

# THE CRYSTALLOGRAPHY OF BASTNAESITE, PARISITE, ROENTGENITE, AND SYNCHISITE\*

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## ABSTRACT

An *x*-ray study of parisite and related minerals from many localities establishes the existence of four species: bastnaesite,  $\text{CeFCO}_3$ ; parisite,  $2\text{CeFCO}_3 \cdot \text{CaCO}_3$ ; roentgenite,  $3\text{CeFCO}_3 \cdot 2\text{CaCO}_3$ ; and synchisite,  $\text{CeFCO}_3 \cdot \text{CaCO}_3$ . Bastnaesite has space group  $C\bar{6}2c$ ,  $a=7.16$ ,  $c=9.79$  Å,  $Z=6$ . Parisite has space group  $R\bar{3}$ ,  $a=7.18$  for the ordered form,  $a=7.12$  for the disordered form,  $c=84.1$  Å,  $Z=18$ . Roentgenite has space group  $R\bar{3}$ ,  $a=7.13$ ,  $c=69.4$  Å,  $Z=9$ . Synchisite is orthorhombic or monoclinic with  $\beta=90^\circ$ , pseudo-hexagonal axes  $a=7.11$ ,  $c=54.7$  Å,  $Z=18$ . All structures show a pronounced pseudo-H-centering of the lattice, which leads to a pseudo-axis  $a'=a/2 \cos 30^\circ$ . When the structures are referred to the  $a'$  cell, several pseudo-periods along  $c$  are observed: one,  $c'''=c/2$ , in the case of bastnaesite; three,  $c'=c/3$ ,  $c''=c/6$ , and  $c'''=c/18$ , for parisite; two,  $c'=c/3$ , and  $c'''=c/15$ , for roentgenite; three,  $c'=c/3$ ,  $c''=c/6$ , and  $c'''=c/12$ , for synchisite. Oftedal's structural scheme is confirmed in its essentials. A linear relationship exists between  $c'''$  and the chemical composition.

Single crystals occur rarely. Most specimens are "polycrystals," syntactic intergrowths of two species. Every pair has been observed, except bastnaesite-synchisite. Differential solubility in nitric acid reveals that the two species are in contact along an irregular surface or along repeated planes parallel to (0001). The complex morphology of these minerals, repeatedly discussed in the literature, is fully explained when the faces on the polycrystals are referred to the appropriate species. Indices of refraction are predicted in two ways:

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(1) from ionic refractivities; (2) from form-birefringence formulas, by letting slabs of the crystal structure be the components of Wiener's "mixed body." The indices thus predicted agree with those measured on specimens identified by  $x$ -rays.

### INTRODUCTION

The main reason for undertaking this study was the promise it held of yielding additional information on syntactic intergrowth, a phenomenon often mentioned in the literature since Ungemach coined the term *syntaxy* in 1935, and recently encountered in the andorite series (Donnay and Donnay, 1952). We are indebted to H. T. Evans, of the U. S. Geological Survey, for suggesting the problem to us. This work was completed as part of a program undertaken by the Geological Survey on behalf of the Division of Research of the Atomic Energy Commission.

The baffling morphology of parisite and related minerals, long a problem to crystallographers, had never been completely solved (Cesàro, 1907; Palache and Warren, 1911; Ungemach, 1935; Parker *et al.*, 1939).

The question of the chemical composition of synchisite also required clarification. The formula for the mineral had been written<sup>1</sup>  $CeFeCO_3 \cdot CaCO_3$ . It remained somewhat doubtful because, as was pointed out by H. Jaffe of the U. S. Geological Survey, the indices of refraction reported in the literature for synchisite ( $CeFeCO_3 \cdot CaCO_3$ ) and parisite ( $2CeFeCO_3 \cdot CaCO_3$ ) were almost identical. This similarity could not be reconciled with the difference in chemical composition. Moreover, Oftedal's calculations (1931b), by means of which he claimed to have confirmed the synchisite formula, were based on a specific gravity of 3.9 (determined by Flink, 1901, p. 35), whereas specific gravities as high as 4.15 were measured by us on crystals, small fragments of which were identified as synchisite by  $x$ -rays.

C. Frondel kindly placed at our disposal the material of the Harvard Museum, including some of the specimens measured and described by Palache and Warren (1911). G. Switzer made available to us all the relevant specimens of the U. S. National Museum. About 35 crystals were studied. They come from the following localities: Narsarsuk, Greenland; Quincy, Mass.; Muzo, Colombia; Pyrites, Mont.; Mountain Pass, Calif.; Bear Paw Mountains, Mont. More than 120  $x$ -ray patterns were interpreted. They are rotation, precession, and Weissenberg photographs, taken with  $MoK\alpha$  ( $\lambda = 0.7107 \text{ \AA}$ ) or  $CuK\alpha$  ( $\lambda = 1.5418 \text{ \AA}$ ) radiation.

One of the results obtained was the discovery of a new mineral, roentgenite,  $3CeFeCO_3 \cdot 2CaCO_3$ , the description of which has already been published (Gabrielle Donnay, 1953b).

<sup>1</sup> In this and all other formulas appearing in this paper, Ce actually stands for the rare earths of the cerium group.

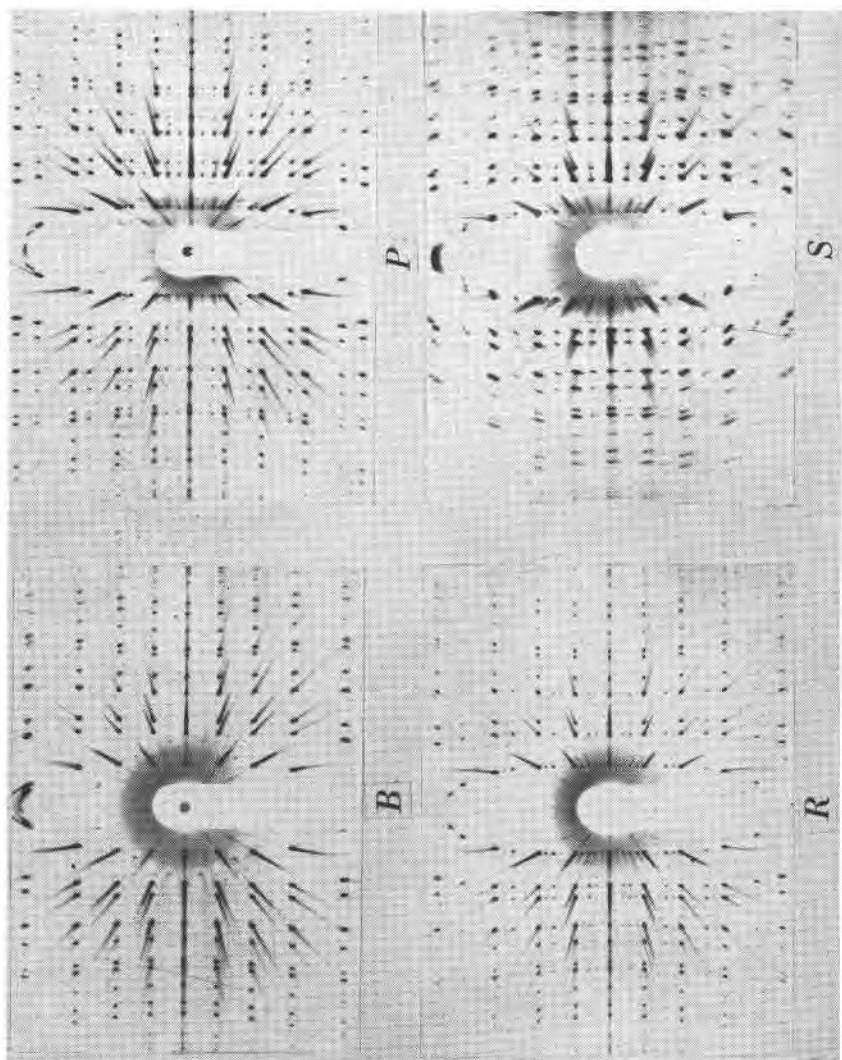


FIG. 1. Rotation photographs about the  $c$  axes of bastnaesite (B), parisite (P), roentgenite (R), synchisite (S). Note faint row lines which give true  $c$  axis. ( $\text{MoK}\alpha$  radiation; 20-hour exposures.)

UNIT-CELL DIMENSIONS AND SYMMETRY

Some of the specimens examined give *x*-ray diffraction patterns that reveal only one crystalline species in a specimen. The data obtained from these single crystals will be presented first.

Rotation photographs about the *c* axes of the four species (Fig. 1) show the following *similarities*. (1) Intense row lines (reciprocal-lattice rows parallel to *c*<sup>\*</sup>) lead to a pseudo-axis *a'* of length 4.1 Å; very faint row lines give the true *a*, at 30° to *a'* and of length  $4.1 \times 2 \cos 30^\circ = 7.1 \text{ \AA}$ .

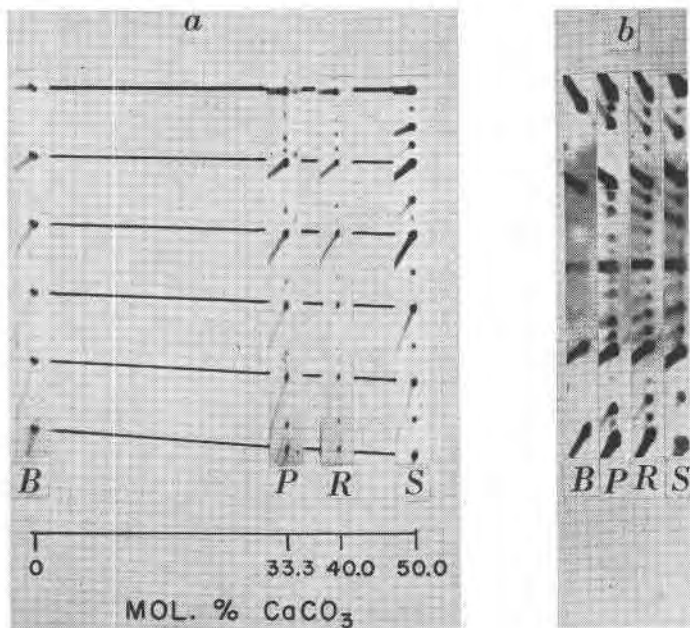


FIG. 2. Reflections  $11\bar{2}l$  showing pseudo-period *c'*. (MoK $\alpha$  radiation; about 24-hour exposures.) (a) Reciprocal lattice rows from precession films mounted against composition to show linear variation of *c'''*. (b) Row lines of *c* axis rotation photographs.

The *a* lengths,<sup>2</sup> accurately determined from precession films, differ very little from species to species (Table 1). (2) The strongest layer lines (reciprocal-lattice nets perpendicular to *c*) correspond to a pseudo-period *c'''* decreasing from 4.9 to 4.6 Å from bastnaesite to synchisite. (3) An-

<sup>2</sup> Oftedal recognized the true *a* of bastnaesite (1931a), but erroneously ascribed the faint row lines shown by parisite rotation patterns to a parallel intergrowth with a hypothetical substance, which he also postulated in synchisite (1931b, p. 444). Strunz (1949, p. 130) correctly transformed Oftedal's cells to the alternate setting, presumably to bring out the relationship with bastnaesite.

TABLE 1. CRYSTAL DATA  
 (All lengths expressed in Å units and known to  $\pm 0.3\%$ )

	Bastnaesite CeFCO <sub>3</sub>	Parisite 2CeFCO <sub>3</sub> · CaCO <sub>3</sub>	Roentgenite 3CeFCO <sub>3</sub> · 2CaCO <sub>3</sub>	Synchisite CeFCO <sub>3</sub> · CaCO <sub>3</sub>
$a'$	4.13 <sub>5</sub>	4.14 <sub>5</sub> <sup>a</sup> 4.11 <sub>5</sub> <sup>b</sup>	4.11 <sub>7</sub>	4.10 <sub>3</sub>
$a = a' \sqrt{3}$	7.16 <sub>1</sub>	7.17 <sub>8</sub> <sup>a</sup> 7.12 <sub>5</sub> <sup>b</sup>	7.13 <sub>1</sub>	7.10 <sub>7</sub>
$c''$	4.89 <sub>4</sub>	4.67 <sub>3</sub>	4.62 <sub>7</sub>	4.56 <sub>0</sub>
$c''$	—	14.0 <sub>2</sub> $c'' = 3c'''$	—	9.12 <sub>0</sub> $c'' = 2c'''$
$c'$	—	28.0 <sub>1</sub> $c' = 6c'''$	23.1 <sub>4</sub> $c' = 5c'''$	18.2 <sub>4</sub> $c' = 4c'''$
$c$	9.78 <sub>7</sub> $c = 2c'''$	84.1 <sub>1</sub> $c = 18c'''$	69.4 <sub>1</sub> $c = 15c'''$	54.7 <sub>2</sub> $c = 12c'''$
$c/a$	1.36 <sub>3</sub>	11.7 <sub>5</sub> <sup>a</sup> 11.8 <sub>5</sub> <sup>b</sup>	9.73 <sub>3</sub>	7.69 <sub>6</sub>
Diffraction aspect (pseudo-cell $a'c'$ )	C***	C***	C***	C***
Diffraction aspect (cell $ac$ )	C**c	R**	R**	—
Laue class (cell $ac$ )	$\frac{6}{m} \frac{2}{m} \frac{2}{m}$	$\bar{3}$	$\bar{3}$	—
Space group (cell $ac$ )	C62c	R3	R3	Orthorhombic or monoclinic
Vol. of unit cell, V	217.4 × 2	208.4 × 18 <sup>a</sup> 205.4 × 18 <sup>b</sup>	203.8 × 15	199.5 × 12
Molecular weight, MW	219.14	538.37	857.60	319.23
Formula units/cell, Z	6	18	9	18
Spec. grav., G(calc.)	5.02	4.29 <sup>a</sup> 4.35 <sup>b</sup>	4.19	3.99
Spec. grav., G(obs.) <sup>e</sup>	4.78 ± 0.1 4.99 <sup>d</sup> 4.9–5.2 <sup>e</sup>	4.05, 4.26, 4.39, 4.41, all ± 0.1	—	4.05, 4.15, all ± 0.1 3.902 <sup>f</sup>

<sup>a</sup> Ordered.

<sup>b</sup> Disordered.

<sup>c</sup> Data probably obtained on polycrystals.

<sup>d</sup> Glass and Smalley (1945).

<sup>e</sup> Palache *et al.* (1951, p. 289).

<sup>f</sup> Flink (1901).

other well-marked pseudo-period,  $c''$ , is found in two of the species: in parisite,  $c'' = 3c'''$ , and in synchisite,  $c'' = 2c'''$ . (4) Every species save bastnaesite possesses yet another pseudo-period,  $c'$ , which is not so easily recognized as  $c''$  and is best seen on the first strong row line, consisting of reflections  $11\bar{2}l$  (Fig. 2). Between the zero and first  $c'''$  layer line no such reflection appears on the bastnaesite pattern ( $c''' = c'$ ); five reflections of medium-strong intensity appear on the parisite pattern

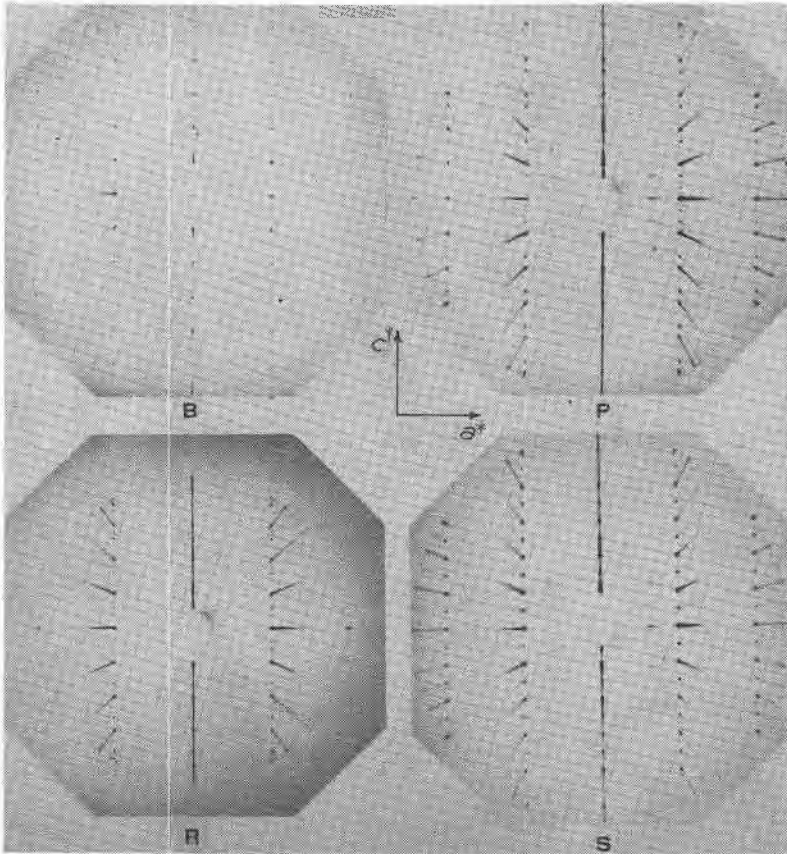


FIG. 3. Precession films of reciprocal lattice planes (010)\*. Faint reflections, which lead to the true  $c$  period, appear on the row lines parallel to  $c^*$  for which  $h \neq 3n$ : (MoK $\alpha$  radiation; 24-hour exposures.)

( $c' = 6c'''$ ); four on the roentgenite pattern ( $c' = 5c'''$ ), and three on the synchisite pattern ( $c' = 4c'''$ ). The true  $c$  values can be obtained from reflections that appear only on the faint row lines. They were measured on precession films exposed for about 24 hours (Fig. 3). For bastnaesite,  $c = 2c'$ . For the other three species  $c = 3c'$ . In terms of  $c'''$ ,  $c = 18c'''$  for parisite,  $c = 15c'''$  for roentgenite, and  $c = 12c'''$  for synchisite.

The chemical compositions of only bastnaesite,  $\text{CeFCO}_3$ , and parisite,  $2\text{CeFCO}_3 \cdot \text{CaCO}_3$ , were well established when this study was begun (Palache, Berman and Frondel, 1951, p. 289, 282). With these formulas and the observed specific gravities (Table 1), the number of formula

units in the true cell is found to be 6 for bastnaesite and 18 for parisite.

The diffraction aspect of every pseudo-cell  $a'c'$  is  $C^{***}$ ; that of the true cell  $ac$  is  $C^{**}c$  for bastnaesite and  $R^{**}$  for parisite and roentgenite. The Laue class is  $6/m\ 2/m\ 2/m$  for bastnaesite and  $\bar{3}$  for parisite and roentgenite. The symmetry of synchisite is lower than hexagonal, either orthorhombic C-centered or monoclinic with an angle of  $120^\circ$ ; even 40-hour exposures of upper levels on the precession camera did not bring out the faint reflections sufficiently to enable a decision to be reached. Morphological observations in the literature (Palache and Warren, 1911, Fig. 3), however, can be interpreted as due to orthorhombic symmetry. Bastnaesite is very strongly piezoelectric (Wooster, in Oftedal, 1931a, p. 465), so that the absence of a center of symmetry in its space group is established. The structural resemblance of the other three minerals to bastnaesite rules out centrosymmetric space groups for all of them. The symmetry of the true cell,  $C\bar{6}2c$  ( $D_{3h}^4$ ) for bastnaesite,  $R3$  ( $C_3^4$ ) for parisite and roentgenite, is obtained from structural considerations (see below, under crystal structure).

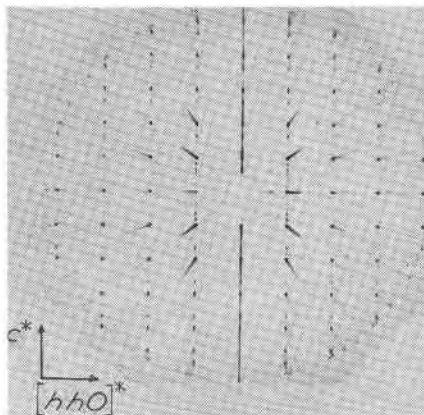


FIG. 4. Precession photograph of disordered parisite crystal from Quincy, Mass. (U. S. Nat. Mus. R2610). Note streaks, in addition to sharp reflections, parallel to  $c^*$ . (MoK $\alpha$  radiation;  $16\frac{1}{2}$ -hour exposure.)

Two parisite crystals give diffraction patterns on which diffuse reflections, drawn out parallel to  $c^*$ , are superposed on the usual sharp reflections of every reciprocal-lattice row parallel to  $c^*$  (Fig. 4). Jagodzinski (1949) has shown that such a pattern is produced by disorder in the sequence of chemically different ordered layers. In this case the layers are perpendicular to  $c^*$ . Thus the two crystals examined belong to a dis-

ordered form. It is probable that they crystallized at elevated temperature. The  $c$  length is the same for the disordered form as for the ordered one, but the  $a$  lengths show a difference which, although small, is beyond experimental error (Table 1). It is probably accidental that disorder was observed in parisite only; roentgenite can be expected to show it too.

#### SYNTAXIC INTERGROWTHS

The term *syntaxy* was coined by Ungemach (1935a, p. 184 and 187) by analogy with the term *epitaxy* previously introduced by Royer. Whereas epitaxy referred to an oriented overgrowth of one substance crystallizing on another, syntaxy was meant to designate the oriented intergrowth of two *chemically identical substances* alternating with each other, but having crystallized simultaneously, whose corresponding cell edges are in the ratio of small integers. Ungemach coined the term to describe the intergrowths of coquimbite and paracoquimbite (1935a, p. 165–190). As the surfaces of junction between constituents are planes (0001), he considered this kind of intergrowth a special case of epitaxy; in fact he writes (1935a, p. 184) that if the two minerals constituting the edifice were of different chemical compositions, he would consider the assemblage an example of repeated epitaxy.

In a subsequent paper Ungemach (1935b) looks for other examples of syntaxy. He stresses the fact that the phenomenon was already known in carborundum. He then describes parisite-synchisite intergrowths in great detail (1935b, pp. 6–18) and attempts to prove that they are syntaxic intergrowths, in his sense of the word. In effect he feels that the very occurrence of the intergrowths is in itself proof that the chemical compositions of the constituents must be identical. He, who as early as 1925 had suggested that synchisite as originally described should be split into two species (one of them parisite), now reverses his previous conclusion. A very searching morphological analysis leads him to distinguish three "polymorphs": "parisite  $\alpha$ " (= synchisite), "parisite  $\gamma$ " (= parisite), and "parisite  $\beta$ ," which we have been able to identify as being roentgenite.

Ungemach's definition of syntaxic intergrowth seems to be unduly restrictive, as this kind of intergrowth is found to occur also with *constituent substances that are chemically different*. We therefore propose to abandon the condition of identity of chemical compositions. Henceforth we shall use the term *syntaxy* in this extended meaning.

A large proportion of the "crystals" examined give x-ray patterns (Fig. 5) that reveal the co-existence of *two* crystalline species in syntaxic intergrowth. Indeed, the specimens discussed above, which give the diffraction pattern of a single species, may do so because only their very tip



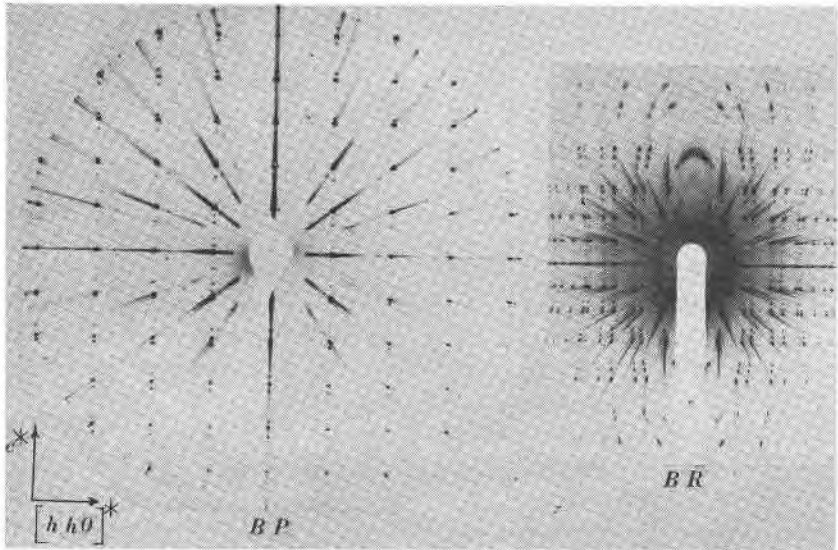


FIG. 5. Diffraction patterns of polycrystals. Left: Precession photograph of bastnaesite-parisite. Right:  $c$ -axis rotation photograph of bastnaesite-roentgenite. (MoK $\alpha$  radiation; 20-hour exposures.)

is bathed in the  $x$ -ray beam. There is morphological evidence that many of these specimens are also intergrowths. With the exception of the bastnaesite-synchisite combination, which has never been observed, all possible pairs have been found. It appears therefore that, in the group of minerals under investigation, syntactic intergrowth can occur when the pseudo-periods  $c'''$  differ by as much as 5.5%, but it no longer occurs when the difference reaches 7.0%. The mutual orientation of the two species consists in a perfect parallelism of their coordinate axes. The  $a$  axes of the two species may have the same sense or opposite senses. In the latter case the two species are related to each other, like the two individuals of a twin, by a  $60^\circ$ -rotation about the 3-fold axis  $c$ . A parisite-roentgenite polycrystal (Harvard Museum No. 84233, labeled "synchisite" from Narsarsuk) showed this phenomenon. The  $x$ -ray reflections are sharp spots, indicating that each individual crystalline domain is at least 0.1 micron across. Streaks parallel to  $c^*$ , like those observed for pure parisite, were produced by one specimen from Montana (U. S. National Museum No. R84440), which was identified as an intergrowth of parisite and roentgenite. Whether the disorder occurs in the parisite, in the roentgenite, or in both was not determined.

Morphologically many of these intergrowths are not readily distinguishable from single crystals and have in fact often been referred to as

"crystals" in the literature. As they are not homogeneous solids, this usage is unfortunate. To such an edifice, composed of two or more structurally distinct species in syntaxial intergrowth and simulating a single crystal, it has been found convenient to apply the term "polycrystal" (Gabrielle Donnay, 1953a).

The confusion that characterizes many morphological descriptions and which is emphasized by the etymology of the name synchisite—from the Greek word for "confounding"—is due to the prevalence of polycrystals. Most of the specimens lent to us, even carefully measured "crystals" described in the literature, were found on *x*-ray examination to be mislabeled. The morphology of these intergrowths is fully described by Ungemach (1935b); it will be explained in the section on morphology.

Occasionally an intergrowth is recognizable as such. In one case (Harvard Museum No. 84233 Narsarsuk) a parisite rhombohedron was attached to a bastnaesite prism. The surface of contact, henceforth referred to as interface, was planar but not a parting plane. The crystals were separated by a method described below. The other case is a group of a dozen intergrowths from a hand specimen of the U. S. National Museum (cordylite, R2613, Narsarsuk). Each intergrowth consists of a pyramid topped by a thin plate. The appearance, on a vastly smaller scale, recalls that of a tiny erosion pillar capped by a miniature mesa. The pyramid is a polycrystal of parisite and roentgenite, the plate is bastnaesite. Attempts to isolate the plate were unsuccessful because again the interface was not a plane of parting.

There exists a simple method of studying the physical nature of the intergrowth, based on the known differential solubility of the constituents. Bastnaesite is insoluble in concentrated cold acid, parisite dissolves slowly in it, roentgenite dissolves readily, and synchisite very quickly, even in dilute acid. The bastnaesite plate comes off easily when the intergrowth is dipped in concentrated nitric acid for a few minutes. Many of the polycrystals were observed under a binocular microscope while submerged in acid and afterwards again studied by *x*-rays. The dissolving of one of the forms could thus be verified. Two different types of syntaxial intergrowth appear, often on one and the same polycrystal. The one type (Fig. 6a) may be described as two-dimensional, because the interface is planar, parallel to (0001). The constituent layers vary from very thin to thick. This type of intergrowth is to be expected because the interface is the plane controlled only by the *a* axes, which are nearly identical for any two species. The second type (Fig. 6b) is three-dimensional; that is, the interface is irregular. By dissolving out the more soluble constituent, the acid leaves a deep crevice or a "drill hole" randomly oriented with respect to crystallographic directions.

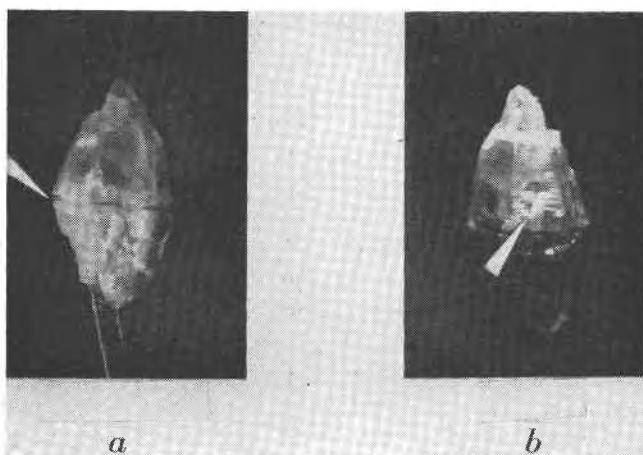


FIG. 6. Roentgenite-synchisite from Narsarsuk, Greenland. The polycrystal was dipped into dilute nitric acid for 10 minutes and synchisite dissolved out. (a) Two-dimensional intergrowth; interface is planar. (b) Three-dimensional intergrowth; interface is irregular.

The frequent occurrence of polycrystals is an expression of the inability of the system  $\text{CeFCO}_3\text{-CaCO}_3$  to form solid solutions. One crystal precipitates until the conditions in the solution have changed sufficiently for the next compound to separate out. The latter crystallizes on the original crystal. The two species alternate, so that periodic changes in the conditions of the system must be postulated. The two species do not necessarily have neighboring compositions on the phase diagram. These observations require conditions to change not only periodically but also discontinuously. Variations in the pH of the solution may well be the controlling factor during crystal growth, and dissolution of a large part of the polycrystal may account for the mushroom-like specimens. They would, at one time, have had the appearance of a single crystal yet have consisted of three species, a situation which would be expected to occur frequently but which has not been observed.

Another intergrowth of bastnaesite, namely with fluocerite (tysonite),  $\text{CeF}_3$ , has been described by Oftedal (1931c). Bastnaesite, in this case, is an alteration product of fluocerite.

#### THE CHEMICAL COMPOSITIONS OF ROENTGENITE AND SYNCHISITE

Because of the predominant occurrence of polycrystals, it would have been necessary to examine innumerable small crystals by  $x$ -rays in order to obtain enough pure material for chemical or spectroscopic analysis. Practical considerations, among them a lack of specimens, rendered such

a procedure impossible. Crystallographic deductions, however, led to the chemical formulas.

All reflections  $hkl$  with  $(h-k)$  divisible by 3 are at least 100 times as intense as those for which  $(h-k)$  is not divisible by 3. It is safe to infer that the heavy cerium and calcium atoms do not contribute to the weak reflections and are therefore responsible for the pseudo-H-centering of the cell, which gives rise to the pronounced pseudo-period  $a'$ , common to all four species. In other words, for every cerium or calcium atom in any site  $x, y, z$ , there must be two other atoms of the same metal at  $x+\frac{2}{3}, y+\frac{1}{3}, z$  and  $x+\frac{1}{3}, y+\frac{2}{3}, z$ . The structure thus contains horizontal layers of calcium and cerium for each other is ruled out because it would result in an increased intensity of the weak reflections. The pronounced pseudo-period  $c'$  indicates that the heavy atoms repeat along the  $z$  axis at intervals close in length to  $c'$ . In consequence the number of cerium atoms and that of calcium atoms in the pseudo-cell  $ac'$  must be multiples of 3.

The possibility must be considered that roentgenite and synchisite might be polymorphic forms of parisite, so that their compositions would

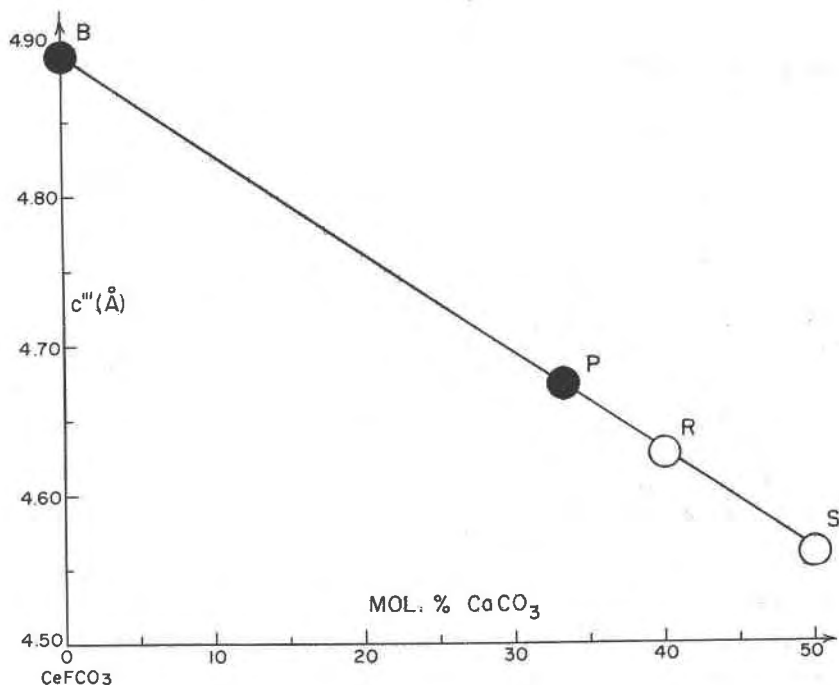


FIG. 7. Pseudo-period  $c'''(\text{Å})$  plotted against composition expressed in mol. %  $\text{CaCO}_3$ . Black circles used to construct line; size of circles gives uncertainty of measurements.

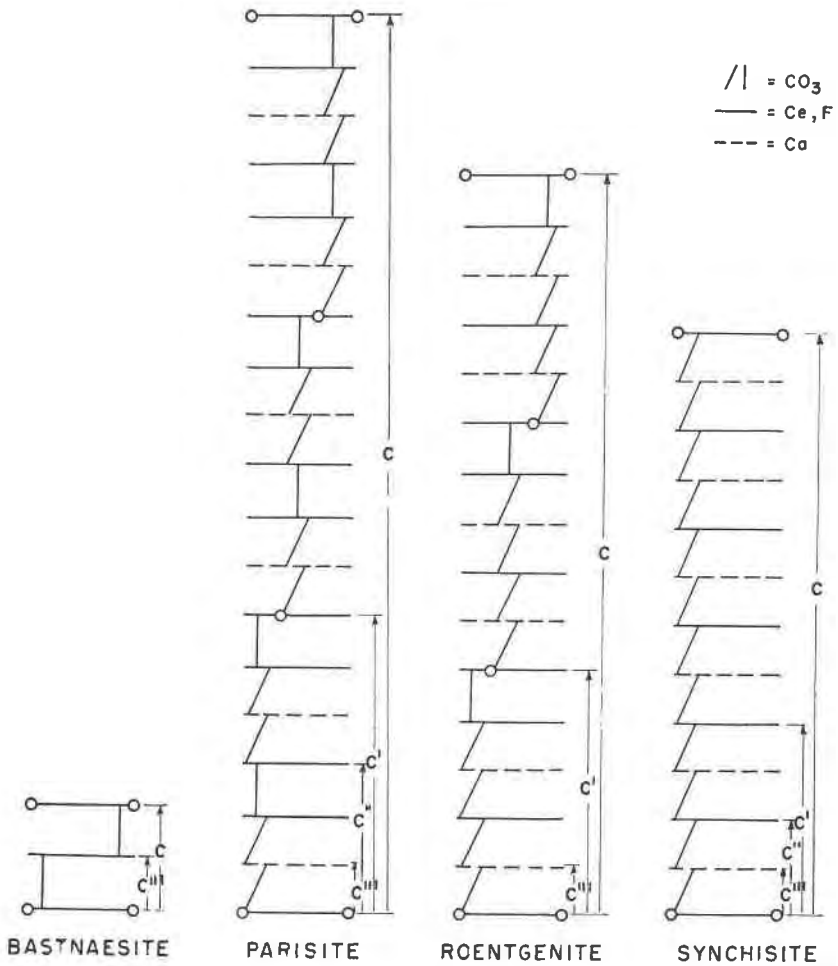


FIG. 8. Schematic representation of crystal structures. Rhombohedral lattice points are shown along diagonal of the rectangle.

be  $\text{Ce}_2\text{CaF}_2(\text{CO}_3)_3$  or very close to it. With the measured specific gravities (Table 1), the contents of the cell  $ac'$  turn out to be  $\text{Ce}_{10}\text{Ca}_5\text{F}_{10}(\text{CO}_3)_{15}$  for roentgenite and  $\text{Ce}_8\text{Ca}_4\text{F}_8(\text{CO}_3)_{12}$  for synchisite. Neither 10, nor 5, nor 8, nor 4 is a multiple of 3! The hypothesis of polymorphism is thus ruled out.

How does the cell volume vary with composition? The smallest cell, namely  $a'c'''$ , is used as it probably contains the same number of cations in every species. The variation in  $a'$  from mineral to mineral is insignificant but that of  $c'''$  is not. Consequently  $c'''$  is plotted against composi-

tion, the latter being expressed in mol. %  $\text{CaCO}_3$  (Fig. 7). A straight line is drawn through the two known points corresponding to bastnaesite and parisite. The observed  $c'''$  value of roentgenite, when placed on this line, gives a composition of 40.0 mol. %  $\text{CaCO}_3$ ; that of synchisite gives 50.0%  $\text{CaCO}_3$ . In order to check the integral values found graphically,  $c'''$  values for these compositions are calculated from the equation of the line. For  $3\text{CeFeCO}_3 \cdot 2\text{CaCO}_3$  the calculated  $c'''$  is 4.629 Å as compared with the observed value of  $4.627 \pm 0.015$  Å; the calculated  $c'''$  value for  $\text{CeFeCO}_3 \cdot \text{CaCO}_3$  is 4.563 Å as compared with  $4.560 \pm 0.015$  Å.

The contents of the  $ac'$  cells calculated with the formulas thus obtained lead to 5 layers, 3 of cerium and 2 of calcium, in roentgenite; and 4 layers, 2 of cerium and 2 of calcium, in synchisite (Fig. 8). The observed pseudo-periods  $c'$  are thus explained, a fact which in itself is a confirmation of the chemical formulas. The chemical formulas are further supported by the excellent agreement between calculated and observed indices of refraction (to be discussed below). The comparison of observed and calculated specific gravities, commonly used to check the composition, cannot be relied on, because specimens large enough to be weighed on the Berman balance are likely to be polycrystals. The wide range of densities reported for bastnaesite and parisite (Table 1) bears out this statement.

### CRYSTAL STRUCTURE

The structural scheme of bastnaesite, parisite, and synchisite was predicted by Oftedal (1931a, b), and our structural hypothesis is essentially in agreement with his. He refers bastnaesite to the true cell  $ac$  and gives the correct space group  $C\bar{6}2c$ , which is derived from the morphological point group  $\bar{6}2m$  and the space group extinctions ( $hh2hl$  absent when  $l$

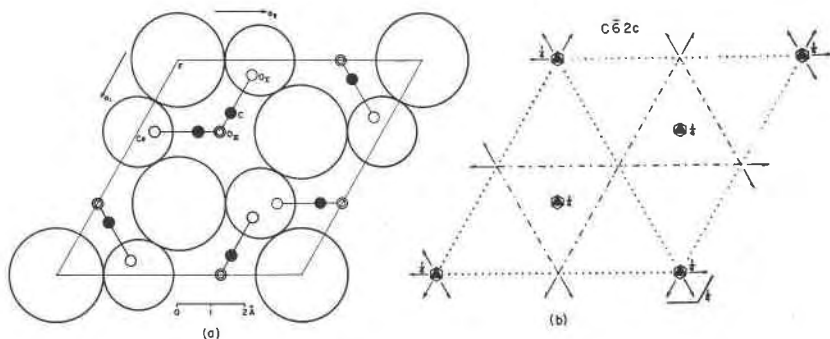


FIG. 9. (a) Fluorine-cerium layer at levels 0 and  $\frac{1}{2}$  schematically showing positions of carbonate groups at heights  $\frac{1}{4}$  (faint line) and  $\frac{3}{4}$  (heavy line). (b) Space-group symmetry of  $C\bar{6}2c$ .

is odd). He refers parisite and synchisite to pseudo-cells, namely cells  $a'c'$ , but he does not specify the space groups.

In all four species cerium and fluorine ions alternate at the vertices of regular hexagons (Fig. 9). With the radii given in the literature,  $r(\text{Ce}^{3+}) = 1.02 \text{ \AA}$  (Zachariasen, 1948) and  $r(\text{F}^-) = 1.36 \text{ \AA}$  (Wells, 1950, p. 70), the side of the hexagon has length  $1.02 + 1.36 = 2.38 \text{ \AA}$  and the predicted cell edge  $a$  is  $3 \times 2.38 = 7.14 \text{ \AA}$ . The observed  $a$  ranges from  $7.11 \pm 0.02 \text{ \AA}$  for synchisite to  $7.18 \pm 0.02 \text{ \AA}$  for parisite. In the crystal structures of parisite, roentgenite, and synchisite, calcium layers alternate with cer-

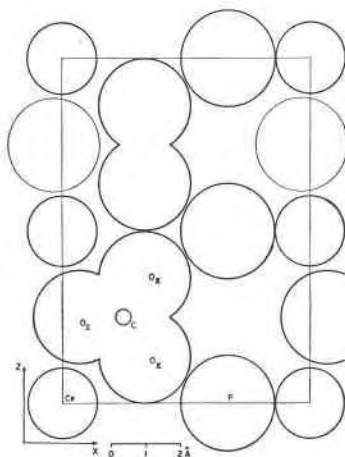


FIG. 10. Vertical section of bastnaesite structure by plane  $y = \frac{1}{3}$ . Light circles show neighboring oxygen atoms.

ium-fluorine layers in various proportions (Fig. 8). Calcium ions are placed above cerium ions. The disordered crystal structures of parisite (Fig. 4) and of the parisite-roentgenite polycrystal result from irregularities in the sequence of these layers. The decreased  $a$  value of the disordered parisite may be ascribed to a slight displacement of fluorine and cerium with respect to each other, so they no longer lie in one horizontal plane. Bastnaesite cannot show disorder of this kind as it contains only cerium-fluorine layers. In synchisite the disordered form would have to contain calcium layers adjacent to each other; the nearly vertical positions of the carbonate groups between the layers (see below) makes such an arrangement very unlikely.

Oftedal refers the crystal structure of parisite to a pseudocell ( $a'$ ,  $c'/2$ ) that contains one formula unit of  $2\text{CeFCO}_3 \cdot \text{CaCO}_3$ . He states (1931b, p. 448, last line) that calcium at the origin is the only choice permitted by all possible space groups:  $\overline{C}6m2(D_{3h}^1)$ ,  $\overline{C}62m(D_{3h}^3)$ ,  $C6mm$

( $C_{6v}^1$ ),  $C622(D_6^1)$ , and  $C6/mmm(D_{6h}^1)$ . He overlooks the fact that  $C\bar{6}m2$  offers the one-fold position  $1(c): \frac{1}{3}, \frac{2}{3}, 0$ . He places cerium (not fluorine) above the origin, at  $00u$  in parisite, at  $00\frac{1}{4}$  in synchisite. In his paper on bastnaesite he stated that interchanging the cerium and the fluorine ions

TABLE 2. ATOMIC PARAMETERS PREDICTED FOR BASTNAESITE  
Space Group  $C\bar{6}2c (D_{3d}^4)$ ;  $6CeFCO_3$  per Cell

Atom	Position	Atomic sites	Atomic parameters	
			Oftedal	Donnay
F	2(a)	0, 0, 0; 0, 0, $\frac{1}{2}$	—	—
F	4(f)	$\frac{1}{3}, \frac{2}{3}, z; \frac{2}{3}, \frac{1}{3}, \bar{z}; \frac{2}{3}, \frac{1}{3}, \frac{1}{2}+z; \frac{1}{3}, \frac{2}{3}, \frac{1}{2}-z$ .	$z \approx 0$	$z \approx 0$
Ce	6(g)	$x, 0, 0; 0, x, 0; \bar{x}, x, 0;$ $x, 0, \frac{1}{2}; 0, x, \frac{1}{2}; \bar{x}, \bar{x}, \frac{1}{2}$ .	$x = \frac{1}{3}$	$x = 0.333$
C	6(h)	$x, y, \frac{1}{4}; \bar{y}, x-y, \frac{1}{4}; y-x, \bar{x}, \frac{1}{4};$ $y, x, \frac{3}{4}; \bar{x}, y-x, \frac{3}{4}; x-y, \bar{y}, \frac{3}{4}$ .	—	$x = 0.245; y = 0.333$
O <sub>I</sub>	6(h)	as above	—	$x = 0.067; y = 0.333$
O <sub>II</sub>	12(i)	$x, y, z; \bar{y}, x-y, z; y-x, \bar{x}, z;$ $x, y, \frac{1}{2}-z; \bar{y}, x-y, \frac{1}{2}-z; y-x, \bar{x}, \frac{1}{2}-z;$ $y, x, \frac{1}{2}+z; \bar{x}, y-x, \frac{1}{2}+z; x-y, \bar{y}, \frac{1}{2}+z;$ $y, x, \bar{z}; \bar{x}, y-x, z; x-y, \bar{y}, \bar{z}$ .	—	$x = 0.333; y = 0.333$ $z = 0.138$

would not appreciably alter his proposed structure (1931a, p. 469). We are very grateful to H.T. Evans, of the Geological Survey, for pointing out to us that the two resulting structures differ radically. Indeed with cerium above the origin no structure will give acceptable interatomic distances for any of the species, whereas with fluorine above the origin the intensities of medium-strong and strong reflections are accounted for and reasonable interatomic distances are obtained (Table 3). The frequent occurrence of intergrowths also supports the identical choice of origin in the four crystal structures, because with this choice a number of cerium-fluorine layers can extend throughout the polycrystal (Fig. 6).

As the symmetry of bastnaesite is such that, with the help of known ionic radii, one can predict the coordinates of *all* the atoms (Table 2), we shall discuss this structure in detail. There are six formula units of  $CeFCO_3$  per cell. Fluorine ions are placed at 2(a): 0, 0, 0 and 4(f):  $\frac{1}{3}, \frac{2}{3}, z$ , with  $z = 0.0$  (Int. Tab. 1935, I, 279); cerium ions are in position 6(g):  $x, 0, 0$  with  $x = 0.333$ . The three oxygen atoms of the carbonate group are assumed to be at the vertices of an equilateral triangle, with carbon at the center and the carbon-oxygen distance  $d_{C-O}$  equal to 1.27 Å (Zachariasen, 1933). The six carbon atoms and six of the oxygen atoms, referred to as O<sub>I</sub>'s, can only be placed in positions 6(h):  $x, y, \frac{1}{4}$ , on the horizontal mirrors. The remaining twelve oxygen atoms lie in 12 (i):  $x, y, z$ . Let us



TABLE 3. INTERATOMIC DISTANCES PREDICTED FOR BASTNAESITE  
(Lengths in Å units)

Ce-F	2.39
Ce-O <sub>I</sub>	2.49
Ce-O <sub>II</sub>	2.74
F-O <sub>II</sub>	2.74
C-O <sub>I</sub> or O <sub>II</sub> (intra-ionic)	1.27
O <sub>II</sub> -O <sub>I</sub> or O <sub>II</sub> (intra-ionic)	2.19
O <sub>II</sub> -O <sub>II</sub> (inter-ionic)	2.70

The same distances are also predicted for corresponding layers in the structures of parisite and roentgenite.

call them O<sub>II</sub>. From the size of the carbonate group, their  $z$  parameter is fixed at 0.138. O<sub>II</sub> is placed on the vertical line through the center of the hexagon outlined by fluorine and cerium (Fig. 10), namely at  $x=0.333$ ,  $y=0.333$ ,  $z=0.138$ , O<sub>II</sub> is thus at a distance of 2.74 Å from each of the three fluorine ions. Using the fluorine radius of 1.36 Å, the oxygen radius found by subtraction is 1.38 Å, a value that falls well within the range reported in the literature (Evans, 1946, p. 171, gives 1.32 Å; Wells, 1950, p. 70, gives 1.40 Å, for the radius of O<sup>=</sup> in 6-fold coordination). Any displacement of O<sub>II</sub> in  $x$  or  $y$  would decrease one of the fluorine-oxygen distances, but any distance smaller than 2.74 Å is unlikely. The O<sub>II</sub>'s should thus lie on (or very nearly on) the  $c$  glide plane. As far as they are concerned, the glide reduces to a translation  $c/2$ . In consequence the O<sub>II</sub>'s will not contribute to reflections  $hkl$  with  $l$  odd; only O<sub>I</sub> and carbon are responsible for these reflections. This conclusion agrees with the observation that the ratio of faint to strong intensities is considerably smaller for bastnaesite than for the other species, in which some of the O<sub>II</sub>'s do contribute to the faint reflections. Equating the height of the carbonate group to the pseudo-period  $c'''$  yields an oxygen radius for O<sub>II</sub> equal to 1.35 Å (Fig. 10).

The distance between O<sub>II</sub> and Ce is, of course, the same as between O<sub>II</sub> and F, namely 2.74 Å. This is a large value, as the sum of the ionic radii of O<sup>=</sup> and Ce<sup>3+</sup> is only 2.40 Å. The bonds Ce-O<sub>II</sub> must therefore be weak ones; to a first approximation, they may be neglected. Ce is thus left with two positive charges after we subtract the three bonds of strength  $\frac{1}{3}+$  each, which go to the neighboring fluorine ions. Cerium can therefore be expected to attract O<sub>I</sub> as close to itself as possible. In other words C and O<sub>I</sub> will, in horizontal projection, lie on a straight line O<sub>II</sub>-Ce.

There are three such lines: two of them parallel to the cell edges and the third one coincident with the trace of the *c* glide plane. Placing the carbonate group on the *c* glide plane would mean that it would be repeated, by translation *c*/2, in the upper half of the cell. The cell height would then have to be *c*/2 instead of *c*, which would be contrary to fact. The carbonate group will, therefore, be projected on a line parallel to a cell edge; another carbonate group, related to the first one by a glide-reflection, will be projected parallel to the other cell edge. C is placed at *x* = 0.245, *y* = 0.333, *z* =  $\frac{1}{4}$  and O<sub>I</sub> at *x* = 0.067, *y* = 0.333, *z* =  $\frac{1}{4}$ . The Ce—O<sub>I</sub> distance is 2.49 Å. The Ce—O<sub>I</sub> bonds form vertical zigzag chains: one in the plane *y* =  $\frac{1}{3}$ , others in the planes symmetrical to it (Fig. 10). Again ignoring the bonds Ce—O<sub>II</sub>, each Ce gives two bonds, each of strength 1+, to the O<sub>I</sub>'s, and each O<sub>I</sub> receives two such bonds. Thus the total charge on the carbonate group is neutralized. The strength of these vertical bonds accounts for the observed lack of cleavage (0001). It was not possible to check by structure-factor calculations the proposed orientation of the carbonate groups because the reflections *hkil* with *l* odd are barely visible and the contributions of C and O<sub>I</sub> to the other reflections

TABLE 4. ATOMIC PARAMETERS PREDICTED FOR PARISITE AND ROENTGENITE  
 space groups R3 (*C*<sub>3</sub><sup>4</sup>): 18,[2CeFCO<sub>3</sub>·CaCO<sub>3</sub>] per unit cell of parisite;  
 9[3CeFCO<sub>3</sub>·2CaCO<sub>3</sub>] per unit cell of roentgenite

Atom	Position	Atomic sites (0, 0, 0; $\frac{1}{3}, \frac{2}{3}, \frac{1}{2}$ ; $\frac{2}{3}, \frac{1}{3}, \frac{1}{2}$ ) +	Atomic parameters	
			Parisite	Roentgenite
F	3(a)	0, 0, <i>z</i> .	<i>z</i> = 0; 0.333; 0.667; 0.108; 0.441; 0.775; 0.167; 0.500; 0.834; 0.275; 0.608; 0.942.	<i>z</i> = 0; 0.333; 0.667; 0.131; 0.464; 0.798; 0.263; 0.596; 0.930.
Ce	9(b)	<i>x</i> , <i>y</i> , <i>z</i> ; $\bar{y}$ , <i>x</i> − <i>y</i> , <i>z</i> ; <i>y</i> − <i>x</i> , $\bar{x}$ , <i>z</i> .	<i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.108; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.167; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.275.	<i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.131; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.263.
Ca	9(b)	<i>x</i> , <i>y</i> , <i>z</i> ; $\bar{y}$ , <i>x</i> − <i>y</i> , <i>z</i> ; <i>y</i> − <i>x</i> , $\bar{x}$ , <i>z</i> .	<i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = .054; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.221.	<i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.066; <i>x</i> = 0.333, <i>y</i> = 0, <i>z</i> = 0.197.
C	9(b)	<i>x</i> , <i>y</i> , <i>z</i> ; $\bar{y}$ , <i>x</i> − <i>y</i> , <i>z</i> ; <i>y</i> − <i>x</i> , $\bar{x}$ , <i>z</i> .	<i>x</i> = 0.245, <i>y</i> = 0.333, <i>z</i> = 0.138; <i>x</i> = 0.245, <i>y</i> = 0.333, <i>z</i> = 0.304; <i>x</i> <sub><i>n</i></sub> , <i>y</i> <sub><i>n</i></sub> , <i>z</i> <sub><i>n</i></sub> ; <i>n</i> = 1 to 4.	<i>x</i> = 0.245, <i>y</i> = 0.333, <i>z</i> = 0.298; <i>x</i> <sub><i>n</i></sub> , <i>y</i> <sub><i>n</i></sub> , <i>z</i> <sub><i>n</i></sub> ; <i>n</i> = 1 to 4.
O	9(b)	<i>x</i> , <i>y</i> , <i>z</i> ; $\bar{y}$ , <i>x</i> − <i>y</i> , <i>z</i> ; <i>y</i> − <i>x</i> , $\bar{x}$ , <i>z</i> .	<i>x</i> = 0.067, <i>y</i> = 0.333, <i>z</i> = 0.138; <i>x</i> = 0.067, <i>y</i> = 0.333, <i>z</i> = 0.304; <i>x</i> = 0.333, <i>y</i> = 0.333, <i>z</i> = 0.124; <i>x</i> = 0.333, <i>y</i> = 0.333, <i>z</i> = 0.151; <i>x</i> = 0.333, <i>y</i> = 0.333, <i>z</i> = 0.291; <i>x</i> = 0.333, <i>y</i> = 0.333, <i>z</i> = 0.317; <i>x</i> <sub><i>n</i></sub> , <i>y</i> <sub><i>n</i></sub> , <i>z</i> <sub><i>n</i></sub> ; <i>n</i> = 1 to 12.	<i>x</i> = 0.067, <i>y</i> = 0.333, <i>z</i> = 0.298; <i>x</i> = 0.333, <i>y</i> = 0.333, <i>z</i> = 0.282; <i>x</i> = 0.333, <i>y</i> = 0.333, <i>z</i> = 0.314; <i>x</i> <sub><i>n</i></sub> , <i>y</i> <sub><i>n</i></sub> , <i>z</i> <sub><i>n</i></sub> ; <i>n</i> = 1 to 12.

are insignificant as compared with the contributions of cerium.

For the same reason it is impossible to determine all the atomic coordinates in the three remaining structures. In the cases of parisite and roentgenite, the true space group can be derived as follows. The Laue class is  $\bar{3}$  and the diffraction aspect  $R^{**}$ . The atomic arrangements in the cerium-fluorine layer, in the calcium layer, and in the interlayer rule out the center of symmetry. The space group is uniquely determined as  $R\bar{3}$ .

For the crystal structures of parisite and roentgenite, the atomic coordinates of fluorine, cerium, calcium, and those carbonate groups that lie between two cerium-fluorine layers can be predicted (Table 4). If the distance between cerium-fluorine layers in bastnaesite, equal to 4.89 Å, is assumed to hold true for parisite, roentgenite, and synchisite, the calculated distance between cerium-fluorine layer and calcium layer is 4.56 Å in all three structures. The normal to the plane of a carbonate group between two chemically different layers must therefore make an angle of about  $79^\circ$  with the vertical ( $\sin 11^\circ \approx 4.56/4.89$ ).

Considering the structural scheme of these minerals,  $m\text{CeFCO}_3 \cdot n\text{CaCO}_3$ , one can see no reason why values of  $m/n$  different from  $\infty$ , 2,  $\frac{3}{2}$ , and 1 cannot exist. Even solid solution resulting in a disordered crystal structure can be envisioned. There exists, however, no  $x$ -ray evidence of more than four species. Compositions richer in calcium than synchisite with analogous crystal structures are not to be expected. Their structures would have to contain adjacent calcium layers, and with the nearly vertical arrangement of the carbonate groups, the calcium coordination number would be too low. Carbonate groups parallel to the calcium layers, as for example in calcite, would be predicted.

#### CRYSTAL MORPHOLOGY

Now that the four species can be unequivocally referred to their true cells by  $x$ -rays, it is possible in each case to ascertain which cell governs the morphological development. The goniometric data of Palache and Warren (1911) are interpreted in Table 5. The polycrystals they measured did not contain any bastnaesite, but their measured angles are found among the calculated angles of all three remaining species. In view of the difficulties encountered in measurement, the agreement between their measured angles and our calculated angles is truly remarkable. It bears witness to the skill and accuracy of these old-time crystallographers.

##### *Bastnaesite*

The morphology is controlled by the true cell  $ac$  and has point-group symmetry  $\bar{6}2m$ . Most crystals are tabular on  $c$  {0001}. One crystal from

Gaines Mountain, Muzo, Colombia (Harvard Museum No. 11019), is short prismatic in habit, with a large trigonal prism  $a \{2\bar{1}\bar{1}0\}$ , a small hexagonal prism  $m \{10\bar{1}0\}$ , and three hexagonal dipyramids:  $q \{10\bar{1}1\}$ ,  $p \{10\bar{1}2\}$ ,  $t \{10\bar{1}3\}$ . Measurements from excellent reflections give  $\rho (10\bar{1}1) = 57^\circ 42'$  and  $\rho (10\bar{1}2) = 38^\circ 19'$ , from which we calculate  $c/a = 1.369$ , in agreement with 1.367, the ratio of the cell edges obtained by x-rays.

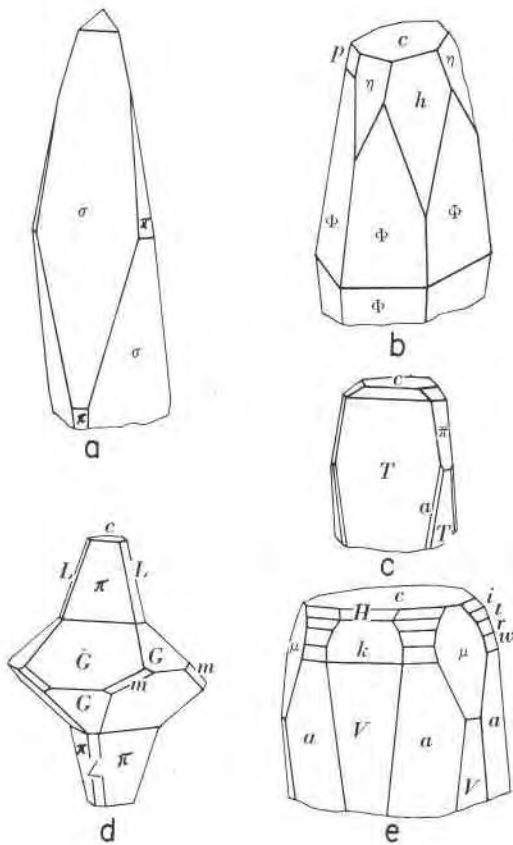


FIG. 11. (a) Parisite (after Palache and Warren, 1911).—(b) Parisite (after Cesàro, 1907).—(c) Roentgenite (after Palache and Warren, 1911).—(d) Intergrowth: synchisite between roentgenite tips (Flink, 1901).—(e) Synchisite (after Palache and Warren, 1911).

The morphological space group is  $C\bar{6}2c(D_{3h}^4)$ , found as follows: the lattice cannot be rhombohedral because the dipyramids violate the R-criterion, “ $(-h+k+l)$  divisible by 3”; the  $c$  glide plane is required by the form  $s \{11\bar{2}2\}$ , which is reported in the literature and obeys the  $L$ -criterion, “ $l$  even.” Palache *et al* (1951, p. 289) erroneously gives  $C\bar{6}c2(D_{3h}^2)$  as the space group, with  $\bar{6}m2(?)$  as the point group.

*Parasite*

There is no obvious morphological feature that would necessitate lowering the point-group symmetry below  $\bar{3}2/m$ . The morphology is *not* controlled by the true cell  $ac$ ; the morphological axial ratio is either  $\frac{1}{2}c/a$  or  $\frac{1}{4}c/a$ . In each case the indices can be made to obey the R-criterion. The series of forms obtained with  $\frac{1}{2}c/a$  (see Table 5) contain several forms that have not been reported by Palache and Warren (1911). The alternate axial ratio,  $\frac{1}{4}c/a$ , would bring about some simplification, but it is difficult to decide between the two. Tentatively we have adopted  $\frac{1}{2}c/a$ . The rhombohedra that are positive with the ratio  $c/a$  or  $\frac{1}{4}c/a$  must become negative with the ratio  $\frac{1}{2}c/a$  in order to obey the R-lattice criterion (see Table 5). For example:  $h\{10\bar{1}4\}$  or  $\{10\bar{1}1\}$  becomes  $\{01\bar{1}2\}$ .

A typical parasite crystal from Quincy Mass. (Fig. 11a), is reproduced from Palache and Warren (1911). Its dominant rhombohedron should be lettered  $\sigma\{10\bar{1}1\}$ . The parasite crystal from Muzo (Fig. 11b) described by Cesàro (1907) can be indexed as follows:  $\phi\{22\bar{4}3\}$ ,  $h\{01\bar{1}2\}$ ,  $\eta\{2025\}$ ,  $\sigma\{10\bar{1}1\}$ ,  $c\{0003\}$ ,  $p\{11\bar{2}3\}$ .

The dominant rhombohedron of a crystal from Narsarsuk, Greenland (Harvard Museum No. 84233) gave a  $\rho$  angle of  $73^\circ 38'$ . It should be indexed  $h\{01\bar{1}2\}$ . It yields an axial ratio equal to  $\frac{1}{2} \times 11.796$ , as compared with 11.722 and 11.807, found by  $x$ -rays for the ordered and the disordered form, respectively, which correspond to  $\rho$  angles of  $73^\circ 32'$  and  $73^\circ 39'$ . We checked by  $x$ -rays that the symbol of this rhombohedron would be  $\{10\bar{1}4\}$  if it were referred to the structural  $c/a$ .

This parasite specimen is twinned by reticular merohedry. The twinned crystal consists of at least three horizontal slices intercalated in the main crystal. The composition surface is planar, parallel to the base. The rhombohedron  $\sigma\{10\bar{1}1\}$ , with measured  $\rho$  of  $81^\circ 35'$ , is brought in zone with  $h\{01\bar{1}2\}$  by twinning.

*Roentgenite*

The rhombohedral lattice obtained by  $x$ -rays manifests itself in the morphology. Referred to the true cell  $ac$ , determined by  $x$ -rays, the forms are:  $T\{10\bar{1}1\}$  dominant,  $\pi\{01\bar{1}2\}$ ,  $c\{0001\}$  subordinate. These forms obey the R-criterion, " $(-h+k+l)$  divisible by 3," provided the base is written  $\{0003\}$ , as it should be, in view of its relatively small size. This indexing of forms is compatible with the diffraction aspect  $R^{**}$ .

The same relative importance of the forms could, however, also be expressed by indexing them as follows:  $T\{01\bar{1}2\}$ ,  $\pi\{10\bar{1}4\}$ ,  $c\{0006\}$ , and by doubling the axial ratio. In this case the aspect would have to be  $R^*c$ .

The morphological  $c$  cannot be twice the structural  $c$ , because crystal

TABLE 5. INTERPRETATION OF MORPHOLOGICAL DATA

Measured $\rho$ angles and no. of crystals (Palache and Warren, 1911)	Calculated $\rho$ angles			Synchisite
	Bastnaesite	Parisite	Roentgenite	
	$\frac{c}{a} = 1.369$	$\frac{1}{2} \left( \frac{c}{a} \right) = \frac{11.722}{2} = \frac{11.807}{2}$ to $\frac{11.807}{2}$	$\frac{c}{a} = 9.732$	$\frac{c''}{a'} = 2.223$
A 29° 9'(57'-35') 4		1.1.2.21 29°10'	0.1.1.20 29°20'	A 1128 29° 5'
B 30°44'(28'-56') 8			1.1.2.33 30°32'	
B 30°34'(24'-49') 11			30°36'	
C 31°41'(30'-50') 9		C 0.1.1.11 31°36'-47'	B 1.0.1.19	
$\gamma$ 32°29'(20'-41') 8				$\gamma$ 1127 32°25' 32°41'
D 33°36'(20'-51') 4		1.1.2.18 33° 7'-16'	1.1.2.30 32°59'	
$\delta$ 33°45'(22'-34') 19		$\delta$ 1.0.1.10 34° 5'-17'	D 0.1.1.17 33°28'	
E 34°57'(20'-80') 10	1124 34°25'		E 1.0.1.16 35° 5'	
F 36°54'(. . . . .) 3		1.1.2.15 38° 0'-13'	1.1.2.27 35°48'	F 1126 36°33'
$\epsilon$ 38°47'(25'-62') 23	1012 38°20'		$\epsilon$ 0.1.1.14 38°45'	
d 40° 2'(53'-21') 1		d 0118 40°13'-27'	1.1.2.24 39° 3'	
G 40°48'(40'-56') 6			G 1.0.1.13 40°51'	G 1013 40°33'
H 41°40'(25'-58') 8	1123 42°24'	1017 44° 2'-13' 44°20'-33'	1.1.2.21 42°50'	H 1125 41°39'
b 45°47'(. . . . .) 1			0.1.1.11 45°37'	b 2025 45°45'

TABLE 5. (Continued)

Measured $\rho$ angles and no. of crystals (Palache and Warren, 1911)	Calculated $\rho$ angles			
	Bastnaesite	Parisite	Roentgenite	Synchisite
	$\frac{c}{a} = 1.369$	$\frac{1}{2} \left( \frac{c}{a} \right) = \frac{11.722}{2}$ to $\frac{11.807}{2}$	$\frac{c}{a} = 9.732$	$\frac{c''}{a'} = 2.223$
2023	46°31'		1.1.2.18 47°15'	} 1124 5
e 48°4'(54'-14') 5				
f 47°54'(47'-60') 5				
i 52°10'(45'-36') 18		i 1129 52°30'-42' 53°33'-45'	1.0.1.10 i 1.1.2.15 48°20' 52°21'	i 1012 52°5'
theta 54°35'(13'-52') 4	1122 53°52'		theta 0118 54°35'	1123 55°59'
l 57°33'(30'-43') 4		l 2.2.4.15 57°24'-36'		
I 58°21'(18'-24') 5	1011 57°42'		I 1017 58°5'	
n 58°23'(11'-36') 9			n 1.1.2.12 58°21'	
f 59°44'(. . . . .) 3				
l 59°45'(34'-60') 10		f 1014 59°24'-36'		
J 62°54'(43'-58') 3		J 0227 62°39'-49'		i 2023 59°42'
q 62°47'(. . . . .) 4		q 1126 62°54'-64'		
4043	64°38'			
u 65°14'(11'-18') 2				
g 65°53'(48'-57') 3			g 0115 65°11' 66°1'	g 1122 65°47'
r 68°48'(36'-60') 10		r 2249 69°0'-8' 69°44'-52'		r 1011 68°43'
eta 69°47'(42'-52') 1		eta 2025		
L 70°22'(56'-40') 5	1121 69°57'		L 1014 70°25' lambda 0.3.3.12 70°25'	
lambda 70°39'(26'-53') 1				





structure restricts symmetry more—not less—than crystal morphology. It is difficult to see, from the structural scheme considered above, how  $a/2$  could be a pseudo-period. It follows that the morphological  $c/a$  is the same as the structural  $c/a$ . The series of forms obtained with the structural  $c/a$  (Table 5) are satisfactory.

The angle measurement on a crystal from Narsarsuk (U. S. National Museum No. R 2613) were poor on T faces, good on  $\pi$  faces. Whereas the axial ratio  $c/a=9.733$  obtained by  $x$ -rays requires  $\rho$  (01 $\bar{1}2$ ) to be  $79^\circ54'$ , the measured value is equal to  $79^\circ55\frac{1}{2}' \pm 2'$ . The corresponding morphological ratio is  $9.749 \pm 0.033$ . The value of  $\rho$  (10 $\bar{1}1$ ), calculated from  $c/a=9.733$  is  $84^\circ55'$ .

One of the crystal drawings of Palache and Warren (1911), reproduced here as figure 11c, shows a specimen that is essentially roentgenite, with T {10 $\bar{1}1$ } dominant, truncated by  $\pi$  {01 $\bar{1}2$ } and  $a$  {11 $\bar{2}0$ }.

The two ends of the peculiar "crystal" of Flink (1901), reproduced as figure 11d, consist of roentgenite. The three forms called  $\gamma$  {03 $\bar{3}1$ },  $\alpha$  {30 $\bar{3}2$ },  $y$  {03 $\bar{3}4$ } by Flink should be interpreted as referring to the ratio  $5c'''/a=3.244$  of roentgenite, not to  $3c'''/a'=3.365$  of parisite. The confusion is easily explained— $a=a'\sqrt{3}$ , so that  $3c'''/a'=(3\sqrt{3})c'''/a=5c'''/a$ , since  $3\sqrt{3}$  is equal to 5 within 4 percent. When  $\gamma$ ,  $\alpha$ , and  $y$  are referred to the structural ratio  $c/a=15c'''/a$  of roentgenite, they become after interchanging the signs of the rhombohedra, T {10 $\bar{1}1$ },  $\pi$  {01 $\bar{1}2$ } L {10 $\bar{1}4$ }.

Ungemach (1935b) predicted a new species, which he called parisite  $\beta$ , with an axial ratio of 10.094, that is to say  $9c'''/a'$ . Parisite  $\beta$  is therefore roentgenite.

The point group cannot be determined from morphology alone as no doubly terminated single crystal has been found, and it is therefore impossible to decide whether {10 $\bar{1}1$ } and {01 $\bar{1}2$ } are rhombohedra or trigonal pyramids.

The apparent point-group symmetry,  $\bar{6}m2$ , of some of the "crystals" is incompatible with the rhombohedral lattice that is clearly indicated by the morphology. The symmetry  $\bar{6}m2$  is obviously that of a twin, in which reflection in (0001) is the twin operation. As the lattice is rhombohedral, we are dealing with twinning by reticular merohedry. What information does twinning yield on the point-group symmetry? If the point group were  $\bar{3}2/m$ , it would give the twin symmetry  $6/m2/m2/m$ . This is contrary to fact, and point group  $\bar{3}2/m$  is ruled out. If the point group were  $32$ ,  $3m$ , or  $\bar{3}$ , the same twin reflection would lead to the following twin symmetry:  $\bar{6}2m$ ,  $\bar{6}m2$ , or  $6/m$ , respectively. On this basis, only one of the three hemihedries, namely  $3m$ , is a possible point group. Finally, if the point group were  $3$ , twinning by reflection in (0001) would

lead to  $\bar{6}$  as symmetry of the twin. Further twinning could raise the twin symmetry to  $\bar{6}m2$ . Point group 3 is also possible on this count.

Evidence of striations or other surface features that would rule out the mirrors of  $3m$  was looked for under the binocular microscope at high magnification, but to no avail. The distinction between  $3m$  and 3 must rest on the Laue class determined by  $x$ -rays.

### *Synchisite*

Although synchisite has been found by  $x$ -rays to have lower symmetry, it is markedly pseudo-hexagonal. Its morphology is controlled by the pseudo-cell  $a'c''$  (see under unit-cell dimensions, above), with axial ratio  $c''/a' = 2.223$ . The series of forms are satisfactory (Table 5), judging from the data taken from Palache and Warren (1911). These authors give a crystal drawing (Fig. 11e) that shows only synchisite forms, namely:  $c, a, k, \mu, V, H, r, w, i, t$ . This crystal looks orthorhombic. If one wishes to refer synchisite to the orthorhombic system, the transformation  $100/120/001$  must be applied to the hexagonal indices (third index omitted). The  $\{h.h.2\bar{h}.l\}$ 's become  $\{2h.0.l\}$ 's and  $\{h.3h.l\}$ 's; the  $\{h0\bar{h}l\}$ 's become  $\{hhl\}$ 's and  $\{0.2h.l\}$ 's. The orthorhombic lattice is C-centered, with axial ratios

$$a_{or}:b_{or}:c_{or} = a':a:c'' = \frac{1}{\sqrt{3}}:1:\frac{2.223}{\sqrt{3}} = 0.577:1:1.283.$$

One intergrowth from Muzo (U. S. National Museum No. 106295) gave good measurements on faces of the synchisite "dipyramids"  $o$   $\{30\bar{3}1\}$  and  $Q$   $\{5.5.\bar{1}0.3\}$ , namely:  $82^\circ33'$  to  $82^\circ37'$  ( $82^\circ37'$ , calc.) and  $82^\circ17'$  ( $82^\circ18'$ , calc.). Two crystals of synchisite found on this intergrowth are turned  $30^\circ$  with respect to each other around the  $c$  axis, as shown by the following readings.:

$\phi$ :	$20^\circ35'$ ,	$50^\circ38'$ ,	$80^\circ30'$ ,	$140^\circ30'$ ,	$200^\circ35'$ ,
$\rho$ :	$82^\circ33'$ ,	$82^\circ33'$ ,	$82^\circ17'$ ,	$82^\circ36'$ ,	$82^\circ37'$ .

This is, however, the only example we found of this phenomenon. None of the polycrystals examined by  $x$ -rays shows it.

### *Morphology of the intergrowths*

The following relationship has been established: the  $a$ -axes of two intergrown species are rigorously parallel within the limits of observation, but they are not in every case directed in the same sense. As mentioned above,  $x$ -rays show that they are directed in opposite senses in a roentgenite-parisite polycrystal from Narsarsuk. On such an edifice positive rhombohedra of roentgenite occur in zone with negative rhombohedra of parisite and *vice versa*.

When synchisite occurs with either parisite or roentgenite, the  $\{h.h.2h.l\}$ 's of synchisite referred to  $c''/a'$  occur in zone with the  $\{h0\bar{h}l\}$ 's and the  $\{0h\bar{h}l\}$ 's of the other species, whose indices are referred to the axes  $c$  and  $a$ . The arrangement in columns of Table 5 is intended to bring out tautozonal faces of the polycrystals. Occasionally twinning introduces additional complication.

The central part of Flink's intergrowth (Fig. 11d) could be synchisite showing  $G \{10\bar{1}3\}$  and  $m \{11\bar{2}0\}$ , in which case the  $a'$  axes of synchisite would parallel the  $a$  axes of roentgenite.

#### CRYSTAL OPTICS

The indices of refraction of the four species were measured by H. Jaffe on small grains identified by  $x$ -rays. Indices of refraction were calculated in two ways: (1) from ionic refractivities; (2) from form birefringence.

##### *Ionic refractivities*

The following equations, which are known to be fair approximations for ionic structures, are used to calculate the indices of refraction;

$$\frac{\epsilon^2 - 1}{\epsilon^2 + 2} \cdot \frac{MW}{G} = \sum_i (R_\epsilon)_i n_i \quad (1)$$

$$\frac{\omega^2 - 1}{\omega^2 + 2} \cdot \frac{MW}{G} = \sum_i (R_\omega)_i n_i \quad (2)$$

$n_i$  is the number of atoms of the  $i$ th element in the molecule,  $R_\epsilon$  and  $R_\omega$  are the ionic refractivities. They are given by Bragg (1939) for all but the cerium ion (Table 6). For the carbonate ion the refractivities are referred to vibration directions parallel and perpendicular to the plane of the ion,  $R_{\parallel} = 11.32$  and  $R_{\perp} = 8.38$ . In bastnaesite  $R_\epsilon$  equals  $R_{\parallel}$ , because all carbonates are parallel to  $c$  and are therefore parallel to the vibration direction of  $\epsilon$ . In all other structures this same value of  $R_\epsilon$  was used as a first approximation. To obtain the value of  $R_\omega = 9.58$ , we assume elliptic variation of refractivity with vibration direction, and calculate the average radius vector of the ellipse whose half-axes are  $R_{\parallel}$  and  $R_{\perp}$ .

TABLE 6. IONIC REFRACTIVITIES

Ion	$R_\omega$	$R_\epsilon$	Source
Ce <sup>+3</sup>	5.51	5.55	Calculated
Ca <sup>++</sup>	1.99	1.99	Bragg, 1939, p. 185
F <sup>-</sup>	2.20	2.20	Bragg, 1939, p. 182
CO <sub>3</sub> <sup>-</sup>	9.58	11.32	$R_\omega$ calculated from $R_{\perp} = 8.38$ , $R_\epsilon = R_{\parallel}$ , Bragg, 1939, p. 185

The refractivities of cerium are obtained by substituting the observed indices of refraction of bastnaesite into equations (1) and (2) and solving for  $(R_\epsilon)_{\text{Ce}^{+3}}$  and  $(R_\omega)_{\text{Ce}^{+3}}$ . Bastnaesite was chosen because, having the highest indices of the four species, the highest reliable indices reported for it in the literature were likely to be those of single crystal grains. With  $\epsilon_{\text{bast.}} = 1.8242$  and  $\omega_{\text{bast.}} = 1.7225$  (Geijer, 1921),  $R_\epsilon$  and  $R_\omega$  of cerium are calculated to equal 5.55 and 5.51, respectively. These refractivities together with those of the other ions lead to calculated refractive indices of parisite, roentgenite, and synchisite in good agreement with observed values. The latter were obtained by H. Jaffe, of the Geological Survey, on grains previously identified by  $x$ -rays (Table 7). Contrary to custom the calculated and observed birefringences are not listed, because they are not computed directly. Any systematic error in calculated values of  $\epsilon$  and  $\omega$  would cancel in the expression  $\epsilon - \omega$ . Good agreement in birefringences could therefore be misleading.

*Form birefringence*

The crystal structures of the four species can be visualized (Fig. 12) as consisting of two kinds of "slabs" stacked in different proportions: bastnaesite slabs (B) and synchisite slabs (S).

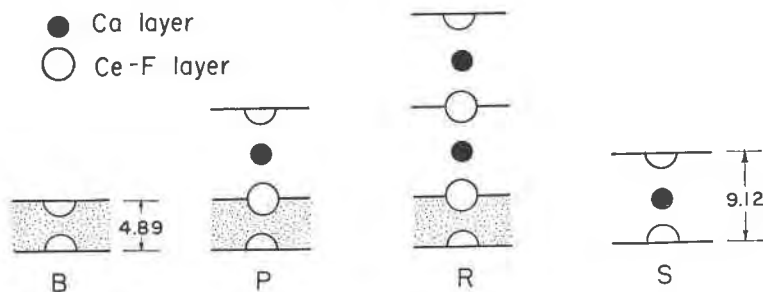


FIG. 12. Stacking of slabs in crystal structures: Bastnaesite = B =  $\text{CeFCO}_3$ , parisite = B.S., roentgenite = B.2S, synchisite = S =  $\text{CeFCO}_3 \cdot \text{CaCO}_3$ .

Crystals of parisite or of roentgenite and polycrystals of any two species may be considered *mixed bodies*, in Wiener's sense (1912), for slab thicknesses are smaller than the wavelength of light. Let  $v_1$  and  $v_2$  be the volume fractions of the two constituents ( $v_1 + v_2 = 1$ ). Let  $\epsilon_1, \omega_1$  be the indices of refraction of bastnaesite;  $\epsilon_2, \omega_2$  those of synchisite. The stack of slabs is optically uniaxial and its indices  $e, o$  are given by Wiener's formulas for negative form birefringence (see, for instance, Ambronn and Frey, 1926, or Donnay, 1936, 1945):

$$e^2 = \frac{\epsilon_1^2 \epsilon_2^2}{v_1 \epsilon_2^2 + v_2 \epsilon_1^2}, \quad o^2 = v_1 \omega_1^2 + v_2 \omega_2^2.$$

TABLE 7. INDICES OF REFRACTION AND SPECIFIC GRAVITY

No.	Locality	$\epsilon$	$\omega$	$\lambda$	Observer	Reference	G
<i>Bastnaesite</i>							
1	Bastnäs, Sweden	1.8242	1.7225		Geijer ('21)	Glass & Smalley ('45)	4.93
	Bastnäs, Sweden	1.8235	1.7220		Glass	Glass & Smalley ('45)	
2	Belgian Congo	1.823	1.722		Glass	Glass & Smalley ('45)	ca. 5
3	Gallinas, N. Mex.	1.819	1.718		Glass	Glass & Smalley ('45)	
4	Narsarsuk, Greenld.	1.818	1.718	(Na)	Jaffe	Jan. 23, 1953 (pr. com.)	4.99
5	Pikes Peak, Colo.	1.818	1.717		Larsen and Berman	Larsen and Berman (1934)	4.948
	Pikes Peak, Colo.	1.818	1.717		Glass	Glass & Smalley ('45)	
6	Madagascar	1.818	1.717		Glass	Glass & Smalley ('45)	
7	Jamestown, Colo.	1.817	1.716		Glass	Glass & Smalley ('45)	4.36
8	Belgian Congo	ca. 1.815	ca. 1.713		Buttgenbach	('47)	
<i>Parisite</i>							
9	Narsarsuk, Greenld.	1.7701	1.6742		Flink ('01)	in Palache, <i>et al.</i> (1951)	4.36
10	Narsarsuk, Greenld.	1.7690	1.6730	(589)	Quercigh ('12)	in Palache, <i>et al.</i> (1951)	
11	Muzo, Colombia	1.7712	1.6717	(589)	Quercigh ('12)	in Palache, <i>et al.</i> (1951)	4.29
12	Narsarsuk, Greenld.	1.765	1.670	(Na)	Jaffe	Jan. 16, 1953 (pr. com.)	
	(2CeFCO <sub>3</sub> · CaCO <sub>3</sub> )	(± 5)	(± 2)		Calcd. from ionic refractivities		
<i>Roentgenite</i>							
13	Narsarsuk	1.756	1.662	(Na)	Jaffe	Jan. 23, 1953 (pr. com.)	4.19
	(3CeFCO <sub>3</sub> · 2CaCO <sub>3</sub> )	1.755	1.654		Calcd. from ionic refractivities		
<i>Synchisite</i>							
14	Narsarsuk, Greenld.	1.745	1.650	(Na)	Jaffe	Jan. 16, 1953 (pr. com.)	3.94
	Switzerland	(± 5)	(± 2)		Parker <i>et al.</i> ('39)		
15	Switzerland	> 1.74	1.649				3.90
16	Transvaal	1.744	1.644		Söhngé ('44)	in Palache, Berman and Frondel (1951)	
	(CeFCO <sub>3</sub> · CaCO <sub>3</sub> )	1.744	1.644		Calcd. from ionic refractivities		3.99

The height of the B slab, obtained by x-rays, is  $h_1 = 4.89 \text{ \AA}$ ; that of the S slab,  $h_2 = 9.12 \text{ \AA}$ . Because the unit mesh in the basal plane has the same area in each case, the volume fractions are proportional to the height fractions. The general formula  $Bn_1Sn_2$  of a polycrystal corresponds to the chemical composition  $(n_1 + n_2) \text{ CeFCO}_3 \cdot n_2 \text{ CaCO}_3$ . The volume fractions are

$$v_1 = n_1 h_1 / (n_1 h_1 + n_2 h_2), \quad v_2 = n_2 h_2 / (n_1 h_1 + n_2 h_2).$$

For pure parisite  $n_1 = n_2 = 1$  and  $v_2 = 0.6508$ ; for pure roentgenite  $n_1 = 1$ ,  $n_2 = 2$ , and  $v_2 = 0.7885$ .

The highest values on record for the indices of refraction of bastnaesite are those obtained by Geijer (1921) on material from the type locality:  $\epsilon_1 = 1.8242$ ,  $\omega_1 = 1.7225$ . It is difficult to decide which measured values to use for the synchisite indices. Söhngé's values (Palache, Berman and

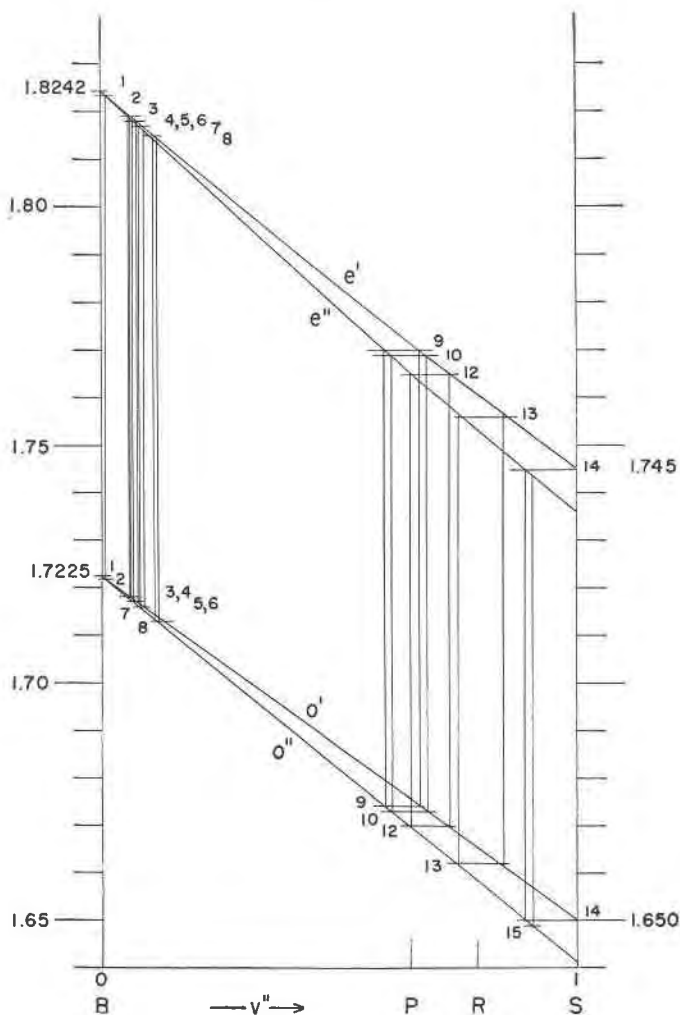


FIG. 13. Indices of refraction vs. composition expressed as volume fraction of S slabs. Curves  $e'o'$  obtained from Wiener's formula with Geijer's values for the indices of bastnaesite and Jaffe's values for those of synchisite. Curves  $e''o''$  obtained with the same indices for bastnaesite and Jaffe's values for parisite.

Fron del, 1951, p. 287),  $\epsilon_2 = 1.744$ ,  $\omega_2 = 1.644$ , were tried first. They give  $e$  and  $o$  curves which account fairly well for the experimental data. But the measurements that H. Jaffe made on our material,  $\epsilon_2 = 1.745$ ,  $\omega_2 = 1.650$ , give better curves ( $e'$  and  $o'$ , Fig. 13) on which measured indices of various sources (numbered in Table 7) can be seen to lie vertically above each other, each pair ( $\epsilon_i$ ,  $\omega_i$ ) corresponding to one and the same

abscissa. The parisite values (Nos. 9–12), however, all fall to the right of the theoretical composition; likewise the roentgenite indices (No. 13) fall to the right of their abscissa. This lack of agreement in position is taken as an indication that the indices used for synchisite were not measured on pure synchisite.

By assuming that the indices measured on our parisite (No. 12) by H. Jaffe are those of pure parisite, we can calculate much lower indices for pure synchisite, namely 1.736 and 1.641. The corresponding curves ( $e''$  and  $o''$ , Fig. 13) likewise show that any pair of measured indices lie on a vertical line. Now, however, the roentgenite pair falls to the left of its composition and the group of parisite values (other than No. 12) also falls to the left of the parisite composition. The correct curves should probably lie between  $e'$  and  $e''$  and between  $o'$  and  $o''$ . Such uncertainties will not be resolved until the indices can be measured on two crystals of known compositions. From the  $e$  and  $o$  curves that could then be drawn, it would be possible to read the composition corresponding to any pair of measured indices.

The variations observed in the indices of the bastnaesite specimens (Nos. 1 to 8) indicate the presence of some S slabs, either as part of a little intercalated parisite or roentgenite or as intersitial S slabs that do *not* constitute synchisite crystals. In the first case the specimen is a polycrystal. In the second case we are dealing with a special kind of solid solution in which not random isolated atoms but whole structural layers at a time substitute for each other. For a given composition the type of disorder observed by  $x$ -rays, namely faults in the stacking of layers, should not affect the indices of refraction.

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