Details of Data Input for Program RFINE90

1. File Name Card: Format(12A1)

   Columns 1-12 contain an alphanumeric label to be written into a new data set. This label will be printed when the file is read.

2. Title Card: Format(72A1)

   Any desired Hollerith information in columns 1-72. This information appears at the top of most pages of the output.

3. Control Card: Format(15I1,2F5.0,3I1,3I2,4I1,F5.0,22I1)

Column	Variable	Description
1	MFSEL	Function selector 0 - Structure factors only 1 - Least-squares refinement 2 - Skip structure factor calculation – used for bonds, etc.
2	MSF	Structure Factor Output Selector 0 - No structure factor output 1 - Printed structure factors 2 - Printed and written (For a Fourier program) on file ISC4 3 - Written on file ISC4
3	MCORR	Correlation matrix [1] 0 - Not printed 1 - Printed
4	MBODAN	Bond distance, angle selector 0 - none computed 1 - Distances only 2 - Distances and angles computed
5	MELVIB	Ellipsoids of vibration 0 - None computed 1 - Ellipsoids computed for anisotropic atoms 2 - Ellipsoids and thermal corrections to bonds computed
6	MREF	Refinement type indicator 0 - Refine on F 1 - Refine on F^2
7	IFILE	Rewrite data set indicator 0 - Data set rewritten if this is the last cycle of the current run 1 - Data set not rewritten 2 - Data set rewritten. On input the standard deviations of parameters refined in previous cycles will be read in. 3 - Data set not rewritten. Standard deviations read in.

[1] The values of the 20 largest off-diagonal correlation coefficients will be printed for every least-squares cycle.

8	NEW(1)	If non-zero, read new cell parameters [2]
9	NEW(2)	If non-zero, read new bond distance limits
10	NEW(3)	If non-zero, read new symmetry operators 1 - Read x, y, z cards 2 - Read space group symbol
11	NEW(4)	If non-zero, read new scattering factors [3]
12	NEW(5)	If non-zero, read new scale factors and atom parameters 1 - All atoms will be read 2 - Only selected atoms will be read 3 - Only scale and extinction will be read 4 - Only special parameters will be read
13	NEW(6)	If non-zero, read temperature factor type card
14	NEW(7)	If non-zero, read parameter selection card(s) and parameter dependency card(s)
15	NEW(8)	If non-zero, read new structure factors 1 - Read data in ABSORB format 2 - Read data in first alternate format 3 - Read data in second alternate format

For all zero values in columns 8-15, the old values are read from file ISCl or the values in memory are used. The above integers are required for all cycles.

16-20	DMIN	Minimum bond distance in Angstroms. Needed only if NEW(2) $\neq$ 0.
21-25	DMAX	Maximum bond distance in Angstroms. Needed only if NEW(2) $\neq$ 0.
26	ICENT	Centric indicator, needed only if NEW(3) = 1. 0 - Centric 1 - Acentric
27	ICELL	Cell type indicator, needed only if NEW(3) = 1. 0 - Primitive cell 1 - A-centered cell 2 - B-centered cell 3 - C-centered cell 4 - I-centered cell 5 - F-centered cell 6 - R-centered cell
28	MANOD	Anomalous dispersion indicator, needed only if NEW(4) $\neq$ 0. 0 - No corrections are to be used 1 - Include anomalous dispersion corrections
29-30	NSCAT	Number of scattering curves. This is needed only if NEW(4) $\neq$ 0. ($1 \leq$ NSCAT ≤ 20)

[2] Note that if the new cell parameters will affect $\sin\theta/\lambda$, and the atomic scattering factors for various reflections, structure factors must be read in.

[3] Warning: New scattering factor curves *must* be read only in the first cycle of a given computer run or when new structure factors are being read.

31-32	NATOM	Number of atoms in asymmetric unit. This is needed only if NEW(5) $\neq$ 0. ($1 \leq$ NATOM ≤ 60)
33-34	NSF	Number of scale factors. This is needed only if NEW(5) $\neq$ 0. ($1 \leq$ NSF ≤ 10)
35	NEWEXT	Extinction change indicator (needed only if NEW(5) $\neq$ 0) 0 - New extinction parameters not read 1 - Extinction parameters read from cards
36	IWT	Weight type, needed only if NEW(8) $\neq$ 0. 0 - Standard deviation of F on cards 1 - Standard deviation of F^2 on cards 2 - Weight on cards 3 - Sigma for F is to be computed in WEIGHT 4 - Compute weight in subroutine WEIGHT 5 - Standard deviation of F on cards will be altered by "ignorance factor" to $\sigma'^2 = \sigma^2 + (pF)^2$
37	INN	Structure factor source indicator, needed if NEW(8) $\neq$ 0 0 - Structure factors on cards 1 - New structure factors from file ISC3
38	IREJ	Rejection parameter, needed only if NEW(8) $\neq$ 0. 0,5 - No reflections rejected 1,6 - Reject if I less than the minimum observable 2,7 - Reject if I less than the minimum observable or $\lvert Fo - Fc\rvert/\sigma_F$ greater than DELMAX 3,8 - Reject if $\lvert Fo - Fc\rvert/\sigma_F$ greater than DELMAX 4,9 - Use special routine REJECT

In cases 2 and 3, discepancies will be altered by $\Delta' = \Delta(1 - x^2)$, where $x = w\Delta^2/(DELMAX)^2$, Δ is F_o-F_c for refinement on F or F_o^2-F_c^2 for refinement on F^2, and w is the weight. If the Fourier structure factor file is also written (MSF=2 or 3), the value of DELMAX is set to be nine times the median value of $\lvert Fo - Fc\rvert/\sigma_F$, thereby invoking the robust/resistant refinement procedure. Note: Values of IREJ from 5 to 9 allow the rejection mode to be changed even though NEW(8) = 0.

39-43	DELMAX	Maximum allowable difference between observed and calculated structure factors on an absolute scale. This is needed only if IREJ is 7 or 8, or if NEW(8) $\neq$ 0 and IREJ is 2 or 3.
44-53	JSCRF	Scale factor refinement switches (needed only if NEW(7) $\neq$ 0.
54-59	KEXTRF	Extinction factor refinement switches (needed only if NEW(7) $\neq$ 0
60	JXPAR	Extra parameter indicator (needed only if NEW(5) $\neq$ 0) 0 - No extra parameters 1 - Extra parameters will be read
61	IFSGN	Orientation determination parameter (needed only if NEW(7) $\neq$ 0) 0 - Do not determine the orientation 1 - Change to orientation with lower weighted <u>R</u>
62	NFILE	Non-zero if file for robust/resistant weighting factors exists
63	ILVG	Non-zero if leverage file should be written
64	ITWN	Non-zero if twin description should be read
65	NABS	Non-zero if spherical harmonic absorption should be calculated

4. Cell card: Format(9F8.0) – Needed only if NEW(1) $\neq$ 0.

Column	Variable	Description
1-8	A(1)	a axis length in angstroms
9-16	A(2)	b axis length
17-24	A(3)	c axis length
25-32	A(4)	Alpha in degrees
33-40	A(5)	Beta in degrees
41-48	A(6)	Gamma in degrees

5a. XYZ Symmetry Cards: Format(3(A3,2(1X,A2),1X),I4) – Needed only if NEW(3) = 1.

Column	Variable	Description
1-3	JTNS(1)	Translational part of x'
5-6	IRMAT(1,1)	First positional part of x'
8-9	IRMAT(2,1)	Second positional part of x'
11-13	JTNS(2)	Translational part of y'
15-16	IRMAT(1,2)	First positional part of y'
18-19	IRMAT(2,2)	Second positional part of y'
21-23	JTNS(3)	Translational part of z'
25-26	IRMAT(1,3)	First positional part of z'
28-29	IRMAT(2,3)	Second positional part of z'
34	IEF	End flag, non-zero on last card only
		0 - Another symmetry card follows
		1 - This is the last card

The translational operators are of the following form: bbb, 1/2, 1/3, 2/3, 1/4, 3/4, 1/6 or 5/6. The positional operators are of the form bW, +W, or -W, with W equal to X, Y, and Z and b signifies a blank. The symmetry postion X Y Z must be included. Symmetry positions corresponding to centers of inversion or lattice centering should not be included. The maximum number of symmetry cards needed or allowed is 24.

5b. Space Group Symbol: Format(A80) – Needed only if NEW(3) = 2.

In this form of symmetry information, the space group symbol is input and the various rotational and translational operators, as well as the lattice centering and the centric indicator, are generated from an alphanumeric string. The lattice type and the various axial directions are separated by spaces. Sample space group symbols include C -1, P 21/C, P N M A, P 4/N M M, R -3 M, and I A 3 D. These routines will always generate the unique axis parallel to b for monoclinic space groups.

6a. Number of Twin Operations: Format(I4,4F10.0) – Needed only if NEW(3) $\neq$ 0 and ITWN $>$ 0.

Column	Variable	Description
1-4	NT	Number of twin operations (NT $\leq$ 20)
5-14	TTHW	Window in 2θ (degrees)
15-24	OMGW	Window in ω (degrees)
25-34	CHIW	Vertical counter aperture/Crystal to Detector Distance
35-44	WAVE	Wavelength in Å

6b. Orientation Matrix: Format(3(3F10.0/)) – Needed only if NEW(3) $\neq 0$ and ITWN > 0.
This item consists of three lines containing the orientation matrix UB used to collect the original data, one row per card.

6c. Twin Operations: Format(F10.0,I1/3(3F10.0))

Column	Variable	Description
Card 1:		
1-10	TFRAC	Volume fraction for this twin component
11	ITRF	Refinement Switch for Twin Fraction
Card 2:		
1-10	T_{11}	Components of twin matrix relating the
11-20	T_{12}	indices of the parent to the twin.
21-30	T_{13}	
Card 3:		
1-10	T_{21}	
11-20	T_{22}	
21-30	T_{23}	
Card 4:		
1-10	T_{31}	There should be a set of items 6b for each of the
11-20	T_{32}	NT twin components.
21-30	T_{33}	

7. Scattering Factor Cards: Format(9F8.0,2A4) – Needed only if NEW(4) $\neq 0$.

Column	Variable	Description
1-8	a_1	These coefficients must
9-16	b_1	satisfy the equation
17-24	a_2	
25-32	b_2	$f(x) = c + \sum_{i=1}^{4} a_i e^{-b_i x^2}$
33-40	a_3	
41-48	b_3	with $x = \sin\theta/\lambda$
49-56	a_4	
57-64	b_4	
65-72	c	
73-80	IDENT	Identification

Coefficients for scattering curves of this form have been tabulated in *International Tables*, Volume IV, pp. 99-101, 1974. If anomalous dispersion coefficients are being used (MANOD = 1), the above card is followed by one with the real part of the anomalous dispersion in col.1-8 and the imaginary part in col.9-16. Note: The scattering curves should be changed only on the first cycle of a given computer run or when NEW(8) = 1. New structure factors *must* be read (NEW(8) = 1) if the number of scattering curves is to be changed.

8. Scale Factor Cards: Needed only if NEW(5) $\neq 0$.

The NSF scale factors are punched with 8 columns per value and 9 values per card and should be the values needed to place the calculated structure factors on the same scale as the observed.

9a. Extinction Cards: Format(3I1,F8.0,7F10.0) – Needed only if NEW(5) $\neq 0$ and NEWEXT $\neq 0$.

Column	Variable	Description
1	NIEXT	Extinction type indicator (1-6 for Becker-Coppens) 1 - Isotropic Type 1 correction 2 - Isotropic Type 2 correction 3 - Isotropic General Case correction 4 - Anisotropic Type 1 correction 5 - Anisotropic Type 2 correction 6 - Anisotropic General Case correction 0 - Zachariasen Isotropic correction
2	NDISTR	Becker-Coppens Distribution Type 0 - Gaussian Distribution 1 - Lorentzian Distribution
3	INEUT	Source Type Indicator 0 - Neutron source 1 - X-ray source without monochrometer 2 - X-ray source with monochrometer
4-11	WVLNTH	Wavelength in Å (default value is 0.7093 Å)
12-21	Z(11)	First extinction coefficient $\times 10^4$
22-31	Z(22)	Second extinction coefficient
32-41	Z(33)	Etc.
42-51	Z(12)	
52-61	Z(13)	
62-71	Z(23)	
72-81		Primary extinction coefficient for Anisotropic General Case

9b. Monochrometer Card: Format(2F8.0) – Needed only if INEUT=2

Column	Variable	Description
1-8	DIHED	Dihedral angle in degrees between monochrometer crystal and sample
9-16	THETM	Monochrometer angle θ in degrees (default value is 6.081)

If the values of the Z's are all zero and the type of extinction does not match the internally stored value, a conversion is initiated. It is not possible, however, to convert from a Becker-Coppens model to a Zachariasen form, or vice-versa.

10. Analytical Absorption: – Needed if NABS $\neq 0$ and NEW(5) $\neq 0$

Card 1: Control – Format(I2)

Column	Variable	Description
1-2	NABSRN	Maximum value of n in C_{nm} and S_{nm} $(n \leq 6)$

Cards 2-n: Coefficients – Format(6E12.0)

Card 2: C_{10}, C_{11}, S_{11}, C_{20}, C_{21}, and C_{22}
Card 3: S_{21}, S_{22}, C_{30}, C_{31}, C_{32}, and C_{33}

Card 4:	S_{31}, S_{32}, S_{33}, C_{40}, C_{41}, and C_{42}
Card 5:	C_{43}, C_{44}, S_{41}, S_{42}, S_{43}, and S_{44}
Card 6:	C_{50}, C_{51}, C_{52}, C_{53}, C_{54}, and C_{55}
Card 7:	S_{51}, S_{52}, S_{53}, S_{54}, S_{55}, and C_{60}
Card 8:	C_{61}, C_{62}, C_{63}, C_{64}, C_{65}, and C_{66}
Card 9:	S_{61}, S_{62}, S_{63}, S_{64}, S_{65}, and S_{66}

11. Atom Parameters: Needed only if NEW(5) $\neq$ 0.

If NEW(5) = 1, the entire atom list is read with two cards per atom. If NEW(5) = 2, the Atom Number is read with Format (I2) followed by the two cards for that atom. Card reading is terminated by a blank Atom Number card.

Column	Variable	Description
Card 1: Format(A6,3F6.0)		
1-6	TAG	Alphanumeric identifier
7-12	SITE	Equipoint fraction
13-18	OCCUP	Total occupancy of site
19-24	OCCA	Occupancy of species A in this site
		Needed only if multiple occupancy
Card 2: Format(2I2,4X,9F7.0,I2)		
1-2	ISCAT(1)	Number of scattering curve for species A
2-4	ISCAT(2)	Number of scattering curve for species B
		(blank if single element in site).
9-15	XYZ(1)	x of atom (fractional coordinates)
16-22	XYZ(2)	y
23-29	XYZ(3)	z
30-36	BETA(1)	B for an isotropic atom, or
		β_{11} for an anisotropic atom
37-43	BETA(2)	β_{22}
44-50	BETA(3)	β_{33}
51-57	BETA(4)	β_{12}
58-64	BETA(5)	β_{13}
65-71	BETA(6)	β_{23}
73	IEF	Indicator of third cumulant
Card 3: Format(9F8.0,F6.0,I2) – Needed if IEF $\neq$ 0.		
1-8	C_{111}	
9-16	C_{222}	
17-32	C_{333}	
25-32	C_{112}	
33-40	C_{122}	
41-48	C_{113}	
49-56	C_{133}	
57-64	C_{223}	
65-72	C_{233}	

73-78	C_{123}	
80	IEF	Fourth cumulant indicator

Card 4-5: Format(10F8.0) – Needed if IEF $\neq$ 0.

Enter d_{1111}, d_{2222}, d_{3333}, d_{1112}, d_{1113}, d_{1122}, d_{1123}, d_{1133}, d_{1222}, d_{1223}, d_{1233}, d_{1333}, d_{2223}, d_{2233}, d_{2333}, in order with 8 columns per value.

12. Special parameter cards: Needed only if NEW(5) $\neq$ 0 and JXPAR $\neq$ 0.

Column	Variable	Description
Card 1: Format(I4)		
1-4	NPAR	Number of special parameters
Cards 2 — NPAR+1: Format(A8,3I2,F10.0)		
1-8	PARNAM	Name of parameter, up to 8 alphanumeric characters
9-10	IPRTYP	Type of parameter 0- Linear function with coefficient given on constraint card. 1- Rigid body system 2- Defined by user in subroutines SPVAL and SPDERI
11-12	IRNUM	Integer indicating which rigid-body system this parameter belongs
13-14	IPDSG	Parameter designator. If IPRTYP is 1, the code is as follows 1- X coordinate of origin (in crystal axes system) 2- Y coordinate of origin 3- Z coordinate of origin 4- ω angle for transformation to standard, orthonormal coordinate system 5- χ angle for transformation 6- ϕ angle for transformation 7- X coordinate of an atom in the special system 8- Y coordinate of an atom 9- Z coordinate of an atom 10- T_{11} 11- T_{22} 12- T_{33} 13- T_{12} 14- T_{13} 15- T_{23} 16- L_{11} 17- L_{22} 18- L_{33} 19- L_{12} 20- L_{13} 21- L_{23} 22- S_{11} 23- S_{22} 24- S_{33}

		25- S_{12}
		26- S_{13}
		27- S_{23}
		28- S_{21}
		29- S_{31}
		30- S_{32}
15-24	SPPAR	Initial value of this parameter

13. Temperature Factor Type Card: Needed only if NEW(5) or NEW(6) $\neq 0$.

The temperature factor type code is punched with one column per atom beginning in Col. 2 with the following code:

0 - Now isotropic, do not change
1 - Now anisotropic, do not change
2 - Now isotropic, change to anisotropic
3 - Now anisotropic, change to isotropic
4 - Now 3rd cumulant, do not change
5 - Now anisotropic, change to 3rd cumulant
6 - Now 3rd cumulant, change to anisotropic
7 - Now 4th cumulant, do not change
8 - Now 3rd cumulant, change to 4th
9 - Now 4th cumulant, change to 3rd

14. Analytical Absorption Selection Card: Format(48I1) - Needed only if NEW(7) $\neq 0$ and NABSRN > 0. The refinement switches for the analytical absorption coefficients are typed in order with a total of NABSRN*(NABSRN+2) values needed. A zero in a column means that the corresponding parameter will not be refined and a one means that it will.

15. Parameter Selection Cards. Format(80I1) – Needed only if NEW(7) $\neq 0$.

If JXPAR $\neq 0$ the first line contains refinement selection for the special parameters in the first NPAR columns. The following lines have the refinement indicators for the conventional parameters punched in columns 1-80. If the number of atom parameters is such that more than 80 values are required, the parameter selection information should be continued in column 1 of the next line. The order of parameters is:

a) occupancy - If single occupancy, the total occupancy is refined. If multiple occupancy, the occupancy of species A is refined.
b) <u>x</u> coordinate
c) <u>y</u> coordinate
d) <u>z</u> coordinate
e) thermal parameters, one for isotropic atoms, six for anisotropic atoms. The thermal coefficients occur in the same order as on the Atom Parameter Card 2.
f) 3rd and 4th cumulant parameters (if needed).

16. Parameter Constraint Cards(s): Needed only if NEW(7) $\neq 0$.

<u>Column</u> <u>Description</u>

1-3	Integer specifying the dependent atom number.
5-6	Dependent parameter number. This number is 1 for occupancy, 2-4 for x through z respectively, 5 for B or β_{11}, and 6, 7, 8, 9, 10 for $\beta_{22}, \beta_{33}, \beta_{12}, \beta_{13}$, and β_{23} respectively. Values of 11 to 20 treat 3rd cumulant parameters and 21-35 are the 4th cumulants.
7-9	Atom number of first independent parameter. Special parameters are arbitrarily indicated as atom number 0.
10-12	Parameter number of first independent parameter. Atom 0 parameter 0 designates an initializing constant in a linear constraint function.
13-20	Coefficient of first relationship.
21-24	Atom number of second independent parameter
25-28	Second independent parameter number
29-36	Second coefficient
37-40	Third independent atom number
41-44	Third independent parameter number
45-52	Third coefficient
53-56	Fourth independent atom number
57-60	Fourth independent parameter number
61-68	Fourth coefficient

If the independent atom number is zero, designating a special parameter, and the constraints are nonlinear, the second and fourth independent parameter numbers may be negative to denote the end of an inclusive list beginning with the first (third) independent parameter number.

There should be one (1) blank line to signify the end of the constraint list. This line is needed even if there are no constraints.

17. Structure Factor Cards: Needed only if NEW(8) $\neq$ 0.

a) Format Card: this card contains the format of the input structure factor data. The number and order of variables on a card (or tape record) depends on the value of NEW(8), as explained in detail below. All records must contain at least four integer fields, for the indices and an observed/unobserved indicator, and two floating point fields for F_{obs} and σ_F or other weight information. The records may contain other information, such as a scale factor group identifier, the factor $\beta(\theta)$ used in extinction corrections, and the setting angles, needed for anisotropic extinction corrections and analytic absorption corrections.

b) If IWT=5, include a card with the "ignorance factor" punched as a real number in the first 8 columns.

c) If NEW(8)=3, include the wavelength in Å in the first 8 columns. For neutron data, an approximation of $\beta(\theta)$ will be computed according to the formula $\beta(\theta) = [2\lambda^2/(V^2 \sin^2\theta)] \times 10^8$, where V is the unit cell volume in $Å^3$. The refined quantity then will be the product Tr*, with T assumed to be approximately constant.

d) Data Cards in a form consistent with format Card: If New(8)=1 the list is in the following order:

Variable	Description
IH(1)	<u>h</u> for reflection

IH(2)	k
IH(3)	l
FOBS	Observed structure factor
SIGMA	Weight information consistent with IWT on Control Card
EXBETA	Factor $\beta(\theta)$ for extinction
I	Number of scale factor for this reflection. If zero, the scale factor is the same as the preceding reflection. Scale factor 1 is assumed for the first reflection.
MREJ	Minimum Observable Parameter 1 - Intensity above minimum observable. 2 - Intensity below minimum observable.
IEF	End-of-list marker, zero on all data cards The line following the last data card should contain a nonzero value in this field. Alternatively, if permitted by the operating system, an end-of-file marker will proxy for this item.
TTH	2θ
OMEG	ω
CHI	χ
PHI	ϕ These angles are needed only if anisotropic extinction is to be calculated and must conform to the diffractometer conventions given by Busing and Levy (1967, p.458).

If NEW(8) = 2 the input list is IH(1), IH(2), IH(3), MREJ, I, FOBS, EXBETA, SIGMA, with the same meanings as above. The end of the data must be marked by an end-of-file marker, not by a data item.

If NEW(8) = 3 the list is IH(1), IH(2), IH(3), MREJ, FOBS, SIGMA. EXBETA is computed, and again the end of data must be marked by an end-of-file.

e) End-of-list card - A card with a non-zero value in the field of variable IEF or an end-of-file card appropriate to the system will signify the end of the structure factors.

18. Run Continuation and Parameter Punch Card Card: Needed for all cycles.

Column	Variable	Description
1	ICONT	Run continuation 0 - No more cycles. The data set on file ISCI will be updated if IFILE from the Control Card is zero 1 - Read a new title card and start another cycle
2	JPUN	Parameter Output Control 0 - Do not output any atom parameters 1 - Output atom cards in form required by 'X-RAY67' 2 - Output atom cards in form required by this program 3 - Output atom cards in both forms
3-4	ISC1	Binary output unit number If this field is non-zero its value will be substituted as the unit number for the binary master file, making it possible to try new courses of refinement without destroying previous stages.