NBS TECHNICAL NOTE 600

Corrections and Calculations on an X-ray Diffraction Line Profile: A Computer Program

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NBS TECHNICAL NOTE 600

ISSUED JUNE 1971

Nat. Bur. Stand. (U.S.), Tech. Note 600, 34 pages (June 1971)
CODEN: NBTNA

Corrections and Calculations on an X-ray Diffraction Line Profile: A Computer Program

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For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402
(Order by SD Catalog No. C13.46:600), Price 40 cents

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Corrections and Calculations on an X-ray Diffraction Line Profile: A Computer Program

Raymond E. Schramm

This computer program was written to perform corrections and make calculations on an x-ray diffraction profile before Fourier analysis. The corrections are for background and for variations of intensity with the Bragg angle. Also calculated are the separation of the $K\alpha_1$ - $K\alpha_2$ doublet and the centroid and position of peak maximum with their standard deviations. There is also an option to smooth the profile.

Key Words: Computer programs, Fourier analysis, nickel steels, x-ray analysis, x-ray diffraction, statistics.

1. Introduction

Annealed and heavily strained x-ray diffraction line profiles (fig. 1) can be compared to provide information on parameters such as particle size, dislocation density, and stacking fault energy. One scheme to do this is Fourier analysis. Gazzara, Stiglich, Meyer, and Hansen[1] wrote a computer program "UNFOLD" to perform the mathematical operations; several other programs are also available.[2,3] In working with a series of iron-nickel alloys, it became necessary to make corrections and calculations on the x-ray data in addition to those available in the program of Gazzara, et al. The program "PREP" was written to unify the preparation for programs such as "UNFOLD" by making all of the following calculations.

Within subroutines corrections are made for:

1. Background intensity.
2. Variation of intensity with the Bragg angle due to geometrical factors.

Subroutines are also used to determine:

1. Rachinger separation[4] of the $K\alpha_1$ - $K\alpha_2$ doublet.
2. Centroids of the $K\alpha$ and $K\alpha_1$ profiles with estimated standard deviations.
3. Peak maxima of the $K\alpha$ and $K\alpha_1$ profiles with estimated standard deviations.

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[1] Figures in brackets indicate the literature references at the end of this paper.

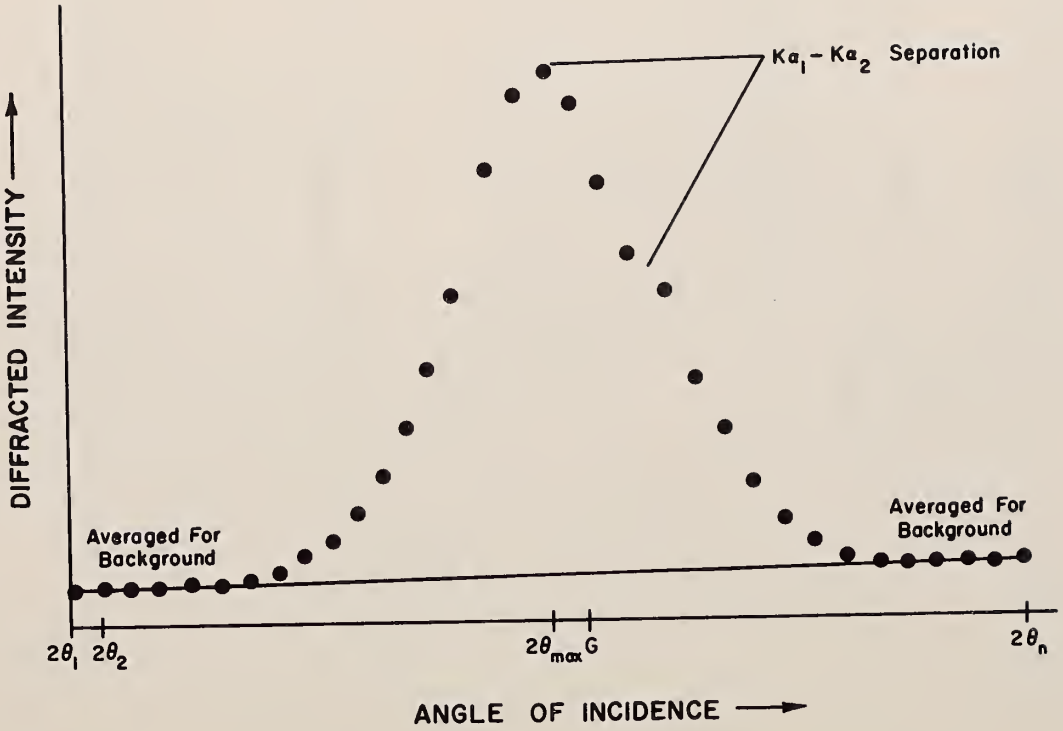

Fig.1. X-ray diffraction line profile.

Table 1. List of Symbols Used.

Symbol	Page of First Appearance	Meaning
a	4	Background intensity on low angle side of diffraction lines.
b	4	Background intensity at an arbitrary point.
c	7	Composite angular correction factor.
d	8	Angular separation of $K\alpha_1$ - $K\alpha_2$ doublet.
f	5	Atomic scattering factor.
h	7	Cylindrical specimen height.
m	7	Specimen density.
n	4	Number of experimental points or pairs of $2\theta_i$ and $I(2\theta_i)$.
r	5	Cylindrical specimen radius.
x	7	Mass fraction of Nickel
z	4	Background intensity on high angle side of diffraction line.
A	5	Sample absorption factor.
B, C, D,	13	Parameters of parabolic fit to peak.
G	2	Profile center of gravity.
I, I_1, I_2	4	Reflected intensity (counts/unit time) due to the radiations $K\alpha$, $K\alpha_1$, and $K\alpha_2$ respectively.
$K\alpha$, $K\alpha_1$, $K\alpha_2$	1	Characteristic radiations of the X-ray tube target.
LP	5	Lorentz-polarization factor.
α	5	Bragg angle of crystal monochromator.
$\delta 2\theta$	14	Increment in 2θ between individual measurements.
$\Delta 2\theta$	10	Angular span between first and last measurements.
θ	1	Specimen Bragg angle of reflection; 2θ is twice this angle. $2\theta_{max}$ is the position of the peak maximum.
λ, λ_1, λ_2	8	Wavelengths of $K\alpha$, $K\alpha_1$, and $K\alpha_2$ radiations respectively.
μ	5	Linear X-ray absorption coefficients.
ρ	7	Specimen mass density
ρ_s	7	Mass density of powder sample.
σ	11	Standard deviation of center of gravity and peak maximum.

It has been found advantageous to smooth the profiles of cold-worked samples before Fourier analysis so an option to do this has been provided. It is also possible to have the corrected profile data punched in a format ready to enter "UNFOLD".

Angular positions are entered in values of 2 θ (twice the Bragg angle). The diffraction intensities must be in the fixed-time mode (intensity count at each angular increment determined for a fixed length of time) and measured at equal increments of 2 θ across the entire profile. Several constants are written into the program assuming Debye-Scherrer geometry, the use of a monochromator with Co K α radiation, and iron-nickel or silver samples. The changes necessary when using other geometries, radiations, or samples are listed in the Appendix.

This program has been written in FORTRAN language and executed on a CDC 3800* computer.

2. Background Calculation, "BACK"

The background intensity is the measured radiation that is scattered into the detector by means other than primary Bragg reflection of the Kα component by the specimen. It generally arises from a number of sources: fluorescent radiation from the specimen, diffraction of the continuous spectrum, incoherent scattering from the specimen, and diffraction or scatter from something other than the specimen itself (e.g., collimator, air, etc.).

This calculation is made by arbitrarily averaging the intensities of the first five points and the last five points and then drawing a straight line through these two averaged points.

Line Profile

Angular Position (2 θ)	Intensity (counts/unit time)
$2\theta_1$	$I(2\theta_1)$
.	.
.	.
.	.
$2\theta_n$	$I(2\theta_n)$

*The use of trade names in this paper in no way implies endorsement or approval by NBS and is included only to define the procedure.

Set:

$$a = \frac{1}{5} \sum_{i=1}^{5} I(2\theta_i) ,$$

$$z = \frac{1}{5} \sum_{i=0}^{4} I(2\theta_{n-i}) .$$

Then

$$b_i = \left(\frac{z - a}{2\theta_n - 2\theta_1} \right) 2\theta_i + \frac{a(2\theta_n) - z(2\theta_1)}{2\theta_n - 2\theta_1} ,$$

where b_i is the background intensity at $2\theta_i$.

Background subtraction is an available option in "UNFOLD".

3. Angular Correction, "ANG"

There are several factors affecting the diffracted intensity which are functions of the Bragg angles. This angular correction is divided into three parts.

3.1. Lorentz-polarization Factor (LP)

When a monochromator is used [5],

$$LP = \frac{1 + \cos^2 2\alpha \cos^2 2\theta}{\sin^2 \theta \cos \theta} ,$$

where α is the Bragg angle of the crystal in the monochromator and θ is the Bragg angle of the specimen.

3.2. Atomic Scattering Factor (f)

The atomic scattering factor (f) is a function of $\frac{\sin\theta}{\lambda}$, λ being the wavelength of the radiation being used. Tables of this factor for various values of $\frac{\sin\theta}{\lambda}$ and for various atomic models are available [6]. The values used are given in table 2 in a normalized form (from self-consistent wave functions) for iron, nickel, and silver.

To determine f at intermediate values of $\frac{\sin\theta}{\lambda}$ a power series polynomial was fitted to the values in table 2. For a better fit $\frac{\sin\theta}{\lambda}$ was divided into three ranges. The values for this fit are given in table 3.

Table 2. - Normalized atomic scattering factors, f

$\frac{\sin\theta}{\lambda}$	Fe	Ni	Ag
0.00	1.000	1.000	1.000
0.05	0.9731	0.9768	0.9817
0.10	0.9108	0.9214	0.9336
0.15	0.8404	0.8568	0.8698
0.20	0.7727	0.7925	0.8017
0.25	0.7077	0.7300	0.7362
0.30	0.6450	0.6689	0.6762
0.35			0.6234
0.40	0.5323	0.5557	0.5781
0.50	0.4412	0.4611	0.5066
0.60	0.3735	0.3875	0.4547
0.70	0.3258	0.3332	0.4151

Table 3. - Power series coefficients for the atomic scattering factors

$$f = a + b\left(\frac{\sin\theta}{\lambda}\right) + c\left(\frac{\sin\theta}{\lambda}\right)^2 + d\left(\frac{\sin\theta}{\lambda}\right)^3$$

Coefficient	Fe	Ni	Ag	Range of $\frac{\sin\theta}{\lambda}$
a	+1.000	+1.000	+1.000	$0 \le \frac{\sin\theta}{\lambda} \le 0.12$
b	-0.184	-0.142	-0.068	
c	-7.080	-6.644	-5.960	
a	+1.0637002	+1.0645004	+1.0685171	$0.12 < \frac{\sin\theta}{\lambda} \le 0.35$
b	-1.6056701	-1.4746725	-1.2033810	
c	+0.86001593	+0.68002691	-1.2685714	
d	-0.53335684	-0.53337307	+3.0666667	
a	+1.1987014	+1.1781009	+1.2061000	$\frac{\sin\theta}{\lambda} > 0.35$
b	-2.3833411	-2.1006716	-2.4973333	
c	+2.0200144	+4.4750092	+2.8050000	
d	-0.56667538	-0.28333892	-1.2166667	

In the case of the iron-nickel alloys it was arbitrarily decided to use the atomic scattering factor for Fe for all bcc alloys and the atomic scattering factor for Ni for all fcc alloys. This gives a reasonable approximation to the electron density distribution in the alloy.

3.3. Sample Absorption (A)

For a flat specimen in a focusing geometry the sample absorption factor is independent of 2θ[7]. However, for a Debye-Scherrer geometry[8,9] a cylindrical sample is used and Bradley[10] gives the absorption factor:

$$A = \frac{1}{\pi\mu r}\left\{1 - \frac{\ell n(\sec\theta + \tan\theta)}{\sec\theta\tan\theta}\right\} + \frac{\sin 2\theta}{2\pi\mu^2 r^2} + \frac{1}{\pi\mu^3 r^3}\left[-\frac{1}{4} + \frac{3}{4}\cos^2\theta\left\{\frac{1}{2} + \frac{1}{2}\frac{\ell n(\sec\theta + \tan\theta)}{\sec\theta\tan\theta}\right\}\right],$$

for $\mu r > 10$, where μ is the linear absorption coefficient and r is the radius of the cylindrical specimen.

Using trigonometric identities this factor can be restated:

$$A = \frac{1}{\pi\mu r}\left\{1 - \frac{\cos^2\theta\,\ell n\left(\frac{1+\sin\theta}{\cos\theta}\right)}{\sin\theta}\right\} + \frac{\sin 2\theta}{2\pi(\mu r)^2} + \frac{1}{\pi(\mu r)^3}\left[-\frac{1}{4} + \frac{3}{8}\cos^2\theta\left\{1 + \frac{\cos^2\theta\,\ell n\left(\frac{1+\sin\theta}{\cos\theta}\right)}{\sin\theta}\right\}\right].$$

Bradley notes that for a diluted specimen (as in this case, we have used a powder mixed with cement):

$$\mu r = \frac{\mu}{\rho}\frac{m}{\pi r h},$$

where $\frac{\mu}{\rho}$ is the mass absorption coefficient, m is the mass of the powder in a specimen of height h and radius r. For a powder with three components (as Fe, Ni, and inactive cement)

$$\mu r = \left[\left(\frac{\mu}{\rho}\right)_{Ni} x + \left(\frac{\mu}{\rho}\right)_{Fe}(1 - x)\right]\frac{m}{\pi r h},$$

where x is the mass fraction of the Ni. Now:

$$\frac{m}{\pi r h} = r\rho_s, \quad \rho_s = \text{the mass density of the final sample.}$$

mass absorption coefficients here for Co Kα radiation are[11]:

$$\left(\frac{\mu}{\rho}\right)_{Ni} = 75.1, \quad \left(\frac{\mu}{\rho}\right)_{Fe} = 59.5, \quad \left(\frac{\mu}{\rho}\right)_{Ag} = 332.$$

Also: $r = 0.1$ cm, ρ_s (Fe-Ni alloys) $= 3.65$ g/cm^3, ρ_s (Ag) $= 3.92$ g/cm^3.

The total angular correction factor used to multiply the intensity is composed of the preceeding three elements.

$$c = \frac{1}{f^2(LP)A}.$$

This is normalized to unity at $2\theta_1$.

"UNFOLD" has the option to make this correction taking into account the Lorenz-polarization and atomic scattering factors. This is all that is necessary when using a flat sample but the Debye-Scherrer geometry requires the inclusion of the absorption factor.

4. Rachinger Separation, "RACH"

The Rachinger method [4] is a graphical means of separating the $K\alpha_1$-$K\alpha_2$ line doublet.

$$I_1(2\theta_i) = I(2\theta_i) - I_2(2\theta_i) \text{ where}$$

I = intensity due to $\alpha_1 + \alpha_2$ (recorded intensity),

I_1 = " " " α_1,

I_2 = " " " α_2.

The usual assumption [12] is made that the $K\alpha_2$ intensity is half that of the $K\alpha_1$ so that:

$$I_2(2\theta_i) = \tfrac{1}{2} I_1(2\theta - d),$$

where d is the doublet separation. Now

$$I_1(2\theta_i) = I(2\theta_i) - \tfrac{1}{2} I_1(2\theta_i - d).$$

It is important that data be collected sufficiently far out on the low angle side of the profile so that the calculation is started at a diffraction angle sufficiently small that $I_2 \approx 0$, or the measured intensity is due mainly to the $K\alpha_1$ component. Values of I_1 at higher angles can then be determined by successive calculations.

The doublet separation, d, is determined from an approximate peak position of the $K\alpha$ curve and the wavelengths λ, λ_1, and λ_2 of the $K\alpha$, $K\alpha_1$, and $K\alpha_2$ radiations respectively.

From the Bragg equation:

$$\lambda = 2d' \sin\theta \text{ and } \lambda_1 = 2d' \sin\theta_1$$

(d' = interplanar spacing),

so

$$\sin\theta_1 = \frac{\lambda_1}{\lambda} \sin\theta.$$

Similarly for the $K\alpha_2$ component:

$$\sin\theta_2 = \frac{\lambda_2}{\lambda} \sin\theta,$$

and the doublet separation in 2θ is

$$d = 2\theta_2 - 2\theta_1$$

or

$$d = 2\left\{\mathrm{Sin}^{-1}\left[\frac{\lambda_2}{\lambda} \sin\theta\right] - \mathrm{Sin}^{-1}\left[\frac{\lambda_1}{\lambda} \sin\theta\right]\right\}.$$

For cobalt radiation

$$\lambda = 1.79021 \text{ Å},$$

$$\lambda_1 = 1.78892 \text{ Å},$$

$$\lambda_2 = 1.79278 \text{ Å}.$$

In general, the angle $(2\theta_i - d)$ will not coincide with any of the angular values $2\theta_j$ so it is necessary to interpolate. Assume:

$$2\theta_j \leq 2\theta_i - d \leq 2\theta_k.$$

A straight line is fitted between $I_1(2\theta_j)$ and $I_1(2\theta_k)$ and:

$$I_1(2\theta_i - d) = \frac{I_1(2\theta_k) - I_1(2\theta_j)}{2\theta_k - 2\theta_j}(2\theta_i - d) + \frac{[I_1(2\theta_j)](2\theta_k) - [I_1(2\theta_k)](2\theta_j)}{2\theta_k - 2\theta_j}.$$

Although it is not apparent, the Rachinger separation has a tendency to oscillate or become assymmetrical on the high angle side of the profile. This is especially true for a sharp, well separated $K\alpha$ line. For this reason the options for card punching and mathematical smoothing are applied only to the corrected $K\alpha$ profile in this program. This section is still included, however, for those occasions when it might be useful.

5. Center of Gravity Calculation, "CENTROID"

The centroid or center of gravity is one measure of the position of the diffraction line. It is of value in measuring lattice parameters [13], twin fault probabilities [14], etc. According to Pike and Wilson [13] the center of gravity is determined by

$$G = \frac{\sum_i (I_i - b_i)\, c_i (2\theta_i)}{\sum_i (I_i - b_i)\, c_i} \quad , \tag{1}$$

where

G = center of gravity,

$2\theta_i$ = angular position,

I_i = measured intensity,

b_i = background (calculated in sec. 2),

c_i = angular correction factor (calculated in sec. 3).

The summations are over all points.

Equation (1) is modified from the equation given by Pike and Wilson by the presence of the c_i

From the calculation of the background (sec. 2)

setting $\Delta 2\theta = 2\theta_n - 2\theta_1$,

$$b_i = \left(\frac{z - a}{\Delta 2\theta}\right) 2\theta_i + \frac{a(2\theta_n) - z(2\theta_1)}{\Delta 2\theta} \quad ;$$

or

$$b_i = z\left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) - a\left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) \quad . \tag{2}$$

Substituting eq (2) into eq (1)

$$G = \frac{\sum_i \left[I_i - z\left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) + a\left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right)\right] c_i (2\theta_i)}{\sum_i \left[I_i - z\left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) + a\left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right)\right] c_i} \quad ,$$

or

$$G = \frac{\sum_i I_i c_i^{\bullet}(2\theta_i) - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i^{\bullet}(2\theta_i) + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i^{\bullet}(2\theta_i)}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i} \quad . \tag{3}$$

An estimate of the standard deviation is given by the standard propagation of error.

$$\sigma^2(G) = \sum_i \left(\frac{\partial G}{\partial I_i}\right)^2 \sigma^2\left(I_i\right) + \left(\frac{\partial G}{\partial a}\right)^2 \sigma^2(a) + \left(\frac{\partial G}{\partial z}\right)^2 \sigma^2(z) \quad . \tag{4}$$

Now

$$\frac{\partial G}{\partial I_i} = \frac{(2\theta_i)\, c_i}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i}$$

$$- \frac{c_i \left[\sum_i I_i c_i^{\bullet}(2\theta_i) - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i(2\theta_i) + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i^{\bullet}(2\theta_i)\right]}{\left[\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i c_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right)\right]^2} \quad .$$

On substituting from eq (3)

$$\frac{\partial G}{\partial I_i} = \frac{(2\theta_i - G)\, c_i}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i} \quad . \tag{5}$$

Also

$$\frac{\partial G}{\partial a} = \frac{\sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i^{\bullet}(2\theta_i)}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i}$$

$$- \frac{\sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i \left[\sum_i I_i c_i^{\bullet}(2\theta_i) - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i^{\bullet}(2\theta_i) + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i^{\bullet}(2\theta_i)\right]}{\left[\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i\right]^2} \quad .$$

On substituting from eq (3)

$$\frac{\partial G}{\partial a} = \frac{\sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i \cdot (2\theta_i) - G \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i} ,$$

Or

$$\frac{\partial G}{\partial a} = \frac{\sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i \cdot (2\theta_i - G)}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i} . \tag{6}$$

Also:

$$\frac{\partial G}{\partial z} = \frac{-\sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i \cdot (2\theta_i)}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i}$$

$$+ \frac{\sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i \cdot \left[\sum_i I_i c_i \cdot (2\theta_i) - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i \cdot (2\theta_i) + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i \cdot (2\theta_i)\right]}{\left[\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i\right]^2} ,$$

or

$$\frac{\partial G}{\partial z} = \frac{\sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i \cdot (G - 2\theta_i)}{\sum_i I_i c_i - z \sum_i \left(\frac{2\theta_i - 2\theta_1}{\Delta 2\theta}\right) c_i + a \sum_i \left(\frac{2\theta_i - 2\theta_n}{\Delta 2\theta}\right) c_i} . \tag{7}$$

And, since the standard deviation of a random variable is the square root of that variable,

$$\sigma^2(I_i) = I_i , \tag{8}$$

also $a = \frac{1}{5} \sum_{i=1}^{5} I_i$ (see sec. 2) ,

$$\sigma^2(a) = \sum_i \left(\frac{\partial a}{\partial I_i}\right)^2 \sigma^2(I_i) \quad \text{where} \quad \frac{\partial a}{\partial I_i} = \frac{1}{5} ,$$

so $$\sigma^2(a) = \frac{1}{25} \sum_{i=1}^{5} I_i = \frac{1}{5} a \cdot \tag{9}$$

In a similar manner for z,

$$\sigma^2(z) = \frac{1}{5} z \cdot \tag{10}$$

The center of gravity is calculated from eq (1) and the estimate of its standard deviation is determined from eq (4) using eq (5) through eq (10).

6. Peak Calculation, "PEAK"

The position of peak maximum is another measure of the profile's angular location. It also is used to measure lattice parameter and twin fault probabilities, as well as stacking fault probabilities [15].

A portion of the profile on either side of the maximum (we generally used a total of 7 to 9 points) is fitted to a parabola[16] and the maximum of that curve is determined.

$$I_i = B + C \cdot (2\theta_i) + D \cdot (2\theta_i)^2 .$$

Differentiating:

$$\frac{dI_i}{d(2\theta_i)} = C + 2D \cdot (2\theta_i) = 0 ,$$

so that

$$\text{peak} = 2\theta_{max} = -\frac{C}{2D} , \tag{11}$$

where B, C, and D are the parameters of the fitted curve.

The standard propagation of error with correlation between the variables C and D gives:

$$\sigma^2(2\theta_{max}) = \left(\frac{\partial(2\theta_{max})}{\partial C}\right)^2 \sigma^2(C) + \left(\frac{\partial(2\theta_{max})}{\partial D}\right)^2 \sigma^2(D)$$

$$+ 2\left(\frac{\partial(2\theta_{max})}{\partial C}\right)\left(\frac{\partial(2\theta_{max})}{\partial D}\right) \sigma(C)\sigma(D) .$$

Differentiating eq (11) this becomes

$$\sigma^2(2\theta_{max}) = \left(\frac{1}{4D^2}\right)\sigma^2(C) + \left(\frac{C^2}{4D^4}\right)\sigma^2(D) + 2\left(\frac{-1}{2D}\right)\left(\frac{C}{2D^2}\right)\sigma(C)\sigma(D),$$

$$= \frac{(2\theta_{max})^2}{C^2}\sigma^2(C) + \frac{(2\theta_{max})^2}{D^2}\sigma^2(D) - 2\frac{(2\theta_{max})^2}{CD}\sigma(C)\sigma(D).$$

A modification of a computer program written by W. J. Hall was used here to compute the curve parameters and their standard deviations. This program performs an orthonormal curve fitting using Bjork's modification[17, 18] of the Gram-Schmidt technique.

7. Curve Smoothing, "SMOOTH"

In the course of experimenting on several iron-nickel alloys, it was found that the Fourier analyses of the diffraction profiles were far more repeatable if the data from the cold-worked sample were first smoothed. This subroutine affords the option of performing this operation.

The intensity curve is mathematically smoothed by a least-squares fit of a quartic equation to seven points, taking the value at the midpoint and then advancing the group of points by one unit and repeating the process.[19]

$$I_i(\text{smoothed}) = (I_{i-3} - 18I_{i-2} + 63I_{i-1} + 164I_i + 63I_{i+1} - 18I_{i+2} + I_{i+3})/256 .$$

Three points are added before the initial intensity and three after the final intensity and they are all set equal to zero.

This smoothing operation can be performed repeatedly (limited to 99 times by format). It is possible to have up to ten of these smoothed curves printed and to have one of them card punched for input to "UNFOLD".

8. Data Input and Output and a Sample

Card Input

Card	Information	Format
1	Identification	50H
2	a. Number of points (intensity with angular position) in profile. b. Initial 2θ, i.e., $2\theta_1$ c. 2θ increment, $\delta 2\theta$ d. 2θ position of peak (must equal $2\theta_1 + k \times \delta 2\theta$, k an integer). e. Angular correction added to each 2θ*. f. Ni fraction in an Fe-Ni alloy. g. 1 for bcc phase, 2 for fcc phase in an Fe-Ni sample (e, f, and g are necessary only when they are applicable).	I10, 5F10.2, I10
3	Punch options for input to "UNFOLD" a. 0 for no card punch, 1 for punch of corrected Kα profile, 2 for punch of corrected Kα profile after smoothing. b. Number of smoothings to be printed (max = 10) c. Smoothing number to be punched (0 for no punch). (b and c are necessary only if 3a = 2)	3I5
4	Smoothing numbers to be printed.(This card is omitted if 3a $\neq$ 2. The number in 3c must be one of these numbers).	10I5
5	Lower and upper angular limits for parabolic fit to determine profile maximum.	2F10.2
6.	Intensities	10F7.0

Output data includes:

1. Identification
2. Table containing angular positions, recorded intensities, backgrounds, angular corrections, and the corrected Kα and Kα_1 profiles.

---

* Using the Debye-Scherrer technique, it is possible to calibrate the diffractometer by examining the reflection on both positive and negative sides of the diffraction cone [6, 8]. This gives a correction factor to the angle read from the instrument.

3. Line maximum and center of gravity with their standard deviations for $K\alpha$ and $K\alpha_1$ profiles.

4. Peak value used for punching "UNFOLD" input and smoothing number used (if applicable).

5. Table containing angular positions and smoothed $K\alpha$ profile (if applicable).

A sample of input and output follows.

Punched Card Input

```
FE-28NI    COLD-WORKED  (111)
     111        50.60        0.02        51.92                    0.28          2
   2     4     4
   2     4     6     8
    51.72        52.02
 270.    260.    260.    270.    260.    290.    240.    290.    310.    310.
 290.    290.    310.    300.    270.    260.    320.    280.    330.    330.
 300.    330.    360.    340.    370.    330.    350.    360.    340.    350.
 390.    400.    400.    370.    420.    450.    490.    470.    510.    510.
 530.    600.    620.    710.    740.    740.    830.    910.    950.    940.
1010.   1170.   1260.   1330.   1410.   1550.   1620.   1720.   1840.   1940.
2050.   2130.   2220.   2270.   2350.   2380.   2390.   2390.   2360.   2320.
2280.   2190.   2040.   1940.   1840.   1600.   1330.   1160.   1050.    950.
 860.    810.    760.    720.    670.    610.    560.    510.    490.    470.
 440.    420.    400.    390.    380.    370.    360.    350.    340.    330.
 320.    310.    300.    290.    280.    270.    270.    270.    270.    270.
 270.
```

Printed Output

FE-28NI COLD-WORKED (111)

2 THETA	REC CNT	BACK	ANG COR	ALPHA	ALPHA 1
50.60	270	264	1.0000	6	0
50.62	260	264	1.0004	-4	0
50.64	260	264	1.0009	-4	0
50.66	270	264	1.0013	6	0
50.68	260	264	1.0017	-4	0
50.70	290	264	1.0022	26	0
50.72	240	264	1.0026	-24	0
50.74	290	264	1.0030	26	0
50.76	310	264	1.0035	46	46
50.78	310	264	1.0039	46	46
50.80	290	265	1.0044	26	26
50.82	290	265	1.0048	26	26
50.84	310	265	1.0052	46	46
50.86	300	265	1.0057	35	35
50.88	270	265	1.0061	5	-17
50.90	260	265	1.0065	-5	-28
50.92	320	265	1.0070	56	43
50.94	280	265	1.0074	15	2
50.96	330	265	1.0078	66	43
50.98	330	265	1.0083	66	48
51.00	300	265	1.0087	35	43
51.02	330	265	1.0092	65	79
51.04	360	265	1.0096	96	75

51.06	340	265	1.0100	75	74
51.08	370	265	1.0105	106	85
51.10	330	265	1.0109	65	42
51.12	350	265	1.0113	86	64
51.14	360	265	1.0118	96	56
51.16	340	266	1.0122	75	38
51.18	350	266	1.0127	85	48
51.20	390	266	1.0131	126	84
51.22	400	266	1.0135	136	115
51.24	400	266	1.0140	136	104
51.26	370	266	1.0144	106	77
51.28	420	266	1.0148	156	137
51.30	450	266	1.0153	187	163
51.32	490	266	1.0157	228	186
51.34	470	266	1.0162	207	150
51.36	510	266	1.0166	248	196
51.38	510	266	1.0170	248	209
51.40	530	266	1.0175	268	200
51.42	600	266	1.0179	340	259
51.44	620	266	1.0184	360	267
51.46	710	266	1.0188	452	377
51.48	740	266	1.0192	483	385
51.50	740	266	1.0197	483	378
51.52	830	267	1.0201	575	475
51.54	910	267	1.0206	657	528
51.56	950	267	1.0210	698	564
51.58	940	267	1.0214	688	500
51.60	1010	267	1.0219	760	567
51.62	1170	267	1.0223	923	734
51.64	1260	267	1.0228	1016	779
51.66	1330	267	1.0232	1088	824
51.68	1410	267	1.0236	1170	888
51.70	1550	267	1.0241	1314	1063
51.72	1620	267	1.0245	1386	1103
51.74	1720	267	1.0250	1489	1123
51.76	1840	267	1.0254	1613	1224
51.78	1940	267	1.0258	1716	1304
51.80	2050	267	1.0263	1830	1386
51.82	2130	267	1.0267	1912	1382
51.84	2220	267	1.0272	2006	1454
51.86	2270	267	1.0276	2058	1496
51.88	2350	267	1.0280	2141	1530
51.90	2380	268	1.0285	2173	1521
51.92	2390	268	1.0289	2184	1491
51.94	2390	268	1.0294	2185	1494
51.96	2360	268	1.0298	2155	1428
51.98	2320	268	1.0303	2114	1366
52.00	2280	268	1.0307	2074	1309
52.02	2190	268	1.0311	1982	1221
52.04	2040	268	1.0316	1828	1082
52.06	1940	268	1.0320	1726	979
52.08	1840	268	1.0325	1623	908
52.10	1600	268	1.0329	1376	692
52.12	1330	268	1.0334	1097	442
52.14	1160	268	1.0338	922	311
52.16	1050	268	1.0342	809	266
52.18	950	268	1.0347	705	215
52.20	860	268	1.0351	612	158
52.22	810	268	1.0356	561	213
52.24	760	268	1.0360	509	286
52.26	720	269	1.0365	468	312
52.28	670	269	1.0369	416	283
52.30	610	269	1.0374	354	246
52.32	560	269	1.0378	302	223

52.34	510	269	1.0382	250	144
52.36	490	269	1.0387	230	87
52.38	470	269	1.0391	209	53
52.40	440	269	1.0396	178	36
52.42	420	269	1.0400	157	34
52.44	400	269	1.0405	136	25
52.46	390	269	1.0409	126	53
52.48	380	269	1.0414	115	71
52.50	370	269	1.0418	105	78
52.52	360	269	1.0422	95	76
52.54	350	269	1.0427	84	67
52.56	340	269	1.0431	74	61
52.58	330	269	1.0436	63	37
52.60	320	269	1.0440	53	17
52.62	310	270	1.0445	42	3
52.64	300	270	1.0449	32	-6
52.66	290	270	1.0454	21	-12
52.68	280	270	1.0458	11	-20
52.70	270	270	1.0463	0	-18
52.72	270	270	1.0467	0	-9
52.74	270	270	1.0471	0	-2
52.76	270	270	1.0476	0	3
52.78	270	270	1.0480	0	6
52.80	270	270	1.0485	0	10

ALPHA CURVE
PEAK MAX = 51.9298
STD DEV = 0.1335
PEAK FIGURED FROM 51.72 TO 52.02

LINE CG = 51.8653 STD DEV = 0.0045

ALPHA 1 CURVE
PEAK MAX = 51.8958
STD DEV = 0.2744
PEAK FIGURED FROM 51.68 TO 51.98

LINE CG = 51.8257 STD DEV = 0.0065

PUNCH PEAK FOR ALPHA = 51.92
SMOOTHING 4

ANGLE	INPUT	SMOOTH 2	SMOOTH 4	SMOOTH 6	SMOOTH 8
50.60	6	2	0	-0	-0
50.62	-4	-1	-1	-1	-1
50.64	-4	-3	-2	-1	-1
50.66	6	1	2	2	2
50.68	-4	7	5	4	3
50.70	26	4	2	2	2
50.72	-24	-1	3	5	7
50.74	26	17	19	20	20
50.76	46	43	39	36	35
50.78	46	43	41	40	39
50.80	26	29	33	35	36
50.82	26	30	32	34	35
50.84	46	40	37	34	33
50.86	35	31	28	26	25

50.88	5	8	12	14	16
50.90	-5	10	12	13	14
50.92	56	29	26	25	25
50.94	15	40	42	41	41
50.96	66	54	53	52	51
50.98	66	57	55	54	54
51.00	35	50	54	56	57
51.02	65	64	65	66	67
51.04	96	85	82	81	80
51.06	75	91	91	89	88
51.08	106	88	87	88	88
51.10	65	80	82	84	84
51.12	86	83	83	83	82
51.14	96	87	84	82	82
51.16	75	81	83	84	85
51.18	85	91	94	97	98
51.20	126	120	118	117	116
51.22	136	137	132	129	127
51.24	136	128	128	128	128
51.26	106	122	126	130	132
51.28	156	149	150	152	153
51.30	187	191	187	185	184
51.32	228	215	213	212	210
51.34	207	224	225	226	225
51.36	248	236	235	235	236
51.38	248	251	251	251	251
51.40	268	278	279	280	281
51.42	340	323	324	325	326
51.44	360	380	381	381	380
51.46	452	439	435	432	429
51.48	483	474	471	471	471
51.50	483	502	510	514	517
51.52	575	571	574	576	576
51.54	657	652	643	637	633
51.56	698	685	680	677	675
51.58	688	700	708	714	718
51.60	760	775	782	787	790
51.62	923	905	899	895	893
51.64	1016	1014	1008	1004	1001
51.66	1088	1090	1095	1097	1097
51.68	1170	1184	1187	1189	1190
51.70	1314	1295	1291	1290	1289
51.72	1386	1394	1394	1393	1392
51.74	1489	1493	1496	1497	1498
51.76	1613	1607	1607	1607	1608
51.78	1716	1721	1720	1719	1719
51.80	1830	1825	1825	1824	1824
51.82	1912	1918	1917	1917	1917
51.84	2006	1998	1998	1999	1999
51.86	2058	2069	2071	2071	2072
51.88	2141	2133	2132	2131	2131
51.90	2173	2173	2172	2171	2171
51.92	2184	2187	2187	2187	2187
51.94	2185	2181	2180	2181	2181
51.96	2155	2156	2157	2159	2160
51.98	2114	2120	2123	2123	2122
52.00	2074	2069	2064	2061	2059
52.02	1982	1971	1969	1968	1969
52.04	1828	1844	1852	1858	1861
52.06	1726	1734	1736	1736	1736
52.08	1623	1599	1587	1579	1574
52.10	1376	1373	1369	1365	1363
52.12	1097	1114	1124	1130	1134
52.14	922	924	929	933	938

52.16	809	803	799	797	797
52.18	705	703	701	699	698
52.20	612	618	620	621	621
52.22	561	557	558	559	560
52.24	509	511	511	510	510
52.26	468	466	465	465	464
52.28	416	415	414	413	412
52.30	354	356	356	356	356
52.32	302	300	300	301	302
52.34	250	255	257	258	259
52.36	230	228	228	228	228
52.38	209	206	205	204	203
52.40	178	180	180	180	180
52.42	157	156	156	157	157
52.44	136	138	138	138	139
52.46	126	125	125	125	125
52.48	115	115	115	115	115
52.50	105	105	105	105	105
52.52	95	95	95	95	95
52.54	84	84	84	84	84
52.56	74	74	74	74	74
52.58	63	63	63	63	63
52.60	53	53	53	53	53
52.62	42	42	42	42	42
52.64	32	32	32	32	32
52.66	21	21	21	21	21
52.68	11	10	10	10	10
52.70	0	2	2	3	3
52.72	0	-0	-0	-0	-0
52.74	0	-0	-0	-1	-1
52.76	0	0	0	-0	-0
52.78	0	0	0	0	0
52.80	0	0	0	0	0

Punched Card Output For "UNFOLD"

2187.	2180.	2157.	2123.	2064.	1969.	1852.	1736.	1587.	1369.
1124.	929.	799.	701.	620.	558.	511.	465.	414.	356.
300.	257.	228.	205.	180.	156.	138.	125.	115.	105.
95.	84.	74.	63.	53.	42.	32.	21.	10.	2.
-0.	-0.	0.	0.	0.					
2187.	2172.	2132.	2071.	1998.	1917.	1825.	1720.	1607.	1496.
1394.	1291.	1187.	1095.	1008.	899.	782.	708.	680.	643.
574.	510.	471.	435.	381.	324.	279.	251.	235.	225.
213.	187.	150.	126.	128.	132.	118.	94.	83.	84.
83.	82.	87.	91.	82.	65.	54.	55.	53.	42.
26.	12.	12.	28.	37.	32.	33.	41.	39.	19.
3.	2.	5.	2.	-2.	-1.	0.			

9. Program Listing

The listing which follows includes two versions on the subroutine "ANG", one for the two phase Fe-Ni alloys and one for Ag. Of course, only one version should appear in the program deck. The program has been written in FORTRAN.

```
      PROGRAM PREP
      DIMENSION Y(300), Y1(300), YC(300),NS(10)
      COMMON X(300), B(300), C(300), N
      DOUBLE PRECISION PK,PK1,CG,CG1,SDPK,SDPK1,SDCG,SDCG1
111   READ 6
      IF (EOF,60) 999,100
100   READ 2, N,AI,AINC,APK,V,FN,IG
      READ 1, IPUN,NP,NUMP
      IF (IPUN-2) 121,120,121
120   READ 1,(NS(I),I=1,NP)
121   DO 101 J=1,N
101   X(J) = AI + (J-1)*AINC
      READ 3, R1,R2
      READ 4, (Y(J),J=1,N)
      CALL BACK(Y,A,Z,D)
      CALL ANG(IG,V,FN)
      CALL RACH(Y,Y1,APK,A1)
      CALL CENTROID(Y,CG,SDCG,A,Z,D)
      CALL CENTROID(Y1,CG1,SDCG1,A,Z,D)
      CALL PEAK(Y,PK,SDPK,R1,R2)
      NN = XFIXF((PK - A1*360./3.1415927)/AINC)
      R3 = R1 - NN*AINC
      R4 = R2 - NN*AINC
      CALL PEAK(Y1,PK1,SDPK1,R3,R4)
      DO 103 J=1,N
      IF (APK-X(J))103,102,103
102   NF = J
      GO TO 104
103   CONTINUE
104   DO 105 J=1,N
      Y1(J) = (Y1(J)-B(J))*C(J)
105   YC(J) = (Y(J)-B(J))*C(J)
      PRINT 14
      PRINT 6
      PRINT 7
      PRINT 8, (X(J),Y(J),B(J),C(J),YC(J),Y1(J),J=1,N)
      PRINT 9, PK,SDPK
      PRINT 10, R1,R2
      PRINT 11, CG,SDCG
      PRINT 12, PK1, SDPK1
      PRINT 10, R3,R4
      PRINT 11,CG1,SDCG1
      IF (IPUN) 107,107,108
108   IF (IPUN-2) 110,109,109
109   CALL SMOOTH(YC,NUMP,NS,NP,NCEN)
      GO TO 107
110   PP = AI + (NF-1)*AINC
      PRINT 13, PP
      DO 106 K=1,NF
106   C(K) = YC(NF+1-K)
      PUNCH 5,(YC(J),J=NF,N)
      PUNCH 5, (C(J),J=1,NF)
107   GO TO 111
1     FORMAT (10I5)
```

```
2     FORMAT (I10,5F10.2,I10)
3     FORMAT (2F10.2)
4     FORMAT (10F7.0)
5     FORMAT (10(F6.0,$,$))
6     FORMAT (50H                                                    )
7     FORMAT (///3X,$2 THETA$,3X,$REC CNT$,6X,$BACK$,3X,$ANG COR$,5X,
     1$ALPHA$,3X,$ALPHA 1$/)
8     FORMAT (F10.2,2F10.0,F10.4,2F10.0)
9     FORMAT (///$ ALPHA CURVE $/$ PEAK MAX = $,F9.4/$  STD DEV = $,
     1F9.4)
10    FORMAT (1X,$PEAK FIGURED FROM $,F7.2,2X,$TO$,F7.2)
11    FORMAT (/$ LINE CG = $,F9.4,$ STD DEV = $,F9.4)
12    FORMAT (///$ ALPHA 1 CURVE $/$ PEAK MAX = $,F9.4/$  STD DEV = $,
     1F9.4)
13    FORMAT (///1X,$PUNCH PEAK FOR ALPHA = $,F7.2)
14    FORMAT (//////)
999   END



      SUBROUTINE BACK(Y,A,Z,D)
C
C      BACKGROUND CALCULATION
C
      COMMON X(300), B(300), C(300), N
      DIMENSION Y(300)
      A = (Y(1)+Y(2)+Y(3)+Y(4)+Y(5))/5.
      Z = (Y(N-4)+Y(N-3)+Y(N-2)+Y(N-1)+Y(N))/5.
      D = X(N) - X(1)
      DO 200 J=1,N
200   B(J) = (Z-A)*X(J)/D + (A*X(N)-Z*X(1))/D
      RETURN
      END



      SUBROUTINE ANG(IG,V,FN)
C
C     ANGULAR CORRECTION  -  FOR FE-NI ALLOY SAMPLES AND CO RADIATION
C
      COMMON X(300), B(300), C(300), N
      PI = 3.1415927
      DA = COS(31.2333*PI/180.)**2                                    A
      DO 313 J=1,N
      SL = SIN((X(J)+V)*PI/360.)/1.79021                              B
      IF (IG-1) 306,306,301
301   IF (SL-0.12) 302,302,303
302   F = 1.-0.142*SL-6.44*SL**2                                      C-FCC
      GO TO 311
303   IF (SL-0.35) 304,304,305
304   F=1.0645004-1.4746725*SL+0.68002691*SL**2-0.53337307*SL**3      D-FCC
      GO TO 311
305   F=1.1781009-2.1006716*SL+1.4750092*SL**2-0.28333892*SL**3       E-FCC
      GO TO 311
306   IF (SL-0.12) 307,307,308
307   F = 1. - 0.184*SL - 7.08*SL**2                                  C-BCC
      GO TO 311
308   IF (SL-0.35) 309,309,310
309   F=1.0637002-1.6056701*SL+0.86001593*SL**2-0.53335684*SL**3      D-BCC
      GO TO 311
310   F=1.1987014-2.2833411*SL+2.0200144*SL**2-0.56667538*SL**3       E-BCC
311   A = SIN((X(J)+V)*PI/360.)
```

```
      BB= COS((X(J)+V)*PI/360.)                                          F
      CC= SIN((X(J)+V)*PI/180.)
      D = COS((X(J)+V)*PI/180.)
      C(J) = (A**2)*BB/(F*F*(1.+DA*D**2))                                G
      E = BB*BB*LOGF((1.+A)/BB)/A                                        H
      RM = (75.1*FN + 59.5*(1.-FN))*0.365                                I
      ABS = (1.-E)/(PI*RM)+CC/(2.*PI*RM*RM)+(-.25+.375*BB*BB*(1.+E))/    I
     1(PI*RM**3)
313   C(J) = C(J)/ABS
      CN = C(1)
      DO 312 J=1,N
312   C(J) = C(J)/CN
      RETURN
      END



      SUBROUTINE ANG(IG,V,FN)
C
C     ANGULAR CORRECTION  -  FOR AG SAMPLES AND CO RADIATION
C
      COMMON X(200),B(200),C(200),N
      PI = 3.1415927
      DA = COS(31.2333*PI/180.)**2                                       A
      DO 313 J=1,N                                                       B
      SL = SIN((X(J)+V)*PI/360.)/1.79021
306   IF (SL-0.12) 307,307,308                                           C
307   F = 1. - 0.068*SL - 5.96*SL**2
      GO TO 311
308   IF (SL-0.35) 309,309,310                                           D
309   F=1.0685171-1.2033810*SL-1.2685714*SL**2+3.0666667*SL**3
      GO TO 311                                                          E
310   F=1.2061-2.4973333*SL+2.805*SL**2-1.2166667*SL**3
311   A = SIN((X(J)+V)*PI/360.)
      BB= COS((X(J)+V)*PI/360.)                                          F
      CC= SIN((X(J)+V)*PI/180.)
      D = COS((X(J)+V)*PI/180.)
      C(J) = (A**2)*BB/(F*F*(1.+DA*D**2))                                G
      E = BB*BB*LOGF((1.+A)/BB)/A                                        H
      RM = 130.14                                                        I
      ABS = (1.-E)/(PI*RM)+CC/(2.*PI*RM*RM)+(-.25+.375*BB*BB*(1.+E))/    I
     1(PI*RM**3)
313   C(J) = C(J)/ABS
      CN = C(1)
      DO 312 J=1,N
312   C(J) = C(J)/CN
      RETURN
      END


      SUBROUTINE RACH(Y,Y1,APK,P1)
C
C     RACHINGER SEPARATION  -  FOR CO RADIATION
C
      COMMON X(300), B(300), C(300), N
      DIMENSION Y(300),Y1(300)
      P1 = 1.78892/1.79021*SIN(APK*3.1415927/360.)                       A
      P2 = 1.79278/1.79021*SIN(APK*3.1415927/360.)                       B
       P1 = ATAN(P1/SQRT(1.-P1**2))
      P2 = ATAN(P2/SQRT(1.-P2**2))
      D = (P2-P1)*360./3.1415927
      DO 606 I=2,N
```

```
      IF (X(I)-X(1)-D) 606,605,605
605   DO 600 J=1,I
600   Y1(J) = B(J)
      IA = I + 1
      GO TO 607
606   CONTINUE
607   IB = N - 1
      DO 603 J=IA,N
      DO 604 K=1,IB
      IF (X(K)-X(J)+D) 601,601,604
601   IF (X(K+1)-X(J)+D) 604,604,602
602   Z = ((Y1(K+1)-B(K+1))*C(K+1)-(Y1(K)-B(K))*C(K))/(X(K+1)-X(K))
     1*(X(J)-D)+((Y1(K)-B(K))*C(K)*X(K+1)-(Y1(K+1)-B(K+1))*C(K+1)*X(K))
     1/(X(K+1)-X(K))
      GO TO 608
604   CONTINUE
608   Y1(J) = (Y(J)-B(J))*C(J) - Z/2.
603   Y1(J) = Y1(J)/C(J) + B(J)
      RETURN
      END



      SUBROUTINE CENTROID(Y,CG,SDCG,A,Z,D)
C
C     CENTROID CALCULATION
C
      COMMON X(300), B(300), C(300), N
      DIMENSION Y(300)
      DOUBLE PRECISION S(6),P1
      DO 500 J=1,6
500   S(J) = 0.
      P1 = 0.
      DO 501 J=1,N
      S(1) = S(1)+Y(J)*X(J)*C(J)
      S(2) = S(2)+(X(J)-X(1))/D*C(J)*X(J)
      S(3) = S(3)+(X(J)-X(N))/D*C(J)*X(J)
      S(4) = S(4)+Y(J)*C(J)
      S(5) = S(5)+(X(J)-X(1))/D*C(J)
501   S(6) = S(6)+(X(J)-X(N))/D*C(J)
      CG = (S(1)-Z*S(2)+A*S(3))/(S(4)-Z*S(5)+A*S(6))
      DO 502 J=1,N
502   P1 = P1+Y(J)*(X(J)-CG)*(X(J)-CG)*C(J)*C(J)/(S(4)-Z*S(5)+A*S(6))/
     1(S(4)-Z*S(5)+A*S(6))
      SDCG = P1+A/5.*(S(3)-CG*S(6))*(S(3)-CG*S(6))/(S(4)-Z*S(5)+A*S(6))
     1/(S(4)-Z*S(5)+A*S(6))
      SDCG=SDCG+Z/5.*(CG*S(5)-S(2))*(CG*S(5)-S(2))/(S(4)-Z*S(5)+A*S(6))/
     1(S(4)-Z*S(5)+A*S(6))
      SDCG = DSQRT(SDCG)
      RETURN
      END



      SUBROUTINE PEAK(Z,PK,SDPK,R1,R2)
C
C     PARABOLIC FIT TO PEAK MAX
C
      COMMON X(300), BG(300), COR(300), N
      DIMENSION A(99,3),B(99),C(3,3),D(3),R(200),Y(3),Z(300),E(3,3),
     1  AA(3)
```

```
      TYPE DOUBLE A,B,C,D,R,Y,AA,PK,SDPK
      DO 406 J=1,N
      IF (R1-X(J)) 403,403,406
403   IR1 = J
      GO TO 411
406   CONTINUE
411   DO 404 J=IR1,N
      IF (R2-X(J)) 407,405,404
405   IR2 = J
      GO TO 412
407   IR2 = J-1
      GO TO 412
404   CONTINUE
412   NP = IR2-IR1+1
      DO 401 I=IR1,IR2
      A(I-IR1+1,1) = 1.0
      A(I-IR1+1,2) = X(I)
      A(I-IR1+1,3) = X(I)*X(I)
401   B(I-IR1+1) = (Z(I)-BG(I))*COR(I)
      D(1)=0.0
      Y(1)=0.0
      SS=0.
      DO 1 I=1,NP
      SS=B(I)*B(I)+SS
      D(1)=A(I,1)*A(I,1)+D(1)
    1 Y(1)=A(I,1)*B(I)+Y(1)
      Y(1)=Y(1)/D(1)
      IR=0
      DO 5 K=2,3
      DO 3 J=K,3
      IR=IR+1
      R(IR)=0.0
      DO 2 I=1,NP
    2 R(IR)=A(I,K-1)*A(I,J)+R(IR)
      R(IR)=R(IR)/D(K-1)
      DO 3 I=1,NP
    3 A(I,J)=A(I,J)-A(I,K-1)*R(IR)
      D(K)=0.0
      Y(K)=0.0
      DO 4 I=1,NP
      B(I)=B(I)-A(I,K-1)*Y(K-1)
      Y(K)=A(I,K)*B(I)+Y(K)
    4 D(K)=A(I,K)*A(I,K)+D(K)
    5 Y(K)=Y(K)/D(K)
      DO 55 I=1,3
      AA(I)=Y(I)*D(I)**0.5
      SS=SS-AA(I)*AA(I)
      FN=NP-I
      IF(FN) 51,51,52
 51   SD = 0.
      GO TO 53
 52   SD=SQRT(SS/FN)
 53   DO 55 J=1,I
   55 E(I,J)=SD
      IRS=-3
      DO 8 K=1,3
      IRS=IRS-K+4
      IR=IRS
      DO 8 JJ=1,K
      J=K-JJ+1
      C(K,J)=Y(J)
      IF(JJ-1) 8,8,6
    6 DO 7 I=2,JJ
      C(K,J)=C(K,J)-C(K,K-I+2)*R(IR)
```

```
      E(K,J)=E(K,J)+ABSF(R(IR)*E(K,K-I+2))
    7 IR=IR-1
    8 IR=IR-3 +K
      PK = -C(3,2)/(2.*C(3,3))
      SDPK = DSQRT((PK*E(3,2)/C(3,2))**2 + (PK*E(3,3)/C(3,3))**2 -
     1  2*(PK**2)*E(3,2)*E(3,3)/C(3,2)/C(3,3))
      RETURN
      END



      SUBROUTINE SMOOTH(YC,NUMP,NS,NP,NCEN)
      DIMENSION S(11,500),D1(500),D2(500),NS(10),YC(300)
      COMMON X(300), B(300), C(300), NUM
      X1 = X(NCEN)
      DO 22 N=1,NUM
22    S(1,N+3) = YC(N)
      NUM = NUM + 3
      DO 20 N=1,3
      D2(N) = 0.
20    D2(NUM+N) = 0.
      DO 5 N=4,NUM
5     D1(N) = S(1,N)
      K = 0
      DO 12 I=1,NP
      J=NS(I)
8     DO 9 N=4,NUM
9     D2(N) = D1(N)
6     K = K + 1
      DO 7 L=4,NUM
7     D1(L)=(D2(L-3)-18.*D2(L-2)+63.*D2(L-1)+164.*D2(L)+63.*D2(L+1)
     1-18.*D2(L+2)+D2(L+3))/256.
      IF (K-J) 8,10,8
10    M = I + 1
      DO 11 N=4,NUM
11    S(M,N) = D1(N)
12    CONTINUE
      IF (NUMP) 13,15,13
13    NCEN = NCEN + 3
      DO 18 N=1,NP
      IF (NUMP-NS(N)) 18,17,18
17    NUMP = N + 1
      GO TO 19
18    CONTINUE
19    PUNCH 105,(S(NUMP,N),N=NCEN,NUM)
      M = NCEN - 3
      DO 14 N=1,M
      K = NCEN - N + 1
14    D2(N) = S(NUMP,K)
      PUNCH 105, (D2(N),N=1,M)
      NUMP = NUMP - 1
      PRINT 109, X1,NS(NUMP)
15    PRINT 107, (NS(N),N=1,NP)
      M = NP + 1
      DO 16 N=4,NUM
16    PRINT 108, X(N-3),(S(J,N),J=1,M)
105   FORMAT (10(F6.0,$,$))
107   FORMAT (////1X,$ANGLE$,5X,$INPUT$,3X,10($SMOOTH $,I2,1X))
108   FORMAT (1X,F7.2,11F10.0)
109   FORMAT (//1X,$PUNCH PEAK FOR ALPHA = $,F7.2/1X,$SMOOTHING$,I3)
      END
```

10. Appendix: Corrections Necessary When Using Other Radiations, Samples and Geometries.

Specific program steps are referred to by the letter identification which appears in the identification columns.

A. Other radiations

In "ANG":

The value of the diffraction angle in the monochromator must be inserted into the calculation of DA (step A). In this case, for Co $K\alpha$ radiation and a quartz crystal, $2\alpha = 31.2333°$. It is also necessary to use the $K\alpha$ wavelength to determine SL (step B); here $\lambda = 1.79021\text{Å}$.

In "RACH":

The values of the $K\alpha$, $K\alpha_1$, and $K\alpha_2$ wavelengths (i.e., λ, λ_1, and λ_2) are necessary to evaluate P1 and P2 (steps A and B) as follows:

$$P1 = \frac{\lambda_1}{\lambda} \sin\left[APK\left(\frac{\pi}{360}\right)\right]$$

$$P2 = \frac{\lambda_2}{\lambda} \sin\left[APK\left(\frac{\pi}{360}\right)\right]$$

B. Other samples

In "ANG":

The parameters of the power series fit to the atomic scattering factors must be used in steps C, D, and E. RM in step H is the factor μr in section 3 and must be evaluated as noted there.

C. Other geometries

In "ANG":

When no monochromator is used, DA (step A) should be set equal to one. For a flat sample, eliminate CC, E, and RM (steps F, G, and H respectively) and set ABS (step I) equal to one.

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