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On the crystal structure of kernite, $Na_2B_4O_7.4H_2O$.*

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As part of a program to correlate X-ray and nuclear magnetic resonance data on the structures of oxy-anions, it was found necessary to re-investigate the crystal structure of kernite. The results of this study indicate that the originally proposed space group and structure (Garrido, 1932; Minder, 1935; Amoros, 1945) are inconsistent with the results of nuclear magnetic resonance studies (Blood & Proctor, 1954; Waterman, 1954; Waterman & Volkoff, 1955; Das, 1957).

Synthetic crystals prepared by Dr W. T. Schaller, U.S. Geological Survey, were examined by the Weissenberg method using Cu $K\alpha$ and Mo $K\alpha$ radiation. The correct space group was found to be $P2_1/c$ not $P2/c$, owing to the presence of a screw axis since there were systematic extinctions of $0k0$ for $k \neq 2n$. The unit-cell dimensions, obtained from zero-layer line photographs of rotations about the a and b axes yielded the following values:

$$a = 7{\cdot}022,\ b = 9{\cdot}151 \pm 0{\cdot}002,\ c = 15{\cdot}676\ \text{Å};\quad \beta = 108^\circ\ 50' \pm 5'\ .$$

The originally proposed structure of kernite (Amoros) consisted of B_3O_6 rings of three $[BO_3]^{-3}$ triangles linked by halved B_2O_3 ions to form infinite chains along the b axial direction. The sodium atoms were proposed to lie in the two-fold special positions: (e) and (f) ($P2/c$). The original structure determination was found to give imperfect agreement between the observed and calculated intensities.

Waterman & Volkoff (1955) have determined from nuclear magnetic resonance analysis of single crystals of kernite that there are four non-equivalent sets of boron atoms per unit cell, all of which are in general positions. One-half of the boron atoms are tetrahedrally coordinated —the others triangularly coordinated to oxygen. The sodium atoms also were found to lie in general positions.

Preliminary X-ray analyses indicate that the basic structure of kernite is related to that of borax, $Na_2B_4O_7.10\,H_2O$ which consists of $[B_4O_5(OH)_4]^{-2}$ rings of tetrahedrally and triangularly coordinated boron (Morimoto, 1956). Christ & Clark (1957) have postulated that kernite is composed of infinite chains of composition $[B_4O_6(OH)_2]^{-2n}$ resulting from the polymerization of the $[B_4O_5(OH)_4]^{-2}$ rings of borax. X-ray three-dimensional data have been collected for kernite and refinement studies are being carried out in cooperation with the U.S. Geological Survey.

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Structure of *s*-triphenyltriazine.*

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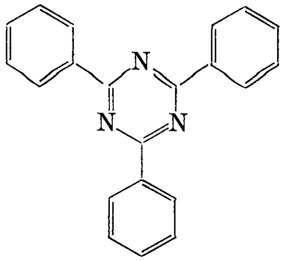
Recently great interest has been devoted to the study of molecular and electronic structure of conjugated heterocyclics containing nitrogen. Results of theoretical and experimental work (Bertinotti *et al.*, 1956; Hameka *et al.*, 1956; Herbstein *et al.*, 1955; Wheatley, 1955, 1957) have in particular indicated several interesting features of the structure of azines. We have therefore undertaken the X-ray structure determination of *s*–triphenyltriazine.

The unit-cell dimensions are:

$$a = 10{\cdot}94,\ b = 3{\cdot}91,\ c = 35{\cdot}84\ \text{Å};\quad \beta = 90^\circ\ 38',\ Z = 4;\ \text{space group}\ P2_1/c\ .$$



The Fourier transform method (Lipson & Cochran, 1953) was used in order to determine the orientation of the molecules with respect to the crystallographic axes from the weighted $h0l$ equatorial section.

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* This short communication was read at the VIII Congresso Nazionale della Società Chimica Italiana, Turin, 1958.

The molecules in the unit cell were then located by calculating a Patterson function modified according to Giglio, Liquori & Ripamonti (1958). The signs of a high percentage of the structure amplitudes were thus established.

A first Fourier synthesis allowed us to determine the coordinates of all the atoms with a sufficient accuracy. After some refinement according to the usual methods, the projection of the electron density along the 010 shown in Fig. 1 was obtained. From the Fourier projection of Fig. 1 it appears—as foreseen—that the molecule is planar or nearly planar. However, only a detailed structure determination now in progress may permit the determination of accurate bond lengths and bond angles and the detection of any small rotations of the benzene rings around the bonds connecting them to the triazine ring. This effect, in the case of 1,2,5-triphenylbenzene (Farag, 1954), proved to be very strong.

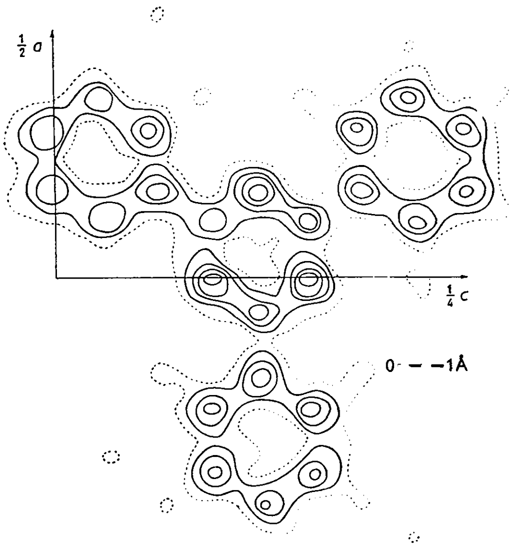

Fig. 1. Fourier projection on 010.

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Monoclinic glycine sulfate: Optical parameters.

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The morphology of monoclinic triglycine sulfate (Wood & Holden, 1957) clearly reveals the orientation of the unique *b*-axis. The *b*-axis coincides with the acute bisectrix *X* (vibration direction of the fast ray, lowest index refraction) and is normal to the planes of easy cleavage. The obtuse bisectrix *Z* (vibration direction of the slow ray, highest index of refraction) is 3° from the perpendicular to (100) in a plane normal to the *b*-axis, while the optic normal *Y* (vibration direction of intermediate ray, intermediate index of refraction) is approximately perpendicular to ($\bar{1}02$) and forms an angle of 3° with *c*.

The refractive indices and the optic angle have been determined from microscopical examination using calibrated immersion liquids and the Becke line method, and also using larger polished sections and a Bausch and Lomb Abbé-type refractometer. The averaged values of the three indices are:

$$n_p = 1{\cdot}484 \pm 0{\cdot}000$$
$$n_m = 1{\cdot}556_5 \pm 0{\cdot}000_5$$
$$n_g = 1{\cdot}584_5 \pm 0{\cdot}000_5$$

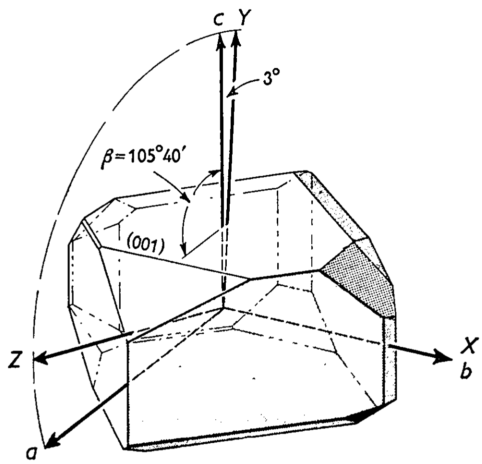

Fig. 1. Crystal axes (*a*, *b*, *c*), obtuse bisectrix *Z* and optic normal *Y* of monoclinic glycine sulfate.