Short Communications

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The unit cell and space group of sodium acetate trihydrate, $NaC_2H_3O_2.3H_2O$.

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Sodium acetate trihydrate crystals, which appear generally in the form of long but very thin needles, belong to the monoclinic system. The lattice parameters were given with a limited accuracy ($a=12\cdot4$, $b=10\cdot5$, $c=10\cdot3$ Å; $\beta=112\cdot1°$, $Z=8$) by Padmanabhan (1952) who stated its space group to be either Cm or $C2/m$. Working on the determination of the structure of $NaC_2H_3O_2 . 3H_2O$, we found that its space group differs from that given by Padmanabhan.

For our investigations small stubby, well-shaped single crystals were used. These crystals were obtained from an almost saturated aqueous solution by cooling it slowly and rubbing the wall of the glass container with a metal rod. If the mother liquor is carefully diluted with water, some of the small stubby crystals assume an almost perfectly spherical form. These crystals were quite suitable for X-ray diffraction work. Unfortunately the crystals spontaneously disintegrated in the open air; therefore they had to be covered with a suitable protecting lacquer immediately on removal from the mother liquor.

The unit-cell dimensions determined from Buerger precession photographs around b and c axes with Cu $K\alpha$ radiation ($\lambda=1\cdot542$ Å), are:

$$a=12\cdot321\pm0\cdot01\ \text{Å} \qquad b=10\cdot425\pm0\cdot01\ \text{Å}$$
$$c=10\cdot380\pm0\cdot01\ \text{Å} \qquad \beta=111\cdot71\pm0\cdot15° .$$

From these the axial ratios are:

$$a:b:c=1\cdot1818:1:0\cdot9957,$$

which are in good agreement with those obtained optically (Groth, 1909).

$$a:b:c=1\cdot1809:1:0\cdot9963 \quad \beta=111°42' .$$

The unit cell contains 8 molecules from which $D_x=1\cdot458$ g.cm^{-3} ($D_m=1\cdot456$ g.cm^{-3}; Schröder, 1881).

The single-crystal photographs indicate the following conditions for systematic absences of reflexions:

$$\begin{array}{ll} hkl \text{ present only for} & h+k=2n \\ h0l & (h=2n),\ l=2n \\ 0k0 & (k=2n)\,. \end{array}$$

These indicate the space group to be either Cc or $C2/c$.

For the determination of the presence or absence of the centre of symmetry an $N(z)$ statistical test (Howells, Phillips & Rogers, 1950) was made of 75 $hk0$ reflexions, obtained by an integrating Weissenberg goniometer, using the multiple-film technique. The intensities measured with a Zeiss fast photometer were corrected for all factors and were put on an absolute scale by Wilson's method. The intensity distribution curve obviously showed the (001) projection to be centrosymmetric. From this the correct space group is $C2/c\,(C_{2h}^6)$, which is also in a good agreement with previous findings that sodium acetate trihydrate belongs to the monoclinic-prismatic crystal class.

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Anomalous dispersion effect in the structure of barium titanate.

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In the structure analysis of barium titanate (Evans, 1961), it was found that the study was indeterminate in that values of the discrepancy index as low as 0·037 were obtained for different sets of structural and thermal parameters. The difficulty was attributed to the strong interaction between some of these parameters in X-ray diffraction and this aspect has been further discussed (Geller, 1961; Megaw, 1962). In view of the interest in this structure analysis, it was felt appropriate to report the effect of the imaginary part of the anomalous scattering, which was neglected in the study.

About 356 reflexions of the type $h0l$ were measured by Evans to obtain on averaging 99 non-equivalent structure amplitudes, of which 16 were omitted owing to serious extinction. The intensities were fairly strong even at the highest angles where $\sin\theta/\lambda$ was about 1·41 for the Mo $K\alpha$ radiation used in the experiment.

However if the imaginary parts of the anomalous scattering from the heavy atoms are also considered, the inverse reflexions *h*0*l* and $\bar{h}0\bar{l}$ with $l \neq 0$ will not be of equal intensity. The relationships, for the present case, will be $|F|_{hol}=|F|_{\bar{h}ol}=|F|_+$ and $|F|_{ho\bar{l}}=|F|_{\bar{h}o\bar{l}}=|F|_-$, where $|F|_+$ and $|F|_-$ can be appreciably different. Such differences can be expected to be very pronounced for reflexions in which the contributions of the Ti and O atoms to the structure factor oppose that of Ba. This gave another criterion for selecting the reflexions, namely *h* even and *l* odd. Calculations were made for these reflexions using the structural parameters of one model, No. 5 (Evans, 1961). The normal and anomalous scattering factors for the neutral atoms were taken from *International Tables for X-ray Crystallography* (1962).

The calculated values of the percentage intensity differences for such selected reflexions are listed in Table 1 for cases where they exceed about 5%. It was noticed that for the particular model employed in the calculations, $|F|_+$ was less than $|F|_-$ in all the *h* even, *l* odd reflexions. The differences are quite significant in a large number of reflexions as seen from Table 1. It seems therefore necessary to take into account the imaginary part of the scattering from the Ba and Ti atoms for the structure analysis following the methods that have been discussed (Patterson, 1963, Ibers & Hamilton, 1964).

In these calculations an overall isotropic temperature effect was assumed for the structure factors and so the intensity differences in the table are independent of temperature. Such a procedure was adopted in comparing the experimental and calculated intensity differences in the structure of L-tyrosine hydrochloride to obtain the imaginary part of the scattering for chlorine (Parthasarathy, 1962). It was there observed that while there was good agreement in the sign of the intensity differences, there were appreciable discrepancies in the magnitudes. In any general case, it can be shown that if anisotropic individual temperature factors are introduced, they would affect the intensity differences, to increase them for some reflexions and decrease them for others, depending on the structural and thermal parameters. Even if it is found that the measured differences are very small, it is clear that the parameters should account for such an observation.

One of us (SKM) would like to acknowledge gratefully the award of a scholarship by the Government of India.

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Table 1. *Percentage intensity differences for* $h0l$, $\bar{h}0\bar{l}$ *(h even, l odd) calculated with allowance for anomalous dispersion, but using an overall isotropic B coefficient*

Reflection *h*0*l*	Percentage difference $\frac{2(\lvert F\rvert_-^2-\lvert F\rvert_+^2)}{\lvert F\rvert_-^2+\lvert F\rvert_+^2}$ %
003	8·4
005	10·3
007	16·0
009	21·7
203	6·9
403	7·0
603	7·5
803	10·0
1003	10·5
205	11·3
405	12·3
605	13·6
805	14·0
207	15·1
407	17·0
607	20·0
807	24·0
209	22·0
409	22·6

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The crystal structure of $MnSO_4$*.

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The structures of most of the anhydrous sulphates of divalent 3*d* transition metals have been established. In the case of $MnSO_4$, the space group and lattice parameters have been reported (Rentzeperis, 1958; Coing-Boyat, 1959; Pistorius, 1960) but not the atomic positions. $MnSO_4$ crystallizes in the orthorhombic space group *Cmcm* (D_{2h}^{17}) with $a=5{\cdot}267$, $b=8{\cdot}046$, $c=6{\cdot}848$ Å, and is isostructural with $NiSO_4$ (Dimaras, 1957; Poljak, 1958) and $MgSO_4$ (Rentzeperis & Soldatos, 1958), and also with $CrVO_4$ and several other chromates of divalent metals. However, in the course of an investigation of the magnetic structure of $MnSO_4$ (Will *et al.*, 1965), it was found that agreement between observed and calculated nuclear intensities in the neutron diffraction powder pattern using the published atomic parameters for $MgSO_4$ or $NiSO_4$ was very poor. We have therefore refined the crystal structure using both X-ray and neutron diffraction data, and the results are reported in this note.*

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* During revision of the manuscript, we became aware of an earlier X-ray study of $MnSO_4$ (Rentzeperis, 1958), where the isotypism of $MnSO_4$ with $MgSO_4$ and $NiSO_4$ is pointed out. The proposed parameters are derived without refinement from $MgSO_4$ by shifting only the sulfur atoms. They are not of sufficient accuracy to describe the structure and leave large discrepancies between calculated and observed intensities of the low angle reflections.