N11—Pt—N31	103.30 (18)	N11—C12—C22	112.9 (5)
N11—Pt—C21	79.1 (2)	N11—C16—C15	123.1 (7)
N11—Pt—C41	177.3 (2)	Pt—C21—C22	115.3 (5)
N31—Pt—C21	176.5 (2)	Pt—C21—C25	136.9 (5)
N31—Pt—C41	79.4 (2)	S23—C22—C12	125.5 (5)
C21—Pt—C41	98.2 (3)	S23—C22—C21	115.2 (5)
C22—S23—C24	89.7 (4)	S23—C24—C25	112.6 (6)
C42—S43—C44	90.9 (3)	N31—C32—C33	122.5 (7)
Pt—N11—C12	113.0 (4)	N31—C32—C42	111.4 (5)
Pt—N11—C16	130.0 (5)	N31—C36—C35	124.5 (7)
C12—N11—C16	116.9 (6)	Pt—C41—C42	113.7 (5)
Pt—N31—C32	113.5 (4)	Pt—C41—C45	137.3 (5)
Pt—N31—C36	130.3 (4)	S43—C42—C32	124.7 (5)
C32—N31—C36	116.1 (6)	S43—C42—C41	113.7 (5)
N11—C12—C13	122.0 (6)	S43—C44—C45	113.2 (5)

H atoms were calculated in their ideal positions and refined using a riding model, with a C—H distance of 0.93 Å and isotropic displacement parameters set to 1.2 times the equivalent isotropic parameters of the atoms to which they are attached.

Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990) and *INSIGHT*II (Biosym Technologies, 1993). Software used to prepare material for publication: *PLATON*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KA1218). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Molecular Adducts of Inorganic Salts. VII. Cadmium Tetraoxorhenium Hexakis(thiourea) Hydrate

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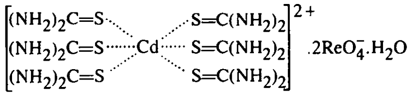
Abstract

The title compound, $[Cd(CH_4N_2S)_6](ReO_4)_2.H_2O$, is built up of isolated centrosymmetric $[Cd\{SC(NH_2)_2\}_6]^{2+}$ octahedra, ReO_4^- tetrahedra and water molecules packed most densely in the *ac* plane. Two of the three non-equivalent thiourea molecules are almost parallel and the third one is perpendicular to the *ac* plane. The structural units are held together by a complex network of intra- and intercluster hydrogen bonds.

Comment

In the system $Cd(ReO_4)_2$–tu–H_2O (tu is thiourea), several different $Cd(ReO_4).n(tu)$ adducts, with $n = 2$, 4 and 6, have been found. For $n = 2$, two polymorphic phases have been identified (Petrova, Angelova & Macíček, 1996). One of them is built up of infinite kröhnkite-type chains (Hawthorne & Ferguson, 1975), while in the other, the chains consist of CdS_3O_3 octahedra linked by alternating double ReO_4 and tu bridges. The crystal structure of $Cd(ReO_4)_2.4tu$ is built up of isolated *trans*-$[M(ReO_4)_2tu_4]$ clusters (Petrova, Angelova, Bakardjieva & Macíček, 1996). In this paper, we discuss the coordination of the Cd atom and the cation–anion packing when $n = 6$, (I).



(I)

The crystal structure of hexakis(thiourea)cadmium tetraoxorhenium monohydrate consists of isolated centrosymmetric $[Cd(tu)_6]^{2+}$ cations, $ReO_4{}^-$ anions and water molecules arranged in 3.1 Å-thick layers extended in the *ac* plane (Figs. 1 and 2). The Cd atom is on a centre of symmetry and is coordinated by six S atoms at the vertices of a trigonal antiprism. The Cd—S distances vary in the range 2.686 (3)–2.734 (2) Å. The basal and side S···S edges are within the ranges 3.481 (3)–3.657 (3) and 4.001 (4)–4.129 (3) Å, respectively. The corresponding basal and side S—Cd—S angles are within the ranges 80.26 (7)–84.85 (7) and 95.15 (7)–99.74 (7)°, respectively. The threefold axis of the CdS_6 trigonal antiprism lies parallel to the *c* axis. Bond lengths and angles in the three tu molecules are in typical ranges (Thanmanivong, Akimov, Andrianov, Struchkov & Molotkin, 1984; Elcombe & Taylor, 1968; Zuñiga *et al.*, 1989). They are almost in-plane coordinated, with Cd—S—C—N torsion angles of 32.3 (8), −24.2 (9) and −21.1 (9)° for the tu1, tu2 and tu3 molecules, respectively. The corresponding angles between the Cd—S bonds and tu planes are 30.3 (1), 21.2 (1) and 18.6 (1)°, respectively, and the Cd atom is out of these planes by 1.37 (1), 0.97 (1) and 0.87 (1) Å, respectively. The tu1 and tu2 molecules are almost parallel, forming a dihedral angle of 17.8 (3)°. Analogous orientation of the thiourea molecules was found in the structure of $Cd(ReO_4)_2.4$tu. In addition, these two tu molecules are inclined to the *ac* plane (the layer plane) by 10.7 (7) and 12.3 (3)°, and by 4.3 (8) and 13.9 (3)° with respect to the S1—S2—S3 face of the CdS_6 antiprism. The tu3 molecule is almost perpendicular to the *ac* plane forming a dihedral angle of 94.2 (2)° and parallel to the $S1^{i}$—S2—S3 face of the CdS_6 antiprism (Table 2) forming a dihedral angle of 15.0 (7)°.

The Re—O bond lengths in the tetraoxorhenium anion are within the range 1.687 (7)–1.747 (8) Å, with an average length of 1.712 (7), in good agreement with the tetraoxorheniums known to date [1.70 (1) Å in $Cd(ReO_4)_2.2$Urea and 1.71 (2) Å in $Cd(ReO_4)_2.2H_2O$ (Angelova, Macíček, Petrova, Todorov & Mihailova, 1996), and 1.71 (8) Å in $Ca(ReO_4)_2.2H_2O$ (Picard, Besse, Chevalier & Gasperin, 1988)]. Its O1—O2—O3 face is almost parallel to the *ac* plane forming a dihedral angle of 9.5 (2)°.

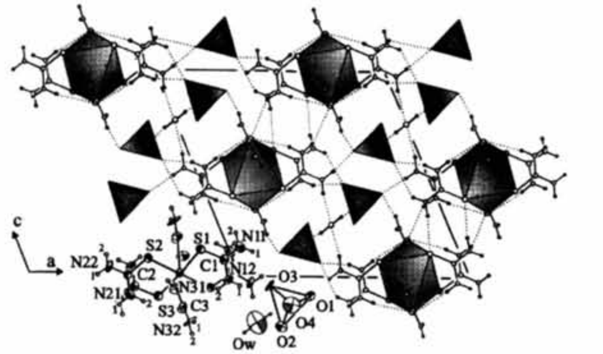


Fig. 1. Projection of the title structure down the *b* axis with the atom-numbering scheme. Displacement ellipsoids are drawn at 50% probability. Hydrogen bonds are denoted by dotted lines.

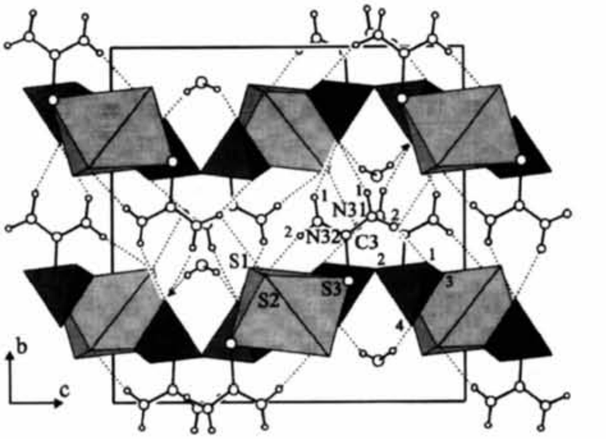


Fig. 2. Projection of the title structure down the *c* axis showing the pseudo-layer packing. For clarity, the tu1 and tu2 molecules are not presented. Hydrogen bonds are denoted by dotted lines.

Although the H atoms were placed in calculated positions, they form a plausible hydrogen-bonding network described in Table 3 and represented in Figs. 1 and 2. There are six intra-cation N—H···S hydrogen bonds with N···S distances ranging between 3.480 (9) and 3.640 (7) Å. Similar internal hydrogen bonding has been found in the structure of $Mg(OCN_2H_4)_6(ClO_4)_2$ (Bondar *et al.*, 1989). The special orientation of the ReO_4 tetrahedron and the tu molecules within the layer leads to many in-layer hydrogen bonds. The inner H atoms of the tu1 molecule take part in six-membered hydrogen-bonded rings to the neighbouring $O3^{ii}$ atom. Similar hydrogen-bonded rings have been found in the structures of some urea adducts $\{[Cr(Urea)_6]^{3+}[Co(NH_3)_2(NO_2)_4]^{-}_{3}.3H_2O$ (Bondar, Kurkutova, Rau, Ilyuhin & Belov, 1979), $CaBr_2.6$Urea (Lebioda & Stadnicka, 1977) and $Ba(ReO_4)_2.3$Urea (Macíček, Angelova & Petrova, 1994)$\}$. Both in-layer tu molecules are hydrogen-bonded to S atoms from an adjacent complex cation (Fig. 1). Hence, the cations can be thought of as ordered in hydrogen-bonded columns along the [101] direction. In addition, tu2 is hydrogen-bonded to the adjacent O*w* acceptor. The perpendicular tu3 molecule is included in analogous types of hydrogen bonds to intra-cation S2 and anionic O1 atoms within the layer and through a six-membered hydrogen-bonded ring to the $O4^{iv}$ atom, as well as to a cationic $S2^{v}$ atom from the adjacent layer.

Of special interest is the structural role of the interstitial water molecule. Its H atoms have been positioned in hydrogen-bonding geometry to the closest O4 atoms and verified by the resulting tetrahedral distribution of all bonds to O*w*. Thus, the O*w* atom acts as an acceptor of two and a donor of two hydrogen bonds (Fig. 1), which suggests that the water molecule serves not merely as a space filler but also as an integral factor in the stability of the structure (Hawthorne, 1992).

Experimental

The title compound was synthesized by slow evaporation of an aqueous solution containing $Cd(ReO_4)_2$ and thiourea in a 1:6 molar ratio at room temperature. Single crystals suitable for the X-ray experiment were obtained after recrystallization from aqueous solution.

Crystal data

$[Cd(CH_4N_2S)_6](ReO_4)_2.H_2O$
$M_r = 1087.53$
Monoclinic
$C2/c$
$a = 15.543\ (2)$ Å
$b = 14.025\ (1)$ Å
$c = 14.676\ (5)$ Å
$\beta = 112.42\ (2)°$
$V = 2957\ (3)$ Å^3
$Z = 4$
$D_x = 2.442$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 22 reflections
$\theta = 20.27–21.76°$
$\mu = 9.45$ mm^{-1}
$T = 292$ K
Cubic
0.16 × 0.16 × 0.16 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.126$, $T_{max} = 0.220$
7381 measured reflections
2452 independent reflections

1798 reflections with $I > 3\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.0°$
$h = 0 \rightarrow 20$
$k = -18 \rightarrow 18$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: 1.5%

Refinement

Refinement on F
$R = 0.032$
$wR = 0.035$
$S = 1.286$
1798 reflections
171 parameters
H atoms refined as riding
$w = 1/[\sigma^2(F) + (0.010F)^2]$

$(\Delta/\sigma)_{max} = 0.826\ [B_{12}$ (Re)]
$\Delta\rho_{max} = 0.827$ e Å^{-3}
$\Delta\rho_{min} = -1.296$ e Å^{-3}
Extinction correction: none
Scattering factors from as coded in *SDP/PDP* (Enraf–Nonius, 1985)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cd	1/4	1/4	1/2	0.0273 (2)
Re	0.33127 (3)	0.33143 (3)	0.14164 (3)	0.0388 (1)
S1	0.3786 (1)	0.3766 (2)	0.6129 (2)	0.0379 (7)
S2	0.1430 (1)	0.3546 (2)	0.5687 (2)	0.0364 (7)
S3	0.1983 (2)	0.3441 (1)	0.3240 (2)	0.0343 (7)
O1	0.2252 (4)	0.3885 (4)	0.1046 (4)	0.045 (2)
O2	0.3998 (5)	0.3736 (7)	0.2540 (5)	0.082 (3)
O3	0.3811 (4)	0.3554 (5)	0.0556 (5)	0.061 (3)
O4	0.3198 (6)	0.2124 (5)	0.1489 (6)	0.097 (4)
O*w*	0	0.3824 (10)	3/4	0.113 (6)
C1	0.4617 (5)	0.3872 (5)	0.5660 (6)	0.034 (3)
C2	0.0289 (5)	0.3593 (5)	0.4893 (6)	0.032 (3)
C3	0.2074 (5)	0.4651 (5)	0.3380 (6)	0.030 (3)
N11	0.5503 (5)	0.4052 (6)	0.6260 (6)	0.067 (3)
N12	0.4447 (5)	0.3793 (5)	0.4704 (5)	0.045 (3)
N21	0.0020 (5)	0.3464 (5)	0.3951 (6)	0.056 (3)
N22	−0.0353 (5)	0.3797 (7)	0.5271 (6)	0.069 (3)
N31	0.2116 (5)	0.5185 (5)	0.2668 (5)	0.050 (3)
N32	0.2085 (5)	0.5075 (5)	0.4198 (6)	0.048 (3)

Table 2. *Selected geometric parameters (Å, °)*

Cd—S1	2.714 (2)	C1—N11	1.345 (10)
Cd—S2	2.686 (3)	C1—N12	1.330 (12)
Cd—S3	2.734 (2)	S2—C2	1.712 (7)
Re—O1	1.724 (6)	C2—N21	1.296 (12)
Re—O2	1.692 (7)	C2—N22	1.344 (14)
Re—O3	1.747 (8)	S3—C3	1.709 (8)
Re—O4	1.687 (7)	C3—N31	1.308 (12)
S1—C1	1.685 (11)	C3—N32	1.334 (12)
S1—Cd—S2	80.26 (7)	O1—Re—O4	111.6 (4)
S1—Cd—S2^i	99.74 (7)	O2—Re—O3	110.4 (4)
S1—Cd—S3	98.25 (6)	O2—Re—O4	109.1 (4)
S1—Cd—S3^i	81.75 (6)	O3—Re—O4	108.8 (4)
S2—Cd—S3	95.15 (7)	N11—C1—N12	116.6 (9)
S2—Cd—S3^i	84.85 (7)	N21—C2—N22	118.8 (7)
O1—Re—O2	108.4 (4)	N31—C3—N32	118.5 (7)
O1—Re—O3	108.5 (3)		

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

Table 3. *Hydrogen-bond geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*	*A*···H···*A*′
N11—H111···O3^{ii}	2.587 (8)	3.302 (13)	132.4 (5)	
N11—H112···S1^{iii}	2.653 (2)	3.583 (9)	166.1 (5)	
N12—H121···O3^{ii}	2.024 (7)	2.894 (12)	151.6 (5)	
N12—H122···S3	2.703 (2)	3.640 (7)	169.1 (5)	
N21—H211···S3^i	2.656 (2)	3.521 (7)	151.7 (6)	
N21—H212···S3	2.673 (3)	3.580 (10)	159.9 (4)	
N22—H221···O1^i	1.899 (5)	2.848 (8)	176.9 (6)	
N22—H222···O*w*	2.175 (1)	3.105 (9)	165.6 (6)	
N31—H311···O4^{iv}	2.281 (9)	3.102 (11)	144.4 (6)	
N31—H312···S2^v	2.839 (2)	3.223 (8)	105.3 (4)	97.4 (2)
N31—H312···O1	2.173 (7)	3.069 (11)	156.7 (4)	
N32—H321···O4^{iv}	2.201 (7)	3.021 (10)	143.9 (5)	
N32—H322···S2	2.544 (3)	3.480 (9)	168.3 (4)	
O*w*—H*w*···O4^{vi}	2.005 (8)	2.941 (10)	167.7 (8)	

Symmetry codes: (i) $-x, y, \frac{1}{2} - z$; (ii) $1 - x, y, \frac{1}{2} - z$; (iii) $1 - x, y, \frac{3}{2} - z$; (iv) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (v) $x, 1 - y, z - \frac{1}{2}$; (vi) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

H atoms were situated at calculated positions and refined as riding with $U_{iso} = 0.0443$ Å^2.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Data reduction: *SDP/PDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SDP/PDP*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Macíček, unpublished).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: SK1053). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 568-570

trans-Chlorobis(diphenylglyoximato)(tri-*n*-butylphosphine)cobalt(III)

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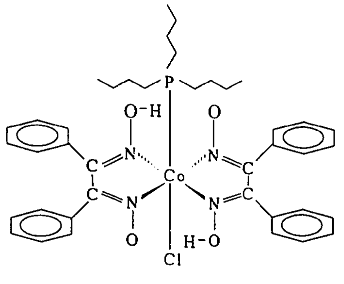
Abstract

The Co atom in the title cobaloxime, $[CoCl(C_{14}H_{11}N_2O_2)_2(C_{12}H_{27}P)]$, is octahedrally coordinated to the two bidentate diphenylglyoximato ligands, approximating a plane with the Cl and P atoms located above and below the plane. The diphenylglyoximato moieties are themselves bent 6.6 (1) and 9.8 (1)° from the central plane of the four N atoms by the bulk of the tri-*n*-butylphosphine ligand. The four phenyl rings of the two diphenylglyoximato groups form a propeller geometry with an average rotation from the central plane of 47 (5)°.

Comment

This analysis was carried out as part of an ongoing study of the reactions between organohaloarsines and cobaloximes. Specifically, the structure of the title compound, (I), can be used to compare the effects of the in-plane methyl groups with the effects of in-plane phenyl groups on the geometry of the cobaloxime. The atom labelling is shown in Fig. 1. The angles between the phenyl rings and the central plane defined by the four N atoms are 42.6 (1), 53.6 (1), 45.6 (1) and 46.3 (1)° for the phenyl rings on atoms C1, C2, C3 and C4, respectively. Short intramolecular hydrogen bonds are formed between atoms O1 and O3 and atoms O2 and O4. The shorter hydrogen bonds are to atoms O2 and O3. The N—C bonds associated with the shorter H bonds are also marginally shorter [0.02 (1) Å] than those associated with the longer hydrogen bonds. However, the N—O bond lengths do not show a consistent pattern.



(I)

Relative to an arsine derivative (Mihichuk, Mombourquette, Einstein & Willis, 1982) and phosphine and phosphite derivatives (Bresciani-Pahor, Calligaris & Randaccio, 1980) of a bis(dimethylglyoximato)-cobaloxime, the C—methyl distances average 0.04 (4) Å longer than the C—phenyl distances reported here. The

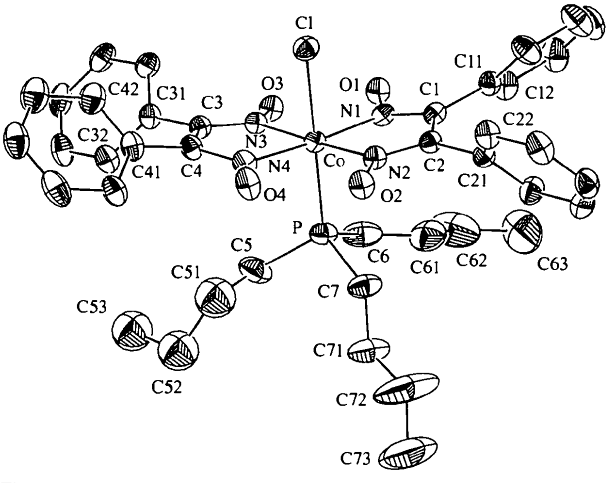


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of the molecule showing the labelling of the non-H atoms with 30% probability displacement ellipsoids. The ring C atoms are labelled C*ij*, where *i* = 1–4 and *j* = 1–6.