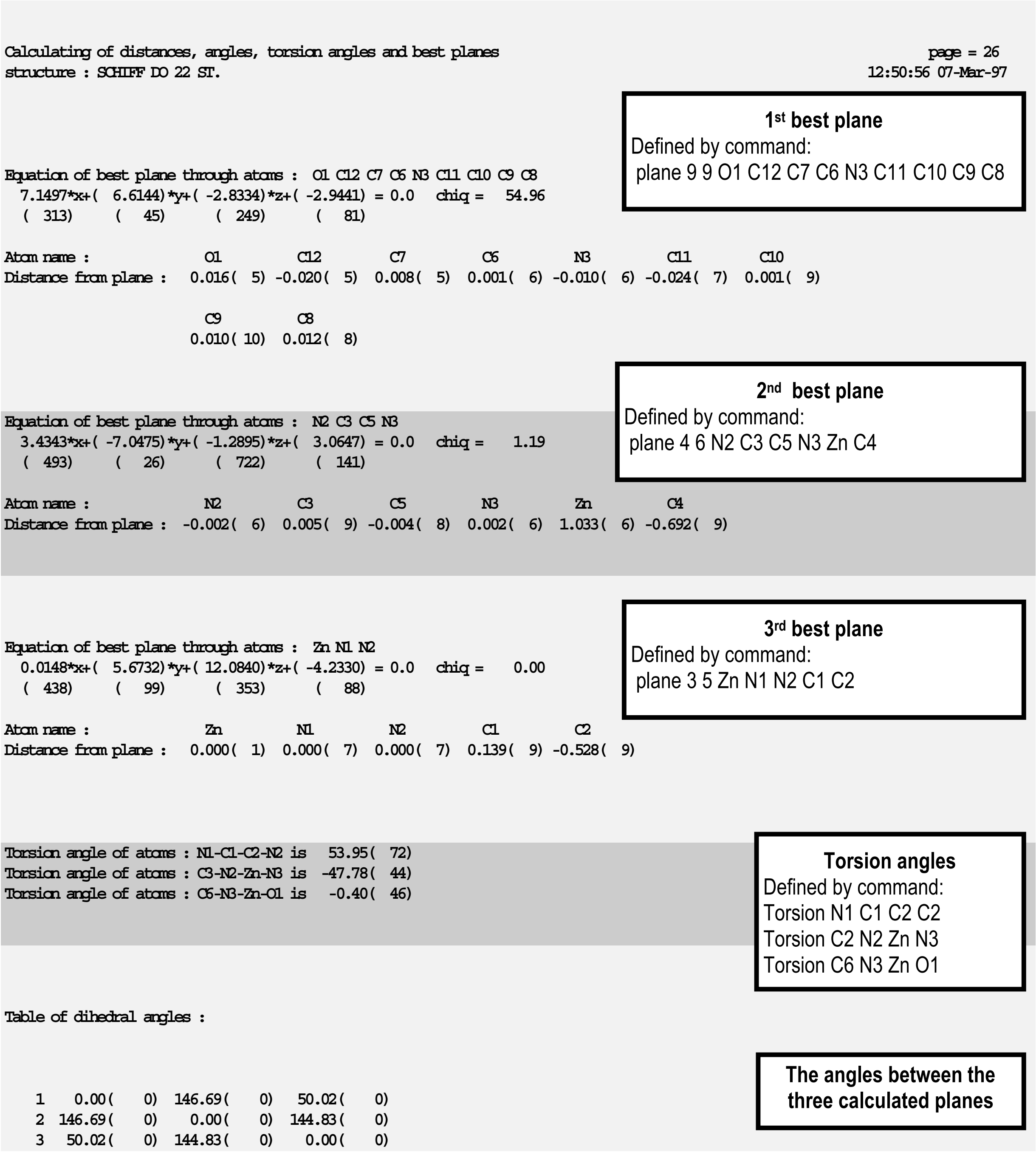
```
**************************      C6.........  68.52(  19)      C1.........  53.80(  20)      H1(n2).....  82.81(  18)
* atom Zn                *      C7.........  43.95(  16)      C2.........  29.58(  21)    C1........... 2.9798(  69)
**************************      C12........  19.48(  15)      C3.........  26.03(  21)      C2.........  28.89(  20)
Br........... 2.4811(   8)      H1(n1).....  96.09(  15)      C4.........  45.87(  20)      C3.........  76.63(  22)
  O1......... 121.44(  12)      H1(n1).....  39.67(  14)      C5.........  72.45(  20)      C4.........  99.64(  20)
  N1.........  96.54(  13)      H2(n1).....  68.01(  15)      C6......... 102.17(  20)      C5......... 126.10(  20)
  N2......... 107.84(  13)      H1(c1).....  92.87(  17)      C7......... 116.76(  17)      C6......... 142.84(  17)
  N3.........  98.18(  13)      H1(c1).....  48.30(  17)      C12........ 127.91(  16)      C7......... 134.73(  17)
  C1......... 106.21(  14)      H1(c2)..... 141.98(  18)      H1(n1).....  89.92(  18)      C12........ 119.29(  18)
  C2.........  97.26(  15)      H1(c3)..... 161.87(  18)      H1(n1).....  90.80(  15)      H1(n1).....  41.26(  17)
  C3.........  90.50(  13)      H1(c5)..... 124.21(  18)      H2(n1).....  93.26(  18)      H1(n1).....  78.07(  16)
  C4.........  95.48(  11)      H1(n2)..... 110.46(  16)      H1(c1).....  53.19(  19)      H2(n1).....  41.23(  17)
  C5.........  84.73(  13)    N1........... 2.1213(  45)      H1(c1)..... 161.00(  19)      H1(c1).....  16.21(  19)
  C6......... 108.46(  11)      N2.........  81.05(  19)      H1(c2).....  40.00(  19)      H1(c1)..... 107.20(  18)
  C7......... 117.74(  10)      N3......... 164.05(  18)      H1(c3).....  38.59(  19)      H1(c2).....  38.23(  20)
  C12........ 121.51(  10)      C1.........  27.41(  19)      H1(c5).....  78.09(  19)      H1(c3).....  79.83(  21)
  H1(n1).....  76.56(  11)      C2.........  53.89(  19)      H1(n2).....  20.53(  16)      H1(c5)..... 127.77(  20)
  H1(n1)..... 159.72(   8)      C3......... 102.28(  21)    N3........... 2.0810(  48)      H1(n2).....  56.92(  18)
  H2(n1)..... 108.08(  11)      C4......... 126.66(  19)      C1......... 139.73(  19)    C2........... 2.9340(  76)
  H1(c1)..... 122.41(  13)      C5......... 152.44(  19)      C2......... 117.77(  21)      C3.........  48.44(  22)
  H1(c1).....  75.06(  13)      C6......... 152.09(  19)      C3.........  71.53(  22)      C4.........  73.11(  20)
  H1(c2).....  79.61(  14)      C7......... 130.21(  17)      C4.........  45.64(  20)      C5.........  98.58(  21)
  H1(c3).....  73.21(  13)      C12........ 107.10(  17)      C5.........  25.33(  21)      C6......... 131.49(  20)
  H1(c5).....  67.23(  12)      H1(n1).....  20.17(  17)      C6.........  20.92(  22)      C7......... 141.36(  17)
  H1(n2)..... 128.08(  11)      H1(n1).....  78.13(  15)      C7.........  45.49(  19)      C12........ 139.82(  18)
O1........... 1.9574(  39)      H2(n1).....  20.19(  16)      C12........  69.99(  19)      H1(n1).....  60.39(  18)
  N1.........  88.11(  16)      H1(c1).....  35.07(  18)      H1(n1)..... 173.81(  18)      H1(n1).....  95.24(  16)
  N2......... 130.43(  17)      H1(c1).....  79.95(  17)      H1(n1).....  89.99(  16)      H2(n1).....  70.03(  18)
  N3.........  89.44(  18)      H1(c2).....  56.34(  19)      H2(n1)..... 151.65(  17)      H1(c1).....  36.76(  19)
  C1......... 103.83(  18)      H1(c3)..... 101.39(  20)      H1(c1)..... 129.45(  18)      H1(c1)..... 132.42(  19)
  C2......... 129.53(  18)      H1(c5)..... 147.66(  19)      H1(c1)..... 109.81(  19)      H1(c2).....  17.65(  20)
  C3......... 145.33(  18)      H1(n2).....  83.29(  18)      H1(c2)..... 120.69(  20)      H1(c3).....  51.15(  22)
  C4......... 127.12(  17)    N2........... 2.1581(  48)      H1(c3).....  77.16(  22)      H1(c5).....  98.89(  20)
  C5......... 114.77(  18)      N3.........  88.60(  20)      H1(c5).....  38.21(  19)      H1(n2).....  42.43(  19)
```

One column in detail:

```
**************************
* atom Zn                *      The central atom
**************************
Br........... 2.4811(   8)      The Zn-Br distance (Å) and its e.s.d.
  O1......... 121.44(  12)      The Br-Zn-O1 angle (°) and its e.s.d.
  N1.........  96.54(  13)      Another Br-Zn-* angles
  N2......... 107.84(  13)
  N3.........  98.18(  13)
  C1......... 106.21(  14)
  C2.........  97.26(  15)
  C3.........  90.50(  13)
  C4.........  95.48(  11)
  C5.........  84.73(  13)
  C6......... 108.46(  11)
  C7......... 117.74(  10)
  C12........ 121.51(  10)
  H1(n1).....  76.56(  11)
  H1(n1)..... 159.72(   8)
```

Figure 186 *DIST listing with best planes and torsion angles*

This information is printed at the end of the listing. It is the same for both ordinary and modulated structures.



```
Calculating of distances, angles, torsion angles and best planes                                    page = 26
structure : SCHIFF DO 22 ST.                                                                  12:50:56 07-Mar-97


Equation of best plane through atoms :  O1 C12 C7 C6 N3 C11 C10 C9 C8
  7.1497*x+(  6.6144)*y+( -2.8334)*z+( -2.9441) = 0.0   chiq =   54.96
  (  313)     (   45)      (  249)      (   81)

Atom name :              O1          C12         C7          C6          N3          C11         C10
Distance from plane :  0.016(  5) -0.020(  5)  0.008(  5)  0.001(  6) -0.010(  6) -0.024(  7)  0.001(  9)

                         C9          C8
                       0.010( 10)  0.012(  8)


Equation of best plane through atoms :  N2 C3 C5 N3
  3.4343*x+( -7.0475)*y+( -1.2895)*z+(  3.0647) = 0.0   chiq =    1.19
  (  493)     (   26)      (  722)      (  141)

Atom name :              N2          C3          C5          N3          Zn          C4
Distance from plane : -0.002(  6)  0.005(  9) -0.004(  8)  0.002(  6)  1.033(  6) -0.692(  9)


Equation of best plane through atoms :  Zn N1 N2
  0.0148*x+(  5.6732)*y+( 12.0840)*z+( -4.2330) = 0.0   chiq =    0.00
  (  438)     (   99)      (  353)      (   88)

Atom name :              Zn          N1          N2          C1          C2
Distance from plane :  0.000(  1)  0.000(  7)  0.000(  7)  0.139(  9) -0.528(  9)


Torsion angle of atoms : N1-C1-C2-N2 is   53.95(  72)
Torsion angle of atoms : C3-N2-Zn-N3 is  -47.78(  44)
Torsion angle of atoms : C6-N3-Zn-O1 is   -0.40(  46)


Table of dihedral angles :


    1    0.00(    0)  146.69(    0)   50.02(    0)
    2  146.69(    0)    0.00(    0)  144.83(    0)
    3   50.02(    0)  144.83(    0)    0.00(    0)
```

1st best plane
Defined by command:
plane 9 9 O1 C12 C7 C6 N3 C11 C10 C9 C8

2nd best plane
Defined by command:
plane 4 6 N2 C3 C5 N3 Zn C4

3rd best plane
Defined by command:
plane 3 5 Zn N1 N2 C1 C2

Torsion angles
Defined by command:
Torsion N1 C1 C2 C2
Torsion C2 N2 Zn N3
Torsion C6 N3 Zn O1

The angles between the three calculated planes

4.2.7 Dist listing of an incommensurate structure

Figure 187 *DIST Listing of a modulated structure*

The beginning of the listing is similar like in the previous examples.

```
**************************
* atom K                 *
**************************
F1a.......... 3.1217(  23)
 ave          3.1331
 min          2.9227
 max          3.3414
 0.000        3.3336
 0.100        3.2964
 0.200        3.1124
 0.300        2.9471
 0.400        2.9413
 0.500        3.0480
 0.600        3.1262
 0.700        3.1300
 0.800        3.1421
 0.900        3.2340
 1.000        3.3336
--------------------------
F1a.......... 2.7594(  26)
 ave          2.7629
 min          2.7041
 max          2.8149
 0.000        2.8138
 0.100        2.7960
 0.200        2.7753
 0.300        2.7657
 0.400        2.7498
 0.500        2.7198
 0.600        2.7041
 0.700        2.7243
 0.800        2.7688
 0.900        2.8061
 1.000        2.8138
--------------------------
F1a.......... 2.7594(  26)
 ave          2.7624
 min          2.7041
 max          2.8149
 0.000        2.7656
 0.100        2.7751
 0.200        2.7956
 0.300        2.8137
 0.400        2.8065
 0.500        2.7695
 0.600        2.7249
 0.700        2.7041
 0.800        2.7193
 0.900        2.7494
 1.000        2.7656
--------------------------
F1a.......... 3.1217(  23)
 ave          3.1293
 min          2.9227
 max          3.3414
 0.000        2.9457
 0.100        3.1091
 0.200        3.2944
 0.300        3.3343
 0.400        3.2358
 0.500        3.1429
 0.600        3.1298
 0.700        3.1268
 0.800        3.0497
 0.900        2.9425
 1.000        2.9457
--------------------------
F2a.......... 2.8314(  25)
 ave          2.8402
 min          2.7629
 max          2.9260
 0.000        2.7661
 0.100        2.7669
 0.200        2.8013
 0.300        2.8589
 0.400        2.9075
 0.500        2.9260
 0.600        2.9086
 0.700        2.8658
 0.800        2.8211
 0.900        2.7871
 1.000        2.7661
--------------------------
F2a.......... 2.7049(  26)
 ave          2.7111
 min          2.6703
 max          2.7523
 0.000        2.7100
 0.100        2.6737
 0.200        2.6799
 0.300        2.7124
 0.400        2.7250
 0.500        2.7130
 0.600        2.6962
 0.700        2.7088
 0.800        2.7443
 0.900        2.7475
 1.000        2.7100
--------------------------
F2a.......... 2.8314(  25)
 ave          2.8411
 min          2.7629
 max          2.9260
 0.000        2.8598
 0.100        2.8021
 0.200        2.7671
 0.300        2.7659
 0.400        2.7866
 0.500        2.8205
 0.600        2.8650
 0.700        2.9081
 0.800        2.9260
 0.900        2.9080
 1.000        2.8598
--------------------------
F2a.......... 2.7049(  26)
 ave          2.7111
 min          2.6703
 max          2.7522
 0.000        2.7128
 0.100        2.6804
 0.200        2.6734
 0.300        2.7093
 0.400        2.7471
 0.500        2.7447
 0.600        2.7093
 0.700        2.6960
 0.800        2.7127
 0.900        2.7250
 1.000        2.7128
--------------------------
O3a.......... 3.2621(  28)
 ave          3.2763
 min          2.9282
 max          3.6887
 0.000        3.6789
 0.100        3.6382
 0.200        3.4357
 0.300        3.1978
 0.400        3.0205
 0.500        2.9355
 0.600        2.9474
 0.700        3.0655
 0.800        3.2789
 0.900        3.5245
 1.000        3.6789
--------------------------
O3a.......... 3.5183(  30)
 ave          3.5207
 min          2.9332
 max          4.0957
 0.000        2.9469
 0.100        2.9867
 0.200        3.2179
 0.300        3.5357
 0.400        3.8441
 0.500        4.0522
 0.600        4.0831
 0.700        3.8995
 0.800        3.5404
 0.900        3.1581
 1.000        2.9469
--------------------------
O3a.......... 3.2621(  28)
 ave          3.2715
 min          2.9282
 max          3.6887
 0.000        3.1945
 0.100        3.4319
 0.200        3.6361
 0.300        3.6798
 0.400        3.5280
 0.500        3.2827
 0.600        3.0682
 0.700        2.9484
 0.800        2.9349
 0.900        3.0185
 1.000        3.1945
--------------------------
O3a.......... 3.5183(  30)
 ave          3.5266
 min          2.9332
 max          4.0957
 0.000        3.5408
 0.100        3.2224
 0.200        2.9891
 0.300        2.9456
 0.400        3.1531
 0.500        3.5341
 0.600        3.8950
 0.700        4.0819
 0.800        4.0542
 0.900        3.8483
 1.000        3.5408
--------------------------
*
**************************
* atom Asa               *
**************************
F1a.......... 1.7332(  23)
 ave          1.7392
 min          1.7337
 max          1.7436
 0.000        1.7397
 0.100        1.7429
 0.200        1.7401
 0.300        1.7381
 0.400        1.7377
 0.500        1.7390
 0.600        1.7432
 0.700        1.7417
 0.800        1.7353
 0.900        1.7343
 1.000        1.7397
--------------------------
F1a.......... 1.7332(  23)
 ave          1.7392
 min          1.7337
 max          1.7436
 0.000        1.7404
 0.100        1.7436
 0.200        1.7398
 0.300        1.7377
 0.400        1.7380
 0.500        1.7394
 0.600        1.7427
 0.700        1.7409
 0.800        1.7351
 0.900        1.7345
 1.000        1.7404
--------------------------
F2a.......... 1.7258(  24)
 ave          1.7447
 min          1.7260
 max          1.7665
 0.000        1.7563
 0.100        1.7653
 0.200        1.7451
 0.300        1.7286
 0.400        1.7305
 0.500        1.7438
 0.600        1.7589
 0.700        1.7556
 0.800        1.7323
 0.900        1.7298
 1.000        1.7563
--------------------------
F2a.......... 1.7258(  24)
```

One column in detail:

```
**************************
* atom K                 *     Central atom
**************************
F1a......... 3.1217(  23)      The distance K-F1a calculated from the basic structure[1]
 ave         3.1331                    The average K-F1a distance for all used t values
 min         2.9227                    The minimum K-F1a distance for all used t values
 max         3.3414                    The maximum K-F1a distance for all used t values
 0.000       3.3336                    The K-F1a distance for t=0
 0.100       3.2964                    The K-F1a distance for t=0.1
 0.200       3.1124            etc.
 0.300       2.9471
 0.400       2.9413            The distances are calculated for 100 equidistant t values. This
 0.500       3.0480            setting can be changed by the nooft control command
 0.600       3.1262
 0.700       3.1300            The distances are printed for each 10th t value. This setting can
 0.800       3.1421            be changed by each control command
 0.900       3.2340
 1.000       3.3336
--------------------------
```

Figure 188 *DIST listing of a modulated structure with angles*
(one column in detail)

```
**************************
* atom K                 *     central atom
**************************
F1a......... 3.1217(  23)      The distances for the first atom of the K coordination
 ave         3.1331            (see previous example for explanation)
 min         2.9227
 max         3.3414
 0.000       3.3336
 0.100       3.2964
 0.200       3.1124
 0.300       2.9471
 0.400       2.9413
 0.500       3.0480
 0.600       3.1262
 0.700       3.1300
 0.800       3.1421
 0.900       3.2340
 1.000       3.3336
--------------------------
  F1a....... 109.86(   6)      The angle F1a-K-F1a calculated from refined coordinates
   ave       109.78            The average F1a-K-F1a angle for all used t values
   min       103.80            The minimum F1a-K-F1a angle for all used t values
   max       115.78            The maximum F1a-K-F1a angle for all used t values
   0.000     104.17
   0.100     104.10            The t grid and complexity of output is controlled by the same
   0.200     106.71            commands as used for distances - see previous example for
   0.300     110.92            explanation
                                       . . . . . .
   1.000     104.17
```

---

[1] The atomic positions in the **basic structure** are the ones refined in the `m40`. The distances in the basic structure don't correspond to any 3d section but they should be close to "ave" distances.

4.2.8 Using *DIST* for commensurate structures

In the case of commensurate structures the modulation function is defined only in discrete points corresponding to the atomic positions in the superstructure and can have arbitrary values between these points. *DIST* automatically chooses the `t` values where the modulation function is defined.

The structure in the following example has q-vector of (0,0,γ) type, the relation between the basic cell and the supercell is 1,1,4 (i.e. the **c** axis of the supercell is four times larger), and the `t` value used for the refinement is 0.188. The corresponding keywords in `m50` are `commen` and `tzero` (see Table 9, page 80). Note: the `tzero` command of *DIST* (see modulation commands, page 316) has no effect for commensurate refinement.

Figure 189 *DIST Listing for a commensurately modulated structure*

```
***************************
* atom Ca                 *
***************************
C1.......... 2.7561( 53)
 ave         2.6851
 min         2.6398
 max         2.7235
 0.188       2.6760
 0.438       2.7011
 0.688       2.7235
 0.938       2.6398
-------------------------
C1.......... 2.7561( 53)
 ave         2.6851
 min         2.6398
 max         2.7235
 0.188       2.7235
 0.438       2.6398
 0.688       2.6760
 0.938       2.7011
-------------------------
O1.......... 2.2480( 90)
 ave         2.2614
 min         2.1873
 max         2.3356
 0.188       2.3356
 0.438       2.1873
 0.688       2.3356
 0.938       2.1873
-------------------------
O2.......... 2.2432( 90)
 ave         2.2414
 min         2.2099
 max         2.2728
 0.188       2.2099
 0.438       2.2728
 0.688       2.2099
 0.938       2.2728
-------------------------
O3.......... 2.3319( 69)
 ave         2.3874
 min         2.3534
 max         2.4408
 0.188       2.3534
 0.438       2.4408
 0.688       2.3705
 0.938       2.3851
-------------------------
O3.......... 2.3319( 69)
 ave         2.3874
 min         2.3534
 max         2.4408
 0.188       2.3705
 0.438       2.3851
 0.688       2.3534
 0.938       2.4408
-------------------------
D7.......... 3.0034( 65)
 ave         3.0124
 min         2.9580
 max         3.0539
 0.188       3.0539
 0.438       3.0352
 0.688       2.9580
 0.938       3.0026
-------------------------
D7.......... 3.0034( 65)
 ave         3.0124
 min         2.9580
 max         3.0539
 0.188       2.9580
 0.438       3.0026
 0.688       3.0539
 0.938       3.0352
-------------------------
D8.......... 2.9725( 57)
 ave         3.0322
 min         2.9581
 max         3.1082
 0.188       2.9581
 0.438       3.1082
 0.688       3.0285
 0.938       3.0341
-------------------------
D8.......... 2.9725( 57)
 ave         3.0322
 min         2.9581
 max         3.1082
 0.188       3.0285
 0.438       3.0341
 0.688       2.9581
 0.938       3.1082
-------------------------
*
***************************
* atom N                  *
***************************
O1.......... 2.8399( 64)
 ave         2.7877
 min         2.7607
 max         2.8148
 0.188       2.7607
 0.438       2.8148
 0.688       2.7607
 0.938       2.8148
-------------------------
C1.......... 1.4855( 65)
 ave         1.4702
 min         1.4207
 max         1.5198
 0.188       1.4207
 0.438       1.5198
 0.688       1.4207
 0.938       1.5198
-------------------------
C3.......... 1.4453( 65)
 ave         1.4961
 min         1.4847
 max         1.5075
 0.188       1.4847
 0.438       1.5075
 0.688       1.4847
 0.938       1.5075
-------------------------
C4.......... 2.5439( 60)
 ave         2.5619
 min         2.5125
 max         2.6113
 0.188       2.5125
 0.438       2.6113
 0.688       2.5125
 0.938       2.6113
-------------------------
D1.......... 2.0969( 41)
 ave         2.1271
 min         2.0924
 max         2.1691
 0.188       2.1035
 0.438       2.0924
 0.688       2.1436
 0.938       2.1691
-------------------------
D1.......... 2.0969( 41)
 ave         2.1271
 min         2.0924
 max         2.1691
 0.188       2.1436
 0.438       2.1691
 0.688       2.1035
 0.938       2.0924
-------------------------
D2.......... 2.0677( 49)
 ave         2.1299
 min         2.0899
 max         2.1700
 0.188       2.1700
 0.438       2.0899
 0.688       2.1700
 0.938       2.0899
-------------------------
D6.......... 2.0423( 42)
 ave         2.1482
 min         2.0704
 max         2.1910
 0.188       2.0704
 0.438       2.1623
 0.688       2.1689
 0.938       2.1910
-------------------------
D6.......... 2.0423( 42)
 ave         2.1482
 min         2.0704
 max         2.1910
 0.188       2.1689
 0.438       2.1910
 0.688       2.0704
 0.938       2.1623
-------------------------
C2a......... 1.4734( 44)
 ave         1.4751
```

4.2.9 Using *DIST* for composite structures

In composite crystals two or more structures coexist with different periodicities. The influence on distances calculation is that a distance between atoms from different composite parts calculated like function of `t` has no upper limit. This feature is clearly visible in the `t` plots (see page 337).

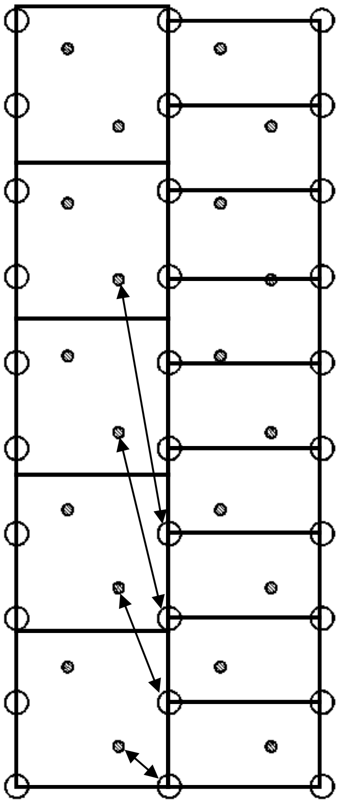


Figure 190 Example of a composite structure.

In this example the small shaded rings denote atoms belonging to the first composite part with larger cell. The second composite part with smaller cell comprises atoms indicated by large white rings. The cell parameters along the vertical direction are incommensurate.
The double arrow shows distances between one pair of atoms from different composite parts for various `t` values.

Like for other modulated structures *DIST* prints in the listing the minimum and maximum distance found in the `t` interval used for calculation. If the `t` function of distances does not have minimum in this interval *DIST* prints the value with a “<” sign.

Figure 191 *DIST listing of a composite structure*

```
Calculating of distances, angles, torsion angles and best planes                                        page =  1
structure : Beznix - incommen                                                                      11:28:04 18-04-99

The following lines were read as a control data :
=>fullcoor 1 each 5 ttables 1<=
=>select ni1 sr<=
=>end<=
```

DIST **commands for this run. The atoms (Ni1 and Sr) are selected as the central ones. They are from different composite parts.**

```
Centrosymmetric super-space group : R-3m(00g)0s

Wave length :   0.71073

Composite part #1

           | 1.000  0.000  0.000  0.000|
    W(1) = | 0.000  1.000  0.000  0.000|
           | 0.000  0.000  1.000  0.000|
           | 0.000  0.000  0.000  1.000|
```

1st composite part: cell parameters and symmetry. Ni1 is from this part.

```
Cell parameters        :    9.4985   9.4985   2.5688    90.00    90.00   120.00    Volume :      200.7
Modulation vector q(1) :   0.00000  0.00000  0.64359

List of centring vectors :

  0.000000  0.000000  0.000000  0.000000
  0.666667  0.333333  0.333333  0.000000
  0.333333  0.666667  0.666667  0.000000

Symmetry operators :

     x1     x2     x3     x4
    -x2  x1-x2     x3     x4
    -x2    -x1     x3 1/2+x4
 -x1+x2    -x1     x3     x4
     x1  x1-x2     x3 1/2+x4
 -x1+x2     x2     x3 1/2+x4

Composite part #2

           | 1.000  0.000  0.000  0.000|
    W(2) = | 0.000  1.000  0.000  0.000|
           | 0.000  0.000  0.000  1.000|
           | 0.000  0.000  1.000  0.000|
```

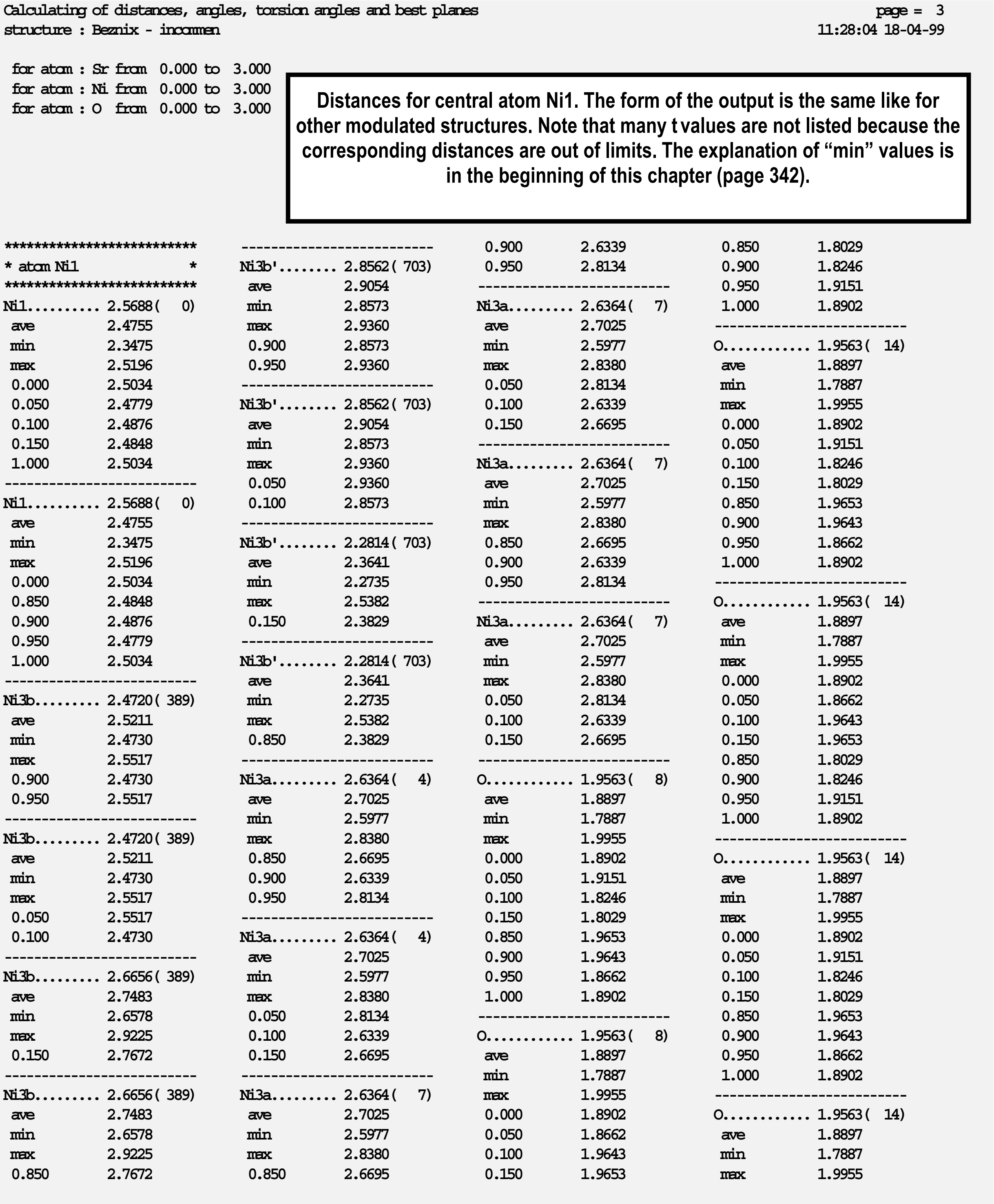
2nd composite part: cell parameters and symmetry. Sr is from this part.

```
Cell parameters        :    9.4985   9.4985   3.9914    90.00    90.00   120.00    Volume :      311.9
Modulation vector q(1) :   0.00000  0.00000  1.55378

List of centring vectors :

  0.000000  0.000000  0.000000  0.000000
  0.666667  0.333333  0.000000  0.333333
  0.333333  0.666667  0.000000  0.666667

Calculating of distances, angles, torsion angles and best planes                                        page =  2
structure : Beznix - incommen                                                                      11:28:04 18-04-99

Symmetry operators :

     x1     x2     x3     x4
    -x2  x1-x2     x3     x4
    -x2    -x1 1/2+x3     x4
 -x1+x2    -x1     x3     x4
     x1  x1-x2 1/2+x3     x4
 -x1+x2     x2 1/2+x3     x4
```

. . . .

```
Full coordination of atoms will be printed
Distances will be calculated
 at 101 equidistant values of t from the interval <0,1> but only each  5th value will be printed
```

Figure 191 *(Continued)*



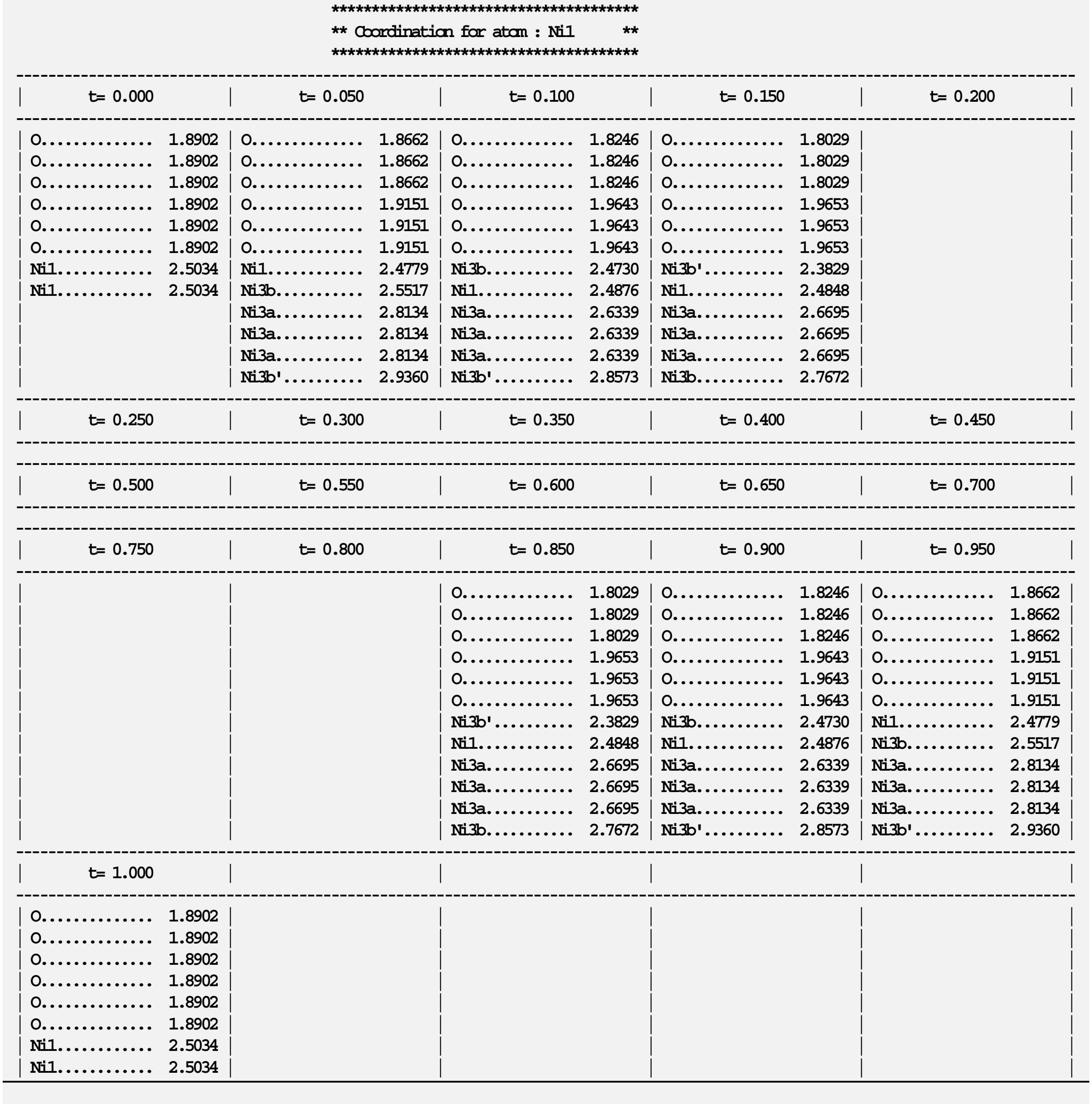
```
Calculating of distances, angles, torsion angles and best planes                                page =  3
structure : Beznix - incommen                                                               11:28:04 18-04-99

 for atom : Sr from  0.000 to  3.000
 for atom : Ni from  0.000 to  3.000
 for atom : O  from  0.000 to  3.000
```

Distances for central atom Ni1. The form of the output is the same like for other modulated structures. Note that many t values are not listed because the corresponding distances are out of limits. The explanation of “min” values is in the beginning of this chapter (page 342).

```
**************************    -------------------------      0.900        2.6339            0.850        1.8029
* atom Ni1               *    Ni3b'........ 2.8562( 703)     0.950        2.8134            0.900        1.8246
**************************     ave          2.9054          -------------------------       0.950        1.9151
Ni1.......... 2.5688(   0)     min          2.8573          Ni3a......... 2.6364(   7)      1.000        1.8902
 ave          2.4755           max          2.9360           ave          2.7025           -------------------------
 min          2.3475           0.900        2.8573           min          2.5977           O............ 1.9563(  14)
 max          2.5196           0.950        2.9360           max          2.8380            ave          1.8897
 0.000        2.5034          -------------------------      0.050        2.8134            min          1.7887
 0.050        2.4779          Ni3b'........ 2.8562( 703)     0.100        2.6339            max          1.9955
 0.100        2.4876           ave          2.9054           0.150        2.6695            0.000        1.8902
 0.150        2.4848           min          2.8573          -------------------------       0.050        1.9151
 1.000        2.5034           max          2.9360          Ni3a......... 2.6364(   7)      0.100        1.8246
-------------------------      0.050        2.9360           ave          2.7025            0.150        1.8029
Ni1.......... 2.5688(   0)     0.100        2.8573           min          2.5977            0.850        1.9653
 ave          2.4755          -------------------------      max          2.8380            0.900        1.9643
 min          2.3475          Ni3b'........ 2.2814( 703)     0.850        2.6695            0.950        1.8662
 max          2.5196           ave          2.3641           0.900        2.6339            1.000        1.8902
 0.000        2.5034           min          2.2735           0.950        2.8134           -------------------------
 0.850        2.4848           max          2.5382          -------------------------       O............ 1.9563(  14)
 0.900        2.4876           0.150        2.3829          Ni3a......... 2.6364(   7)      ave          1.8897
 0.950        2.4779          -------------------------      ave          2.7025            min          1.7887
 1.000        2.5034          Ni3b'........ 2.2814( 703)     min          2.5977            max          1.9955
-------------------------      ave          2.3641           max          2.8380            0.000        1.8902
Ni3b......... 2.4720( 389)     min          2.2735           0.050        2.8134            0.050        1.8662
 ave          2.5211           max          2.5382           0.100        2.6339            0.100        1.9643
 min          2.4730           0.850        2.3829           0.150        2.6695            0.150        1.9653
 max          2.5517          -------------------------     -------------------------       0.850        1.8029
 0.900        2.4730          Ni3a......... 2.6364(   4)    O............ 1.9563(   8)      0.900        1.8246
 0.950        2.5517           ave          2.7025           ave          1.8897            0.950        1.9151
-------------------------      min          2.5977           min          1.7887            1.000        1.8902
Ni3b......... 2.4720( 389)     max          2.8380           max          1.9955           -------------------------
 ave          2.5211           0.850        2.6695           0.000        1.8902           O............ 1.9563(  14)
 min          2.4730           0.900        2.6339           0.050        1.9151            ave          1.8897
 max          2.5517           0.950        2.8134           0.100        1.8246            min          1.7887
 0.050        2.5517          -------------------------      0.150        1.8029            max          1.9955
 0.100        2.4730          Ni3a......... 2.6364(   4)     0.850        1.9653            0.000        1.8902
-------------------------      ave          2.7025           0.900        1.9643            0.050        1.9151
Ni3b......... 2.6656( 389)     min          2.5977           0.950        1.8662            0.100        1.8246
 ave          2.7483           max          2.8380           1.000        1.8902            0.150        1.8029
 min          2.6578           0.050        2.8134          -------------------------       0.850        1.9653
 max          2.9225           0.100        2.6339          O............ 1.9563(   8)      0.900        1.9643
 0.150        2.7672           0.150        2.6695           ave          1.8897            0.950        1.8662
-------------------------     -------------------------      min          1.7887            1.000        1.8902
Ni3b......... 2.6656( 389)    Ni3a......... 2.6364(   7)     max          1.9955           -------------------------
 ave          2.7483           ave          2.7025           0.000        1.8902           O............ 1.9563(  14)
 min          2.6578           min          2.5977           0.050        1.8662            ave          1.8897
 max          2.9225           max          2.8380           0.100        1.9643            min          1.7887
 0.850        2.7672           0.850        2.6695           0.150        1.9653            max          1.9955
```

Figure 191 *(Continued)*

The type of output can be selected in the Modulation Commands form (see page 316). Atom Ni1 is coordinated by 6 oxygen atoms but not for every value of t. This is a characteristic feature of composite structures.



```
                                     ***************************************
                                     ** Coordination for atom : Ni1      **
                                     ***************************************
-------------------------------------------------------------------------------------------------------------------------
|       t= 0.000        |       t= 0.050        |       t= 0.100        |       t= 0.150        |       t= 0.200        |
-------------------------------------------------------------------------------------------------------------------------
| O............. 1.8902 | O............. 1.8662 | O............. 1.8246 | O............. 1.8029 |                       |
| O............. 1.8902 | O............. 1.8662 | O............. 1.8246 | O............. 1.8029 |                       |
| O............. 1.8902 | O............. 1.8662 | O............. 1.8246 | O............. 1.8029 |                       |
| O............. 1.8902 | O............. 1.9151 | O............. 1.9643 | O............. 1.9653 |                       |
| O............. 1.8902 | O............. 1.9151 | O............. 1.9643 | O............. 1.9653 |                       |
| O............. 1.8902 | O............. 1.9151 | O............. 1.9643 | O............. 1.9653 |                       |
| Ni1........... 2.5034 | Ni1........... 2.4779 | Ni3b.......... 2.4730 | Ni3b'......... 2.3829 |                       |
| Ni1........... 2.5034 | Ni3b.......... 2.5517 | Ni1........... 2.4876 | Ni1........... 2.4848 |                       |
|                       | Ni3a.......... 2.8134 | Ni3a.......... 2.6339 | Ni3a.......... 2.6695 |                       |
|                       | Ni3a.......... 2.8134 | Ni3a.......... 2.6339 | Ni3a.......... 2.6695 |                       |
|                       | Ni3a.......... 2.8134 | Ni3a.......... 2.6339 | Ni3a.......... 2.6695 |                       |
|                       | Ni3b'......... 2.9360 | Ni3b'......... 2.8573 | Ni3b.......... 2.7672 |                       |
-------------------------------------------------------------------------------------------------------------------------
|       t= 0.250        |       t= 0.300        |       t= 0.350        |       t= 0.400        |       t= 0.450        |
-------------------------------------------------------------------------------------------------------------------------
-------------------------------------------------------------------------------------------------------------------------
|       t= 0.500        |       t= 0.550        |       t= 0.600        |       t= 0.650        |       t= 0.700        |
-------------------------------------------------------------------------------------------------------------------------
-------------------------------------------------------------------------------------------------------------------------
|       t= 0.750        |       t= 0.800        |       t= 0.850        |       t= 0.900        |       t= 0.950        |
-------------------------------------------------------------------------------------------------------------------------
|                       |                       | O............. 1.8029 | O............. 1.8246 | O............. 1.8662 |
|                       |                       | O............. 1.8029 | O............. 1.8246 | O............. 1.8662 |
|                       |                       | O............. 1.8029 | O............. 1.8246 | O............. 1.8662 |
|                       |                       | O............. 1.9653 | O............. 1.9643 | O............. 1.9151 |
|                       |                       | O............. 1.9653 | O............. 1.9643 | O............. 1.9151 |
|                       |                       | O............. 1.9653 | O............. 1.9643 | O............. 1.9151 |
|                       |                       | Ni3b'......... 2.3829 | Ni3b.......... 2.4730 | Ni1........... 2.4779 |
|                       |                       | Ni1........... 2.4848 | Ni1........... 2.4876 | Ni3b.......... 2.5517 |
|                       |                       | Ni3a.......... 2.6695 | Ni3a.......... 2.6339 | Ni3a.......... 2.8134 |
|                       |                       | Ni3a.......... 2.6695 | Ni3a.......... 2.6339 | Ni3a.......... 2.8134 |
|                       |                       | Ni3a.......... 2.6695 | Ni3a.......... 2.6339 | Ni3a.......... 2.8134 |
|                       |                       | Ni3b.......... 2.7672 | Ni3b'......... 2.8573 | Ni3b'......... 2.9360 |
-------------------------------------------------------------------------------------------------------------------------
|       t= 1.000        |                       |                       |                       |                       |
-------------------------------------------------------------------------------------------------------------------------
| O............. 1.8902 |                       |                       |                       |                       |
| O............. 1.8902 |                       |                       |                       |                       |
| O............. 1.8902 |                       |                       |                       |                       |
| O............. 1.8902 |                       |                       |                       |                       |
| O............. 1.8902 |                       |                       |                       |                       |
| O............. 1.8902 |                       |                       |                       |                       |
| Ni1........... 2.5034 |                       |                       |                       |                       |
| Ni1........... 2.5034 |                       |                       |                       |                       |
```

Figure 191 *(Continued)*

```
                                   ***************************************
                                   ** Coordination for atom : Sr       **
                                   ***************************************

-------------------------------------------------------------------------------------------------------------------------
|        t= 0.000       |        t= 0.050       |        t= 0.100       |        t= 0.150       |        t= 0.200       |
-------------------------------------------------------------------------------------------------------------------------
| O............. 2.4096 | O............. 2.2406 | O............. 2.5155 | O............. 2.4940 | O............. 2.3287 |
| O............. 2.4215 | O............. 2.4977 | O............. 2.5408 | O............. 2.5386 | O............. 2.4355 |
| O............. 2.6116 | O............. 2.5079 | O............. 2.6319 | O............. 2.6424 | O............. 2.5615 |
| O............. 2.6319 | O............. 2.6724 | O............. 2.6501 | O............. 2.6899 | O............. 2.6795 |
| O............. 2.6513 | O............. 2.7194 | O............. 2.7696 | O............. 2.7522 | O............. 2.7130 |
| O............. 2.6776 | O............. 2.7727 | O............. 2.7974 | O............. 2.8043 | O............. 2.7138 |
| O............. 2.7142 | O............. 2.7823 | O............. 2.7994 | O............. 2.8115 | O............. 2.7305 |
| O............. 2.7355 | O............. 2.8385 | O............. 2.8353 | O............. 2.8510 | O............. 2.7551 |
|                       | O............. 2.8771 | O............. 2.9071 | O............. 2.8649 |                       |
|                       | Ni3a.......... 2.9298 | O............. 2.9260 | O............. 2.9400 |                       |
|                       | Ni3a.......... 2.9895 |                       |                       |                       |
-------------------------------------------------------------------------------------------------------------------------
|        t= 0.250       |        t= 0.300       |        t= 0.350       |        t= 0.400       |        t= 0.450       |
-------------------------------------------------------------------------------------------------------------------------
| O............. 2.4299 | O............. 2.4403 | O............. 2.3554 | O............. 2.4943 | O............. 2.5234 |
| O............. 2.4853 | O............. 2.4896 | O............. 2.4244 | O............. 2.5304 | O............. 2.5351 |
| O............. 2.6098 | O............. 2.6071 | O............. 2.5766 | O............. 2.6494 | O............. 2.6346 |
| O............. 2.6260 | O............. 2.6218 | O............. 2.6621 | O............. 2.6994 | O............. 2.6427 |
| O............. 2.6314 | O............. 2.6361 | O............. 2.6947 | O............. 2.7546 | O............. 2.7779 |
| O............. 2.6366 | O............. 2.6388 | O............. 2.7220 | O............. 2.8072 | O............. 2.7915 |
| O............. 2.6689 | O............. 2.6562 | O............. 2.7284 | O............. 2.8187 | O............. 2.8060 |
| O............. 2.7242 | O............. 2.7155 | O............. 2.7321 | O............. 2.8313 | O............. 2.8235 |
| Ni3a.......... 2.9780 | Ni3a.......... 2.9677 |                       | O............. 2.8630 | O............. 2.9145 |
|                       |                       |                       | O............. 2.9462 | O............. 2.9225 |
-------------------------------------------------------------------------------------------------------------------------
|        t= 0.500       |        t= 0.550       |        t= 0.600       |        t= 0.650       |        t= 0.700       |
-------------------------------------------------------------------------------------------------------------------------
| O............. 2.2297 | O............. 2.3973 | O............. 2.4582 | O............. 2.4167 | O............. 2.2889 |
| O............. 2.5005 | O............. 2.4127 | O............. 2.4881 | O............. 2.4695 | O............. 2.4575 |
| O............. 2.5119 | O............. 2.5993 | O............. 2.6089 | O............. 2.6202 | O............. 2.5381 |
| O............. 2.6628 | O............. 2.6408 | O............. 2.6207 | O............. 2.6250 | O............. 2.6987 |
| O............. 2.7126 | O............. 2.6671 | O............. 2.6319 | O............. 2.6325 | O............. 2.7122 |
| O............. 2.7942 | O............. 2.6973 | O............. 2.6371 | O............. 2.6392 | O............. 2.7298 |
| O............. 2.7963 | O............. 2.7212 | O............. 2.6546 | O............. 2.6884 | O............. 2.7418 |
| O............. 2.8512 | O............. 2.7319 | O............. 2.6983 | O............. 2.7336 | O............. 2.7907 |
| O............. 2.8512 |                       | Ni3a.......... 2.9580 |                       | O............. 2.9562 |
|                       |                       |                       |                       | Ni3a.......... 2.9765 |
-------------------------------------------------------------------------------------------------------------------------
|        t= 0.750       |        t= 0.800       |        t= 0.850       |        t= 0.900       |        t= 0.950       |
-------------------------------------------------------------------------------------------------------------------------
| O............. 2.4986 | O............. 2.5041 | O............. 2.2672 | O............. 2.4116 | O............. 2.4689 |
| O............. 2.5454 | O............. 2.5457 | O............. 2.4730 | O............. 2.4540 | O............. 2.4827 |
| O............. 2.6346 | O............. 2.6320 | O............. 2.5248 | O............. 2.6250 | O............. 2.6138 |
| O............. 2.6740 | O............. 2.6642 | O............. 2.6885 | O............. 2.6265 | O............. 2.6227 |
| O............. 2.7543 | O............. 2.7588 | O............. 2.7271 | O............. 2.6303 | O............. 2.6265 |
| O............. 2.7921 | O............. 2.7907 | O............. 2.7350 | O............. 2.6516 | O............. 2.6282 |
| O............. 2.8120 | O............. 2.8088 | O............. 2.7588 | O............. 2.6995 | O............. 2.6664 |
| O............. 2.8597 | O............. 2.8520 | O............. 2.8113 | O............. 2.7363 | O............. 2.6861 |
| O............. 2.8775 | O............. 2.8909 | O............. 2.9243 |                       | Ni3a.......... 2.9548 |
| O............. 2.9334 | O............. 2.9305 | Ni3a.......... 2.9572 |                       |                       |
-------------------------------------------------------------------------------------------------------------------------
|        t= 1.000       |                       |                       |                       |                       |
-------------------------------------------------------------------------------------------------------------------------
| O............. 2.4096 |                       |                       |                       |                       |
| O............. 2.4215 |                       |                       |                       |                       |
| O............. 2.6116 |                       |                       |                       |                       |
| O............. 2.6319 |                       |                       |                       |                       |
| O............. 2.6513 |                       |                       |                       |                       |
| O............. 2.6776 |                       |                       |                       |                       |
| O............. 2.7142 |                       |                       |                       |                       |
| O............. 2.7355 |                       |                       |                       |                       |
-------------------------------------------------------------------------------------------------------------------------
```

4.2.10 *DIST* listing m61

Together with the wide listing *DIST* writes the results also in one column form to the `m61` file. The information of these two output files is complementary; `m61` lists the symmetry codes which are not present in the wide listing, but it doesn't contain the values of distances and angles as a function of the `t` coordinate.

Figure 192 *The* `m61` *file of ordinary structure.*

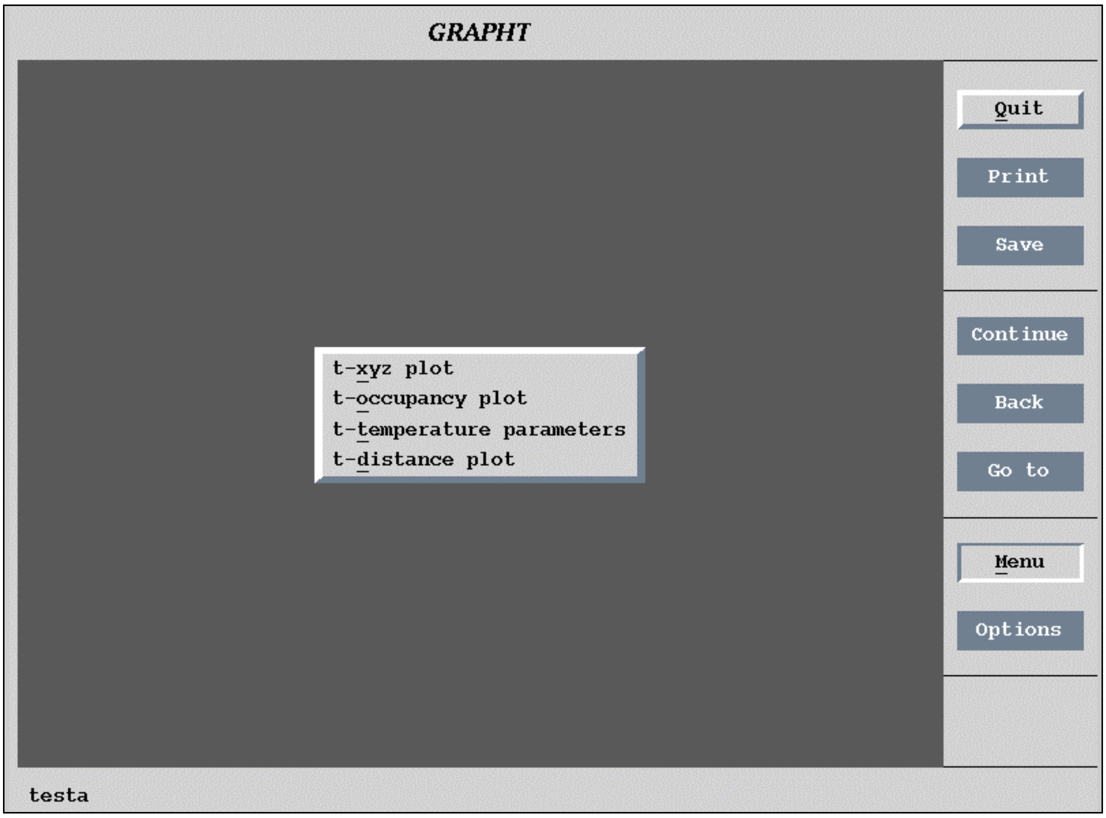
```
Distances and angles concerning atom Zn
=============================================
Zn       - Br             2.4811(   8)  symmetry : x,y,z#
Br       - Zn       - O1            121.44(  12)  symmetry : x,y,z
Br       - Zn       - N1             96.54(  13)  symmetry : x,y,z
Br       - Zn       - N2            107.84(  13)  symmetry : x,y,z
Br       - Zn       - N3             98.18(  13)  symmetry : x,y,z
Br       - Zn       - C1            106.21(  14)  symmetry : x,y,z
Br       - Zn       - C2             97.26(  15)  symmetry : x,y,z
Br       - Zn       - C3             90.50(  13)  symmetry : x,y,z
Br       - Zn       - C4             95.48(  11)  symmetry : x,y,z
Br       - Zn       - C5             84.73(  13)  symmetry : x,y,z
Br       - Zn       - C6            108.46(  11)  symmetry : x,y,z
Br       - Zn       - C7            117.74(  10)  symmetry : x,y,z
Br       - Zn       - C12           121.51(  10)  symmetry : x,y,z
Br       - Zn       - H1(n1)         76.56(  11)  symmetry : x,y,z
Br       - Zn       - H1(n1)        159.72(   8)  symmetry : 1/2-x,1/2+y,1/2-z
Br       - Zn       - H2(n1)        108.08(  11)  symmetry : x,y,z
Br       - Zn       - H1(c1)        122.41(  13)  symmetry : x,y,z
Br       - Zn       - H1(c1)         75.06(  13)  symmetry : 1/2-x,-1/2+y,1/2-z
Br       - Zn       - H1(c2)         79.61(  14)  symmetry : x,y,z
```

Figure 193 *The* `m61` *file for modulated structure.*
See page 27 for description of the special symmetry codes starting with the "#" character.

```
Distances concerning atom Sr1
==================================
Sr1      - O31            2.2636(   0)  symmetry : 1/2-x,y,-1+z#s2t0,0,-1
Sr1      - O31            2.2636(   0)  symmetry : 1/2-x,y,z#s2
Sr1      - O31            2.2636(   0)  symmetry : 1/2-x,y,1+z#s2t0,0,1
Sr1      - O31            2.2636(   0)  symmetry : 1/2+x,1-y,1-z#s-2t0,1,1
Sr1      - O31            2.2636(   0)  symmetry : 1/2+x,1-y,2-z#s-2t0,1,2
Sr1      - O31            2.2636(   0)  symmetry : 1/2+x,1-y,3-z#s-2t0,1,3
Sr1      - O11            2.6068(  89)  symmetry : 1/2-x,y,-1/2+z#s2t0,0,-1
Sr1      - O11            2.6068(  89)  symmetry : 1/2+x,1/2-y,1-z#s-2c2
Sr1      - O2             2.6168(  14)  symmetry : x,y,-1+z#t0,0,-1
Sr1      - O2             2.6168(  14)  symmetry : x,y,z#
-----------------------------------------------------------------------------
```

4.3 Program *GRAPHT*

Input: `m40`, `m50`
Output: HPGL ,PostScript, PCX or numerical form
M50 control keywords: none

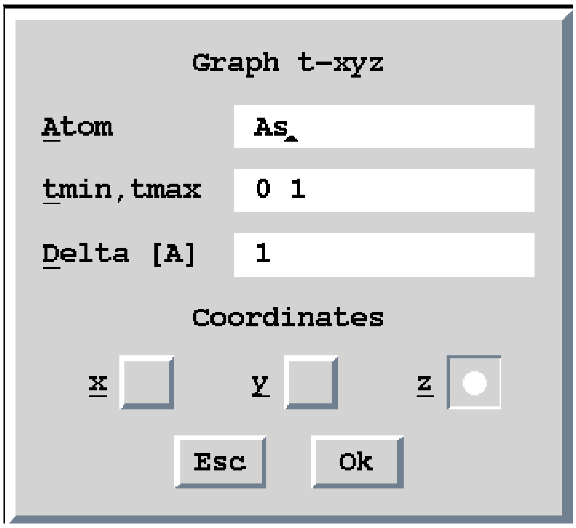


4.3.1 Description of *GRAPHT*

GRAPHT plots coordinates, occupancies, distances and bond valences as a function of the `t` coordinate. It can save the pictures in HPGL, PostScript and PXC format or in a numerical form suitable as an input for other programs.

4.3.2 Coordinate plots

Figure 194 The t-xyz form of GRAPHT



Graph t-xyz

Atom: As

tmin,tmax: 0 1

Delta [A]: 1

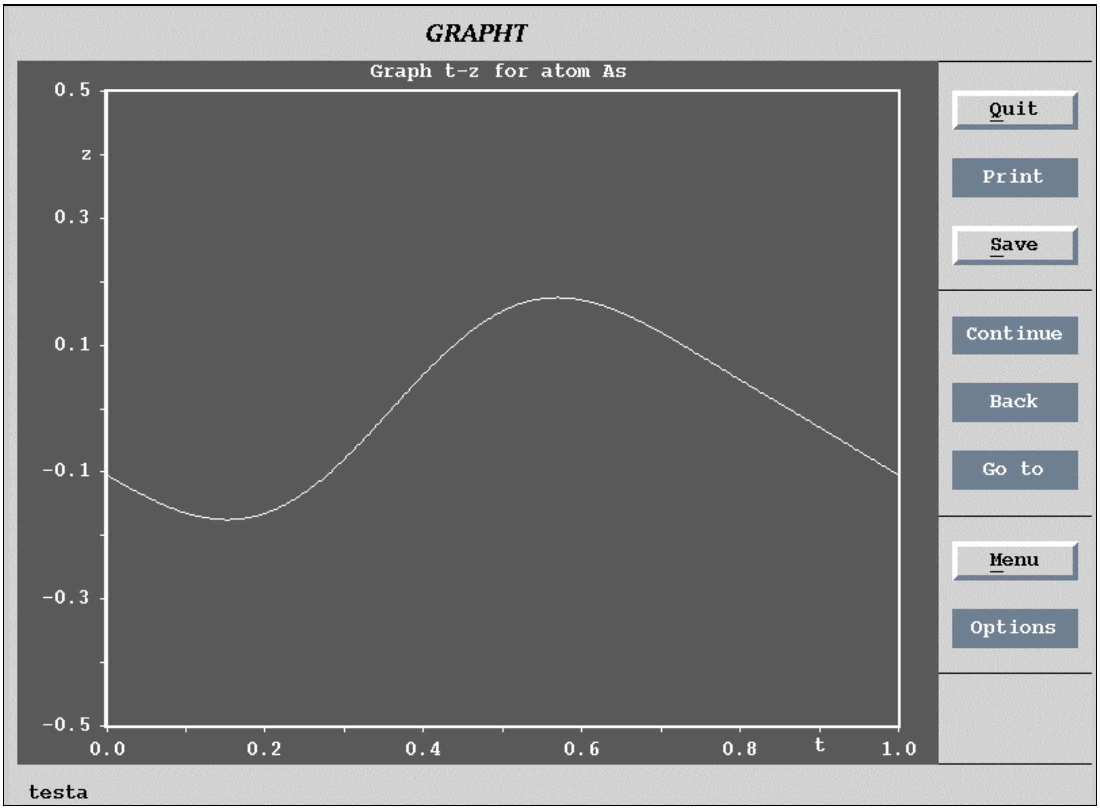
Coordinates

x ☐ y ☐ z ●

This tool plots the difference between selected coordinate and its basic value as a function of `t`.

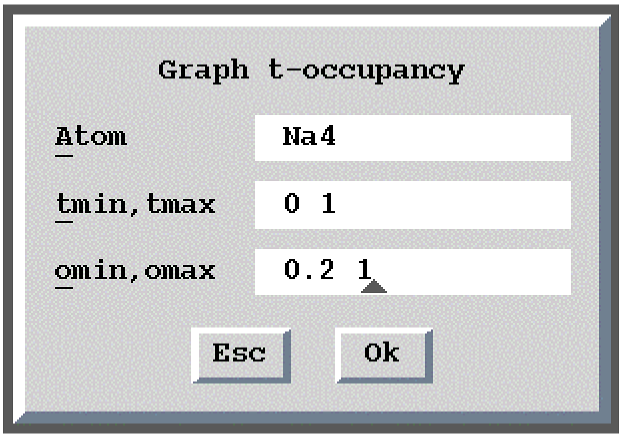
`Delta` in the form defines the length of the vertical axis of the plot in Å.

Figure 195 Example of a coordinate plot



4.3.3 Occupancy plots

Figure 196 *The t-occupancy form of GRAPHT*



Graph t-occupancy

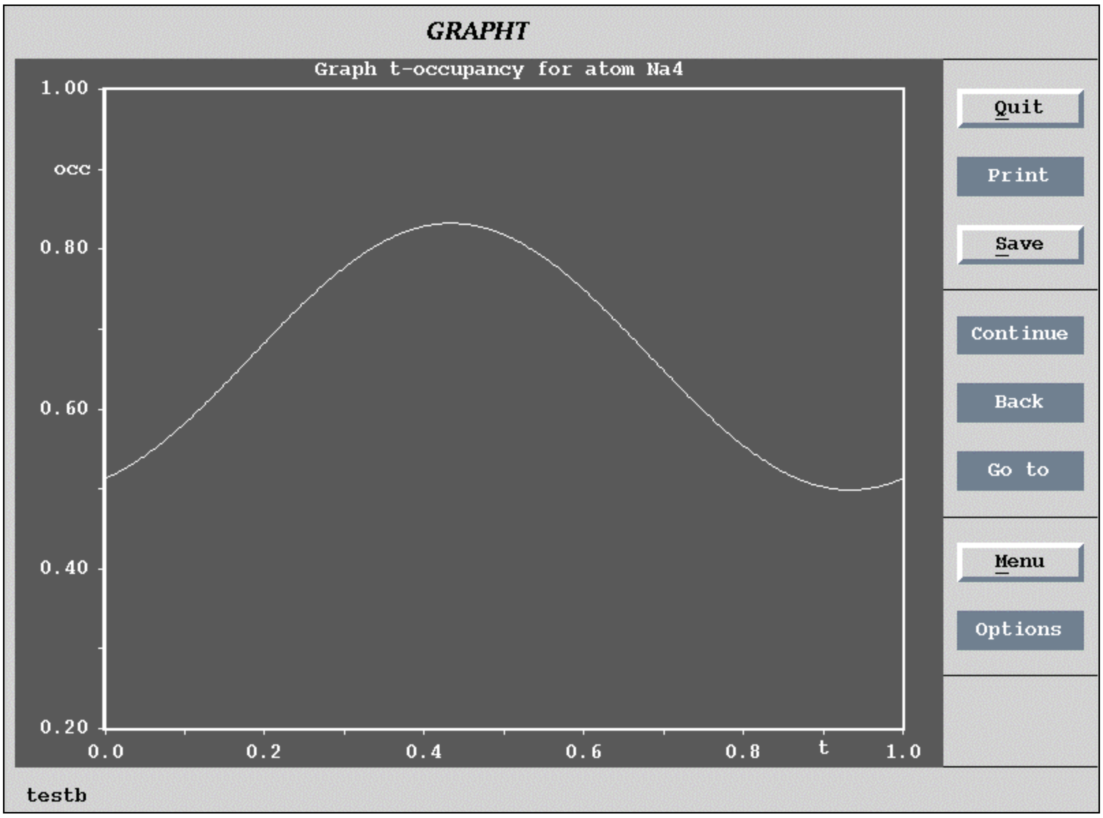
Atom: Na4

tmin,tmax: 0 1

omin,omax: 0.2 1

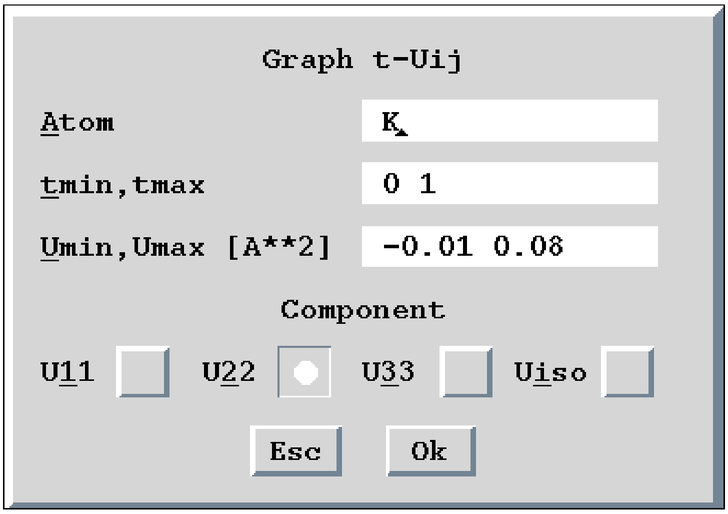
This tool plots the occupancy of an atom as a function of `t`.
The `omin` and `omax` define the length of the vertical axis of the plot in the positive and negative direction, respectively.

Figure 197 *Example of an occupancy plot*



4.3.4 Temperature plots

Figure 198 *The t-temperature parameters form of GRAPHT*



Graph t-Uij

Atom K

tmin,tmax 0 1

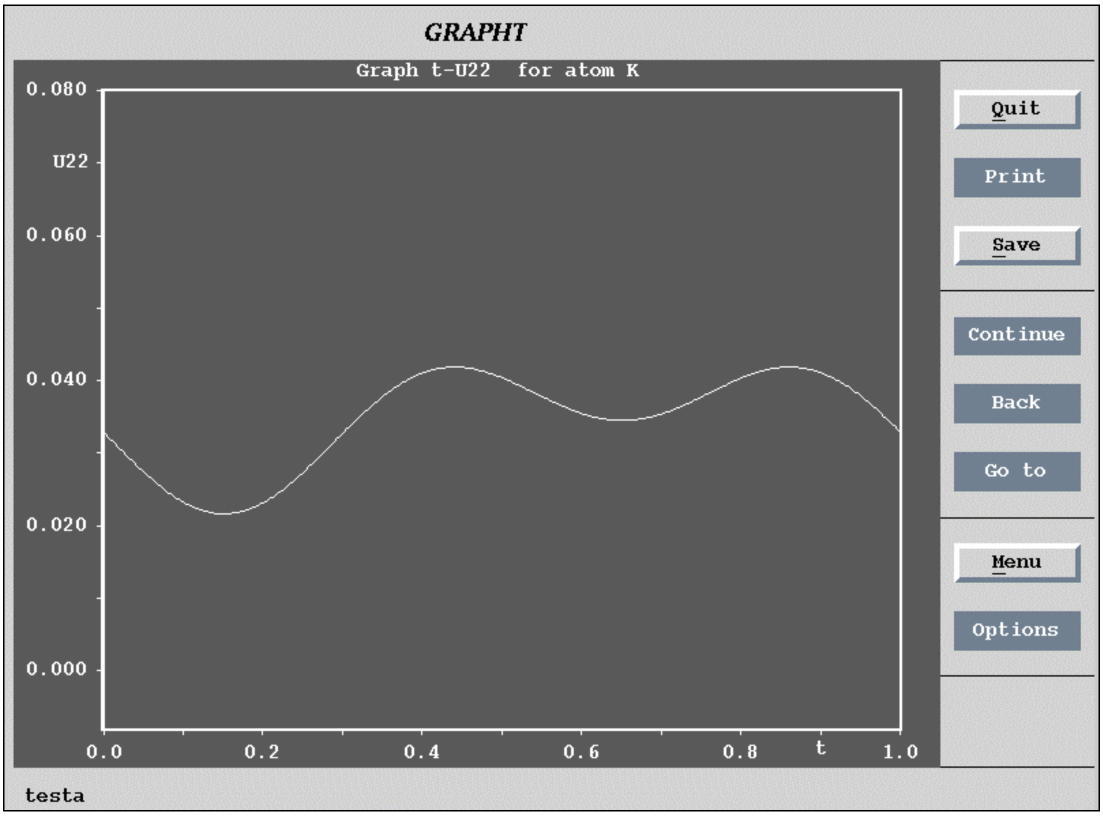
Umin,Umax [A**2] -0.01 0.08

Component

U11 ☐ U22 ● U33 ☐ Uiso ☐

This tool plots selected temperature parameters of an atom as a function of `t`. The `Umin` and `Umax` define the minimal and maximal value on the vertical axis of the plot, respectively.

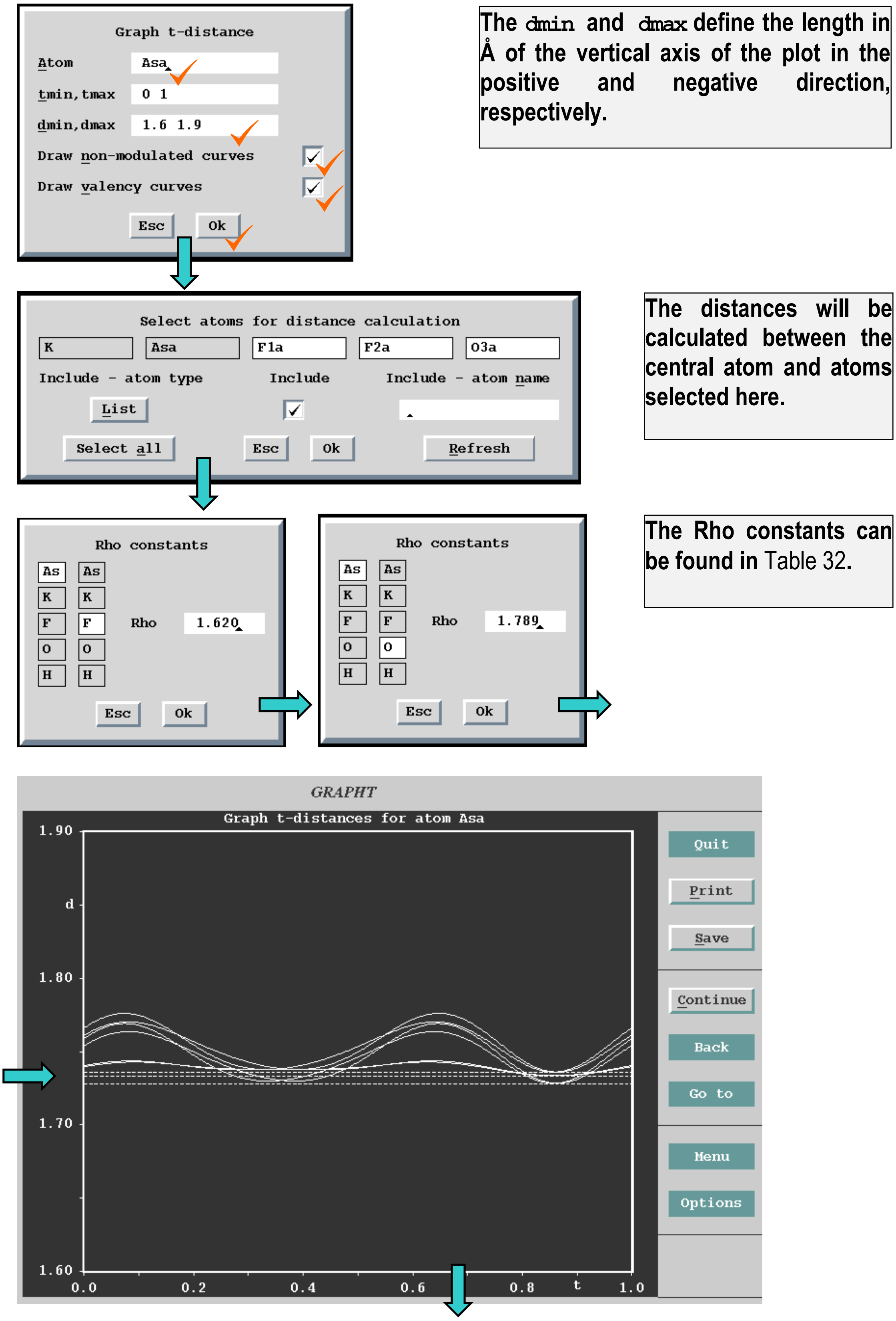
Figure 199 *Example of a t-temperature parameters plot.*



4.3.5 Distance and valence plots

This tool plots distances between a central and other selected atoms and bond valences defined according to Brown & Altermatt,1985 as a function of `t`.

Figure 200 *Plotting t-distance and t-valence plots*



Graph t-distance

Atom: Asa

tmin,tmax: 0 1

dmin,dmax: 1.6 1.9

Draw non-modulated curves ☑

Draw valency curves ☑

The `dmin` and `dmax` define the length in Å of the vertical axis of the plot in the positive and negative direction, respectively.

Select atoms for distance calculation

K | Asa | F1a | F2a | O3a

Include - atom type

Include ☑

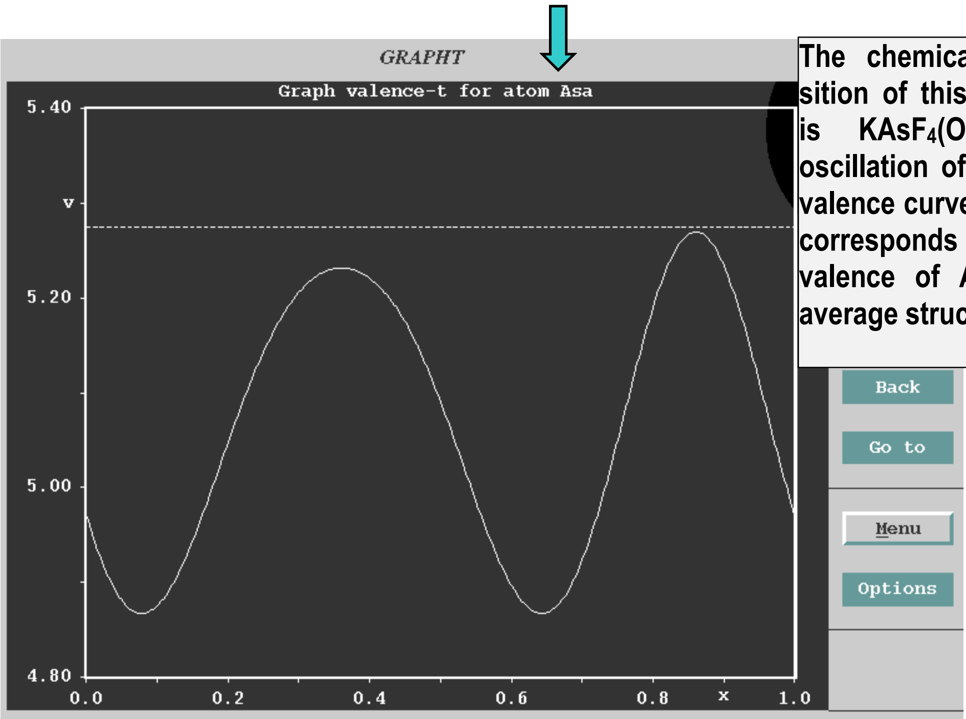
Include - atom name

The distances will be calculated between the central atom and atoms selected here.

Rho constants: As As, K K, F F, O O, H H; Rho 1.620

Rho constants: As As, K K, F F, O O, H H; Rho 1.789

The Rho constants can be found in Table 32.



The chemical composition of this structure is $KAsF_4(OH)_2$. The oscillation of the bond valence curve around 5 corresponds to the valence of As in the average structure.

Table 32 *Bond-Valence parameters*

This is output of the Bond valence calculator program[1]*. It is based on Brown & Altermatt,1985.*

Ag	1	O	-2	1.842	0.370
Ag	1	S	-2	2.119	0.370
Al	3	Cl	-1	2.032	0.370
Al	3	F	-1	1.545	0.370
Al	3	O	-2	1.620	0.370
As	3	O	-2	1.789	0.370
As	3	S	-2	2.272	0.370
As	5	F	-1	1.620	0.370
As	5	O	-2	1.767	0.370
B	3	F	-1	1.281	0.370
B	3	O	-2	1.371	0.370
Ba	2	F	-1	2.188	0.370
Ba	2	O	-2	2.285	0.370
Ba	2	S	-2	2.769	0.370
Be	2	F	-1	1.281	0.370
Be	2	O	-2	1.381	0.370
Bi	3	O	-2	2.094	0.370
Bi	3	S	2	2.570	0.370
C	4	N	-3	1.442	0.370
C	4	O	-2	1.390	0.370
Ca	2	Br	-1	2.507	0.370
Ca	2	F	-1	1.842	0.370
Ca	2	O	-2	1.967	0.370
Cd	2	Cl	-1	2.212	0.370
Cd	2	O	-2	1.904	0.370
Ce	4	O	-2	2.090	0.370
Cd	2	S	-2	2.304	0.370
Cl	7	O	-2	1.632	0.370
Co	2	Cl	-1	2.033	0.370
Co	2	F	-1	1.649	0.370
Co	2	O	-2	1.692	0.370
Co	3	C	2	1.634	0.370
Cr	3	F	-1	1.657	0.370
Cr	3	O	-2	1.724	0.370
Cr	6	O	-2	1.794	0.370
Cs	1	Cl	-1	2.791	0.370
Cs	1	O	-2	2.417	0.370
Cu	1	I	-1	2.108	0.370
Cu	1	O	-2	1.600	0.370
Cu	1	S	-2	1.898	0.370
Cu	2	F	-1	1.594	0.370
Cu	2	O	-2	1.679	0.370
Cu	2	S	-2	2.054	0.370
Cu	3	O	-2	1.739	0.370
D	1	O	-2	0.927	0.370
Dy	3	O	-2	2.001	0.370
Er	3	F	-1	1.904	0.370
Er	3	O	-2	1.988	0.370
Eu	2	S	-2	2.584	0.370
Eu	3	O	-2	2.074	0.370
Fe	2	O	-2	1.734	0.370
Fe	3	C	2	1.689	0.370
Fe	3	F	-1	1.679	0.370
Fe	3	O	-2	1.759	0.370
Fe	3	S	-2	2.149	0.370
Ga	3	O	-2	1.730	0.370
Ga	3	S	-2	2.163	0.370
Ge	4	O	-2	1.748	0.370
Ge	4	S	-2	2.217	0.370
H	1	N	-3	0.885	0.370
H	1	O	-2	0.882	0.370
Hg	2	O	-2	1.972	0.370
Hg	2	S	-2	2.308	0.370
Ho	3	O	-2	2.025	0.370
I	5	O	-2	2.003	0.370
I	7	O	-2	1.910	0.370
In	3	F	-1	1.792	0.370
In	3	O	-2	1.902	0.370
In	3	S	-2	2.370	0.370
K	1	Cl	-1	2.519	0.370
K	1	F	-1	1.992	0.370
K	1	O	-2	2.132	0.370
La	3	O	-2	2.172	0.370
La	3	S	-2	2.643	0.370
Li	1	F	-1	1.360	0.370
Li	1	O	-2	1.466	0.370
Mg	2	O	-2	1.693	0.370
Mn	2	Br	-1	2.335	0.370
Mn	2	Cl	-1	2.133	0.370
Mn	2	F	-1	1.698	0.370
Mn	2	O	-2	1.790	0.370
Mn	3	O	-2	1.760	0.370
Mn	4	O	-2	1.753	0.370
Mo	6	O	-2	1.907	0.370
N	3	O	-2	1.361	0.370
N	5	O	-2	1.432	0.370
Na	1	F	-1	1.677	0.370
Na	1	O	-2	1.803	0.370
Na	1	S	-2	2.300	0.370
Nb	5	O	-2	1.911	0.370
Nd	3	O	-2	2.105	0.370
Ni	2	F	-1	1.596	0.370
Ni	2	O	-2	1.654	0.370
P	5	N	-3	1.704	0.370
P	5	O	-2	1.617	0.370
P	5	S	-2	2.145	0.370
Pb	2	O	-2	2.112	0.370
Pb	2	S	-2	2.541	0.370
Pb	2	Se	-2	2.686	0.370
Pb	4	O	-2	2.042	0.370
Pr	3	O	-2	2.138	0.370
Pt	2	C	2	1.760	0.370
Pt	4	O	-2	1.879	0.370
Rb	1	Cl	-1	2.652	0.370
Rb	1	O	-2	2.263	0.370
S	2	N	-2	1.597	0.370
S	2	N	-3	1.682	0.370
S	4	N	-3	1.762	0.370
S	4	O	-2	1.644	0.370
S	6	O	-2	1.624	0.370
Sb	3	F	-1	1.883	0.370
Sb	3	O	-2	1.973	0.370
Sb	3	S	-2	2.474	0.370
Sb	3	Se	-2	2.602	0.370
Sb	5	F	-1	1.797	0.370
Sb	5	O	-2	1.942	0.370
Sc	3	O	-2	1.849	0.370
Sc	3	S	-2	2.321	0.370
Se	4	O	-2	1.811	0.370
Se	6	O	-2	1.788	0.370
Si	4	C	-4	1.883	0.370
Si	4	N	-3	1.724	0.370
Si	4	O	-2	1.640	0.370
Si	4	S	-2	2.126	0.370
Sn	2	F	-1	1.925	0.370
Sn	4	Cl	-1	2.276	0.370
Sn	4	F	-1	1.843	0.370
Sn	4	O	-2	1.905	0.370
Sn	4	S	-2	2.399	0.370
Sr	2	O	-2	2.118	0.370
Ta	5	O	-2	1.920	0.370
Tb	3	O	-2	2.032	0.370
Te	4	O	-2	1.977	0.370
Te	6	O	-2	1.917	0.370
Th	4	F	-1	2.068	0.370
Ti	4	O	-2	1.815	0.370
Tl	1	I	-1	2.822	0.370
Tl	1	O	-2	2.226	0.340
Tl	1	S	-2	2.545	0.370
Tl	3	O	-2	2.007	0.370
U	4	F	-1	2.038	0.370
U	6	O	-2	2.075	0.370
V	3	O	-2	1.743	0.370
V	4	O	-2	1.784	0.370
V	5	O	-2	1.803	0.370
W	6	O	-2	1.917	0.370
Y	3	O	-2	2.019	0.370
Yb	3	O	-2	1.965	0.370
Zn	2	Cl	-1	2.027	0.370
Zn	2	O	-2	1.704	0.370
Zr	4	F	-1	1.846	0.370
Zr	4	O	-2	1.928	0.370

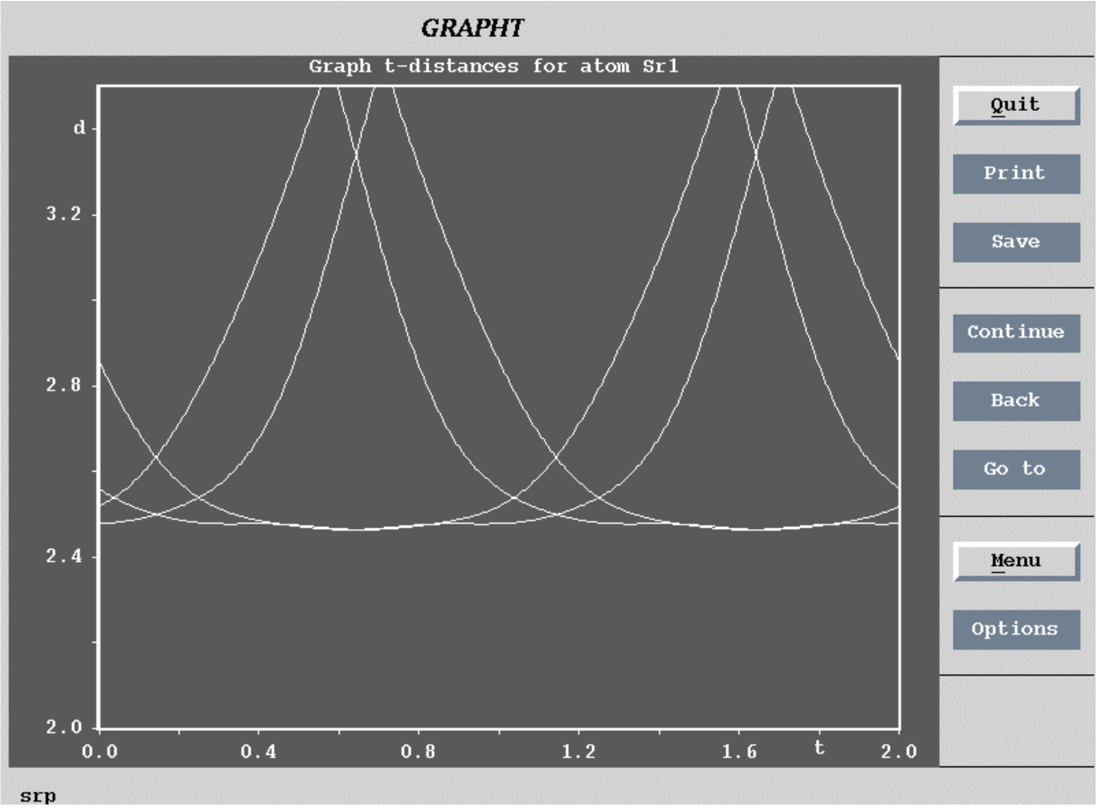
---

[1] http://www.ccp14.ac.uk/ccp/web-mirrors/valence/valence.txt

4.3.6 The distance plots of composite structures

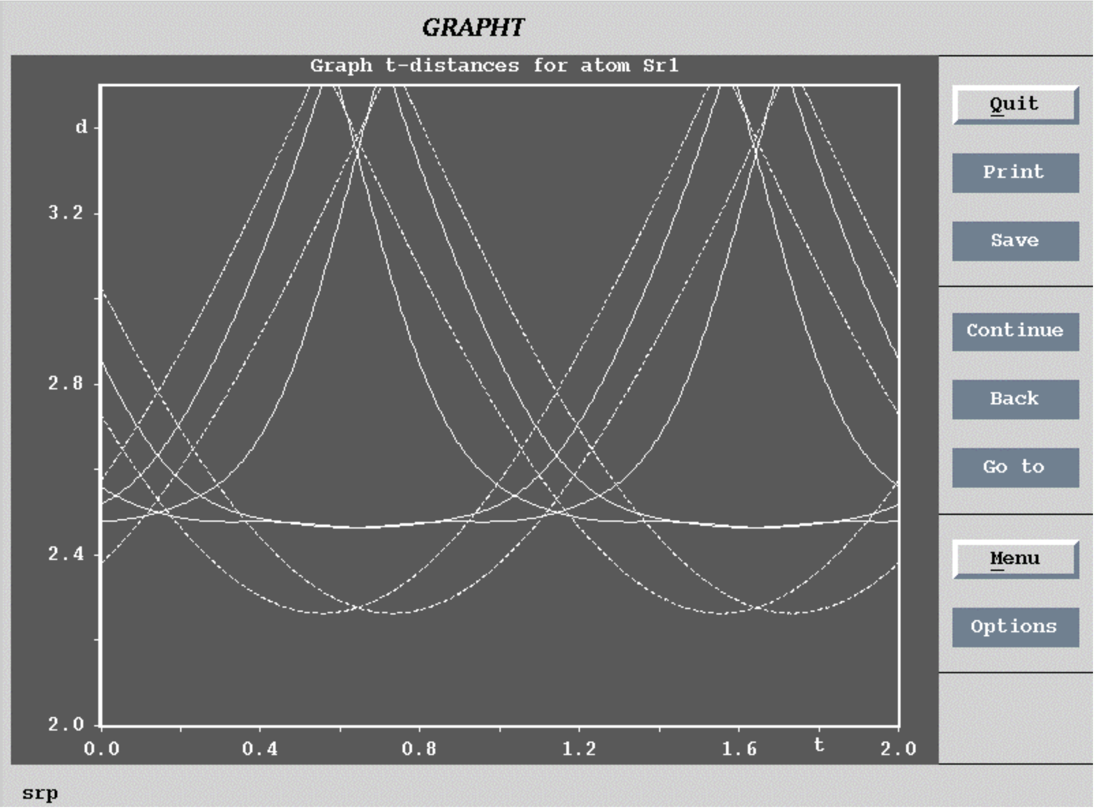
Calculation of distances for composite structures is discussed in page 325. The program makes systematic search to find and draw all distances as a function of t within the limits defined in the form.

Figure 201 *Example of a distance plot for a composite structure.*
This plot is for two atoms belonging to different composite parts.



The t-distance form (page 335) contains an option for drawing of non-modulated curves. They are calculated from basic positions of atoms as refined in the `m40` file. In the case of two atoms belonging to different composite parts the non-modulated curves are hyperbolic and can be used as an estimation how modulation influences distances. For instance, in the following figure the minimal distance without modulation could be too short.

Figure 202 *Example of a distance plot with non-modulated curves*



References

Axe,J.D.(1980). Phys.Rev. B, 21, 4181-4190

Becker, P.J. & Coppens, P. (1974).Acta Cryst. A30,129-147
Extinction within the limit of validity of the Darwin transfer equations, part I

Becker, P.J. & Coppens, P. (1974).Acta Cryst. A30,148-152
Extinction within the limit of validity of the Darwin transfer equations, part II

Brown, I.D. & Altermatt, D. (1985).Acta Cryst. B41,244-247

De Wolf, P.M., Janssen, T. & Janner, A.(1981).Acta Cryst. A37, 625-636
The superspace groups for incommensurate crystal structures with a one-dimernsional modulation

Hall, S.R.(1981). Acta Cryst. A37, 517
Space group notation with an explicit origin

International Tables for Crystallography (1993). Vol. B

International Tables for Crystallography (1992). Vol. C

Kuhs, W.F. (1992). Acta Cryst. A48, 80-98

Madariaga,G. (1994). Proc. international conference on aperiodic crystals Aperiodic'94, pp 425-429. World Scientific.

Petricek, V., Gao, Y., Lee, P. & Coppens, P. (1990). Phys.Rev.B, 42, 387-392
X-ray analysis of the incommensurate modulation in the 2:2:1:2 Bi-Sr-Ca-Cu-O superconductor including the oxygen atoms.

Petricek,V., Lee, A. Van Der & Evain, M. (1995). Acta Cryst A51, 529-535
On the use of crenel functions for occupationally modulated structures.

Schomaker, V. & Trueblood, K.N. (1968). *Acta Cryst.* B24, 63 - 76.
On the rigid-body motion of molecules in crystals

Van Smaalen, S. (1991), Phys.Rev.B, 43, 11330-11341
Symmetry of composite crystals.

Van Smaalen, S. & Petricek, V. (1992).Acta Cryst A48,610
Refinement of incommensurate structures against diffraction data from a twinned crystal.

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