

range from 1.960 (4) to 1.985 (4) Å. The C atoms of the butadiene moieties are planar (0.0001 and 0.006 Å r.m.s.d.). Each butadiene plane is perpendicular [89.8 (1)°] to the basal plane of the tetragonal pyramid. Fe(2)—C(24), 2.110 (4) Å, is marginally shorter than the other three Fe—terminal C-atom distances [2.123 (4) to 2.130 (5) Å]. The terminal H atoms of the butadiene groups are not coplanar with these units, and the interplanar angles between the terminal H—C—H groups and the butadiene planes range from 37 (2) to 42 (2)°. The three C—C bonds of each butadiene moiety are statistically equivalent [1.401 (5) to 1.414 (5) Å]. Similar effects have been reported for other Fe(CO)<sub>3</sub>-butadiene complexes (Cotton, Day, Frenz, Hardcastle & Troup, 1973; Meier, Cherpillod, Boschi, Roulet, Vogel, Mahaim, Pinkerton, Schwarzenbach & Chapuis, 1980; Meier, Pinkerton, Roulet, Vogel & Schwarzenbach, 1981; Pinkerton, Chapuis, Vogel, Hanisch, Narbel, Boschi & Roulet, 1979; Sustmann, Bohm & Saver, 1979). Least-squares planes and other interplanar angles are given in the supplementary material.

The cyclopentanone ring system is planar (0.03 Å r.m.s.d.) and the two norbornane rings are fused to it in an *exo-trans-exo* arrangement. The methylene bridges C(20) and C(21) are bent toward the cyclopentanone portion of the molecule as indicated by the non-equivalent interplanar angles, *e.g.* C(9)C(20)C(19) makes angles of 130.0 (3) and 121.3 (3)° with C(7)C(8)C(9)C(19) and C(9)C(10)C(18)C(19), respectively. All chemically equivalent bonds in the organic portion of the complex are statistically equivalent.

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## Structure of Bis(oxamato-*O,O'*)lead(II) Monohydrate

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**Abstract.** [Pb(C<sub>2</sub>H<sub>2</sub>NO<sub>3</sub>)<sub>2</sub>].H<sub>2</sub>O, *M<sub>r</sub>* = 401.3, triclinic, *P* $\bar{1}$ , *a* = 6.727 (1), *b* = 7.293 (1), *c* = 9.814 (1) Å,  $\alpha$  = 81.70 (1),  $\beta$  = 75.93 (1),  $\gamma$  = 64.06 (1)°, *V* = 420 Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 3.18 Mg m<sup>-3</sup>,  $\lambda$ (Ag *K*α),  $\mu$  = 10.5 mm<sup>-1</sup>, *F*(000) = 362, *R* = 0.027, *wR* = 0.031, *S* = 0.93. The coordination polyhedron of the metal ion is a distorted pentagonal dipyramid. Each Pb<sup>2+</sup> ion is

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bonded to seven O atoms with six belonging to four oxamate ions and one to the water molecule. The water molecule is also involved in three hydrogen bonds that reinforce the stability of the hydrate and the cohesion of the structure.

**Introduction.** This X-ray structure determination is part of a more general study on the interaction of metals with small ligands of the type *RCOCOR'* (*R* = OH, NH<sub>2</sub>, *R'* = OH, NH<sub>2</sub>). Crystals of the title compound

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Table 1. Atomic coordinates of non-H atoms and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

	x	y	z	$B_{eq}(\text{\AA}^2)$
Pb	0.57040 (2)	0.90442 (2)	0.70303 (2)	1.50 (1)
O(11)	0.6058 (6)	0.7777 (5)	0.4698 (4)	1.94 (9)
O(12)	0.6761 (7)	0.5090 (5)	0.3500 (4)	2.10 (11)
O(13)	0.7926 (7)	0.5098 (6)	0.6750 (4)	2.27 (11)
N(11)	0.8777 (7)	0.2484 (6)	0.5436 (5)	2.00 (11)
C(11)	0.6805 (7)	0.5881 (6)	0.4489 (5)	1.47 (10)
C(12)	0.7901 (8)	0.4421 (7)	0.5670 (5)	1.62 (11)
O(21)	0.1931 (6)	0.9393 (6)	0.7121 (4)	1.97 (10)
O(22)	-0.0978 (7)	0.8768 (9)	0.8301 (5)	3.39 (15)
O(23)	0.4230 (6)	0.7427 (6)	0.9205 (4)	2.18 (11)
N(21)	0.1168 (8)	0.6916 (8)	1.0416 (5)	2.35 (13)
C(21)	0.0952 (8)	0.8651 (7)	0.8130 (5)	1.78 (12)
C(22)	0.2251 (8)	0.7585 (7)	0.9345 (6)	1.84 (12)
OW	0.3687 (8)	1.2199 (7)	0.8696 (5)	2.58 (12)

were prepared by the silica-gel method at pH values 5.2–7.8 (Michaelides & Skoulika, 1988).

**Experimental.** Transparent  $0.3 \times 0.2 \times 0.2$  mm crystal used. Automated CAD-4 Enraf-Nonius diffractometer with incident-beam (Ag  $K\alpha$ ) graphite monochromator. 25 centered reflections within  $10 < 2\theta < 24^\circ$  used for determining lattice parameters. 2935 reflections measured,  $[(\sin\theta)/\lambda]_{\max} = 0.75 \text{ \AA}^{-1}$ , range of  $hkl$ :  $-10 < h < 10$ ,  $-11 < k < 11$ ,  $0 < l < 14$ ;  $\omega, 2\theta$  scan mode. 2585 reflections used [ $I > 3\sigma(I)$ ]. Data corrected for Lorentz and polarization effects; empirical absorption correction based on  $\psi$  scans, empirical isotropic correction for secondary extinction  $x = 9.7 (3) \times 10^{-3}$  [ $F'_c = F_c (1 - 0.0001xF_c^2/\sin\theta)$ ]. Data reduction with *SDP* package (Enraf-Nonius, 1979). Structure solved by Patterson and Fourier methods. Least-squares program *SHELX76* (Sheldrick, 1976),  $\sum w(|F_o| - |F_c|)^2$  minimized where  $w = 0.6189/[\sigma^2(F) + 0.00185F^2]$ . Positions of H atoms of oxamate ions calculated from standard geometry, anisotropic thermal motion for all non-H atoms,  $(\Delta/\sigma)_{\max} = 0.007$ ,  $R(F) = 0.027$ ,  $wR(F) = 0.031$ ,  $S = 0.93$ , maximum residual density  $3.03 \text{ e \AA}^{-3}$  close to Pb and  $0.73 \text{ e \AA}^{-3}$  elsewhere. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** Atomic coordinates are given in Table 1, interatomic distances and angles in Table 2.\* Each Pb atom is bonded to seven O atoms. The two oxamate ions of the asymmetric unit, one tridentate and the other bidentate, chelate to Pb through amidic and carboxylic O atoms (Fig. 1). The remaining coordination positions are occupied by the water O atom, the second

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44730 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

Oxamate ions			
O(11)–C(11)	1.276 (6)	O(21)–C(21)	1.243 (6)
O(12)–C(11)	1.211 (7)	O(22)–C(21)	1.233 (8)
O(13)–C(12)	1.239 (7)	O(23)–C(22)	1.259 (7)
C(11)–C(12)	1.546 (7)	C(21)–C(22)	1.560 (7)
N(11)–C(12)	1.302 (6)	N(21)–C(22)	1.289 (7)
Coordination polyhedron of Pb			
Pb–O(11)	2.510 (4)	Pb–O(23)	2.489 (4)
Pb–O(11 <sup>ii</sup> )	2.663 (3)	Pb–O(22')	2.732 (6)
Pb–O(13)	2.618 (4)	Pb–OW	2.662 (5)
Pb–O(21)	2.418 (4)		
O(21)–O(13)	3.843 (4)	O(22)–OW	3.322 (5)
O(21)–O(23)	2.717 (5)	O(22)–O(11 <sup>ii</sup> )	4.680 (6)
O(21)–O(11)	3.080 (4)	O(13)–O(23)	3.073 (4)
O(21)–OW	3.448 (7)	O(11)–O(13)	2.719 (4)
O(21)–O(11 <sup>ii</sup> )	3.078 (5)	O(11)–O(11 <sup>ii</sup> )	2.999 (6)
O(22)–O(13)	3.670 (8)	OW–O(11 <sup>ii</sup> )	3.291 (5)
O(22)–O(23)	3.640 (7)	OW–O(23)	3.311 (7)
O(22)–O(11)	4.735 (7)		
O(11)–Pb–O(22')	129.1 (1)	OW–Pb–O(21)	85.4 (1)
O(11)–Pb–O(21)	77.3 (1)	OW–Pb–O(23)	79.9 (1)
O(11)–Pb–O(13)	64.0 (1)	O(23)–Pb–O(22')	88.3 (2)
O(11)–Pb–O(11 <sup>ii</sup> )	70.8 (1)	O(23)–Pb–O(21)	67.2 (1)
O(11 <sup>ii</sup> )–Pb–O(22')	120.3 (1)	O(23)–Pb–O(13)	73.9 (1)
O(11 <sup>ii</sup> )–Pb–O(21)	74.4 (1)	O(13)–Pb–O(22')	86.6 (2)
O(11 <sup>ii</sup> )–Pb–OW	76.3 (1)	O(13)–Pb–O(21)	99.4 (1)
OW–Pb–O(22')	76.0 (1)	O(22')–Pb–O(21)	151.6 (1)

## Probable hydrogen bonds

OW–H...O(23 <sup>iii</sup> )	2.858
OW–H...O(12 <sup>ii</sup> )	2.685
OW...H–N(21 <sup>iv</sup> )	2.970

Symmetry code: (i)  $1 + x, y, z$ ; (ii)  $1 - x, 2 - y, 1 - z$ ; (iii)  $1 - x, 2 - y, 2 - z$ ; (iv)  $-x, 2 - y, 2 - z$ .

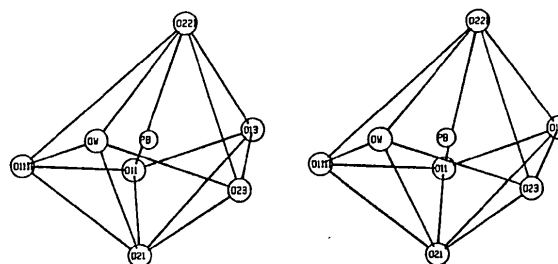


Fig. 1. Stereoscopic view of the coordination polyhedron about the Pb atom.

carboxylic O atom of the tridentate oxamate unit translated along  $[100]$  and the carboxylic O atom of the bidentate unit obtained with the symmetry operation  $1 - x, 2 - y, 1 - z$ . The coordination polyhedron is a distorted pentagonal dipyramid (Fig. 1). The O(21) and O(22) atoms form the apexes of the dipyramid; the distance Pb–O(21) is the shortest (2.420  $\text{\AA}$ ) and Pb–O(22) the longest (2.732  $\text{\AA}$ ). The basal Pb–O

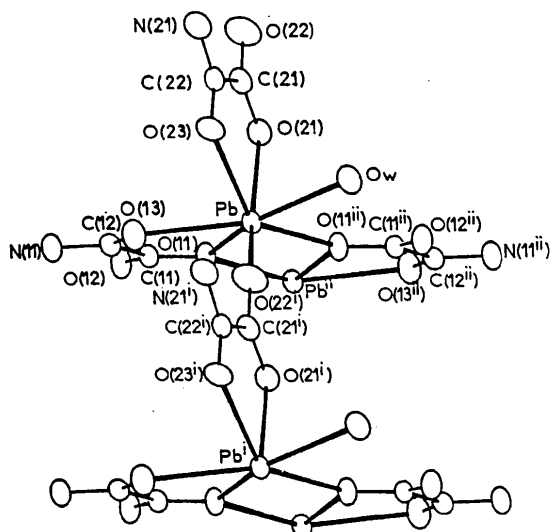


Fig. 2. Part of the polymeric 'double chain', showing the atom labelling.

lengths are in the range 2.489–2.662 Å. There is a distortion of the basal atoms away from O(22) and towards O(21): all O(21)–Pb–O (basal) angles, with the exception of O(21)–Pb–O(13) (Table 2), are  $< 90^\circ$ . This feature is also observed in other cases and can be explained by the assumption that the stereochemically active lone pair of Pb is directed towards O(22) and pushes all O atoms to the other side of the metal atom (Rajaram & Mohana Rao, 1982; Clegg, Little & Straughan, 1986).

The geometry of the oxamate ions is not unusual (Pellinghelli, Tiripicchio & Tiripicchio Camellini, 1972; Braibanti, Pellinghelli, Tiripicchio & Tiripicchio Camellini, 1971; Skoulika, Michaelides & Aubry, 1988). The two oxamates of the asymmetric unit fill different

structural roles. One is tridentate and ensures, by the coordination of its second carboxylic O atom O(22) to Pb, the formation of polymeric chains parallel to *a*. The second oxamate ion is bidentate and, by bridging, through O(11), two Pb atoms related by the center of symmetry, permits the interaction between two parallel chains and the formation of 'double chains' (Fig. 2). The Pb<sub>2</sub>O<sub>2</sub> parallelograms formed in this way are almost coplanar with the oxamate ions containing the O atoms (distance of the Pb atoms from the mean plane of the two oxamates –0.323 and 0.257 Å).

The water molecule that is coordinated to Pb is also involved in three hydrogen bonds. For this purpose it uses both H atoms to form hydrogen bonds to two O atoms of the oxamate ions and acts as acceptor in an additional hydrogen bond to an N atom. By means of these interactions the 'double chains' are hydrogen bonded between them and the overall structure of the hydrate stabilized.

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## Structure of (2,2'-Bipyridyl)carbonyl( $\eta$ -3,4-diphenylcyclobutene-1,2-dione)nickel(0) Toluene Solvate

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**Abstract.** [Ni(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)(C<sub>16</sub>H<sub>10</sub>O<sub>2</sub>)(CO)].0.5C<sub>7</sub>H<sub>8</sub>, *M<sub>r</sub>* = 523.3, monoclinic, *P*2<sub>1</sub>/*a*, *a* = 15.230 (4), *b* = 9.489 (2), *c* = 17.652 (2) Å,  $\beta$  = 96.95 (1)°, *V* = 2532.2 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.38 Mg m<sup>-3</sup>,  $\lambda$ (Mo *K* $\alpha$ ) = 0.71069 Å,  $\mu$  = 0.80 mm<sup>-1</sup>, *F*(000) = 1084, *T* =

291 K. Final *R* = 0.056 (*wR* = 0.066) for 1992 unique observed reflections. The cyclobutenedione ring is coordinated to the Ni atom *via* its olefinic bond, with the plane of the cyclobutene ring making an angle of 87° to that of the three-membered ring defined by the