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Dilead mercury chromate(VI), Pb_2HgCrO_6

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The structure of Pb_2HgCrO_6 (space group $P\overline{1}$) can be described as consisting of isolated $[CrO_4]^{2-}$ tetrahedra and nearly linear $[HgO_2]^{2-}$ dumb-bells, which form layers of composition $[HgCrO_6]^{4-}$. These are intercalated with corrugated pseudo-hexagonal Pb^{2+} layers. The Pb^{2+} cation is stereochemically active and has coordination 3+5.

Comment

Pb_2HgCrO_6 was first obtained as a by-product of different syntheses aiming to produce ternary silver oxides in steel autoclaves with perchloric acid as an accelerator (Curda *et al.*, 2001). Subsequently, single crystals of Pb_2HgCrO_6 have been prepared by solid-state reaction of a mixture of HgO, PbO_2 and elemental Cr under an elevated oxygen pressure.

The structure of Pb_2HgCrO_6 can be described as consisting of three basic building units: isolated $[CrO_4]^{2-}$ tetrahedra and nearly linear $[HgO_2]^{2-}$ dumb-bells form layers of composition $[HgCrO_6]^{4-}$, which are intercalated with corrugated pseudo-hexagonal Pb^{2+} layers perpendicular to the $[\overline{1}10]$ direction (Fig. 1).

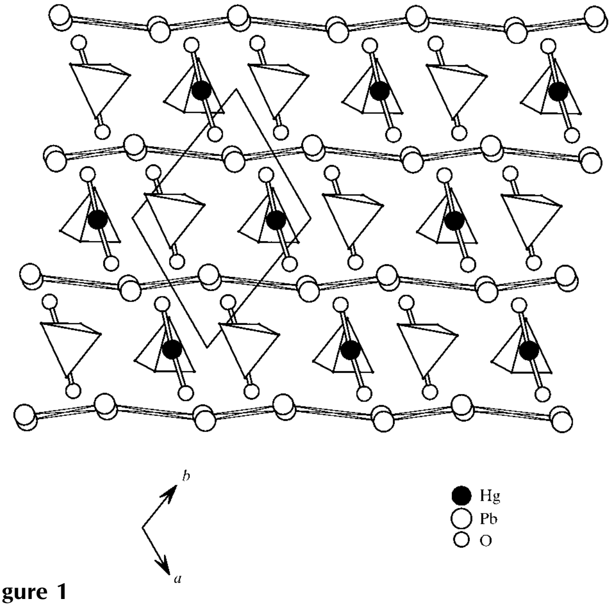

Figure 1
A projection on to *ab* of the structure of Pb_2HgCrO_6. The $[CrO_4]^{2-}$ tetrahedra are shown.

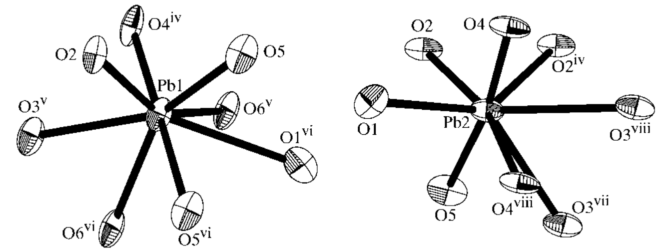

Figure 2
Views of the two environments of the Pb^{2+} cations in Pb_2HgCrO_6. Symmetry codes are as given in Table 1.

The Pb^{2+} cation is stereochemically active. Every Pb^{2+} cation has a first coordination sphere formed by three O atoms at distances ranging from 2.24 to 2.45 Å. The second coordination sphere is formed by five further O atoms at distances of 2.64–3.66 Å (Table 1 and Fig. 2).

Bond-valence sums for the cations, calculated according to Brese & O'Keeffe (1991), are 6.16 for Cr, 1.98 for Hg, and 2.14 and 2.21 for Pb. While for atoms O1, O5 and O6, the bond-valence sums are within the normal range (2.09, 2.11 and 1.96, respectively), for O2 (2.22), O3 (1.79) and O4 (2.30), higher deviations from the ideal value are observed.

Experimental

To synthesize Pb_2HgCrO_6, a mixture of HgO, PbO_2 and elemental Cr (molar ratio 1:2:1), under an elevated oxygen pressure of 11 MPa, was annealed for 120 h at 773 K in silver crucibles placed in stainless steel autoclaves (Linke & Jansen, 1997) using H_2O (2 ml) as an accelerator. Single crystals of Pb_2HgCrO_6 were formed.

Crystal data

Pb_2HgCrO_6
$M_r = 762.98$
Triclinic, $P\overline{1}$
$a = 6.505$ (2) Å
$b = 7.201$ (3) Å
$c = 7.605$ (3) Å
$\alpha = 91.82$ (3)°
$\beta = 92.17$ (3)°
$\gamma = 111.33$ (3)°
$V = 331.2$ (2) Å^3
$Z = 2$
$D_x = 7.651$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8070 reflections
$\theta = 2.6$–24.5°
$\mu = 75.35$ mm^{-1}
$T = 293$ (2) K
Prismatic, red
0.20 × 0.05 × 0.02 mm

Data collection

Stoe IPDS-II diffractometer
Image-plate scans
Absorption correction: numerical (Coppens, 1970)
$T_{min} = 0.011$, $T_{max} = 0.081$
3475 measured reflections
1838 independent reflections
1583 reflections with $I > 2\sigma(I)$
$R_{int} = 0.070$
$\theta_{max} = 29.5°$
$h = -9 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -10 \rightarrow 10$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.127$
$S = 0.97$
1838 reflections
92 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0857P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 3.68$ e Å^{-3}
$\Delta\rho_{min} = -3.30$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0073 (7)

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Table 1
Selected geometric parameters (Å, °).

Hg1—O4	1.993 (9)	Pb1—O1^{vi}	3.209 (12)
Hg1—O2^{i}	2.019 (9)	Pb2—O4	2.236 (9)
Hg1—O1^{i}	2.673 (9)	Pb2—O2	2.384 (10)
Hg1—O6^{ii}	2.749 (9)	Pb2—O2^{iv}	2.395 (11)
Hg1—O3	3.150 (9)	Pb2—O1	2.642 (14)
Hg1—O3^{iii}	3.193 (9)	Pb2—O5	2.795 (14)
Hg1—O1	3.459 (9)	Pb2—O3^{vii}	3.109 (14)
Pb1—O4^{iv}	2.240 (11)	Pb2—O3^{viii}	3.215 (14)
Pb1—O2	2.375 (10)	Pb2—O4^{viii}	3.571 (14)
Pb1—O5	2.448 (12)	Cr1—O1	1.609 (12)
Pb1—O3^{v}	2.765 (12)	Cr1—O6	1.619 (11)
Pb1—O6^{vi}	2.893 (12)	Cr1—O5^{ix}	1.650 (13)
Pb1—O6^{v}	2.895 (12)	Cr1—O3	1.661 (12)
Pb1—O5^{vi}	3.080 (12)		
O4—Hg1—O2^{i}	174.5 (4)	O1—Cr1—O6	107.5 (7)
O4^{iv}—Pb1—O2	75.7 (4)	O1—Cr1—O5^{ix}	111.0 (6)
O4^{iv}—Pb1—O5	90.6 (4)	O6—Cr1—O5^{ix}	112.2 (6)
O2—Pb1—O5	78.9 (3)	O1—Cr1—O3	109.5 (7)
O4—Pb2—O2	93.6 (4)	O6—Cr1—O3	108.5 (6)
O4—Pb2—O2^{iv}	75.4 (4)	O5^{ix}—Cr1—O3	108.0 (5)
O2—Pb2—O2^{iv}	71.5 (4)		

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x, y, 1+z$; (iii) $2-x, 2-y, 1-z$; (iv) $-x, 1-y, 1-z$; (v) $x-1, y-1, z$; (vi) $-x, 1-y, -z$; (vii) $x-1, y, z$; (viii) $1-x, 2-y, 1-z$; (ix) $1+x, y, z$.

Refinement was also carried out in space group $P1$, but did not lead to better agreement factors and was therefore discarded.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1998–2000).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1350). Services for accessing these data are described at the back of the journal.

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