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Structure of the 1:1 Complex of Mercury(II) Saccharinate with 2,2′-Bipyridyl*

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(*Received* 26 *May* 1988; *accepted* 6 *October* 1988)

Abstract. $[Hg(C_7H_4NO_3S)_2(C_{10}H_8N_2)]$, $M_r = 721\cdot13$, monoclinic, $P2_1/c$, $a = 12\cdot238\ (1)$, $b = 14\cdot298\ (2)$, $c = 14\cdot237\ (3)$ Å, $\beta = 106\cdot65\ (1)°$, $V = 2386\cdot73$ Å^3, $Z = 4$, D_m (by flotation) $= 1\cdot988$, $D_x = 2\cdot007$ Mg m^{-3}, $\lambda(Cu\ K\alpha) = 1\cdot54178$ Å, $\mu = 13\cdot51$ mm^{-1}, $F(000) = 1392$, room temperature, final $R = 0\cdot031$ for 3703 independent observed reflections. The structure is built up of discrete molecules. Hg has a distorted tetrahedral coordination being bonded to two saccharinate N atoms [2·141 (4) and 2·120 (4) Å] and two N atoms belonging to the bipyridyl ligand [2·393 (3) and 2·328 (4) Å]. The N—Hg—N angles in the tetrahedron range from 70·7 (1) to 135·4 (2)°.

Introduction. This investigation is part of our broader research on the complexing properties of *o*-sulfobenzoimide, commonly known as saccharin. So far the crystal structures of sodium and magnesium saccharinates (Jovanovski & Kamenar, 1982), manganese(II) saccharinate (Kamenar & Jovanovski, 1982), mercury(II) saccharinates (Kamenar, Jovanovski & Grdenić, 1982; Jovanovski, Kamenar, Ferguson & Kaitner, 1988) and lead(II) saccharinate (Jovanovski, Hergold-Brundić & Kamenar, 1988) have been studied. In order to extend this investigation to the Hg chelate complexes we have prepared the 1:1 complex of mercury(II) saccharinate with 2,2′-bipyridyl. This is even more interesting because, contrary to the transition-metal complexes, there are not many examples of Hg complexes with chelating N donor ligands. Up to now complexes with such ligands as ethylenediamine (Duplančić, Grdenić, Kamenar, Matković & Sikirica, 1976; Cannas, Cristini & Marongiu, 1976; Grdenić, Sikirica & Vicković, 1977; Cannas, Cristini & Marongiu, 1978), 1,10-phenanthroline (Beauchamp, Saperas & Rivest, 1974; Grdenić, Kamenar & Hergold-Brundić, 1978*b*), 1,8-naphthyridine (Epstein, Dewan, Kepert & White, 1974) and bis(pyrazolyl)alkanes (Cingolani, Lorenzotti, Lobbia, Leonesi, Bonati & Bovio, 1987) have been prepared and structurally characterized. A few different complexes of Hg with 2,2′-bipyridyl as the bidentate ligand are also known (Craig, Farhangi, Graddon & Stephenson, 1974; Canty & Gatehouse, 1976; Grdenić, Kamenar & Hergold-Brundić, 1978*a*; Grdenić, Kamenar & Hergold-Brundić, 1979; Halfpenny, 1982).

Experimental. The complex was prepared by successive addition of an equimolar quantity of 2,2′-bipyridyl to a warm mixture of aqueous solutions of saccharin and mercury(II) acetate. Transparent needle-shaped crystals were obtained by recrystallization from water. Diffraction data collected on a Philips PW 1100 diffractometer, graphite-monochromated Cu *Kα* radiation. Crystal of dimensions 0·33 × 0·09 × 0·07 mm used for measurement of unit-cell parameters

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

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

$$B_{\rm eq} = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Hg	23128 (2)	30006 (1)	−3384 (1)	3·14 (1)
S(1)	3370 (1)	839 (1)	−163 (1)	2·92 (3)
N(11)	2354 (3)	1530 (3)	−49 (3)	3·13 (10)
C(11)	2862 (4)	−150 (3)	309 (3)	2·83 (11)
C(12)	3284 (4)	−1056 (3)	422 (4)	3·72 (13)
C(13)	2723 (5)	−1690 (4)	844 (5)	4·30 (17)
C(14)	1766 (5)	−1432 (4)	1138 (5)	4·46 (18)
C(15)	1346 (4)	−532 (3)	1006 (4)	3·76 (14)
C(16)	1907 (4)	111 (3)	588 (3)	2·86 (11)
C(17)	1600 (4)	1117 (3)	364 (3)	2·84 (11)
O(11)	4461 (3)	1134 (2)	475 (3)	3·98 (9)
O(12)	3312 (3)	720 (3)	−1174 (3)	4·38 (11)
O(13)	783 (3)	1501 (2)	524 (3)	3·88 (10)
S(2)	3947 (1)	3719 (1)	−1715 (1)	2·95 (3)
N(21)	3348 (3)	3951 (3)	−845 (3)	3·16 (10)
C(21)	4369 (4)	4879 (3)	−1814 (3)	2·69 (10)
C(22)	4977 (4)	5241 (4)	−2403 (4)	3·49 (13)
C(23)	5225 (4)	6192 (4)	−2319 (4)	3·79 (12)
C(24)	4883 (5)	6748 (4)	−1651 (4)	4·22 (16)
C(25)	4293 (4)	6370 (3)	−1047 (4)	3·55 (13)
C(26)	4036 (4)	5423 (3)	−1120 (3)	2·85 (11)
C(27)	3442 (4)	4864 (3)	−553 (3)	2·74 (11)
O(21)	3101 (3)	3412 (3)	−2579 (3)	4·50 (11)
O(22)	4910 (3)	3114 (2)	−1353 (3)	4·22 (11)
O(23)	3039 (3)	5163 (3)	88 (3)	3·83 (10)
N(1)	494 (3)	3466 (3)	−1390 (3)	3·11 (11)
C(1)	233 (4)	3495 (4)	−2373 (4)	2·89 (10)
C(2)	−878 (5)	3715 (4)	−2957 (4)	3·60 (14)
C(3)	−1676 (5)	3950 (4)	−2487 (5)	4·31 (15)
C(4)	−1397 (4)	3953 (3)	−1485 (4)	4·55 (16)
C(5)	−294 (4)	3709 (3)	−937 (3)	3·56 (13)
N(2)	1194 (3)	3572 (3)	609 (3)	2·84 (11)
C(6)	75 (4)	3710 (3)	147 (4)	2·88 (12)
C(7)	−694 (5)	3864 (4)	688 (4)	4·22 (15)
C(8)	−313 (5)	3880 (4)	1684 (5)	4·82 (18)
C(9)	832 (5)	3750 (4)	2166 (4)	4·13 (16)
C(10)	1545 (4)	3586 (4)	1586 (4)	3·74 (14)

(from 18 reflections with θ between 20 and 27°) and diffraction-data collection. Intensities of reflections with indices $h = -14$ to 14, $k = 0$ to 17, $l = 0$ to 17, ω-scan technique, scan speed 0·04° s^{-1}, scan range 1·20°, background 5 s before and after each scan, $\theta = 3$ to 70°, 3923 unique reflections of which 3703 with $I \geq 2\sigma(I)$ used in subsequent calculations. Three check reflections measured every 2 h showed no significant change with time. Data corrected for Lorentz, polarization and absorption effects (Harkema, 1978), transmission factors 0·472–0·162. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Structure solved by means of Fourier synthesis based upon the Hg-atom coordinates obtained from the Patterson synthesis. H atoms visible in a difference Fourier map. Refinement by block-diagonal least-squares method with anisotropic thermal parameters for all non-H atoms. In the final rounds of calculations H atoms were positioned on geometrical grounds (C—H 0·95 Å) and as riding atoms included in the structure-factor calculations with an overall isotropic temperature factor $B_{\rm iso} = 5{\cdot}4$ Å^2. Three secondary-extinction-affected reflections (110, 002, 202) were rejected in the final cycles of refinement. The final least-squares refinement (on F) gave the following discrepancy factors: $R = 0{\cdot}031$, $wR = 0{\cdot}036$, $w^{-1} = \sigma^2|F_o| + 0{\cdot}002609|F_o|^2$. $(\Delta/\sigma)_{\rm max} = 0{\cdot}27$. Max. and min. height in final difference Fourier map 0·51 and −1·51 e Å^{-3} (close to Hg), respectively. All calculations were performed using a Univac 1110 of the SRCE, University Computing Centre, Zagreb, with programs written by Domenicano, Spagna & Vaciago (1969) and Sheldrick (1976).

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* A view of the structure and the atom-labelling scheme are shown in Fig. 1. Interatomic distances and angles are given in Table 2. The crystal structure consists of individual (bpy)Hg(sac)$_2$ molecules (sac denoting the saccharinate ion). The Hg atom has tetrahedral characteristic coordination being bonded to two saccharinate N atoms and two N atoms belonging to the bipyridyl ligand. The saccharinate N to Hg bonds, Hg—N(11) and Hg—N(21), of 2·141 (4) and 2·120 (4) Å are longer than the corresponding bonds in the structure of mercury(II) saccharinate itself (2·03–2·06 Å) (Kamenar, Jovanovski & Grdenić, 1982) and chloromercury(II) saccharinate (2·021 Å) (Jovanovski, Kamenar, Ferguson & Kaitner, 1988) as well as in other structures with Hg—N covalent bonds (*e.g.* Breitinger & Brodersen, 1970; Oppolzer & Weber, 1972; Brauer, 1979), but in which digonal characteristic coordination of Hg was found. In the present structure the longer Hg—N bonds were expected due to the increase of the coordination number of Hg (Grdenić, 1965). The two bipyridyl N atoms are significantly farther from Hg: the bonds Hg—N(1) and

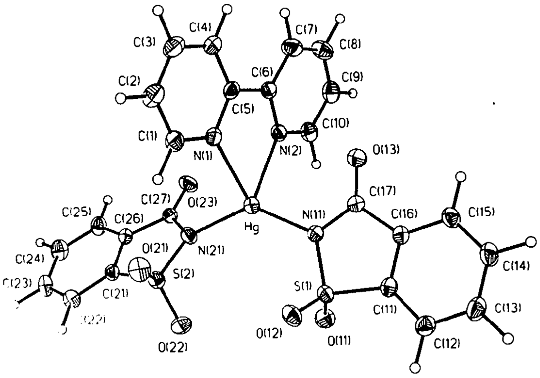

Fig. 1. Molecular structure with the atomic numbering scheme. Thermal ellipsoids are drawn at the 34% probability level. H atoms are represented as spheres of arbitrary size.

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51484 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Hg—N(1)	2·393 (3)	Hg—N(11)	2·141 (4)
Hg—N(2)	2·328 (4)	Hg—N(21)	2·120 (4)
S(1)—N(11)	1·632 (4)	S(2)—N(21)	1·644 (5)
S(1)—O(11)	1·445 (3)	S(2)—O(21)	1·432 (4)
S(1)—O(12)	1·432 (4)	S(2)—O(22)	1·434 (4)
S(1)—C(11)	1·754 (5)	S(2)—C(21)	1·756 (4)
N(11)—C(17)	1·363 (7)	N(21)—C(27)	1·364 (6)
C(17)—O(13)	1·217 (6)	C(27)—O(23)	1·233 (6)
C(16)—C(17)	1·498 (6)	C(26)—C(27)	1·468 (7)
C(11)—C(16)	1·390 (7)	C(21)—C(26)	1·407 (7)
C(11)—C(12)	1·387 (7)	C(21)—C(22)	1·371 (8)
C(12)—C(13)	1·374 (9)	C(22)—C(23)	1·392 (7)
C(13)—C(14)	1·401 (9)	C(23)—C(24)	1·393 (9)
C(14)—C(15)	1·378 (7)	C(24)—C(25)	1·382 (9)
C(15)—C(16)	1·380 (8)	C(25)—C(26)	1·387 (6)
C(1)—N(1)	1·344 (6)	C(6)—N(2)	1·353 (6)
N(1)—C(5)	1·350 (7)	N(2)—C(10)	1·334 (6)
C(1)—C(2)	1·412 (7)	C(6)—C(7)	1·393 (9)
C(2)—C(3)	1·374 (10)	C(7)—C(8)	1·360 (8)
C(3)—C(4)	1·368 (8)	C(8)—C(9)	1·384 (8)
C(4)—C(5)	1·396 (6)	C(9)—C(10)	1·382 (9)
C(5)—C(6)	1·479 (7)		
N(1)—Hg—N(2)	70·7 (1)	N(2)—Hg—N(11)	102·7 (2)
N(1)—Hg—N(11)	111·1 (1)	N(2)—Hg—N(21)	118·7 (1)
N(1)—Hg—N(21)	98·5 (1)	N(11)—Hg—N(21)	135·4 (2)
S(1)—N(11)—C(17)	114·2 (3)	S(2)—N(21)—C(27)	113·9 (3)
C(11)—S(1)—N(11)	95·0 (2)	C(21)—S(2)—N(21)	94·3 (2)
O(11)—S(1)—O(12)	115·9 (3)	O(21)—S(2)—O(22)	116·2 (2)
O(11)—S(1)—C(11)	111·1 (2)	O(21)—S(2)—C(21)	112·2 (2)
O(11)—S(1)—N(11)	110·7 (2)	O(21)—S(2)—N(21)	110·0 (2)
O(12)—S(1)—N(11)	110·6 (2)	O(22)—S(2)—N(21)	110·4 (2)
O(12)—S(1)—C(11)	111·6 (2)	O(22)—S(2)—C(21)	111·6 (2)
N(11)—C(17)—C(16)	110·4 (4)	N(21)—C(27)—C(26)	111·5 (4)
N(11)—C(17)—O(13)	125·4 (4)	N(21)—C(27)—O(23)	122·7 (5)
O(13)—C(17)—C(16)	124·2 (5)	O(23)—C(27)—C(26)	125·8 (4)
C(11)—C(16)—C(17)	112·2 (4)	C(21)—C(26)—C(27)	112·0 (4)
C(11)—C(16)—C(15)	120·5 (4)	C(21)—C(26)—C(25)	119·1 (5)
C(15)—C(16)—C(17)	127·3 (5)	C(25)—C(26)—C(27)	128·9 (5)
S(1)—C(11)—C(16)	108·2 (3)	S(2)—C(21)—C(26)	108·3 (4)
S(1)—C(11)—C(12)	129·5 (4)	S(2)—C(21)—C(22)	129·0 (4)
C(12)—C(11)—C(16)	122·3 (5)	C(22)—C(21)—C(26)	122·6 (4)
C(11)—C(12)—C(13)	116·8 (5)	C(21)—C(22)—C(23)	117·3 (5)
C(12)—C(13)—C(14)	121·5 (5)	C(22)—C(23)—C(24)	121·1 (6)
C(13)—C(14)—C(15)	121·1 (6)	C(23)—C(24)—C(25)	120·9 (5)
C(14)—C(15)—C(16)	117·9 (5)	C(24)—C(25)—C(26)	118·9 (5)
C(1)—N(1)—C(5)	120·0 (4)	C(5)—C(6)—N(2)	118·1 (5)
N(1)—C(1)—C(2)	121·4 (5)	C(7)—C(6)—N(2)	120·2 (4)
C(1)—C(2)—C(3)	117·8 (5)	C(5)—C(6)—C(7)	121·6 (4)
C(2)—C(3)—C(4)	120·6 (5)	C(6)—C(7)—C(8)	119·7 (5)
C(3)—C(4)—C(5)	119·6 (5)	C(7)—C(8)—C(9)	120·7 (6)
C(4)—C(5)—C(6)	122·7 (5)	C(8)—C(9)—C(10)	116·7 (5)
C(4)—C(5)—N(1)	120·4 (4)	C(9)—C(10)—N(2)	123·7 (5)
N(1)—C(5)—C(6)	116·9 (4)	C(10)—N(2)—C(6)	118·9 (5)

Hg—N(2) are 2·393 (3) and 2·328 (4) Å, both longer than the sum of corresponding covalent radii [1·48 Å for tetracoordinated Hg (Grdenić, 1969) and 0·70 Å for N (Pauling, 1960)]. However, they are in agreement with the values (2·27–2·34 Å) found in the previously determined structures with tetrahedrally coordinated Hg (*e.g.* Duplančić *et al.*, 1976; Grdenić *et al.*, 1978*b*; Grdenić *et al.*, 1979). In a rather distorted tetrahedron the angle between the shorter Hg—N bonds is considerably larger [135·4 (2)°] than the angle between the longer bonds [70·7 (1)°]. This is due to the fixed 'bite' of the bidentate bipyridyl ligand.

Both saccharinate ligands are planar within experimental errors; the angles between the least-squares best planes through the five- and six-membered rings within the saccharinate groups are 1·5 and 1·1°. The corresponding interatomic distances and angles within the two independent saccharinate ligands are similar and in general close to those found in other metal saccharinates. The pyridine rings of the bipyridyl ligand are also planar but twisted to each other by an angle of 6·8°.

This work was supported by the Foundation for Scientific Research of SR Croatia, Zagreb, and SR Macedonia, Skopje.

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