

Fig. 2. Stereographic packing diagram of the title compound (H atoms omitted for clarity).

As in other Ph_3PAuX complexes, there are no short $\text{Au}\cdots\text{Au}$ contacts of the kind often observed for Au^{I} . The molecular packing is shown in Fig. 2.

We thank the Fonds der Chemischen Industrie and the Deutsche Forschungsgemeinschaft for financial assistance, and Degussa AG for a gift of gold metal. Calculations were performed with the program system *SHELX76* (Sheldrick, 1976), locally modified by its

author. Professor P. L. Bellon kindly sent us the atom coordinates of his earlier structure determination.

References

- AHRLAND, S., DREISCH, K., NORÉN, B. & OSKARSSON, Å. (1987). *Acta Chem. Scand. Ser. A*, **41**, 173–177.
- BARRON, P. F., ENGELHARDT, L. M., HEALY, P., ODDY, J. & WHITE, A. H. (1987). *Aust. J. Chem.* **40**, 1545–1555.
- BELLON, P. L., MANASSERO, M. & SANSONI, M. (1969). *Ric. Sci.* **39**, 173–175.
- CLEGG, W. (1981). *Acta Cryst.* **A37**, 22–28.
- HORMANN, A. L., SHAW, C. F. III, BENNETT, D. W. & REIFF, W. M. (1986). *Inorg. Chem.* **25**, 3953–3957.
- JONES, P. G. (1981). *Gold Bull.* **14**, 102–118.
- JONES, P. G. (1983). *Gold Bull.* **16**, 114–124.
- JONES, P. G. (1984a). *Acta Cryst.* **C40**, 1320–1322.
- JONES, P. G. (1984b). *Acta Cryst.* **A40**, 660–662.
- JONES, P. G. (1986). *Gold Bull.* **19**, 46–57.
- NICHOLS, D. I. & CHARLESTON, A. S. (1969). *J. Chem. Soc. A*, pp. 2581–2583.
- ROGERS, D. (1981). *Acta Cryst.* **A37**, 734–741.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- VICENTE, J., CHICOTE, M. T., BERMÚDEZ, M. D., SANCHEZ-SANTANO, M. J. & JONES, P. G. (1988). *J. Organomet. Chem.* In the press.

Acta Cryst. (1988). **C44**, 2093–2095

Structure of Lithium Phthalate Hemitrihydrate

By H. KÜPPERS

Mineralogisches Institut der Universität Kiel, Olshausenstrasse 40, D-2300 Kiel 1, Federal Republic of Germany

(Received 23 March 1988; accepted 14 July 1988)

Abstract. $2(2\text{Li}^+\cdot\text{C}_8\text{H}_4\text{O}_4^{2-}\cdot\frac{3}{2}\text{H}_2\text{O})$, $M_r=410.0$, triclinic, $P\bar{1}$, $a=7.411(2)$, $b=9.849(2)$, $c=12.511(2)$ Å, $\alpha=93.60(2)$, $\beta=91.45(1)$, $\gamma=98.18(3)^\circ$, $V=901.6(3)$ Å³, $Z=2$, $D_x=1.5103(6)$ Mg m⁻³, $\lambda(\text{Mo K}\alpha)=0.71069$ Å, $\mu=0.114$ mm⁻¹, $F(000)=420$, room temperature, $wR=0.031$ for 1565 reflections [$I > 2.5\sigma(I)$]. The structure shows a layer-like sandwich arrangement [parallel to (001)] such that two layers of phthalate ions direct their carboxylic groups towards an inner layer of Li ions. The weak van der Waals forces between these sandwiches cause the observed perfect cleavage along that plane. Of four independent Li ions two have fourfold and two have fivefold coordination.

Introduction. The system Li_2O –phthalic acid– H_2O has gained special interest because of the occurrence of two acid salts containing extremely short hydrogen bonds (Gonschorek & Küppers, 1975; Küppers, Taku-

sagawa & Koetzle, 1985). The existence of the neutral compound with composition $2[\text{Li}_2(\text{C}_6\text{H}_4)(\text{COO})_2]\cdot 3\text{H}_2\text{O}$ was reported by Smith, Sturm & Ely (1935) in a study on equilibria in the system mentioned above.

Experimental. Single crystals were grown from an aqueous solution of a stoichiometric mixture of Li_2CO_3 and phthalic acid by slow evaporation of the solvent. The crystals grow as (001) plates which are terminated by small faces of types $\{100\}$, $\{011\}$, $\{101\}$, $\{1\bar{1}0\}$, $\{1\bar{1}1\}$, $\{\bar{1}01\}$ and $\{0\bar{1}1\}$. The crystal used for the present study measured $0.08 \times 0.15 \times 0.25$ mm. Lattice parameters were determined by a least-squares fit to 20 diffractometrically measured Bragg angles in the range $7 < 2\theta < 30^\circ$. 7474 reflections were collected with a Siemens AED2 diffractometer in the θ range $2\text{--}30^\circ$ using $\theta\text{--}2\theta$ scanning technique. 1476 of these were rejected as unobserved. Among the remaining 5998 were 4074 unique (internal R value was 0.013).

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) of non-H atoms

U_{eq} is one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	5590 (5)	4030 (3)	7595 (3)	200 (13)
C(2)	5309 (6)	3909 (4)	8684 (4)	308 (16)
C(3)	6723 (6)	3822 (4)	9418 (3)	380 (17)
C(4)	8484 (6)	3880 (4)	9045 (4)	330 (17)
C(5)	8796 (6)	4000 (4)	7966 (4)	291 (15)
C(6)	7372 (5)	4077 (3)	7224 (3)	180 (12)
C(7)	3928 (5)	4107 (4)	6863 (3)	214 (16)
C(8)	-2204 (5)	4243 (4)	6063 (3)	218 (13)
O(1)	-646 (3)	4015 (2)	5757 (2)	260 (8)
O(2)	-3383 (3)	4616 (2)	5443 (2)	276 (9)
O(3)	3235 (3)	5187 (2)	6949 (2)	360 (10)
O(4)	3338 (3)	3060 (2)	6253 (2)	251 (8)
C(11)	1941 (4)	275 (3)	2378 (3)	196 (12)
C(12)	2606 (5)	-301 (4)	1442 (4)	307 (16)
C(13)	2516 (5)	332 (4)	483 (4)	369 (15)
C(14)	1761 (5)	1528 (4)	441 (4)	372 (16)
C(15)	1141 (5)	2141 (5)	1371 (4)	321 (15)
C(16)	1243 (4)	1521 (3)	2343 (3)	198 (13)
C(17)	1825 (6)	-520 (4)	3369 (3)	274 (15)
C(18)	689 (5)	2247 (4)	3364 (3)	235 (15)
O(11)	3307 (3)	-724 (3)	3820 (2)	368 (10)
O(12)	256 (3)	-1006 (2)	3649 (2)	375 (10)
O(13)	1756 (3)	2343 (2)	4173 (2)	306 (9)
O(14)	-777 (3)	2760 (2)	3327 (2)	301 (9)
OW(1)	5612 (5)	1763 (3)	4319 (3)	321 (11)
OW(2)	4123 (6)	7515 (5)	8146 (3)	450 (13)
OW(3)	2958 (6)	368 (4)	7345 (3)	474 (13)
Li(1)	810 (8)	2494 (6)	5622 (5)	276 (21)
Li(2)	4259 (8)	3377 (7)	4735 (5)	322 (21)
Li(3)	6959 (9)	1532 (7)	2933 (5)	360 (22)
Li(4)	1443 (8)	5646 (6)	5875 (5)	281 (22)

Index ranges were $h-10/10$, $k-14/14$, $l0/18$. Three standard reflections monitored every 60 min showed no systematic decay. No absorption correction was made. Scattering factors for neutral atoms were taken from Cromer & Mann (1968). The positions of the non-hydrogen atoms were determined by direct methods. Difference Fourier syntheses yielded the positions of the H atoms. Refinement on F with 1565 reflections with $I > 2.5\sigma(I)$ yielded a final $wR = 0.031$ ($R = 0.056$), with weights $w = 1/\sigma^2$ and maximum $\Delta/\sigma = 0.017$. Anisotropic temperature factors were applied for non-H atoms and individual isotropic temperature factors for H atoms. Residual electron density ranged between -0.46 and $+0.37 \text{ e \AA}^{-3}$. Programs used: *SHELX76* (Sheldrick, 1976), *SHELXS86* (Sheldrick, 1986), *ORFFE* (Busing, Martin & Levy, 1964) and *ORTEPII* (Johnson, 1976). Atomic coordinates and equivalent isotropic thermal parameters U_{eq} for the non-H atoms are listed in Table 1.*

Discussion. The packing within the unit cell is shown by a stereoscopic *ORTEP* plot in Fig. 1. The coordination spheres of all eight Li ions within the unit cell were

* Lists of structure factors, anisotropic temperature factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51245 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

complemented by also including O atoms of carboxylic groups belonging to phthalate ions in adjacent cells. The C atoms of these carboxylic groups are marked by dashed contours in order to indicate the incomplete phthalate ion. The structure shows a layer-like set-up. Regions of heteropolar bonds (Li—O) extend within a slab parallel to the ab plane in the middle of the chosen unit cell, whereas within slabs around the terminating ab faces of the chosen unit cell the 'back sides' of the phthalate ions, *i.e.* the benzene rings, are connected by comparatively weak van der Waals bonds. This finding is in accord with the observed perfect cleavage along (001).

Distances and angles within the phthalate ions are in the usual range. The angles between the carboxylic groups and mean planes through the benzene rings are as follows: C(1) to C(6) with O(1)—C(8)—O(2) 14.6° and with O(3)—C(7)—O(4) 72.5° , C(11) to C(16) with O(11)—C(17)—O(12) 67.0° and with O(13)—C(18)—C(14) 45.4° . Distances and angles within the coordination polyhedra around the Li ions are listed in Table 2. The coordination numbers of Li(1) and Li(3) are both four, and those of Li(2) and Li(4) are both five. The polyhedron around Li(1) is formed exclusively by O atoms of carboxylic groups and is a heavily distorted tetrahedron (the six O—Li—O angles range between 91 and 124°). The polyhedron around Li(3), formed by three water O atoms and only one carboxylic O atom, is a more regular tetrahedron (O—Li—O angles ranging between 99 and 119°), possibly because the water molecules can more easily move to achieve a regular environment than the O atoms belonging to the unwieldy phthalate molecules. The Li(2) polyhedron can be approximately characterized as being a tetragonal pyramid with an O(4) as apex. The angles O(4)—Li(2)—O (O standing for the remaining four atoms forming the base of the pyramid) are near 90° [86.6 to O(13), 88.6 to O(2), 98.2 to O(2)', 106.1° to OW(1)] and the angles O—Li—O within the base range between

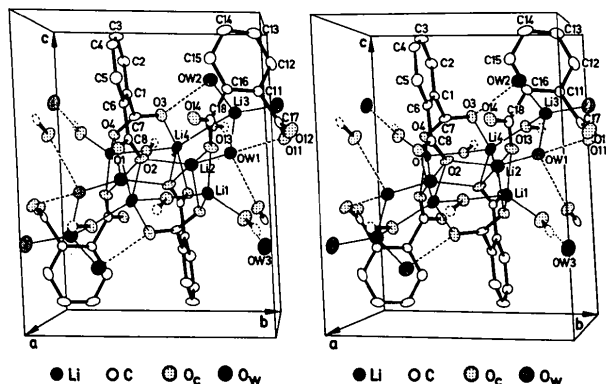


Fig. 1. Stereoscopic view of one unit cell, projected along a^* (*ORTEPII*; Johnson, 1976). H atoms were omitted. Hydrogen bonds are represented by dashed lines. O_c are oxygen atoms of carboxylic groups and O_w are oxygen atoms of water molecules.

Table 2. Distances (Å) and angles (°) in the Li—O polyhedra

Li(1)—O(12)	1.865 (7)	Li(3)—OW(3 ^{iv})	1.892 (8)
Li(1)—O(13)	1.964 (7)	Li(3)—OW(2 ^v)	1.914 (8)
Li(1)—O(1)	1.969 (7)	Li(3)—O(14 ⁱⁱ)	1.956 (6)
Li(1)—O(4)	2.004 (6)	Li(3)—OW(1)	2.040 (8)
All others	> 3.11	All others	> 3.24
Li(2)—OW(1)	2.044 (8)	Li(4)—O(14 ⁱⁱⁱ)	1.936 (7)
Li(2)—O(4)	2.061 (7)	Li(4)—O(3)	1.983 (7)
Li(2)—O(13)	2.069 (6)	Li(4)—O(1)	2.062 (6)
Li(2)—O(2 ⁱⁱ)	2.122 (6)	Li(4)—O(1 ⁱⁱⁱ)	2.170 (7)
Li(2)—O(2 ⁱⁱⁱ)	2.189 (7)	Li(4)—O(2 ⁱⁱⁱ)	2.243 (7)
All others	> 3.11	All others	> 3.13
O(12)—Li(1)—O(13)	124.4 (3)	OW(3 ^{iv})—Li(3)—OW(2 ^v)	118.0 (4)
O(12)—Li(1)—O(1)	110.5 (3)	OW(3 ^{iv})—Li(3)—O(14 ⁱⁱ)	119.1 (4)
O(12)—Li(1)—O(4)	107.3 (3)	OW(3 ^{iv})—Li(3)—OW(1)	107.5 (3)
O(13)—Li(1)—O(1)	110.3 (3)	OW(2 ^v)—Li(3)—O(14 ⁱⁱ)	102.7 (3)
O(13)—Li(1)—O(4)	91.1 (3)	OW(2 ^v)—Li(3)—OW(1)	108.7 (4)
O(1)—Li(1)—O(4)	111.3 (3)	O(14 ⁱⁱ)—Li(3)—OW(1)	99.0 (3)
OW(1)—Li(2)—O(4)	106.1 (3)	O(14 ⁱⁱⁱ)—Li(4)—O(3)	97.1 (3)
OW(1)—Li(2)—O(13)	93.5 (3)	O(14 ⁱⁱⁱ)—Li(4)—O(1)	112.4 (3)
OW(1)—Li(2)—O(2 ⁱⁱ)	94.0 (3)	O(14 ⁱⁱⁱ)—Li(4)—O(1 ⁱⁱⁱ)	102.4 (3)
OW(1)—Li(2)—O(2 ⁱⁱⁱ)	155.3 (4)	O(14 ⁱⁱⁱ)—Li(4)—O(2 ⁱⁱⁱ)	133.3 (3)
O(4)—Li(2)—O(13)	86.6 (2)	O(3)—Li(4)—O(1)	106.1 (3)
O(4)—Li(2)—O(2 ⁱⁱ)	88.6 (2)	O(3)—Li(4)—O(1 ⁱⁱⁱ)	151.4 (3)
O(4)—Li(2)—O(2 ⁱⁱⁱ)	98.2 (3)	O(3)—Li(4)—O(2 ⁱⁱⁱ)	90.8 (3)
O(13)—Li(2)—O(2 ⁱⁱ)	172.0 (4)	O(1)—Li(4)—O(1 ⁱⁱⁱ)	85.7 (2)
O(13)—Li(2)—O(2 ⁱⁱⁱ)	92.2 (3)	O(1)—Li(4)—O(2 ⁱⁱⁱ)	109.3 (3)
O(2 ⁱⁱ)—Li(2)—O(2 ⁱⁱⁱ)	82.1 (2)	O(1 ⁱⁱⁱ)—Li(4)—O(2 ⁱⁱⁱ)	60.5 (2)
		O(14 ⁱⁱⁱ)—Li(4)—C(8 ⁱⁱⁱ)	120.4 (4)
		O(3)—Li(4)—C(8 ⁱⁱⁱ)	120.9 (3)
		O(1 ⁱⁱⁱ)—Li(4)—C(8 ⁱⁱⁱ)	99.7 (2)

Symmetry codes: (i) $-x, -y, 1-z$; (ii) $1+x, y, z$; (iii) $-x, 1-y, 1-z$; (iv) $1-x, -y, 1-z$; (v) $1-x, 1-y, 1-z$.

82 and 94°. In the polyhedron around Li(4) the two farthest O atoms [O(1) and O(2)] belong to the same carboxylic group. Since the relatively short distance between O(1) and O(2) is restrained by the covalent forces within the carboxylic group the angle O(1)—Li(4)—O(2) is only 60.5°. The remaining O atoms of this polyhedron are arranged such that if the closely spaced O(1) and O(2) are regarded as a 'single' anion, then using the bisector of O(1)—Li(4)—O(2) [*i.e.*

approximately the line Li(4)—C(8)] the polyhedron has roughly the appearance of a distorted tetrahedron. The angles between the appropriate bonds emanating from Li(4) range between 97.1 and 120.9°.

OW(1) is the only water oxygen atom exhibiting two well defined hydrogen bonds [OW(1)—O(11) = 2.747 (5) and OW(1)—O(11)' = 2.806 (4) Å] and the appropriate H atoms could be resolved clearly in the course of the structure determination. In contrast, both OW(2) and OW(3) form only one distinct hydrogen bond [OW(2)—O(3) = 2.650 (5) and OW(3)—O(12)' = 2.827 (5) Å, respectively] and only the corresponding H atoms could be clearly located. The other two H atoms seem to be distributed irregularly and show unrealistic temperature factors.

Thanks are due to Dr K.-F. Hesse for collecting the intensities and to Mrs U. Bennwitz for retouching the drawings.

References

- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.
- GONSCHOREK, W. & KÜPPERS, H. (1975). *Acta Cryst.* B31, 1068–1072.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KÜPPERS, H., TAKUSAGAWA, F. & KOETZLE, T. F. (1985). *J. Chem. Phys.* 82, 5636–5647.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure refinement. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.
- SMITH, S. B., STURM, W. A. & ELY, E. C. (1935). *J. Am. Chem. Soc.* 57, 2406–2408.

Acta Cryst. (1988). C44, 2095–2097

The Structure of {*N,N*-Bis[(3,5-dimethyl-1-pyrazolyl)methyl]aminoethane}iodocopper(I)

BY YVON C. M. PENNING, WILLEM L. DRIESSEN* AND JAN REEDIJK

Department of Chemistry, Gorlaeus Laboratories, Leiden University, PO Box 9502, 2300 RA Leiden, The Netherlands

(Received 29 March 1988; accepted 14 July 1988)

Abstract. [Cu(C₁₄H₂₃N₅)I], $M_r = 451.82$, monoclinic, $C2/c$, $a = 24.324$ (6), $b = 8.875$ (2), $c = 17.020$ (5) Å, $\beta = 95.89$ (2)°, $V = 3654.6$ Å³, $Z = 8$, $D_x = 1.64$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu =$

28.71 cm⁻¹, $F(000) = 891$, $T = 293$ K, final $R = 0.036$ for 2570 significant reflections. The copper(I) ion is coordinated to three ligand N atoms and to the I⁻ ion. The I⁻ ion is non-bridging. The geometry of the Cu environment can be described as a distorted trigonal pyramid, in which the amine N atom forms the top of

* Author to whom correspondence should be addressed.