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Program for Refinement of Powder Diffraction Data

(POWDER LEAST SQUARES)

von

G. Will, E. Jansen und W. Schäfer

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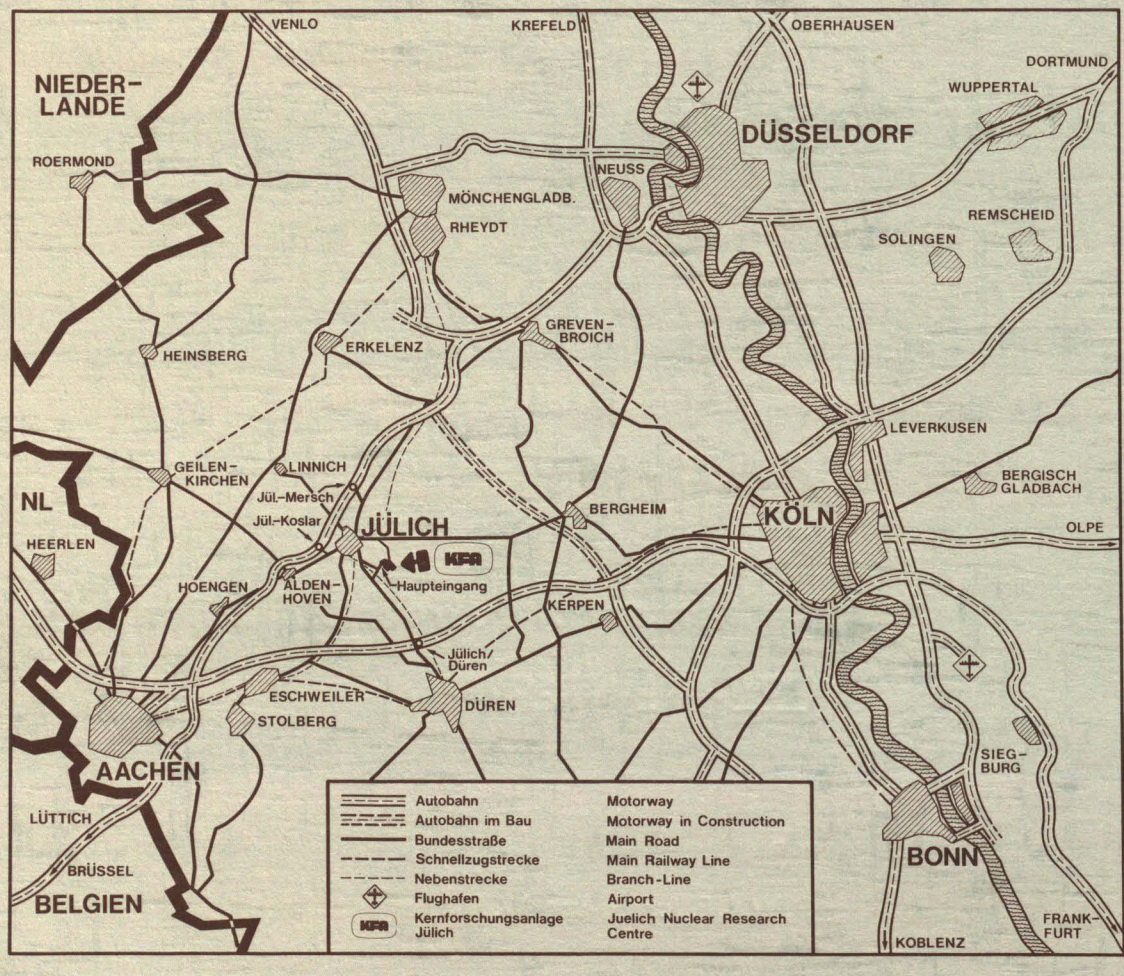

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POWLS-60

Program for Refinement of Powder Diffraction Data

(POWDER LEAST SQUARES)

Mitteilung aus dem Mineralogischen Institut
der Universität Bonn in Zusammenarbeit mit dem Institut
für Festkörperforschung der KFA Jülich

von

G. Will, E. Jansen und W. Schäfer

Abstract:

A computer program POWLS - 60, based on least-squares calculations, for crystallographic and magnetic structure refinements from X-ray or neutron diffraction powder data is reported. Great flexibility is achieved by a special problem oriented subroutine to be supplied by the user defining any functions in terms of any parameters. Three examples are given together with the source listing of the whole program.

Contents:

I Introduction

POWLS is a reiterative least-squares FORTRAN program for refinement of crystal and magnetic structures from neutron or X-ray diffraction powder data. There is an option available to calculate Bragg angles and structure factors.

Observed quantities may be structure factors, integrated intensities, or any function of intensity. In case of overlapping peaks, the total intensity may be treated as one observation, and (or) the individual estimated values may be treated as additional observations. Provision is made for use of a non-diagonal weight matrix in such cases.

Any type of parameter can be used. In addition to the usual positional and thermal parameters one can easily refine multiplicities, occupancy factors, magnetic parameters (including those for non-collinear and spiral structures), form factor parameters, unusual descriptions of thermal motion etc. This flexibility is achieved by a special problem oriented subroutine to be supplied by the user defining any functions in terms of any parameters.

II Princip of Least Squares Calculation

POWLS forms and solves the matrix equations

$$(A^T P A)X = A^T P F,$$

where X is the m * 1 dimensional matrix of m parameter adjustments to be determined:

$$\begin{bmatrix} x_1 \\ \vdots \\ x_j \\ \vdots \\ x_m \end{bmatrix}.$$

F is the (n * 1) matrix of the n (> m) residual functions

$$f_i = f(obs)_i - f(calc)_i$$

to be minimized. P, the weight matrix, is the inverse of the variance - covariance matrix of F:

$$\begin{bmatrix} \sigma^2(f_1) & \sigma_1\sigma_2\varrho_{12} & \cdots & \sigma_1\sigma_n\varrho_{1n} \\ \sigma_1\sigma_2\varrho_{12} & \sigma^2(f_2) & & \\ \vdots & & \ddots & \\ \sigma_1\sigma_n\varrho_{1n} & & & \sigma^2(f_n) \end{bmatrix}.$$

A is the matrix of partial derivatives of the functions with respect to the parameter adjustments:

$$a_{ij} = \frac{\partial f_i}{\partial x_j} .$$

The a_{ij} are obtained by a numerical method. A^T is the transposed of A.

III Description of POWLS Flow Scheme

The MAIN program reads the input data to be described later. There is an option for reading form factor tables. A weight matrix P is formed by an input correlation matrix ϱ_{ij} of F or by an unit matrix using standard deviations of the observations.

Bragg angles and trigonometric functions are calculated from Miller indices, cell constants, and wave length for use in subroutines.

With this input information the refinement loop starts. In each cycle different parameters may be refined. Therefore a parameter switch card is read at the beginning of each cycle. These switches indicate whether a parameter is to be varied or not. The number of the switches set determines the dimension of the actual refinement problem.

To calculate the (n x 1) matrix F(calc), the MAIN program calls subroutine FUNNY, which is to be written by the user in order to obtain individual model functions with the parameters. With F(calc) FUNNY returns to MAIN and the matrix F = F(obs) - F(calc) is stored for further use.

The partial derivatives are calculated by changing the free parameters one by one by small increments dx_j and forming a neighbour matrix F'. The partial derivatives a_{ij} are calculated as $(f_i' - f_i)/dx_j$ and their matrix is stored as A.

With A, P, and F the MAIN program calls subroutine WEAST, which calculates the matrix products $A^TPA = B$ and $A^TPF = V'$. To solve the equation $BX = V'$, the subroutine SMI inverts the matrix B to B^{-1} and the parameter adjustment matrix is obtained by $X = B^{-1}V'$. WEAST returns to MAIN yielding, together with X and B, the matrix V of the remaining residuals v_i, the standard deviation of an observation of unit weight $FAX = V^TPV/(n-m)$, and the weighted error matrix $XM = B^{-1}/FAX$.

MAIN adjusts the parameters and calculates their standard deviations. The increments dx_j are adapted to the deviations for use in further refinement cycles.

At the end of each refinement cycle subroutine OUTPUT lists a current parameter output, which will be described later, and a new cycle may be started by reading a new parameter switch card.

After the last refinement cycle MAIN calls OUTPUT for final listings depending on options, whereafter a new start is possible at different points of MAIN, also controlled by option switches.

IV The Subroutine FUNNY(PIZ,FZ)

FUNNY, supplied by the user, obtains the current parameters PIZ from MAIN and produces the model functions FZ. Useful crystallographic quantities are prepared in COMMON blocks by MAIN facilitating the programming of FZ, like

AS,BS,CS,CAS,CBS,CCS	: the reciprocal cell constants $a^*,b^*,c^*,\cos(\alpha^*),\cos(\beta^*),\cos(\gamma^*)$;
WAVE	: the wave lenght;
IH,IK,IL,SINT,SIN2T	: Miller indices and trigonometric functions;
FFZ,DELZ	: form factor tables and help for interpolation;
NP,NF,NI,NZ	: maximum numbers of parameters, functions, sets of Miller indices, form factor tables.

Because of the great variety for constructing model functions by FUNNY, we will limit the description to some suggestions listed in appendix. The examples consist of a proved common part and three problem oriented inserts, one for magnetic, the others for crystal structure refinements from X-ray and neutron diffraction measurements.

The common part of FUNNY, which may be used for most problems, includes COMMON declarations and a control output. The calculated model functions are listed in comparison with the observed and R-values are printed defined by

$$R'' = \frac{\sum |v_i|}{\sum |f(obs)_i|} .$$

The R-values are calculated including and omitting zero observations.

The problem oriented sections of FUNNY show a common flow. A definition of general parameters is followed by the loop of reflections. Parameters depending on scattering angle are introduced here. Within each reflection loop, an atomic loop includes atomic parameters. Finally the calculated values are summed up to the model functions to be compared with the observations.

V Description of Input Data

1. Title card FORMAT(18A4).

   Cols. 1 -72 Text.

2. Option card FORMAT(24I3).

   Cols. 1 - 3 INTEG(1) = 0 : Weight matrix is diagonal.
   ≠ 0 : Weight matrix is non-diagonal.

   4 - 6 " (2) < 0 : Only Bragg angles output.
   = 0 : Bragg angles and functions output.
   > 0 : Total number of refinement cycles.

   7 - 9 " (3) = 0 : No output of matrices.
   ≠ 0 : Output of matrices.

   10-12 " (4) = 0 : No form factor tables.
   ≠ 0 : Total number of form factor tables.

   13-15 " (5) Not used.
   :
   66-69 " (23) " "

   70-72 " (24) = 0 : Final halt.
   ≠ 0 : Restart after final cycle.

3. Form factor tables, four cards FORMAT(8F9.3) in each table, if INTEG(4) > 0.

   First card cols. 1 - 9 Form factor for $\sin\theta/\lambda$ = 0.00.
   10-18 " " " " = 0.05.
   :
   64-72 " " " " = 0.35.

   Second " " 1 - 9 " " " " = 0.40.
   :

   Fourth " " 64-72 " " " " = 1.55.

4. Function cards FORMAT(I3,2F12.5,6A4,I21).

   Cols. 1 - 3 Function identification number.

   4 -15 Observed values of functions.

   16-27 Standard deviation of functions.

   28-51 Description of function.

   52-72 = 0 : A further function card follows.
   ≠ 0 : This is the last function card.

5. Correlation cards FORMAT(2I2,F12.6,I56), if INTEG(1) ≠ 0.

   Cols. 1 - 2 Function identification number i.

   3 - 4 Function identification number j.

   5 -16 Correlation coefficient between functions i and j.

   17-72 = 0 : A further correlation card follows.
   ≠ 0 : This is the last correlation card.

6. Reflection cards FORMAT(4I3,I60).

Cols. 1 - 3 Reflection identification number.
4 - 6 Miller index h.
7 - 9 " " k.
10-12 " " l.
13-72 = 0 : A further reflection card follows.
≠ 0 : This is the last reflection card.

7. Parameter cards FORMAT(I3,10A4,2F12.7,I5).

Cols. 1 - 3 Parameter identification number.
4 -43 Description of parameter.
44-55 Initial value of parameter.
56-67 Initial increment for calculating derivative.
68-72 = 0 : A further parameter card follows.
≠ 0 : This is the last parameter card.

8. Cell data card FORMAT(7F10.5).

Cols. 1 -10 Wave length in Angstroem.
11-20 a_o " " .
21-30 b_o " " .
31-40 c_o " " .
41-50 Cosine of alpha.
51-60 " " beta.
61-70 " " gamma.

9. Parameter switch cards FORMAT(72I1), one card for each refinement cycle.

Col. 1 = 0 : Parameter number 1 is not to be refined.
≠ 0 : " " 1 is " " " .
2 = 0 : " " 2 is not " " " .
≠ 0 : " " 2 is " " " .
:
72 = 0 : " " 72 is not " " " .
≠ 0 : " " 72 is " " " .

VI Description of Printed Output

The printed output is preceeded by a listing of the input data. Controlled by INTEG(2) a parameter output after each cycle is listed. It is indicated in the first column, whether the parameters were changed in the preceeding cycle or not. The further columns list identification numbers, old values, applied adjustments, new values, and the standard deviations of parameters, respectively. Subsequently the estimated $V^TPV/(n-m)$, the standard deviation of an observation of unit weight, is printed followed by a special FUNNY output (see description of FUNNY).

If INTEG(2) = 0 an output of matrices may be obtained. The matrices printed out are the matrix of the correlation coefficients $\varrho_{ij} = xm_{ij}/(xm_{ii} * xm_{jj})^{1/2}$ of the parameters, the matrix of errors in the parameters XM, and the normalized matrix of normal equations XM^{-1}. The latter is useful for hypothesis test according to W. C. Hamilton (Statistics in Physical Science, New York: The Ronald Press, 1964).

The functions output, also caused by INTEG(2) consists of six columns, representing the identification number of function, its description, the observed and calculated values, their difference and the weighted remaining residuals. The printed R-values are defined as

$$R' = \left[\frac{\sum p_{ii}(f(obs)_i - f(calc)_i)^2}{\sum p_{ii}(f(obs)_i)^2} \right]^{1/2}$$

and

$$R = \frac{V^TPV}{F(obs)^TPF(obs)}.$$

If INTEG(2) < 0 the output of Bragg angles and related crystallographic quantities is listed like Miller indices, $\sin\Theta$, $\sin(2\Theta)$, Θ and 2Θ.

APPENDIX A LISTING OF SOURCE TEXT POWLS
(WITHOUT PROBLEM ORIENTED SECTION OF FUNNY)

```
C     POWLS-60
C     MAIN PROGRAM FOR REFINEMENT OF POWDER DIFFRACTION DATA
C
C     DEVELOPED BY G. WILL & D. E. COX (1962)
C     IN COOPERATION WITH W. C. HAMILTON
C     PUBLISHED IN J. APPL. CRYST. 12, 483 (1979)
C     DOCUMENTED BY E. JANSEN & W. SCHAEFER, BONN/JUELICH
C
      COMMON /FCT/ NF,NO(50),FOBS(50),FCALC(50),SIGF1(50),DELF1(50)
      COMMON /HKL/ NI,IH(200),IK(200),IL(200),SINT(200),SIN2T(200)
      COMMON /INT/ INTEG(24),ISEL(72),NCOUNT,ICYC
      COMMON /LTC/ AS,BS,CS,CAS,CBS,CCS,WAVE
      COMMON /LSQ/ V(50),X(20),XM(20,20),B(20,20),RHO(50,50),FAX
      COMMON /MGN/ NZ,FFZ(15,32),DELZ(15,31)
      COMMON /PRM/ NP,NPVAR,PVALUE(72),PNEW(72),ADJUST(72),SIGMA(72)
      COMMON /TXT/ HOLLER(18),WHAT(72,10),HOW(50,6)
C
      DIMENSION AFCALC(50),MARK(50),PNOW(50),PINCR(50),AMAT(50,20)
C
      FLOATF(II)=FLOAT(II)
      SQRTF(XX)=SQRT(XX)
C
C-----READ & WRITE TITLE & OPTIONS
  782 READ(5,1)(HOLLER(I),I=1,18)
      WRITE(6,105)(HOLLER(I),I=1,18)
      READ(5,2)(INTEG(I),I=1,24)
      WRITE(6,106)
      WRITE(6,2)(INTEG(I),I=1,24)
      ICYC=INTEG(2)
      NZ=INTEG(4)
C-----IF INTEG(4) .= 0 READ & WRITE FORM FACTOR TABLES
      IF(NZ)322,322,321
  321 WRITE(6,104)
      DO306IZ=1,NZ
      READ(5,307)(FFZ(IZ,INZ),INZ=1,32)
      WRITE(6,307)(FFZ(IZ,INZ),INZ=1,32)
      DO308ISZ=1,31
  308 DELZ(IZ,ISZ)=(FFZ(IZ,ISZ)-FFZ(IZ,ISZ+1))/0.05
  306 CONTINUE
  322 CONTINUE
C-----READ & WRITE OBSERVATIONS & RELATED MARGINS OF ERROR
      NCOUNT=0
      WRITE (6,100)
    5 READ(5,3)IF,FOBS(IF),SIGF1(IF),(HOW(IF,J),J=1,6),ITEST
      WRITE(6,9)IF,FOBS(IF),SIGF1(IF),(HOW(IF,J),J=1,6),ITEST
      NCOUNT=NCOUNT+1
      MARK(IF)=NCOUNT
      NO(NCOUNT)=IF
      IF(ITEST)6,5,6
    6 NF=NCOUNT
C-----SET DEFAULT CORRELATIONS IN OBSERVATIONS
      DO7I=1,NF
```

```
      DO7J=1,NF
      RHO(I,J)=0.
    7 RHO(J,J)=1.
C-----IF INTEG(1) ¬= 0 READ ADDITIONAL CORRELATIONS IN OBS.
      IF(INTEG(1))10,11,10
   10 READ(5,4)I,J,RHOIJ,ITEST
      K=MARK(I)
      L=MARK(J)
      RHO(K,L)=RHOIJ
      IF(ITEST)11,10,11
C-----DEVELOP ERROR MATRIX OF OBSERVATIONS
   11 DO15I=1,NF
      DO15J=1,NF
      IF(RHO(I,J))8,15,8
    8 K=NO(I)
      L=NO(J)
      RHO(I,J)=RHO(I,J)*SIGF1(K)*SIGF1(L)
   15 CONTINUE
C-----INVERT ERROR MATRIX INTO WEIGHT MATRIX
 1000 IF(INTEG(1))1002,1001,1002
 1002 CALLSMI50(RHO,NF)
      GOTO505
 1001 DO1003I=1,NF
 1003 RHO(I,I)=1./RHO(I,I)
C-----READ MILLER INDICES
  505 NCOUNT=0
      WRITE(6,101)
   18 READ(5,16)IRUN,IH(IRUN),IK(IRUN),IL(IRUN),ITEST
      WRITE(6,16)IRUN,IH(IRUN),IK(IRUN),IL(IRUN),ITEST
      NCOUNT=NCOUNT+1
      IF(ITEST)17,18,17
   17 NI=NCOUNT
C-----READ START PARAMETERS AND RELATED INCREMENTS FOR DERIVATIVES
      NCOUNT=0
      WRITE(6,102)
   19 READ(5,20)IP,(WHAT(IP,J),J=1,10),PVALUE(IP),PINCR(IP),ITEST
      WRITE(6,20)IP,(WHAT(IP,J),J=1,10),PVALUE(IP),PINCR(IP),ITEST
      NCOUNT=NCOUNT+1
      IF(ITEST)21,19,21
   21 NP=NCOUNT
C-----READ CELL CONSTANTS (COSINES OF ANGLES)
      WRITE(6,103)
      READ(5,22)WAVE,AO,BO,CO,CA,CB,CC
      WRITE(6,22)WAVE,AO,BO,CO,CA,CB,CC
C-----CALCULATE RECIPROCAL CELL CONSTANTS
      SA=SQRTF(1.-CA*CA)
      SB=SQRTF(1.-CB*CB)
      SC=SQRTF(1.-CC*CC)
      CAS=(CB*CC-CA)/(SB*SC)
      CBS=(CA*CC-CB)/(SA*SC)
      CCS=(CA*CB-CC)/(SA*SB)
      SAS=SQRTF(1.-CAS*CAS)
      SBS=SQRTF(1.-CBS*CBS)
      AS=1./(AO*SBS*SC)
      BS=1./(BO*SAS*SC)
      CS=1./(CO*SAS*SB)
C-----CALCULATE TABLE OF TRIGONOMETRIC FUNCTIONS
```

```
      DO25I=1,NI
      DUM1=FLOATF(IH(I))*AS
      DUM2=FLOATF(IK(I))*BS
      DUM3=FLOATF(IL(I))*CS
C     ---SIN(THETA)
      SINT(I)=(WAVE/2.)*SQRTF(DUM1*DUM1+DUM2*DUM2
     1+DUM3*DUM3+2.*(DUM1*DUM2*CCS+DUM1*DUM3
     2*CBS+DUM2*DUM3*CAS))
C     ---SIN(2*THETA)
   25 SIN2T(I)=2.*SINT(I)*SQRTF(1.-SINT(I)*SINT(I))
C
C     *** START OF REFINEMENT PROCEDURE ***
C
C-----IF INTEG(2) < 0 CALL BRAGG ANGLE OUTPUT
C     IF INTEG(2) = 0 FINAL CALCULATION OF MODEL FUNCTIONS
   90 IF(INTEG(2))28,60,26
C-----READ PARAMETERS TO BE VARIED
   26 READ(5,29)(ISEL(I),I=1,72)
C-----DEVELOP SYSTEM OF EQUATIONS IN PARAMETER ADJUSTMENTS
C     ---CALCULATE MODEL FUNCTIONS FROM PARAMETERS
   60 NCOUNT=0
      CALLFUNNY(PVALUE,FCALC)
C     ---IF INTEG(2) <= 0 CALL FINAL OUTPUT
      IF(INTEG(2))28,28,27
C     ---CALCULATE NEIGHBOUR FUNCTIONS BY VARYING ONE PARAMETER ONLY
   27 NCOUNT=0
      DO30I=1,NP
      IF(ISEL(I)-1)30,33,30
   33 NCOUNT=NCOUNT+1
      DO34J=1,NP
   34 PNOW(J)=PVALUE(J)
      PNOW(I)=PNOW(I)+PINCR(I)
      CALLFUNNY(PNOW,AFCALC)
      DO35J=1,NF
      K=NO(J)
C     ---RESIDUAL FUNCTIONS
      DELF1(J)=FOBS(K)-FCALC(K)
C     ---MATRIX OF DERIVATIVES
   35 AMAT(J,NCOUNT)=(AFCALC(K)-FCALC(K))/PINCR(I)
   30 CONTINUE
C-----WEIGHT & SOLVE EQUATIONS
      NPVAR=NCOUNT
      CALLWEAST(DELF1,AMAT,X,XM,B,FAX,NPVAR,NF,RHO,V)
C-----ADJUST PARAMETERS, DETERMINE MARGINS OF ERROR
      NCOUNT=0
      DO40I=1,NP
      IF(ISEL(I)-1)42,41,42
   41 NCOUNT=NCOUNT+1
      ADJUST(I)=X(NCOUNT)
      PNEW(I)=ADJUST(I)+PVALUE(I)
      SIGMA(I)=SQRTF(XM(NCOUNT,NCOUNT))
      GOTO40
   42 ADJUST(I)=0
      PNEW(I)=PVALUE(I)
      SIGMA(I)=0
   40 CONTINUE
C-----CALL CURRENT PARAMETER OUTPUT
```

```
   28 CALLOUTPUT
C-----IF INTEG(2) < 0 ASK FOR EXIT OR NEW START
C      IF INTEG(2) = 0 DECREMENT INTEG(2) ONLY
       IF(INTEG(2))333,1234,334
C-----REPLACE PARAMETERS. CALCULATE INCREMENTS FOR DERIVATIVES
C      & DECREMENT INTEG(2)
  334 DO43I=1,NP
       PVALUE(I)=PNEW(I)
       IF(SIGMA(I))43,43,510
  510 PINCR(I)=0.1*SIGMA(I)
   43 CONTINUE
 1234 INTEG(2)=INTEG(2)-1
C
C      *** END OF REFINEMENT PROCEDURE ***
C
C-----START NEW REFINEMENT PROCEDURE WITH READING NEW PARAMETERS
C      TO BE VARIED
       GOTO90
C-----IF INTEG(24) > 0 START PROGRAM AGAIN WITH NEW DATA SET
C      IF INTEG(24) = 0 START AGAIN WITH NEW OPTIONS & PARAMETERS ONLY
  333 IF(INTEG(24)-1)777,778,779
  777 CALLEXIT
C-----CALL EXIT
  778 READ(5,2)(INTEG(I),I=1,24)
       ICYC=INTEG(2)
  781 READ(5,20)IP,(WHAT(IP,J),J=1,7),PVALUE(IP),PINCR(IP),ITEST
       IF(ITEST)90,781,90
  779 GOTO782
C
    1 FORMAT(18(A4))
    2 FORMAT(24I3)
    3 FORMAT(I3,2F12.5,6A4,I21)
    4 FORMAT(2I2,F12.6,I56)
    9 FORMAT(I3,2F12.5,4X,6A4,I17)
   16 FORMAT(4I3,I60)
   20 FORMAT(I3,10A4,2F12.7,I5)
   22 FORMAT(7F10.5)
   29 FORMAT(72I1)
  100 FORMAT('0OBSERVATIONS')
  101 FORMAT('0MILLER INDICES')
  102 FORMAT('0PARAMETERS')
  103 FORMAT('0CELL CONSTANTS')
  104 FORMAT('0FORM FACTOR TABLES')
  105 FORMAT(1X,18A4//)
  106 FORMAT('0OPTIONS')
  307 FORMAT(8F9.3)
C
       END



C      POWLS-60
C      OUTPUT
C
       SUBROUTINEOUTPUT
       COMMON /FCT/ NF,NO(50),FOBS(50),FCALC(50),SIGF1(50),DELF1(50)
       COMMON /HKL/ NI,IH(200),IK(200),IL(200),SINT(200),SIN2T(200)
```

```
      COMMON /INT/ INTEG(24),ISEL(72),NCOUNT,ICYC
      COMMON /LTC/ AS,BS,CS,CAS,CBS,CCS,WAVE
      COMMON /LSQ/ V(50),X(20),XM(20,20),B(20,20),RHO(50,50),FAX
      COMMON /PRM/ NP,NPVAR,PVALUE(72),PNEW(72),ADJUST(72),SIGMA(72)
      COMMON /TXT/ HOLLER(18),WHAT(72,10),HOW(50,6)
C
      DIMENSION NODUM(50),YES(2),DUMMY(20,20)
      DATA YES/'YES','NO'/
C
      FLOATF(II)=FLOAT(II)
      SQRTF(XX)=SQRT(XX)
C
C-----WRITE TITLE
      WRITE(6,1)
      WRITE(6,2)(HOLLER(I),I=1,18)
C-----IF INTEG(2) > 0 CURRENT PARAMETER OUTPUT
C     IF INTEG(2) = 0 FINAL OUTPUT
      IF(INTEG(2))101,102,103
C
C     *** BRAGG ANGLE OUTPUT ***
C
C-----CALCULATE & LIST BRAGG ANGLES & RELATED FUNCTIONS OF H,K,L
  101 WRITE(6,3)
      DO201I=1,NI
      ODD1=ARSIN(SINT(I))*57.3
      ODD2=2.*ODD1
  201 WRITE(6,4)IH(I),IK(I),IL(I),SINT(I),SIN2T(I),ODD1,ODD2
      RETURN
C
C     *** FINAL OUTPUT ***
C
C-----ACCUMULATE RESIDUALS & OBSERVATIONS WEIGHTED DUE TO ERRORS IN OBS.
  102 WRITE(6,5)
      WRITE(6,6)
      WRITE(6,7)
      WRITE(6,6)
      ODD5=0
      ODD6=0
      DO202I=1,NF
      K=NO(I)
      DELF1(K)=FOBS(K)-FCALC(K)
      ODD4=DELF1(K)/SIGF1(K)
      ODD5=ODD5+ODD4**2
      ODD6=ODD6+(FOBS(K)/SIGF1(K))**2
C-----LIST OBSERVATIONS, FINAL RELATED CALCULATIONS & RESIDUALS
  202 WRITE(6,8)K,(HOW(K,KK),KK=1,6),FOBS(K),FCALC(K),DELF1(K),ODD4
C-----CALCULATE & WRITE R-FACTOR WITHOUT CORRELATIONS IN OBSERVATION
      AKIM=SQRTF(ODD5/ODD6)
      WRITE(6,20)AKIM
C-----ACCUMULATE RESIDUALS & OBSERVATIONS WEIGHTED BY WEIGHT MATRIX
      ODD4=0
      ODD5=0
      DO203I=1,NF
      DO203J=1,NF
      ODD5=ODD5+RHO(I,J)*FOBS(I)*FOBS(J)
  203 ODD4=ODD4+RHO(I,J)*DELF1(I)*DELF1(J)
C-----CALCULATE & WRITE R-FACTOR WITH CORRELATIONS IN OBSERVATIONS
```

```
      ODD5=SQRTF(ODD4/ODD5)
      WRITE(6,25)ODD5
C-----WRITE VPV (CORRELATION INCLUDED)
      WRITE(6,9)ODD4
C-----LIST FINAL PARAMETERS
      WRITE(6,10)
      WRITE(6,11)
      DO204I=1,NP
  204 WRITE(6,12)I,(WHAT(I,J),J=1,10),PVALUE(I)
      RETURN
C
C     *** CURRENT PARAMETER OUTPUT ***
C
  103 IODD5=ICYC-INTEG(2)+1
      WRITE(6,13)IODD5,ICYC
      WRITE(6,14)
      WRITE(6,6)
      DO205I=1,NP
C-----IF PARAMETER TO BE VARIED YES = 'YES'
      IF(ISEL(I))270,270,271
  271 NYES=1
      GOTO205
  270 NYES=2
  205 WRITE(6,15)YES(NYES),I,PVALUE(I),ADJUST(I),PNEW(I),SIGMA(I)
C-----WRITE VPV/(N-M)
      WRITE(6,16)FAX
C-----IF INTEG(2) ¬= 0 RETURN
      IF(INTEG(2)-1)206,207,206
C-----IF INTEG(3) = 0 RETURN
  207 IF(INTEG(3))208,206,208
  206 RETURN
C
C     *** MATRIX OUTPUT ***
C
C-----CORRELATION MATRIX FOR PARAMETERS
  208 WRITE(6,17)
      K=1
      DO250I=1,NP
      IF(ISEL(I))250,250,252
  252 NODUM(K)=I
      K=K+1
  250 CONTINUE
      WRITE(6,18)(NODUM(K),K=1,NPVAR)
      DO209I=1,NPVAR
      DO210J=1,NPVAR
  210 DUMMY(I,J)=XM(I,J)/SQRTF(XM(I,I)*XM(J,J))
  209 WRITE(6,19)NODUM(I),(DUMMY(I,J),J=1,NPVAR)
C-----ERROR MATRIX FOR PARAMETERS
      WRITE(6,21)
      WRITE(6,23)(NODUM(K),K=1,NPVAR)
      DO211I=1,NPVAR
  211 WRITE(6,22)NODUM(I),(XM(I,J),J=1,NPVAR)
C-----NORMALIZED MATRIX OF NORMAL EQUATIONS
      WRITE(6,24)
      WRITE(6,23)(NODUM(K),K=1,NPVAR)
      DO212I=1,NPVAR
      DO213J=1,NPVAR
```

```
  213 B(I,J)=B(I,J)/FAX
  212 WRITE(6,22)NODUM(I),(B(I,J),J=1,NPVAR)
      RETURN
    1 FORMAT(88H1STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPP
     1ING POWDER DATA.                    )
    2 FORMAT(1X,18(A4))
    3 FORMAT(72HO TRIGONOMETRIC FUNCTIONS  H   K   L   SINTHETA SIN2THET
     1A THETA  2THETA    )
    4 FORMAT(26X,I3,1X,I3,1X,I3,F9.5,1X,F9.5,2F8.2)
    5 FORMAT(1HO,25X,22HFUNCTIONS OF INTENSITY)
    6 FORMAT(1HO)
    7 FORMAT(2X,3HNO.,8X,11HDESCRIPTION,12X,54HOBSERVED     CALCULATED
     1     DELTA        DELTA/SIGMA       )
    8 FORMAT(2X,I2,2X,6A4,4E15.4)
    9 FORMAT(6HO VPV=E11.4,24H (CORRELATIONS INCLUDED))
   10 FORMAT(69H1THE F VALUES ABOVE ARE CALCULATED FOR THE FOLLOWING PAR
     1AMETER VALUES           )
   11 FORMAT(10HOPARAMETER,16X,11HDESCRIPTION,23X,5HVALUE)
   12 FORMAT(1HO,I4,8X,10A4,E16.6)
   13 FORMAT(22HOLEAST SQUARES. CYCLE I2,5H OF  I2,8H CYCLES.)
   14 FORMAT(68HOREF$  PAM.      OLD VALUE       CHANGE         NEW VALUE
     1        SIGMA    )
   15 FORMAT(1H ,A3,3X,I2,2X,4E16.6)
   16 FORMAT(21HOESTIMATED VPV/(N-M)=E11.4)
   17 FORMAT(19HOCORRELATION MATRIX)
   18 FORMAT(5HO    ,20I5)
   19 FORMAT(1HO,I2,3X,20F5.2)
   20 FORMAT(38HOWEIGHTED R FACTOR FOR FUNCTIONS IS   F6.4,46H.   CORREL
     1ATIONS IN OBSERVATIONS NOT INCLUDED.)
   21 FORMAT(25HOMATRIX OF SECOND MOMENTS)
   22 FORMAT(1HO,I2,3X,10E11.4)
   23 FORMAT(1HO,10I11)
   24 FORMAT(66HONORMALIZED MATRIX OF NORMAL EQUATIONS FOR USE IN HYPOTH
     1ESIS TESTS       )
   25 FORMAT(36HODITTO BUT CORRELATIONS INCLUDED. R=F7.4,1H.)
C
       END



C      POWLS-60
C      WEAST (WEIGHTED LEAST SQUARES)
C
       SUBROUTINEWEAST(F,A,X,XM,B,FAX,NP,NO,P,V)
       DIMENSIONF(50),A(50,20),X(20),XM(20,20)
       DIMENSIONB(20,20),P(50,50),V(50),DUMMY(50,50)
C
       DO1I=1,NP
       DO1J=1,NO
       DUMMY(I,J)=0
       DO1K=1,NO
 1     DUMMY(I,J)=DUMMY(I,J)+A(K,I)*P(K,J)
       DO2I=1,NP
       DO2J=1,NP
       B(I,J)=0
       DO2K=1,NO
 2     B(I,J)=B(I,J)+DUMMY(I,K)*A(K,J)
```

```
      DO4I=1,NP
      V(I)=0
      DO4K=1,NQ
 4    V(I)=V(I)+F(K)*DUMMY(I,K)
      DO5I=1,NP
      DO5J=1,NP
 5    DUMMY(I,J)=B(I,J)
      CALLSMI5O(DUMMY,NP)
      DO6I=1,NP
      X(I)=0
      DO6K=1,NP
 6    X(I)=X(I)+DUMMY(I,K)*V(K)
      VPV=0
      DO7J=1,NQ
      V(J)=F(J)
      DO7I=1,NP
 7    V(J)=V(J)-X(I)*A(J,I)
      DO8I=1,NQ
      DO8J=1,NQ
 8    VPV=VPV+V(J)*V(I)*P(I,J)
      FAX=VPV/FLOAT(NQ-NP)
      DO9I=1,NP
      DO9J=1,NP
 9    XM(I,J)=DUMMY(I,J)*FAX
      RETURN
      END


C     POWLS-60
C     SMI 50 (SYMMETRIC MATRIX INVERSION)
C
      SUBROUTINE SMI5O(D,N)
      DIMENSION D(50,50),S(50)
C
      DO2J=1,N
      DO1I=1,J
      D(I,J)=-D(I,J)
    1 D(J,I)=D(I,J)
    2 D(J,J)=1.+D(J,J)
      DO8LR=1,N
      IF(1.-D(LR,LR).EQ.0.) GO TO 9
      D(LR,LR)=1./(1.-D(LR,LR))
      DO4J=1,N
      S(J)=D(LR,J)
      IF(J-LR) 3,4,3
    3 D(J,LR)=D(J,LR)*D(LR,LR)
      D(LR,J)=D(J,LR)
    4 CONTINUE
      DO8J=1,N
      IF(J-LR) 5,8,5
    5 DO7I=1,J
      IF(I-LR) 6,7,6
    6 D(I,J)=D(I,J)+D(I,LR)*S(J)
      D(J,I)=D(I,J)
    7 CONTINUE
    8 CONTINUE
```

```
      RETURN
    9 WRITE(6,10)
   10 FORMAT('0ERROR IN SMI50')
      CALL EXIT
      END



C     POWLS-60
C     FUNNY (MODEL FUNCTIONS)
C
      SUBROUTINEFUNNY(PIZ,FZ)
      COMMON /FCT/ NF,NO(50),FOBS(50),FCALC(50),SIGF1(50),DELF1(50)
      COMMON /HKL/ NI,IH(200),IK(200),IL(200),SINT(200),SIN2T(200)
      COMMON /INT/ INTEG(24),ISEL(72),NCOUNT,ICYC
      COMMON /LTC/ AS,BS,CS,CAS,CBS,CCS,WAVE
      COMMON /MGN/ NZ,FFZ(15,32),DELZ(15,31)
C
      DIMENSION PIZ(72),FZ(50)
C
C-----SET CONSTANTS
      PZ=6.2831853
      RADZ=57.295779
      BG=0.017
C*******************************************************************************
C
C     *** PROBLEM ORIENTED PART ***
C
C     TO BE PREPARED INDIVIDUALLY
C     EXAMPLES SEE APPENDIX B
C
C*******************************************************************************
C-----IF FUNNY IS CALLED FOR CALCULATING NEIGHBOUR FUNCTIONS RETURN
      IF(NCOUNT)603,604,603
C
C     *** CURRENT CONTROL OUTPUT ***
C-----WRITE TABLES OF CALCULATED INTENSITIES & RELATED OBSERVATIONS
  604 ICON = INTEG(5)
  610 WRITE(6,381)
      DO375JO=1,10
      JO1=JO+10
      JO2=JO+20
      JO3=JO+30
      JO4=JO+40
      WRITE(6,305)JO,FZ(JO),FOBS(JO),JO1,FZ(JO1),FOBS(JO1),JO2,FZ(JO2),
     1FOBS(JO2),JO3,FZ(JO3),FOBS(JO3),JO4,FZ(JO4),FOBS(JO4)
  375 CONTINUE
C-----CALCULATE & WRITE R-FACTORS INCLUDING & OMITTING ZERO OBS.
      FRZZ=0.0
      FRNZ=0.0
      FRZY = 0.0
      FRNY = 0.0
      DO304J=1,NF
      FRZZ=FRZZ+ABS(FOBS(J)-FZ(J))
      FRNZ=FRNZ+FOBS(J)
      ICHEK = FOBS(J)
      IF(ICHEK)    620,620,621
```

```
  621 FRZY = FRZY + ABS(FOBS(J) - FZ(J))
      FRNY = FRNY + FOBS(J)
  620 CONTINUE
  304 CONTINUE
      FRZ=FRZZ/FRNZ
      IF (FRNY.EQ.0.)GO TO 1000
      FRY = FRZY / FRNY
 1000 WRITE(6,312)FRZ
      WRITE(6,314)FRY
  603 RETURN
C
  305 FORMAT(1I5,2F9.2,1I5,2F9.2,1I5,2F9.2,1I5,2F9.2,1I5,2F9.2)
  312 FORMAT('0',40X,34HR - FACTOR  INCLUDING  ZEROS      =F10.4)
  314 FORMAT('0',40X,34HR - FACTOR  WITHOUT    ZEROS      =F10.4)
  381 FORMAT(117H1  NO  I(CALC)  I (OBS)   NO  I(CALC)  I (OBS)   NO  I(
     1CALC)  I (OBS)   NO  I(CALC)  I (OBS)   NO  I(CALC)  I (OBS)  ///)
C
      END
```

APPENDIX B LISTING OF SOURCE TEXT FUNNY
(PROBLEM ORIENTED SECTIONS)

EXAMPLE 1

```
C     FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION
C     SPACE GROUP PNMA (NO. 62 INT. TABLES)
C     PHYS. STAT. SOL. (A) 40, 447 (1977)
C
      DIMENSION SFZ(100)
C
C-----SET ACUTAL OVERALL PARAMETERS
C     ---SCALE FACTOR SCZ
      SCZ=PIZ(24)
C     ---OVERALL TEMPERATURE FACTOR OTZ
      OTZ=PIZ(25)
C
C     *** LOOP OF REFLECTIONS ***
      DO 200 JHKL=1,NI
C-----SET ACTUAL MILLER INDICES AH,AK,AL
      AH=IH(JHKL)
      AK=IK(JHKL)
      AL=IL(JHKL)
C-----RESET STRUCTURE FACTOR
      SFZ(JHKL)=0.0
C
C       * LOOP OF ATOMS IN ASYMMETRIC UNIT *
      NAT = 5
      DO 300 JAT=1,NAT
C     ---SET ACTUAL ATOMIC PARAMETERS
C        POSITIONS AX,AY,AZ
      AX=PIZ(JAT)
      AY=PIZ(JAT+NAT)
      AZ=PIZ(JAT+2*NAT)
C        SITE OCCUPANCIES WZ
      WZ=PIZ(JAT+3*NAT)
C        SCATTERING LENGTHS BZ
      BZ=PIZ(JAT+4*NAT)
      IF(JAT.GT.3)BZ=PIZ(23)
C     ---CALCULATE STRUCTURE FACTOR (FORMULA FROM INTERNATIONAL TABLES)
      CC=8.0*COS(PZ*(AH*AX-(AH+AK+AL)/4.0))*COS(PZ*(AK*AY+AK/4.0))*COS(P
     FZ*(AL*AZ+(AH+AL)/4.0))
C        ACCUMULATE STRUCTURE FACTOR
      SFZ(JHKL)=SFZ(JHKL)+BZ*WZ*CC
  300 CONTINUE
C       * END OF ATOM LOOP *
C
C-----CALCULATE INDIVIDUAL INTENSITIES
      SFZ(JHKL)=SFZ(JHKL)**2
      SFZ(JHKL)=SFZ(JHKL) * SCZ / (SINT(JHKL)*SIN2T(JHKL))
      SFZ(JHKL)=SFZ(JHKL)*EXP(-2.0 * OTZ * (SINT(JHKL)/WAVE)**2)
  200 CONTINUE
C     *** END OF REFLECTION LOOP ***
C
```

```
C-----CALCULATE BLOCK INTENSITIES INCLUDING MULTIPLICITIES
C      (MODEL FUNCTIONS)
       FZ(1)=2.0*SFZ(1)+4.0*SFZ(2)
       FZ(2)=4.0*SFZ(3)
       FZ(3)=4.0*SFZ(4)+8.0*SFZ(5)+2.0*SFZ(6)
       FZ(4)=4.0*SFZ(7)
       FZ(5)=8.0*SFZ(8)
       FZ(6)=4.0*SFZ(9)+8.0*SFZ(10)
       FZ(7)=4.0*SFZ(11)
       FZ(8)=8.0*SFZ(12)+2.0*SFZ(13)
       FZ(9)=4.0*SFZ(14)+4.0*SFZ(15)
       FZ(10)=4.0*SFZ(16)+8.0*SFZ(17)+8.0*SFZ(18)
       FZ(11)=8.0*SFZ(19)+4.0*SFZ(20)+4.0*SFZ(21)
       FZ(12)=8.0*SFZ(22)+8.0*SFZ(23)
       FZ(13)=8.0*SFZ(24)+2.0*SFZ(25)+8.0*SFZ(26)
       FZ(14)=4.0*SFZ(27)+8.0*SFZ(28)+8.0*SFZ(29)+4.0*SFZ(30)+8.0*SFZ(31)
      F+4.0*SFZ(32)+8.0*SFZ(33)+4.0*SFZ(34)+4.0*SFZ(35)+8.0*SFZ(36)
       FZ(15)=2.0*SFZ(37)+8.0*SFZ(38)+8.0*SFZ(39)+4.0*SFZ(40)
       FZ(16)=4.0*SFZ(41)+4.0*SFZ(42)+8.0*SFZ(43)+8.0*SFZ(44)+8.0*SFZ(45)
      F+4.0*SFZ(46)



       APPENDIX B EXAMPLE 2


C      FESO4 STRUCTURE REFINEMENT BY X-RAY DIFFRACTION
C      SPACE GROUP PNMA (NO. 62 INT. TABLES)
C      PHYS. STAT. SOL. (A) 40, 447 (1977)
C
       DIMENSION SFZ(100)
C
C-----SET ACTUALL OVERALL PARAMETERS
C      ---SCALE FACTOR SCZ
       SCZ=PIZ(21)
C      ---OVERALL TEMPERATURE FACTOR OTZ
       OTZ=PIZ(22)
C
C      *** LOOP OF REFLECTIONS ***
       DO 200 JHKL=1,NI
C-----SET ACTUAL MILLER INDICES AH,AK,AL
       AH=IH(JHKL)
       AK=IK(JHKL)
       AL=IL(JHKL)
C-----RESET STRUCTURE FACTOR
       SFZ(JHKL)=0.0
C
C        * LOOP OF ATOMS IN ASYMMETRIC UNIT *
       NAT = 5
       DO 300 JAT=1,NAT
C      ---SET ACTUAL ATOMIC PARAMETERS
C         POSITIONS AX,AY,AZ
       AX=PIZ(JAT)
       AY=PIZ(JAT+NAT)
       AZ=PIZ(JAT+2*NAT)
C         SITE OCCUPANCIES WZ
       WZ=PIZ(JAT+3*NAT)
```

```
C         SCATTERING LENGTHS BZ
        NT=20.*SINT(JHKL)/WAVE
        DT=SINT(JHKL)/WAVE-.05*FLOAT(NT)
        BZ=FFZ(JAT,NT+1)-DT*DELZ(JAT,NT+1)
C       ---CALCULATE STRUCTURE FACTOR (FORMULA FROM INTERNATIONAL TABLES)
        CC=8.0*COS(PZ*(AH*AX-(AH+AK+AL)/4.0))*COS(PZ*(AK*AY+AK/4.0))*COS(P
       FZ*(AL*AZ+(AH+AL)/4.0))
C         ACCUMULATE STRUCTURE FACTOR
        SFZ(JHKL)=SFZ(JHKL)+BZ*WZ*CC
  300 CONTINUE
C       * END OF ATOM LOOP *
C
C-----CALCULATE INDIVIDUAL INTENSITIES
        SFZ(JHKL)=SFZ(JHKL)**2
        SFZ(JHKL)=SFZ(JHKL) * SCZ / (SINT(JHKL)*SIN2T(JHKL))
        SFZ(JHKL)=SFZ(JHKL)*EXP(-2.0 * OTZ * (SINT(JHKL)/WAVE)**2)
  200 CONTINUE
C       *** END OF REFLECTION LOOP ***
C
C-----CALCULATE BLOCK INTENSITIES INCLUDING MULTIPLICITIES
C       (MODEL FUNCTIONS)
        FZ(1)=2.*SFZ(1)
        FZ(2)=4.*SFZ(2)
        FZ(3)=4.*SFZ(4)
        FZ(4)=8.*SFZ(5)
        FZ(5)=4.*SFZ(9)
        FZ(6)=8.*SFZ(10)
        FZ(7)=4.*SFZ(11)
        FZ(8)=2.*SFZ(37)
        FZ(9)=8.*SFZ(38)
        FZ(10)=4.*SFZ(40)
        FZ(11)=2.*SFZ(13)
        FZ(12)=4.*SFZ(14)
        FZ(13)=4.*SFZ(15)
        FZ(14)=4.*SFZ(41)
        FZ(15)=8.*SFZ(44)
        FZ(16)=8.*SFZ(45)
        FZ(17)=4.*SFZ(16)
        FZ(18)=4.*SFZ(21)
        FZ(19)=8.*(SFZ(23)+SFZ(22))
        FZ(20)=2.*SFZ(25)
        FZ(21)=4.*SFZ(27)
        FZ(22)=4.*SFZ(30)
        FZ(23)=8.*SFZ(31)
        FZ(24)=8.*SFZ(33)
        FZ(25)=8.*SFZ(36)+4.*(SFZ(34)+SFZ(35))



        APPENDIX B EXAMPLE 3


C       DYAS04 MAGNETIC STRUCTURE ANALYSIS BY NEUTRON DIFFRACTION
C       J. PHYS. C: SOLID ST. PHYS. 4, 3224 (1971)
C
        DIMENSION BZ(5),SFZ(50)
        REAL H,K,L
```

```
C
C      --SCALE AND TEMPERATURE FACTOR PARAMETERS
       SC=PIZ(17)
       TF=PIZ(18)
C      --MAGNETIC STRUCTURE CONSTANTS AND PARAMETERS
       FF=.2695
       AM=PIZ(19)
       PA=PIZ(20)*BG
       PB=PZ/4.-PA
       PC=PIZ(21)*BG
C
C       *** LOOP OF REFLECTIONS ***
C
       DO 200 JH=1,NI
C          --CALCULATE MAGNETIC FORM FACTORS
           NT=20.*SINT(JH)/WAVE
           DT=SINT(JH)/WAVE-.05*FLOAT(NT)
           DO 100 I=1,NZ
  100         BZ(I)=FFZ(I,NT+1)-DT*DELZ(I,NT+1)
C          --SET ACTUAL PARAMETERS H,K,L
           H=IH(JH)
           K=IK(JH)
           L=IL(JH)
C          --RESET STRUCTURE FACTOR COMPONENTS
           A=0.
           B=0.
C
C          *** LOOP OF ATOMS ***
C
           NA=4
           DO 300 JA=1,NA
C             --SET ACTUAL ATOMIC PARAMETERS
              X=PIZ(JA)
              Y=PIZ(JA+NA)
              Z=PIZ(JA+2*NA)
              S=PIZ(JA+3*NA)
C             --ACCUMULATE STRUCTURE FACTOR
              A=A+S*COS(PZ*(H*X+K*Y+L*Z))
              B=B+S*SIN(PZ*(H*X+K*Y+L*Z))
  300      CONTINUE
C
C          *** END OF ATOMS LOOP ***
C
C          --INCLUDE TEMPERATURE CORRECTION
           AB=SQRT(A**2+B**2)*EXP(-TF*(SINT(JH)/WAVE)**2)
C          --COMPLETE MAGNETIC INTENSITIES
           QQ=1.-((H*AS*COS(PA))**2
     +            +(K*BS*COS(PB))**2
     +            +(L*CS*COS(PC))**2)*(WAVE/(2*SINT(JH)))**2
           AB=(AB*FF*AM*BZ(1))**2*QQ*SC/(SINT(JH)*SIN2T(JH))
C          --INCLUDE MULTIPLICITIES
           IF(H.NE.0)AB=2*AB
           IF(K.NE.0)AB=2*AB
           IF(L.NE.0)AB=2*AB
C          --STORE AB, CLEAR  MODEL FUNCTIONS
           SFZ(JH)=AB
           FZ(JH)=0.
```

```
  200 CONTINUE
C
C      *** END OF REFLECTIONS LOOP ***
C
C      --CALCULATE BLOCK INTENSITIES (MODEL FUNCTIONS)
     1 FZ(1)=SFZ(1)
     2 FZ(2)=SFZ(2)
     3 FZ(3)=SFZ(3)
       DO 4 I=4,6
     4 FZ(4)=FZ(4)+SFZ(I)
     5 FZ(5)=SFZ(7)
     6 FZ(6)=SFZ(8)
       DO 7 I=9,11
     7 FZ(7)=FZ(7)+SFZ(I)
       DO 8 I=12,14
     8 FZ(8)=FZ(8)+SFZ(I)
     9 FZ(9)=SFZ(15)+SFZ(16)
    10 FZ(10)=SFZ(17)+SFZ(18)
```

APPENDIX C LISTING OF PRINTED OUTPUT

EXAMPLE 1

```
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION


OPTIONS
 0  6  0  0  1  1  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0

OBSERVATIONS
 1   5000.00000    100.00000     200 101                                0
 2     20.00000     20.00000     011                                    0
 3   8650.00000    100.00000     210 111 020                            0
 4    280.00000     20.00000     201                                    0
 5     50.00000     20.00000     211                                    0
 6   4400.00000    100.00000     220 121                                0
 7   2050.00000     50.00000     301                                    0
 8   1830.00000     50.00000     112 400                                0
 9    520.00000     30.00000     202 410                                0
10    200.00000     50.00000     022 122 411                            0
11   2600.00000     50.00000     231 302 420                            0
12   3430.00000     60.00000     222 312                                0
13   2150.00000     50.00000     421 040 331                            0
14  12000.0000     120.00000     501 322 ... 412                        1

MILLER INDICES
 1   2  0  0                                                            0
 2   1  0  1                                                            0
 3   0  1  1                                                            0
 4   2  1  0                                                            0
 5   1  1  1                                                            0
 6   0  2  0                                                            0
 7   2  0  1                                                            0
 8   2  1  1                                                            0
 9   2  2  0                                                            0
10   1  2  1                                                            0
11   3  0  1                                                            0
12   1  1  2                                                            0
13   4  0  0                                                            0
14   2  0  2                                                            0
15   4  1  0                                                            0
16   0  2  2                                                            0
17   1  2  2                                                            0
18   4  1  1                                                            0
19   2  3  1                                                            0
20   3  0  2                                                            0
21   4  2  0                                                            0
22   2  2  2                                                            0
23   3  1  2                                                            0
24   4  2  1                                                            0
25   0  4  0                                                            0
26   3  3  1                                                            0
27   5  0  1                                                            0
28   3  2  2                                                            0
29   1  3  2                                                            0
30   4  0  2                                                            0
31   5  1  1                                                            0
32   2  4  0                                                            0
33   1  4  1                                                            0
34   1  0  3                                                            0
35   4  3  0                                                            0
36   4  1  2                                                            0
37   0  0  2                                                            0
38   2  2  1                                                            0
39   3  1  1                                                            0
40   1  0  2                                                            0
41   0  3  1                                                            0
42   2  3  0                                                            0
43   2  1  2                                                            0
44   3  2  1                                                            0
45   1  3  1                                                            0
46   4  0  1                                                            1
```

PARAMETERS

1	X FE	0.0	0.0010000	0
2	X S	0.1740000	0.0010000	0
3	X O1	0.1210000	0.0010000	0
4	X O2	0.3470000	0.0010000	0
5	X O3	0.1260000	0.0010000	0
6	Y FE	0.0	0.0010000	0
7	Y S	0.2500000	0.0010000	0
8	Y O1	0.2500000	0.0010000	0
9	Y O2	0.2500000	0.0010000	0
10	Y O3	0.0610000	0.0010000	0
11	Z FE	0.0	0.0010000	0
12	Z S	0.4880000	0.0010000	0
13	Z O1	0.7530000	0.0010000	0
14	Z O2	0.4630000	0.0010000	0
15	Z O3	0.3340000	0.0010000	0
16	WZ FE	0.5000000	0.0010000	0
17	WZ S	0.5000000	0.0010000	0
18	WZ O1	0.5000000	0.0010000	0
19	WZ O2	0.5000000	0.0010000	0
20	WZ O3	1.0000000	0.0010000	0
21	BZ FE	0.9510000	0.0010000	0
22	BZ S	0.2487000	0.0010000	0
23	BZ O	0.5803000	0.0010000	0
24	SCALE FACTOR	1.0000000	0.0010000	0
25	OVERALL TEMPERATURE FACTOR	0.3000000	0.0010000	1

CELL CONSTANTS

1.20300 8.71500 6.80400 4.79500 0.0 0.0 0.0

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	1222.72	5000.00	11	635.87	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	3.80	20.00	12	979.63	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	2178.05	8650.00	13	787.46	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	90.36	280.00	14	3197.17	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	12.43	50.00	15	2545.28	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	1245.02	4400.00	16	3575.45	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	622.32	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	383.25	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	159.29	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	91.73	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.7311

R - FACTOR WITHOUT ZEROS = 0.7311

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 1 OF 6 CYCLES.

REF?	PAM.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174000E+00	0.0	0.174000E+00	0.0
NO	3	0.121000E+00	0.0	0.121000E+00	0.0
NO	4	0.347000E+00	0.0	0.347000E+00	0.0
NO	5	0.126000E+00	0.0	0.126000E+00	0.0
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
NO	10	0.610000E-01	0.0	0.610000E-01	0.0
NO	11	0.0	0.0	0.0	0.0
NO	12	0.488000E+00	0.0	0.488000E+00	0.0
NO	13	0.753000E+00	0.0	0.753000E+00	0.0
NO	14	0.463000E+00	0.0	0.463000E+00	0.0
NO	15	0.334000E+00	0.0	0.334000E+00	0.0
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.951000E+00	0.0	0.951000E+00	0.0
NO	22	0.248700E+00	0.0	0.248700E+00	0.0
NO	23	0.580300E+00	0.0	0.580300E+00	0.0
YES	24	0.100000E+01	0.267667E+01	0.367667E+01	0.118777E+00
NO	25	0.300000E+00	0.0	0.300000E+00	0.0

ESTIMATED VPV/IN-MIN 0.3426E+02

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	4495.55	5000.00	11	2337.88	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	13.96	20.00	12	3601.78	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	8007.96	8650.00	13	2895.25	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	332.22	280.00	14	11754.91	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	45.68	50.00	15	9358.14	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	4577.54	4400.00	16	13145.74	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2288.08	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	1409.07	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	585.66	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	337.26	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0851

R - FACTOR WITHOUT ZEROS = 0.0851

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 2 OF 6 CYCLES.

REFS	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174000E+00	0.0	0.174000E+00	0.0
YES	3	0.121000E+00	-0.733158E-03	0.120267E+00	0.658508E-02
NO	4	0.347000E+00	0.0	0.347000E+00	0.0
NO	5	0.126000E+00	0.0	0.126000E+00	0.0
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
NO	10	0.610000E-01	0.0	0.610000E-01	0.0
NO	11	0.0	0.0	0.0	0.0
NO	12	0.488000E+00	0.0	0.488000E+00	0.0
YES	13	0.753000E+00	0.990911E-03	0.753991E+00	0.897032E-02
NO	14	0.463000E+00	0.0	0.463000E+00	0.0
NO	15	0.334000E+00	0.0	0.334000E+00	0.0
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.951000E+00	0.0	0.951000E+00	0.0
NO	22	0.248700E+00	0.0	0.248700E+00	0.0
NO	23	0.580300E+00	0.0	0.580300E+00	0.0
NO	24	0.367667E+01	0.0	0.367667E+01	0.0
NO	25	0.300000E+00	0.0	0.300000E+00	0.0

ESTIMATED VPV/IN-MI= 0.3696E+02

NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)
1	4539.43	5000.00	11	2346.67	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	13.92	20.00	12	3632.44	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	7972.46	8650.00	13	2897.15	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	333.01	280.00	14	11705.41	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	51.21	50.00	15	9366.33	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	4535.49	4400.00	16	13157.57	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2278.62	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	1411.77	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	582.15	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	330.47	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0849

R - FACTOR WITHOUT ZEROS = 0.0849

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 3 OF 6 CYCLES.

REFS	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174000E+00	0.0	0.174000E+00	0.0
NO	3	0.120267E+00	0.0	0.120267E+00	0.0
YES	4	0.347000E+00	0.549819E-02	0.352498E+00	0.435375E-02
NO	5	0.126000E+00	0.0	0.126000E+00	0.0
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
NO	10	0.610000E-01	0.0	0.610000E-01	0.0
NO	11	0.0	0.0	0.0	0.0
NO	12	0.488000E+00	0.0	0.488000E+00	0.0
NO	13	0.753991E+00	0.0	0.753991E+00	0.0
YES	14	0.463000E+00	0.474257E-02	0.467743E+00	0.132246E-01
NO	15	0.334000E+00	0.0	0.334000E+00	0.0
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.951000E+00	0.0	0.951000E+00	0.0
NO	22	0.248700E+00	0.0	0.248700E+00	0.0
NO	23	0.580300E+00	0.0	0.580300E+00	0.0
NO	24	0.367667E+01	0.0	0.367667E+01	0.0
NO	25	0.300000E+00	0.0	0.300000E+00	0.0

ESTIMATED VPV/IN-MI= 0.3267E+02

NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)
1	4851.25	5000.00	11	2498.73	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	24.26	20.00	12	3483.78	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	7783.21	8650.00	13	2845.20	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	361.95	280.00	14	11502.21	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	37.84	50.00	15	9258.04	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	4244.94	4400.00	16	13265.00	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2340.48	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	1522.05	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	605.39	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	358.07	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0801

R - FACTOR WITHOUT ZEROS = 0.0801

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 4 OF 6 CYCLES.

REFS	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174000E+00	0.0	0.174000E+00	0.0
NO	3	0.120267E+00	0.0	0.120267E+00	0.0
YES	4	0.352498E+00	-0.118721E-02	0.351311E+00	0.446315E-02
NO	5	0.126000E+00	0.0	0.126000E+00	0.0
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
NO	10	0.610000E-01	0.0	0.610000E-01	0.0
NO	11	0.0	0.0	0.0	0.0
NO	12	0.488000E+00	0.0	0.488000E+00	0.0
NO	13	0.753991E+00	0.0	0.753991E+00	0.0
YES	14	0.467743E+00	-0.298784E-02	0.464755E+00	0.124576E-01
NO	15	0.334000E+00	0.0	0.334000E+00	0.0
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.951000E+00	0.0	0.951000E+00	0.0
NO	22	0.248700E+00	0.0	0.248700E+00	0.0
NO	23	0.580300E+00	0.0	0.580300E+00	0.0
NO	24	0.367667E+01	0.0	0.367667E+01	0.0
NO	25	0.300000E+00	0.0	0.300000E+00	0.0

ESTIMATED VPV/(N-M)= 0.3322E+02

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	4774.83	5000.00	11	2477.00	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	17.41	20.00	12	3501.89	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	7807.00	8650.00	13	2855.56	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	340.81	280.00	14	11541.95	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	42.36	50.00	15	9375.78	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	4310.60	4400.00	16	13174.71	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2330.92	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	1491.50	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	602.07	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	340.50	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0794

R - FACTOR WITHOUT ZEROS = 0.0794

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 5 OF 6 CYCLES.

REFS	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174000E+00	0.0	0.174000E+00	0.0
NO	3	0.120267E+00	0.0	0.120267E+00	0.0
NO	4	0.351311E+00	0.0	0.351311E+00	0.0
YES	5	0.126000E+00	-0.162565E-02	0.124374E+00	0.268144E-02
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
YES	10	0.610000E-01	0.103795E-01	0.713795E-01	0.198155E-02
NO	11	0.0	0.0	0.0	0.0
NO	12	0.488000E+00	0.0	0.488000E+00	0.0
NO	13	0.753991E+00	0.0	0.753991E+00	0.0
NO	14	0.464755E+00	0.0	0.464755E+00	0.0
YES	15	0.334000E+00	-0.202521E-02	0.331975E+00	0.428818E-02
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.951000E+00	0.0	0.951000E+00	0.0
NO	22	0.248700E+00	0.0	0.248700E+00	0.0
NO	23	0.580300E+00	0.0	0.580300E+00	0.0
NO	24	0.367667E+01	0.0	0.367667E+01	0.0
NO	25	0.300000E+00	0.0	0.300000E+00	0.0

ESTIMATED VPV/(N-M)= 0.9592E+01

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	4916.32	5000.00	11	2828.83	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	0.08	20.00	12	3471.43	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	8358.29	8650.00	13	2235.31	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	356.93	280.00	14	12141.31	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	34.83	50.00	15	8447.14	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	4576.69	4400.00	16	13415.86	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2238.27	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	1537.28	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	589.09	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	272.84	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0413

R - FACTOR WITHOUT ZEROS = 0.0413

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FES04 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 6 OF 6 CYCLES.

REFS	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174000E+00	0.0	0.174000E+00	0.0
NO	3	0.120267E+00	0.0	0.120267E+00	0.0
NO	4	0.351311E+00	0.0	0.351311E+00	0.0
YES	5	0.124374E+00	0.852372E-03	0.125227E+00	0.272587E-02
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
YES	10	0.713795E-01	0.386542E-03	0.717660E-01	0.217429E-02
NO	11	0.0	0.0	0.0	0.0
NO	12	0.488000E+00	0.0	0.488000E+00	0.0
NO	13	0.753991E+00	0.0	0.753991E+00	0.0
NO	14	0.464755E+00	0.0	0.464755E+00	0.0
YES	15	0.331975E+00	-0.113147E-02	0.330843E+00	0.435151E-02
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.951000E+00	0.0	0.951000E+00	0.0
NO	22	0.248700E+00	0.0	0.248700E+00	0.0
NO	23	0.580300E+00	0.0	0.580300E+00	0.0
NO	24	0.367667E+01	0.0	0.367667E+01	0.0
NO	25	0.300000E+00	0.0	0.300000E+00	0.0

ESTIMATED VPV/(N-M)= 0.9913E+01

NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)	NO	I(CALC)	I(OBS)
1	4929.58	5000.00	11	2806.62	2600.00	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	0.32	20.00	12	3495.04	3430.00	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	8364.78	8650.00	13	2202.50	2150.00	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	365.93	280.00	14	12122.09	12000.00	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	39.08	50.00	15	8403.36	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	4557.63	4400.00	16	13485.89	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2244.28	2050.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	1534.18	1830.00	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	595.68	520.00	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	262.51	200.00	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0395

R - FACTOR WITHOUT ZEROS = 0.0395

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FES04 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

FUNCTIONS OF INTENSITY

NO.	DESCRIPTION	OBSERVED	CALCULATED	DELTA	DELTA/SIGMA
1	200 101	0.5000E+04	0.4930E+04	0.7042E+02	0.7042E+00
2	011	0.2000E+02	0.3204E+00	0.1968E+02	0.9840E+00
3	210 111 020	0.8650E+04	0.8365E+04	0.2852E+03	0.2852E+01
4	201	0.2800E+03	0.3659E+03	-0.8593E+02	-0.4298E+01
5	211	0.5000E+02	0.3908E+02	0.1092E+02	0.5458E+00
6	220 121	0.4400E+04	0.4558E+04	-0.1578E+03	-0.1578E+01
7	301	0.2050E+04	0.2244E+04	-0.1943E+03	-0.3886E+01
8	112 400	0.1830E+04	0.1534E+04	0.2958E+03	0.5916E+01
9	202 410	0.5200E+03	0.5957E+03	-0.7568E+02	-0.2523E+01
10	022 122 411	0.2000E+03	0.2625E+03	-0.6251E+02	-0.1250E+01
11	231 302 420	0.2600E+04	0.2807E+04	-0.2066E+03	-0.4132E+01
12	222 312	0.3430E+04	0.3495E+04	-0.6504E+02	-0.1084E+01
13	421 040 331	0.2150E+04	0.2203E+04	-0.5250E+02	-0.1050E+01
14	501 322 ... 412	0.1200E+05	0.1212E+05	-0.1221E+03	-0.1017E+01

WEIGHTED R FACTOR FOR FUNCTIONS IS 0.0573. CORRELATIONS IN OBSERVATIONS NOT INCLUDED.

DITTO BUT CORRELATIONS INCLUDED. R= 0.0573.

VPV= 0.1093E+03 (CORRELATIONS INCLUDED)

THE F VALUES ABOVE ARE CALCULATED FOR THE FOLLOWING PARAMETER VALUES

PARAMETER	DESCRIPTION	VALUE
1	X FE	0.0
2	X S	0.174000E+00
3	X 01	0.120267E+00
4	X 02	0.351311E+00
5	X 03	0.175227E+00
6	Y FE	0.0
7	Y S	0.250000E+00
8	Y 01	0.250000E+00
9	Y 02	0.250000E+00
10	Y 03	0.176600E 01
11	Z FE	0.0
12	Z S	0.488000E+00
13	Z 01	0.754991E+00
14	Z 02	0.464755E+00
15	Z 03	0.230E43E+00
16	WZ FE	0.500000E+00
17	WZ S	0.500000E+00
18	WZ 01	0.500000E+00
19	WZ 02	0.500000E+00
20	WZ 03	0.100000E+01
21	BZ FE	0.951000E+00
22	BZ S	0.248700E+00
23	BZ O	0.580300E+00
24	SCALE FACTOR	0.367667E+01
25	OVERALL TEMPERATURE FACTOR	0.300000E+00

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA
FESO4 STRUCTURE REFINEMENT BY NEUTRON DIFFRACTION

TRIGONOMETRIC FUNCTIONS	H	K	L	SINTHETA	SIN2THETA	THETA	2THETA
	2	0	0	0.13804	0.27343	7.93	15.87
	1	0	1	0.14318	0.28340	8.23	16.46
	0	1	1	0.15346	0.30329	8.83	17.66
	2	1	0	0.16392	0.32341	9.44	18.87
	1	1	1	0.16827	0.33174	9.69	19.38
	0	2	0	0.17681	0.34804	10.18	20.37
	2	0	1	0.18652	0.36650	10.75	21.50
	2	1	1	0.20641	0.40393	11.91	23.83
	2	2	0	0.22431	0.43719	12.96	25.93
	1	2	1	0.22751	0.44309	13.15	26.30
	3	0	1	0.24209	0.46978	14.01	28.02
	1	1	2	0.27481	0.52847	15.95	31.90
	4	0	0	0.27608	0.53069	16.03	32.05
	2	0	2	0.28635	0.54872	16.64	33.28
	4	1	0	0.28988	0.55487	16.85	33.70
	0	2	2	0.30693	0.58423	17.88	35.75
	1	2	2	0.31459	0.59724	18.34	36.68
	4	1	1	0.31586	0.59938	18.41	36.83
	2	3	1	0.32423	0.61344	18.92	37.84
	3	0	2	0.32529	0.61520	18.98	37.97
	4	2	0	0.32784	0.61944	19.14	38.28
	2	2	2	0.33654	0.63382	19.67	39.34
	3	1	2	0.33709	0.63473	19.70	39.40
	4	2	1	0.35102	0.65737	20.55	41.10
	0	4	0	0.35362	0.66154	20.71	41.42
	3	3	1	0.35909	0.67028	21.05	42.09
	5	0	1	0.36719	0.68308	21.54	43.09
	3	2	2	0.37024	0.68786	21.73	43.46
	1	3	2	0.37154	0.68989	21.81	43.62
	4	0	2	0.37204	0.69223	21.91	43.81
	5	1	1	0.37768	0.69941	22.19	44.38
	2	4	0	0.37960	0.70238	22.31	44.62
	1	4	1	0.38150	0.70530	22.43	44.86
	1	0	3	0.38261	0.70699	22.50	44.99
	4	3	0	0.38282	0.70732	22.51	45.02
	4	1	2	0.38338	0.70817	22.54	45.09
	0	0	2	0.25089	0.48572	14.53	29.06
	2	2	1	0.25700	0.49674	14.89	29.79
	3	1	1	0.25773	0.49804	14.94	29.87
	1	0	2	0.26021	0.50249	15.08	30.17
	0	3	1	0.29338	0.56094	17.06	34.12
	2	3	0	0.29898	0.57062	17.40	34.80
	2	1	2	0.29969	0.57183	17.44	34.88
	3	2	1	0.29978	0.57199	17.45	34.89
	1	3	1	0.30139	0.57475	17.54	35.08
	4	2	1	0.30324	0.57792	17.65	35.31

APPENDIX C EXAMPLE 2

FESO4 STRUCTURE REFINEMENT BY X-RAY DIFFRACTION

OPTIONS

0 11 0 5 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0

FORM FACTOR TABLES

24.000	23.710	22.890	21.660	20.150	18.510	16.870	15.310
13.840	11.470	9.710	8.740	7.600	6.990	6.510	6.120
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18.000	17.190	15.160	12.730	10.740	9.450	8.660	8.210
7.890	7.220	6.470	5.690	4.930	4.230	3.620	3.130
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8.000	7.796	7.250	6.482	5.634	4.814	4.093	3.492
3.010	2.338	1.944	1.714	1.566	1.462	1.374	1.296
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8.000	7.796	7.250	6.482	5.634	4.814	4.093	3.492
3.010	2.338	1.944	1.714	1.566	1.462	1.374	1.296
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8.000	7.796	7.250	6.482	5.634	4.814	4.093	3.492
3.010	2.338	1.944	1.714	1.566	1.462	1.374	1.296
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

OBSERVATIONS

1	15.40000	3.00000	200	0
2	43.50000	5.00000	101	0
3	45.00000	5.00000	210	0
4	100.00000	6.00000	111	0
5	84.00000	6.00000	220	0
6	68.50000	6.00000	121	0
7	65.00000	5.00000	301	0
8	24.00000	4.00000	002	0
9	20.00000	4.00000	221	0
10	10.00000	3.00000	102	0
11	1.00000	2.00000	400	0
12	6.70000	3.00000	202	0
13	3.30000	2.00000	410	0
14	7.00000	3.00000	031	0
15	33.00000	4.00000	321	0
16	2.00000	2.00000	131	0
17	1.00000	2.00000	022	0
18	23.00000	4.00000	420	0
19	50.00000	5.00000	312,222	0
20	20.50000	4.00000	040	0
21	3.50000	2.00000	501	0
22	11.00000	3.00000	402	0
23	9.00000	3.00000	511	0
24	12.50000	3.00000	141	0
25	14.30000	3.00000	103,412,043	1

MILLER INDICES

1	2	0	0	0
2	1	0	1	0
3	0	1	1	0
4	2	1	0	0
5	1	1	1	0
6	0	2	0	0
7	2	0	1	0
8	2	1	1	0
9	2	2	0	0
10	1	2	1	0
11	3	0	1	0
12	1	1	2	0
13	4	0	0	0
14	2	0	2	0

```
15  4  1  0                                                          0
16  0  2  2                                                          0
17  1  2  2                                                          0
18  4  1  1                                                          0
19  2  3  1                                                          0
20  3  0  2                                                          0
21  4  2  0                                                          0
22  2  2  2                                                          0
23  3  1  2                                                          0
24  4  2  1                                                          0
25  0  4  0                                                          0
26  3  3  1                                                          0
27  5  0  1                                                          0
28  3  2  2                                                          0
29  1  3  2                                                          0
30  4  0  2                                                          0
31  5  1  1                                                          0
32  2  4  0                                                          0
33  1  4  1                                                          0
34  1  0  3                                                          0
35  4  3  0                                                          0
36  4  1  2                                                          0
37  0  0  2                                                          0
38  2  2  1                                                          0
39  3  1  1                                                          0
40  1  0  2                                                          0
41  0  3  1                                                          0
42  2  3  0                                                          0
43  2  1  2                                                          0
44  3  2  1                                                          0
45  1  3  1                                                          0
46  4  0  1                                                          1

PARAMETERS
 1  X FE                              0.0          0.0010000       0
 2  X S                               0.1860000    0.0100000       0
 3  X O1                              0.1300000    0.0100000       0
 4  X O2                              0.3670000    0.0100000       0
 5  X O3                              0.1300000    0.0100000       0
 6  Y FE                              0.0          0.0100000       0
 7  Y S                               0.2500000    0.0100000       0
 8  Y O1                              0.2500000    0.0100000       0
 9  Y O2                              0.2500000    0.0100000       0
10  Y O3                              0.0720000    0.0100000       0
11  Z FE                              0.0          0.0100000       0
12  Z S                               0.4580000    0.0100000       0
13  Z O1                              0.7500000    0.0100000       0
14  Z O2                              0.4580000    0.0100000       0
15  Z O3                              0.3190000    0.0100000       0
16  WZ FE                             0.5000000    0.0010000       0
17  WZ S                              0.5000000    0.0010000       0
18  WZ O1                             0.5000000    0.0010000       0
19  WZ O2                             0.5000000    0.0010000       0
20  WZ O3                             1.0000000    0.0010000       0
21  SCALE FACTOR                      0.0100000    0.0010000       0
22  OVERALL TEMPERATURE FACTOR        0.2000000    0.0010000       1

CELL CONSTANTS
  1.54180   8.71800   6.81100   4.79800   0.0       0.0       0.0
```

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	567.86	15.40	11	3.31	1.00	21	133.51	3.50	31	0.0	0.0	41	0.0	0.0
2	2963.19	43.50	12	445.08	6.70	22	740.30	11.00	32	0.0	0.0	42	0.0	0.0
3	1773.69	45.00	13	237.05	3.30	23	189.24	9.00	33	0.0	0.0	43	0.0	0.0
4	4679.52	100.00	14	231.89	7.00	24	772.95	12.50	34	0.0	0.0	44	0.0	0.0
5	3333.04	84.00	15	1127.86	33.00	25	821.73	14.30	35	0.0	0.0	45	0.0	0.0
6	2766.76	68.50	16	364.40	2.00	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	2904.15	65.00	17	110.77	1.00	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	716.23	24.00	18	834.68	23.00	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	731.55	20.00	19	2081.37	50.00	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	488.10	10.00	20	878.51	20.50	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 43.4990

R - FACTOR WITHOUT ZEROS = 43.4990

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY X-RAY DIFFRACTION

LEAST SQUARES. CYCLE 1 OF 11 CYCLES.

REF?	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.186000E+00	0.0	0.186000E+00	0.0
NO	3	0.130000E+00	0.0	0.130000E+00	0.0
NO	4	0.367000E+00	0.0	0.367000E+00	0.0
NO	5	0.130000E+00	0.0	0.130000E+00	0.0
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
NO	10	0.720000E-01	0.0	0.720000E-01	0.0
NO	11	0.0	0.0	0.0	0.0
NO	12	0.458000E+00	0.0	0.458000E+00	0.0
NO	13	0.750000E+00	0.0	0.750000E+00	0.0
NO	14	0.458000E+00	0.0	0.458000E+00	0.0
NO	15	0.319000E+00	0.0	0.319000E+00	0.0
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
YES	21	0.100000E-01	-0.978299E-02	0.217013E-03	0.950068E-05
NO	22	0.200000E+00	0.0	0.200000E+00	0.0

ESTIMATED VPV/IN-MI= 0.2448E+01

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	12.32	15.40	11	0.07	1.00	21	2.90	3.50	31	0.0	0.0	41	0.0	0.0
2	64.31	43.50	12	9.66	6.70	22	16.07	11.00	32	0.0	0.0	42	0.0	0.0
3	38.49	45.00	13	5.14	3.30	23	4.11	9.00	33	0.0	0.0	43	0.0	0.0
4	101.55	100.00	14	5.03	7.00	24	16.77	12.50	34	0.0	0.0	44	0.0	0.0
5	72.33	84.00	15	24.48	33.00	25	17.83	14.30	35	0.0	0.0	45	0.0	0.0
6	60.04	68.50	16	7.91	2.00	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	63.02	65.00	17	2.40	1.00	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	15.54	24.00	18	19.42	23.00	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	15.88	20.00	19	45.17	50.00	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	10.59	10.00	20	19.06	20.50	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.1767

R - FACTOR WITHOUT ZEROS = 0.1767

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY X-RAY DIFFRACTION

LEAST SQUARES. CYCLE 10 OF 11 CYCLES.

REF$	PAM.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.174068E+00	0.0	0.174068E+00	0.0
NO	3	0.131576E+00	0.0	0.131576E+00	0.0
NO	4	0.345786E+00	0.0	0.345786E+00	0.0
NO	5	0.125026E+00	0.0	0.125026E+00	0.0
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
NO	10	0.682637E-01	0.0	0.682637E-01	0.0
NO	11	0.0	0.0	0.0	0.0
NO	12	0.493716E+00	0.0	0.493716E+00	0.0
NO	13	0.780243E+00	0.0	0.780243E+00	0.0
NO	14	0.450851E+00	0.0	0.450851E+00	0.0
NO	15	0.347300E+00	0.0	0.347300E+00	0.0
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
YES	21	0.217016E-03	0.590682E-05	0.222923E-03	0.502378E-05
NO	22	0.200000E+00	0.0	0.200000E+00	0.0

ESTIMATED VPV/IN-M:= 0.6703E+00

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	14.12	15.40	11	0.33	1.00	21	7.06	3.50	31	0.0	0.0	41	0.0	0.0
2	47.54	43.50	12	8.29	6.70	22	10.69	11.00	32	0.0	0.0	42	0.0	0.0
3	47.05	45.00	13	2.39	3.30	23	7.55	9.00	33	0.0	0.0	43	0.0	0.0
4	104.22	100.00	14	6.08	7.00	24	15.80	12.50	34	0.0	0.0	44	0.0	0.0
5	78.38	84.00	15	29.29	33.00	25	13.19	14.30	35	0.0	0.0	45	0.0	0.0
6	60.17	68.50	16	6.07	2.00	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	65.58	65.00	17	2.70	1.00	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	24.22	24.00	18	22.25	23.00	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	20.71	20.00	19	46.51	50.00	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	9.45	10.00	20	20.53	20.50	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0819

R - FACTOR WITHOUT ZEROS = 0.0819

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY X-RAY DIFFRACTION

LEAST SQUARES. CYCLE 11 OF 11 CYCLES.

REF$	PAM.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
YES	2	0.174068E+00	-0.425163E-04	0.174025E+00	0.393366E-02
YES	3	0.131576E+00	0.240518E-02	0.133981E+00	0.962636E-02
YES	4	0.345786E+00	0.172118E-02	0.347508E+00	0.756417E-02
YES	5	0.125026E+00	-0.373283E-04	0.124988E+00	0.482838E-02
NO	6	0.0	0.0	0.0	0.0
NO	7	0.250000E+00	0.0	0.250000E+00	0.0
NO	8	0.250000E+00	0.0	0.250000E+00	0.0
NO	9	0.250000E+00	0.0	0.250000E+00	0.0
YES	10	0.682637E-01	-0.898803E-03	0.673649E-01	0.743131E-02
NO	11	0.0	0.0	0.0	0.0
YES	12	0.493716E+00	-0.161363E-02	0.492102E+00	0.153117E-01
YES	13	0.780243E+00	0.223576E-02	0.782479E+00	0.137436E-01
YES	14	0.450851E+00	-0.101442E-02	0.449836E+00	0.228965E-01
YES	15	0.347300E+00	0.315636E-03	0.347615E+00	0.912016E-02
NO	16	0.500000E+00	0.0	0.500000E+00	0.0
NO	17	0.500000E+00	0.0	0.500000E+00	0.0
NO	18	0.500000E+00	0.0	0.500000E+00	0.0
NO	19	0.500000E+00	0.0	0.500000E+00	0.0
NO	20	0.100000E+01	0.0	0.100000E+01	0.0
NO	21	0.222923E-03	0.0	0.222923E-03	0.0
NO	22	0.200000E+00	0.0	0.200000E+00	0.0

ESTIMATED VPV/IN-M:= 0.9882E+00

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	13.98	15.40	11	0.35	1.00	21	6.90	3.50	31	0.0	0.0	41	0.0	0.0
2	48.07	43.50	12	8.62	6.70	22	10.47	11.00	32	0.0	0.0	42	0.0	0.0
3	46.31	45.00	13	2.73	3.30	23	7.45	9.00	33	0.0	0.0	43	0.0	0.0
4	102.41	100.00	14	5.87	7.00	24	15.77	12.50	34	0.0	0.0	44	0.0	0.0
5	78.68	84.00	15	29.55	33.00	25	13.19	14.30	35	0.0	0.0	45	0.0	0.0
6	59.32	68.50	16	5.93	2.00	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	65.44	65.00	17	2.70	1.00	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	24.29	24.00	18	22.27	23.00	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	20.69	20.00	19	45.77	50.00	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	10.01	10.00	20	20.77	20.50	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0803

R - FACTOR WITHOUT ZEROS = 0.0803

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
FESO4 STRUCTURE REFINEMENT BY X-RAY DIFFRACTION

FUNCTIONS OF INTENSITY

NO.	DESCRIPTION	OBSERVED	CALCULATED	DELTA	DELTA/SIGMA
1	200	0.1540E+02	0.1398E+02	0.1420E+01	0.4732E+00
2	101	0.4350E+02	0.4807E+02	-0.4569E+01	-0.9138E+00
3	210	0.4500E+02	0.4631E+02	-0.1307E+01	-0.2614E+00
4	111	0.1000E+03	0.1024E+03	-0.2409E+01	-0.4016E+00
5	220	0.8400E+02	0.7868E+02	0.5319E+01	0.8865E+00
6	121	0.6850E+02	0.5932E+02	0.9183E+01	0.1530E+01
7	301	0.6500E+02	0.6544E+02	-0.4385E+00	-0.8770E-01
8	002	0.2400E+02	0.2429E+02	-0.2917E+00	-0.7293E-01
9	221	0.2000E+02	0.2069E+02	-0.6870E+00	-0.1717E+00
10	102	0.1000E+02	0.1001E+02	-0.5894E-02	-0.1965E-02
11	400	0.1000E+01	0.3501E+00	0.6499E+00	0.3249E+00
12	202	0.6700E+01	0.8623E+01	-0.1923E+01	-0.6410E+00
13	410	0.3300E+01	0.2730E+01	0.5702E+00	0.2851E+00
14	031	0.7000E+01	0.5869E+01	0.1131E+01	0.3770E+00
15	321	0.3300E+02	0.2955E+02	0.3449E+01	0.8622E+00
16	131	0.2000E+01	0.5933E+01	-0.3933E+01	-0.1967E+01
17	022	0.1000E+01	0.2696E+01	-0.1696E+01	-0.8481E+00
18	420	0.2300E+02	0.2227E+02	0.7334E+00	0.1834E+00
19	312.222	0.5000E+02	0.4577E+02	0.4233E+01	0.8465E+00
20	040	0.2050E+02	0.2077E+02	-0.2664E+00	-0.6660E-01
21	501	0.3500E+01	0.6903E+01	-0.3403E+01	-0.1701E+01
22	402	0.1100E+02	0.1047E+02	0.5341E+00	0.1780E+00
23	511	0.9000E+01	0.7448E+01	0.1552E+01	0.5175E+00
24	141	0.1250E+02	0.1577E+02	-0.3273E+01	-0.1091E+01
25	103.412.043	0.1430E+02	0.1319E+02	0.1111E+01	0.3703E+00

WEIGHTED R FACTOR FOR FUNCTIONS IS 0.1088. CORRELATIONS IN OBSERVATIONS NOT INCLUDED.

DITTO BUT CORRELATIONS INCLUDED. R= 0.1088.

VPV= 0.1581E+02 (CORRELATIONS INCLUDED)

THE F VALUES ABOVE ARE CALCULATED FOR THE FOLLOWING PARAMETER VALUES

PARAMETER	DESCRIPTION	VALUE
1	X FE	0.0
2	X S	0.174025E+00
3	X O1	0.133981E+00
4	X O2	0.347508E+00
5	X O3	0.124988E+00
6	Y FE	0.0
7	Y S	0.250000E+00
8	Y O1	0.250000E+00
9	Y O2	0.250000E+00
10	Y O3	0.673649E-01
11	Z FE	0.0
12	Z S	0.492102E+00
13	Z O1	0.782479E+00
14	Z O2	0.449836E+00
15	Z O3	0.347615E+00
16	WZ FE	0.500000E+00
17	WZ S	0.500000E+00
18	WZ O1	0.500000E+00
19	WZ O2	0.500000E+00
20	WZ O3	0.100000E+01
21	SCALE FACTOR	0.222923E-03
22	OVERALL TEMPERATURE FACTOR	0.200000E+00

APPENDIX C EXAMPLE 3

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DYAS04 MAGNETIC STRUCTURE ANALYSIS BY NEUTRON DIFFRACTION


OPTIONS
 0  2  1  1  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0  0

FORM FACTOR TABLES
   1.000     0.980     0.950     0.890     0.830     0.760     0.690     0.620
   0.560     0.490     0.430     0.380     0.290     0.250     0.210     0.180
   0.0       0.0       0.0       0.0       0.0       0.0       0.0       0.0
   0.0       0.0       0.0       0.0       0.0       0.0       0.0       0.0

OBSERVATIONS
 1   3730.00000     60.00000     100                                        0
 2   1782.00000     40.00000     001                                        0
 3   1800.00000     40.00000     111                                        0
 4   1205.00000     35.00000     120+201+021                                0
 5    978.00000     30.00000     021                                        0
 6   1178.00000     35.00000     212                                        0
 7    880.00000     30.00000     311+131+003                                1

MILLER INDICES
 1   1  0  0                                                                0
 2   0  0  1                                                                0
 3   1  1  1                                                                0
 4   1  2  0                                                                0
 5   2  0  1                                                                0
 6   0  2  1                                                                0
 7   0  1  2                                                                0
 8   2  1  2                                                                0
 9   3  1  1                                                                0
10   1  3  1                                                                0
11   0  0  3                                                                0
12   3  2  0                                                                0
13   1  1  3                                                                0
14   0  3  2                                                                0
15   3  0  0                                                                0
16   2  2  1                                                                0
17   2  0  3                                                                0
18   0  2  3                                                                1

PARAMETERS
 1   X DY1                          0 0           0.0010000                0
 2   X DY2                          0.0           0.0010000                0
 3   X DY3                          0.5000000     0.0010000                0
 4   X DY4                          0.5000000     0.0010000                0
 5   Y DY1                          0.0           0.0010000                0
 6   Y DY2                          0.5000000     0.0010000                0
 7   Y DY3                          0.5000000     0.0010000                0
 8   Y DY4                          0.0           0.0010000                0
 9   Z DY1                          0.0           0.0010000                0
10   Z DY2                          0.2500000     0.0010000                0
11   Z DY3                          0.5000000     0.0010000                0
12   Z DY4                          0.7500000     0.0010000                0
13   S DY1                          1.0000000     0.0010000                0
14   S DY2                          1.0000000     0.0010000                0
15   S DY3                         -1.0000000     0.0010000                0
16   S DY4                         -1.0000000     0.0010000                0
17   SCALE FACTOR                   0.2670000     0.0100000                0
18   TEMPERATURE FACTOR             0.2000000     0.0010000                0
19   MAGNETIC MOMENT                7.0000000     0.0010000                0
20   PHIA                          45.0000000     0.0100000                0
21   PHIC                          90.0000000     0.0100000                1

CELL CONSTANTS
  0.97000    7.06900    7.06900    6.33400    0.0        0.0        0.0
```

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	1450.69	3730.00	11	0.0	0.0	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	1200.01	1782.00	12	0.0	0.0	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	1158.51	1800.00	13	0.0	0.0	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	1098.65	1205.00	14	0.0	0.0	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	739.30	978.00	15	0.0	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	613.21	1178.00	16	0.0	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	475.37	880.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	570.45	0.0	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	374.40	0.0	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	194.27	0.0	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.4170

R - FACTOR WITHOUT ZEROS = 0.4170

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
GYASD4 MAGNETIC STRUCTURE ANALYSIS BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 1 OF 2 CYCLES.

REFS	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.0	0.0	0.0	0.0
NO	3	0.500000E+00	0.0	0.500000E+00	0.0
NO	4	0.500000E+00	0.0	0.500000E+00	0.0
NO	5	0.0	0.0	0.0	0.0
NO	6	0.500000E+00	0.0	0.500000E+00	0.0
NO	7	0.500000E+00	0.0	0.500000E+00	0.0
NO	8	0.0	0.0	0.0	0.0
NO	9	0.0	0.0	0.0	0.0
NO	10	0.250000E+00	0.0	0.250000E+00	0.0
NO	11	0.500000E+00	0.0	0.500000E+00	0.0
NO	12	0.750000E+00	0.0	0.750000E+00	0.0
NO	13	0.100000E+01	0.0	0.100000E+01	0.0
NO	14	0.100000E+01	0.0	0.100000E+01	0.0
NO	15	-0.100000E+01	0.0	-0.100000E+01	0.0
NO	16	-0.100000E+01	0.0	-0.100000E+01	0.0
NO	17	0.267000E+00	0.0	0.267000E+00	0.0
NO	18	0.200000E+00	0.0	0.200000E+00	0.0
YES	19	0.700000E+01	0.174547E+01	0.874547E+01	0.189287E+00
YES	20	0.450000E+02	0.323676E+02	0.773676E+02	0.395048E+01
NO	21	0.900000E+02	0.0	0.900000E+02	0.0

ESTIMATED VPV/IN-MI= 0.1256E+02

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	4419.61	3730.00	11	0.0	0.0	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	1873.07	1782.00	12	0.0	0.0	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	1808.29	1800.00	13	0.0	0.0	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	1286.12	1205.00	14	0.0	0.0	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	1058.09	978.00	15	0.0	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	1133.97	1178.00	16	0.0	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	742.00	880.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	851.39	0.0	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	754.00	0.0	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	303.23	0.0	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0980

R - FACTOR WITHOUT ZEROS = 0.0980

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
DYA504 MAGNETIC STRUCTURE ANALYSIS BY NEUTRON DIFFRACTION

LEAST SQUARES. CYCLE 2 OF 2 CYCLES.

REF?	PAR.	OLD VALUE	CHANGE	NEW VALUE	SIGMA
NO	1	0.0	0.0	0.0	0.0
NO	2	0.0	0.0	0.0	0.0
NO	3	0.500000E+00	0.0	0.500000E+00	0.0
NO	4	0.500000E+00	0.0	0.500000E+00	0.0
NO	5	0.0	0.0	0.0	0.0
NO	6	0.500000E+00	0.0	0.500000E+00	0.0
NO	7	0.500000E+00	0.0	0.500000E+00	0.0
NO	8	0.0	0.0	0.0	0.0
NO	9	0.0	0.0	0.0	0.0
NO	10	0.250000E+00	0.0	0.250000E+00	0.0
NO	11	0.500000E+00	0.0	0.500000E+00	0.0
NO	12	0.750000E+00	0.0	0.750000E+00	0.0
NO	13	0.100000E+01	0.0	0.100000E+01	0.0
NO	14	0.100000E+01	0.0	0.100000E+01	0.0
NO	15	-0.100000E+01	0.0	-0.100000E+01	0.0
NO	16	-0.100000E+01	0.0	-0.100000E+01	0.0
NO	17	0.267000E+00	0.0	0.267000E+00	0.0
NO	18	0.200000E+00	0.0	0.200000E+00	0.0
YES	19	0.874547E+01	-0.176720E+00	0.855875E+01	0.151127E+00
YES	20	0.773676E+02	-0.104896E+02	0.668780E+02	0.590372E+01
NO	21	0.900000E+02	0.0	0.900000E+02	0.0

ESTIMATED VPV/(N-M)= 0.1257E+02

CORRELATION MATRIX

	19	20
19	1.00	-0.50
20	-0.50	1.00

MATRIX OF SECOND MOMENTS

	19	20
19	0.2284E-01	-0.4424E+00
20	-0.4424E+00	0.3485E+02

NORMALIZED MATRIX OF NORMAL EQUATIONS FOR USE IN HYPOTHESIS TESTS

	19	20
19	0.5806E+02	0.7370E+00
20	0.7370E+00	0.3805E-01

NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)	NO	I(CALC)	I (OBS)
1	3731.41	3730.00	11	0.0	0.0	21	0.0	0.0	31	0.0	0.0	41	0.0	0.0
2	1798.13	1782.00	12	0.0	0.0	22	0.0	0.0	32	0.0	0.0	42	0.0	0.0
3	1735.95	1800.00	13	0.0	0.0	23	0.0	0.0	33	0.0	0.0	43	0.0	0.0
4	1336.40	1205.00	14	0.0	0.0	24	0.0	0.0	34	0.0	0.0	44	0.0	0.0
5	1038.51	978.00	15	0.0	0.0	25	0.0	0.0	35	0.0	0.0	45	0.0	0.0
6	1046.65	1178.00	16	0.0	0.0	26	0.0	0.0	36	0.0	0.0	46	0.0	0.0
7	712.32	880.00	17	0.0	0.0	27	0.0	0.0	37	0.0	0.0	47	0.0	0.0
8	826.58	0.0	18	0.0	0.0	28	0.0	0.0	38	0.0	0.0	48	0.0	0.0
9	683.59	0.0	19	0.0	0.0	29	0.0	0.0	39	0.0	0.0	49	0.0	0.0
10	291.10	0.0	20	0.0	0.0	30	0.0	0.0	40	0.0	0.0	50	0.0	0.0

R - FACTOR INCLUDING ZEROS = 0.0496

R - FACTOR WITHOUT ZEROS = 0.0496

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
DYASD4 MAGNETIC STRUCTURE ANALYSIS BY NEUTRON DIFFRACTION

FUNCTIONS OF INTENSITY

NO.	DESCRIPTION	OBSERVED	CALCULATED	DELTA	DELTA/SIGMA
1	100	0.3730E+04	0.3731E+04	-0.1414E+01	-0.2357E-01
2	001	0.1782E+04	0.1798E+04	-0.1613E+02	-0.4034E+00
3	111	0.1800E+04	0.1736E+04	0.6405E+02	0.1601E+01
4	120+201+021	0.1205E+04	0.1336E+04	-0.1314E+03	-0.3754E+01
5	021	0.9780E+03	0.1039E+04	-0.6051E+02	-0.2017E+..
6	212	0.1178E+04	0.1047E+04	0.1314E+03	0.3753E+01
7	311+131+003	0.8800E+03	0.7123E+03	0.1677E+03	0.5589E+01

WEIGHTED R FACTOR FOR FUNCTIONS IS 0.0739. CORRELATIONS IN OBSERVATIONS NOT INCLUDED.

DITTO BUT CORRELATIONS INCLUDED. R= 0.0739.

VPV= 0.6621E+02 (CORRELATIONS INCLUDED)

THE F VALUES ABOVE ARE CALCULATED FOR THE FOLLOWING PARAMETER VALUES

PARAMETER	DESCRIPTION	VALUE
1	X DY1	0.0
2	X DY2	0.0
3	X DY3	0.500000E+00
4	X DY4	0.500000E+00
5	Y DY1	0.0
6	Y DY2	0.500000E+00
7	Y DY3	0.500000E+00
8	Y DY4	0.0
9	Z DY1	0.0
10	Z DY2	0.250000E+00
11	Z DY3	0.500000E+00
12	Z DY4	0.750000E+00
13	S DY1	0.100000E+01
14	S DY2	0.100000E+01
15	S DY3	-0.100000E+01
16	S DY4	-0.100000E+01
17	SCALE FACTOR	0.267000E+00
18	TEMPERATURE FACTOR	0.200000E+00
19	MAGNETIC MOMENT	0.856875E+01
20	PHIA	0.668780E+02
21	PHIC	0.900000E+02

STRUCTURE FACTOR AND LEAST SQUARES PROGRAM FOR OVERLAPPING POWDER DATA.
DYASD4 MAGNETIC STRUCTURE ANALYSIS BY NEUTRON DIFFRACTION

TRIGONOMETRIC FUNCTIONS	H	K	L	SINTHETA	SIN2THETA	THETA	2THETA
	1	0	0	0.06861	0.13690	3.93	7.87
	0	0	1	0.07657	0.15269	4.39	8.78
	1	1	1	0.12360	0.24531	7.10	14.20
	1	2	0	0.15342	0.30320	8.83	17.65
	2	0	1	0.15714	0.31037	9.04	18.08
	0	2	1	0.15714	0.31037	9.04	18.08
	0	1	2	0.16781	0.33086	9.66	19.32
	2	1	2	0.21677	0.42323	12.52	25.04
	3	1	1	0.23008	0.44781	13.30	26.61
	1	3	1	0.23008	0.44781	13.30	26.61
	0	0	3	0.22971	0.44714	13.28	26.56
	3	2	0	0.24737	0.47937	14.32	28.65
	1	1	3	0.24936	0.48297	14.44	28.88
	0	3	2	0.25655	0.49593	14.87	29.73
	3	0	0	0.20583	0.40284	11.88	23.76
	2	2	1	0.20862	0.40805	12.04	24.08
	2	0	3	0.26758	0.51564	15.52	31.04
	0	2	3	0.26758	0.51564	15.52	31.04