Somayazulu, M. S., Roy, A. P. & Deb, S. K. (1993). *J. Phys. Condens. Matter*, **5**, 4557–4562.
Somayazulu, M. S., Sastry, P. U. M. & Wadhawan, V. K. (1988). *Solid State Commun.* **67**, 757–761.
Wehe, D. J., Busing, W. R. & Levy, H. A. (1962). *ORABS*. Report ORNL-TM-229. Oak Ridge National Laboratory, Tennessee, USA.
Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1994). C**50**, 1857–1859

$InBO_3$

James R. Cox and Douglas A. Keszler*

Department of Chemistry and Center for Advanced Materials Research, Oregon State University, Gilbert Hall 153, Corvallis, Oregon 97331 4003, USA

(*Received* 1 *November* 1993; *accepted* 13 *April* 1994)

Abstract

The crystal structure of indium borate, $InBO_3$, has been determined by single-crystal X-ray methods. The structure is similar to that of the mineral calcite, $CaCO_3$. Layers of distorted InO_6 octahedra are interleaved by layers of triangular-planar BO_3 units. The resulting indium-centered O octahedron exhibits a small trigonal elongation. The structure is compared and contrasted with other borates containing indium.

Comment

As part of a continuing effort to synthesize materials that possess promising optical properties, the compounds $Sr_3In(BO_3)_3$ and $Ba_3In(BO_3)_3$ were synthesized recently (Cox, Schaffers & Keszler, 1994). While attempting to delineate the crystal chemistry of these compounds and their relationships with the corresponding Sc derivatives, we discovered that the crystal structure of $InBO_3$ had not been refined. All references describing the structural characteristics of $InBO_3$ can ultimately be traced to the original X-ray work on powdered samples of materials isostructural with the mineral calcite, $CaCO_3$ (Goldschmidt & Hauptmann, 1932).

The unique optical properties exhibited by $InBO_3$ have resulted in several interesting applications. Tb^{3+}-doped samples have been proposed for potential use as green cathode ray tube (CRT) phosphors in color televisions (Welker, 1991) and as real-time solar neutrino detectors (Chaminade *et al.*, 1990) on the basis of the inverse β-decay reaction $^{115}In \rightarrow {}^{115}Sn^*$ (Raghavan, 1976). To characterize this material more completely, we report here its single-crystal structure refinement.

Single crystals were grown in a platinum crucible from a melt (Chaminade *et al.*, 1990) containing 3 g $LiBO_2$ (AESAR 99.9), 0.800 g In_2O_3 (AESAR 99.9) and 0.200 g B_2O_3 (Alpha 99.98). The melt was cooled from 1423 to 1113 K at 5 K h^{-1} and then rapidly to room temperature. The flux was dissolved in hot distilled water to leave block-shaped crystals ranging in size from 0.2 to 0.8 mm. A suitable crystal was mounted on a glass fiber for data collection.

$InBO_3$ is isostructural with the mineral calcite, $CaCO_3$, as was predicted from X-ray powder diffraction photographs (Goldschmidt & Hauptmann, 1932). The present X-ray analysis verifies this prediction and also provides data on refined metrical parameters. In general, the structure is composed of alternating layers of In atoms and triangular planar BO_3 units as can be seen from the ball-and-stick representation of the unit cell (Fig. 1), with the In atoms occupying distorted octahedral sites. A similar type of layered structural coale-

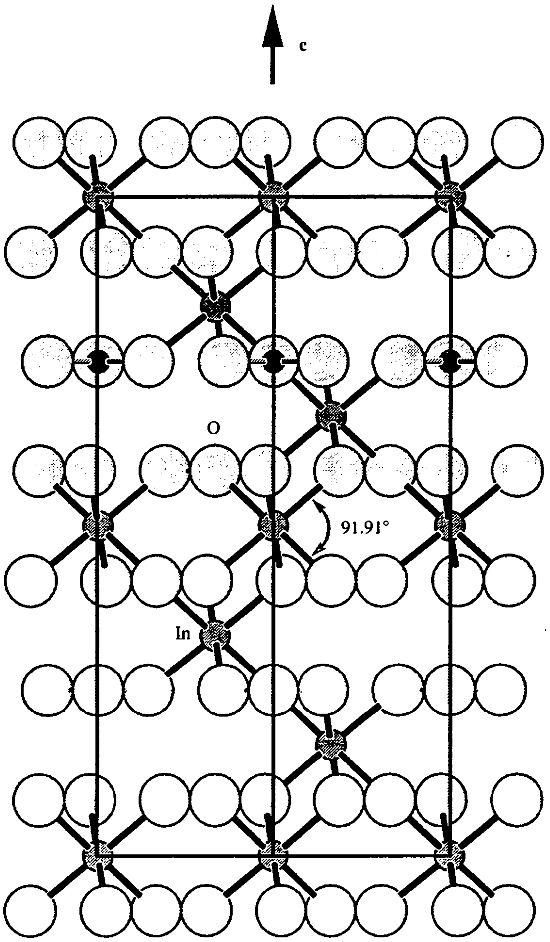

Fig. 1. Ball-and-stick representation of the $InBO_3$ unit cell with a [110] orientation. Filled black circles depict B atoms, medium shaded circles with dark bonds depict In atoms and the largest circles represent O atoms. This diagram also illustrates the elongation experienced by the InO_6 octahedra.

scence is common to a variety of borate and carbonate structures. Structural connectivity between the InO_6 and BO_3 groups occurs *via* corner sharing only. Across a BO_3 layer, each InO_6 polyhedron shares corners with three other InO_6 octahedra and with three BO_3 units sandwiched between the adjacent InO_6 layers (Fig. 2). Therefore, each InO_6 polyhedron shares corners with twelve polyhedra, namely, six BO_3 groups and six InO_6 octahedra.

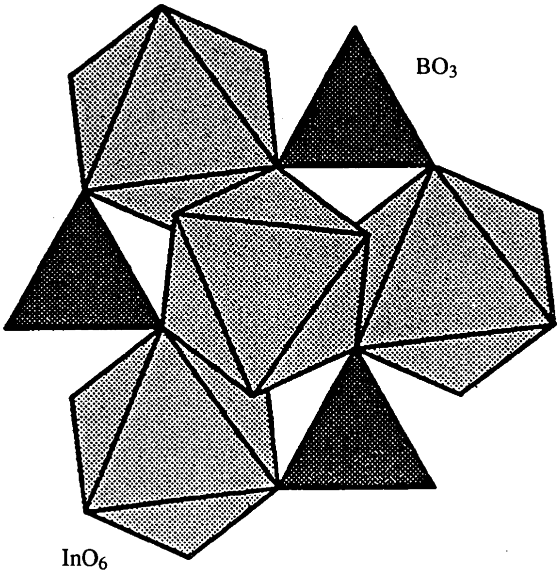

Fig. 2. View of the unit cell down the c axis illustrating how the layers are connected together. In descending order down the c axis, the center InO_6 octahedra represents the first layer, the BO_3 units are next, lying between the O_6 environments, and the outer three InO_6 polyhedra are the lowest layer in the diagram.

The In environment has S_6 site symmetry and a trigonal elongation of the InO_6 octahedron which produces an O—In—O angle of 91.91 (2)° (*cf.* Fig. 1). This deviation from O_h symmetry is not as extensive as that found in the isostructural borate $ScBO_3$ (Sun & Keszler, 1988) where the corresponding O—Sc—O angle is 92.28 (1)°. Comparisons with the STACK (Schaffers, Thompson, Alekel, Cox & Keszler, 1990) compounds $Sr_3In(BO_3)$ and $Ba_3In(BIO_3)_3$, are useful since these materials have both elongated and compressed InO_6 sites that both maintain S_6 site symmetry. For $Sr_3In(BO_3)_3$, the distortions in O—In—O angles range from the compressed 83.0 (2) to the elongated 93.4 (2)°, and for $Ba_3In(BO_3)_3$, the corresponding angles are 84.3 (3) and 94.0 (3)°.

The In—O distance of 2.1575 (7) Å is longer than the Sc—O distance of 2.1200 (4) Å in $ScBO_3$, as expected from the crystal radii of Sc^{3+} (0.885 Å) and In^{3+} (0.940 Å) (Shannon, 1976). For the oxide In_2O_3, an average In—O distance of 2.18 (1) Å is derived from two sixfold sites having approximate cubic symmetry (Marezio, 1966). The O—In—O angles and In—O distances in the materials $Sr_3In(BO_3)_3$ [In—O 2.127 (4)–2.186 (4) Å] and $Ba_3In(BO_3)_3$ [In—O 2.147 (6)–2.211 (8) Å] are largely dictated by the sizes of the large dipositive cations, Sr^{2+} and Ba^{2+}, and by the binding constraints imposed by the STACK structure. All of these distances, and the distance of 2.17 Å calculated from the crystal radius for an In atom in an octahedral O site, indicate that the In—O interaction in $InBO_3$ is typical of other oxides containing indium.

The BO_3 triangular-planar environment centers on a site having D_3 point symmetry. The O—B—O angle is 120° and the B—O bond distance is 1.380 (1) Å, a value that is statistically equivalent to the distance of 1.3752 (5) Å in $ScBO_3$. The less symmetric BO_3 units in $Sr_3In(BO_3)_3$ and $Ba_3In(BO_3)_3$ have C_1 point symmetries with angles and distances ranging from 118.8 (6) to 121.4 (5)° and from 1.34 (1) to 1.44 (1) Å, respectively. Within a given BO_3 layer of $InBO_3$, all BO_3 groups have an identical orientation. This alignment alternates from layer to layer by a rotation of 60° about the trigonal axis. Each BO_3 group shares vertices with six InO_6 octahedra; two at each O atom from layers above and below the BO_3 plane.

The O-atom coordination number is also three. As noted above, each O atom is common to a BO_3 unit and to two InO_6 octahedra. In contrast to the complex In borates, $Ba_3In(BO_3)_3$, $Sr_3In(BO_3)_3$ and $Sr_2LiInB_4O_{10}$ (Schaffers & Keszler, 1993), that possess similar triangular BO_3 groups, the O atom in $InBO_3$ occupies a site with a high degree of symmetry (C_2).

Experimental

Crystal data

$InBO_3$
$M_r = 173.63$
Rhombohedral
$R\bar{3}c$ (hexagonal axes)
$a = 4.8217$ (8) Å
$c = 15.438$ (1) Å
$V = 310.82$ (7) Å^3
$Z = 6$
$D_x = 5.565$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 15 reflections
$\theta = 13.3$–$19.45°$
$\mu = 10.908$ mm^{-1}
$T = 296$ K
Irregular block
0.25 (2) × 0.15 (2) × 0.15 (2) mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.60$, $T_{\max} = 1.00$
1190 measured reflections
236 independent reflections
918 observed reflections $[F_o^2 \geq 3\sigma(F_o^2)]$

$R_{\rm int} = 0.037$
$\theta_{\max} = 37.5°$
$h = 0 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -26 \rightarrow 26$
3 standard reflections $\{0,0,12, 0\bar{2}8, 1,1,\overline{12}\}$ monitored every 300 reflections
intensity variation: 2.5%

Refinement

Refinement on F
$R = 0.020$
$wR = 0.024$
$S = 1.48$
164 reflections
$w = 1/\sigma(F_o)$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 1.713$ e Å^{-3}
$\Delta\rho_{\min} = 1.216$ e Å^{-3}

Extinction correction: *TEXSAN* (Molecular Structure Corporation, 1989)
Extinction coefficient: 0.47 (4) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
In	0	0	0	0.21 (1)
O	0.7138 (3)	0	1/4	0.30 (4)
B	0	0	1/4	0.6 (1)

Table 2. *Selected geometric parameters* (Å, °)

In—O	2.1575 (7)	O···O	2.390 (2)
B—O	1.380 (1)		
O—In—O	91.91 (2)	In—O—In	122.92 (6)
O—B—O	120	B—O—In	118.54 (3)

A weighting scheme $w = 1/\sigma(F_o)$ was employed in which $\sigma(F_o^2) = [C + \frac{1}{4}(t_c/t_b)^2(b_1 + b_2) + (pxl)^2]^{1/2}$, where C is the total number of counts per peak, t_c is the time spent counting peak intensity, t_b is the time spent counting one side of background, b_1 are the high-angle background counts, b_2 are the low-angle background counts, p is the ignorance factor, and l is $C - \frac{1}{2}(t_c/t_b)(b_1 + b_2)$.

This work was supported by the National Science Foundation (DMR-9221372). Acknowledgement is also made to the Donors of the Petroleum Research Fund, administrated by the American Chemical Society, for partial support of this research. DAK thanks the Alfred P. Sloan Foundation for a fellowship.

Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: BR1064). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chaminade, J. P., Garcia, A., Pouchard, M., Fouassier, C., Jacquier, B., Perret-Gallix, D. & Gonzales-Mestres, L. (1990). *J. Cryst. Growth*, **99**, 799–804.
Cox, J. R., Schaffers, K. I. & Keszler, D. A. (1994). In preparation.
Goldschmidt, V. M. & Hauptmann, H. (1932). *Nachr. Ges. Wiss. Göttingen*, pp. 53–72.
Marezio, M. (1966). *Acta Cryst.* **20**, 723–728.
Molecular Structure Corporation (1989). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Raghavan, R. S. (1976). *Phys. Rev. Lett.* **37**, 259–262.
Schaffers, K. I. & Keszler, D. A. (1993). *Acta Cryst.* C**49**, 211–214.
Schaffers, K. I., Thompson, P. D., Alekel III, T., Cox, J. R. & Keszler, D. A. (1990). *J. Am. Chem. Soc.* **112**, 7068–7069.
Shannon, R. D. (1976). *Acta Cryst.* A**32**, 751–767.
Sun, H. & Keszler, D. A. (1988). *Acta Cryst.* C**44**, 1505–1507.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.
Welker, T. (1991). *J. Lumin.* **48/49**, 49–56.

Acta Cryst. (1994). C**50**, 1859–1861

$Sr_{11}Sb_{10}$

ANETTE REHR AND SUSAN M. KAUZLARICH

Department of Chemistry, University of California, Davis, California 95616, USA

(*Received* 7 *December* 1993; *accepted* 12 *April* 1994)

Abstract

Single crystals of tetragonal (*I*4/*mmm*) $Sr_{11}Sb_{10}$ can be prepared by reaction of the elements in a welded Nb container. Although the existence of $Sr_{11}Sb_{10}$ has been reported, to date no crystal structure determination has been reported.

Comment

The Sr–Sb binary system contains a number of compounds (Scott, 1990) such as SrSb, $SrSb_3$, Sr_3Sb_2, Sr_2Sb (Martinez-Ripoll, Haase & Brauer, 1973), $SrSb_2$ (Deller & Eisenmann, 1976*b*), Sr_2Sb_3 (Eisenmann, 1979) and Sr_5Sb_3 (Martinez-Ripoll & Brauer, 1973; Hurng & Corbett, 1989; Rehr & Kauzlarich, 1994). Of these, only the latter four have been characterized by a single-crystal structure determination. Although $Sr_{11}Sb_{10}$ is not shown in the phase diagram (Scott, 1990), it has been postulated from X-ray powder patterns that it also crystallizes with the $Ho_{11}Ge_{10}$ structure type (Schmelczer, Schwarzenbach & Hulliger, 1979). $Sr_{11}Sb_{10}$ has been reported as an impurity in reactions to produce Sr_2Sb_3 (Eisenmann, 1979) but no crystal structure has been reported.

Single crystals of $Sr_{11}Sb_{10}$ have been obtained regularly during our investigations of ternary intermetallic compounds of Sr, Sb and a transition metal (Kauzlarich, 1990). In order to provide complete structural information the crystal structure was determined. The structure refinement indicated that the compound is $Sr_{11}Sb_{10}$ and that it is isotypic with $Ho_{11}Ge_{10}$ (Smith, Johnson & Tharp, 1967). Relatively few compounds crystallize with this structure type; examples include $Eu_{11}Sb_{10}$ (Schmelczer, Schwarzenbach & Hulliger, 1979), $Yb_{11}Sb_{10}$ (Clark, Simpson & Steinfink, 1970), $Ca_{11}Sb_{10}$ (Deller & Eisenmann, 1976*a*), $Ca_{11}Bi_{10}$ (Deller & Eisenmann, 1976*a*) and $Ho_{11}Ge_{8.75}Si_{1.25}$ (Schmelczer, Schwarzenbach & Hulliger, 1979). In all cases, including $Sr_{11}Sb_{10}$, the R value was still rather large after anisotropic refinement.

The structure type has been described in detail by Smith, Johnson & Tharp (1967) and Deller & Eisenmann (1976*a*). The structure consists of square-planar Sb rings which are connected three dimensionally by Sb_2 'dumbbells'. Such compounds can be classified as