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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<td></td>
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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 637 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:

\[
\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \quad \text{(tetragonal),} \quad \frac{1}{d^2} = \frac{4(h^2 + hk + k^2)}{3a^2} + \frac{l^2}{c^2} \quad \text{(hexagonal).}
\]

Let \(D = \frac{1}{d^2}\), \(A = \frac{1}{a^2}\), \(C = \frac{1}{c^2}\), \(r = h^2 + k^2\) or \(\frac{4}{3}(h^2 + hk + k^2)\), \(s = l^2\). Then eqn. 1 becomes a linear equation in two variables, \(A\) and \(C\):

\[
D = rA + sC
\]

The "normal" equations for least-squares solution of this linear equation are the following:

\[
\begin{align*}
\sum \xi r_i^2 A & + \sum \xi r_i s_i C = \sum \xi r_i D_i \\
\sum \xi r_i s_i A & + \sum \xi s_i^2 C = \sum \xi s_i D_i
\end{align*}
\]
Solution of these two equations yields \( A \) and \( C \), from which \( a \) and \( c \) can be obtained. Let the matrix \( B_{ij} = \begin{bmatrix} \ell \cdot r^2 & \ell \cdot r^3 \\ \ell \cdot r^3 & \ell \cdot s^3 \end{bmatrix} \) 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:

\[
\sigma_a^2 = B_{11}^{-1} \left( \frac{f}{n-2} \right), \quad \sigma_c^2 = B_{22}^{-1} \left( \frac{f}{n-2} \right),
\]

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, \( \sigma_a \) and \( \sigma_c \), using the equations \( \sigma_a^2 = \left( \frac{\partial f}{\partial a} \right)^2 \sigma_a^2 \) and \( \sigma_c^2 = \left( \frac{\partial f}{\partial c} \right)^2 \sigma_c^2 \)

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

**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)
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 \( B_{11} \) 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 \( B_{21} \) 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), \( \Delta \), and \( d_c \).

6. Calculate \( f \).

7. Calculate \( a, c, \sigma_a \), and \( \sigma_c \)

ALL WORK SHOULD BE RECORDED TO AT LEAST 5 SIGNIFICANT FIGURES; RESULTS OF \( a, c, \sigma_a \), and \( \sigma_c \) SHOULD BE GIVEN TO 3 FIGURES TO THE RIGHT OF THE DECIMAL POINT.
EXAMPLE FOR LEAST-SQUARES REFINEMENT
OF CELL PARAMETERS

| Substance: Tetrahedron System: Tetragonal |

<table>
<thead>
<tr>
<th>hkl</th>
<th>d₀</th>
<th>r</th>
<th>s</th>
<th>D₀</th>
<th>Dₑ</th>
<th>Δ</th>
<th>dₑ</th>
</tr>
</thead>
<tbody>
<tr>
<td>101</td>
<td>4.72</td>
<td>1</td>
<td>1</td>
<td>0.044887</td>
<td>0.044807</td>
<td>0.00086</td>
<td>4.72419</td>
</tr>
<tr>
<td>200</td>
<td>3.85</td>
<td>4</td>
<td>0</td>
<td>0.079249</td>
<td>0.078988</td>
<td>0.00236</td>
<td>3.85811</td>
</tr>
<tr>
<td>211</td>
<td>2.842</td>
<td>5</td>
<td>1</td>
<td>1.123608</td>
<td>1.123795</td>
<td>0.00087</td>
<td>2.84212</td>
</tr>
<tr>
<td>112</td>
<td>2.676</td>
<td>2</td>
<td>4</td>
<td>1.199446</td>
<td>1.199774</td>
<td>0.00038</td>
<td>2.67616</td>
</tr>
<tr>
<td>220</td>
<td>2.518</td>
<td>8</td>
<td>0</td>
<td>1.157974</td>
<td>1.157964</td>
<td>0.00010</td>
<td>2.51874</td>
</tr>
<tr>
<td>202</td>
<td>2.361</td>
<td>4</td>
<td>4</td>
<td>1.179249</td>
<td>1.179228</td>
<td>0.00015</td>
<td>2.36209</td>
</tr>
<tr>
<td>301</td>
<td>2.222</td>
<td>9</td>
<td>1</td>
<td>1.202541</td>
<td>1.202893</td>
<td>0.00092</td>
<td>2.22847</td>
</tr>
<tr>
<td>103</td>
<td>2.019</td>
<td>1</td>
<td>9</td>
<td>1.245281</td>
<td>1.245281</td>
<td>0.00003</td>
<td>2.01423</td>
</tr>
</tbody>
</table>

\[
\begin{align*}
D₀ &= \frac{1}{\sqrt[4]{s}} \\
Δ &= D₀ - Dₑ \\
Σ r^2 &= \frac{20B}{r} \\
Σ rD₀ &= \frac{5.3101}{r} \\
Σ s^2 &= \frac{4B}{s} \\
Σ sD₀ &= \frac{3.8552}{s} \\
Σ t^2 &= \frac{11C}{t} \\
f^2 &= \Sigma A^2 = 3.3 \times 10^{-7}
\end{align*}
\]

\[
\begin{align*}
\Sigma \left(\frac{1}{A}\right)^{1/6} \sigma_6 &= \sqrt{2} \left( \frac{B_{11}^{1/4}f}{A^3 (n-2)} \right)^{1/2} \\
\Sigma \left(\frac{1}{C}\right)^{1/6} \sigma_6 &= \sqrt{2} \left( \frac{B_{33}^{1/4}f}{C^3 (n-2)} \right)^{1/2}
\end{align*}
\]

\[
\begin{align*}
a &= 7.11422 \pm \text{0.0257} \\
c &= 6.31648 \pm \text{0.02441}
\end{align*}
\]
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-alpha₁ and K-alpha₂, 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 $\text{ThSiO}_4$; 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 1.5418Å. 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 $0.245^\circ$ off, i.e., the difference between the calculated and observed 2-theta values is $0.245^\circ$. The output also shows that the calculated cell dimensions of $a = 7.1339Å \pm .0013$ and $c = 6.3199Å \pm .0017$ match quite well with the cell dimensions listed on the JCPDS card, $a = 7.142Å$ and $c = 6.327Å$. Because the cell dimensions fit so well and because $0.245^\circ$ 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 $\text{Cu}_3\text{V}_4\text{S}_4$ 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, $a = 5.3951$Å ± .0012 with the cell dimension listed on the JCPDS card $a = 5.391$Å, the JCPDS card for sulvanite contains no errors.

Xenotime

The chemical formula for yttrium, erbium phosphate (xenotime) is $(\text{Y,Er})\text{PO}_4$ 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 $a = 6.9026$Å ± .0010 and $c = 6.0360$Å ± .0016 also fit very well with the cell dimensions listed on the...
JCPDS card \( a = 6.904\text{A} \) and \( c = 6.033\text{A} \). 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 \( \text{ZnO} \) 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 \( a = 3.2495\text{A} \pm .0012 \) and \( c = 5.2050\text{A} \pm .0022 \) fit exactly with the cell dimensions given on the JCPDS card \( a = 3.249\text{A} \) and \( c = 5.205\text{A} \). 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 1.237Å was the correct value and that the d-value given on the JCPDS card of 1.2250Å 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 1.235Å and not 1.225Å.

The final conclusion was that a typographical error was made on the original card that was in the JCPDS file.
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 1.54050Å 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 $a = 10.0579Å ± 0.0013$ and $c = 8.4169Å ± 0.0017$ fit equally well with the cell dimensions listed on the JCPDS card $a = 10.060Å$ and $c = 8.417Å$. 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 1.7902Å 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, $a = 8.1122Å ± 0.0023$ and $c = 8.8258Å ± 0.0061$ fit quite well with the cell dimensions given on the JCPDS card, $a = 8.111Å$ and $c = 8.824Å$. Therefore, the JCPDS card for phosgenite contains no errors.
Potassium Alum

The chemical formula for potassium aluminum sulfate hydrate (potassium alum) is KAl(SO$_4$)$_2$ · 12H$_2$O 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 1.5405Å 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 1.3853Å 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 1.3853Å. 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 1.3853Å.

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
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 $\text{Ca}_2\text{Al}_2\text{Si}_3\text{O}_{10}(\text{OH})_2$ 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 1.7902Å. 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 $a = 4.6368\text{Å} \pm 0.0013$, $b = 5.4876\text{Å} \pm 0.0016$ and $c = 18.4863\text{Å} \pm 0.0070$ fit equally well with the cell dimensions listed on the JCPDS card $a = 4.642\text{Å}$, $b = 5.487\text{Å}$ and $c = 18.490\text{Å}$. The JCPDS card for prehnite contains no errors.

Stibnite

The chemical formula for antimony sulfide (stibnite) is $\text{Sb}_2\text{S}_3$ 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 1.5405Å 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.
Natrolite

The chemical formula for the zeolite mineral sodium aluminum silicate hydrate (natrolite) is Na₂Al₂Si₃O₁₀ · 2H₂O 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 1.5418 Å 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:

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<tr>
<th>new hkl</th>
<th>d-value</th>
<th>hkl replaced</th>
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<td>800</td>
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<td>911</td>
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<td>1.8240</td>
<td>822</td>
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<tr>
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<td>1.7970</td>
<td>533</td>
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<td>444</td>
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<td>844</td>
</tr>
<tr>
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<td>1.2630</td>
<td>355</td>
</tr>
<tr>
<td>515</td>
<td>1.2370</td>
<td>155</td>
</tr>
<tr>
<td>1151</td>
<td>1.2180</td>
<td>355</td>
</tr>
</tbody>
</table>
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 investi-
gation, 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.

<table>
<thead>
<tr>
<th>JCPDS dimensions</th>
<th>Cell dimensions calculated with new hkl values</th>
<th>Cell dimensions calculated with original hkl values</th>
</tr>
</thead>
<tbody>
<tr>
<td>a = 18.284A</td>
<td>18.2533A ± .0128</td>
<td>18.1897A ± .0526</td>
</tr>
<tr>
<td>b = 18.620</td>
<td>18.6187 ± .0108</td>
<td>18.6078 ± .0697</td>
</tr>
<tr>
<td>c = 6.592</td>
<td>6.5932 ± .0041</td>
<td>6.6141 ± .0132</td>
</tr>
</tbody>
</table>

16.
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 1.3853Å 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.
TABLE I

Non-Linear Least-Squares Output for Thorite

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

<table>
<thead>
<tr>
<th>H K L</th>
<th>2-THETA</th>
<th>D(OBS)</th>
<th>D(CALC)</th>
<th>1/D**2</th>
<th>2-THETA</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 0 0</td>
<td>18.785</td>
<td>4.7200</td>
<td>4.7306</td>
<td>0.00075</td>
<td>0.642</td>
</tr>
<tr>
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<td>2.8420</td>
<td>2.8480</td>
<td>0.00052</td>
<td>0.068</td>
</tr>
<tr>
<td>1 2 0</td>
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<td>2.0760</td>
<td>2.0670</td>
<td>0.00062</td>
<td>0.025</td>
</tr>
<tr>
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<td>35.655</td>
<td>2.5160</td>
<td>2.5220</td>
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<td>0.691</td>
</tr>
<tr>
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<td>2.2610</td>
<td>2.2653</td>
<td>0.00065</td>
<td>0.672</td>
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<tr>
<td>2 0 1</td>
<td>40.566</td>
<td>2.3220</td>
<td>2.2956</td>
<td>0.00066</td>
<td>0.069</td>
</tr>
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<td>2.0190</td>
<td>2.0244</td>
<td>0.00034</td>
<td>0.032</td>
</tr>
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<td>0.00039</td>
<td>0.031</td>
</tr>
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REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 5.3951
+/− 0.0012

SSR (DEGREES 2-THETA): 3.33160-01
RMS RESIDUALS (DEGREES 2-THETA): 1.25960-01

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<th>D(CALC)</th>
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TABLE 2

Non-Linear Least-Squares Output for Sulvanite
### Table 3

HKL Generating Output for Sulvanite

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<th>L</th>
<th>D</th>
</tr>
</thead>
<tbody>
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Non-Linear Least-Squares Output for Xenotime

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\[ \text{VS RESIDUALS (DEGREES 2-THETA)}: 1.8492D-01 \]

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TABLE 5

Non-Linear Least-Squares Output for Zincite
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### REFERENCE MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

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### TABLE 7
Non-Linear Least-Squares Output for Zincite Using Experimental 2-Theta Values

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**TABLE 8**

Non-Linear Least-Squares Output for Nepheline

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### Table 9

Non-Linear Least-Squares Output for Phosgenite

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**TABLE 10**

Non-Linear Least-Squares Output for Potassium Alum
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**TABLE 11**

HKL Generating Output for Potassium Alum

29.
Non-Linear Least-Squares Output for Potassium Alum with Corrections

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**TABLE 12**

Non-Linear Least-Squares Output for Potassium Alum with Corrections
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### Non-Linear Least-Squares Output for Potassium Alum Using Experimental \( \theta \) Values

31.
REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

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\[ +/\ - 0.0013 \quad 0.0016 \quad 0.0076 \]

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\[ \text{RMS RESIDUALS (DEGREES 2-THETA):} \quad 2.2733 \quad 0.02 \]

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TABLE 14

Non-Linear Least-Squares Output for Prehnite

32.
### TABLE 15

Non-Linear Least-Squares Output for Stibnite
### Table 16

#### Non-Linear Least-Squares Output for Natrolite

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#### Table 17

Non-Linear Least-Squares Output for Natrolite...
## Table 17

**HKL Generating Output for Natrolite**

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**TABLE 18**

Non-Linear Least-Squares Output for Natrolite with Corrections

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