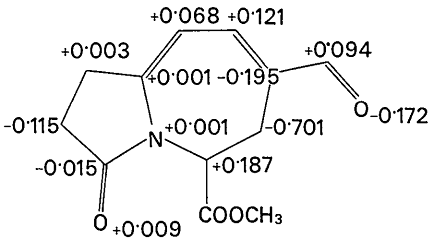


Fig. 5. Deviations (Å) of atoms from the least-squares plane denoted by equation (3).

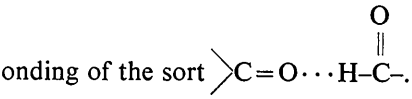
where the value on the right-hand side is the origin-to-plane distance in Å (Schomaker, Waser, Marsh & Bergman, 1959). Deviations from this plane are shown in Fig. 5. It is seen that the deviations are as large as ± 0·2 Å, except, of course, for C(6) at $-0·7$ Å, which was not expected to be in the plane. The three bonds to the nitrogen are not completely coplanar since C(5), the saturated carbon atom, is 0·2 Å out of the plane formed by the other atoms. In a diazepinone derivative (Karle & Karle, 1967), one of the bonds to a nitrogen atom situated in a somewhat similar environment deviated from a plane by the same amount.

The five atoms in the ester group form a plane whose equation is

$$6·1604x-5·0797y+1·4152z=5·0579 . \quad (4)$$

The maximum deviation of any of the five atoms is 0·02 Å. The dihedral angle formed by the plane of the ester group with the approximate plane of the molecule is 78·5°.

The packing of the molecules is shown in Figs. 2 and 3. The closest intermolecular approaches are between O···O and C···O pairs at 3·4 Å and above, except for the carbonyl oxygen, O(2), and the aldehyde carbon, C(13), in the molecule above in the **a** direction (Fig. 3). The distance C(13′)–O(2) is only 3·05 Å. The aldehyde hydrogen atom, attached to C(13′), is not oriented directly toward O(2) and the distance between them is 2·66 Å, a van der Waals separation. The orientation of the atoms do not indicate any possible hydrogen bonding of the sort



$$\rangle C=O\cdots H-\overset{O}{\overset{\|}{C}}-.$$

The y and z coordinates for O(3) and O(4) are nearly the same while their x coordinates differ by nearly one-half. This pair of atoms is responsible for the fact that the hkl reflections with h even are considerably stronger than those with h odd.

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Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

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A note on the structure of pinnoite.

By J. KROGH-MOE, *Institute of Chemistry, NLHT, Trondheim, Norway*

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The structure of pinnoite has been studied by Paton & MacDonald, utilizing a pseudo space group. Using the true space group according to Paton and MacDonald and the structure factors observed by these authors, a least-squares refinement based on their structure proposal has been attempted. The overall reliability index came down from 22 % to 15·7 %. The original structure proposal seems to be roughly correct.

The crystal structure of the borate mineral pinnoite, $MgO.B_2O_3$ $3H_2O$, has been studied by Paton & MacDonald (1957). The space group of pinnoite was determined as $P4_2$ in this study. The reported structure, however, was derived on the basis of the space group $P4_2/n$. This was possible because of the presence of a pseudo-symmetry. The computational burden was greatly reduced by working with the pseudo space group, but the overall reliability

Table 1. *Atomic position parameters, with standard deviations*

Values are given as fractions, × 10^4, of the unit cell edge. The last two columns give the parameter B (together with its standard deviation) of the temperature factor $\exp\{-B(\sin\theta/\lambda)^2\}$.

	x/a	$\sigma_{x/a}$	y/b	$\sigma_{y/b}$	z/c	$\sigma_{z/c}$	B	σ_B
Mg	2487	7	2487	7	2290	24	0·64	0·10
B(1)	3883	18	3761	18	8738	34	0·21	0·24
B(2)	1119	22	1091	21	5830	40	0·95	0·31
O(1)	0000	—	0000	—	9841	36	0·85	0·22
O(2)	5000	—	5000	—	4512	31	0·65	0·23
O(3)	3352	14	2388	13	9916	27	0·84	0·22
O(4)	1598	15	2638	14	4709	28	0·95	0·21
O(5)	4795	11	2913	12	7300	30	0·32	0·17
O(6)	0176	13	1979	13	7183	29	0·88	0·20
O(7)	2450	12	4823	13	8028	24	0·54	0·19
O(8)	2704	13	0176	12	6387	23	0·43	0·18

factor as a consequence remained high. The final value of 22% left room for doubts about the essential correctness of the proposed structure. Moreover, no assessment of the accuracy of the atomic coordinates was possible under the circumstances. It was therefore considered worth while to attempt a least-squares refinement based on space group $P4_2$, utilizing the experimental data of Paton & MacDonald.

A modified version of the Busing, Martin & Levy (1962) least-squares refinement program was used. (The observed structure factors were weighted according to the scheme of Hughes, setting $2F_{\min} = 15$.) The atomic coordinates given by Paton & MacDonald served as a starting point (with the exception of the z parameter of Mg, which was eventually found to have an incorrect sign).

By initially refining a fraction of the atoms at a time, the parameters were found to converge, and the reliability factor came down from 22% to 15·7%. The parameters obtained in this way were not entirely satisfactory as far as the atoms O(1) and O(2) (in special positions) were concerned. The resulting z parameter of these two atoms led to differing boron–oxygen bond lengths (1·32 and 1·54 Å) for chemically equivalent bonds. By adjustment of these z parameters to give boron–oxygen bond distances of 1·47 Å and continuing the refinement process, the parameters converged to new values. The reliability factor remained at 15·7%, but less disparate values for the boron–oxygen bond lengths ensued.

Parameters, with their standard deviations, from the last refinement cycle are given in Table 1. The boron–oxygen bond lengths calculated from these parameters average 1·47 Å. This agrees with the accepted value for the boron–oxygen bond, when the boron atom is in four-fold coordination. The individual bond lengths fall within a range around the average value, corresponding to about ± 3 times the standard deviation (3 × 0·03 Å).

The structure contains an isolated borate polyanion, $(OH)_3B–O–B(OH)_3^{2-}$, roughly described as two BO_4 tetrahedra sharing a corner. The B–O–B bond angle, calculated from the original parameter values of Paton & MacDonald, was found to be 111·6°. As observed by Takeuchi (1958) and Krogh-Moe (1962), this value for the B–O–B angle is considerably smaller than the values usually reported for the B–O–B angle between planar BO_3 triangles sharing a corner. Thus the B–O–B angle of the $O_2B–O–BO_2^{4-}$ polyanion in magnesium pyroborate is 134·5°, with estimated standard deviation 1·3° (Block, Burley, Perloff & Mason, 1959). However, from the present refinement the values 120·5 and 127·0°, with estimated standard deviations 3·5°, are obtained for the two different B–O–B angles in pinnoite. Hence the experimental evidence for a more acute B–O–B bond angle in pinnoite than in magnesium pyroborate is not entirely conclusive.

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Six new zinc sulphide polytypes of the family 16*L*–48*R*.

By S. Mardix and O. Brafman, *Department of Physics, The Hebrew University, Jerusalem, Israel.*

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Six hitherto unknown ZnS polytypes were found. Their unit cell and Zhdanov symbols are: 16*L* (14 2); 16*L* (5 3 3 5); 16*L* (3 3 2 2 3 3); 48*R* $(13\ 3)_3$; 48*R* $(8\ 4\ 2\ 2)_3$; and 48*R* $(10\ 6)_3$. X-ray oscillation photographs of their (10.*l*) column are shown; the calculated and observed intensities are compared.

The concept of polytype families has already been introduced in a previous publication (Mardix, Alexander, Brafman & Steinberger, 1967); five polytypes of the family 16*L*–48*R* were reported there. Six other polytypes of the same family were found in two vapour-phase-grown zinc sulphide crystals. The crystal 175 *S*23 contained polytype regions