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Indium tellurium trioxide chloride, $InTeO_3Cl$

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The new oxychloride $InTeO_3Cl$ was synthesized from a mixture of In_2O_3, $InCl_3$ and TeO_2. Its structure has been determined from single-crystal X-ray diffraction data. The structure is composed of layers separated by a van der Waals gap. The layers consist of edge-sharing chains of $[InO_4Cl_2]$ octahedra linked through $[TeO_3]$ trigonal pyramids. No free Cl atoms are located between the layers.

Comment

One of the most common oxychlorides is FeOCl. This layered compound possesses various interesting chemical properties (Rouxel *et al.*, 1987). Indeed, it is possible to intercalate alkali metals or molecules between the layers. It is also possible to substitute the chlorine layers by nucleophilic alkali metal salts, these grafting or pillaring reactions leading to new layered compounds. Some authors have developed the family of oxyhalide compounds with the synthesis of metal–tellurium oxyhalides (see, for instance, Jerez *et al.*, 1987; Alonso, 1998; Nikiforov *et al.*, 1999). All these compounds, as with FeOCl, exhibit lamellar organization. The structural differences observed depend on the nature of the metal and the oxidation state of Te. The title compound is the first to be synthesized with a group 13 metal. The charge balance proposed for this compound is $In^{3+}Te^{4+}O_3^{2-}Cl^-$.

The structure is composed of puckered layers separated by a van der Waals gap, as shown in Fig. 1. The layers consist of edge-sharing chains of $[InO_4Cl_2]$ octahedra running along the *a* direction and linked through $[TeO_3]$ trigonal pyramids (Fig. 2). Such threefold coordination is usual for Te with a +4 oxidation state. The Te^{4+} lone electron pair points toward the van der Waals gap.

The In—O and In—Cl distances are comparable with the average distances encountered in InOCl (2.15 and 2.53 Å, respectively; Forsberg, 1956), and with the values reported by Shannon (1976) of 2.27 and 2.73 Å, respectively. The Te—O distances in the $[TeO_3]$ trigonal pyramid [1.897 (5)–1.907 (4) Å] are close to the value of 1.87 Å expected from the Shannon table, and to the average distance found in $SbTeO_3Cl$ (1.92 Å; Alonso, 1998). Concerning the two long Te—O distances of 2.586 Å between Te and O2 and O3, their bond-valence contributions are equal to 0.19 [the values for the bond valence calculation are taken from Brown & Altermatt (1985)]. Thus, they can be considered as weak or secondary bonds. When these bonds are included in the bond-valence sums, the sum around Te is 4.05 and that around O2 and O3 is 1.98, and these are in good agreement with the charge balance proposed.

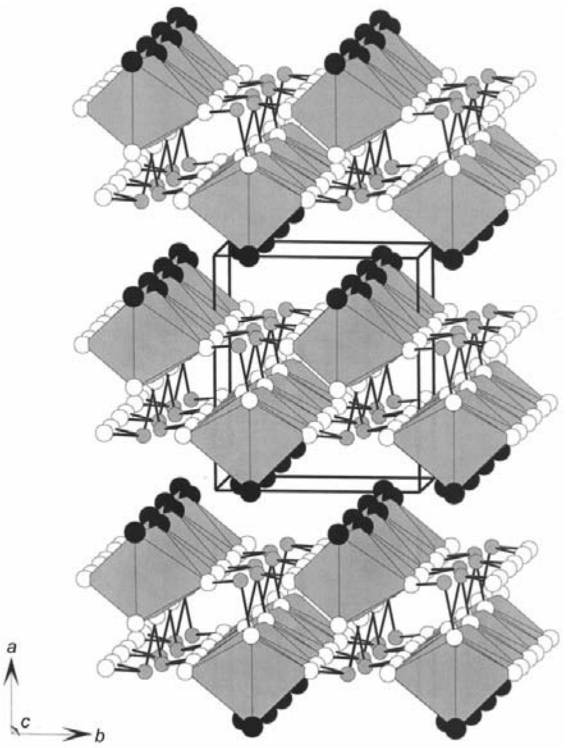

Figure 1
A representation of the $InTeO_3Cl$ structure along the *c* axis showing its layered nature. The $[InO_4Cl_2]$ octahedra are outlined. Grey, white and black circles represent Te, O and Cl atoms, respectively.

Among the family of metal tellurium oxyhalides, $InTeO_3Cl$ is an unusual case because of the lack of free Cl atoms between the layers. Indeed, the Cl atoms belong to the In coordination polyhedra, while in $SbTeO_3Cl$ and $NdTe_2O_5Cl$, for example, Sb and Nd are surrounded only by O atoms (Alonso, 1998; Nikiforov *et al.*, 1999). Moreover, an orthorhombic symmetry is generally observed for this kind of compound, and the lowest symmetry observed for $InTeO_3Cl$ (monoclinic) is certainly due to the asymmetry of the In environment, with four O and two Cl atoms. The $InTeO_3Cl$ structure is closest to the layered structural type of FeOCl. Indeed, iron(III) is also in an octahedral coordination composed of four O and two Cl atoms, and the outer part of the slabs is built up with chlorine layers which belong to the metal coordination sphere. Thus, as in the case of FeOCl, some chemical reactions can be expected with $InTeO_3Cl$. If no intercalation chemistry seems to be possible, as the +2 oxidation state for the In is not stable, some grafting or pillaring reactions can be imagined.

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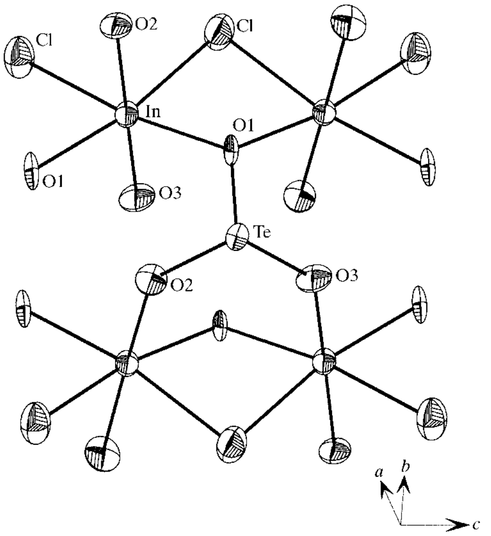

Figure 2
A view of the $[InO_4Cl_2]$ octahedra linked through $[TeO_3]$ trigonal pyramids. Displacement ellipsoids are shown at the 90% probability level.

Experimental

The title compound was obtained during the synthesis of $In_2Si_2O_7$ single crystals by chemical vapour transport, using $TeCl_4$ as the transport agent. In addition to the silicate crystals, which are interesting for their scintillation properties (Garcia *et al.*, 1995), very thin platelets were recovered in the cooler zone. Chemical analysis by electron probe microanalysis revealed a new compound containing In, Te, Cl and O in the ratio 1:1:1:3. Direct synthesis of $InTeO_3Cl$ from a stoichiometric mixture of In_2O_3, $InCl_3$ and TeO_2 heated at 723 K in an evacuated silica tube for 15 h was successful. The experimental density of 5.34 Mg m^{-3} was measured by the hydrostatic pressure method (Rabardel *et al.*, 1971). Because of the lamellar shape of the $InTeO_3Cl$ crystals, it was very difficult to find a real single crystal. Eventually, a crystal was cleaved, yielding a good single crystal.

Crystal data

$InTeO_3Cl$
$M_r = 325.87$
Monoclinic, $P2_1/c$
$a = 8.2596$ (4) Å
$b = 6.8752$ (3) Å
$c = 7.1394$ (3) Å
$\beta = 103.121$ (2)°
$V = 394.84$ (3) Å^3
$Z = 4$
$D_x = 5.48$ Mg m^{-3}
$D_m = 5.34$ Mg m^{-3}
D_m measured hydrostatically
Mo $K\alpha$ radiation
Cell parameters from 12 538 reflections
$\theta = 2.5$–35.0°
$\mu = 13.74$ mm^{-1}
$T = 293$ K
Plate, pale yellow
0.40 × 0.16 × 0.04 mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
CCD scans
Absorption correction: Gaussian (Templeton & Templeton, 1978)
$T_{min} = 0.064$, $T_{max} = 0.610$
6506 measured reflections
1128 independent reflections
944 reflections with $I > 2\sigma(I)$
$R_{int} = 0.100$
$\theta_{max} = 29.8°$
$h = -11 \rightarrow 11$
$k = -9 \rightarrow 9$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R(F) = 0.037$
$wR(F^2) = 0.093$
$S = 1.30$
1128 reflections
56 parameters
$w = 1/(\sigma^2 I + 0.0004I^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.18$ e Å^{-3}
$\Delta\rho_{min} = -2.38$ e Å^{-3}
Extinction correction: Becker & Coppens (1974), type II
Extinction coefficient: 0.07 (2)

Table 1
Selected geometric parameters (Å, °).

$In-Cl^{i}$	2.592 (2)	$Te-O1^{v}$	1.897 (5)
$In-Cl^{ii}$	2.577 (2)	$Te-O2$	1.907 (4)
$In-O1^{iii}$	2.174 (5)	$Te-O2^{vi}$	2.586 (4)
$In-O1^{iv}$	2.176 (4)	$Te-O3^{v}$	1.905 (4)
$In-O2^{ii}$	2.109 (4)	$Te-O3^{vi}$	2.586 (4)
$In-O3$	2.111 (4)		
$Cl^{i}-In-Cl^{ii}$	88.69 (7)	$O1^{iv}-In-O3$	92.23 (16)
$Cl^{i}-In-O1^{iii}$	79.65 (14)	$O2^{ii}-In-O3$	175.4 (2)
$Cl^{i}-In-O1^{iv}$	168.59 (17)	$O1^{v}-Te-O2$	92.0 (2)
$Cl^{i}-In-O2^{ii}$	88.23 (13)	$O1^{v}-Te-O2^{vi}$	78.9 (2)
$Cl^{i}-In-O3$	88.44 (13)	$O1^{v}-Te-O3^{v}$	92.7 (2)
$Cl^{ii}-In-O1^{iii}$	168.27 (13)	$O1^{v}-Te-O3^{vi}$	79.50 (18)
$Cl^{ii}-In-O1^{iv}$	79.96 (16)	$O2-Te-O2^{vi}$	169.9 (2)
$Cl^{ii}-In-O2^{ii}$	89.13 (17)	$O2-Te-O3^{v}$	101.9 (2)
$Cl^{ii}-In-O3$	87.69 (17)	$O2-Te-O3^{vi}$	74.43 (17)
$O1^{iii}-In-O1^{iv}$	111.7 (2)	$O2^{vi}-Te-O2$	169.9 (2)
$O1^{iii}-In-O2^{ii}$	91.82 (19)	$O2^{vi}-Te-O3^{v}$	74.45 (16)
$O1^{iii}-In-O3$	90.6 (2)	$O2^{vi}-Te-O3^{vi}$	107.82 (14)
$O1^{iv}-In-O2^{ii}$	90.42 (16)	$O3^{v}-Te-O3^{vi}$	171.1 (2)

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

Data collection: *KappaCCD Software* (Nonius, 1999); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *JANA*2000 (Petricek & Dusek, 2000); program(s) used to refine structure: *JANA*2000; software used to prepare material for publication: *JANA*2000.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1338). Services for accessing these data are described at the back of the journal.

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