increase in the formal coordination number of the Pb atom in $Pb(S_2COCH_3)_2$ compared with the higher homologues. The structure determination reported herein provides another example of a different coordination environment in a metal xanthate compound when the nature of the *R* substituent in the $^{-}S_2COR$ anion is changed.

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Structures of Dibromobis(tetrahydroselenophene)mercury(II) and Diiodobis(tetrahydroselenophene)mercury(II)

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Abstract. $[HgBr_2(C_4H_8Se)_2]$, $M_r = 630{\cdot}53$, monoclinic, $C2/c$, $a = 23{\cdot}551\ (3)$, $b = 5{\cdot}1288\ (5)$, $c = 14{\cdot}596\ (2)$ Å, $\beta = 128{\cdot}14\ (1)^\circ$, $V = 1386{\cdot}6\ (2)$ Å^3, $Z = 4$, $D_x = 3{\cdot}02$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 220$ cm^{-1}, $F(000) = 1000$, $T = 295$ K, $R = 0{\cdot}025$ for 1083 observed unique reflections. $[HgI_2(C_4H_8Se)_2]$, $M_r = 724{\cdot}53$, triclinic, $P\bar{1}$, $a = 5{\cdot}1653\ (8)$, $b = 9{\cdot}0009\ (12)$, $c = 16{\cdot}884\ (4)$ Å, $\alpha = 92{\cdot}21\ (2)$, $\beta = 91{\cdot}29\ (2)$, $\gamma = 101{\cdot}75\ (2)^\circ$, $V = 767{\cdot}6\ (2)$ Å^3, $Z = 2$, $D_x = 3{\cdot}13$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 187$ cm^{-1}, $F(000) = 572$, $T = 295$ K, $R = 0{\cdot}031$ for 2122 observed unique reflections. Both compounds consist of monomeric species with the Hg atoms four-coordinated by two halogen atoms and two tetrahydroselenophene ligands. The Hg coordination polyhedron is a distorted tetrahedron with [I]Br—Hg—Br[I] and Se—Hg—Se bond angles of 110·06 (4) [120·79 (3)] and 114·79 (4)° [109·62 (4)°]. The Hg—Br[I] and Hg—Se distances are 2·604 (1) [2·730 (1), 2·755 (1)] and 2·648 (1) Å [2·688 (1), 2·718 (1) Å].

Introduction. Compounds of mercury(II) halides with neutral organic sulfur, selenium or tellurium ligands show a remarkable variety in composition, structure and spectroscopic properties. The composition of thioether complexes ($L = RSR'$) can, for example, range from $(HgX_2)_2.L$ to $HgX_2.2L$ with $3HgX_2.2L$ and $HgX_2.L$ being the common forms between the extremes.

As has been confirmed by numerous crystallographic investigations, monomeric as well as halogen-bridged dimeric and polymeric structures occur. Brändén (1964) reported the crystal structures of $(HgCl_2)_2.SEt_2$ and $HgCl_2$.THT (THT = tetrahydrothiophene), both consisting of polymeric chains built up by halogen bridges. Sandström & Persson (in preparation) have investigated the structures of HgX_2.2THT (X = Cl, Br) in the solid state and, together with Goggin (Sandström, Persson & Goggin, 1986), the bromide and iodide in THT solution. They found, both in the solid compounds and in the THT solutions, a distorted tetrahedral arrangement around Hg with two halogens and two THT S atoms.

Experimental. Summary of experiments and refinements are given in Table 1. Crystals were grown from a solution of mercury(II) bromide (iodide) in warm acetonitrile on addition of excess of tetrahydroselenophene (THSe), followed by slow cooling to room temperature. Single crystals of the two compounds were

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sealed in Lindemann capillaries together with a little mother liquid. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scans. Structure solved by direct methods with *MULTAN*80 (Main *et al.*, 1980). In the full-matrix least-squares refinements a parameter was included to correct for secondary extinction (Zachariasen, 1967). Scattering factors and anomalous-dispersion factors from *International Tables for X-ray Crystallography* (1974). Programs used: see Lundgren (1982).

Table 1. *Summary of experiments and refinements*

	[$HgBr_2(C_4H_8Se)_2$]	[$HgI_2(C_4H_8Se)_2$]
Crystal dimensions (mm)	0·48 × 0·12 × 0·08	0·29 × 0·12 × 0·10
Number of reflections for lattice parameters	50	50
and their 2θ range (°)	$20 < 2\theta < 35$	$22 < 2\theta < 40$
Data collection $2\theta_{max}$ (°)	55	50
Scan width (°)	0·70 + 0·5 tanθ	0·75 + 0·5 tanθ
Standard reflections	712, 425, 934	318, 155
(% variation)	(4%)	(5%)
Number of measured reflections	1620	2878
h, k, l range	0→28, 0→6, −17→17	−6→10, −10→10, −19→19
Number of unique reflections	1365	2458
R_{int}	0·038	0·035
Reflections with $I > 3\sigma(I)$	1083	2122
Least-squares parameters	61	119
Transmission-factor range	0·09–0·36	0·13–0·25
Weights (w^{-1})	$\sigma^2(F_o) + (0{\cdot}015F_o)^2 + 2{\cdot}0$	$\sigma^2(F_o) + (0{\cdot}030F_o)^2 + 1{\cdot}0$
Largest Δ/σ in final cycle	0·02	0·02
$\Delta\rho_{max}$ (e Å^{-3})	0·59	1·63
$R(F)$	0·025	0·031
$wR(F)$	0·031	0·042
S	1·08	0·99
Secondary-extinction parameter, g	0·25 (3) × 10^4	0·52 (2) × 10^4

Table 2. *Atomic coordinates and isotropic temperature factors* (Å^2)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
[$HgBr_2(C_4H_8Se)_2$]				
Hg	0·0000	0·1297 (1)	0·2500	0·0419 (2)
Br	0·0879 (1)	0·4208 (2)	0·2429 (1)	0·0502 (5)
Se	0·0797 (1)	−0·1485 (2)	0·4440 (1)	0·0415 (4)
C1	0·1772 (4)	−0·0625 (18)	0·4967 (7)	0·050 (4)
C2	0·1968 (4)	0·1896 (18)	0·5644 (8)	0·055 (5)
C3	0·1701 (5)	0·1656 (20)	0·6385 (7)	0·061 (5)
C4	0·0899 (4)	0·1129 (17)	0·5557 (6)	0·049 (4)
[$HgI_2(C_4H_8Se)_2$]				
Hg	0·16239 (8)	0·03722 (4)	0·26345 (2)	0·0458 (1)
I(1)	0·37058 (14)	−0·14047 (7)	0·15566 (4)	0·0556 (2)
I(2)	0·48276 (14)	0·22684 (8)	0·37154 (4)	0·0592 (2)
Se(1)	−0·07544 (20)	0·22441 (12)	0·17788 (6)	0·0529 (3)
Se(2)	−0·20108 (19)	−0·14482 (11)	0·34660 (6)	0·0520 (3)
C(1)	0·2275 (29)	0·4014 (12)	0·1689 (7)	0·077 (5)
C(2)	0·3141 (35)	0·3983 (19)	0·0841 (9)	0·102 (7)
C(3)	0·1888 (44)	0·2669 (25)	0·0386 (10)	0·160 (10)
C(4)	−0·0218 (22)	0·1563 (13)	0·0674 (5)	0·061 (4)
C(5)	−0·1707 (20)	−0·3460 (10)	0·3023 (6)	0·054 (3)
C(6)	0·0424 (20)	−0·3934 (11)	0·3487 (7)	0·059 (4)
C(7)	0·0208 (29)	−0·3439 (13)	0·4351 (8)	0·082 (5)
C(8)	0·0201 (29)	−0·1732 (13)	0·4407 (6)	0·074 (5)

Discussion. Final atomic coordinates and temperature factors are listed in Table 2,* selected interatomic distances and angles are given in Table 3. The [$HgBr_2(C_4H_8Se)_2$] molecule is drawn in Fig. 1 and Fig. 2 shows the packing of the [$HgI_2(C_4H_8Se)_2$] molecules in the unit cell.

Table 3. *Selected bond lengths* (Å) *and angles* (°)

	[$HgBr_2(C_4H_8Se)_2$]	[$HgI_2(C_4H_8Se)_2$]
Hg–I(2)		2·730 (1)
Hg–Br,I(1)	2·604 (1)	2·755 (1)
Hg–Se(2)		2·688 (1)
Hg–Se(1)	2·648 (1)	2·718 (1)
Se(1)–C(1)	1·97 (1)	2·01 (1)
Se(1)–C(4)	2·01 (1)	1·98 (1)
C(1)–C(2)	1·52 (1)	1·51 (2)
C(2)–C(3)	1·56 (1)	1·42 (3)
C(3)–C(4)	1·51 (1)	1·43 (2)
Se(2)–C(5)		1·97 (1)
Se(2)–C(8)		1·99 (1)
C(5)–C(6)		1·48 (1)
C(6)–C(7)		1·52 (2)
C(7)–C(8)		1·54 (2)
Br,I(1)–Hg–Br,I(2)	110·06 (4)	120·79 (3)
Se(1)–Hg–Se(2)	114·79 (4)	109·62 (4)
Br,I(1)–Hg–Se(1)	108·78 (3)	106·62 (3)
Br,I(1)–Hg–Se(2)	107·20 (3)	108·46 (3)
I(2)–Hg–Se(1)		104·47 (3)
I(2)–Hg–Se(2)		106·54 (3)
C(1)–Se(1)–C(4)	90·4 (3)	89·7 (5)
Se(1)–C(1)–C(2)	104·9 (5)	106·8 (9)
C(1)–C(2)–C(3)	107·1 (7)	114·0 (14)
C(2)–C(3)–C(4)	107·5 (7)	122·9 (15)
C(3)–C(4)–Se(1)	103·4 (5)	106·1 (9)
C(5)–Se(2)–C(8)		90·0 (4)
Se(2)–C(5)–C(6)		106·7 (7)
C(5)–C(6)–C(7)		108·0 (9)
C(6)–C(7)–C(8)		109·2 (10)
C(7)–C(8)–Se(2)		103·3 (8)

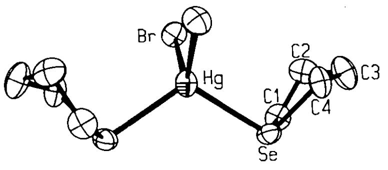

Fig. 1. The [$HgBr_2(C_4H_8Se)_2$] molecule.

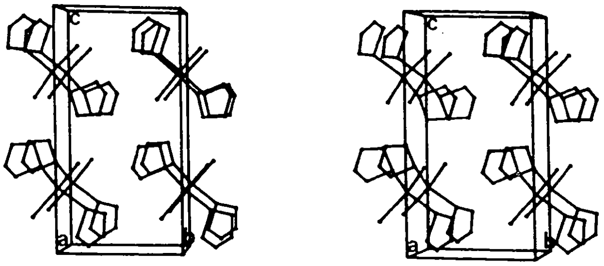

Fig. 2. Stereoview of the molecular packing in [$HgI_2(C_4H_8Se)_2$].

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44435 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *A comparison of bond lengths* d(Hg—X), (X = Cl, Br, I) *and* d(Hg—L) (L = *pyridine, THT, THSe*) *with the bond angles* X—Hg—X *and* L—Hg—L *in pseudotetrahedral* HgX_2L_2 *complexes*

Mean values are given for the distances in the solids.

	d(Hg—X) (Å)	d(Hg—L) (Å)	∠(X—Hg—X) (°)	∠(L—Hg—L) (°)
$HgCl_2$ in pyridine[a]	2·375 (10)	2·47 (2)	150	
$HgCl_2(THT)_2$[b]	2·459	2·57	115·4	109·1
$HgBr_2$ in pyridine[a]	2·497 (2)	2·45 (2)	147 (2)	
$[HgBr_2(py)_2]$[c]	2·481	2·39	141·2 (1)	90·7 (7)
$HgBr_2$ in THT[d]	2·535 (6)	2·62	132 (2)	
$[HgBr_2(THT)_2]$[b]	2·553	2·60	117·6 (1)	107·7
$[HgBr_2(THSe)_2]$[e]	2·604 (1)	2·648 (1)	110·06 (4)	114·79 (4)
HgI_2 in pyridine[a]	2·665 (2)	2·43 (2)	141 (2)	
$[HgI_2(py)_2]$[a]	2·666	2·424 (9)	142·7 (1)	93·8 (4)
HgI_2 in THT[d]	2·670 (4)	2·72	143 (2)	
$[HgI_2(THSe)_2]$[e]	2·742	2·703	120·79 (3)	109·62 (4)

References: (*a*) Persson, Sandström, Goggin & Mosset (1985); (*b*) Sandström & Persson (In preparation); (*c*) Canty, Raston, Skelton & White (1982); (*d*) Sandström, Persson & Goggin (1986); (*e*) Present work.

The present compounds consist of discrete molecular complexes of approximate C_{2v} point symmetry with the Hg atoms coordinating two halogens and two THSe Se atoms. In $HgBr_2.2THSe$, the angles Br—Hg—Br and Se—Hg—Se are 110·06 (4) and 114·79 (4)° and the Hg—Se distance is 2·648 (1) Å. Corresponding angles in the iodide are 10·7° wider and 5·2° smaller and the average Hg—Se distance is shortened by 0·05 Å. Distances and angles in similar pseudotetrahedral complexes HgX_2L_2, with L = THT, THSe and pyridine (py), are listed in Table 4, which also includes complexes found in THT and pyridine solutions. In the solid compounds $HgBr_2L_2$ a considerable decrease in the Br—Hg—Br angle (141·2 to 110·1°) is, as expected, associated with an increasing Hg—Br bond length (2·481 to 2·604 Å) in the sequence L = py, THT and THSe. The $[HgCl_2(py)_2]$ structure (Canty, Raston, Skelton & White, 1982) is of a different type, with Hg octahedrally coordinated. Crystals of $[HgCl_2THSe]$ and not $[HgCl_2(THSe)_2]$ are obtained from a solution of $HgCl_2$ in excess THSe. In $[HgCl_2THSe)]$ (Stålhandske & Zintl, 1986), the coordination around Hg is a distorted trigonal bipyramid.

Some of the distances and angles in one of the THSe ligands in the $[HgI_2(C_4H_8Se_2)_2]$ molecule deviate from the expected values (Nahlovska, Nahlovsky & Seip, 1970), which might indicate conformational disorder.

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Structure of Dichlorobis(pentane-2,4-dionato)bis(tetrahydrofuran)uranium(IV)

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Abstract. $[U(C_5H_7O_2)_2Cl_2(C_4H_8O)_2]$, M_r = 655·4, monoclinic, $C2/c$, a = 14·873 (4), b = 8·728 (1), c = 17·518 (3) Å, β = 97·62 (2)°, V = 2254 (1) Å^3, Z = 4, D_x = 1·919 Mg m^{-3}, Mo $K\alpha$ radiation, λ = 0·71073 Å, μ = 7·095 mm^{-1}, $F(000)$ = 1264, T = 295 K, final R_F = 0·039 for 1429 unique reflections. The structure is of molecular type: the U atom is eight coordinated by two Cl [U—Cl 2·684 (3) Å] and six O atoms from two bidentate diketonato ligands [U—O 2·265 (8), 2·366 (9) Å] and from two tetrahydrofuran ligands [U—O 2·618 (8) Å]. The stereochemistry of the U atom corresponds to a dodecahedral environment.

Introduction. Recent interest in studies of the high coordination numbers of U^{IV} has turned our attention to the synthesis of compounds such as $[UCl_2(acac)_2L_n]$ (acac ≡ $CH_3COCHCOCH_3$) in the hope of understanding the influence of the nature of the ligand L on

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