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MERCURY(II) SACCHARINATE, $Hg(C_7H_4NO_3S)_2$

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Preliminary information. In continuation of our research on crystal structure of metal saccharinates (Jovanovski & Kamenar, 1981a) we determined also the crystal and molecular structure of mercury(II) saccharinate. The compound was prepared for the first time by warming aqueous solution of Na saccharinate and Hg(II) acetate. The crystals were obtained by recrystallization from ethanol.

Crystal data. From single crystal diffractometry, Mo$K\alpha$, λ = 0.7107 Å: a = 14.460(9), b = 13.054(10), c = 10.471(10) Å, α = 106.56(2), β = 94.96(3), γ = 111.09(2)°, V = 1727.7 Å^3, Z = 4, D_{obs} = 2.21, D_{calc} = 2.170 g cm^{-3}, F(000) = 1064, μ(Mo$K\alpha$) = 93.49 cm^{-1}, space group $P\bar{1}$.

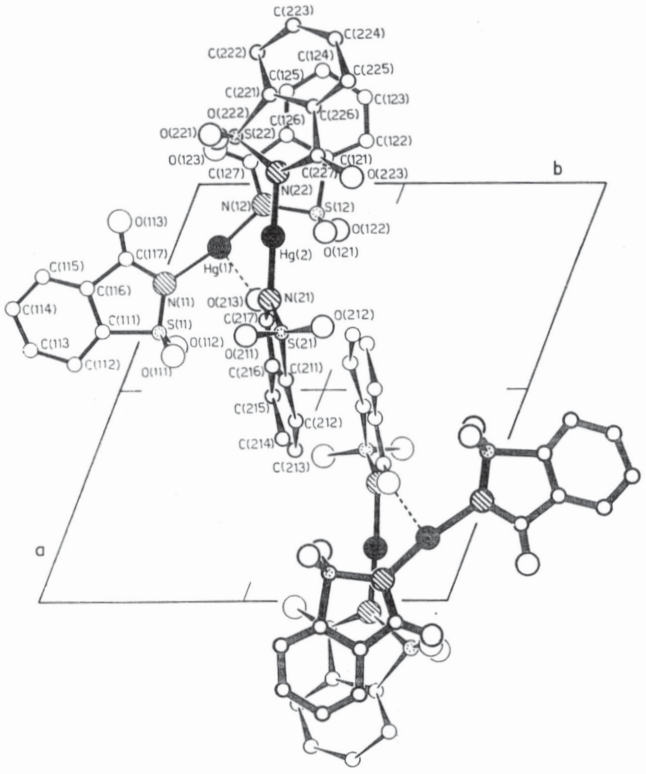
Intensity data, structure determination and refinement. Intensity data were collected on a Philips PW 1100 computer controlled automatic diffractometer using graphite monochromatized Mo$K\alpha$ radiation. 6481 independent reflections were measured from prismatic specimen (0.14 x 0.15 x 0.35 mm^3) in the range $4<2\theta<60°$ employing θ-2θ scan technique (scan width 1.60°, scan speed 0.08°s^{-1}). Absorption correction was applied. The structure was solved by means of three-dimensional Fourier synthesis based upon the mercury atom coordinates obtained

Table 1. Atomic coordinates ($x10^4$) with their e.s.d.'s

	x/a	*y/b*	*z/c*
Hg(1)	1536(0)	1057(1)	1829(1)
N(11)	2335(10)	37(13)	1677(14)
S(11)	3579(3)	416(5)	1810(5)
C(111)	3504(15)	-1012(19)	1260(20)
C(112)	4273(20)	-1384(27)	1028(25)
C(113)	3987(23)	-2603(32)	519(29)
C(114)	2967(24)	-3423(24)	317(27)
C(115)	2202(18)	-2995(19)	559(21)
C(116)	2507(15)	-1790(18)	1031(19)
C(117)	1817(14)	-1160(17)	1276(18)
O(113)	892(9)	-1624(11)	1098(14)
O(112)	3933(10)	994(15)	851(16)
O(111)	4106(11)	1013(14)	3207(15)
N(12)	499(10)	1791(12)	2097(14)
S(12)	686(3)	3167(4)	2865(5)
C(121)	-612(12)	2892(15)	2723(17)
C(122)	-1032(15)	3670(19)	3185(22)
C(123)	-2101(16)	3228(21)	2981(24)
C(124)	-2685(14)	2008(21)	2330(22)
C(125)	-2220(12)	1264(17)	1891(17)
C(126)	-1170(12)	1699(15)	2054(16)
C(127)	-515(12)	1077(14)	1644(15)
O(123)	-824(9)	32(10)	990(11)
O(122)	1129(10)	3840(13)	2042(16)
O(121)	1185(10)	3582(12)	4256(16)
Hg(2)	1277(0)	2303(1)	6119(1)
N(21)	2803(9)	2855(12)	6319(13)
S(21)	3626(3)	3457(5)	7787(5)
C(211)	4697(12)	3936(14)	7083(19)
C(212)	5683(13)	4614(19)	7751(24)
C(213)	6391(14)	4890(18)	6961(28)
C(214)	6119(14)	4457(17)	5492(28)
C(215)	5080(13)	3777(17)	4812(25)
C(216)	4383(12)	3518(14)	5674(21)
C(217)	3273(12)	2938(14)	5198(17)
O(213)	2804(9)	2571(11)	4044(12)
O(212)	3470(12)	4414(15)	8643(16)
O(211)	3648(11)	2574(16)	8300(15)
N(22)	-268(10)	1817(12)	5812(16)
S(22)	-1129(3)	496(4)	5071(5)
C(221)	-2138(13)	925(17)	5140(20)
C(222)	-3168(14)	214(19)	4521(24)
C(223)	-3798(16)	810(27)	4728(28)
C(224)	-3455(19)	1983(25)	5465(28)
C(225)	-2409(19)	2694(24)	6051(27)
C(226)	-1788(14)	2088(17)	5859(20)
C(227)	-636(14)	2663(17)	6266(21)
O(223)	-124(12)	3652(13)	6891(19)
O(222)	-1051(10)	-19(12)	3700(12)
O(221)	-1159(10)	-240(12)	5853(13)

from the Patterson synthesis. The structure was refined by block diagonal least-squares method with anisotropic thermal parameters for all atoms except hydrogen atoms. The final R factor for 6127 reflections with $I>3\sigma(I)$ was 0.062. The anomalous dispersions for mercury and sulphur atoms were included in the F_c calculations (International Tables for X-ray Crystallography, 1974).

Comments. Tables 1 and 2 give atomic coordinates and interatomic distances and angles. The structure is built up of two crystallographically independent molecules with different geometries (see Figure). The least-squares best planes through the atoms of saccharinate ligands intersect at an angle of 11° in molecule (1) and at 71° in molecule (2). Both mercury atoms have digonal characteristic coordination being bonded to two saccharinate nitrogen atoms. The Hg-N bond lengths are 2.04 and 2.05 Å in molecule (1) and 2.03 and 2.06 Å in molecule (2). All these values agree with those previously found in the structures with covalent Hg-N bond (*e.g.* Oppolzer and Weber, 1972). The N-Hg-N angles deviate from linearity. In molecule (1) this angle amounts to 167° and in molecule (2) 175°, respectively. The smaller angle could be explained by the Hg(1)...O(213) contact of 2.59 Å which is significantly shorter than the sum of van der Waals radii for mercury and oxygen atom (Grdenić, 1981). The Hg(1) atom has other two additional Hg...O contacts (2.80 and 2.93 Å) with the oxygen atoms from neighbouring molecules. Since the Hg(2) atom has also three Hg...O contacts (2.83, 2.93 and 2.94 Å) with the neighbouring molecules, both mercury atoms are effectively five-coordinated (Grdenić, 1965). Saccharinate ligands are planar within the experimental errors. Bond lengths and angles in these ions are close to the values found in sodium and magnesium saccharinate (Jovanovski & Kamenar, 1982b) as well as in manganese(II) saccharinate (Kamenar & Jovanovski, 1982).



Figure

Table 2. Interatomic distances and angles with their e.s.d.'s

Distances (Å)

Hg(1)-N(11)	2.04(2)	C(111)-C(112)	1.38(4)
Hg(1)-N(12)	2.05(2)	C(112)-C(113)	1.41(5)
S(11)-N(11)	1.67(1)	C(113)-C(114)	1.43(4)
S(11)-C(111)	1.75(3)	C(114)-C(115)	1.42(5)
S(11)-O(111)	1.44(2)	C(115)-C(116)	1.39(3)
S(11)-O(112)	1.44(2)	S(12)-N(12)	1.65(2)
N(11)-C(117)	1.38(2)	S(12)-C(121)	1.76(2)
C(117)-O(113)	1.22(2)	S(12)-O(121)	1.42(2)
C(117)-C(116)	1.50(4)	S(12)-O(122)	1.43(2)
C(111)-C(116)	1.38(3)	N(12)-C(127)	1.37(2)

Table 2. (cont.)

C(127)-O(123)	1.23(2)	C(211)-C(212)	1.37(2)
C(127)-C(126)	1.47(3)	C(212)-C(213)	1.35(3)
C(121)-C(126)	1.40(2)	C(213)-C(214)	1.44(4)
C(121)-C(122)	1.36(3)	C(214)-C(215)	1.43(2)
C(122)-C(123)	1.41(3)	C(215)-C(216)	1.43(3)
C(123)-C(124)	1.42(3)	S(22)-N(22)	1.63(1)
C(124)-C(125)	1.37(4)	S(22)-C(221)	1.74(2)
C(125)-C(126)	1.39(2)	S(22)-O(221)	1.42(2)
Hg(2)-N(21)	2.03(1)	S(22)-O(222)	1.43(1)
Hg(2)-N(22)	2.06(1)	N(22)-C(227)	1.38(3)
S(21)-N(21)	1.65(1)	C(227)-O(223)	1.13(2)
S(21)-C(211)	1.76(2)	C(227)-C(226)	1.52(3)
S(21)-O(211)	1.41(2)	C(221)-C(226)	1.36(3)
S(21)-O(212)	1.42(2)	C(221)-C(222)	1.41(2)
N(21)-C(217)	1.42(2)	C(222)-C(223)	1.39(4)
C(217)-O(213)	1.20(2)	C(223)-C(224)	1.36(4)
C(217)-C(216)	1.47(2)	C(224)-C(225)	1.43(3)
C(211)-C(216)	1.39(3)	C(225)-C(226)	1.39(4)
$Hg(1)\ldots O(213)_{i}$	2.59(1)	$Hg(2)\ldots O(121)_{ii}$	2.93(2)
$Hg(1)\ldots O(123)_{ii}$	2.80(1)	$Hg(2)\ldots O(222)_{ii}$	2.94(2)
$Hg(1)\ldots O(221)_{ii}$	2.93(1)	$Hg(2)\ldots O(221)_{ii}$	2.83(2)

(i = -x, -y, -z; ii = -x, -y, 1 - z)

Angles (°)

N(11)-Hg(1)-N(12)	167(1)	C(121)-S(12)-N(12)	95(1)
S(11)-N(11)-Hg(1)	129(1)	O(121)-S(12)-O(122)	117(1)
C(117)-N(11)-Hg(1)	119(1)	O(121)-S(12)-C(121)	111(1)
S(11)-N(11)-C(117)	111(1)	O(121)-S(12)-N(12)	112(1)
C(111)-S(11)-N(11)	95(1)	O(122)-S(12)-N(12)	109(1)
O(111)-S(11)-O(112)	117(1)	O(122)-S(12)-C(121)	111(1)
O(111)-S(11)-C(111)	111(1)	N(12)-C(127)-O(123)	123(2)
O(111)-S(11)-N(11)	111(1)	N(12)-C(127)-C(126)	113(1)
O(112)-S(11)-N(11)	110(1)	C(126)-C(127)-O(123)	125(1)
O(112)-S(11)-C(111)	111(1)	C(121)-C(126)-C(127)	112(1)
N(11)-C(117)-O(113)	122(2)	C(121)-C(126)-C(125)	118(2)
N(11)-C(117)-C(116)	112(2)	C(125)-C(126)-C(127)	130(2)
C(116)-C(117)-O(113)	126(2)	S(12)-C(121)-C(126)	108(2)
C(111)-C(116)-C(117)	111(2)	S(12)-C(121)-C(122)	128(1)
C(111)-C(116)-C(115)	123(2)	C(122)-C(121)-C(126)	124(2)
C(115)-C(116)-C(117)	126(2)	C(121)-C(122)-C(123)	117(2)
S(11)-C(111)-C(116)	110(2)	C(122)-C(123)-C(124)	120(2)
S(11)-C(111)-C(112)	128(2)	C(123)-C(124)-C(125)	121(2)
C(112)-C(111)-C(116)	122(2)	C(124)-C(125)-C(126)	120(2)
C(111)-C(112)-C(113)	116(2)	N(21)-Hg(2)-N(22)	175(1)
C(112)-C(113)-C(114)	123(3)	S(21)-N(21)-Hg(2)	125(1)
C(113)-C(114)-C(115)	119(3)	C(217)-N(21)-Hg(2)	122(1)
C(114)-C(115)-C(116)	117(2)	S(21)-N(21)-C(217)	113(1)
S(12)-N(12)-Hg(1)	129(1)	C(211)-S(21)-N(21)	95(1)
C(127)-N(12)-Hg(1)	119(1)	O(211)-S(21)-O(212)	119(1)
S(12)-N(12)-C(127)	112(1)	O(211)-S(21)-C(211)	110(1)

Table 2. (cont.)

O(211)-S(21)-N(21)	109(1)	C(221)-S(22)-N(22)	94(1)
O(212)-S(21)-N(21)	110(1)	O(221)-S(22)-O(222)	112(1)
O(212)-S(21)-C(211)	111(1)	O(221)-S(22)-C(221)	112(1)
N(21)-C(217)-O(213)	123(1)	O(221)-S(22)-N(22)	113(1)
N(21)-C(217)-C(216)	110(1)	O(222)-S(22)-N(22)	113(1)
C(216)-C(217)-O(213)	127(2)	O(222)-S(22)-C(221)	113(1)
C(211)-C(216)-C(217)	114(2)	N(22)-C(227)-O(223)	124(2)
C(211)-C(216)-C(215)	122(2)	N(22)-C(227)-C(226)	108(2)
C(215)-C(216)-C(217)	124(2)	C(226)-C(227)-O(223)	127(2)
S(21)-C(211)-C(216)	108(1)	C(221)-C(226)-C(227)	112(2)
S(21)-C(211)-C(212)	128(2)	C(221)-C(226)-C(225)	123(2)
C(212)-C(211)-C(216)	124(2)	C(225)-C(226)-C(227)	124(2)
C(211)-C(212)-C(213)	117(2)	S(22)-C(221)-C(226)	110(1)
C(212)-C(213)-C(214)	122(2)	S(22)-C(221)-C(222)	127(2)
C(213)-C(214)-C(215)	120(2)	C(222)-C(221)-C(226)	124(2)
C(214)-C(215)-C(216)	116(2)	C(221)-C(222)-C(223)	114(2)
S(22)-N(22)-Hg(2)	126(1)	C(222)-C(223)-C(224)	123(2)
C(227)-N(22)-Hg(2)	119(1)	C(223)-C(224)-C(225)	122(3)
S(22)-N(22)-C(227)	115(1)	C(224)-C(225)-C(226)	114(2)

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