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Structure of Thallium(I) Iodate

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Abstract. $TlIO_3$, $M_r = 379{\cdot}3$, rhombohedral, $R3m$, $a = 6{\cdot}352\,(3)$, $c = 7{\cdot}945\,(1)$ Å, $V = 277{\cdot}6\,(3)$ Å^3, $Z = 3$, $D_x = 6{\cdot}806$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 502$ cm^{-1}, $F(000) = 474$, $T = 295$ K. Final $R(F) = 0{\cdot}025$ for 202 independent observed reflections. The iodate ion has an I—O bond distance of 1·820 (6) Å and a bond angle of 100·2 (3)°. The Tl···O distances are 3·214 (6), 3·485 (6) and 3·016 (6) Å and the I···O contacts are 2·735 (6) Å.

Experimental. Thallium(I) iodate crystals prepared as previously described (Bergman & Wood, 1976). Diffractometer used: Enraf–Nonius CAD-4 equipped with graphite monochromator. Cell dimensions: from 24 reflections in range $20 < 2\theta \leq 56°$. Crystal dimensions: 0·165 × 0·072 × 0·185 mm. Prismatic crystal bounded by 15 faces having the forms $\{110\}$, $\{\bar{1}\bar{1}0\}$, $\{10\bar{2}\}$, $\{01\bar{1}\}$ and $\{0\bar{1}1\}$ (hexagonal indices). Absorption corrections: analytical method (de Meulenaer & Tompa, 1965), max. and min. transmission factors 0·113 and 0·015. Intensity measurements: $\omega/2\theta$ step scans (96 steps) in octants hkl, $h\bar{k}l$, $\bar{h}kl$ and $\bar{h}\bar{k}l$ in range $(\sin\theta)/\lambda < 0{\cdot}85$ Å^{-1}. Max. scan time 102 s. Reflections recorded: 993 total, 202 independent (h −10→10; k −10→10; l 0→13); internal agreement factors on averaging, $R(F^2) = 0{\cdot}057$, $wR = 0{\cdot}062$. Five standards measured every 50 reflections, max. variation 1·5% from mean. Step scans analyzed using *PROFILE* (Blessing, Coppens & Becker, 1974). Standard deviations assigned as $\sigma(I) = [\sigma^2(I)_{\text{count}} + (0{\cdot}03I)^2]^{1/2}$ where $\sigma(F^2)_{\text{count}}$ is based on counting statistics. Refinement carried out in $R3m$ with initial coordinates $0,0,\frac{1}{2}$ for I, $\frac{1}{6},\frac{1}{3},\frac{1}{3}$ for O and Tl origin defining. Least-squares refinement (based on F) carried out using *LINEX*, a modified version of *ORFLS* (Busing, Martin & Levy, 1962); function minimized $\sum w(|F_o| - |F_c|)^2$ with weights $w = [2\text{Lp}F_o/\sigma(I)]^2$. All 202 independent observations used. Refinement over a total of 13 variables included an isotropic extinction parameter for a type II crystal with a Lorentzian distribution of mosaicity (Becker & Coppens, 1974). Attempts to refine extinction anisotropically led to no additional improvement. Final $R = 0{\cdot}025$, $wR = 0{\cdot}026$ and $S = 1{\cdot}21$. At convergence Δ/σ max. = 0·05. Largest extinction correction $1{\cdot}61F_o$, eight reflections having values greater than $1{\cdot}10F_o$. Atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); anomalous-dispersion corrections for Tl and I included (Cromer & Liberman, 1970). Calculations carried out on a CDC Cyber 175 computer. Atomic coordinates and thermal parameters are listed in Table 1.† An *ORTEP* view (Johnson, 1965) of the structure is given in Fig. 1.

Related literature. Structures of $LiIO_3$ (Svensonn, Albertsson, Liminga, Kvick & Abrahams, 1983; Coquet, Crettez, Pannetier, Bouillot & Damien, 1983), KIO_3 (Lucas, 1984) and an early study of $TlIO_3$ (Rivoir & Abbad, 1947).

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^4) *for* $TlIO_3$

	x	y	z	U_{eq}*
Tl	0	0	0	256 (4)
I	0	0	4953 (1)	109 (2)
O	1465 (7)	2930	3890 (8)	331 (36)

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

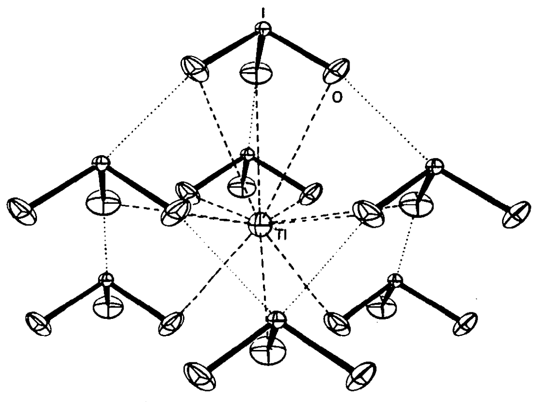

Fig. 1. An *ORTEP* (Johnson, 1965) perspective view of the packing arrangement in $TlIO_3$. Broken lines indicate Tl···O contacts and dotted lines I···O contacts.

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† Lists of anisotropic thermal parameters and observed and calculated structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44028 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Dimer of Dichlorodioxobis(tetrahydrofuran)uranium(VI)

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Abstract. $[UO_2Cl_2(C_4H_8O)_2]$, $M_r = 485{\cdot}1$, orthorhombic, *Pnma*, $a = 23{\cdot}585$ (7), $b = 9{\cdot}602$ (2), $c = 12{\cdot}377$ (3) Å, $V = 2803$ (2) Å^3, $Z = 8$ (unit formulae), $D_x = 2{\cdot}30$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71073$ Å, $\mu = 11{\cdot}36$ mm^{-1}, $F(000) = 1712$, $T = 295$ K. Final $R = 0{\cdot}038$ for 470 observed reflections. Di-μ-Cl-$[UO_2Cl_2(THF)_2]_2$ dimeric units exist (THF = tetrahydrofuran), in which each U atom is seven-coordinated in a pentagonal-bipyramidal geometry. The two independent U atoms and their eight equatorial atoms (four O of the THF molecules and four Cl atoms), all on special positions (*m*) of the *Pnma* space group, are coplanar. The linear UO_2^{2+} ions and the four planar THF groups lie perpendicularly to this plane. The U(1)···U(2) distance is 4·619 (5) Å, the U—Cl distances range from 2·65 (2) to 2·83 (2) Å (Cl atoms shared by U atoms) and the average U—O_{THF} distance is 2·40 (3) Å.

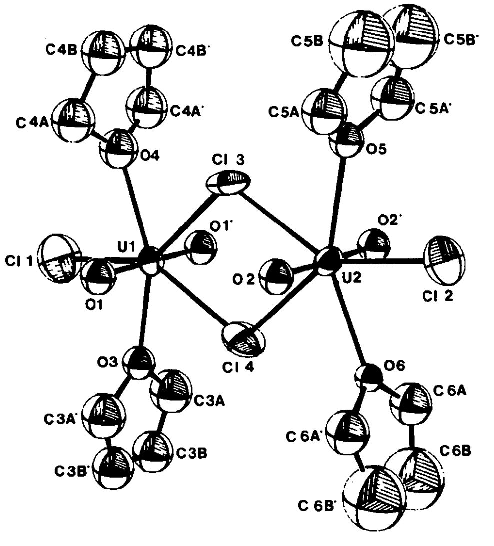

Fig. 1. Drawing of the title compound showing the atom numbering (primed atoms are related by the symmetry plane at $y = \frac{1}{4}$).

Table 1. *Positional and isotropic thermal parameters and their e.s.d.'s*

	x	*y*	*z*	*B*(Å^2)
U(1)	0·2093 (1)	0·250	0·5040 (2)	3·03 (8)*
Cl(1)	0·099 (1)	0·250	0·453 (2)	3·4 (8)*
O(1)	0·209 (1)	0·434 (7)	0·504 (3)	4·5 (7)
O(3)	0·206 (2)	0·250	0·317 (3)	5 (1)
C(3*A*)	0·212 (3)	0·368 (6)	0·242 (5)	8 (2)
C(3*B*)	0·210 (3)	0·321 (5)	0·134 (4)	9 (2)
O(4)	0·160 (2)	0·250	0·679 (4)	5 (1)
C(4*A*)	0·146 (2)	0·377 (5)	0·739 (4)	7 (1)
C(4*B*)	0·160 (2)	0·331 (5)	0·854 (4)	7 (2)
Cl(3)	0·286 (1)	0·250	0·679 (1)	4·7 (6)*
Cl(4)	0·321 (1)	0·250	0·427 (1)	6·1 (7)*
U(2)	0·3982 (2)	0·250	0·6027 (2)	3·12 (8)*
O(2)	0·397 (1)	0·431 (3)	0·602 (2)	3·5 (6)
Cl(2)	0·506 (1)	0·250	0·659 (2)	6·0 (7)*
O(5)	0·402 (2)	0·250	0·804 (3)	4 (1)
C(5*A*)	0·409 (2)	0·376 (5)	0·862 (4)	6 (1)
C(5*B*)	0·423 (3)	0·331 (7)	0·972 (6)	14 (3)
O(6)	0·450 (2)	0·250	0·440 (3)	3 (1)
O(6*A*)	0·469 (2)	0·367 (5)	0·377 (4)	7 (2)
O(6*B*)	0·494 (3)	0·323 (7)	0·273 (6)	14 (3)

$* B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

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