SHORT-FORMAT PAPERS

Structure of (Tetrahydroselenophene)mercury(II) Chloride

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Abstract. [Hg(C$_4$H$_8$Se)]Cl$_2$, $M_r = 406 \cdot 6$, triclinic, $P\bar{1}$, $a = 9 \cdot 554$ (1), $b = 7 \cdot 619$ (1), $c = 5 \cdot 926$ (1) Å, $\alpha = 97 \cdot 57$ (1), $\beta = 104 \cdot 48$ (2), $\gamma = 89 \cdot 72$ (1)°, $V = 413 \cdot 8$ (1) Å^3, $Z = 2$, $D_x = 3 \cdot 26$ Mg m^{-3}, λ(Mo $K\alpha$) = $0 \cdot 7107$ Å, $\mu = 24 \cdot 2$ mm^{-1}, $F(000) = 360$, $T = 295$ K, $R = 0 \cdot 042$ for 1747 observed reflections. Coordination around Hg is a distorted trigonal bipyramid with two short bonds Hg–Se $2 \cdot 535$ (1), Hg–Cl $2 \cdot 354$ (2) and three longer Hg–Cl $2 \cdot 63$–$3 \cdot 16$ Å. The compound is isomorphous with [Hg(C$_4$H$_8$S)]Cl$_2$.

Experimental. Transparent needles crystallized from a methanol solution of tetrahydroselenophene and HgCl$_2$. Crystal size $0 \cdot 40 \times 0 \cdot 07 \times 0 \cdot 06$ mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scans. Cell dimensions from setting angles of 25 reflections, $22 < 2\theta < 36°$. Data collected to $\sin\theta/\lambda = 0 \cdot 47$ Å^{-1}, h –12 to 12, k –9 to 9, l –7 to 0, 2923 reflections collected, 2052 unique ($R_{int} = 0 \cdot 03$), 1747 with $I > 3\sigma(I)$ used in the refinement, standard reflections $\bar{5}10$, $12\bar{3}$, $2\bar{4}\bar{2}$, 4% random variation. Lp and absorption corrections, transmission factors $0 \cdot 15$–$0 \cdot 34$. Structure refined with starting parameters from [Hg(C$_4$H$_8$S)]Cl$_2$ (Brändén, 1964). Full-matrix least-squares techniques minimizing

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$\sum w(\Delta F)^2$; $w^{-1} = [\sigma^2(F_o) + (0 \cdot 05 F_o)^2]$. Final refinements with H atoms included in calculated positions with isotropic thermal parameters set to $1 \cdot 2$ times B_{eq} of the bonded atom. Model converged with 1747 reflections, 74 variables, $R = 0 \cdot 042$, $wR = 0 \cdot 054$, $(\Delta/\sigma)_{max} = 0 \cdot 01$, $S = 0 \cdot 99$, isotropic extinction parameter

Table 2. *Selected distances (Å), angles (°) and torsion angles (°)*

Hg–Se	2·535 (1)	Se–C(1)	1·969 (10)
Hg–Cl(2)	2·353 (2)	Se–C(4)	1·986 (11)
Hg–Cl(1^i)	2·634 (2)	C(1)–C(2)	1·508 (16)
Hg–Cl(1^{ii})	2·842 (2)	C(2)–C(3)	1·516 (16)
Hg–Cl(1)	3·165 (2)	C(3)–C(4)	1·472 (14)
Se–Hg–Cl(1^i)	104·94 (5)	Hg^i–Cl(1)–Hg^{iii}	94·97 (6)
Se–Hg–Cl(1^{ii})	97·94 (5)	Hg^i–Cl(1)–Hg	99·22 (6)
Se–Hg–Cl(1)	74·02 (5)	Hg–Cl(1)–Hg^{iii}	161·12 (8)
Se–Hg–Cl(2)	144·37 (7)		
Cl(1^i)–Hg–Cl(1^{ii})	85·03 (6)	C(1)–Se–C(4)	89·6 (4)
Cl(1^i)–Hg–Cl(1)	80·78 (6)	Se–C(1)–C(2)	104·7 (7)
Cl(1^{ii})–Hg–Cl(1)	161·12 (8)	C(1)–C(2)–C(3)	107·8 (8)
Cl(1^i)–Hg–Cl(2)	107·63 (9)	C(2)–C(3)–C(4)	111·0 (10)
Cl(1^{ii})–Hg–Cl(2)	98·87 (9)	C(3)–C(4)–Se	105·7 (7)
Cl(1)–Hg–Cl(2)	97·25 (8)		
Hg–Se–C(1)–C(2)	–125·4 (7)	C(2)–C(3)–C(4)–Se	33·4 (10)
Hg–Se–C(4)–C(3)	95·3 (7)	C(3)–C(4)–Se–C(1)	–7·3 (8)
Se–C(1)–C(2)–C(3)	41·5 (10)	C(4)–Se–C(1)–C(2)	–19·3 (8)
C(1)–C(2)–C(3)–C(4)	–51·5 (12)		

Symmetry code: (i) $-x, -y, 1-z$; (ii) $x, y, z-1$; (iii) $x, y, z+1$.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\beta_{12}\cos\gamma + ac\beta_{13}\cos\beta + bc\beta_{23}\cos\alpha)$.

	x	y	z	B_{eq}(Å^2)
Hg	0·06393 (3)	0·18614 (4)	0·28375 (5)	2·98 (1)
Se	–0·1612 (1)	0·3558 (1)	0·3129 (2)	3·02 (2)
Cl(1)	0·0189 (2)	0·1501 (2)	0·7870 (3)	3·31 (5)
Cl(2)	·0·3184 (2)	0·1993 (4)	0·3912 (5)	4·32 (7)
C(1)	–0·3142 (11)	0·1695 (14)	0·2036 (19)	4·3 (3)
C(2)	–0·4226 (11)	0·2390 (15)	0·0061 (23)	4·8 (3)
C(3)	–0·3391 (11)	0·3241 (14)	–0·1397 (20)	4·7 (3)
C(4)	–0·2288 (10)	0·4524 (12)	0·0100 (20)	3·9 (2)

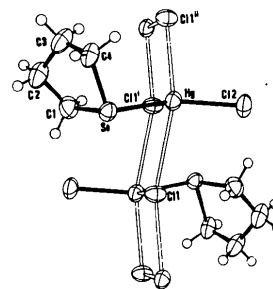Fig. 1. The infinite double chains in the structure and atomic numbering.

0·59 (4) × 10⁻⁴, final $\Delta\rho$ excursions $\leq 3\cdot 2$ e Å⁻³. Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used: see Lundgren (1982).

Atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Selected bond distances, angles and torsion angles are presented in Table 2. The building elements of the structure, infinite double chains, are shown in Fig. 1.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43102 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The crystal structures of $HgCl_2.C_4H_8S$ (Brändén, 1964), $HgCl_2.2C_4H_8S$ and $HgBr_2.2C_4H_8S$ (Sandström & Persson, in preparation).

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Tetraaquatrinitratoeuropium(III) Monohydrate

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Abstract. $[Eu(NO_3)_3(H_2O)_4].H_2O$, $M_r = 428\cdot 05$, triclinic, $P\bar{1}$, $a = 10\cdot 638\,(3)$, $b = 9\cdot 568\,(3)$, $c = 6\cdot 704\,(2)$ Å, $\alpha = 76\cdot 12\,(3)$, $\beta = 84\cdot 68\,(2)$, $\gamma = 63\cdot 72\,(2)°$, $V = 595\cdot 1\,(4)$ Å³, $Z = 2$, $D_x = 2\cdot 388$ Mg m⁻³, $\lambda(\text{Mo }K\alpha) = 0\cdot 71073$ Å, $\mu = 5\cdot 35$ mm⁻¹, $F(000) = 412$, $T = 293$ K, final $R = 0\cdot 079$ for 2521 observed reflections. Three bidentate nitrates and four coordinated water molecules produce a ten-coordinated Eu atom. The coordination polyhedron around Eu approximates a bicapped square antiprism. The crystal structure is built up by the H-bridge-bonded layers of $[Eu(NO_3)_3(H_2O)_4]$ clusters. Between these layers there are the extra water molecules. The structure is isomorphous with its yttrium analogue [Eriksson (1982). *Acta Chem. Scand. Ser. A*, **36**, 186–188].

Experimental. A crystal (Merck, art. 12461) approximating a sphere of diameter 0·2 mm sealed in Lindemann-glass capillary. Philips PW 1100 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Accurate cell constants by least-squares fit for 21 well-distributed general reflections in θ range 9–31°. Data collected by θ–2θ scan method, 3433 reflections, $0\cdot 0768 \leq \sin\theta \leq 0\cdot 7033$, $h\ -12\rightarrow 12$, $k\ -13\rightarrow 13$, $l\ 0\rightarrow 9$, 2521 reflections taken as observed with $I > 10\sigma(I)$. Three check reflections every 2 h, max. and min. correction factors 1·063 and 0·988. Data were corrected for Lorentz and polarization effects and for a small spherical absorption effect $(\mu R = 0\cdot 53)$. Structure solved by Patterson and successive structure-factor and Fourier calculations. Full-matrix refinement. $\sum w(\Delta F)^2$ minimized 18 heavy atoms (164 parameters). Final $R = 0\cdot 079$, $wR = 0\cdot 094$. The large R value is the consequence of the poor quality of the crystal and the difference of the real shape of the crystal from the ideal sphere. $S = 2\cdot 11$, $w = [\sigma^2(F_o) + (2 \times 10^{-4})(F_o)^2]^{-1}$. Extinction coefficient $4\cdot 84 \times 10^{-6}$. No H positions could be located. The final difference map revealed four peaks around the Eu atom forming an irregular rectangle at an average distance of 0·79 (3) Å with a mean electron density of 4 (1) e Å⁻³, $(\Delta/\sigma)_{max} = 0\cdot 27$. Scattering factors from *International Tables for X-ray Crystallography* (1962). Enraf–Nonius *SDP* with local modifications adapted to a PDP 11/34 minicomputer (64K). Final coordinates are given in Table 1. Interatomic distances and selected bond angles are reported