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$PbCl(ReO_4)$, a derivative of the matlockite (PbFCl) structure

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(e–O) = 0.014 Å
R factor = 0.035
wR factor = 0.089
Data-to-parameter ratio = 12.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Single crystals of lead(II) chloride perrhenate(VII), (I), were obtained from a reaction of $PbCl_2$ and $Pb(ReO_4)_2$. Its structure can be considered as an alternation of lead–chlorine slices, $[Pb_2Cl_2]^{2+}$, formed of edge-sharing $ClPb_4$ tetrahedra, and double layers of perrhenate anions, $[ReO_4{}^-]_2$. Except for one O atom, all other atoms in (I) are situated on mirror planes. The arrangement is derived from a distorted matlockite (PbFCl) structure by replacing F with Cl, and introducing the ReO_4 group instead of Cl. The structure of (I) is the first PbFCl derivative containing tetrahedral anions.

Comment

Lead oxide halides often form complicated layered structures. A large family of structurally related compounds with general formula $(Pb,M)_8O_{8+x}X_2$ is observed for *M* = Si, Ge, Ti, V, Nb, Ta, P, As, S and Cr (Aurivillius, 1982, 1983; Cooper & Hawthorne, 1994). Our investigation of the systems PbO–PbX_2–$Pb(ReO_4)_2$, in the search for possible Re-containing analogues, resulted in two new compounds $PbX(ReO_4)$, *X* = Cl and Br, and we present the results for the Cl member, (I), here.

The crystal structure of (I) is made up of $[Pb_2Cl_2]^{2+}$ layers and $[ReO_4]_2$ double layers alternating along the *b* axis (Fig. 1). Compound (I) can be derived from a distorted matlockite (PbFCl) structure (Pasero & Perchiazzi, 1996) by replacing F^- with Cl^-, and introducing the $ReO_4{}^-$ group instead of Cl^-. The $[Pb_2Cl_2]^{2+}$ layers can be considered as slices from a fluorite-type structure where one-half of the anions are removed, which results in a net of condensed $ClPb_4$ tetrahedra. The stability of such anion-centred tetrahedra (*e.g.* OCu_4, OLn_4, OPb_4, OHg_4) has been addressed previously (Krivovichev & Filatov, 1999; Borisov *et al.*, 2005). The $[Pb_2Cl_2]^{2+}$ slices are distorted in a way reminiscent of $AgCuI_2$ (Avilov & Baranova, 1972), but the Pb—Cl distances [2.834 (6)–3.186 (6) Å] are similar to those observed in the related $PbSbO_2Cl$ (3.137–3.255 Å; Giuseppetti & Tadini, 1973) whose structure may alternatively be described as a sequence of $[Pb_2Cl_2]^{2+}$ fluorite-type slices and $[SbO_2]^-$ ribbons. The coordination polyhedron around Pb^{2+} in (I) is a distorted tricapped trigonal prism (4Cl + 5O; Fig. 2).

There are no structural analogies between $PbX(ReO_4)$ and any of the structurally characterized $AX(MO_4)$ compounds (*A* = Ln, Bi, Fe; *X* = Hal, OH; *M* = S, Cr, Mo, W) (Aurivillius & Lowenhielm, 1964; Brixner *et al.*, 1982; Bueno *et al.*, 1989, 1991; Johansson, 1962; Klevtsova & Borisov, 1969; Wang *et al.*, 2000; Wickleder, 1999). However, formation of isostructural compounds was detected during exploration of alkaline earth analogues of (I).

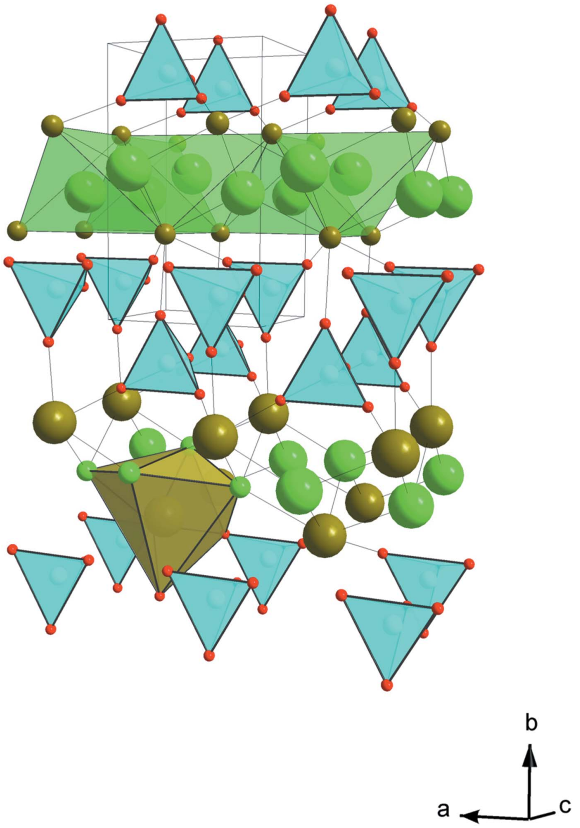

Figure 1
The crystal structure of (I). Pb atoms are brown, ReO_4 tetrahedra are blue, Cl atoms are green, and O atoms are red.

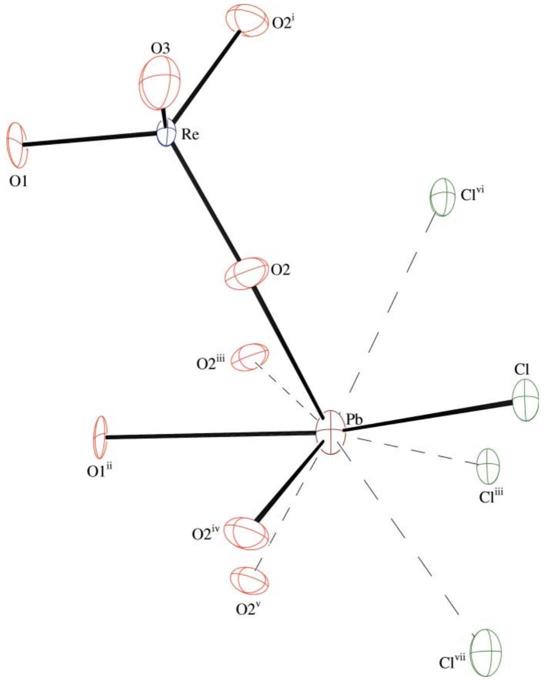

Figure 2
The environment around the Pb and Re atoms. Displacement ellipsoids are plotted at the 50% probability level. Solid lines are given for bonds shorter than 2.84 Å, whereas dashed lines represent bonds longer than 2.84 Å. [Symmetry codes: (i) $-x, y, z$; (ii) $\frac{1}{2} - x, -y, -\frac{1}{2} + z$; (iii) $x, y, -1 + z$; (iv) $1 - x, y, z$; (v) $1 - x, y, -1 + z$; (vi) $\frac{3}{2} + x, 1 - y, -\frac{1}{2} + z$; (vii) $\frac{1}{2} - x, 1 - y, -\frac{1}{2} + z$].

Experimental

Polycrystalline samples of $PbCl(ReO_4)$ and $PbBr(ReO_4)$ were obtained by annealing PbX_2 (X = Cl, Br) and $Pb(ReO_4)_2$ in evacuated silica tubes at 670–520 K for 1–2 d. $Pb(ReO_4)_2$ was obtained by annealing a 1:2 mixture of PbO and NH_4ReO_4 first at 723 K for 24 h and, after regrinding, at 823 K for 55 h. All starting compounds were of purity 99% or higher. Single crystals were mechanically extracted from $3PbX_2$:$Pb(ReO_4)_2$ mixtures melted at 620–670 K for 36–48 h. The crystals were brown to almost black thick platelets or prisms. Ground powders are off-white. Second harmonic generation (SHG) tests were positive for both halide perrhenates. The cell parameters of the isostructural $PbBr(ReO_4)$ are a = 5.7473 (6) Å, b = 9.6512 (9) Å and c = 4.5268 (6) Å. Compound (I) decomposes upon heating in air around 680 K, yielding black amorphous products. $PbBr(ReO_4)$ melts incongruently at about 670 K. Attempts to prepare lead perrhenate analogues with X = F or I, as well as $BiXMO_4$ (X = halogen, M = Mo or W), were not successful, leaving unreacted PbX_2 and $Pb(ReO_4)_2$ or $BiOX$ and MO_3.

Crystal data

$PbCl(ReO_4)$
M_r = 492.84
Orthorhombic, $Pmn2_1$
a = 5.6800 (3) Å
b = 9.4389 (5) Å
c = 4.4656 (2) Å
V = 239.41 (2) Å^3
Z = 2
D_x = 6.837 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 21 reflections
θ = 4.2–32.6°
μ = 60.82 mm^{-1}
T = 173 (2) K
Prism, black
0.07 × 0.06 × 0.03 mm

Data collection

Bruker–Nonius X8 APEX CCD area-detector diffractometer
φ scans
Absorption correction: numerical (*XPREP* in *SHELXTL*; Bruker, 2004)
T_{min} = 0.016, T_{max} = 0.163
1798 measured reflections

518 independent reflections
517 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.3°
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = -5 \rightarrow 5$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.089$
S = 1.27
518 reflections
41 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.5031P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 5.68 e Å^{-3}
$\Delta\rho_{min}$ = −2.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.109 (7)
Absolute structure: Flack (1983), 160 Friedel pairs
Flack parameter: 0.07 (2)

Table 1
Selected geometric parameters (Å, °).

Re—O3	1.67 (2)	Pb—Cl	2.834 (6)
Re—O1	1.718 (15)	Pb—O2^{iii}	3.084 (13)
Re—O2	1.741 (10)	Pb—Cl^{viii}	3.147 (2)
Pb—O2	2.537 (12)	Pb—Cl^{iii}	3.186 (6)
Pb—O1^{ii}	2.657 (16)		
O3—Re—O1	106.3 (9)	O3—Re—O2^{i}	106.1 (6)
O3—Re—O2	106.1 (6)	O1—Re—O2^{i}	113.3 (5)
O1—Re—O2	113.3 (5)	O2—Re—O2^{i}	111.0 (7)

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

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The highest peak is located 0.79 Å from Re, and the deepest hole 0.96 Å from Pb.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

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