formation of only one of two possible isomers of diketone (**1**) of the previously mentioned reaction scheme. Determination of the analogous *trans* stereochemistry for dialcohol (**3**) [which was studied rather than diketone (**1**) (or **2**) of the scheme given elsewhere (Sapienza *et al.*, 1976) because of ease of crystal growth] by single-crystal methods supports this suggestion.

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Lithium Hydrogen Phthalate Monohydrate

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Abstract. $LiHC_8O_4H_4.H_2O$, $C_8H_5LiO_4.H_2O$, triclinic, $P\bar{1}$, $a = 11\cdot932\,(2)$, $b = 8\cdot874\,(2)$, $c = 7\cdot943\,(2)$ Å, $\alpha = 97\cdot13\,(5)$, $\beta = 95\cdot88\,(5)$, $\gamma = 93\cdot12\,(5)°$, $Z = 4$, $D_x = 1\cdot524$ Mg m^{-3}. The structure has been solved by direct methods and refined to $R = 0\cdot032$ for 3446 independent reflexions. The Li ions are coordinated by distorted O tetrahedra. In the two symmetrically independent hydrogen *o*-phthalate ions very short intramolecular hydrogen bonds between the two adjacent carboxylic groups are formed with O—O distances 2·400 (2) and 2·390 (2) Å.

Introduction. Recent structure analyses of the acid lithium phthalates $LiHC_8O_4H_4.2H_2O$ (Gonschorek & Küppers, 1975) and $LiHC_8O_4H_4.CH_3OH$ (Adiwidjaja & Küppers, 1978) revealed intramolecular hydrogen bonds with extremely short O—O distances. The aim of the present investigation is to check whether similarly short hydrogen bonds occur in the monohydrate. In the course of growth experiments on large single crystals of orthorhombic lithium hydrogen phthalate dihydrate, a triclinic crystal species appeared by chance during one experiment which was found to be the monohydrate. The predominating faces of these crystals as grown from aqueous solution are {110}, {001}, and {$1\bar{1}0$}. Additionally, small faces of type {100} and {$\bar{1}11$} occur. Perfect cleavage is observed parallel to (110). The optical character is negative and the acute bisectrix is nearly perpendicular to (110).

X-ray measurements were carried out by an automatic Philips PW 1100 four-circle diffractometer using Mo $K\alpha$ radiation. Lattice parameters were determined by a least-squares treatment of 48 reflexions. Using a specimen of approximately spherical shape with a mean diameter of 0·4 mm intensities of all 7884 reflexions in the range $2 < \theta < 28°$ were collected. 848 of these were not centred or had negative intensities. Averaging the Friedel pairs yielded 3629 unique reflexions with an internal consistency index $R = 0\cdot039$ (Sheldrick, 1976). No correction for absorption seemed necessary ($\mu = 0\cdot118$ mm^{-1}).

The approximate location of the C and O atoms of the phthalate molecules was found by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Further calculations were carried out using the *SHELX* 76 system (Sheldrick, 1976). The approximate positions of the remaining atoms were determined using a difference Fourier map. Full-matrix least-squares refinement with anisotropic temperature factors for the non-hydrogen atoms resulted in a weighted R value of 0·032 (non-weighted $R = 0\cdot039$). Squared reciprocal standard deviations were used as weights. Atomic scattering factors including correction for anomalous dispersion were taken from *International Tables for X-*

ray Crystallography (1974). 180 reflexions with $F_o < 2\sigma$ were eliminated. The three strongest reflexions which apparently suffered from extinction ($|F_c| > |F_o|$) were also removed. Thus, 3446 reflexions were used for the final refinement. Positional parameters are presented in Table 1.*

Discussion. The Li ions are coordinated by four O atoms which form slightly distorted tetrahedra. Distances and angles in these tetrahedra are given in Table 2.

Table 2. *Bond distances* (Å) *and angles* (°) *involving the* Li *ions*

Li(1)–O(1)	1·933 (3)	O(1)–Li(1)–O(4)	114·6 (2)
Li(1)–O(4)	1·935 (3)	O(1)–Li(1)–O(11)	105·6 (2)
Li(1)–O(11)	1·954 (3)	O(1)–Li(1)–O(14)	108·2 (2)
Li(1)–O(14)	1·905 (3)	O(4)–Li(1)–O(11)	108·9 (2)
		O(4)–Li(1)–O(14)	108·0 (2)
		O(11)–Li(1)–O(14)	111·6 (2)
Li(2)–O(12)	2·069 (3)	O(12)–Li(2)–O(13)	94·1 (2)
Li(2)–O(13)	2·043 (3)	O(12)–Li(2)–O*W*(1)	126·3 (2)
Li(2)–O*W*(1)	1·914 (3)	O(12)–Li(2)–O*W*(2)	102·0 (2)
Li(2)–O*W*(2)	1·931 (3)	O(13)–Li(2)–O*W*(1)	100·8 (2)
		O(13)–Li(2)–O*W*(2)	121·3 (2)
		O*W*(1)–Li(2)–O*W*(2)	112·8 (2)

Li(1), which is linked to the four outer O atoms of the carboxylic groups (Fig. 1), shows a more regular environment. Li(2) is connected to the water O and two of the inner O atoms of the carboxylic groups of one of the hydrogen phthalate ions. The latter O atoms are less ionized because they form the intramolecular hydrogen bond; the respective Li–O distances are longer and, as a consequence, the tetrahedron is more distorted.

The water molecules form the following weak hydrogen bonds:

O*W*(1)–H*W*(11)···O(3)	2·807 (3) Å
O*W*(1)–H*W*(12)···O(4)	2·792 (3)
O*W*(2)–H*W*(21)···O(1)	2·857 (3).

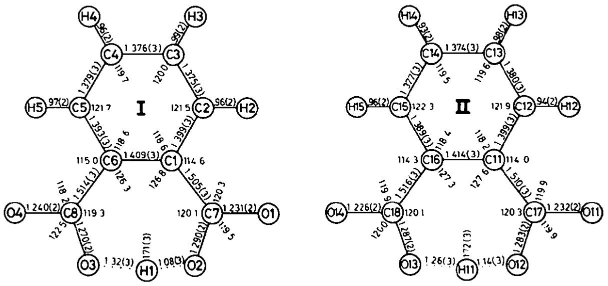
H*W*(22) is not involved in a hydrogen bond; it shows the largest temperature factor.

Distances and angles within the two hydrogen phthalate ions are presented in Fig. 1. The standard deviations of the angles are uniformly 0·2°. Similar to $LiHC_8O_4H_4.2H_2O$ and $LiHC_8O_4H_4.CH_3OH$, but in contrast to all other acid alkali phthalates, of which the structures are known, the hydrogen phthalate ions contain very short hydrogen bonds and are, consequently, quasi-planar. The present structure, therefore, presents a further example proving the previously stated assumption (Gonschorek & Küppers, 1975) that

Table 1. *Fractional atomic coordinates and, for* H, *isotropic thermal parameters* (*all* × 10^4)

Standard deviations given in parentheses refer to the last significant digits in the parameter values.

	x	*y*	*z*	*U* (Å^2)
C(1)	3743 (1)	3871 (1)	5130 (1)	
C(2)	3526 (1)	4517 (1)	6752 (1)	
C(3)	4050 (1)	4063 (2)	8211 (2)	
C(4)	4810 (1)	2953 (2)	8093 (2)	
C(5)	5062 (1)	2329 (1)	6510 (1)	
C(6)	4544 (1)	2765 (1)	5010 (1)	
C(7)	3068 (1)	4458 (1)	3673 (1)	
C(8)	4940 (1)	2005 (1)	3383 (1)	
O(1)	2648 (1)	5694 (1)	3893 (1)	
O(2)	2896 (1)	3646 (1)	2195 (1)	
O(3)	4250 (1)	1744 (1)	2037 (1)	
O(4)	5929 (1)	1635 (1)	3430 (1)	
C(11)	−278 (1)	7902 (1)	5426 (1)	
C(12)	−36 (1)	6960 (2)	3980 (2)	
C(13)	−640 (1)	6961 (2)	2403 (2)	
C(14)	−1518 (1)	7890 (2)	2236 (2)	
C(15)	−1799 (1)	8786 (1)	3653 (2)	
C(16)	−1203 (1)	8824 (1)	5257 (1)	
C(17)	559 (1)	7876 (1)	6981 (1)	
C(18)	−1615 (1)	9925 (1)	6637 (1)	
O(11)	1503 (1)	7419 (1)	6813 (1)	
O(12)	294 (1)	8338 (1)	8475 (1)	
O(13)	−1306 (1)	9835 (1)	8220 (1)	
O(14)	−2260 (1)	886 (1)	6244 (1)	
Li(1)	2644 (2)	7658 (2)	5252 (3)	
Li(2)	−1792 (2)	1519 (3)	9934 (3)	
O*W*(1)	3217 (1)	9360 (1)	9630 (1)	
O*W*(2)	1872 (1)	6418 (1)	584 (2)	
H(1)	3449 (15)	2716 (20)	2117 (20)	922 (55)
H(2)	2990 (10)	5285 (13)	6801 (14)	364 (32)
H(3)	3880 (11)	4523 (15)	9342 (17)	571 (40)
H(4)	5168 (10)	2591 (14)	9098 (16)	488 (37)
H(5)	5595 (10)	1551 (14)	6393 (14)	445 (35)
H(11)	9504 (16)	9007 (21)	8422 (21)	1091 (61)
H(12)	568 (11)	6329 (15)	4139 (16)	528 (39)
H(13)	420 (11)	3719 (16)	8556 (17)	598 (41)
H(14)	−1901 (11)	7932 (14)	1168 (17)	527 (39)
H(15)	−2429 (10)	9412 (14)	3511 (15)	444 (35)
H*W*(11)	3609 (14)	25 (19)	362 (20)	823 (58)
H*W*(12)	3577 (13)	−886 (17)	−1243 (20)	727 (51)
H*W*(21)	2085 (16)	6154 (21)	1538 (24)	1006 (71)
H*W*(22)	1872 (21)	5738 (27)	−125 (27)	1458 (108)



(*a*) (*b*)

Fig. 1. Distances (Å) and angles (°) in the hydrogen phthalate molecules, (*a*) molecule (I), (*b*) molecule (II).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33906 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

low coordination numbers of the cation favour the development of the intramolecular hydrogen bond. The planes of the carboxylic groups make the following angles with the least-squares planes through the C atoms:

[C(1) to C(8)]	with C(7)O(1)O(2)	23·4°
[C(1) to C(8)]	with C(8)O(3)O(4)	31·7
[C(11) to C(18)]	with C(17)O(11)O(12)	20·7
[C(11) to C(18)]	with C(18)O(13)O(14)	16·6.

The larger torsion angles of the first two carboxylic groups seem to cause the hydrogen bond O(2)···O(3) = 2·400 (2) to be longer than O(12)···O(13) = 2·390 (2) Å. Similar to the other acid lithium phthalates, the distances and angles within the benzene ring and the carboxylic groups are modified by the stress exerted by the short hydrogen bond.

The fact that H(2) is found to be located closer to O(2) than to O(3) within the hydrogen bond seems quite reasonable with regard to the environment of the carboxylic groups of molecule (I). The distances Li(1)–O(1) and Li(1)–O(4) are equal within the standard deviations. But O(4) and O(3) are involved in the hydrogen bonds to the water molecules previously mentioned. H(1), therefore, should be more closely attached to O(2) than to O(3) because O(2) shows no intermolecular linkages. Thus, O(2)–C(7) is longer than O(3)–C(8) and, consequently, O(1)–C(7) is shorter than O(4)–C(8).

In molecule (II), the higher symmetry with respect to linked neighbours makes it difficult to decide which of O(12) or O(13) the hydrogen H(11) should be closer to. A neutron diffraction study is in progress.

The quasi-planar hydrogen phthalate ions are packed in the crystal structure in a layer-like fashion. The angle between the two least-squares planes through molecules (I) and (II) is 15·5°. These mean planes have very roughly the orientation of the cleavage plane (110).

The topological configuration of the constituent groups in the present structure shows a close resemblance to that of $LiHC_8O_4H_4.CH_3OH$ if the methanol molecules are replaced by water molecules. This replacement causes an increase in density (from 1·474 to 1·524 Mg m^{-3}) and a distortion of the unit cell. If the lattice parameters in $LiHC_8O_4H_4.CH_3OH$ as reported by Adiwidjaja & Küppers (1978) are properly interchanged ($a \rightarrow -a$, $b \rightarrow c$, $c \rightarrow b$) to allow a comparison, the values to be compared with the present lattice parameters are: $a = 13{\cdot}692$, $b = 8{\cdot}467$, $c = 8{\cdot}023$ Å, $\alpha = 95{\cdot}29$, $\beta = 93{\cdot}28$, $\gamma = 83{\cdot}69°$.

The diffractometer was supplied by the Deutsche Forschungsgemeinschaft. Thanks are due to Dr K.-F. Hesse for collecting the X-ray intensities, to Dr Ilse Pallas for installing the computer programs, to Mrs U. Bennewitz for technical assistance, to Mr P. Drissen for aid in performing the calculations, and to Dr M. Barton for checking the English text. Computations were carried out at the Rechenzentrum der Universität Kiel (PDP 10).

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Tetramethylammonium Tri-μ-hydrido-dodecacarbonyltetraosmate

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Abstract. $[(CH_3)_4N][Os_4(CO)_{12}H_3]$, orthorhombic, *Pccn*, $a = 11{\cdot}647$ (8), $b = 12{\cdot}742$ (2), $c = 16{\cdot}865$ (3) Å, $U = 2502{\cdot}9$ Å^3, $Z = 4$, $D_c = 3{\cdot}115$ Mg m^{-3}, μ(Mo $K\alpha$) = 20·29 mm^{-1}. The structure was refined to an R of 0·037 for 1034 unique observed diffractometer data. The Os atoms define a distorted tetrahedron with crystallographic C_2 symmetry. The carbonyl ligands are all terminal, and their arrangement suggests that the hydrides bridge three Os–Os edges.

Introduction. Low-temperature IR and NMR studies on $[H_3Os_4(CO)_{12}]^-$ have indicated the existence of two