Orthorhombic
Immm
$a = 4.7034\,(10)$ Å
$b = 7.1735\,(13)$ Å
$c = 15.643\,(2)$ Å
$V = 527.78\,(16)$ Å^3
$Z = 2$
$D_x = 4.654$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 10.48$–$21.33°$
$\mu = 23.43$ mm^{-1}
$T = 293\,(2)$ K
Wedge
0.08 × 0.07 × 0.05 mm
Silver/light grey

Data collection

Nonius CAD-4 diffractometer
$\omega-\frac{1}{3}\theta$ scans
Absorption correction: numerical (Sheldrick, 1976)
$T_{\min} = 0.176$, $T_{\max} = 0.332$
463 measured reflections
463 independent reflections

430 reflections with $I > 3\sigma(I)$
$\theta_{\max} = 29.89°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 21$
3 standard reflections every 100 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.107$
$S = 1.217$
463 reflections
23 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 15.1135P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.031$

$\Delta\rho_{\max} = 2.651$ e Å^{-3}
$\Delta\rho_{\min} = -1.776$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0045 (8)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^ia^j\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
Ba1	0	0	0	0.0081 (3)
Ba2	0	1/2	0.13150 (4)	0.0094 (3)
As1	1/2	−0.32666 (17)	0	0.0073 (3)
As2	0	0	0.21219 (7)	0.0082 (3)
Li	1/2	−0.199 (3)	−0.1789 (11)	0.019 (3)

Table 2. *Selected bond lengths (Å)*

Ba1—As2	3.3191 (13)	Ba2—As1$^{\rm ii}$	3.3627 (7)
Ba1—As2$^{\rm i}$	3.3191 (13)	Ba2—As2$^{\rm x}$	3.3925 (10)
Ba1—As1$^{\rm ii}$	3.3199 (10)	Ba2—As2$^{\rm xi}$	3.3925 (10)
Ba1—As1$^{\rm iii}$	3.3199 (10)	As1—As1$^{\rm xii}$	2.487 (2)
Ba1—As1	3.3199 (10)	As1—Li$^{\rm xiii}$	2.944 (19)
Ba1—As1$^{\rm i}$	3.3199 (10)	As1—Li	2.944 (19)
Ba2—Li$^{\rm iv}$	3.277 (15)	As2—Li$^{\rm vii}$	2.751 (18)
Ba2—Li$^{\rm i}$	3.277 (15)	As2—Li$^{\rm xiv}$	2.751 (18)
Ba2—Li$^{\rm v}$	3.277 (15)	As2—Li$^{\rm xv}$	2.800 (12)
Ba2—Li$^{\rm ii}$	3.277 (15)	As2—Li$^{\rm ii}$	2.800 (12)
Ba2—Li$^{\rm vi}$	3.292 (19)	As2—Li$^{\rm i}$	2.800 (12)
Ba2—Li$^{\rm vii}$	3.292 (19)	As2—Li$^{\rm xiii}$	2.800 (12)
Ba2—As1$^{\rm i}$	3.3627 (7)	Li—Ba2$^{\rm i}$	3.277 (15)
Ba2—As1$^{\rm viii}$	3.3627 (7)	Li—Ba2$^{\rm ii}$	3.277 (15)
Ba2—As1$^{\rm ix}$	3.3627 (7)	Li—Ba2$^{\rm xvi}$	3.292 (19)

Symmetry codes: (i) $-x, -y, -z$; (ii) $1-x, -y, -z$; (iii) $x-1, y, z$; (iv) $1-x, 1+y, -z$; (v) $-x, 1+y, -z$; (vi) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z$; (vii) $x-\frac{1}{2}, \frac{1}{2}+y, \frac{1}{2}+z$; (viii) $x-1, 1+y, z$; (ix) $x, 1+y, z$; (x) $\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}-z$; (xi) $-\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}-z$; (xii) $1-x, -1-y, -z$; (xiii) $1-x, y, -z$; (xiv) $x-\frac{1}{2}, -\frac{1}{2}-y, \frac{1}{2}+z$; (xv) $-x, y, -z$; (xvi) $\frac{1}{2}+x, y-\frac{1}{2}, z-\frac{1}{2}$.

The parameters and crystallographic space group were initially determined by oscillation and Weissenberg techniques. The space group was found to be *Immm* (No. 71) and no violation of the I-centering conditions ($h+k+l=2n$) was observed.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: local program. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ATOMS* (Dowty, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1246). Services for accessing these data are described at the back of the journal.

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Thallium titanium phosphate, $Tl_3Ti_3O(PO_4)_3(P_2O_7)$

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Abstract

The title compound, trithallium trititanium oxide tris(phosphate) diphosphate, consists of a three-dimensional network of vertex-sharing TiO_6, PO_4 and P_2O_7 groups [$d_{\rm av}$(Ti—O) = 1.940 (3) and $d_{\rm av}$(P—O) = 1.536 (4) Å]. Extra-framework Tl^+ cations [$d_{\rm av}$(Tl—O) = 3.090 (7) Å] complete the structure, which is isostructural with $M_3Ti_3O(PO_4)_3(P_2O_7)$ (M = K, Rb).

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Comment

$Tl_3Ti_3O(PO_4)_3(P_2O_7)$ (Fig. 1) is isostructural with $K_3Ti_3O(PO_4)_3(P_2O_7)$ (Nagornyi & Kapshuk, 1993) and $Rb_3Ti_3O(PO_4)_3(P_2O_7)$ (Harrison *et al.*, 1994; Duhlev, 1994). These phases are built up from a three-dimensional network of vertex-sharing TiO_6, PO_4 and P_2O_7 groups, connected *via* Ti—O—Ti and Ti—O—P bonds. Extra-framework univalent cations complete the structure. Polyhedral '3-rings' (one tetrahedron and two octahedra) and '5-rings' (two tetrahedra and three octahedra) are apparent (Fig. 1). These $M_3Ti_3O(PO_4)_3(P_2O_7)$ phases are of interest for their relationship to the technologically important MTiOPO$_4$ [M = K (Tordjman *et al.*, 1974), Rb (Thomas *et al.*, 1992) and Tl (Harrison *et al.*, 1995)] family of phases (Stucky *et al.*, 1989).

The Tl1 [d_{av}(Tl1—O) = 3.121 (7) Å] and Tl2 [d_{av}(Tl2—O) = 3.091 (7) Å] atoms are nine-coordinate to O atoms, and Tl3 [d_{av}(Tl3—O) = 3.055 (7) Å] is eight-coordinate, assuming a maximum Tl—O contact of 3.4 Å in each case. All three Tl coordination polyhedra are irregular, and the stereochemical role of the Tl lone pair, if any, is hard to gauge. Considered together, the TlO_8/TlO_9 polyhedra link together through vertex-, edge- and face-sharing into a three-dimensional network. The Rb^+ ions in $Rb_3Ti_3O(PO_4)_3(P_2O_7)$ occupy very similar sites (within ~0.1 Å) to those of the Tl^+ species in the title compound.

The three distinct TiO_6 octahedra [d_{av}(Ti1—O) = 1.943 (3), d_{av}(Ti2—O) = 1.936 (4) and d_{av}(Ti3—O) = 1.940 (4) Å] are somewhat distorted. However, the displacements (Δ_{oct}) of the Ti^{IV} species from the best centres (Balić Žunić & Makovicky, 1996) of their octahedra [Δ_{oct}(Ti1) = 0.05 (1), Δ_{oct}(Ti2) = 0.10 (1) and Δ_{oct}(Ti3) = 0.11 (1) Å] are significantly smaller than the typical displacements observed for the Ti species (Δ_{oct} ~0.3 Å) in the MTiOPO$_4$ (M = K, Rb, Tl) phases, where Ti=O double bonds are considered to occur.

There are three phosphate groups (central atoms P1, P2 and P3) and a pyrophosphate group (P4 and P5 linked by O18) in this phase. The tetrahedral geometries of the phosphate species are unexceptional [d_{av}(P1—O) = 1.536 (4), d_{av}(P2—O) = 1.532 (5) and d_{av}(P3—O) = 1.530 (4) Å]. The P—O bonds involved in the pyrophosphate bridge show their typical lengthening relative to the other pyrophosphate P—O bonds [d_{av}(P4—O) = 1.540 (4) and d_{av}(P5—O) = 1.541 (5) Å] (Durif, 1995). The P_2O_7 group in $Tl_3Ti_3O(PO_4)_3(P_2O_7)$ is almost perfectly eclipsed, with a twist angle (Brown & Calvo, 1970) of 2.5°. Atoms P4, P5, O19 and O20 are planar to within 0.01 Å. The P—O—P bond angle of 134.9 (4)° and the P···P separation of 2.977 (4) Å both fall within the range of values summarized for similar species by Durif (1995).

Of the 20 O atoms in the structure, one forms a Ti2—O9—Ti3 bond, one a P4—O18—P5 bond, and 16 form Ti—O—P links (11 link Ti to a phosphate group and five link Ti to the pyrophosphate group), with θ_{av}(Ti—O—P) = 150.4 (5)°. Three of the Ti—O—P links (*via* O3,

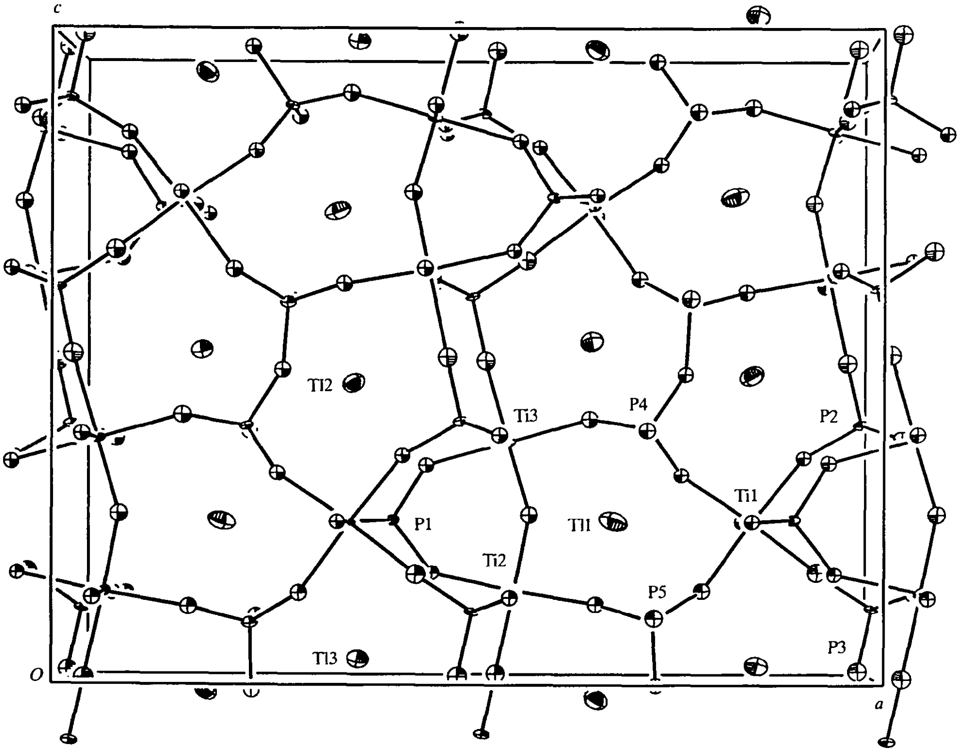

Fig. 1. View of $Tl_3Ti_3O(PO_4)_3(P_2O_7)$ down [010]. Ellipsoids are at the 50% probability level.

O8 and O14) are close to being linear. The remaining two O atoms, O19 and O20, form short terminal P—O links. All the O atoms, except O1, O3 and O16, also bond to one or more Tl^+ cations. The short P4—O20 and P5—O19 bonds link to all three Tl species.

The polyhedral connectivity in this phase is the same as in $Rb_3Ti_3O(PO_4)_3(P_2O_7)$ (Harrison *et al.*, 1994). The most important structural features are the three-dimensional network of vertex-sharing octahedra and tetrahedra, and the resulting [010] channels occupied by the univalent cations. Further work will be required to determine if these species show mobility in these channels, akin to the situation of the highly mobile guest cations in the $KTiOPO_4$ isomorphs (Thomas *et al.*, 1992).

Experimental

Single crystals of $Tl_3Ti_3O(PO_4)_3(P_2O_7)$ were prepared from 85% H_3PO_4 (11.76 g), TiO_2 (2.75 g) and Tl_2CO_3 (35 g) (starting ratio of Tl:Ti:P $\simeq$ 4:1:4). These components were warmed in a platinum crucible to remove water and CO_2, then heated to 1173 K in a muffle furnace. After soaking for 12 h, the furnace was cooled slowly to 800 K over a period of 24 h, and then to room temperature over a period of a few hours. Transparent rod-like crystals of the title compound were separated from the yellowish flux by washing in hot water.

Crystal data

$Tl_3Ti_3O(PO_4)_3(P_2O_7)$
$M_r = 1231.66$
Orthorhombic
$Pca2_1$
$a = 18.2534 (9)$ Å
$b = 6.3271 (3)$ Å
$c = 14.7428 (7)$ Å
$V = 1702.7 (2)$ Å^3
$Z = 4$
$D_x = 4.81$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8192 reflections
$\theta = 2.5–28.5°$
$\mu = 30.21$ mm^{-1}
$T = 293 (2)$ K
Rod
0.30 × 0.02 × 0.02 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
Area-detector scans
Absorption correction: empirical (*SADABS* in *SMART*; Bruker, 1997)
$T_{min} = 0.166$, $T_{max} = 0.694$
9849 measured reflections
2254 independent reflections (plus 1405 Friedel-related reflections)

3405 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 28.53°$
$h = -24 \rightarrow 21$
$k = -8 \rightarrow 6$
$l = -18 \rightarrow 18$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.044$

Extinction correction: *SHELXL97*
Extinction coefficient: 0.00101 (7)

3659 reflections
182 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 6.0577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 3.12$ e Å^{-3}
$\Delta\rho_{min} = -2.34$ e Å^{-3}

Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.00 (7)

Table 1. *Selected geometric parameters (Å, °)*

Tl1—O20	2.722 (8)	Ti2—O9	1.815 (9)
Tl1—O19	2.859 (8)	Ti2—O11	1.919 (8)
Tl1—O12^{i}	2.959 (7)	Ti2—O10	1.930 (7)
Tl1—O2^{i}	3.119 (5)	Ti2—O7	1.963 (8)
Tl1—O6^{i}	3.229 (7)	Ti2—O12	1.972 (8)
Tl1—O17^{i}	3.279 (8)	Ti2—O8	2.015 (8)
Tl1—O10^{i}	3.288 (8)	Ti3—O9	1.801 (9)
Tl1—O9^{i}	3.294 (6)	Ti3—O16	1.912 (9)
Tl1—O11	3.338 (7)	Ti3—O15	1.964 (8)
Tl2—O13	2.722 (8)	Ti3—O17	1.965 (8)
Tl2—O19^{ii}	2.813 (8)	Ti3—O13	1.987 (8)
Tl2—O18^{iii}	3.021 (6)	Ti3—O14	2.014 (8)
Tl2—O12^{iii}	3.064 (7)	P1—O13	1.529 (8)
Tl2—O20^{iv}	3.086 (8)	P1—O1^{iv}	1.534 (5)
Tl2—O14	3.213 (9)	P1—O7	1.536 (8)
Tl2—O10^{iii}	3.219 (8)	P1—O2^{v}	1.545 (5)
Tl2—O4^{v}	3.305 (7)	P2—O8^{viii}	1.503 (8)
Tl2—O2^{v}	3.380 (12)	P2—O4	1.537 (8)
Tl3—O7	2.805 (8)	P2—O17^{ix}	1.543 (9)
Tl3—O20^{vi}	2.878 (8)	P2—O16^{x}	1.545 (9)
Tl3—O17^{vii}	2.997 (8)	P3—O14^{xi}	1.517 (8)
Tl3—O19^{iv}	3.003 (8)	P3—O3	1.527 (8)
Tl3—O15^{vii}	3.008 (7)	P3—O11^{x}	1.534 (8)
Tl3—O5^{v}	3.119 (7)	P3—O12^{ix}	1.541 (8)
Tl3—O2^{v}	3.302 (12)	P4—O20	1.486 (9)
Tl3—O8	3.330 (9)	P4—O15	1.530 (8)
Ti1—O1	1.895 (5)	P4—O6	1.534 (7)
Ti1—O3	1.903 (8)	P4—O18^{viii}	1.609 (7)
Ti1—O4	1.941 (7)	P5—O19	1.492 (9)
Ti1—O6	1.971 (7)	P5—O10	1.525 (8)
Ti1—O5	1.972 (7)	P5—O5	1.534 (8)
Ti1—O2	1.973 (5)	P5—O18	1.614 (7)
P1^{x}—O1—Ti1	142.3 (4)	P5—O10—Ti2	149.8 (5)
P1^{ix}—O2—Ti1	137.2 (3)	P3^{iv}—O11—Ti2	143.9 (5)
P3—O3—Ti1	174.4 (5)	P3^{v}—O12—Ti2	143.3 (5)
P2—O4—Ti1	159.1 (5)	P1—O13—Ti3	136.9 (5)
P5—O5—Ti1	142.6 (5)	P3^{viii}—O14—Ti3	175.9 (6)
P4—O6—Ti1	144.0 (4)	P4—O15—Ti3	144.7 (5)
P1—O7—Ti2	138.9 (5)	P2^{iv}—O16—Ti3	148.8 (5)
P2^{xi}—O8—Ti2	179.0 (6)	P2^{v}—O17—Ti3	145.2 (5)
Ti3—O9—Ti2	153.2 (4)	P4^{xi}—O18—P5	134.9 (4)

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

Comparison of intensities extracted from the initial 75 frames with duplicates collected at the end of the experiment indicated that negligible crystal decay had occurred. The starting model was taken from the isostructural $Rb_3Ti_3O(PO_4)_3(P_2O_7)$ (Harrison *et al.*, 1994), with Tl substituting for Rb. The highest difference peak is 0.75 Å from Tl1 and the deepest difference hole is 0.74 Å from Tl3. The 15 most significant difference Fourier peaks/holes are all within 1.0 Å of one of the thallium species. Anisotropic refinement in the $Pca2_1$ space group was unsuccessful for the O atoms, with one or two refining to non-physical values, and several refining to an unreasonable degree of ellipsoidal elongation. $Tl_3Ti_3O(PO_4)_3(P_2O_7)$ is pseudo-centrosymmetric, as was also observed for $Rb_3Ti_3O(PO_4)_3(P_2O_7)$ (Harrison *et al.*, 1994; Duhlev, 1994). Refinements in the centrosymmetric space group *Pcam* (No. 57), which contains 2 × Tl, 2 × Ti, 3 × P and 12 × O in the asymmetric unit, led to much higher residuals of $R[F > 2\sigma(F)] = 0.067$ and $wR(F^2) = 0.180$. Very elongated ellipsoids resulted from two O atoms which form Ti—O—P(pyrophosphate) [principal axis $U_{\min}/U_{\max}$ ratio for this atom = 5.3] and P—O—P [$U_{\min}/U_{\max} = 9.7$] bonds. These *Pcam* species correspond to the O atoms involved in the Ti1—O5—P5, Ti1—O6—P4 and P4—O18—P5 bonds in the $Pca2_1$ model.

The geometries of the TiO_6 octahedra are relatively unaffected by the symmetry reduction from *Pcam* to $Pca2_1$. In particular, the conformation of the Ti—O—Ti bridge is barely altered when the twofold symmetry constraint on a bridging O atom is removed. In $Pca2_1$, the unusual situation where both the Ti atoms are displaced towards the central O atom remains essentially unchanged compared with that occurring in *Pcam*. This situation is totally different to that found in phases such as $KTiOPO_4$, containing infinite chains of vertex-sharing TiO_6 octahedra, where a very distinctive alternation of Ti—O bond lengths along the chain occurs (Stucky *et al.*, 1989; Thomas *et al.*, 1992).

The application of a symmetry check (Farrugia, 1998) revealed that the atom least compatible (by ~0.39 Å) with higher symmetry was O18, which forms the pyrophosphate P—O—P bridge. The P—O—P angle is reduced from 145 (2)° in *Pcam* to 134.9 (4)° in $Pca2_1$. In *Pcam*, the bridging O atom lies on a twofold axis, which restricts the value of the P—O—P angle, given that four of the terminal P—O bonds have their position constrained by Ti atoms. The P—O—P angles in pyrophosphate groups cover a wide range, ~120–180° (Durif, 1995), thus in itself, the driving force for a symmetry-breaking effect involving removal of the twofold axis of the P_2O_7 group is probably a relatively weak one (Brown, 1999). Another effect may be involved, namely, optimizing the Tl^+ cation coordinations. In *Pcam*, the Tl11 species (corresponding to Tl1 in $Pca2_1$) is relatively underbonded, with only six O-atom neighbours within 3.4 Å, and a bond valence sum (BVS) of 0.66 (Brown, 1996). Tl12 (corresponding to Tl2 and Tl3) has nine O-atom neighbours within the same limits (BVS = 0.79). In $Pca2_1$, Tl1 increases its coordination number to nine and its BVS to 0.80, Tl2 (nine-coordinate) increases its BVS to 0.86, whilst that for Tl3 (eight-coordinate) is essentially unchanged from that of Tl12 at 0.79. In *Pcam*, the P—O—P bridge O atom bonds to two equivalent Tl12 cations, with d(Tl—O) = 3.23 (2) Å. In $Pca2_1$, the two distinct Tl—O bond lengths are 3.021 (7) Å for Tl2 and 3.493 (7) Å for Tl3. In *Pcam*, Tl11 makes a very long bond of 3.47 (2) Å to the Ti—O—P(pyrophosphate) O atom noted above. In $Pca2_1$, Tl1—O6 is much shorter at 3.229 (7) Å, whereas Tl1—O5 is longer at 3.693 (6) Å. To a first approximation, these improved low-symmetry thallium coordinations are achieved by distorting the P_2O_7 group and breaking the mirror symmetry of *Pcam*. Diffraction measurements as a function of temperature would be required to further substantiate this explanation.

Data collection: *SMART* (Bruker, 1997). Cell refinement: *SMART*. Data reduction: *SMART*. Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*-3 (Farrugia, 1997). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1228). Services for accessing these data are described at the back of the journal.

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Zinc mercury thiocyanate (ZMTC)

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Abstract

The title complex, zinc mercury tetrathiocyanate, $[ZnHg(SCN)_4]_n$, consists of slightly flattened ZnN_4 and

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