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Crystal data for anhydrous lithium acetate.

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A crystallographic study has been made of anhydrous lithium acetate which is apparently the less common form of lithium acetate; most standard chemical works give data for the dihydrate only. The source of our material was a commercial compound sold as 'anhydrous lithium acetate'. This consisted of a fine hygroscopic powder with which was mixed a relatively few tiny colorless crystals. A chemical analysis of the crystals after they had been permitted to take on water from the atmosphere showed them to be the dihydrate. It was not possible to get a good chemical analysis of the original crystals, but their hygroscopic character suggested that they were either the anhydrous compound or a lower hydrate. They proved to have a lower density than the 1·30 g.cm.$^{-3}$ of the dihydrate (Amirthalingam & Padmanabhan, 1958), and by sifting the commercial powder and then floating the crystals in a liquid with a density lower than 1·30 g.cm.$^{-3}$, it was possible to obtain good crystals of the hygroscopic material. By heavy liquids the best specific gravity was determined to be 1·227 g.cm.$^{-3}$.

X-ray precession photographs using Cu radiation were taken of several of the crystals which were coated with a thin layer of transparent nail polish or of a plastic waterproof spray, Krylon, in order to protect them from moisture in the atmosphere. The crystals proved to be triclinic with:

$$a = 9{\cdot}29,\ b = 12{\cdot}13,\ c = 6{\cdot}76\ \text{Å}.$$
$$\alpha = 101^\circ\ 0',\ \beta = 100^\circ\ 19',\ \gamma = 105^\circ\ 5'.$$

The statistical tests of Howells, Phillips & Rogers (1950) showed the space group to be $P\bar{1}$. These cell dimensions yield, for an assumed composition of anhydrous lithium acetate and the measured specific gravity of 1·227 g.cm.$^{-3}$, 8·04 formula units in the unit cell. The density calculated for $Z = 8$ is 1·221 g.cm.$^{-3}$, and this agreement between calculated and observed densities is our basis for concluding that the crystals are anhydrous lithium acetate and not some hydrate lower than the dihydrate. Powder data for this compound and for the dihydrate have been submitted for publication in the *A.S.T.M. Powder Data Card File*.

We attempted an analysis of the crystal structure by 2-dimensional Fourier methods, but we were unable to deduce a structure that would explain the observed intensities. Details of the investigation are to be found in Saunderson (1960). We plan no further work on this crystal.

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A note on the crystal structure of anhydrous copper sulphate.

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The crystal structure of $CuSO_4$, which crystallizes with the cell constants

$$a = 8{\cdot}39,\ b = 6{\cdot}69,\ c = 4{\cdot}83\ \text{Å}$$

in the space group *Pnma* and $Z = 4$, has been determined by Kokkoros & Rentzeperis (1958) by the method of trial and error. A (2 + 2 + 2) coordination has been attributed to Cu in this structure, but copper has mostly been found to have four nearest coplanar neighbours, with two more at a rather greater distance, which complete a distorted octahedral coordination group (Wells, 1950; *cf.* also Gattow & Zemann, 1958). Recently Knox (1959) has reported a new kind of distortion for the octahedral copper(II) in K_2CuF_4, where Cu has been found to have a (2 + 4) coordination with two nearest neighbours (Cu–F = 1·95 Å (2 ×)) and four at a slightly greater distance (Cu–F = 2·08 Å (4 ×)). Edwards & Peacock (1959) have also reported an identical coordination for Cu in $KCuF_3$ (Cu–F = 1·96 Å (2 ×) and 2·07 Å (4 ×)). Therefore it is of interest to investigate whether Cu has the usual (4 + 2) coordination or the rarer type of (2 + 4) coordination in $CuSO_4$. While there is no doubt that the structure determined by Kokkoros & Rentzeperis (K&R) is correct in principle it needs further refinement to establish the exact nature of the coordination polyhedron around copper.

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