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CRYSTAL STRUCTURE OF CHLOROMERCURY(II) SACCHARINATE, $C_7H_4ClHgNO_3S$

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Experimental. The compound was prepared for the first time by warming equimolar aqueous solutions of Na saccharinate and Hg(II) chloride. The crystals were recrystallized from water solution.

Crystal data: $C_7H_4ClHgNO_3S$, Mr=418.22, orthorhombic, a=20.530(5) b=4.767(2), c=9.467(2) Å, V=926.5 Å^3, Z=4, ρ_o=2.98, ρ_c=3.00 g.cm^{-3}, MoKα radiation, λ=0.7107 Å, F(000)=760, μ(MoKα)=171.1 cm^{-1}.

A colourless thin plate crystal having approximate dimensions of 0.23 x 0.10 x 0.05 mm was used for data collection. Three-dimensional intensity data were collected on an Enraf-Nonius CAD4 computer controlled diffractometer with a graphite monochromatized MoKα radiation. A total of 1283 reflections were collected, of which 1066 were unique. Lorentz and polarization as well as absorption and secondary extinction corrections were applied.

The structure was solved using heavy-atom method and refined by full-matrix least-squares procedure. Only 818 reflections having intensities greater than 3.0 times their standard deviation were used in the refinement. The final cycle of refinement with anisotropic thermal parameters for non-H atoms converged at R=0.028 (R_w=0.035).

Discussion. Interatomic distances and angles in the structure are given in the Table.

Distances (Å)

Hg-Cl	2.263(3)	O3-C7	1.23(2)
Hg-N	2.021(8)	N-C7	1.45(2)
Hg...O1(i)	2.793(8)	C1-C2	1.39(2)
Hg...O2(ii)	2.811(9)	C1-C6	1.43(2)
Hg...O3(iii)	2.750(10)	C2-C3	1.36(2)
S-O1	1.426(9)	C3-C4	1.38(3)
S-O2	1.433(10)	C4-C5	1.42(2)
S-N	1.622(13)	C5-C6	1.35(2)
S-C1	1.750(12)	C6-C7	1.44(2)

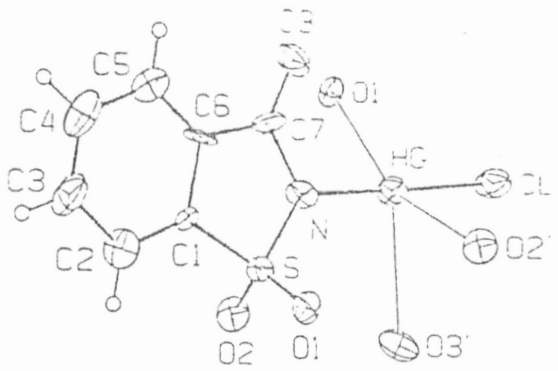
Angles (°)

Cl-Hg-N	177.6(3)	C2-C1-C6	122(1)
O1-S-O2	117.2(6)	C1-C2-C3	118(1)
O1-S-N	110.1(5)	C2-C3-C4	122(1)
O1-S-C1	109.1(5)	C3-C4-C5	120(1)
O2-S-N	109.8(5)	C4-C5-C6	120(1)
O2-S-C1	112.4(5)	C1-C6-C5	118(1)
N-S-C1	96.2(5)	C1-C6-C7	112(1)
Hg-N-S	128.2(7)	C5-C6-C7	130(1)
Hg-N-C7	120.4(9)	O3-C7-N	122(1)
S-N-C7	111.2(7)	O3-C7-C6	126(1)
S-C1-C2	129.(1)	N-C7-C6	112(1)
S-C1-C6	108.8(9)		

(i)=1/2-x, y, z-1/2; (ii)=x, y-1, z; (iii)=1/2-x, y, 1/2+z

The structure is built up of the discrete $C_7H_4ClHgNO_3S$ molecules. The mercury atom has digonal characteristic coordination being bonded to the saccharinate N atom and Cl atom. The Hg-N and Hg-Cl bond lengths are 2.021(8) and 2.263(3) Å, respectively. The value of 2.021(8) Å for Hg-N distance agree well with the values of 2.03-2.06 Å found in Hg(II) saccharinate (Kamenar, Jovanovski & Grdenić, 1982) as well as with those found in the structures with covalent Hg-N bond (e.g. Oppolzer and Weber, 1972). The Cl-Hg-N angle is 177.6°. There are three O atoms from three different saccharinate ligands at Hg...O distances of 2.793(8), 2.811(9) and 2.750(10) Å (see Figure).

Since the O...Hg...O angles range from 69.20° to 159.01°, the coordination polyhedron of the effectively five-coordinated Hg atom (Grdenić, 1965) can be described as a strongly irregular trigonal bipyramid. Saccharinate ligand is planar within the experimental errors. Bond lengths and angles in the saccharinate ligand are close to the values found in Hg(II) sac-



charinate (Kamenar, Jovanovski & Grdenić, 1982) as well as in Na and Mg saccharinates (Jovanovski & Kamenar, 1982) and Mn(II) saccharinate (Kamenar & Jovanovski, 1982).

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