An Investigation to Locate and Correct Errors
in the Card File Prepared by
the Joint Committee on Powder Diffraction Standards (JCPDS)

by

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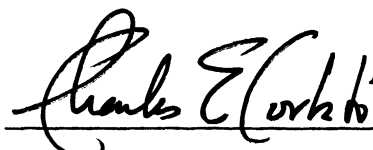

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I. TABLE OF CONTENTS

II. LIST OF TABLES

III. INTRODUCTION

The objective of this research was to search for errors in the card file of X-ray diffraction data prepared by the Joint Committee on Powder Diffraction Standards (JCPDS) and, where possible, to find answers for the errors. Expected errors include incorrect indexing of reflections, incorrect d-values and/or cell dimensions, reflections attributable to impurities or a combination of these.

PROCEDURE

The procedure used in this investigation was as follows: Initially, a mineral in either the tetragonal, hexagonal, orthorhombic or cubic crystal system was picked at random from a list of minerals available in the Mineralogy museum. The reason for only using minerals in the Mineralogy museum was that in order to determine the cause of some errors it may be necessary to produce an X-ray diffraction pattern in which case the mineral would have to be readily available. Next, the JCPDS card was found for the mineral chosen. All the hkl values and corresponding d-values listed on that card were fed into a least-squares computer program, prepared by C.E. Corbato, for the refinement of cell parameters for a comparison with the cell dimensions given on the card. The program includes a least-squares procedure minimizing the sum of the squares of the residuals of $1/d^2$ and a non-linear least-squares procedure minimizing the sum of the squares of the residuals of theta. At present the program can handle tetragonal, hexagonal, orthorhombic and cubic crystal systems only. Therefore, minerals

in the triclinic and monoclinic crystal systems were not considered for this investigation.

The least-squares method is based on the assumption that the variable for which the sum of the squares of the residuals to be minimized will be randomly distributed about its mean value. In X-ray diffraction this variable is usually theta or 2-theta; however, the cell parameters are not linearly related to theta. Thus, the least-squares method is only used to give a first approximation of the refined cell parameters when the parameter minimized is $1/d^2$. The following example, taken from G&M 837 Coursework, illustrates the method of least-squares as applied to cell parameter refinement:

LEAST-SQUARES REFINEMENT OF CELL PARAMETERS
TETRAGONAL AND HEXAGONAL

THEORY

Consider the d-spacing formulas:

(eqn. 1) $\quad \dfrac{1}{d^2} = \dfrac{h^2+k^2}{a^2} + \dfrac{1^2}{c^2}$ (tetragonal), $\dfrac{1}{d^2} = \dfrac{4(h^2+hk+k^2)}{3a^2} + \dfrac{1^2}{c^2}$ (hexagonal).

Let $D = \dfrac{1}{d^2}$, $A = \dfrac{1}{a^2}$, $C = \dfrac{1}{c^2}$, $r = h^2+k^2$ or $\dfrac{4}{3}(h^2+hk+k^2)$, $s = 1^2$. Then

eqn. 1 becomes a linear equation in two variables, A and C:

(eqn. 2) $\qquad D = rA + sC$

The "normal" equations for least-squares solution of this linear equation are the following: $\qquad \left[\sum r_i^2\right] A + \left[\sum r_i s_i\right] C = \sum r_i D_i$

$\left[\sum r_i s_i\right] A + \left[\sum s_i^2\right] C = \sum s_i D_i$

2.

Solution of these two equations yields A and C, from which a and c can be obtained. Let the matrix $B_{ij} = \begin{bmatrix} \sum r^2 & \sum rs \\ \sum rs & \sum s^2 \end{bmatrix}$ and B_{ij}^{-1} its inverse so that $B \cdot B^{-1} = 1$.

The variance of the unknowns, A and C, is then given by the following:

$$\sigma_a^2 = B_{11}^{-1} \left(\frac{f}{n-2} \right), \qquad \sigma_c^2 = B_{22}^{-1} \left(\frac{f}{n-2} \right), \qquad \text{where f is the sum of the}$$

squares of the "residuals", i.e., the differences between D_o (derived from the observed d's) and D_c (calculated using the values of A and C). From these relationships, it is possible to determine the errors of the cell parameters, σ_a and σ_c, using the equations $\sigma_a^2 = \left(\frac{\partial a}{\partial A} \right)^2 \sigma_A^2$ and $\sigma_c^2 = \left(\frac{\partial c}{\partial C} \right)^2 \sigma_C^2$

so that $\sigma_a = \pm 1/2 \left[\dfrac{B_{11}^{-1} f}{A^3 (n-2)} \right]^{\frac{1}{2}}$ and $\sigma_c = \pm 1/2 \left[\dfrac{B_{22}^{-1} f}{C^3 (n-2)} \right]^{\frac{1}{2}}$

METHOD

1. Enter n values of hkl and d_o (observed).

2. Calculate n values of r, s, and D_o.

3. Calculate the five sums which are functions of r,s, and D_o and enter into matrix (1).

4. Solve for unknowns and inverse of matrix (using Gauss-Jordan method):

   a. Divide each term of 1st row of matrix (1) by B_{11} and place quotients in 1st row of matrix (2); divide each term of 2nd row of matrix (1) by B_{21} and place quotients in 2nd row of matrix (2)

3.

b. Rewrite 1st row of matrix (2) in 1st row of matrix (3); subtract terms of 1st row of matrix (2) from corresponding terms of 2nd row and place in 2nd row of matrix (3).

c. Divide each term in 1st row of matrix (3) by the term now in B_{12} position of matrix (3) and place quotients in 1st row of matrix (4); divide each term in 2nd row of matrix (3) by term now in B_{22} position of matrix (3) and place quotients in 2nd row of matrix (4).

d. Subtract terms of 2nd row of matrix (4) from corresponding terms of 1st row and place in 1st row of matrix (5); rewrite 2nd row of matrix (4) in 2nd row of matrix (5) and of matrix (6).

e. Divide each term in 1st row of matrix (5) by term now in B_{11} position and place quotients in 1st row of matrix (6).

5. Calculate n values of D_c (calculated), $\triangle$, and d_c.

6. Calculate f.

7. Calculate a, c, σ_a, and σ_c

ALL WORK SHOULD BE RECORDED TO AT LEAST 5 SIGNIFICANT FIGURES; RESULTS OF a, c, σ_a, and σ_c SHOULD BE GIVEN TO 3 FIGURES TO THE RIGHT OF THE DECIMAL POINT.

EXAMPLE FOR LEAST-SQUARES REFINEMENT OF CELL PARAMETERS

Substance: __Thorite__   System: __Tetragonal__

hkl	d_o	r	s	D_o	D_c	Δ	d_c
101	4.72	1	1	.044887	.044807	.00008	4.72419
200	3.55	4	0	.079249	.078988	.000361	3.55811
211	2.842	5	1	.123808	.123795	.000013	2.84216
112	2.676	2	4	.139646	.139734	-.000088	2.67516
220	2.516	8	0	.157971	.157976	-.000005	2.51596
202	2.361	4	4	.179394	.179228	.000166	2.36209
301	2.222	9	1	.202541	.202783	-.000242	2.22067
103	2.019	1	9	.245217	.245287	.00003	2.019123

$r = h^2 + k^2$ (tetragonal)
$= \frac{1}{3}(h^2 + hk + k^2)$ (hexagonal)
$s = l^2$
$D_o = 1/d_o^2$

$D_c = rA + sC$
$\Delta = D_o - D_c$
$d_c = \left(1/D_c\right)^{1/2}$

$\Sigma r^2 = \underline{208}$ $\Sigma r D_o = \underline{5.3101}$
$\Sigma rs = \underline{48}$ $\Sigma s D_o = \underline{3.8552}$
$\Sigma s^2 = \underline{116}$ $f = \Sigma \Delta^2 = \underline{2.3 \times 10^{-7}}$

(1)

Σr^2 208	Σrs 48	$\Sigma r D_o$ 5.3101	1	0
Σrs 48	Σs^2 116	$\Sigma s D_o$ 3.8552	0	1

(2)

1	.23077	.02553	.004807	0
1	2.41666	.08031	0	.02083

(3)

1	.23077	.02553	.004807	0
0	2.18589	.05478	-.004807	.02083

(4)

4.33219	1	.110629	.02083	0
0	1	.02506	-.002199	.009529

(5)

4.33219	0	.085569	.02303	-.009529
0	1	.02506	-.002199	.009529

(6)

1	0	A= .019747	$B_{11}^{-1}=$ .005318	$B_{12}^{-1}=$ .00220
0	1	C= .02506	$B_{12}^{-1}=$ -.002199	$B_{22}^{-1}=$ .009529

$a = \left(\dfrac{1}{A}\right)^{1/2}, \quad \sigma_a = \pm \dfrac{1}{2}\left[\dfrac{B_{11}^{-1}\, f}{A^3 (n-2)}\right]^{1/2}$

$c = \left(\dfrac{1}{C}\right)^{1/2}, \quad \sigma_c = \pm \dfrac{1}{2}\left[\dfrac{B_{22}^{-1}\, f}{C^3 (n-2)}\right]^{1/2}$

$a = \underline{7.11622} \pm \underline{.00257}$

$c = \underline{6.31648} \pm \underline{.00241}$

The output from the least-squares program, especially the non-linear least-squares case, shows whether the JCPDS card contains any possible errors. If no major errors were encountered, a new mineral was chosen and the least-squares procedure was repeated. When the JCPDS card contained a possible error it usually showed up as a large deviation between calculated and observed residuals of 2-theta. A deviation of about 0.3° 2-theta or greater was taken to indicate that the line in question was incorrectly indexed, not read properly on the original diffraction pattern, or that the line was very weak and its 2-theta value was hard to record accurately. When a possible error showed up in the output the next step in the procedure was to run a second computer program, commonly referred to as the "hkl generator". This program determined the Laue class from the space group number, generated all appropriate hkl values and corresponding d-values and ordered the data by decreasing d-value.

The observed d-spacing from the least-squares output, which showed a residual of 2-theta of about 0.3° or greater, was then compared to the d-values that were calculated in the hkl generator. The d-spacing from the least-squares output was matched as closely as possible to a d-value from the hkl generator and the corresponding hkl values were compared. If the hkl values differed, it was likely that the line was incorrectly indexed on the JCPDS card. This was checked by inputting the hkl from the hkl generator and observed d-spacing from the JCPDS card into the least-squares program. If the residual of 2-theta value for that line was now less than 0.3°, then it was reasonable to assume that the line in question was incorrectly indexed and that the new hkl is a better choice for that line. Unfortunately, the new hkl may yield a residual of 2-theta value that was no better or even worse

than the original or perhaps the hkl generator would show that the hkl originally used on the JCPDS card was the only hkl or at least the best hkl for that particular d-spacing. In either case, the next step was to find the mineral in the Mineralogy museum, make a powder sample and run an X-ray diffraction pattern for that mineral. The 2-theta values could then be read directly from this experimental X-ray pattern and the corresponding hkl's could be found in two easy steps. First, the d-spacings that correspond to the 2-theta values taken from the X-ray pattern must be found. This was accomplished by using a table, prepared by R.T. Tettenhorst, that lists 2-theta values from 1.00° to 179.99° and the corresponding d-spacing values in increments of 0.01°. The table is divided into two sections "Copper K-alpha" and "Copper K-alpha 1". The Copper K-alpha 1 section was used when the X-ray peaks showed resolution between K-alpha$_1$ and K-alpha$_2$, otherwise the Copper K-alpha section was used. Once the d-spacings had been found in the table, they were matched as closely as possible to the d-spacings calculated by the hkl generator and the appropriate hkl for each d-spacing was subsequently found. Finally, the hkl's and their corresponding 2-theta values were fed into the least-squares program. If the output showed that the line in question now gave a residual of 2-theta value considerably less than the original value then it was assumed that the 2-theta value was incorrectly read on the original diffraction pattern. However, if the line still gave a residual of 2-theta value of about 0.3° or greater, then perhaps it represented a weak peak that could not be read accurately, in which case no reasonable correction could be offered in this investigation.

IV.   ANALYSIS OF MINERALS

The minerals investigated in this paper include thorite, sulvanite, xenotime, zincite, nepheline, phosgenite, potassium alum, prehnite, stibnite and natrolite.   Each mineral was tested according to the procedure described in Section III.   In addition, thorite, sulvanite, xenotime and zincite were initially tested using a least-squares computer program that was found to be inferior to the least-squares program that was ultimately used.   The initial program minimized the sum of the squares of the residuals of $1/d^2$ but did not contain a non-linear least-squares procedure minimizing the sum of the squares of the residuals of theta and for this reason it was abandoned. Thorite, sulvanite, xenotime and zincite were subsequently retested with the superior program.

Thorite

The chemical formula for thorium silicate (thorite) is $ThSiO_4$; it has the same structure as zircon and is a member of the tetragonal crystal system. The diffraction work was done in 1958 using Copper K-alpha radiation with a wavelength of 1.5418A.   The non-linear least-squares output for thorite is given in Table 1, page 20.   The residuals of 2-theta show that all the lines fit very well with the possible exception of the 640 line which is 0.245° off, i.e., the difference between the calculated and observed 2-theta values is 0.245°.   The output also shows that the calculated cell dimensions of a = 7.1339A ± .0013 and c = 6.3199A ± .0017 match quite well with the cell dimensions listed on the JCPDS card, a = 7.142A and c = 6.327A.   Because the cell dimensions fit so well and because 0.245° is not considered a major error, no attempt was made in this investigation to improve the residual of 2-theta value for the 640 line.

Sulvanite

The chemical formula for copper vanadium sulfide (sulvanite) is

Cu_3VS_4 and it is a member of the cubic crystal system. The diffraction

work was done using Copper K-alpha radiation with a wavelength of 1.5418A

and with a nickel filter. The non-linear least-squares output for sulvanite

is given in Table 2, page 21. The residuals of 2-theta show that most of

the lines fit reasonably well although three lines are greater than 0.2°

(calculated minus observed 2-theta), the largest discrepancy being the 311

line at 0.275°. Even though 0.275° is not considered a major error, the hkl

generator was run for sulvanite to ascertain that none of the lines showing

0.2° or greater was incorrectly indexed. The output from the hkl generator

is given in Table 3, page 22 and shows that in each case when the d-spacings

are matched as closely as possible, the proper hkl had been chosen on the

JCPDS card. Therefore, on the basis that the proper hkl's had been used

and the close fit of the calculated cell dimension, a = 5.3951A ± .0012 with

the cell dimension listed on the JCPDS card a = 5.391A, the JCPDS card for

sulvanite contains no errors.

Xenotime

The chemical formula for yttrium, erbium phosphate (xenotime) is $(Y,Er)PO_4$

and it is a member of the tetragonal crystal system. The diffraction work was

done in 1958 using Copper K-alpha radiation with wavelengths of 1.5418A and

1.5405A and with a nickel filter. The non-linear least-squares output for

xenotime is given in Table 4, page 23. The residuals of 2-theta show that

all the lines fit very well with the 543 line showing the greatest discrepancy

at 0.186°. The calculated cell dimensions of a = 6.9026A ± .0010 and

c = 6.0360A ± .0016 also fit very well with the cell dimensions listed on the

JCPDS card a = 6.904A and c = 6.033A. On the basis that the cell dimensions
and the residuals of 2-theta fit so well (0.186° 2-theta is not considered
an error) the JCPDS card for xenotime contains no errors.

Zincite

The chemical formula for zinc oxide (zincite) is ZnO and it is a
member of the hexagonal crystal system. The diffraction work was done in
1957 using Copper radiation with a wavelength of 1.5405A and with a nickel
filter. The non-linear least-squares output for zincite is given in Table 5,
page 24. The residuals of 2-theta show that all the lines fit extremely
well except for the 202 line which shows a major error of 0.951°. In spite
of the fact that the 202 line is off by 0.951° 2-theta, the calculated cell
dimensions a = 3.2495A ± .0012 and c = 5.2050A ± .0022 fit exactly with the
cell dimensions given on the JCPDS card a = 3.249A and c = 5.205A. In order
to determine if the hkl was incorrectly picked for the 202 line, the hkl
generating program was run and the output is given in Table 6, page 25.
The observed d-spacing for the line in error is 1.2250A and when this was
matched as closely as possible to the d-values given by the hkl generator
it showed that 202 was the correct hkl for that particular d-spacing. Since
the hkl was properly picked for the line in error, the next step was to make
a powder sample of zincite and run an X-ray diffraction pattern. Once the
diffraction pattern was obtained the peaks were assigned 2-theta values and
the corresponding d-spacings were found. At this point it should be noted
that a d-spacing of 1.237A was obtained from the diffraction pattern for the
line in error which was considerably different then the d-value of 1.2250A which
was listed on the JCPDS card. To ascertain whether 1.237A was the correct d-spacing
for the 202 line, 13 hkl's and corresponding 2-theta values from the diffraction

pattern were input into the least-squares program and the non-linear
output from this program is given in Table 7, page 25. The residuals
of 2-theta show that the line that originally gave a discrepancy of 0.951°
now gives a discrepancy of only 0.013°. The conclusion was that the d-value
of 1.237A was the correct value and that the d-value given on the JCPDS
card of 1.2250A was incorrect.

NOTE: The hkl's and observed d-values used in this analysis were
taken from the JCPDS card file. By coincidence zincite was
looked up in the recent JCPDS reference book of minerals which
is an update of all JCPDS cards. In the JCPDS reference book
the d-spacing that corresponds to the 202 line is given as
1.235A and not 1.225A.

5-0 664

d	2.48	2.82	2.60	2.81t	ZnO				
I/I₁	100	71	56	71	Zinc Oxide		Zincite		

Rad. Cu   λ 1.5405   Filter Ni
Dia.   Cut off   Coll.
I/I₁   Counter   d corr. abs.?
Ref. _n_ and Fuyat, NBS Circular 539, Vol. 2, 25 (1953)

Sys. Hexagonal   S.G. C⁴₆ᵥ - C6mc
a₀ 3.249 b₀   c₀ 5.205 A   C 1.60
α   β   γ   Z 2
Ref. Ibid.

2V   Dₓ 5.680 mp   Color
Ref. Ibid.

Sample from New Jersey Zinc Co.
Spect. analysis shows < 0.001% each of Mg, Si and Ca.
X-ray pattern at 26°C.

To replace 1-1136, 3-0752, 3-0888, 3-0891.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
2.816	71	100	0.9069	12	213
2.602	56	002	.8826	6	302
2.476	100	101	.8675	1	006
1.911	29	102	.8369	6	205
1.626	40	110	.8290	2	106
1.477	35	103	.8237	2	214
1.407	6	200	.8125	5	220
1.379	28	112			
1.359	14	201			
1.301	3	004			
1.225	5	202			
1.1812	3	104			
1.0929	10	203			
1.0639	4	210			
1.0422	10	211			
1.0158	5	114			
0.9848	4	212			
.9764	7	105			
.9555	1	204			
.9382	4	300			

S-664

d	2.48	2.82	2.60	2.82	ZnO				
I/I₁	100	71	56	71	Zinc Oxide		(Zincite)		

Rad. CuKα₁ λ 1.5405   Filter Ni   Dia.
Cut off   I/I₁   Diffractometer   I/I cor.
Ref. Swanson and Fuyat, NBS Circular 539, Vol. 2, 25 (1953)

Sys. Hexagonal   S.G. P6₃mc (186)
a₀ 3.249 b₀   c₀ 5.205 A   C 1.60
α   β   γ   Z 2   Dx 5.680
Ref. Ibid.

2V   D   mp   Color Colorless
Ref. Ibid.

Sample from New Jersey Zinc Co.
Spect. analysis: <0.001% each of Mg, Si and Ca.
X-ray pattern at 26°C.
Merck Index, 8th Ed., p. 1129.
Wurtzite group.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
2.816	71	100	.9069	12	213
2.602	56	002	.8826	6	302
2.476	100	101	.8675	1	006
1.911	29	102	.8369	6	205
1.626	40	110	.8290	2	106
1.477	35	103	.8237	2	214
1.407	6	200	.8125	5	220
1.379	28	112			
1.359	14	201			
1.301	3	004			
1.238	5	202			
1.1812	3	104			
1.0929	10	203			
1.0639	4	210			
1.0422	10	211			
1.0158	5	114			
0.9848	4	212			
.9764	7	105			
.9555	1	204			
.9382	4	300			

The final conclusion was that a typographical error was made on the original
card that was in the JCPDS file.

11.

Nepheline

The chemical formula for potassium sodium aluminum silicate (nepheline) is $(Na,K)AlSiO_4$ and it is a member of the hexagonal crystal system. The diffraction work was done in 1957 using Copper K-alpha 1 radiation with a wavelength of 1.54050A and with a nickel filter. The non-linear least-squares output for nepheline is given in Table 8, page 26. The residuals of 2-theta show that all the lines fit extremely well with the largest discrepancy being a mere 0.069°. The calculated cell dimensions a = 10.0579A ± .0013 and c = 8.4169A ± .0017 fit equally well with the cell dimensions listed on the JCPDS card a = 10.060A and c = 8.417A. Obviously, the JCPDS card for nepheline contains no errors.

Phosgenite

The chemical formula for lead carbonate chloride (phosgenite) is $Pb_2(CO_3)Cl_2$ and it is a member of the tetragonal crystal system. The diffraction work was done in 1957 using Cobalt K-alpha radiation with a wavelength of 1.7902A and with an iron filter. The non-linear least-squares output for phosgenite is given in Table 9, page 27. The residuals of 2-theta show that all the lines fit very well with the largest discrepancy being 0.171° for the 401 line. A value of 0.171° 2-theta is not considered a large enough error to warrant further investigation. In addition, the calculated cell dimensions, a = 8.1122A ± .0023 and c = 8.8258A ± .0061 fit quite well with the cell dimensions given on the JCPDS card, a = 8.111A and c = 8.824A. Therefore, the JCPDS card for phosgenite contains no errors.

Potassium Alum

The chemical formula for potassium aluminum sulfate hydrate (potassium alum) is $KAl(SO_4)_2 \cdot 12H_2O$ and it is a member of the isometric crystal system. The diffraction work for potassium alum was done in 1956 using Copper K-alpha 1 radiation with a wavelength of 1.5405A and with a nickel filter. The non-linear least-squares output for potassium alum is given in Table 10, page 28. The residuals of 2-theta show that all the lines fit extremely well except for the 752 line which shows a major discrepancy of 0.464°. In order to find out whether 752 is the correct hkl for the observed d-spacing the hkl generating program was run for potassium alum and the output from that program is given in Table 11, page 29. When the observed d-spacing of 1.3853A was matched as closely as possible to the d-values given by the hkl generator it was observed that there are two hkl's that could be equally correct for a d-spacing of 1.3853A. The acceptable hkl's are 832 and 654, both have $h^2+k^2+l^2$ equal to 77. To check this an hkl of 832 was input into the least-squares program in place of 752 and the non-linear least-squares output from this program is given in Table 12, page 30. The residuals of 2-theta show that using 832 in place of 752 reduces the discrepancy from 0.464° to 0.019°. It would appear that 832 and 654 are the correct hkl values for a d-spacing of 1.3853A.

As a final check, a powder sample of potassium alum was made and an X-ray diffraction pattern was run. The peaks were assigned 2-theta values and corresponding d-values and hkl values were subsequently found. Finally 53 hkl and corresponding 2-theta values were input into the least-squares program. The non-linear least-squares output from that program is given in Table 13, page 31. The residuals of 2-theta show that all the lines fit

13.

extremely well including the 832 line which shows a discrepancy of only 0.011°. This was taken as conclusive evidence that the JCPDS card for potassium alum was in error and that the hkl value of 752 should be replaced by either an hkl value of 832 or 654.

Prehnite

The chemical formula for calcium aluminum silicate hydroxide (prehnite) is $Ca_2Al_2Si_3O_{10}(OH)_2$ and it is a member of the orthorhombic crystal system. The diffraction work for prehnite was done in 1975 using Cobalt K-alpha radiation with a wavelength of 1.7902A. The non-linear least-squares output for prehnite is given in Table 14, page 32. The residuals of 2-theta show that all the lines fit exceptionally well with the largest discrepancy being a slight 0.042°. The calculated cell dimensions a = 4.6368A ± .0013, b = 5.4876A ± .0016 and c = 18.4863A ± .0070 fit equally well with the cell dimensions listed on the JCPDS card a = 4.642A, b = 5.487A and c = 18.490A. The JCPDS card for prehnite contains no errors.

Stibnite

The chemical formula for antimony sulfide (stibnite) is Sb_2S_3 and it is a member of the orthorhombic crystal system. The diffraction work for stibnite was completed in 1955 using Copper K-alpha 1 radiation with a wavelength of 1.5405A and with a nickel filter. The non-linear least-squares output for stibnite is given in Table 15, page 33. The residuals of 2-theta show that all the lines fit exceptionally well indicating that no errors are present in the JCPDS card for stibnite. The calculated cell dimensions also fit extremely well with the cell dimensions given on the JCPDS card and this reinforces the probability that no errors occurred in the JCPDS card for stibnite.

14.

JCPDS cell dimensions	Calculated cell dimensions
a = 11.229A	a = 11.2284A ± .0018
b = 11.310	b = 11.3106 ± .0020
c = 3.8389	c = 3.8393 ± .0007

Natrolite

The chemical formula for the zeolite mineral sodium aluminum silicate hydrate (natrolite) is $Na_2Al_2Si_3O_{10} \cdot 2H_2O$ and it is a member of the orthorhombic crystal system. The diffraction work for natrolite was done in 1966 using Copper K-alpha radiation with a wavelength of 1.5418A and with a nickel filter. The nonlinear least-squares output for natrolite is given in Table 16, page 34. The residuals of 2-theta show that although many of the lines fit very well there are also numerous major discrepancies including one line of 3.074°. The hkl generating program was run because of the apparent indexing problems and the output from that program is given in Table 17, page 35. When the observed d-spacings of all the bad lines were matched as closely as possible to the d-values given by the hkl generator, the following hkl's appear to be better choices:

new hkl	Corresponding d-value	hkl replaced
371	2.2880	800
153	1.9070	911
2100	1.8240	822
10$\overline{2}$0	1.7970	533
$\overline{4100}$	1.7240	951
$\overline{791}$	1.5740	10$\underline{22}$
410$\overline{2}$	1.5270	424
41$\overline{20}$	1.4680	444
$\overline{953}$	1.3850	464
412$\underline{2}$	1.3420	084
$\underline{1280}$	1.2740	844
$\underline{1262}$	1.2630	355
$\overline{515}$	1.2370	155
11$\underline{51}$	1.2180	355

The new hkl values were input into the least-squares program and the non-linear portion of the least-squares analysis is given in Table 18, page 36. The residuals of 2-theta now show that all the lines fit very well with the exception of the 153 line. The hkl value of 153 replaced the hkl value of 911 but in doing so increased the discrepancy from 0.408° 2-theta to 0.657° 2-theta indicating that 153 is not a better choice for that line. However, taking the observed d-spacing for the 911 line (1.9070A) and matching it as closely as possible to the d-values given by the hkl generator (see Table 17, page 35), it can be seen that the closest matches are the 911 (1.9310A) and the 153 (1.8824A). Since neither the 911 nor the 153 fit the observed d-spacing of 1.9070A very closely, it may indicate that particular peak on the diffraction pattern was very weak (the intensity for that peak given on the JCPDS card was 4, on a scale of 100 being equal to the intensity of the strongest peak) and an accurate 2-theta value was unobtainable. Therefore, for the purpose of this investigation, the 911 line (on the basis that it gave a smaller discrepancy) was taken as the correct hkl for the observed d-spacing of 1.9070A. All the other hkl values, which were tried as replacements for hkl values listed on the JCPDS card, were considered the correct hkl values for those particular d-spacings. It is also important to note that the calculated cell dimensions fit the cell dimensions given on the JCPDS card much closer when the new hkl values were input into the least-squares program.

JCPDS dimensions	Cell dimensions calculated with new hkl values	Cell dimensions calculated with original hkl values
a = 18.284A	18.2533A ± .0128	18.1897A ± .0526
b = 18.620	18.6187 ± .0108	18.6078 ± .0697
c = 6.592	6.5932 ± .0041	6.6141 ± .0132

As a final note, had time permitted, an experimental diffraction pattern would have been run for natrolite in order to ascertain the true nature of the discrepancy of the 911 line.

V. CONCLUSIONS

On the basis of my results in this investigation, the minerals which were analyzed can be classified into one of two categories based on the amount of discrepancy between the calculated and observed 2-theta values. The first category included the minerals analyzed which showed a maximum discrepancy of 0.280° 2-theta, and since a discrepancy of 0.300° 2-theta was taken as the minimum value which constituted a major error, the JCPDS cards for all the minerals in category 1 (for the purposes of this investigation) contained no errors. The minerals in category 1 are thorite, sulvanite, xenotime, nepheline, phosgenite, prehnite and stibnite. The second category contained the minerals which were analyzed that had at least one line which showed a discrepancy of 0.300° 2-theta or greater. The minerals in category 2 include zincite, potassium alum and natrolite. The JCPDS card for each of these minerals contained at least one major error.

The major discrepancy in zincite was found to be a typographical error in the d-value given on the original JCPDS card for the 202 line.

The major discrepancy in potassium alum was due to an indexing problem with the 752 line. The correct hkl value for a d-spacing of 1.3853A was found to be either 832 or 654, not 752 as given on the JCPDS for potassium alum.

There were numerous major discrepancies in natrolite and at least all but one were due to improper indexing of hkl values. All of the improperly indexed hkl values were eventually corrected with the possible exception of the 153 line, which may represent more than an indexing problem. However, due to lack of time no further investigation of the 153 line was possible.

VI. SUGGESTIONS FOR FURTHER RESEARCH

A further investigation of natrolite, specifically the running of an experimental X-ray diffraction pattern, would likely give the reason for the large discrepancy in the 911 line, which remained unanswered in this investigation. Also, due to the number of discrepancies between calculated and observed 2-theta encountered in this investigation (three out of ten minerals studied contained major errors), it is highly probable that further research into the JCPDS card file would yield numerous errors. Many of those errors could be easily corrected using methods identical to those described in this investigation.

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 7.1339A   C= 6.3199A
+/- 0.0013   0.0017

SSR (DEGREES 2-THETA):   1.8443D-01
RMS RESIDUALS (DEGREES 2-THETA):   6.8768D-02

H K L	2-THETA	D(OBS)	D(CALC)	$1/D^{**2}$	RESIDUALS 2-THETA
1 0 1	18.785	4.7200	4.7306	0.00020	0.042
2 0 0	25.063	3.5500	3.5669	0.00075	0.121
2 1 1	31.452	2.8420	2.8480	0.00052	0.068
1 1 2	33.458	2.6760	2.6779	0.00020	0.025
2 2 0	35.655	2.5160	2.5222	0.00078	0.091
2 0 2	38.083	2.3610	2.3653	0.00065	0.072
3 0 1	40.566	2.2220	2.2256	0.00066	0.069
1 0 3	44.855	2.0190	2.0204	0.00034	0.032
3 2 1	48.238	1.8850	1.8882	0.00095	0.087
3 1 2	49.669	1.8340	1.8360	0.00066	0.059
4 0 0	51.222	1.7820	1.7835	0.00052	0.045
2 1 3	52.005	1.7570	1.7580	0.00035	0.031
4 1 1	55.042	1.6670	1.6688	0.00078	0.065
4 2 0	57.794	1.5940	1.5952	0.00058	0.047
0 0 4	58.436	1.5780	1.5800	0.00100	0.080
3 3 2	62.538	1.4840	1.4844	0.00024	0.018
2 0 4	64.476	1.4440	1.4446	0.00040	0.030
5 0 1	67.196	1.3920	1.3917	-0.00019	-0.014
2 2 4	70.297	1.3380	1.3390	0.00080	0.058
5 2 1	72.933	1.2960	1.2965	0.00050	0.036
5 1 2	73.995	1.2800	1.2793	-0.00068	-0.048
4 4 0	75.232	1.2620	1.2611	-0.00090	-0.063
1 0 5	76.442	1.2450	1.2446	-0.00042	-0.029
6 0 0	80.758	1.1890	1.1890	-0.00003	-0.002
4 0 4	81.336	1.1820	1.1826	0.00077	0.053
2 1 5	81.924	1.1750	1.1751	0.00014	0.010
6 1 1	83.925	1.1520	1.1531	0.00145	0.099
5 3 2	84.923	1.1410	1.1409	-0.00011	-0.008
6 2 0	86.137	1.1280	1.1280	-0.00005	-0.004
4 2 4	86.615	1.1230	1.1225	-0.00064	-0.044
5 4 1	89.203	1.0970	1.0972	0.00031	0.021
3 2 5	92.650	1.0650	1.0652	0.00029	0.020
6 3 1	94.496	1.0490	1.0487	-0.00051	-0.034
1 1 6	96.558	1.0320	1.0311	-0.00168	-0.115
6 1 3	97.567	1.0240	1.0247	0.00131	0.090
4 1 5	97.953	1.0210	1.0206	-0.00068	-0.046
7 0 1	99.936	1.0060	1.0061	0.00024	0.017
6 4 0	102.024	0.9910	0.9893	-0.00353	-0.245
4 4 4	102.744	0.9860	0.9856	-0.00078	-0.054

TABLE I

Non-Linear Least-Squares Output for Thorite

SSR = Sum of the Squares of the Residuals
RMS = Root Mean Squares

20.

A = 5.3951A
+/- 0.0012

SSR (DEGREES 2-THETA): 3.3316D-01
RMS RESIDUALS (DEGREES 2-THETA): 1.2596D-01

H K L	2-THETA	D(OBS)	D(CALC)	$1/D**2$	RESIDUALS 2-THETA
1 0 0	16.402	5.4000	5.3951	-0.00006	-0.015
1 1 1	28.587	3.1200	3.1149	-0.00034	-0.048
2 0 0	33.152	2.7000	2.6976	-0.00025	-0.031
2 1 0	37.441	2.4000	2.4128	0.00183	0.206
2 2 0	47.568	1.9100	1.9075	-0.00073	-0.067
3 0 0	50.553	1.8040	1.7984	-0.00192	-0.169
3 1 1	56.251	1.6340	1.6267	-0.00337	-0.275
3 2 0	61.752	1.5010	1.4963	-0.00277	-0.213
4 0 0	69.640	1.3490	1.3488	-0.00017	-0.013
4 1 0	72.158	1.3080	1.3085	0.00046	0.033
3 3 1	77.101	1.2360	1.2377	0.00183	0.128
4 2 0	79.391	1.2060	1.2064	0.00045	0.031
4 2 1	81.755	1.1770	1.1773	0.00039	0.027
4 2 2	88.793	1.1010	1.1013	0.00042	0.029
5 1 1	95.940	1.0370	1.0383	0.00232	0.159
5 2 0	100.482	1.0020	1.0019	-0.00029	-0.020
4 4 0	107.854	0.9530	0.9537	0.00170	0.121
4 4 1	110.059	0.9400	0.9392	-0.00199	-0.144
5 3 1	115.458	0.9110	0.9119	0.00250	0.188
6 2 0	129.117	0.8530	0.8530	0.00015	0.013
6 2 1	132.054	0.8430	0.8426	-0.00140	-0.128

TABLE 2

Non-Linear Least-Squares Output for Sulvanite

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	0	0	5.3910												
1	1	0	3.8120												
1	1	1	3.1125												
2	0	0	2.6955												
2	1	0	2.4109												
2	1	1	2.2009												
2	2	0	1.9060												
3	0	0	1.7970												
2	2	1	1.7970												
3	1	0	1.7048												
3	1	1	1.6254												
2	2	2	1.5562												
3	2	0	1.4952												
3	2	1	1.4408												
4	0	0	1.3477												
4	1	0	1.3075												
3	2	2	1.3075												
3	3	0	1.2707												
4	1	1	1.2707												
3	3	1	1.2368												
4	2	0	1.2055												
4	2	1	1.1764												
3	3	2	1.1494												
4	2	2	1.1004												
5	0	0	1.0782												
4	3	0	1.0782												
5	1	0	1.0573												
4	3	1	1.0573												
5	1	1	1.0375												
3	3	3	1.0375												
5	2	0	1.0011												
4	3	2	1.0011												
5	2	1	0.9843												
4	4	0	0.9530												
4	4	1	0.9385												
5	2	2	0.9385												
5	3	0	0.9245												
4	3	3	0.9245												
5	3	1	0.9112												
6	0	0	0.8985												
4	4	2	0.8985												
6	1	0	0.8863												
6	1	1	0.8745												
5	3	2	0.8745												
6	2	0	0.8524												
5	4	0	0.8419												
6	2	1	0.8419												
4	4	3	0.8419												

TABLE 3

HKL Generating Output for Sulvanite

22.

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 6.9026A   C = 6.3360A
+/- 0.0010   0.0016

SSR (DEGREES 2-THETA):   2.0402D-01
RMS RESIDUALS (DEGREES 2-THETA):   7.2327D-02

| | | | | | | | RESIDUALS | |
H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	2-THETA
1	0	1	19.493	4.5500	4.5438	-0.00013	-0.027
2	0	0	25.802	3.4500	3.4513	0.00006	0.010
2	1	1	32.533	2.7500	2.7484	-0.00016	-0.020
1	1	2	35.022	2.5600	2.5669	0.00082	0.098
2	2	0	36.805	2.4400	2.4404	0.00006	0.007
2	0	2	39.672	2.2700	2.2719	0.00032	0.035
3	0	1	41.988	2.1500	2.1500	-0.00001	-0.001
1	0	3	47.071	1.9290	1.9316	0.00073	0.068
3	2	1	49.960	1.8240	1.8248	0.00028	0.025
3	1	2	51.657	1.7680	1.7687	0.00025	0.021
4	0	0	53.044	1.7250	1.7256	0.00025	0.022
2	1	3	54.440	1.6840	1.6856	0.00066	0.055
4	1	1	56.935	1.6160	1.6132	-0.00132	-0.107
4	2	0	59.895	1.5430	1.5435	0.00025	0.020
0	0	4	61.209	1.5130	1.5090	-0.00231	-0.179
3	3	2	65.082	1.4320	1.4321	0.00008	0.006
2	0	4	67.692	1.3830	1.3826	-0.00028	-0.021
5	0	1	69.818	1.3460	1.3458	-0.00019	-0.014
2	2	4	73.793	1.2830	1.2835	0.00044	0.031
5	1	2	77.175	1.2350	1.2352	0.00016	0.011
4	4	0	78.227	1.2210	1.2202	-0.00086	-0.060
6	0	0	84.105	1.1500	1.1504	0.00057	0.039
5	0	3	85.292	1.1370	1.1383	0.00181	0.123
2	1	5	86.519	1.1240	1.1243	0.00041	0.028
6	1	1	87.491	1.1140	1.1152	0.00179	0.122
5	3	2	88.691	1.1020	1.1020	0.00006	0.004
6	2	0	89.826	1.0910	1.0914	0.00061	0.042
5	2	3	90.996	1.0800	1.0810	0.00166	0.113
3	2	5	97.824	1.0220	1.0211	-0.00161	-0.111
6	3	1	98.866	1.0140	1.0143	0.00066	0.046
6	1	3	102.310	0.9890	0.9884	-0.00122	-0.085
1	1	6	102.905	0.9849	0.9853	0.00083	0.058
6	4	0	107.102	0.9576	0.9572	-0.00087	-0.062
5	4	3	108.503	0.9491	0.9502	0.00260	0.186
5	5	2	112.005	0.9291	0.9288	-0.00075	-0.055
3	1	6	114.904	0.9138	0.9136	-0.00042	-0.031
6	2	4	121.096	0.8946	0.8843	-0.00075	-0.060
7	3	2	125.102	0.8680	0.8681	0.00017	0.014
8	0	0	126.315	0.8633	0.8628	-0.00148	-0.125

TABLE 4

Non-Linear Least-Squares Output for Xenotime

23.

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 3.2495A   C= 5.2050A
+/- 0.0012   0.0022

SSQ (DEGREES 2-THETA):   9.2328D-01
RMS RESIDUALS (DEGREES 2-THETA):   1.8492D-01

H	K	L	2-THETA	D(OBS)	D(CALC)	1/D*2	RESIDUALS 2-THETA
1	0	0	31.750	2.8160	2.8141	-0.00017	-0.022
0	0	2	34.430	2.6020	2.6025	0.00005	0.007
1	0	1	36.251	2.4760	2.4755	-0.00007	-0.008
1	0	2	47.541	1.9110	1.9107	-0.00009	-0.009
1	1	0	56.553	1.6260	1.6247	-0.00059	-0.048
1	0	3	62.868	1.4770	1.4769	-0.00009	-0.007
2	0	0	66.386	1.4070	1.4071	0.00004	0.003
1	1	2	67.915	1.3790	1.3782	-0.00061	-0.045
2	0	1	69.055	1.3590	1.3583	-0.00055	-0.040
0	0	4	72.608	1.3010	1.3012	0.00022	0.015
2	0	2	77.923	1.2250	1.2377	0.01365	0.951
1	0	4	81.463	1.1312	1.1811	-0.00014	-0.009
2	0	3	99.627	1.0929	1.0928	-0.00008	-0.006
2	1	0	92.774	1.0639	1.0636	-0.00043	-0.030
2	1	1	95.308	1.0422	1.0421	-0.00017	-0.012
1	1	4	98.629	1.0158	1.0157	-0.00028	-0.019
2	1	2	102.919	0.9848	0.9846	-0.00046	-0.032
1	0	5	104.165	0.9764	0.9763	-0.00014	-0.010
2	0	4	107.444	0.9555	0.9553	-0.00037	-0.026
3	0	0	110.374	0.9382	0.9380	-0.00038	-0.028
2	1	3	116.283	0.9069	0.9068	-0.00027	-0.021
3	0	2	121.557	0.8826	0.8825	-0.00039	-0.031
0	0	6	125.229	0.8675	0.8675	-0.00002	-0.002
2	0	5	133.969	0.8369	0.8369	-0.00014	-0.013
1	0	6	136.611	0.8290	0.8290	-0.00001	-0.001
2	1	4	139.504	0.8237	0.8235	-0.00063	-0.064
2	2	0	142.897	0.8125	0.8124	-0.00049	-0.056

TABLE 5

Non-Linear Least-Squares Output for Zincite

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	0	0	2.8137												
0	0	2	2.6025												
1	0	1	2.4752												
1	0	2	1.9106												
1	1	0	1.6245												
1	0	3	1.4768												
2	0	0	1.4069												
1	1	2	1.3781												
2	0	1	1.3581												
0	0	4	1.3012												
2	0	2	1.2376												
1	0	4	1.1811												
2	0	3	1.0928												
2	1	0	1.0635												
2	1	1	1.0420												
1	1	4	1.0156												
2	1	2	0.9845												
1	0	5	0.9763												
2	0	4	0.9553												
3	0	0	0.9379												
3	0	1	0.9230												
2	1	3	0.9067												
3	0	2	0.8824												
0	0	6	0.8675												
2	0	5	0.8368												
1	0	6	0.8290												
3	0	3	0.8251												
2	1	4	0.8235												
2	2	0	0.8123												

TABLE 6

HKL Generating Output for Zincite

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 3.2483A   C = 5.2053A
+/- 0.0007   0.0012

SSR (DEGREES 2-THETA):   2.2661D-02
RMS RESIDUALS (DEGREES 2-THETA):   4.1751D-02

H K L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA
1 0 0	31.840	2.8082	2.8131	0.00044	0.057
0 0 2	34.460	2.6005	2.6026	0.00025	0.030
1 0 1	36.370	2.4682	2.4748	0.00088	0.101
1 0 2	47.560	1.9103	1.9104	0.00004	0.003
1 1 0	56.630	1.6240	1.6242	0.00008	0.007
1 0 3	62.950	1.4753	1.4768	0.00093	0.071
1 1 2	68.000	1.3775	1.3779	0.00029	0.022
2 0 2	77.010	1.2372	1.2374	0.00019	0.013
2 0 3	89.650	1.0927	1.0926	-0.00007	-0.005
2 1 1	95.344	1.0419	1.0417	-0.00028	-0.019
1 0 5	104.150	0.9765	0.9763	-0.00034	-0.024
2 1 3	116.310	0.9068	0.9066	-0.00051	-0.039
2 0 5	134.000	0.8368	0.8368	-0.00006	-0.005

TABLE 7

Non-Linear Least-Squares Output for Zincite Using Experimental 2-Theta Values

25.

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 10.0579A   C = 8.4169A

+/- 0.0013   0.0017

SSR (DEGREES 2-THETA):   1.945RD-02
RMS RESIDUALS (DEGREES 2-THETA): 2.3249D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA
1	1	0	17.618	5.0300	5.0290	-0.00002	-0.004
2	0	0	20.399	4.3500	4.3552	0.00013	0.025
1	1	1	20.542	4.3200	4.3171	-0.00007	-0.014
0	0	2	21.085	4.2100	4.2085	-0.00004	-0.008
2	0	1	22.962	3.9700	3.9681	-0.00007	-0.012
1	0	2	23.454	3.7890	3.7894	0.00001	0.002
2	1	0	27.047	3.2940	3.2922	-0.00010	-0.015
2	1	1	29.111	3.0550	3.0660	0.00007	0.010
2	0	2	29.484	3.0270	3.0264	-0.00004	-0.006
3	0	0	30.753	2.9550	2.9035	-0.00013	-0.017
1	0	3	33.536	2.6700	2.6705	0.00006	0.007
2	1	2	34.562	2.5930	2.5930	0.00001	0.001
2	2	0	35.670	2.5150	2.5145	-0.00007	-0.008
3	1	0	37.250	2.4150	2.4158	0.00012	0.013
3	0	2	37.603	2.3900	2.3899	-0.00002	-0.002
2	0	3	38.116	2.3590	2.3586	-0.00006	-0.007
3	1	1	38.749	2.3220	2.3221	0.00001	0.001
4	0	0	41.437	2.1772	2.1776	0.00006	0.006
2	2	2	41.821	2.1582	2.1585	0.00007	0.007
2	1	3	42.297	2.1350	2.1354	0.00008	0.008
0	0	4	42.938	2.1045	2.1042	-0.00008	-0.008
3	1	2	43.210	2.0920	2.0952	0.00069	0.069
3	2	0	45.329	1.9990	1.9983	-0.00017	-0.017
3	2	1	46.661	1.9450	1.9443	-0.00020	-0.019
1	1	4	46.814	1.9390	1.9417	0.00059	0.055
4	1	0	47.834	1.9050	1.9008	0.00022	0.020
2	0	4	47.968	1.8950	1.8947	-0.00010	-0.009
4	1	1	49.135	1.8527	1.8541	0.00043	0.039
3	2	2	50.532	1.8047	1.8051	0.00015	0.013
4	0	3	53.210	1.7200	1.7202	0.00010	0.008
3	0	4	53.712	1.7051	1.7038	-0.00051	-0.043
3	3	1	55.813	1.6453	1.6440	-0.00080	-0.065
3	2	3	56.492	1.6276	1.6277	0.00003	0.002
5	0	2	57.190	1.6094	1.6096	0.00011	0.009
4	1	3	58.599	1.5740	1.5736	-0.00019	-0.015
2	0	5	58.743	1.5705	1.5702	-0.00017	-0.013

TABLE 8

Non-Linear Least-Squares Output for Nepheline

26.

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 8.1122A   C = 8.8258A
+/- 0.0029   0.0072

SSF (DEGREES 2-THETA):   2.1543D-01
RMS RESIDUALS (DEGREES 2-THETA):   7.9600D-02

| | | | | | | | RESIDUALS | |
H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	2-THETA
1	1	0	15.506	5.7100	5.7362	0.00028	0.071
0	0	2	20.165	4.4000	4.4129	0.00030	0.059
2	0	0	21.983	4.0400	4.0561	0.00049	0.088
2	1	0	24.640	3.6100	3.6279	0.00075	0.123
1	1	2	25.427	3.5000	3.4976	-0.00011	-0.018
2	0	2	29.858	2.9900	2.9863	-0.00028	-0.038
2	2	0	31.249	2.8600	2.8681	0.00069	0.090
2	1	2	32.054	2.7900	2.8024	0.00114	0.146
3	1	0	35.022	2.5600	2.5653	0.00063	0.075
2	2	2	37.441	2.4000	2.4048	0.00069	0.078
2	1	3	39.491	2.2800	2.2850	0.00085	0.091
3	1	2	40.796	2.2100	2.2178	0.00144	0.150
4	0	0	44.599	2.0300	2.0281	-0.00047	-0.045
4	0	1	46.034	1.9700	1.9765	0.00170	0.161
2	0	4	46.788	1.9400	1.9382	-0.00049	-0.045
3	3	0	47.568	1.9100	1.9121	0.00059	0.055
2	1	4	48.184	1.8870	1.8852	-0.00055	-0.050
4	0	2	49.468	1.8410	1.8428	0.00056	0.050
4	2	0	50.078	1.8200	1.8139	-0.00202	-0.179
4	1	2	50.673	1.8000	1.7970	-0.00104	-0.091
3	3	2	52.005	1.7575	1.7545	-0.00094	-0.081
3	1	4	54.864	1.6720	1.6728	0.00034	0.028
4	1	3	56.177	1.6360	1.6355	-0.00025	-0.020
2	1	5	58.033	1.5880	1.5872	-0.00038	-0.030
5	2	0	61.434	1.5080	1.5064	-0.00094	-0.072
5	1	2	62.027	1.4950	1.4966	0.00098	0.076
4	1	4	63.250	1.4690	1.4685	-0.00033	-0.025
3	3	4	64.426	1.4450	1.4450	-0.00001	-0.001
5	2	2	65.390	1.4260	1.4256	-0.00026	-0.019
4	2	4	66.654	1.4020	1.4012	-0.00057	-0.042
5	3	0	67.141	1.3930	1.3912	-0.00131	-0.097
4	4	2	68.766	1.3640	1.3638	-0.00012	-0.009
6	0	0	69.463	1.3520	1.3520	0.00003	0.002
5	3	2	70.967	1.3270	1.3269	-0.00013	-0.009

TABLE 9

Non-Linear Least-Squares Output for Phosgenite

27.

+/- 0.0016

H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	RESIDUALS 2-THETA
1	1	1	12.599	7.0200	7.0212	0.00001	0.002
2	1	0	16.280	5.4400	5.4386	-0.00002	-0.004
2	1	1	17.868	4.9600	4.9648	0.00008	0.017
2	2	0	20.649	4.2995	4.2996	0.00004	0.008
2	2	1	21.912	4.0530	4.0537	0.00002	0.004
3	1	1	24.251	3.6670	3.6667	-0.00001	-0.002
3	2	1	27.420	3.2505	3.2502	0.00001	0.002
4	0	0	29.365	3.0390	3.0403	0.00009	0.013
4	1	0	30.272	2.9500	2.9495	-0.00004	-0.005
4	1	1	31.182	2.8660	2.8664	0.00003	0.004
3	3	1	32.065	2.7890	2.7899	0.00009	0.011
4	2	0	32.914	2.7190	2.7193	0.00003	0.004
4	2	1	33.744	2.6540	2.6538	-0.00002	-0.003
3	3	2	34.562	2.5930	2.5928	-0.00003	-0.003
4	2	2	36.160	2.4820	2.4824	0.00005	0.006
4	3	1	37.685	2.3850	2.3850	-0.00000	-0.060
5	1	1	38.438	2.3400	2.3404	0.00006	0.007
4	3	2	39.874	2.2590	2.2583	-0.00013	-0.014
5	2	1	40.605	2.2200	2.2203	0.00006	0.006
4	4	0	41.988	2.1500	2.1498	-0.00004	-0.004
5	2	2	42.653	2.1130	2.1170	-0.00022	-0.022
4	3	3	43.362	2.0850	2.0856	0.00014	0.013
5	3	1	44.028	2.0550	2.0556	0.00014	0.014
6	0	0	44.669	2.0270	2.0269	-0.00004	-0.003
6	1	0	45.305	2.0000	1.9993	-0.00018	-0.017
6	1	1	45.960	1.9730	1.9728	-0.00005	-0.005
6	2	0	47.201	1.9240	1.9228	-0.00033	-0.030
6	2	1	47.861	1.8990	1.8992	0.00007	0.007
5	4	1	48.457	1.8770	1.8765	-0.00015	-0.014
5	3	3	49.070	1.8550	1.8546	-0.00014	-0.013
6	2	2	49.698	1.8330	1.8334	0.00012	0.010
6	3	0	50.314	1.8120	1.8129	0.00029	0.026
6	3	1	50.885	1.7930	1.7931	0.00002	0.002
5	4	3	53.243	1.7190	1.7198	0.00033	0.028
7	1	1	53.818	1.7020	1.7029	0.00036	0.031
6	4	0	54.370	1.6860	1.6864	0.00018	0.015
6	4	1	54.935	1.6700	1.6705	0.00020	0.016
7	2	1	55.512	1.6540	1.6549	0.00041	0.033
6	4	2	56.629	1.6240	1.6251	0.00051	0.042
7	2	2	57.167	1.6100	1.6108	0.00000	0.030
7	3	1	58.274	1.5820	1.5832	0.00063	0.050
6	5	0	59.324	1.5565	1.5571	0.00030	0.024
7	3	2	59.853	1.5440	1.5445	0.00025	0.020
8	1	1	61.962	1.4964	1.4969	0.00032	0.024
8	2	0	62.992	1.4744	1.4748	0.00022	0.017
8	2	1	63.491	1.4640	1.4640	0.00002	0.001
8	2	2	65.042	1.4328	1.4332	0.00027	0.020
8	3	1	66.021	1.4139	1.4137	-0.00014	-0.011
7	5	1	66.531	1.4043	1.4042	-0.00004	-0.003
6	6	2	67.022	1.3952	1.3950	-0.00017	-0.012
7	5	2	67.565	1.3853	1.3770	-0.00632	-0.464
8	4	0	69.043	1.3592	1.3597	0.00036	0.026
8	4	1	69.522	1.3510	1.3512	0.00019	0.014
9	1	1	70.521	1.3343	1.3349	0.00047	0.034
8	4	2	70.985	1.3267	1.3269	-0.00016	0.011
9	2	1	71.929	1.3116	1.3114	-0.00021	-0.015
9	2	2	73.380	1.2892	1.2891	-0.00012	-0.008
8	5	1	73.901	1.2814	1.2819	0.00047	0.033
9	3	2	75.782	1.2542	1.2543	0.00012	0.009
8	4	4	76.748	1.2409	1.2412	0.00041	0.028
9	4	1	77.682	1.2282	1.2285	0.00028	0.019
9	3	3	78.166	1.2218	1.2222	0.00048	0.033

TABLE 10

Non-Linear Least-Squares Output for Potassium Alum

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	1	1	7.0188	5	4	1	1.8759	6	4	4	1.4743	2	6	7	1.2886
2	0	0	6.0785	1	4	5	1.8759	0	2	8	1.4743	3	4	8	1.2886
2	1	0	5.4368	5	3	3	1.8539	8	2	1	1.4635	8	5	1	1.2815
2	1	1	4.9631	6	2	2	1.8327	7	4	2	1.4635	7	5	4	1.2815
2	2	0	4.2981	6	3	0	1.8123	2	4	7	1.4635	4	5	7	1.2815
2	2	1	4.0523	5	4	2	1.8123	1	2	8	1.4635	1	5	8	1.2815
3	1	1	3.6655	2	4	5	1.8123	6	5	3	1.4530	9	3	1	1.2744
2	2	2	3.5094	6	3	1	1.7925	3	5	6	1.4530	1	3	9	1.2744
0	2	3	3.3717	1	3	6	1.7925	6	6	0	1.4327	8	5	2	1.2606
3	2	1	3.2491	4	4	4	1.7547	8	2	2	1.4327	2	5	8	1.2606
1	2	3	3.2491	6	3	2	1.7367	8	3	0	1.4229	9	3	2	1.2539
4	0	0	3.0392	2	3	6	1.7367	6	6	1	1.4229	7	6	3	1.2539
4	1	0	2.9485	5	4	3	1.7193	8	3	1	1.4132	3	6	7	1.2539
3	2	2	2.9485	3	4	5	1.7193	7	4	3	1.4132	2	3	9	1.2539
4	1	1	2.8654	7	1	1	1.7023	3	4	7	1.4132	8	4	4	1.2408
3	3	1	2.7890	5	5	1	1.7023	1	3	8	1.4132	6	6	5	1.2344
4	2	0	2.7184	6	4	0	1.6859	7	5	1	1.4038	0	4	9	1.2344
0	2	4	2.7184	0	4	6	1.6859	5	5	5	1.4038	9	4	1	1.2280
4	2	1	2.6529	6	4	1	1.6699	1	5	7	1.4038	8	5	3	1.2280
1	2	4	2.6529	1	4	6	1.6699	6	6	2	1.3945	3	5	8	1.2280
3	3	2	2.5919	0	2	7	1.6699	8	3	2	1.3854	1	4	9	1.2280
4	2	2	2.4815	7	2	1	1.6544	6	5	4	1.3854	7	7	1	1.2218
4	3	0	2.4314	5	5	2	1.6544	4	5	6	1.3854	9	3	3	1.2218
4	3	1	2.3842	6	3	3	1.6544	2	3	8	1.3854	7	5	5	1.2218
1	3	4	2.3842	1	2	7	1.6544	7	5	2	1.3765	10	0	0	1.2157
5	1	1	2.3396	6	4	2	1.6245	2	5	7	1.3765	8	6	0	1.2157
3	3	3	2.3396	2	4	6	1.6245	8	4	0	1.3592	0	6	8	1.2157
4	3	2	2.2575	7	2	2	1.6102	0	4	8	1.3592	10	1	0	1.2097
2	3	4	2.2575	5	4	4	1.6102	8	4	1	1.3508	8	6	1	1.2097
0	2	5	2.2575	7	3	1	1.5827	6	6	3	1.3508	9	4	2	1.2097
5	2	1	2.2196	5	5	3	1.5827	7	4	4	1.3508	7	6	4	1.2097
1	2	5	2.2196	1	3	7	1.5827	1	4	8	1.3508	4	6	7	1.2097
4	4	0	2.1491	6	5	0	1.5565	8	3	3	1.3425	1	6	8	1.2097
4	4	1	2.1163	6	4	3	1.5565	9	1	1	1.3344	2	4	9	1.2097
5	2	2	2.1163	3	4	6	1.5565	7	5	3	1.3344	10	1	1	1.2037
4	3	3	2.0849	6	5	1	1.5439	3	5	7	1.3344	7	7	2	1.2037
5	3	1	2.0549	7	3	2	1.5439	8	4	2	1.3264				
1	3	5	2.0549	1	5	6	1.5439	2	4	8	1.3264				
6	0	0	2.0262	2	3	7	1.5439	0	6	7	1.3186				
4	4	2	2.0262	8	0	0	1.5196	0	2	9	1.3186				
6	1	0	1.9986	8	1	0	1.5079	9	2	1	1.3109				
6	1	1	1.9721	6	5	2	1.5079	7	6	1	1.3109				
5	3	2	1.9721	2	5	6	1.5079	6	5	5	1.3109				
2	3	5	1.9721	0	4	7	1.5079	1	6	7	1.3109				
6	2	0	1.9222	8	1	1	1.4964	1	2	9	1.3109				
0	2	6	1.9222	7	4	1	1.4964	6	6	4	1.2959				
6	2	1	1.8986	5	5	4	1.4964	8	5	0	1.2886				
4	4	3	1.8986	1	4	7	1.4964	9	2	2	1.2886				
0	4	5	1.8986	7	3	3	1.4852	7	6	2	1.2886				
1	2	6	1.8986	8	2	0	1.4743	8	4	3	1.2886				

TABLE 11

HKL Generating Output for Potassium Alum

29.

A= 12.1589A
+/- 0.0004

SSR (DEGREES 2-THETA): 1.5426D-02
RMS RESIDUALS (DEGREES 2-THETA): 1.5774D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS $1/D^{2}$	RESIDUALS 2-THETA
1	1	1	12.599	7.0200	7.0200	-0.00000	-0.000
2	1	0	16.280	5.4400	5.4376	-0.00003	-0.007
2	1	1	17.868	4.9600	4.9639	0.00006	0.014
2	2	0	20.648	4.2980	4.2988	0.00002	0.004
2	2	1	21.912	4.0530	4.0530	-0.00000	-0.000
3	1	1	24.251	3.6670	3.6661	-0.00004	-0.006
3	2	1	27.420	3.2500	3.2496	-0.00002	-0.003
4	0	0	29.365	3.0390	3.0397	0.00005	0.007
4	1	0	30.272	2.9500	2.9490	-0.00008	-0.011
4	1	1	31.182	2.8660	2.8659	-0.00001	-0.001
3	3	1	32.065	2.7890	2.7894	0.00004	0.005
4	2	0	32.914	2.7190	2.7188	-0.00002	-0.002
4	2	1	33.744	2.6540	2.6533	-0.00008	-0.009
3	3	2	34.562	2.5930	2.5923	-0.00008	-0.010
4	2	2	36.160	2.4820	2.4819	-0.00001	-0.001
4	3	1	37.685	2.3850	2.3846	-0.00006	-0.007
5	1	1	38.438	2.3400	2.3400	-0.00000	-0.000
4	3	2	39.874	2.2590	2.2579	-0.00020	-0.021
5	2	1	40.605	2.2200	2.2199	-0.00002	-0.002
4	4	0	41.988	2.1500	2.1494	-0.00012	-0.012
5	2	2	42.653	2.1180	2.1166	-0.00030	-0.030
4	3	3	43.362	2.0850	2.0852	0.00005	0.005
5	3	1	44.028	2.0550	2.0552	0.00005	0.005
6	0	0	44.669	2.0270	2.0265	-0.00012	-0.012
6	1	0	45.305	2.0000	1.9989	-0.00027	-0.026
6	1	1	45.960	1.9730	1.9724	-0.00015	-0.014
6	2	0	47.201	1.9240	1.9225	-0.00042	-0.039
6	2	1	47.861	1.8990	1.8989	-0.00003	-0.003
5	4	1	48.457	1.8770	1.8762	-0.00025	-0.023
5	3	3	49.070	1.8550	1.8542	-0.00024	-0.022
6	2	2	49.698	1.8330	1.8330	0.00001	0.001
6	3	0	50.314	1.8120	1.8125	0.00018	0.016
6	3	1	50.885	1.7930	1.7927	-0.00009	-0.008
5	4	3	53.243	1.7190	1.7195	0.00021	0.018
7	1	1	53.818	1.7020	1.7020	0.00024	0.020
6	4	0	54.370	1.6860	1.6861	0.00006	0.005
6	4	1	54.935	1.6700	1.6702	0.00007	0.006
7	2	1	55.512	1.6540	1.6546	0.00027	0.023
6	4	2	56.629	1.6240	1.6248	0.00038	0.031
7	2	2	57.167	1.6100	1.6105	0.00023	0.019
7	3	1	58.274	1.5820	1.5830	0.00048	0.039
6	5	0	59.324	1.5565	1.5568	0.00015	0.012
7	3	2	59.853	1.5440	1.5442	0.00010	0.008
8	1	1	61.962	1.4964	1.4967	0.00016	0.012
8	2	0	62.992	1.4744	1.4745	0.00005	0.004
8	2	1	63.491	1.4640	1.4638	-0.00015	-0.011
8	2	2	65.042	1.4328	1.4329	0.00010	0.007
8	3	1	66.021	1.4139	1.4134	-0.00032	-0.024
7	5	1	66.531	1.4043	1.4040	-0.00022	-0.016
6	6	2	67.022	1.3952	1.3947	-0.00035	-0.026
8	3	2	67.565	1.3853	1.3856	0.00025	0.019
8	4	0	69.043	1.3592	1.3594	0.00017	0.012
8	4	1	69.522	1.3510	1.3510	-0.00001	-0.000
9	1	1	70.521	1.3343	1.3346	0.00027	0.019
8	4	2	70.985	1.3267	1.3266	-0.00004	-0.003
9	2	1	71.929	1.3116	1.3111	-0.00042	-0.030
9	2	2	73.380	1.2892	1.2888	-0.00033	-0.024
8	5	1	73.901	1.2814	1.2817	0.00025	0.018
9	3	2	75.782	1.2542	1.2541	-0.00010	-0.007
8	4	4	76.748	1.2408	1.2410	0.00017	0.012
9	4	1	77.682	1.2282	1.2282	0.00004	0.003
9	3	3	78.166	1.2218	1.2220	0.00024	0.017

TABLE 12

Non-Linear Least-Squares Output for Potassium Alum with Corrections

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 12.1601 A
+/- 0.0010 A

SSR (DEGREES 2-THETA): 4.5758D-02
RMS RESIDUALS (DEGREES 2-THETA): 2.9954D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA
1	1	1	12.650	6.9919	7.0206	0.00017	0.052
2	1	0	16.280	5.4401	5.4382	-0.00002	-0.006
2	1	1	17.850	4.9650	4.9643	-0.00001	-0.002
2	2	0	20.650	4.2977	4.2992	0.00004	0.008
2	2	1	21.900	4.0551	4.0534	-0.00005	-0.010
3	1	1	24.250	3.6672	3.6664	-0.00003	-0.005
2	2	2	25.350	3.5105	3.5103	-0.00001	-0.002
0	2	3	26.400	3.3732	3.3726	-0.00003	-0.005
3	2	1	27.400	3.2524	3.2499	-0.00014	-0.021
4	0	0	29.350	3.0406	3.0400	-0.00004	-0.005
4	1	0	30.250	2.9521	2.9493	-0.00022	-0.030
4	1	1	31.200	2.8644	2.8662	0.00015	0.020
3	3	1	32.050	2.7903	2.7897	-0.00005	-0.007
4	2	0	32.900	2.7201	2.7191	-0.00010	-0.013
4	2	1	33.800	2.6497	2.6535	0.00041	0.050
3	3	2	34.600	2.5903	2.5925	0.00026	0.031
4	2	2	36.170	2.4814	2.4822	0.00011	0.012
4	3	1	37.680	2.3853	2.3848	-0.00008	-0.009
5	1	1	38.450	2.3393	2.3402	0.00014	0.016
4	3	2	39.080	2.2586	2.2581	-0.00010	-0.011
5	2	1	40.600	2.2202	2.2201	-0.00002	-0.002
4	4	0	42.000	2.1494	2.1496	0.00004	0.004
5	2	2	42.670	2.1172	2.1168	-0.00008	-0.008
4	3	3	43.300	2.0878	2.0854	-0.00053	-0.053
5	3	1	44.000	2.0562	2.0554	-0.00019	-0.018
6	0	0	44.670	2.0269	2.0267	-0.00006	-0.006
6	1	1	45.950	1.9734	1.9726	-0.00020	-0.019
6	2	0	47.250	1.9221	1.9227	0.00016	0.015
5	4	1	48.500	1.8754	1.8763	0.00027	0.025
5	3	3	49.150	1.8522	1.8544	0.00070	0.063
6	2	2	49.750	1.8312	1.8332	0.00065	0.058
6	3	0	50.330	1.8115	1.8127	0.00042	0.037
6	3	1	50.850	1.7941	1.7929	-0.00043	-0.038
5	4	3	53.250	1.7188	1.7197	0.00035	0.030
7	1	1	53.800	1.7025	1.7028	0.00009	0.008
7	2	1	55.450	1.6557	1.6548	-0.00041	-0.034
6	4	0	54.300	1.6880	1.6863	-0.00072	-0.060
6	4	2	56.550	1.6261	1.6250	-0.00052	-0.043
7	2	2	57.150	1.6104	1.6106	0.00010	0.008
7	3	1	58.230	1.5831	1.5831	0.00000	0.000
6	5	0	59.400	1.5547	1.5569	0.00120	0.095
7	3	2	59.800	1.5452	1.5443	-0.00049	-0.039
8	1	1	61.950	1.4967	1.4968	0.00008	0.006
8	2	0	62.950	1.4753	1.4746	-0.00040	-0.031
8	2	2	65.000	1.4336	1.4331	-0.00036	-0.027
8	3	1	66.050	1.4133	1.4136	0.00017	0.012
7	5	1	66.550	1.4039	1.4041	0.00014	0.010
8	3	2	67.550	1.3856	1.3858	0.00016	0.011
8	4	1	69.500	1.3514	1.3511	-0.00021	-0.015
0	6	7	71.430	1.3195	1.3189	-0.00051	-0.036
10	1	0	79.100	1.2097	1.2100	0.00030	0.021

TABLE 13

Non-Linear Least-Squares Output for Potassium Alum Using Experimental 2-Theta Values

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 4.6368A	B= 5.4876A	C= 18.4863A	
+/- C.0013	0.0016	0.0070	

SSR (DEGREES 2-THETA): 1.2919D-02
RMS RESIDUALS (DEGREES 2-THETA): 2.2733D-02

						RESIDUALS	
H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	2-THETA
0	0	2	9.523	9.2800	9.2432	-0.00009	-0.038
0	1	1	16.819	5.2670	5.2607	-0.00009	-0.020
1	0	0	19.108	4.6410	4.6368	-0.00008	-0.017
1	0	2	21.399	4.1490	4.1446	-0.00012	-0.023
1	1	0	25.107	3.5440	3.5418	-0.00010	-0.016
1	1	1	25.569	3.4810	3.4785	-0.00012	-0.019
1	1	2	26.905	3.3110	3.3073	-0.00021	-0.031
1	0	4	27.198	3.2760	3.2733	-0.00015	-0.023
0	0	6	28.975	3.0790	3.0811	0.00014	0.020
1	1	4	31.808	2.8110	2.8112	0.00002	0.002
0	2	2	34.061	2.6300	2.6303	0.00004	0.005
1	0	6	34.966	2.5640	2.5662	0.00026	0.031
0	1	7	37.767	2.3800	2.3797	-0.00005	-0.005
0	2	4	38.100	2.3600	2.3593	-0.00010	-0.011
1	2	1	38.421	2.3410	2.3423	0.00020	0.022
2	0	0	39.852	2.3160	2.3184	0.00039	0.042
1	2	3	40.913	2.2040	2.2050	0.00018	0.019
1	1	7	42.553	2.1180	2.1171	-0.00018	-0.018
2	0	4	43.648	2.0720	2.0723	0.00006	0.006
0	2	6	44.141	2.0500	2.0491	-0.00022	-0.021
2	1	4	46.839	1.9380	1.9387	0.00018	0.017
2	1	5	49.211	1.8500	1.8493	-0.00022	-0.020
2	2	0	51.532	1.7720	1.7709	-0.00040	-0.035
0	3	4	53.986	1.7000	1.7008	0.00033	0.028
2	1	7	55.294	1.6600	1.6606	0.00026	0.022

TABLE 14

Non-Linear Least-Squares Output for Prehnite

32.

A = 11.2284A   B= 11.3106A   C= 3.8393A
+/- 0.0018   0.0020   0.0007

SSR (DEGREES 2-THETA):   1.6224D-02
RMS RESIDUALS (DEGREES 2-THETA):   2.0396D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA
1	1	0	11.064	7.9900	7.9686	-0.00008	-0.030
0	2	0	15.660	5.6540	5.6553	0.00001	0.004
1	2	0	17.540	5.0520	5.05C8	-0.00002	-0.004
2	2	0	22.279	3.9870	3.9843	-0.00009	-0.015
1	0	1	24.489	3.6320	3.6328	0.00004	0.006
1	3	0	24.900	3.5730	3.5741	0.00005	0.008
3	1	0	25.021	3.5560	3.5533	-0.00012	-0.019
1	1	1	25.742	3.4580	3.4588	0.00004	0.006
0	2	1	28.054	3.1780	3.1765	-0.00009	-0.014
2	3	0	28.512	3.1280	3.1299	0.00013	0.018
2	1	1	29.228	3.0530	3.0516	-0.00010	-0.013
2	2	1	32.363	2.7640	2.7646	0.00006	0.008
3	0	1	33.407	2.6800	2.68C0	0.00000	0.001
3	1	1	34.344	2.6090	2.6078	-0.00013	-0.016
2	4	0	35.524	2.5250	2.5254	0.00005	0.006
2	3	1	37.025	2.4260	2.4259	-0.00001	-0.001
0	4	1	39.545	2.2770	2.2768	-0.00003	-0.004
4	3	0	40.003	2.2520	2.2516	-0.00008	-0.008
1	4	1	40.358	2.2330	2.2314	-0.00029	-0.030
5	1	0	40.951	2.2020	2.2027	0.00013	0.013
3	3	1	41.284	2.1850	2.1844	-0.00012	-0.012
4	2	1	43.015	2.1010	2.1035	0.00053	0.053
5	2	0	43.297	2.0880	2.0871	-0.00019	-0.019
4	4	0	45.497	1.9920	1.9921	0.00004	0.003
4	3	1	46.788	1.9400	1.9422	0.00060	0.056
0	0	2	47.305	1.9200	1.9197	-0.00009	-0.009
0	6	0	48.238	1.8850	1.8851	0.00003	0.003
6	0	0	48.623	1.8710	1.8714	0.00012	0.011
1	6	0	48.985	1.8580	1.8591	0.00034	0.030
6	1	0	49.325	1.8460	1.8463	0.00009	0.008
3	5	1	52.911	1.7290	1.7287	-0.00013	-0.011
5	3	1	53.044	1.7250	1.7239	-0.00042	-0.036
1	3	2	54.210	1.6906	1.6912	0.00024	0.020
2	3	2	56.184	1.6358	1.6364	0.00028	0.023
7	2	0	59.891	1.5431	1.5432	0.00004	0.003
2	4	2	60.545	1.5280	1.5283	0.00015	0.012
3	7	0	62.552	1.4837	1.4835	-0.00014	-0.011
4	3	2	63.603	1.4617	1.4608	-0.00058	-0.044
5	6	0	64.471	1.4441	1.4438	-0.00018	-0.013

TABLE 15

Non-Linear Least-Squares Output for Stibnite

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 18.1857A ————————— B = 18.6073A ————————— C = 6.6141A ————————

+/- 0.0526   0.0697   0.0132

SSR (DEGREES 2-THETA):   1.16660+01

RMS RESIDUALS (DEGREES 2-THETA):   4.37315-01

H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	KESIDUALS 2-THETA
2	2	0	13.633	6.4530	6.5037	0.00010	0.029
1	1	1	15.065	5.9000	5.8957	-0.00004	-0.011
0	4	0	19.070	4.6500	4.6519	0.00004	0.008
4	0	0	19.450	4.5630	4.5474	-0.00027	-0.054
1	3	1	20.365	4.3700	4.3906	0.00049	0.096
3	1	1	20.395	4.3500	4.3458	-0.00010	-0.020
2	4	0	21.393	4.1500	4.1416	-0.00024	-0.044
4	2	0	21.657	4.1000	4.0855	-0.00042	-0.078
3	3	1	24.502	3.6300	3.6261	-0.00016	-0.027
4	4	0	27.249	3.2700	3.2518	-0.00105	-0.155
1	5	1	27.957	3.2000	3.1950	-0.00043	-0.062
5	1	1	28.365	3.1500	3.1418	-0.00055	-0.075
0	2	2	28.775	3.1000	3.1160	0.00107	0.151
2	6	0	30.325	2.9450	2.9355	-0.00076	-0.102
6	5	1	31.249	2.8600	2.8599	-0.00001	-0.002
6	3	1	31.509	2.8570	2.8351	-0.00017	-0.022
6	6	0	34.896	2.5650	2.5622	-0.00081	-0.096
1	7	1	36.785	2.4410	2.4441	0.00043	0.049
7	1	1	37.344	2.4060	2.3984	-0.00110	-0.123
0	8	0	38.705	2.3210	2.3266	0.00079	0.086
8	1	0	39.347	2.2680	2.2737	-0.00241	-0.258
2	8	0	39.929	2.2560	2.2534	-0.00045	-0.047
6	6	2	40.396	2.2310	2.2347	0.00067	0.070
2	6	2	41.206	2.1890	2.1953	0.00120	0.124
6	6	0	41.503	2.1740	2.1679	-0.00115	-0.122
4	8	0	43.070	2.0710	2.0708	-0.00004	-0.004
8	4	0	44.186	2.0450	2.0428	-0.00122	-0.119
7	5	1	44.738	2.0240	2.0279	0.00095	0.092
3	3	3	45.293	1.9560	1.9652	0.00138	0.150
9	1	1	47.647	1.9070	1.9225	0.00442	0.408
5	1	3	48.595	1.8720	1.8759	0.00118	0.107
8	2	2	49.960	1.8240	1.8367	0.00415	0.370
5	3	3	50.764	1.7970	1.8040	0.00239	0.210
7	7	1	50.885	1.7930	1.7889	-0.00142	-0.124
4	8	2	52.100	1.7540	1.7551	0.00041	0.035
8	4	2	52.645	1.7370	1.7379	0.00036	0.031
9	5	1	53.077	1.7240	1.7153	-0.00342	-0.290
10	4	0	53.852	1.7010	1.6941	-0.00283	-0.238
5	5	3	54.637	1.6770	1.6819	0.00208	0.174
0	0	4	55.732	1.6480	1.6535	0.00245	0.202
3	7	3	56.440	1.6290	1.6342	0.00239	0.195
7	3	3	56.781	1.6200	1.6226	0.00122	0.099
2	2	4	57.479	1.6020	1.6025	0.00026	0.021
10	2	2	58.599	1.5740	1.5709	-0.00159	-0.127
4	2	4	60.598	1.5270	1.5327	0.00321	0.251
10	4	2	61.389	1.5090	1.5078	-0.00073	-0.056
9	1	3	62.585	1.4830	1.4851	0.00126	0.096
4	4	4	63.298	1.4680	1.4739	0.00372	0.283
3	9	3	63.538	1.4635	1.4635	0.00098	0.074
9	3	3	64.276	1.4460	1.4486	0.00040	0.030
2	6	4	64.979	1.4360	1.4407	0.00314	0.236
9	5	1	66.016	1.4140	1.4119	-0.00146	-0.108
4	6	4	67.581	1.3850	1.3893	0.00324	0.238
0	8	4	70.056	1.3420	1.3477	0.00467	0.338
8	2	4	71.214	1.3250	1.3237	0.00059	0.042
7	9	3	72.350	1.3050	1.3044	-0.00057	-0.041
4	6	4	73.394	1.2890	1.2901	0.00092	0.207
8	4	4	74.402	1.2740	1.2852	0.01072	0.758
3	5	5	75.162	1.2630	1.2209	-0.04399	-3.074
1	5	5	77.027	1.2370	1.2435	0.00682	0.476
3	5	5	78.457	1.2180	1.2209	0.00315	0.221

TABLE 16

Non-Linear Least-Squares Output for Natrolite

34.

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
2	2	0	6.5230	1	5	3	1.8824	4	10	2	1.5279	5	13	1	1.3072
1	1	1	5.8836	3	9	1	1.8779	12	0	0	1.5237	7	9	3	1.3049
0	4	0	4.6550	5	1	3	1.8739	10	4	2	1.5122	10	10	0	1.3046
4	0	0	4.5710	2	8	2	1.8614	12	2	0	1.5037	9	7	3	1.3011
1	3	1	4.3869	9	3	1	1.8529	1	9	3	1.5012	11	3	3	1.2964
3	1	1	4.3512	6	8	0	1.8497	5	11	1	1.4960	14	2	0	1.2933
2	4	0	4.1482	8	2	2	1.8411	9	1	3	1.4869	13	5	1	1.2903
4	2	0	4.1031	8	6	0	1.8403	11	5	1	1.4791	4	8	4	1.2903
3	3	1	3.6299	2	10	0	1.8245	4	4	4	1.4709	1	3	5	1.2864
4	4	0	3.2615	6	6	2	1.8150	4	12	0	1.4693	3	1	5	1.2855
1	5	1	3.1926	3	5	3	1.8073	3	9	3	1.4623	8	4	4	1.2848
5	1	1	3.1516	5	3	3	1.8023	8	8	2	1.4616	8	12	0	1.2838
0	2	2	3.1070	10	2	0	1.7941	9	3	3	1.4504	4	14	0	1.2770
2	0	2	3.1006	7	7	1	1.7934	12	4	0	1.4481	9	11	1	1.2759
2	2	2	2.9418	4	8	2	1.7555	8	10	0	1.4436	6	12	2	1.2751
2	6	0	2.9386	8	4	2	1.7417	10	8	0	1.4378	12	8	0	1.2748
6	2	0	2.8961	5	9	1	1.7370	2	6	4	1.4374	11	9	1	1.2715
3	5	1	2.8625	4	10	0	1.7244	6	2	4	1.4323	12	6	2	1.2633
5	3	1	2.8426	9	5	1	1.7215	6	10	2	1.4313	3	3	5	1.2617
2	4	2	2.5806	10	4	0	1.7018	10	6	2	1.4213	5	11	3	1.2590
4	2	2	2.5696	1	7	3	1.6369	7	7	3	1.4213	14	4	0	1.2574
4	6	0	2.5675	5	5	3	1.6807	9	9	1	1.4157	11	5	3	1.2489
6	4	0	2.5496	7	1	3	1.6747	1	13	1	1.3956	1	5	5	1.2400
1	7	1	2.4446	0	0	4	1.6480	5	9	3	1.3928	5	1	5	1.2375
5	5	1	2.4261	1	11	1	1.6330	7	11	1	1.3886	7	13	1	1.2337
7	1	1	2.4079	3	7	3	1.6322	2	12	2	1.3876	0	14	2	1.2334
0	8	0	2.3275	8	8	0	1.6307	4	6	4	1.3869	6	8	4	1.2305
4	4	2	2.3183	7	3	3	1.6230	9	5	3	1.3847	8	6	4	1.2277
3	7	1	2.2866	0	10	2	1.6212	6	4	4	1.3840	2	10	4	1.2230
8	0	0	2.2855	6	8	2	1.6130	6	12	0	1.3827	2	14	2	1.2223
7	3	1	2.2614	8	6	2	1.6068	11	7	1	1.3784	13	7	1	1.2218
0	6	2	2.2594	11	1	1	1.6057	13	1	1	1.3718	6	14	0	1.2190
2	8	0	2.2555	10	0	2	1.5989	12	2	2	1.3680	3	5	5	1.2178
6	0	2	2.2375	2	2	4	1.5978	12	6	0	1.3677	1	15	1	1.2172
8	2	0	2.2196	2	10	2	1.5963	3	13	1	1.3641	5	3	5	1.2162
2	6	2	2.1934	6	10	0	1.5889	0	8	4	1.3450	14	0	2	1.2142
6	2	2	2.1756	3	11	1	1.5832	13	3	1	1.3429	10	2	4	1.2137
6	6	0	2.1743	10	2	2	1.5758	4	12	2	1.3420	10	10	2	1.2130
1	1	3	2.1668	10	6	0	1.5753	1	11	3	1.3374	9	9	3	1.2100
4	8	0	2.0741	7	9	1	1.5748	8	0	4	1.3367	14	2	2	1.2040
1	3	3	2.0582	9	7	1	1.5682	2	8	4	1.3307	14	6	0	1.2037
3	1	3	2.0545	11	3	1	1.5600	12	4	2	1.3258				
8	4	0	2.0516	0	4	4	1.5535	8	2	4	1.3232				
5	7	1	2.0450	0	12	0	1.5517	8	10	2	1.3223				
7	5	1	2.0341	4	0	4	1.5503	11	1	3	1.3223				
4	6	2	2.0255	5	7	3	1.5371	10	8	2	1.3179				
6	4	2	2.0167	7	5	3	1.5325	2	14	0	1.3161				
1	9	1	1.9625	2	4	4	1.5316	6	6	4	1.3134				
3	3	3	1.9612	2	12	0	1.5298	1	1	5	1.3117				
9	1	1	1.9310	4	2	4	1.5293	3	11	3	1.3096				

TABLE 17

HKL Generating Output for Natrolite

A= 18.2533A   B= 18.6187A   C= 6.5932A
+/- 0.0128   0.0133   0.0041

SSR (DEGREES 2-THETA): 7.1328D-01
RMS RESIDUALS (DEGREES 2-THETA): 1.0813D-01

| | | | | | | | RESIDUALS |
H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	2-THETA
2	2	0	13.633	6.4960	6.5171	0.00020	0.057
1	1	1	15.003	5.9000	5.8833	-0.00015	-0.043
0	4	0	15.070	4.6500	4.6547	0.00009	0.019
4	0	0	15.450	4.5530	4.5632	0.00007	0.014
1	3	1	20.305	4.3700	4.3866	0.00040	0.078
3	1	1	20.399	4.3500	4.3478	-0.00005	-0.011
2	4	0	21.393	4.1500	4.1465	-0.00010	-0.018
4	2	0	21.657	4.1000	4.0975	-0.00007	-0.013
3	3	1	24.532	3.6300	3.6279	-0.00009	-0.015
4	4	0	27.249	3.2700	3.2596	-0.00022	-0.097
1	5	1	27.857	3.2000	3.1924	-0.00047	-0.068
5	1	1	28.309	3.1500	3.1473	-0.00014	-0.020
0	2	2	28.775	3.1000	3.1075	0.00050	0.071
2	6	0	30.325	2.9450	2.9379	-0.00055	-0.075
2	5	1	31.249	2.8600	2.8614	0.00012	0.016
5	3	1	31.506	2.8570	2.8398	0.00024	-0.032
4	6	0	34.896	2.5690	2.5660	-0.00035	-0.042
1	7	1	36.789	2.4410	2.4444	0.00047	0.054
7	1	1	37.344	2.4060	2.4045	-0.00021	-0.024
0	8	0	38.765	2.3210	2.3273	0.00151	-0.110
3	7	1	39.347	2.2850	2.2850	-0.00034	-0.057
2	8	0	39.925	2.2560	2.2552	-0.00014	-0.015
6	0	2	40.356	2.2310	2.2357	0.00084	0.089
2	6	2	41.204	2.1850	2.1833	0.00162	0.085
6	6	0	41.503	2.1740	2.1724	-0.00032	-0.032
4	8	0	43.070	2.0710	2.0733	0.00015	0.050
8	4	0	44.186	2.0450	2.0486	0.00018	0.017
7	5	1	44.733	2.0240	2.0220	-0.00152	-0.186
3	3	3	46.283	1.9600	1.9611	0.00029	0.028
1	5	3	47.047	1.9070	1.8826	-0.00713	-0.657
5	1	3	48.595	1.8720	1.8735	0.00040	0.036
2	10	0	49.960	1.8290	1.8243	0.00010	0.009
10	2	0	50.764	1.7970	1.7912	-0.00200	-0.176
7	7	1	50.885	1.7930	1.7919	-0.00036	-0.032
4	8	2	52.100	1.7540	1.7550	0.00038	0.033
8	4	2	52.649	1.7370	1.7401	0.00116	0.101
4	10	0	53.077	1.7240	1.7239	-0.00004	-0.003
10	4	0	53.852	1.7010	1.6993	-0.00068	-0.057
5	5	3	54.687	1.6770	1.6803	0.00139	0.116
0	0	4	55.732	1.6480	1.6483	0.00013	0.011
3	7	3	56.440	1.6290	1.6321	0.00144	0.118
7	3	3	56.781	1.6200	1.6221	0.00097	0.079
2	2	4	57.479	1.6020	1.5980	-0.00196	-0.158
7	9	1	58.599	1.5740	1.5738	-0.00009	-0.008
4	10	2	60.588	1.5270	1.5276	0.00036	0.028
10	4	2	61.389	1.5090	1.5105	0.00085	0.066
9	1	3	62.585	1.4830	1.4857	0.00166	0.127
4	12	0	63.298	1.4680	1.4690	0.00061	0.047
3	9	3	63.586	1.4620	1.4622	0.00014	0.010
9	3	3	64.276	1.4480	1.4493	0.00083	0.062
2	6	4	64.979	1.4360	1.4375	0.00102	0.077
6	9	1	66.016	1.4140	1.4145	0.00038	0.028
9	5	3	67.581	1.3850	1.3837	-0.00095	-0.070
4	12	2	70.056	1.3420	1.3418	-0.00018	-0.013
8	2	4	71.214	1.3230	1.3226	-0.00037	-0.027
7	9	3	72.350	1.3050	1.3044	-0.00058	-0.041
4	8	4	73.394	1.2890	1.2902	0.00115	0.081
12	8	0	74.402	1.2740	1.2733	-0.00070	-0.050
12	6	2	75.162	1.2630	1.2618	-0.00117	-0.082
5	1	5	77.027	1.2370	1.2375	0.00055	0.035
11	5	1	78.457	1.2180	1.2171	-0.00099	-0.069

TABLE 18

Non-Linear Least-Squares Output for Natrolite with Corrections

36.