

Table 2. Selected bond lengths (Å) and angles (°) with *e.s.d.'s in parentheses*

Re(1)–Re(2)	2.731 (1)	Re(2)–P(1)	2.371 (6)
Re(1)–Re(3)	2.726 (1)	Re(2)–P(3)	2.370 (6)
Re(2)–Re(3)	2.734 (1)	Re(3)–P(1)	2.359 (6)
Re(1)–P(2)	2.375 (6)	Re(3)–P(2)	2.369 (6)
Re(1)–P(3)	2.372 (6)		
Mean values			
Re–H*	2.15	O–C	1.205 (34)
Re–C	1.836 (33)		
Re(3)–Re(1)–Re(2)	60.1 (1)	P(1)–Re(3)–Re(2)	54.9 (2)
Re(2)–Re(3)–Re(1)	60.0 (1)	P(2)–Re(3)–Re(2)	115.0 (2)
Re(3)–Re(2)–Re(1)	59.9 (1)	C(2)–Re(1)–C(1)	92.0 (13)
P(2)–Re(1)–Re(3)	54.8 (2)	C(4)–Re(2)–C(3)	91.7 (13)
P(3)–Re(1)–Re(3)	114.9 (2)	C(6)–Re(3)–C(5)	87.6 (13)
P(3)–Re(1)–Re(2)	54.8 (2)	Re(3)–P(1)–Re(2)	70.6 (2)
P(2)–Re(1)–Re(2)	115.0 (2)	Re(3)–P(2)–Re(1)	70.2 (2)
P(1)–Re(2)–Re(3)	54.5 (2)	Re(2)–P(3)–Re(1)	70.3 (2)
P(3)–Re(2)–Re(3)	114.7 (2)	P(3)–Re(2)–P(1)	169.2 (2)
P(3)–Re(2)–Re(1)	54.9 (2)	P(3)–Re(1)–P(2)	169.7 (2)
P(1)–Re(2)–Re(1)	114.3 (2)	P(2)–Re(3)–P(1)	169.9 (2)
P(2)–Re(3)–Re(1)	55.0 (2)	Re–H–Re	79*
P(1)–Re(3)–Re(1)	114.9 (2)		

* See text.

0.184 (4) Å shorter. This decided bond shortening is accompanied by a small decrease of the acute Re–P–Re bond angle by 4.1 (1)° and of the average Re–P bond length by a value of about 0.06 (3) Å. The Re–Re bond shortening in the title substance has similarities to the behaviour of such bond lengths in the formal double bond in [Re₂H₂(CO)₈] [2.896 (3) Å] (Bennett, Graham, Hoyano & Hutcheon, 1972), the unsaturated triangular Re₃ core in [Re₃(CO)₈–{P(C₆H₅)₃}(μ-H)₄][–] [2.797 (1), 3.193 (1) and 3.234 (1) Å] and the unsupported bond in [Re₂(CO)₁₀] [3.104 (1) Å] (Churchill, Amoh & Wassermann, 1981) with values of 0.166 (3), 0.067 (1) and 0.374 (1) Å. All

these facts confirm the shortened Re–Re bond in the title compound and agree therefore with an assumed π-bond property for the metal–metal bonds in the Re₃ ring.

The positional parameters of the two hydrides [H(1) and H(2)] could be located from a Δ*F* map and fit well with the expected values for two H atoms positioned *trans* to the CO ligands. This is in accordance with the known extremely marked *trans* influence of a hydride. The average values of the Re–(μ₃-H) bond length and the Re–(μ-H)–Re bond angle are 2.15 Å and 79°. Refinement of the hydride positions yielded no improvement. The estimated Re–(μ₃-H) bond length is elongated relative to that of Re–(μ-H) ascertained by neutron diffraction analysis of [Re₂H₈{P(C₂H₅)₂–(C₆H₅)₄}]₄ [ave. 1.878 (7) Å] (Bau, Carroll, Teller & Koetzle, 1977).

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Structure of Lead(II) Disaccharinate Monohydrate*

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Abstract. [Pb(C₇H₄NO₃S)₂].H₂O, *M_r* = 589.56, monoclinic, *P*2₁/*c*, *a* = 16.037 (3), *b* = 13.356 (5), *c* =

8.132 (3) Å, β = 93.04 (2)°, *V* = 1739.34 Å³, *Z* = 4, *D_m* = 2.14, *D_x* = 2.251 Mg m^{–3}, λ(Mo *K*α) = 0.71069 Å, μ = 9.58 mm^{–1}, *F*(000) = 1112, room temperature, final *R* = 0.050 for 2718 observed unique

* Saccharin is 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide.

reflections. The structure is built up of Pb^{2+} cations, saccharinate anions and water molecules. The Pb^{2+} ion is eight coordinated by one water O atom, five O atoms from CO and SO_2 groups and two N atoms from saccharinate ions with distances ranging from 2.446 (9) to 2.929 (9) Å.

Introduction. *o*-Sulfobenzamide, commonly known as saccharin, forms several types of metal saccharinates. These compounds are interesting for the crystal chemistry of the metals as well as for the ligation properties of saccharin. Up to now, crystal-structure analyses revealed different types of metal-to-ligand bonding in the series of different metal saccharinates. From the arrangement of the saccharinate ligands around the central metal atom and comparison of the interatomic distances between the metal atom and electron-donor atoms of the ligand (N and O atoms from SO_2 and CO groups), some complexes are predominantly ionic, some covalent and some intermediate. In the course of our structural investigations of such compounds, we have reported the crystal structures of the saccharinate complexes of sodium and magnesium (Jovanovski & Kamenar, 1982), manganese(II) (Kamenar & Jovanovski, 1982) and mercury(II) (Kamenar, Jovanovski & Grdenić, 1982; Jovanovski, Kamenar, Ferguson & Kaitner, 1986). In the mean time, the structures have been published of the series of saccharinates of iron(II), cobalt(II), nickel(II), copper(II), chromium(II), zinc(II), cadmium(II) and vanadium(II) (Haider, Malik, Ahmed, Hess, Riffel & Hursthouse, 1983; Cotton, Lewis, Murillo, Schwotzer & Valle, 1984; Haider, Malik, Das & Hursthouse, 1984; Cotton, Falvello, Llusar, Libby, Murillo & Schwotzer, 1986). All are isostructural with Mn^{II} saccharinate. Also published were the structures of dipotassium sodium saccharinate (Malik, Haider, Hosain & Hursthouse, 1984) and of trimethylstannyl saccharinate (Chuprunov, Gromilov, Stolyarova, Tarkhova & Belov, 1982). We present here the structure of lead(II) saccharinate monohydrate and compare its structural characteristics with those of other metal saccharinates.

Experimental. The title compound was prepared by successive addition of an equimolar quantity of PbCO_3 to a warm aqueous solution of saccharin. The single crystals were grown from aqueous solution. D_m by flotation. Diffraction intensities collected on a Philips PW 1100 automatic four-circle diffractometer, graphite-monochromated $\text{Mo K}\alpha$ radiation. Crystal of dimensions $0.2 \times 0.2 \times 0.4$ mm used for measurement of unit-cell parameters (from 18 reflections with θ between 6 and 10°) and data collection. 3899 reflections ($h = -22$ to 22, $k = 0$ to 18, $l = 0$ to 11) measured using the ω - 2θ -scan technique, scan range 1.20° , scan speed $0.04^\circ \text{ s}^{-1}$, $\theta = 3$ to 30° . Three standard reflec-

tions measured every 2 h showed no significant change with time. 2718 reflections with $I \geq 5\sigma(I)$ considered observed. Data corrected for Lorentz and polarization effects but not for absorption. Atomic scattering factors and anomalous-dispersion correction for Pb atom from *International Tables for X-ray Crystallography* (1974). Patterson map indicated position of Pb atom; positions of other atoms (except H) obtained by Fourier-map

Table 1. Fractional atomic coordinates ($\times 10^4$; $\times 10^5$ for Pb) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{4}{3} \sum_i \beta_i a_i^2$$

	x	y	z	B_{eq} (Å ²)
Pb	40354 (2)	7606 (3)	14180 (5)	2.43 (1)
S(1)	5180 (2)	3393 (2)	654 (3)	2.5 (1)
N(1)	5051 (6)	2225 (7)	940 (11)	2.5 (2)
C(11)	6191 (7)	3332 (8)	-81 (13)	2.4 (3)
C(12)	6716 (9)	4054 (11)	-558 (19)	4.3 (4)
C(13)	7499 (10)	3711 (16)	-1090 (23)	6.0 (6)
C(14)	7733 (9)	2774 (18)	-1025 (21)	5.9 (6)
C(15)	7184 (8)	2027 (14)	-561 (20)	5.2 (5)
C(16)	6411 (7)	2317 (10)	-38 (15)	3.0 (3)
C(17)	5714 (7)	1671 (10)	558 (13)	2.8 (3)
O(11)	5194 (6)	3941 (7)	2179 (10)	3.7 (3)
O(12)	4585 (5)	3801 (8)	-541 (10)	3.9 (3)
O(13)	5732 (6)	773 (6)	687 (11)	2.6 (2)
S(2)	1412 (2)	341 (3)	1536 (8)	6.4 (2)
N(2)	2380 (7)	734 (9)	1856 (20)	4.9 (4)
C(21)	945 (8)	1542 (11)	1545 (20)	4.4 (4)
C(22)	117 (9)	1831 (12)	1362 (26)	5.9 (6)
C(23)	-53 (10)	2845 (16)	1338 (28)	7.2 (6)
C(24)	581 (10)	3565 (11)	1597 (27)	6.2 (6)
C(25)	1418 (9)	3265 (10)	1781 (25)	5.6 (5)
C(26)	1582 (7)	2218 (9)	1746 (17)	3.1 (3)
C(27)	2406 (7)	1716 (11)	1887 (19)	3.7 (3)
O(21)	1300 (8)	-96 (11)	-111 (22)	9.3 (6)
O(22)	1185 (8)	-213 (11)	2948 (29)	10.8 (7)
O(23)	3095 (5)	2163 (7)	2013 (14)	4.5 (3)
O(w)	3533 (6)	1109 (9)	-1416 (11)	4.4 (3)

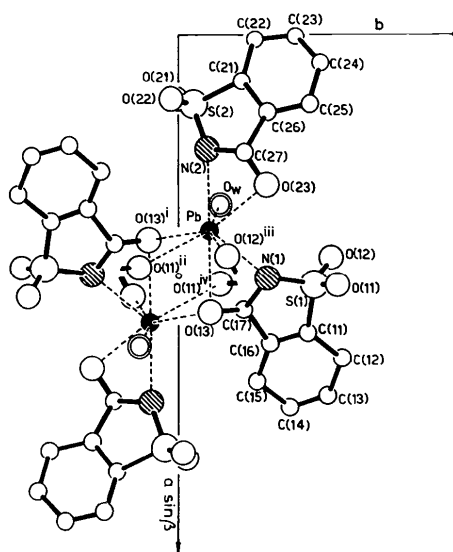


Fig. 1. Crystal structure viewed along c , showing the Pb atom surroundings and the atom-labelling scheme.

calculations; H-atom positions for water molecule and saccharinate ion (except two) from difference Fourier synthesis. In the final rounds of calculations the saccharin H atoms were positioned on geometrical grounds (C—H 0.95 Å) and included (as riding atoms) in the structure-factor calculations. The water molecule was refined as a rigid body (O—H = 1.05 Å, \angle H—O—H = 110°). The final least-squares refinement (on F) assuming anisotropic thermal parameters for non-H atoms and isotropic temperature factors for all H atoms gave the following discrepancy factors: $R = 0.050$, $wR = 0.051$, $w = 2.112/[(\sigma^2)|F_o| + 0.001077|F|^2]$; $(\Delta/\sigma)_{\max} = 0.25$. Max. and min. heights in final difference Fourier map 2.77 and $-3.84 \text{ e } \text{Å}^{-3}$, respectively. All calculations were performed on the Univac 1110 computer of the Zagreb University Computing Centre, SRCE, with programs written by Domenicano, Spagna & Vaciano (1969) and Sheldrick (1976).

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* The arrangement of the structural constituents around the symmetry centre (at 0.5,0,0), the surroundings of the Pb atom and the atom-labelling scheme are shown in Fig. 1. Interatomic distances and angles are given in Table 2.

The structure consists of Pb^{2+} cations, saccharinate anions and water molecules. Each Pb^{2+} ion is irregularly eight coordinated, being surrounded by one O atom from the water molecule [O(*w*) at 2.446 (9) Å], three O atoms from the CO group [O(13) 2.816 (10), O(23) 2.469 (12) and O(13ⁱ) 2.708 (9) Å], two O atoms from the SO₂ group [O(11ⁱⁱ) 2.929 (9) and O(12ⁱⁱⁱ) 2.646 (8) Å], and two N atoms [N(1) 2.588 (9) and N(2) 2.698 (11) Å] belonging to the saccharinate ions. Such values for Pb—O and Pb—N distances agree with those previously found in the structures with a Pb atom surrounded by O and/or N atoms (*e.g.* van der Kooi, den Brinker & de Kok, 1985; Ohba, Nosé & Saito, 1985; Calestani, Andreotti, Montenero & Bettinelli, 1985).

The CO O(13) atom from one of the two crystallographically independent saccharinates is coordinated to two Pb^{2+} ions forming thus a centrosymmetrical bridge $\text{Pb} \cdots \overset{\text{O}}{\text{O}} \cdots \text{Pb}$, while the CO O(23) atom from the other saccharinate is coordinated only to one Pb^{2+} ion but at the same time participates in hydrogen bonding with the water molecule [O(*w*) \cdots O(23^{iv}) = 2.71 (2) Å]. The second proton acceptor involved in

Table 2. Interatomic distances (Å) and angles (°) with *e.s.d.*'s in parentheses

Pb—O(<i>w</i>)	2.446 (9)	Pb—O(11 ⁱⁱ)	2.929 (9)
Pb—O(13)	2.816 (10)	Pb—O(12 ⁱⁱⁱ)	2.646 (8)
Pb—O(23)	2.469 (12)	Pb—N(1)	2.588 (9)
Pb—O(13 ⁱ)	2.708 (9)	Pb—N(2)	2.698 (11)
S(1)—N(1)	1.593 (9)	S(2)—N(2)	1.647 (11)
S(1)—O(11)	1.439 (9)	S(2)—O(21)	1.463 (19)
S(1)—O(12)	1.433 (9)	S(2)—O(22)	1.429 (22)
S(1)—C(11)	1.759 (11)	S(2)—C(21)	1.771 (15)
N(1)—C(17)	1.346 (15)	N(2)—C(27)	1.313 (18)
C(17)—O(13)	1.204 (15)	C(27)—O(23)	1.255 (15)
C(16)—C(17)	1.512 (17)	C(26)—C(27)	1.481 (16)
C(11)—C(16)	1.400 (16)	C(21)—C(26)	1.366 (18)
C(11)—C(12)	1.351 (18)	C(21)—C(22)	1.384 (19)
C(12)—C(13)	1.426 (23)	C(22)—C(23)	1.382 (26)
C(13)—C(14)	1.307 (31)	C(23)—C(24)	1.407 (24)
C(14)—C(15)	1.397 (26)	C(24)—C(25)	1.400 (21)
C(15)—C(16)	1.387 (18)	C(25)—C(26)	1.424 (18)
O(<i>w</i>)—Pb—N(2)	81.3 (4)	O(23)—Pb—O(13)	130.1 (3)
O(<i>w</i>)—Pb—O(23)	82.3 (8)	O(23)—Pb—O(11 ⁱⁱ)	143.4 (8)
O(<i>w</i>)—Pb—N(1)	83.9 (3)	O(23)—Pb—O(13 ⁱ)	143.5 (7)
O(<i>w</i>)—Pb—O(13)	94.2 (3)	O(23)—Pb—O(12 ⁱⁱⁱ)	80.0 (8)
O(<i>w</i>)—Pb—O(11 ⁱⁱ)	129.5 (3)	N(1)—Pb—O(13)	48.8 (3)
O(<i>w</i>)—Pb—O(13 ⁱ)	66.3 (3)	N(1)—Pb—O(11 ⁱⁱ)	115.4 (3)
O(<i>w</i>)—Pb—O(12 ⁱⁱⁱ)	156.3 (4)	N(1)—Pb—O(13 ⁱ)	111.6 (3)
N(2)—Pb—O(23)	50.8 (3)	N(1)—Pb—O(12 ⁱⁱⁱ)	78.0 (3)
N(2)—Pb—N(1)	131.4 (3)	O(13)—Pb—O(11 ⁱⁱ)	72.2 (2)
N(2)—Pb—O(13)	175.4 (4)	O(13)—Pb—O(13 ⁱ)	72.8 (3)
N(2)—Pb—O(11 ⁱⁱ)	109.6 (3)	O(13)—Pb—O(12 ⁱⁱⁱ)	85.3 (3)
N(2)—Pb—O(13 ⁱ)	104.1 (4)	O(11 ⁱⁱ)—Pb—O(13 ⁱ)	63.2 (3)
N(2)—Pb—O(12 ⁱⁱⁱ)	99.3 (4)	O(11 ⁱⁱ)—Pb—O(12 ⁱⁱⁱ)	72.9 (3)
O(23)—Pb—N(1)	81.5 (3)	O(13 ⁱ)—Pb—O(12 ⁱⁱⁱ)	135.0 (3)
S(1)—N(1)—C(17)	113.3 (8)	S(2)—N(2)—C(27)	110.5 (9)
C(11)—S(1)—N(1)	97.6 (5)	C(21)—S(2)—N(2)	96.1 (6)
O(11)—S(1)—O(12)	111.8 (6)	O(21)—S(2)—O(22)	120.4 (10)
O(11)—S(1)—C(11)	110.1 (5)	O(21)—S(2)—C(21)	109.5 (8)
O(11)—S(1)—N(1)	111.6 (5)	O(21)—S(2)—N(2)	109.9 (8)
O(12)—S(1)—N(1)	112.6 (5)	O(22)—S(2)—N(2)	108.4 (9)
O(12)—S(1)—C(11)	112.3 (5)	O(22)—S(2)—C(21)	109.9 (9)
N(1)—C(17)—C(16)	111.4 (10)	N(2)—C(27)—C(26)	115.0 (10)
N(1)—C(17)—O(13)	122.9 (11)	N(2)—C(27)—O(23)	120.3 (11)
O(13)—C(17)—C(16)	125.6 (11)	O(23)—C(27)—C(26)	124.7 (12)
C(11)—C(16)—C(17)	111.8 (10)	C(21)—C(26)—C(27)	111.7 (11)
C(11)—C(16)—C(15)	119.4 (12)	C(21)—C(26)—O(23)	120.9 (11)
C(15)—C(16)—C(17)	128.7 (13)	C(25)—C(26)—C(27)	127.4 (11)
S(1)—C(11)—C(16)	105.8 (8)	S(2)—C(21)—C(26)	106.6 (9)
S(1)—C(11)—C(12)	131.6 (10)	S(2)—C(21)—C(22)	131.0 (12)
C(12)—C(11)—C(16)	122.6 (11)	C(22)—C(21)—C(26)	122.4 (13)
C(11)—C(12)—C(13)	115.5 (14)	C(21)—C(22)—C(23)	117.6 (14)
C(12)—C(13)—C(14)	123.5 (17)	C(22)—C(23)—C(24)	121.8 (15)
C(13)—C(14)—C(15)	120.7 (15)	C(23)—C(24)—C(25)	120.1 (14)
C(14)—C(15)—C(16)	118.0 (16)	C(24)—C(25)—C(26)	117.1 (12)

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

hydrogen bonding is one of the O atoms from the SO₂ group [O(*w*) \cdots O(11ⁱⁱ) = 2.96 (1) Å]. The SO₂ O atoms from saccharinate (1) are coordinated to Pb atoms, those from saccharinate (2) are not. Also they do not participate in hydrogen bonding.

In comparison to other known crystal structures of M^{II} saccharinates, the structure of Pb^{II} saccharinate monohydrate is the first example in which the N atom and carbonyl O atom from the same saccharinate ion are coordinated to the metal atom.

Both saccharinate ions are planar within the experimental errors. The angles between the planes through

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44371 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the six- and five-membered rings within the saccharinate ions (1) and (2) are 0.4 and 0.6° respectively. The atoms belonging to the saccharinate ion (2) have significantly larger thermal parameters than those belonging to the saccharinate ion (1). This is probably due to the different coordinating function of the two crystallographically independent saccharinate ions.

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Structure du Chlorure de *trans*-Dichlorobis(propanediamine-1,3)platine(IV) Tétrahydrate

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Abstract. $[\text{PtCl}_2(\text{C}_3\text{H}_{10}\text{N}_2)_2]\text{Cl}_2 \cdot 4\text{H}_2\text{O}$, $M_r = 557.2$, monoclinic, $P2_1/n$, $a = 9.284$ (1), $b = 13.448$ (2), $c = 7.466$ (2) Å, $\beta = 107.25$ (1)°, $V = 890.2$ (6) Å³, $Z = 2$, $D_x = 2.08$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 8.57$ mm⁻¹, $F(000) = 540$, $T = 294$ (1) K, $R = 0.026$ for 1593 independent reflections. The Pt atom exhibits a slightly distorted octahedral coordination showing no unusual bond lengths or angles. It is linked to two chlorine atoms and to two 1,3-propanediamine molecules. The six-membered Pt-diamine ring has a chair-like conformation and the $[\text{PtCl}_2(\text{C}_3\text{H}_{10}\text{N}_2)_2]^{2+}$ cation adopts approximate C_{2h} symmetry. Anions, water molecules and cations are linked together by O–H...Cl, N–H...Cl and N–H...O hydrogen bonds.

Introduction. Le chlorure de *trans*-dichlorobis(propanediamine-1,3)platine(IV) tétrahydrate a été préparé à la température ambiante par action de l'eau oxygénée sur une solution de chlorure de bis(propanediamine-1,3)platine(II) additionnée d'acide chlorhydrique. Des cristaux ont été obtenus par évaporation de la

solution aqueuse. Compte tenu de la méthode de préparation, la détermination de la structure a été entreprise dans le but de préciser l'environnement de l'atome de platine. Elle a eu aussi pour perspective de vérifier les résultats de l'analyse élémentaire et, en particulier, de la teneur en eau. Cette étude fait suite à celles de l'hexachloroplatinate(IV) de propanediammonium-1,3 (Toffoli, Venumière, Khodadad, Rodier & Julien, 1985), du chlorure hexachloroplatinate(IV) de bis(pentanediammonium-1,5) (Delafontaine, Toffoli, Khodadad, Rodier & Julien, 1987) et du *trans*-diammine-*trans*-dichlorodihydroxoplatine(IV) di(peroxyde d'hydrogène) (Khodadad & Rodier, 1987).

Partie expérimentale. Cristal en forme de plaquette hexagonale: 0,180 × 0,125 × 0,085 mm. Dimensions de la maille déterminées sur monocristal, avec 25 réflexions telles que $7,64 \leq \theta \leq 18,45^\circ$. Diffractomètre Enraf-Nonius CAD-4. $0,039 \leq (\sin\theta)/\lambda \leq 0,661$ Å⁻¹; $-12 \leq h \leq 12$, $0 \leq k \leq 17$ et $0 \leq l \leq 9$. Balayage $\theta/2\theta$ d'amplitude $s(^\circ) = 0,65 + 0,35 \text{ tg}\theta$.