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## Structure of a Lead(II) Complex of a New 13-Membered Macrocycle with Two Pendant Carboxymethyl Groups

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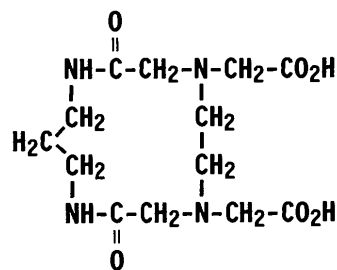
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**Abstract.** (Aqua)[2,9-dioxo- $\kappa^2O, O'-1,4,7,10$ -tetraaza- $\kappa^2N^4, N^7$ -4,7-cyclotridecanediacetato(2-)- $\kappa^2O, O'$ ]-lead(II) dihydrate,  $[\text{Pb}(\text{C}_{13}\text{H}_{20}\text{N}_4\text{O}_6)(\text{H}_2\text{O})_2]\cdot 2\text{H}_2\text{O}$ ,  $M_r = 589.56$ , monoclinic,  $P2_1/c$ ,  $a = 8.999$  (1),  $b = 23.055$  (1),  $c = 9.382$  (1) Å,  $\beta = 108.58$  (1)°,  $V = 1845.1$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.12$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 92.6$  cm<sup>-1</sup>,  $F(000) = 1144$ ,  $T = 294$  K,  $R = 0.026$  for 2829 independent reflections with  $I > 3\sigma(I)$ . A Pb<sup>II</sup> ion is coordinated to six atoms from a macrocyclic ligand and an O atom from water. An additional weak coordination bond with an O atom from an adjacent metal chelate molecule results in a binuclear structure. The resulting eight-coordination geometry around a Pb<sup>II</sup> ion is an unusually distorted triangulated dodecahedron.

**Introduction.** The coordination chemistry of macrocyclic ligands with pendant functional groups has attracted a growing interest (Bernhardt & Lawrance, 1990; Parker, 1990). In our previous study, new 12-membered and 24-membered macrocycles with pendant carboxymethyl groups were synthesized by condensation of ethylenediaminetetraacetic dianhydride with ethylenediamine, and the copper(II)

and the manganese(II) complexes of these ligands were characterized by X-ray crystal analyses (Inoue, Villegas, Asano, Nakamura, Inoue & Fernando, 1992). These metal chelates have novel molecular structures due to the coordination of the amide O atoms. The structure of a metal chelate with a macrocyclic ligand is sensitively dependent on the relative sizes of the metal and the macrocycle. In the present study we have synthesized a new 13-membered macrocycle (I) by a condensation between 1,3-propanediamine and ethylenediaminetetraacetic dianhydride, and carried out an X-ray study of its Pb<sup>II</sup> complex. The coordination polyhedron around the Pb<sup>II</sup> ion is found to be unusually distorted due to the nature of the coordinated ligand.



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**Experimental.** The 13-membered macrocycle was prepared by condensation between ethylenediaminetetraacetic dianhydride and 1,3-propanediamine in high dilution in dimethylformamide. The reaction conditions were similar to those reported for the condensation reaction between ethylenediaminetetraacetic dianhydride and ethylenediamine (Inoue, Villegas, Asano, Nakamura, Inoue & Fernando, 1992). Calc. for C<sub>13</sub>H<sub>22</sub>N<sub>4</sub>O<sub>6</sub>: C, 47.27; H, 6.67; N, 16.97. Found: C, 47.56; H, 6.76; N, 16.91. The Pb<sup>II</sup> complex was obtained by reacting the resulting ligand with lead(II) carbonate in water, and purified by precipitating the lead complex from an aqueous solution by the addition of ethanol. Single crystals suitable for X-ray analysis were grown from an aqueous solution by slow evaporation.

A colorless crystal of approximate dimensions 0.38 × 0.35 × 0.22 mm was sealed in a glass capillary. The data collection was performed with monochromated Mo K $\alpha$  radiation on an Enraf-Nonius CAD-4 diffractometer. The cell constants were determined from 25 reflections in the range  $26 < 2\theta < 36^\circ$ . From the systematic absences ( $h0l$ ,  $l = 2n + 1$ ;  $0k0$ ,  $k = 2n + 1$ ) the space group was determined to be  $P2_1/c$  (No. 14). The data were collected using the  $\omega$ - $2\theta$  scan method at a rate of  $1-7^\circ \text{ min}^{-1}$  in the range  $0 < 2\theta \leq 50^\circ$  ( $h = 0$  to 10,  $k = 0$  to 27,  $l = -11$  to 10),  $(\sin\theta)/\lambda \leq 0.5940 \text{ \AA}^{-1}$ . Three standard reflections were monitored every 60 min and were constant within experimental error. A total of 3567 reflections were collected, 3245 of which were unique and not systematically absent. An empirical absorption correction based on a series of  $\psi$  scans was applied to the data. Relative transmission coefficients ranged from 0.552 to 0.998 with an average value of 0.852. The structure was solved by the Patterson heavy-atom method which revealed the position of the Pb atom. The remaining atoms were located in succeeding difference Fourier syntheses. H atoms bonded to carbon or nitrogen were included in the refinement at standardized positions with a fixed X—H distance of 0.95 Å; those of the water molecules were located in the difference Fourier map, and constrained to ride on the atoms to which they are bonded. Non-H atoms were anisotropically refined. The full-matrix least-squares refinements were carried out using 2829 reflections with  $I > 3\sigma(I)$ ;  $\sum w(|F_o| - |F_c|)^2$  was minimized;  $w$  was calculated from  $4F_o^2/\sigma^2(F_o^2)$ . Scattering factors used were from Cromer & Waber (1974). The anomalous-dispersion effects were included in  $F_c$ ; the values for  $f'$  and  $f''$  were those of Cromer (1974). The final agreement factors for 244 variables were;  $R = 0.026$  and  $wR = 0.038$ . The standard deviation of an observation of unit weight was 1.57. The ratio of  $\Delta_{\text{max}}/\sigma$  was less than 0.01. The highest peak in the final difference Fourier map was found near the Pb atom with a height of

$1.1(1) \text{ e \AA}^{-3}$ ; the most negative peak had a minimum of  $-0.3(1) \text{ e \AA}^{-3}$ . All calculations were performed on a VAX computer with the *MolEN* (Fair, 1990) program.

**Discussion.** Table 1\* contains the positional and the thermal parameters. Selected bond distances, angles and torsion angles are collected in Table 2. Fig. 1 shows the *ORTEP* diagram (Johnson, 1965) of the Pb<sup>II</sup> chelate structure. The crystal packing diagram is shown in Fig. 2.

The crystal analysis demonstrated that the 13-membered macrocycle (I) forms a neutral Pb<sup>II</sup> complex. A ligand molecule is coordinated to the Pb<sup>II</sup> ion with two carboxylate O atoms, O(3) and O(5), two amide O atoms, O(1) and O(2), and two amine N atoms, N(1) and N(2). All these donor atoms are located in half of the coordination sphere, the ligand being unable to occupy all the positions in the coordination sphere of the Pb<sup>II</sup> ion. The remaining coordination positions are occupied by a water O atom O( $w$ 1) and a carboxylate O atom O(4') from the adjacent metal chelate. If the very long Pb—O(4') distance of 3.223 Å is considered to be a Pb—O bond, the resulting molecule is a binuclear chelate (Fig. 2). The water molecule O( $w$ 1) that is coordinated to the Pb<sup>II</sup> ion forms a hydrogen bond with O(3') of the adjacent metal chelate, thereby stabilizing the binuclear chelate. Other hydrogen bonds with the O—O distance less than 2.9 Å are found for the O( $w$ 1)—O(6), O( $w$ 2)—O(4) and O( $w$ 3)—O(6) atomic pairs (Table 2).

The Pb<sup>II</sup> ion has eight coordination bonds when the two distant atoms O(2) and O(4') are included in the coordination sphere (Fig. 3). A coordination geometry usually found in eight-coordination compounds is either the square antiprism or the triangulated dodecahedron (Wells, 1984). The latter polyhedron is formed in the present Pb<sup>II</sup> chelate, but it is highly distorted. For example, the O(4')...O(5) distance [3.836 (7) Å] in the polyhedron is much longer than the O(1)...N(1) distance [2.803 (6) Å]. These distances should be equal in the ideal triangulated dodecahedron with  $D_{2d}$  symmetry. In addition, the O(2)...O( $w$ 1) and O(3)...N(2) distances are quite different from each other being 5.017 (6) and 3.358 (5) Å, respectively. This extreme distortion is caused by the nature of the ligand. The —C—CO—N—C— atoms in each amide group are in the same plane. The largest deviation from the

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, geometry, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55860 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1028]

Table 1. Fractional coordinates and equivalent isotropic temperature factors

$$B_{\text{eq}} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (abc\cos\gamma)\beta_{12} + (accos\beta)\beta_{13} + (bccos\alpha)\beta_{23}]$$

	x	y	z	$B_{\text{eq}}$ ( $\text{\AA}^2$ )
Pb	0.03928 (2)	0.10685 (1)	0.11590 (2)	1.920 (4)
O(1)	0.2813 (4)	0.1693 (2)	0.2529 (4)	2.71 (8)
O(2)	0.0048 (5)	0.2344 (2)	0.1343 (4)	2.89 (8)
O(3)	-0.0309 (4)	0.0108 (2)	0.1786 (4)	2.60 (8)
O(4)	0.0661 (5)	-0.0774 (2)	0.2381 (5)	3.66 (9)
O(5)	-0.2688 (5)	0.1107 (2)	0.0556 (5)	3.5 (1)
O(6)	-0.4315 (4)	0.0680 (2)	0.1586 (6)	5.5 (1)
O(w1)	0.2561 (4)	0.0498 (2)	0.0442 (4)	3.95 (9)
O(w2)	0.7075 (5)	0.1595 (2)	0.7453 (5)	5.0 (1)
O(w3)	0.4721 (6)	0.0544 (3)	0.8008 (5)	5.6 (1)
N(1)	0.2217 (4)	0.0640 (2)	0.3737 (4)	1.99 (8)
N(2)	-0.0599 (4)	0.1398 (2)	0.3341 (4)	1.92 (8)
N(3)	0.1162 (5)	0.2667 (2)	0.3731 (4)	2.50 (9)
N(4)	0.4740 (5)	0.1892 (2)	0.4673 (5)	2.8 (1)
C(1)	0.3725 (5)	0.1537 (2)	0.3744 (5)	2.02 (9)
C(2)	0.3785 (6)	0.0902 (2)	0.4212 (6)	2.4 (1)
C(3)	0.1412 (6)	0.0723 (2)	0.4881 (5)	2.3 (1)
C(4)	0.0642 (5)	0.1307 (2)	0.4783 (5)	2.00 (9)
C(5)	-0.1072 (5)	0.2020 (2)	0.3157 (6)	2.2 (1)
C(6)	0.0113 (6)	0.2372 (2)	0.2673 (5)	2.3 (1)
C(7)	0.2377 (6)	0.3036 (2)	0.3440 (6)	3.0 (1)
C(8)	0.3993 (6)	0.2959 (2)	0.4560 (6)	3.0 (1)
C(9)	0.5048 (6)	0.2477 (3)	0.4262 (6)	3.0 (1)
C(10)	0.2313 (6)	0.0015 (2)	0.3432 (6)	2.6 (1)
C(11)	0.0782 (6)	-0.0238 (2)	0.2476 (6)	2.3 (1)
C(12)	-0.1991 (6)	0.1048 (2)	0.3239 (7)	2.6 (1)
C(13)	-0.3108 (6)	0.0941 (3)	0.1656 (7)	3.3 (1)

Table 2. Selected distances ( $\text{\AA}$ ), angles ( $^\circ$ ), torsion angles ( $^\circ$ ) and hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

Pb—O(1)amide	2.585 (3)	Pb—O(2)amide	2.968 (4)	
Pb—O(3)carboxylato	2.426 (4)	Pb—O(5)carboxylato	2.649 (4)	
Pb—N(1)amine	2.644 (3)	Pb—N(2)amine	2.594 (4)	
Pb—O(w1)water	2.614 (4)	Pb—O(4)*	3.223 (4)	
C(1)—O(1)amide	1.228 (5)	C(1)—N(4)amide	1.325 (6)	
C(6)—O(2)amide	1.232 (6)	C(6)—N(3)amide	1.320 (6)	
C(11)—O(3)carboxylato	1.271 (6)	C(11)—O(4)carboxylato	1.241 (6)	
C(13)—O(5)carboxylato	1.265 (9)	C(13)—O(6)carboxylato	1.225 (8)	
O(1)—Pb—O(2)	60.4 (1)	O(1)—Pb—O(3)	129.1 (1)	
O(1)—Pb—O(4')	123.7 (1)	O(1)—Pb—O(5)	138.3 (1)	
O(1)—Pb—O(w1)	80.1 (1)	O(1)—Pb—N(1)	64.8 (1)	
O(1)—Pb—N(2)	83.8 (1)	O(2)—Pb—O(3)	148.2 (1)	
O(2)—Pb—O(4')	105.47 (9)	O(2)—Pb—O(5)	81.8 (1)	
O(2)—Pb—O(w1)	127.9 (1)	O(2)—Pb—N(1)	111.3 (1)	
O(2)—Pb—N(2)	66.1 (1)	O(3)—Pb—O(4')	92.5 (1)	
O(3)—Pb—O(5)	75.3 (1)	O(3)—Pb—O(w1)	82.9 (1)	
O(3)—Pb—N(1)	64.6 (1)	O(3)—Pb—N(2)	83.9 (1)	
O(4')—Pb—O(5)	81.0 (1)	O(4')—Pb—O(w1)	68.1 (1)	
O(4')—Pb—N(1)	139.2 (1)	O(4')—Pb—N(2)	144.4 (1)	
O(5)—Pb—O(w1)	141.1 (1)	O(5)—Pb—N(1)	120.7 (1)	
O(5)—Pb—N(2)	63.8 (1)	O(w1)—Pb—N(1)	75.6 (1)	
O(w1)—Pb—N(2)	145.4 (1)	N(1)—Pb—N(2)	69.9 (1)	
C(6)—N(3)—C(7)	123.5 (5)	C(1)—N(4)—C(9)	123.3 (4)	
O(2)—C(6)—N(3)	125.6 (5)	O(1)—C(1)—N(4)	123.1 (5)	
O(2)—C(6)—C(5)	117.6 (4)	O(1)—C(1)—C(2)	120.1 (4)	
N(3)—C(6)—C(5)	116.8 (5)	N(4)—C(1)—C(2)	116.8 (4)	
C(7)—N(3)—C(6)—O(2)	3.4 (8)	C(9)—N(4)—C(1)—O(1)	-9.6 (8)	
C(7)—N(3)—C(6)—C(5)	-179.1 (4)	C(9)—N(4)—C(1)—C(2)	167.3 (5)	
D—H...A	D—H	H...A	D...A	D—H...A
O(w1)—H(w1a)...O(3)	0.954 (4)	1.876 (3)	2.780 (5)	157.0 (3)
O(w1)—H(w1b)...O(6)	0.919 (4)	1.859 (4)	2.703 (5)	151.7 (3)
O(w2)—H(w2b)...O(4)	0.903 (5)	1.871 (4)	2.748 (6)	163.1 (3)
O(w3)—H(w3b)...O(6)	0.882 (6)	2.044 (6)	2.886 (8)	159.1 (4)

\* Carboxylato O atom from an adjacent metal chelate.

averaged plane is 0.094 (5)  $\text{\AA}$  for C(9), N(4), C(1), O(1), C(2), and 0.014 (4)  $\text{\AA}$  for C(7), N(3), C(6), O(2), C(5). The near planarity of these groups makes the macrocyclic ring inflexible. Moreover, the macrocycle cannot extend itself to occupy all the coordination positions around the Pb<sup>II</sup> ion. These geometrical constraints of the ligand and the coordination of the amide O atoms result in the novel coordination geometry around the Pb<sup>II</sup> ion.

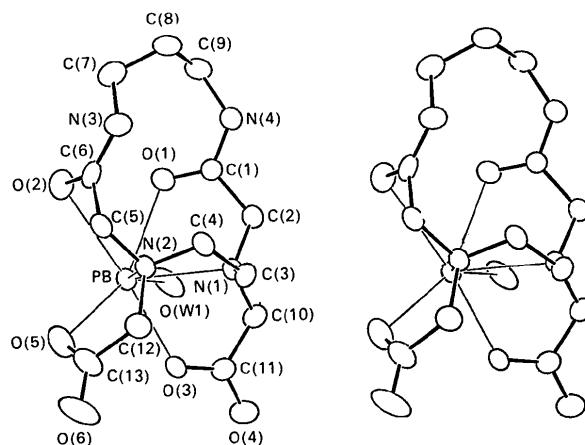
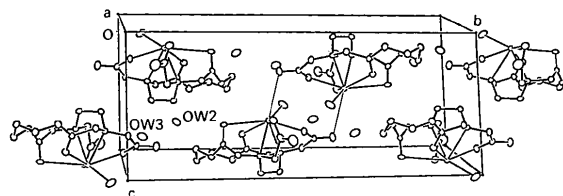
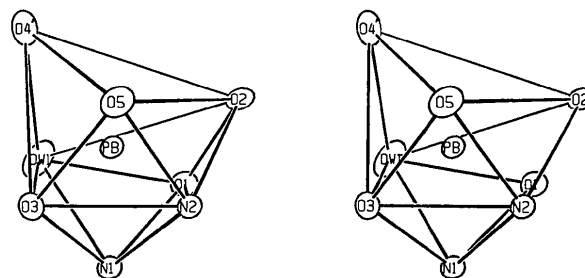
Fig. 1. Stereoscopic view of the Pb<sup>II</sup> chelate. Atoms are given at 50% probability level.

Fig. 2. Packing diagram in the unit cell. Atoms are given at 20% probability level.

Fig. 3. Stereoscopic view of coordination geometry around a Pb<sup>II</sup> ion.

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## Structure of Hydroxo(methyl)bis( $\eta^5$ -pentamethylcyclopentadienyl)tantalum(V) Hydroxotris(pentafluorophenyl)borate

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**Abstract.** [Ta(C<sub>10</sub>H<sub>15</sub>)<sub>2</sub>(OH)(CH<sub>3</sub>)] [B(OH)(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>],  $M_r = 1001.63$ , monoclinic,  $P2_1/n$ ,  $a = 12.217$  (2),  $b = 16.848$  (6),  $c = 18.834$  (3) Å,  $\beta = 100.37$  (2)°,  $V = 3813.1$  (15) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.75$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 29.52$  cm<sup>-1</sup>,  $F(000) = 1972$ , room temperature,  $R = 0.031$  for 3534 reflections with  $F_o^2 > 3\sigma(F_o^2)$ . The Ta cation has the expected geometry, with Ta—C and Ta—O distances 2.211 (6) and 1.865 (5) Å, respectively. The anion has not been characterized previously; its geometry is irregular with tetrahedral angles at boron ranging from 103.6 (6) to 113.8 (6)°, and systematic angular distortions in the C<sub>6</sub>F<sub>5</sub> rings.

**Introduction.** Recent interest in the reactive oxo chemistry of organometallic complexes has increased due to the possible roles of these compounds in the activation of hydrocarbons by the 2 + 2 addition of an aliphatic or aryl C—H bond to the metal oxo moiety. Bent metallocene complexes which would model this reaction chemistry, [( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)<sub>2</sub>Zr(OH)(C<sub>6</sub>F<sub>5</sub>)] (Chaudhari & Stone, 1966), [( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Hf(OH)Ph] (van Asselt, Santarsiero & Bercaw, 1986; Vaughn, Rupert & Hillhouse, 1987), [( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Zr(OH)Ph] (Schrock & Marks, 1988) and [( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Zr(OH){CPh=CPhNH(N=CPh<sub>2</sub>)}]

(Vaughn, Hillhouse & Rheingold, 1990), have been synthesized by routes which do not involve 2 + 2 addition chemistry.

We have been investigating the reaction chemistry of the cation [( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Ta(CH<sub>3</sub>)<sub>2</sub>]<sup>+</sup> with a variety of counterions. Initial attempts to prepare this cation with perfluorotetraphenylborate as the counterion by the metathesis of [( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Ta(CH<sub>3</sub>)<sub>2</sub>]SO<sub>3</sub>CF<sub>3</sub> with [NaB(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>] resulted in the isolation of a crystal found to be [( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Ta(OH)(CH<sub>3</sub>)] [B(OH)(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>]. The crystal structure of this complex is described herein.

**Experimental.** Material synthesized by RWQ; pale orange crystal, irregular chunk with maximum dimensions 0.26 × 0.35 × 0.48 mm; CAD-4 diffractometer,  $\omega$  scans; 25 reflections with  $34 < 2\theta < 37^\circ$  used for cell dimensions; absorption correction based on  $\psi$  scans of three reflections and their Friedel mates, with relative transmission from 0.857 to 1.081;  $(\sin\theta/\lambda)_{\text{max}}$  of 0.54 Å<sup>-1</sup>,  $h$  from -13 to 0,  $k$  from -18 to 18,  $l$  from -20 to 20; three standard reflections ( $\bar{3}03$ , 015, 025) showed no variation greater than predicted by counting statistics; 10 622 reflections measured, 4975 independent; goodness-of-fit for merging was 1.10 ( $R_{\text{merge}}$  for 3831 reflections with exactly two observations 0.032); all reflections used in solution and refinement of the structure.

\* Contribution No. 8514.