

molar amount of tetranactin and CuBr_2) as dark green needles with monoclinic symmetry (No. 11). The complex formation was proved by the infrared spectra which indicated complicated shifts in $\nu_{\text{C}-\text{O}-\text{C}}$ bands, while $\Delta\nu_{\text{C}-\text{O}}$ (ca. 4 cm^{-1}) was rather small compared with those found in K^+ (15 cm^{-1}) and Ba^{2+} (45 cm^{-1}) complexes.

The spectral data (infrared and p.m.r.) on the free and the complexed molecules of the tetranactins will be published elsewhere.

Acta Cryst. (1971) B27, 1682

The crystal structures of racemic 3-benzylamino-4-hydroxypent-2-enoic acid lactone hydrochloride and of spontaneously resolved 3-benzylamino-4-hydroxypent-2-enoic acid lactone hydrobromide. By PEI-TAK CHENG, CHUNG HOE KOO, IAN P. MELLOR, S. C. NYBURG AND JOHN M. YOUNG, *Lash Miller Chemical Laboratories, University of Toronto, Toronto 181, Ontario, Canada*

(Received 27 January 1971)

In *Acta Cryst.* (1970) B26, 1139 the compounds are erroneously named and pent-2-enoic should read pentanoic.

In a paper of the above title (Cheng, Koo, Mellor, Nyburg & Young, 1970), the compounds are erroneously named. 3-Benzylamino-4-hydroxypent-2-enoic acid should read 3-benzylamino-4-hydroxypentanoic acid throughout.

Reference

- CHENG, P.-T., KOO, C. H., MELLOR, I. P., NYBURG, S. C. & YOUNG, J. M. (1970). *Acta Cryst.* B26, 1339.

Acta Cryst. (1971). B27, 1682

Refinement of the crystal structure of lithium hydroxide monohydrate. By N.W. ALCOCK, *School of Molecular Sciences, University of Warwick, Coventry CV4 7AL, England*

(Received 29 April 1971)

The crystal structure of lithium hydroxide monohydrate, $\text{LiOH}\cdot\text{H}_2\text{O}$, has been refined from the data of Rabaud & Gay to locate the hydrogen atoms. The R value was reduced from 0.085 to 0.065 and the hydrogen atoms were shown to be ordered.

Rabaud & Gay (1957) determined the crystal structure of $\text{LiOH}\cdot\text{H}_2\text{O}$ from three-dimensional data, using for refinement difference syntheses in two-dimensional projections. They located the hydroxyl hydrogen atom (in a special position) but could not find that of the water molecule (in a general position) and they suggested that it was disordered. However, the deuteron magnetic resonance (d.m.r.) spectrum of the compound, which has been studied in this laboratory (Clifford, Dixon & Smith, 1967; Smith & Clifford, 1971), indicates strongly that the water molecule is not disordered. The X-ray data has therefore been re-examined to resolve this discrepancy.

Experimental

Crystal data

Monoclinic, $a=7.37$, $b=8.26$, $c=3.19\text{ \AA}$;
 $\beta=110^\circ 18'$; $Z=4$;
 Space group $C2/m$ (from Rabaud & Gay, 1957).

Form factors for Li^+ and O were from *International Tables for X-ray Crystallography*, (1962), for H from McWeeny (1951). Two cycles of full-matrix least-square refinement on the lithium and oxygen atoms with anisotropic temperature factors for each reduced the R value to 0.073 (Rabaud & Gay (1957) quote 0.085 as their final figure). A difference Fourier synthesis showed the largest peak in special position $i(x,0,z)$ with $x=0.26$, $z=0.64$, equivalent to the hydroxyl hydrogen found by Rabaud & Gay. There were two peaks of nearly equal height in general positions, but after one cycle of refinement including the first hydrogen atom, H(1), only one of these two peaks remained strong. The second hydrogen atom, H(2), was therefore added at this position and the data finally refined to $R=0.065$. It was found necessary to damp the hydrogen atom shifts by 0.5 to avoid oscillations; of the 167 reflexions, three (400, 021, 002) showing extinction effects were given zero weight, with the rest having unit weights. The final parameters are shown in Table 1. Following the original nomenclature,

References

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Table 1. Atomic positions, thermal parameters and standard deviations

The expression for the anisotropic temperature factors is

$$\exp [-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$$

	Li	O(1)	O(2)	H(1)	H(2)
<i>X</i>	0.0	0.2857 (5)	0.0	0.237 (21)	0.107 (12)
<i>Y</i>	0.3474 (11)	0.0	0.2066 (4)	0.0	0.118 (10)
<i>Z</i>	0.5	0.3952 (12)	0.0	0.631 (61)	0.004 (38)
<i>B</i>	—	—	—	12.3 (4.4)	14.0 (4.2)
β_{11}	0.0082 (14)	0.0069 (6)	0.0107 (8)	—	—
β_{22}	0.0046 (10)	0.0052 (5)	0.0059 (5)	—	—
β_{33}	0.0543 (99)	0.0422 (43)	0.0396 (42)	—	—
β_{12}	0.0	0.0	0.0	—	—
β_{13}	0.0056 (31)	0.0040 (14)	0.0069 (14)	—	—
β_{23}	0.0	0.0	0.0	—	—

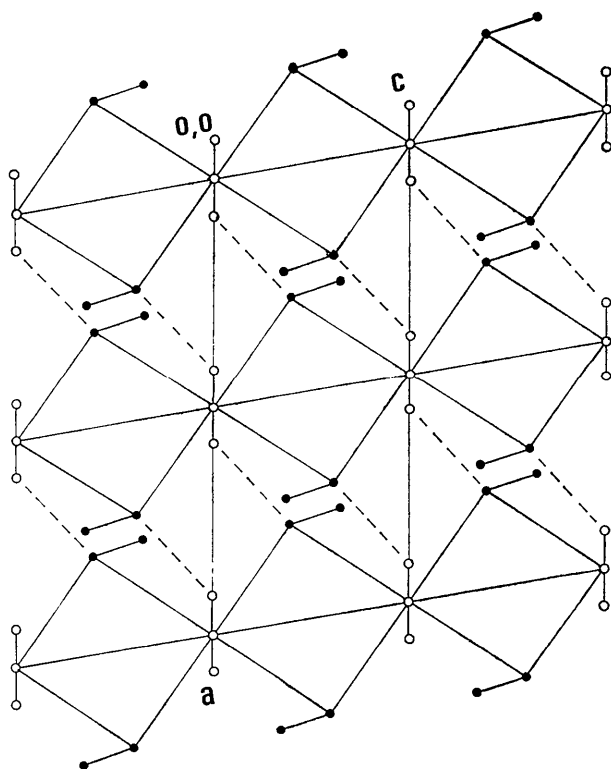


Fig. 1. LiOH·H₂O packing diagram projected down *b*. Sections of three chains of double tetrahedra are shown; each has lower tetrahedra under the visible upper ones (related by a mirror plane). The central chain is at height $\frac{1}{2}$, the outer ones at zero height. O(1) and H(1) are shown by solid circles, O(2) and H(2) by open circles and the hydrogen bonds are dotted. The Li atoms in the middle of each tetrahedron are omitted.

O(1) and H(1) make up the hydroxyl group and lie on a mirror plane, while O(2) of the water molecule is on a two-fold axis; its hydrogen atoms, H(2) and H(2') are related by this axis. The computations were performed with the X-RAY 63 program system running on the SRC Atlas computer at Chilton.

Results

The principal point of importance is that the re-interpreted X-ray data supports the d.m.r. results in finding localized hydrogen atoms. The bond lengths are

O(1)—H(1)	0.94 ± 0.21
O(2)—H(2)	1.07 ± 0.09
O(1)---H(2)	1.76 ± 0.08 (hydrogen bond)

A clearer description of the molecular packing can also be given. Each lithium atom is coordinated by a tetrahedron of oxygen atoms, two from hydroxide groups and two from water molecules. These are linked in pairs by a common edge and share corners to form chains running in the *c* direction. The chains are linked by hydrogen bonds, O(1)---H(2)—O(2), approximately in the $[\bar{1}11]$ and $[1\bar{1}\bar{1}]$ directions. H(1) is not involved in hydrogen bonding. Fig. 1 shows this packing.

The hydrogen bond is bent; this is not significant in view of the errors involved, but is confirmed by the d.m.r. results (Smith & Clifford, 1971).

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