SHORT COMMUNICATIONS

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Potassium tetrachlorosulfatoborate: change in space group.

By Richard E. Marsh, *Arthur Amos Noyes Laboratory of Chemical Physics, California Institute of Technology, Pasadena, California* 91125, *USA** and Verner Schomaker, *Department of Chemistry, University of Washington, Seattle, Washington* 98195, *USA*

Abstract

$K[B(SO_3Cl)_4]$ surely has monoclinic Cc symmetry, rather than the triclinic, $P1$, reported by Mairesse & Drache [*Acta Cryst.* (1978), B34, 1771–1776], their values for the unit-cell dimensions, atomic coordinates, Gaussian amplitudes, and structure factors conforming almost too well to the higher symmetry. The transformed (Cc) dimensions, coordinates, and amplitudes are given.

We wish to point out that the structure of potassium tetrachlorosulfatoborate, $K[B(SO_3Cl)_4]$, which was described and refined in the triclinic space group $P1$ (Mairesse & Drache, 1978; hereafter MD), can be more appropriately described in the monoclinic space group Cc.

The reported triclinic unit cell has dimensions $a = 10\cdot513$ (9), $b = 10\cdot838$ (7), $c = 10\cdot965$ (11) Å, $\alpha = 99\cdot21$ (3), $\beta = 135\cdot48$ (3), $\gamma = 97\cdot15$ (3)°, and $Z = 2$. The vectors 101, 121, and $\bar{1}00$ define a C-centered cell: $a' = 8\cdot147$, $b' = 20\cdot085$, $c' = 10\cdot513$ Å, $\alpha' = 89\cdot99$, $\beta' = 109\cdot32$, $\gamma' = 90\cdot01°$, $Z = 4$. (Lacking covariance values, we cannot give proper e.s.d.'s.) Convincing proof of the monoclinic symmetry comes from several sources: the atomic coordinates (Table 1, MD), which show that corresponding atoms of the two formula units in the triclinic cell are related to each other by the n glide of Cc, well within their e.s.d.'s; the anisotropic β's (obtained as supplementary material;† also, see Fig. 1 of MD), which show similar equivalences; and the table of F_o's (supplementary material), which shows substantial absence of the glide-forbidden reflections and excellent comparisons between $2/m$ Laue-equivalent intensities.

The atomic coordinates in Cc (x',y',z'; Table 1) are obtained from the $P1$ coordinates by the relationships $x' = \frac{1}{2}[(z_a - \frac{1}{2}y_a) + (z_b - \frac{1}{2}y_b \pm 0\cdot5)]$, $y' = \frac{1}{2}(\frac{1}{2}y_a - \frac{1}{2}y_b) + 0\cdot25$, $z' = \frac{1}{2}[(z_a - x_a) + (z_b - x_b - 0\cdot5)]$, the subscript a standing for an atom in the first unit and b in the second, glide-related

Table 1. *Atomic coordinates* ($\times 10^4$) *for space group* Cc

	x'	y'	z'
$K^+(1,2)$	4312	835	−4034
B(1,2)	−6	1189	−3
S(1,5)	−1676	2232	590
Cl(1,5)	−4044	2449	−600
O(1,13)	−1140	1776	−366
O(2,14)	−703	2826	770
O(3,15)	−1755	1890	1726
S(2,6)	996	1486	−2122
Cl(2,6)	−1309	1760	−3437
O(4,16)	467	1009	−1186
O(5,17)	1827	1108	−2853
O(6,18)	1796	2070	−1418
S(3,7)	−2319	186	−524
Cl(3,7)	−1066	−479	−1256
O(7,19)	−834	610	385
O(8,20)	−3055	−172	311
O(9,21)	−3374	564	−1610
S(4,8)	2903	908	2020
Cl(4,8)	4912	1493	2360
O(10,22)	1480	1370	1177
O(11,23)	3098	352	1262
O(12,24)	2718	790	3270

unit, *e.g.* O(1) and O(13); the increment $0\cdot25$ was chosen to translate the origin onto the c glide plane. The averaging (symmetrizing) involved no shift larger than $0\cdot002$ Å for K, S, or Cl or larger than $0\cdot006$ Å for O. The situation is similar for the transformed and averaged β's.‡ Again we are unable to list proper e.s.d.'s for the transformed parameters, lacking covariances among the original parameters.

Absence of the additional covariances between atoms related by the monoclinic symmetry rules out any proper test of whether or not the corresponding coordinates and β's agree as well as they should. For what it may be worth in the circumstances, we remark that the agreement is much *better* than could be expected from the e.s.d.'s, neglecting covar-

‡ A list of the transformed and averaged anisotropic thermal parameters has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34911 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

iances. For example, the goodnesses-of-fit for x',y',z', and β'_{22} are 0·41, 0·48, 0·39, and 0·32, each for 22 degrees of freedom, the polar-axis problem being ignored, the coordinate e.s.d.'s of atom B(1) taken equal to their B(2) counterparts [coordinates of B(1) were held fixed in MD], and a $\cos\beta$ estimate of the x–z covariance (Templeton, 1959) inserted in evaluating the e.s.d.'s for z'. We note that these values are close to the reciprocal of the goodness-of-fit value of 2·08 reported by MD, suggesting that the e.s.d.'s given by MD are considerably greater than required by the random errors in the data and probably reflect errors in the model that are independent of the choice of Laue group.

The glide plane in Cc should result in the systematic absence of reflections $h'0l'$ with l' odd – or, relative to the original $P1$ description, of $h, k, -h - 2k$ with h odd. Of the 65 such reflections in the range of the supplementary tables, five are omitted (presumably because the net count was negative or zero), 52 are starred [presumably because I was less than $3\sigma(I)$], and eight are unstarred. No $\sigma(F_o)$ values are given. We cannot assert that these reflections are really absent; but it is certain that the triclinic structure fails to account for them, as the average value of F_c/F_o for the 60 reflections listed is 0·18 (0·06 for the eight unstarred), and it seems extremely unlikely that any (necessarily) quite different structure could ever be found to agree so well ($R = 0·035$, $R_w = 0·045$) with all the data. Instead, we believe that either the observed 60 are somehow in error or they represent some slight imperfection of the crystal. In any case, they are so weak (the largest F_o among them is only about a fortieth of the largest of all the F_o's) as to be almost certainly undetectable on any photograph.

The Laue symmetry of Cc, $2/m$, requires equivalence of reflections hkl and $h, -h - k - l, l$ in the triclinic indexing. Lack of e.s.d.'s for the reported F_o's precludes a definitive test of the equivalence, and we have carried out only a partial study. For the 84 pairs in the supplementary tables with I equal to 0 or 1 and with one or both F_o's greater than 1000 (for example), the average $|\Delta F_o|/\langle F_o\rangle$ is 0·030, only 50% greater than would correspond to the lack-of-confidence term $(0·04I)^2$ used by MD in assigning observational errors. (Again the triclinic structure fails to account for any apparent deviations from monoclinic symmetry, as the average $|\Delta F_c|/\langle|F_c|\rangle$ for these same 84 pairs is only 0·010.) We gain the impression that the deviations from monoclinic symmetry are at worst no greater than would be expected from the weights (or σ's) assigned by MD, confirming our earlier conclusion that the parameter e.s.d.'s reported are considerably greater than required by the random errors in the data.

The refinement by MD in $P1$ should have led to no problems of singularity or abnormally large covariances other than those due to the polar axes (Schomaker & Marsh, 1979), but we cannot test this point precisely. Altogether, the main advantage in describing this structure in Cc lies in imposing exact, rather than approximate, symmetry relationships between different ions and, in so doing, increasing the accuracy of the determination. Of course, Cc also provides the natural explanation of the near identity of the two molecules in the unit cell (noted but not explained by MD) and the monoclinic metrical symmetry of the lattice, which is pretty well hidden by the triclinic indexing but should have been suggested by the original (MD) pseudotetragonal indexing.

References

MAIRESSE, G. & DRACHE, M. (1978). *Acta Cryst.* B34, 1771–1776.
SCHOMAKER, V. & MARSH, R. E. (1979). *Acta Cryst.* B35, 1933–1934.
TEMPLETON, D. H. (1959). *Acta Cryst.* 12, 771–773.

Is there an oxygen atom in La₂Sb?*

By Y. WANG,† L. D. CALVERT‡ and J. B. TAYLOR, *Chemistry Division, National Research Council of Canada, Ottawa, Canada K1A 0R9*

Abstract

La₂Sb has space group $I4/mmm$, $a = 4·629$ (2), $c = 18·098$ (6) Å, $Z = 4$, $D_x = 6·84$ Mg m⁻³ with La(1) in 4(c) 0,½,0. La(2) in 4(e) 0,0,z [$z = 0·3203$ (1)] and Sb in 4(e) [$z = 0·1377$ (2)] and equivalent isotropic B's of 1·12 (8), 0·70 (6) and 0·74 (7) Å² respectively. Interatomic distances are La–La 3·273 (1), 3·992 (2), 4·146 (3) and La–Sb 3·306 (4), 3·360 (2), 3·400 (2) Å. No significant electron density was found at the sites 2(a) or 2(b), contrary to the

case of Eu₄As₂O, an apparently isostructural compound. The structure was refined by full-matrix least squares using 455 observed diffractometer data.

The structure of La₂Sb was first reported by Stassen, Sato & Calvert (1970) based on photographic data. In 1977, during a diffractometer study of 'Eu₂As', thought to be isostructural with La₂Sb, it was found that the formula was really Eu₄As₂O (Wang, Calvert, Gabe & Taylor, 1977). It was therefore suggested that other structures assigned to the La₂Sb type might also contain an extra atom. This has been confirmed for Ba₄As₂O (von Schnering & Schmettow, 1979). A re-examination of La₂Sb was therefore undertaken. A