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CRYSTAL STRUCTURE OF LEAD(II) SACCHARINATE MONOHYDRATE $Pb(C_7H_4NO_3S)_2.H_2O$

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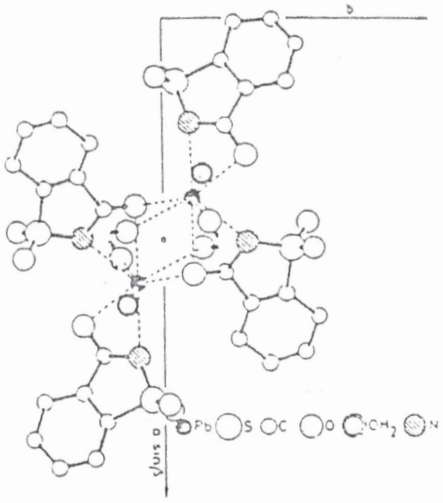
Experimental. The compound was prepared by successive adding of equimolar quantity of $PbCO_3$ to warm aqueous solution of saccharine.

Crystal data: $C_{14}H_{10}O_7S_2Pb$, M=561.54, monoclinic, a=16.037(5), b=13.356(5), c=8.132(3) Å, β=93.04(2)°, V=1739.34 Å^3, D_x = 2.144 gcm^{-3}, Z=4, space group $P2_1/c$.

Intensities were collected on Philips PW1100 automatic diffractometer with MoKα radiation. Reflections were measured using the ω-2θ technique with scan range of 1.2° and scan rate of 0.04°s^{-1}. 3205 reflections with $I \geq 3\sigma(I)$ were considered observed. No absorption correction was applied. The structure was solved by means of three-dimensional Patterson and Fourier syntheses and refined by least-squares procedure using anisotropic temperature factors for all atoms. The final R = 0.085.

Discussion. The structure consists of Pb^{2+} cations, saccharinate anions and water molecules (see Figure). Each Pb^{2+} ion is surrounded by one oxygen atom from the water molecule, five oxygen atoms from CO and SO_2 groups and two nitrogen atoms belonging to saccharinate ions. The Pb-O and Pb-N bond lengths within the irregular coordination polyhedra vary from 2.433 to 2.917 Å and 2.577 to 2.710 Å, respectively.

The CO oxygen atom from one of two crystallographically independent saccharinate ions is coordinated to two Pb^{2+} ions forming a centrosymmetric Pb<O>Pb bridging system with Pb-O distances: 2.665 (2x) and 2.829 Å (2x). The CO oxygen atom from the other saccharinate ion is coordinated to one Pb^{2+} ion



and at the same time participates in hydrogen bonding with the water molecule. The second proton-acceptor involved in hydrogen bonding scheme is one of the oxygens from the SO_2 group. The SO_2 oxygen atoms from the second saccharinate ion are not in contact with Pb^{2+} nor participate in hydrogen bond formation.

Both saccharinate ions are planar with rather large discrepancies between the identical interatomic distances and angles in different ions. This is probably due to the different roles of two crystallographically independent saccharinate ligands in the structure.

In comparison to other known crystal structures of M-saccharinates (M=Na, Mg, Mn, Fe, Co, Ni, Zn, Cd and Hg)[1-3] the structure of Pb(II) saccharinate is the first example in which two atoms (N and O_{CO}) from the same saccharinate ligand are in contact with the metal atom.

References

[1]G. Jovanovski & B. Kamenar, Cryst. Struct. Comm. 11, 247 (1982)
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