

C(7)—C(8)	1.504 (7)	1.514 (6)	1.529 (7)
C(8)—O(3)	1.231 (6)	1.227 (5)	1.220 (5)
C(8)—N(2)	1.328 (5)	1.332 (7)	1.343 (6)
N(2)—C(9)	1.438 (7)	1.445 (6)	1.448 (7)
C(9)—C(10)	1.518 (5)	1.509 (6)	1.527 (7)
C(10)—O(4)	1.314 (6)	1.272 (6)	1.267 (5)
C(10)—O(5)	1.188 (6)	1.244 (5)	1.253 (5)
C(1)—C(2)—C(3)	118.5 (6)	118.6 (6)	118.2 (5)
C(2)—C(3)—C(4)	120.7 (6)	120.8 (7)	120.5 (5)
C(3)—C(4)—C(5)	120.5 (6)	120.7 (6)	120.5 (5)
C(4)—C(5)—C(6)	120.1 (7)	118.9 (6)	120.7 (5)
C(5)—C(6)—C(1)	118.7 (7)	120.3 (6)	118.2 (5)
C(6)—C(1)—C(2)	121.4 (5)	120.7 (5)	121.9 (5)
C(1)—S—O(1)	109.0 (4)	107.4 (3)	107.8 (3)
C(1)—S—O(2)	108.3 (4)	107.1 (3)	107.2 (3)
C(1)—S—N(1)	106.5 (3)	107.7 (3)	108.4 (2)
S—C(1)—C(2)	119.5 (4)	120.6 (4)	118.5 (4)
S—C(1)—C(6)	119.1 (5)	118.6 (4)	119.5 (4)
O(1)—S—N(1)	105.4 (4)	106.4 (3)	107.7 (3)
O(2)—S—N(1)	105.6 (4)	108.4 (3)	105.5 (3)
S—N(1)—C(7)	117.6 (4)	120.1 (4)	119.4 (4)
O(1)—S—O(2)	121.2 (4)	119.5 (3)	119.8 (3)
N(1)—C(7)—C(8)	114.5 (4)	113.4 (4)	113.4 (4)
C(7)—C(8)—O(3)	119.6 (4)	119.5 (4)	119.7 (4)
C(7)—C(8)—N(2)	118.9 (6)	117.0 (4)	116.0 (4)
O(3)—C(8)—N(2)	121.5 (6)	123.5 (5)	124.3 (5)
C(8)—N(2)—C(9)	121.5 (5)	121.9 (4)	122.5 (4)
N(2)—C(9)—C(10)	110.7 (5)	113.6 (4)	114.3 (4)
C(9)—C(10)—O(4)	109.4 (6)	115.2 (4)	114.6 (4)
C(9)—C(10)—O(5)	124.2 (6)	118.5 (4)	119.0 (4)
O(4)—C(10)—O(5)	126.4 (5)	126.3 (4)	126.4 (4)
C(1)—S—N(1)—C(7)	67.1 (5)	-78.9 (4)	-78.9 (4)
O(1)—S—N(1)—C(7)	-177.3 (5)	166.3 (4)	37.5 (5)
O(2)—S—N(1)—C(7)	-47.9 (6)	36.6 (5)	166.6 (4)
S—N(1)—C(7)—C(8)	95.1 (6)	-122.7 (4)	-99.3 (4)
N(1)—C(7)—C(8)—O(3)	-173.5 (5)	-171.4 (5)	-179.5 (5)
N(1)—C(7)—C(8)—N(2)	9.3 (8)	8.2 (7)	2.1 (6)
C(7)—C(8)—N(2)—C(9)	179.8 (5)	-170.6 (5)	171.7 (4)
O(3)—C(8)—N(2)—C(9)	2.7 (9)	8.9 (7)	-6.7 (8)
C(8)—N(2)—C(9)—C(10)	-164.7 (5)	67.6 (6)	96.4 (5)
N(2)—C(9)—C(10)—O(4)	178.2 (5)	175.7 (4)	147.8 (4)
N(2)—C(9)—C(10)—O(5)	-2.3 (8)	-4.0 (6)	-34.2 (6)

Symmetry code: (') 2 - x, 1 - y, 3 - z.

The structures were solved by direct methods and Fourier syntheses using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *SHELX76* (Sheldrick, 1976).

We thank Dr G. N. Mukherjee for the gift of the compounds and the Council of Scientific and Industrial Research for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraamminebis(saccharinato-*N*)cadmium

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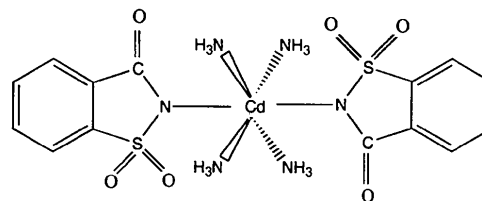
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Abstract

Single crystals of the title compound, [Cd(C₇H₄NO₃S)₂(NH₃)₄], were prepared from tetraaquabis(saccharinato-*N*)cadmium dihydrate using ammonia flux [saccharin is 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide]. The complex displays the same geometry as the parent compound, although its packing in the solid state and hydrogen-bonding network are very different as a result of the absence of the water molecules of crystallization. If exposed to air, the product becomes amorphous; several studies suggest a conversion to the parent hydrogen-bonding pattern by the acquisition of water molecules from the moisture in the air.

Comment

The title compound, (I), crystallizes in space group *P*2₁/*c* with four molecules in the unit cell. The structure consists of neutral molecules comprising Cd atoms bonded to a mutually *trans* pair of saccharinate anions through the (deprotonated) N atom and four equatorial ammonia ligands which complete a slightly distorted octahedral coordination sphere. The Cd atom sits on a crystallographic inversion centre, which relates the two saccharinate ligands.



(I)

The internal arrangement of the complex is analogous to that found in the bis-saccharinato tetra-aqua compounds of first-row transition elements (Haider *et al.*, 1983; Cotton *et al.*, 1986). The packing of the structure in the cell shows a hydrogen-bonding pattern which can be defined as follows. The title compound forms layers that extend in a direction perpendicular to the lattice plane (10 $\bar{1}$), propagated by a stabilizing intermolecular contact (which may or may not be considered to be a hydrogen bond) between the O atom of a saccharinato carbonyl group of one molecule and an ammonia group of another: O(1ⁱ) \cdots N(2) = 3.21 (1) Å, O(1ⁱ) \cdots H(22)—N(2) = 115 (9) $^\circ$ [symmetry code: (i) $x - 1, y, z$]. There is also an interaction, probably energetically favorable, between an O atom of the sulfonyl group of a saccharinato ligand and an H atom of the benzoyl group of another molecule: O(3ⁱⁱ) \cdots H(3)—C(3) = 3.26 (1) Å [symmetry code: (ii) $x + 1, y, z$] (Fig 2). These layers are stacked in a herringbone arrangement, held together by a series of crystallographically equivalent intermolecular contacts which involve the second O atom of the sulfonyl group and the second independent ammonia ligand: O(2ⁱⁱⁱ) \cdots N(2) = 3.17 (1) Å, O(2ⁱⁱⁱ) \cdots H(21)—N(2) = 159 (7) $^\circ$ [symmetry code: (iii) $-x, y - \frac{1}{2}, \frac{1}{2} - z$].

If the complex is stored for several weeks at room temperature and unprotected from the moisture of the air, it evolves into a different compound, with both

aqua and ammonia groups present. This suggests the slow incorporation of water molecules into the system. Studies carried out on the analogous Cu compound, with a much faster evolution of this second product, revealed a hydrated species; studies of this second Cu complex showed a tighter packing arrangement than in the present compound.

Experimental

The title compound was prepared by bubbling ammonia gas into a stirred ethanol solution of [Cd(C₆H₄COSO₂N)₂(OH₂)₄].2H₂O, (II). The white powder that precipitated within 10 min was insoluble in any solvent. Crystals could be obtained from the mother liquor if stored at room temperature for 2 d, or in 8 h if pressurized ammonia gas is introduced to a static very dilute solution of complex (II) in EtOH. The compound undergoes an as yet unidentified chemical change above 573 K. Crystals of the title compound are colorless, with well defined faces. After several weeks exposed to air, spectroscopic studies of the product suggested a very slow conversion to the parent hydrogen-bonding pattern by acquisition of water molecules from the moisture in the air. For X-ray data collection, a small crystal was covered with a thin layer of epoxy; the sample did not show signs of significant change during the course of the data collection.

Crystal data

[Cd(C₇H₄NO₃S)₂(NH₃)₄]
 $M_r = 544.88$
 Monoclinic
 $P2_1/c$
 $a = 7.450 (2) \text{ \AA}$
 $b = 13.184 (2) \text{ \AA}$
 $c = 10.388 (1) \text{ \AA}$
 $\beta = 100.87 (1)^\circ$
 $V = 1002.1 (3) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.806 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 12.05\text{--}15.25^\circ$
 $\mu = 1.343 \text{ mm}^{-1}$
 $T = 298 (2) \text{ K}$
 Well formed needle
 $0.23 \times 0.04 \times 0.04 \text{ mm}$
 Colorless

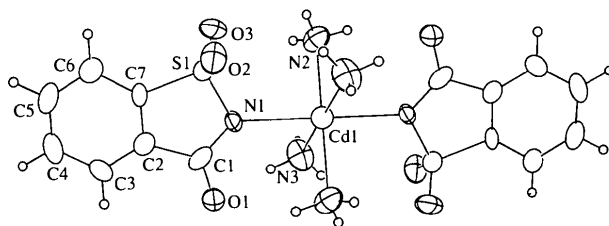


Fig 1. Structure of tetraamminebis(saccharinato-*N*)cadmium. Non-H atoms are represented by their 50% probability ellipsoids. Unlabelled atoms are related to the labelled ones by a crystallographic inversion centre.

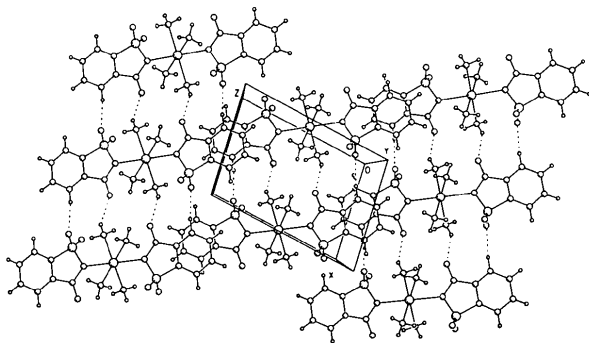


Fig 2. Packing diagram viewed along (010). Note the extension of the two-dimensional layer.

Data collection

Enraf-Nonius CAD-4 four-circle diffractometer
 ω scans
 Absorption correction:
 ψ scan (North, Phillips & Mathews, 1968)
 $T_{\min} = 0.85, T_{\max} = 0.87$
 1898 measured reflections
 1756 independent reflections
 903 observed reflections
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0920$
 $\theta_{\text{max}} = 24.98^\circ$
 $h = 0 \rightarrow 8$
 $k = 0 \rightarrow 15$
 $l = -12 \rightarrow 12$
 3 standard reflections monitored every 400 reflections
 intensity decay: 9.9%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0651$
 $wR(F^2) = 0.0908$

$(\Delta/\sigma)_{\text{max}} = 0.007$
 $\Delta\rho_{\text{max}} = 0.607 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.615 \text{ e \AA}^{-3}$

S = 1.150
 1282 reflections
 166 parameters
 All H-atom parameters
 refined
 $w = 1/[\sigma^2(F_o^2) + (0.0208P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
 Atomic scattering factors
 from *International Tables
 for Crystallography* (1992,
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Cd1	0	0	0	0.0323 (3)
N1	0.1754 (11)	0.0787 (6)	0.1945 (7)	0.033 (2)
C1	0.3585 (17)	0.0771 (8)	0.2334 (10)	0.037 (3)
O1	0.4664 (10)	0.0489 (5)	0.1638 (7)	0.047 (2)
C2	0.4229 (16)	0.1103 (8)	0.3735 (9)	0.033 (3)
C3	0.5954 (16)	0.1146 (10)	0.4475 (12)	0.041 (3)
C4	0.6182 (19)	0.1448 (9)	0.5751 (12)	0.044 (4)
C5	0.4641 (20)	0.1731 (10)	0.6273 (10)	0.044 (3)
C6	0.2932 (18)	0.1696 (9)	0.5564 (11)	0.040 (3)
C7	0.2752 (15)	0.1384 (8)	0.4279 (9)	0.026 (3)
S1	0.0748 (4)	0.1177 (2)	0.3102 (2)	0.0361 (8)
O2	−0.0158 (11)	0.2137 (5)	0.2777 (7)	0.053 (2)
O3	−0.0354 (10)	0.0392 (5)	0.3493 (7)	0.052 (2)
N2	−0.1952 (16)	−0.0860 (9)	0.1122 (11)	0.050 (3)
N3	0.2147 (17)	−0.1313 (8)	0.0250 (12)	0.058 (3)

Table 2. Selected geometric parameters (Å, °)

Cd1—N2	2.324 (11)	Cd1—N1	2.423 (8)
Cd1—N3	2.338 (11)		
N2—Cd1—N3	93.6 (4)	N3—Cd1—N1	88.5 (4)
N2—Cd1—N1	94.6 (3)		

Overall quality of the sample was checked by rotation photography. Lattice parameters were checked by normal-beam oscillation photography and Lorentz–polarization and decay corrections were applied (Sheldrick, 1991)

Saccharinate H atoms were found in difference maps, ammine H atoms were found in a local slant Fourier. The ammine H atoms were constrained to have a common isotropic displacement parameter. Anisotropic displacement parameters were used for all non-H atoms. Refinement was by full-matrix least squares.

Data collection: *CAD-4 PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4 PC*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1152). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Methyldiphenylphosphine)(trimethylsiloxy)gold(I)

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Abstract

Crystals of the title compound, [Au(C₃H₉O₃Si)(C₁₃H₁₃-P)], are composed of monomeric molecules with a nearly linear O—Au—P axis. The molecules are ordered in strings with these O—Au—P axes parallel. The steric bulk of the phosphine ligand and the Me₃SiO group prevent any intermolecular Au···Au contacts. The highly polar Au—O bond results in a short Au—P distance.

Comment

Silicate minerals of Cu and Ag are rare, and it was only with modern sophisticated synthetic methods that metal-rich silicates of the lighter coinage metals could be established (Jansen, 1987). For Au, however, no well defined silicates have been detected, neither for the standard oxidation states I or III, nor for the more unusual II or V (Schmidbaur & Dash, 1982). With the aid of phosphine donor ligands at gold(I) centres and with alkylsilanols as pseudo-silicate components, the first examples of complex molecules with a discrete