Precession photography: orienting crystals by means of the stereographic projection. By M. Rieder, Institute of Geological Sciences, Charles University, Albertov 6, 12843 Prague 2, Czechoslovakia

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A graphical construction on the Wulff net permits corrections on both arcs and the dial to be determined even if neither of the arcs is parallel to the film and/or the outer arc reading is large. The procedure requires that the orientation errors be multiplied by an arbitrary common factor smaller than ten, while the biggest multiplied error should be less than ten degrees; the corrections are obtained graphically and divided by the factor. Corrections thus obtained are within 10% of their true values.

The orientation of a crystal on the precession camera is easy if one of the arcs on the goniometer head is parallel to the film and both arcs read zero degrees (Buerger, 1964). When these conditions are not met, as is often the case, and the corrections on the arcs and the dial are set equal to the orientation errors of the misadjusted reciprocal plane, several orientation photographs have to be taken before the crystal is set. While the principles of a solution with the stereographic projection are straightforward, the chief trouble is that most corrections are too small to be handled graphically with sufficient accuracy. A solution by spherical trigonometry (e.g. Donnay, 1945) is accurate, but may take more time than a couple of new orientation photographs.

A tabulation of solutions for all combinations of the five variables (three orientation errors measured on the photograph plus the angle between the plane of one of the arcs and the plane of the film, plus outer arc reading) would be bulky at best. Without a computer teletype in the laboratory, one has to look for another solution.

The problem can be circumvented if the three orientation errors are multiplied by an arbitrary common factor and the solution found graphically on a Wulff net. The multiplied corrections obtained are then divided by the same factor. A comparison of the resulting approximate corrections thus obtained with the calculated ones shows that they differ from each other as the factor increases and as the multiplied errors get bigger. If the factor does not exceed ten and the multiplied errors are less than about ten degrees, the graphically determined corrections are more than 90% of their true values, which is often within the precision of instrumental setting. The results become gradually worse as the optimum conditions are not satisfied. It should be realized, however, that a residual error of say 40% has an excellent chance of being corrected at a 90% level in the second round, leaving a mere 4% of the original error uncorrected. Experience has shown that once the orientation errors can be measured accurately, the solution found graphically on a Wulff net suffices for a perfect alignment of the crystal.

The graphical procedure (Fig. 1) is rapid (about five minutes) and consists of the following six steps: (i) Multiplied orientation errors represented as rotations about axis A parallel to the X-ray beam (a), about axis B perpendicular to the beam and the dial axis (b), and about the dial axis C (c) are plotted; they define the misadjusted reciprocal plane (1), its pole (1), and vector V1 lying in this plane. (ii) The axis of inner arc (I) is plotted using angle d (measured on the dial) and the reading on outer arc (e); the axis of outer arc (E) is perpendicular to axes I and C. (iii) The multiplied correction of the inner arc reading (i) is equal to the angle between the plane defined by axes I and C and the plane defined by axis I and vector V1. It is read off on a great circle at right angles to I. (iv) Pole 1 and vector V1 are rotated about axis I through the angle i and travel to loci 2 and V2, respectively (e.g. Nuffield, 1966). The plane of misadjustment changes from 1 to 2. (v) The multiplied correction of the outer arc reading (e) is defined by the dial axis C and vector V2. As a control, it can be read off between pole 2 and a point on the equator that lies on the same small circle when E is superimposed on

Fig. 1. A stereographic projection showing multiplied orientation errors a, b, c of a reciprocal plane that are to be corrected by rotation about axes of inner arc (I), outer arc (E), and the dial axis (C). The angle between the plane of inner arc and the plane of the film is labelled d, the outer arc reading, e. The projection is a view down the dial axis. Unrealistically big errors have been chosen to illustrate the procedure: a = 14°, b = -24°, c = 10°, d = 50°, e = -25°; i = -25°40', e = 11°20', v = 15°30'. The - sign refers to clockwise, + to counterclockwise, rotation about positive axes A, B, C and I, E, C chosen to form right-handed systems.
either north or south pole. The rotation about $E$ brings to coincidence the vector and dial axis $C$; the misadjusted reciprocal plane and its pole fall to loci labelled 3. (vi) The angle between the X-ray beam ($A$) and pole 3 (or between plane $BC$ and plane 3) define the multiplied correction of the dial reading ($\gamma$).

Little experience is needed to see that only a small part of Fig. 1 has to be drawn to obtain the corrections sought.

References

Crystal Data


Données cristallographiques sur quatre polyphosphates mixtes du type Ba$^{III}$(PO$_3$)$_4$ pour M$^{II}$= Mn, Cd, Ca et Hg, Par Marie-Thérèse Averbuch-Pouchot, Laboratoire des Rayons X, CNRS, B.P. 166, Centre de Tri, 38042 Grenoble Cedex, France

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Four new long-chain polyphosphates are described: BaMn(PO$_3$)$_4$, BaCd(PO$_3$)$_4$, BaCa(PO$_3$)$_4$, and BaHg(PO$_3$)$_4$. All are monoclinic ($P2_1/n$) and belong to a new type of structure. Chemical preparations and crystal data are given.

Introduction

Les diagrammes d'équilibre Cd(PO$_3$)$_2$-Ba(PO$_3$)$_2$ et Ca(PO$_3$)$_2$-Ba(PO$_3$)$_2$ établis par Bukhalova, Tokman & Shpakova (1970) révèlent l'existence de deux composés définis: BaCd(PO$_3$)$_4$ et BaCa(PO$_3$)$_4$. Ces auteurs ne précisent pas la nature de l'anion condensé et n'ont pas effectué d'étude cristallographique sur ces deux sels. Dans cette étude, nous décrivons une méthode de préparation pour ces deux composés ainsi que des sels isotypes: BaMn(PO$_3$)$_4$ et BaHg(PO$_3$)$_4$ en précisant la nature de leur anion et leurs caractéristiques cristallographiques.

Préparations chimiques

Poudres

Ces quatre sels se préparent facilement par calcination d'un mélange en proportions stœchiométriques de monophosphate biammonique, de carbonate de baryum et de carbonate du métal bivalent correspondant aux différentes formules des composés. Les températures de chauffage sont de 700°C pour MnBa(PO$_3$)$_4$, CdBa(PO$_3$)$_4$ et CaBa(PO$_3$)$_4$, et de 400°C pour HgBa(PO$_3$)$_4$.

Cristaux

Seuls des monocristaux de CdBa(PO$_3$)$_4$ ont été préparés.

On les obtient par chauffage à 350°C, durant une journée, d'une solution de 7 cm$^3$ d'acide monophosphorique à 85% dans laquelle ont été introduits 1 g de carbonate de baryum et 1,2 g de carbonate de cadmium. Les cristaux se présentent sous forme de prismes allongés possédant quatre faces.

Analyse chromatographique

L'analyse chromatographique révèle que tous ces sels sont des polyphosphates à longues chaînes.

Etude cristallographique

L'étude d'un cristal du sel de baryum-cadmium par la méthode de Weissenberg montre que ce composé est monoclinique avec le groupe spatial $P2_1/n$. Les diagrammes de poudre indiquent que ces différents composés sont tous isotypes. Leurs caractéristiques cristallographiques sont rassemblées dans le Tableau 1. Les Tableaux 2, 3, 4 et 5 rassemblent les dépouillements de diffractogrammes effectués à vitesse lente ($\frac{1}{2}$°(θ)/min) à l'aide d'un diffractomètre Philips-Noreleo, utilisant la longueur d'onde du cuivre $K_{α1}$. Les intensités données sont les hauteurs des pics au dessus du fond continu. Les valeurs des paramètres de réseau données dans le Tableau 1 ont été affinées par une

<table>
<thead>
<tr>
<th>Formules</th>
<th>$a$ (Å)</th>
<th>$b$ (Å)</th>
<th>$c$ (Å)</th>
<th>$β$ (°)</th>
<th>$V$ (Å$^3$)</th>
<th>$Z$</th>
<th>$d_ε$</th>
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<tr>
<td>MnBa(PO$_3$)$_4$</td>
<td>14,69 (1)</td>
<td>9,147 (6)</td>
<td>7,201 (4)</td>
<td>90,57 (1)</td>
<td>967,3</td>
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<td>3,49</td>
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<tr>
<td>CdBa(PO$_3$)$_4$</td>
<td>14,94 (1)</td>
<td>9,192 (7)</td>
<td>7,219 (5)</td>
<td>90,79 (1)</td>
<td>991,3</td>
<td>4</td>
<td>3,96</td>
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<tr>
<td>CaBa(PO$_3$)$_4$</td>
<td>15,24 (1)</td>
<td>9,173 (7)</td>
<td>7,231 (5)</td>
<td>90,96 (1)</td>
<td>1010,8</td>
<td>4</td>
<td>3,24</td>
</tr>
<tr>
<td>HgBa(PO$_3$)$_4$</td>
<td>15,05 (1)</td>
<td>9,236 (8)</td>
<td>7,239 (6)</td>
<td>90,62 (1)</td>
<td>1006,4</td>
<td>4</td>
<td>4,30</td>
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