syntheses. The effects of anomalous dispersion were included in F_c (Cromer & Ibers, 1974).

Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71226 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1038]

References

Abrahams, S. C. & Bernstein, J. L. (1967). *J. Chem. Phys.* **46**, 3776–3782.
Bertaut, E. F., Buisson, G., Durif, A., Mareschal, A., Montmory, M. C. & Quezel-Ambrunaz, S. (1965). *Bull. Soc. Chim. Fr.* pp. 1132–1137.
Buisson, G. (1973). *Phys. Status Solidi A*, **17**, 191–198.
Buisson, G. (1977). *Acta Cryst.* B**33**, 1031–1034.
Cromer, D. T. & Ibers, J. A. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Euzen, P., Léone, P., Palvadeau, P., Queignec, M. & Rouxel, J. (1992). *Mater. Res. Bull.* **27**, 1295–1300.
Euzen, P., Léone, P., Mansot, J. L., Bonneau, P., Queignec, M. & Palvadeau, P. (1992). *Mater. Res. Bull.* **27**, 1423–1430.
Euzen, P., Palvadeau, P., Queignec, M. & Rouxel, J. (1991). *C. R. Acad. Sci.* **312**, 367–372.
Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 4. Enraf–Nonius, Delft, The Netherlands.
Gardner, P. P., Wilkinson, C., Forsyth, J. B. & Wanklyn, B. M. (1988). *J. Phys. C*, **21**, 5653–5661.
Quezel-Ambrunaz, S., Bertaut, E. F. & Buisson, G. (1964). *C. R. Acad. Sci.* **258**, 3025–3028.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1993). C**49**, 1877–1879

Structure of $Tl_2Mo_2P_2O_{11}$

A. Guesdon, M. M. Borel, A. Grandin,
A. Leclaire and B. Raveau

Laboratoire CRISMAT, ISMRa,
Université de Caen, Boulevard du Maréchal Juin,
14050 Caen CEDEX, France

(*Received* 22 *December* 1992; *accepted* 29 *April* 1993)

Abstract

Dimolybdenum dithallium diphosphate, $Tl_2Mo_2P_2O_{11}$, is isotypic with $K_2Mo_2P_2O_{11}$. Its framework is built from the original $Mo_2P_2O_{15}$ unit, formed by one Mo_2O_{11} group sharing four of its corners with two PO_4 tetrahedra, leading to $[Mo_2P_2O_{13}]_\infty$ chains running along **c**. The $[Mo_2P_2O_{11}]_\infty$ framework forms two different intersecting tunnels running along **c** and ⟨110⟩, where the Tl^+ ions are located.

Comment

The synthesis and structure determination of the title compound were undertaken in order to test the possible replacement of potassium by thallium in the phase $K_2Mo_2P_2O_{11}$ (Gueho, Borel, Grandin, Leclaire & Raveau, 1993). The crystals of the new phase $Tl_2Mo_2P_2O_{11}$ were synthesized from the nominal composition $Tl_2Mo_2P_2O_{11}$ heated at 1123 K for one day in an evacuated silica ampoule and cooled at $2\ K\ h^{-1}$ to 923 K. Finally, the sample was quenched to room temperature and brown crystals were extracted. The phosphate $Tl_2Mo_2P_2O_{11}$ is isotypic with $K_2Mo_2P_2O_{11}$, *i.e.* its framework is built from $Mo_2P_2O_{15}$ units, formed by one Mo_2O_{11} group sharing four of its corners with two PO_4 tetrahedra along **c**, leading to $[Mo_2P_2O_{13}]_\infty$ chains running along **c**. Laterally in the (001) plane, the $[Mo_2P_2O_{13}]_\infty$ columns share the corners of their polyhedra leading to the three-dimensional framework $[Mo_2P_2O_{11}]_\infty$. This framework forms two different intersecting tunnels running along **c** and ⟨110⟩. Like the potassium cations in $K_2Mo_2P_2O_{11}$, the Tl cations are distributed over three kinds of sites. The Tl(1) cations are located on the symmetry centre and exhibit eightfold coordination with Tl—O distances ranging from 2.73 (1) to 2.90 (1) Å; they are strongly linked to the surrounding O atoms, in agreement with the value of their thermal factors (close to 1.60 Å^2). The Tl(2) cations located in the [001] tunnels are characterized by ninefold coordination with large Tl—O distances ranging from 2.80 (1) to 3.34 (1) Å. The Tl(3) cations

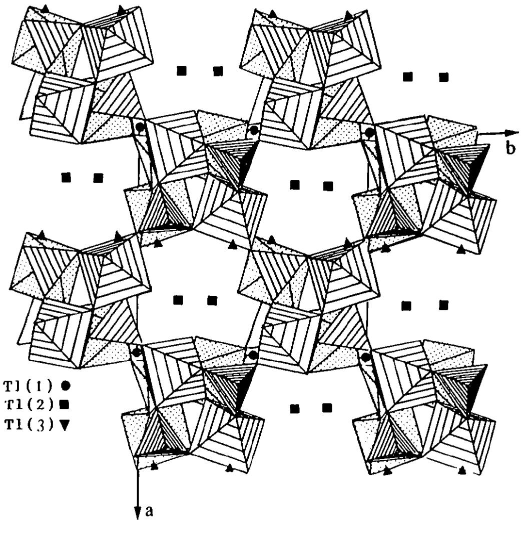

Fig. 1. Projection of $Tl_2Mo_2P_2O_{11}$ along [001].

located near the axis of the ⟨110⟩ tunnels sit in the [001] tunnels and are split over two positions with respect to the symmetry centre. This cation is characterized by sevenfold coordination with Tl—O distances ranging from 2.84 (1) to 3.26 (1) Å. The high values of the thermal factors of Tl(2) and Tl(3) (3.69 and 4.78 Å, respectively) are in agreement with the large Tl—O distances for these cations.

Experimental

Crystal data

$Mo_2Tl_2P_2O_{11}$
M_r = 838.6
Monoclinic
$P2_1/c$
a = 9.945 (1) Å
b = 10.156 (1) Å
c = 9.974 (2) Å
β = 97.64 (1)°
V = 998.5 Å^3
Z = 4
D_x = 5.58 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 18–22°
μ = 35.3 mm^{-1}
T = 294 K
Polyhedron
0.077 × 0.051 × 0.051 mm
Brown
Crystal source: solid-state synthesis

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-2/3θ scans of width (1 + 0.35tanθ)°
Absorption correction: analytical
$T_{\rm min}$ = 0.18009, $T_{\rm max}$ = 0.2665
8805 measured reflections
8805 independent reflections

1364 observed reflections [$I \geq 3\sigma(I)$]
$\theta_{\rm max}$ = 45°
h = −16 → 15
k = 0 → 16
l = 0 → 15
3 standard reflections
frequency: 50 min
intensity variation: <0.5%

Refinement

Refinement on F
Final R = 0.044
wR = 0.047
S = 1.001
1364 reflections
105 parameters
$w = F\sin(\theta/\lambda)$
$(\Delta/\sigma)_{\rm max} < 0.004$

$\Delta\rho_{\rm max}$ = 3.9 e Å^{-3}
$\Delta\rho_{\rm min}$ = −2 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{\rm eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}].$$

	x	y	z	$B_{\rm eq}$
Mo(1)	0.3448 (1)	0.0853 (2)	0.1325 (2)	0.67 (2)
Mo(2)	0.0775 (1)	0.3219 (2)	0.1467 (2)	0.71 (2)
P(1)	0.3962 (4)	0.3755 (5)	0.3154 (5)	0.87 (8)
P(2)	0.1260 (4)	0.0758 (5)	0.3535 (5)	0.69 (7)
Tl(1)	0.000	0.000	0.000	1.60 (2)
Tl(2)	0.7572 (1)	0.1776 (1)	0.2865 (1)	3.69 (2)
Tl(3)	0.5273 (3)	0.0798 (3)	0.5077 (2)	4.78 (5)
O(1)	0.287 (1)	−0.057 (1)	0.061 (1)	1.4 (2)*
O(2)	0.427 (1)	0.152 (1)	−0.033 (1)	1.1 (2)*
O(3)	0.269 (1)	0.054 (1)	0.317 (1)	1.2 (2)*
O(4)	0.526 (1)	0.001 (1)	0.221 (1)	1.0 (2)*
O(5)	0.192 (1)	0.192 (1)	0.095 (1)	0.8 (2)*
O(6)	0.443 (1)	0.258 (1)	0.238 (1)	0.7 (2)*
O(7)	−0.073 (1)	0.265 (1)	0.066 (1)	1.3 (2)*
O(8)	−0.024 (1)	0.480 (1)	0.225 (1)	1.1 (2)*
O(9)	0.135 (1)	0.449 (1)	0.006 (1)	0.9 (2)*
O(10)	0.072 (1)	0.216 (1)	0.320 (1)	1.0 (2)*
O(11)	0.247 (1)	0.408 (1)	0.272 (1)	1.1 (2)*

* Atoms refined isotropically.

Table 2. *Geometric parameters* (Å, °)

Mo(1)—O(1)	1.68 (1)	O(7)—O(11)	3.83 (2)
Mo(1)—O(2)	2.05 (1)	O(8)—O(9)	2.87 (2)
Mo(1)—O(3)	2.10 (1)	O(8)—O(10)	2.96 (2)
Mo(1)—O(4)	2.08 (1)	O(8)—O(11)	2.77 (2)
Mo(1)—O(5)	1.87 (1)	O(9)—O(10)	4.04 (2)
Mo(1)—O(6)	2.20 (1)	O(9)—O(11)	2.77 (2)
Mo(2)—O(5)	1.86 (1)	O(10)—O(11)	2.71 (2)
Mo(2)—O(7)	1.70 (1)	O(2$^{\rm i}$)—O(4$^{\rm ii}$)	2.53 (2)
Mo(2)—O(8)	2.10 (1)	O(2$^{\rm i}$)—O(6)	2.49 (2)
Mo(2)—O(9)	2.04 (1)	O(2$^{\rm i}$)—O(11)	2.54 (2)
Mo(2)—O(10)	2.05 (1)	O(4$^{\rm ii}$)—O(6)	2.52 (2)
Mo(2)—O(11)	2.15 (1)	O(4$^{\rm ii}$)—O(11)	2.44 (2)
P(1)—O(2$^{\rm i}$)	1.53 (2)	O(6)—O(11)	2.53 (2)
P(1)—O(4$^{\rm ii}$)	1.56 (1)	O(3)—O(9$^{\rm i}$)	2.46 (2)
P(1)—O(6)	1.53 (1)	O(3)—O(8$^{\rm iii}$)	2.53 (2)
P(1)—O(11)	1.52 (1)	O(3)—O(10)	2.57 (2)
P(2)—O(3)	1.53 (1)	O(8$^{\rm iii}$)—O(9$^{\rm i}$)	2.52 (2)
P(2)—O(8$^{\rm iii}$)	1.54 (1)	O(8$^{\rm iii}$)—O(10)	2.47 (2)
P(2)—O(9$^{\rm i}$)	1.54 (1)	O(9$^{\rm i}$)—O(10)	2.52 (2)
P(2)—O(10)	1.54 (1)	Tl(1)—O(1$^{\rm iv}$)	2.90 (1)
O(1)—O(2)	2.77 (2)	Tl(1)—O(1)	2.90 (1)
O(1)—O(3)	2.81 (2)	Tl(1)—O(5$^{\rm iv}$)	2.81 (1)
O(1)—O(4)	2.74 (2)	Tl(1)—O(5)	2.81 (1)
O(1)—O(5)	2.74 (2)	Tl(1)—O(7)	2.89 (1)
O(1)—O(6)	3.87 (2)	Tl(1)—O(7$^{\rm iv}$)	2.89 (1)
O(2)—O(3)	4.13 (2)	Tl(1)—O(8$^{\rm v}$)	2.73 (1)
O(2)—O(4)	3.01 (2)	Tl(1)—O(8$^{\rm iii}$)	2.73 (1)
O(2)—O(5)	2.83 (2)	Tl(2)—O(1$^{\rm ii}$)	3.16 (1)
O(2)—O(6)	2.89 (2)	Tl(2)—O(4)	2.93 (1)
O(3)—O(4)	2.89 (2)	Tl(2)—O(6)	3.21 (1)
O(3)—O(5)	2.65 (2)	Tl(2)—O(7$^{\rm vi}$)	3.08 (1)
O(3)—O(6)	2.87 (2)	Tl(2)—O(7$^{\rm viii}$)	3.12 (1)
O(4)—O(5)	3.91 (2)	Tl(2)—O(8$^{\rm ix}$)	3.34 (1)
O(4)—O(6)	2.75 (2)	Tl(2)—O(9$^{\rm ix}$)	3.20 (1)
O(5)—O(6)	2.78 (2)	Tl(2)—O(10$^{\rm vi}$)	3.12 (1)
O(5)—O(7)	2.71 (2)	Tl(2)—O(11$^{\rm ix}$)	2.80 (1)
O(5)—O(8)	3.95 (2)	Tl(3)—O(2$^{\rm i}$)	2.91 (1)
O(5)—O(9)	2.79 (2)	Tl(3)—O(3$^{\rm vi}$)	2.84 (1)
O(5)—O(10)	2.69 (2)	Tl(3)—O(3)	3.00 (1)
O(5)—O(11)	2.82 (2)	Tl(3)—O(4$^{\rm vii}$)	2.94 (1)
O(7)—O(8)	2.70 (2)	Tl(3)—O(4)	2.97 (1)
O(7)—O(9)	2.90 (2)	Tl(3)—O(6)	3.26 (1)
O(7)—O(10)	2.79 (2)	Tl(3)—O(6$^{\rm i}$)	3.04 (1)
O(1)—Mo(1)—O(2)	9.54 (6)	O(7)—Mo(2)—O(9)	101.4 (6)
O(1)—Mo(1)—O(3)	95.8 (6)	O(7)—Mo(2)—O(10)	95.7 (6)
O(1)—Mo(1)—O(4)	92.9 (6)	O(7)—Mo(2)—O(11)	170.4 (6)
O(1)—Mo(1)—O(5)	101.2 (6)	O(8)—Mo(2)—O(9)	87.8 (5)
O(1)—Mo(1)—O(6)	172.8 (6)	O(8)—Mo(2)—O(10)	91.1 (5)
O(2)—Mo(1)—O(3)	168.7 (5)	O(8)—Mo(2)—O(11)	81.5 (5)
O(2)—Mo(1)—O(4)	93.7 (5)	O(9)—Mo(2)—O(10)	162.8 (5)
O(2)—Mo(1)—O(5)	92.6 (5)	O(9)—Mo(2)—O(11)	82.6 (5)
O(2)—Mo(1)—O(6)	85.5 (5)	O(10)—Mo(2)—O(11)	80.3 (5)
O(3)—Mo(1)—O(4)	87.6 (5)	O(2$^{\rm i}$)—P(1)—O(4$^{\rm ii}$)	109.9 (7)
O(3)—Mo(1)—O(5)	83.5 (5)	O(2$^{\rm i}$)—P(1)—O(6)	108.8 (7)
O(3)—Mo(1)—O(6)	83.7 (5)	O(2$^{\rm i}$)—P(1)—O(11)	112.4 (8)
O(4)—Mo(1)—O(5)	164.0 (5)	O(4$^{\rm ii}$)—P(1)—O(6)	109.2 (7)
O(4)—Mo(1)—O(6)	79.9 (5)	O(4$^{\rm ii}$)—P(1)—O(11)	104.6 (7)
O(5)—Mo(1)—O(6)	86.0 (5)	O(6)—P(1)—O(11)	111.9 (7)
O(5)—Mo(2)—O(7)	99.4 (6)	O(3)—P(2)—O(8$^{\rm iii}$)	111.0 (7)
O(5)—Mo(2)—O(8)	170.7 (5)	O(3)—P(2)—O(9$^{\rm i}$)	106.4 (7)
O(5)—Mo(2)—O(9)	91.2 (5)	O(3)—P(2)—O(10)	113.1 (7)
O(5)—Mo(2)—O(10)	87.2 (5)	O(8$^{\rm iii}$)—P(2)—O(9$^{\rm i}$)	110.3 (7)
O(5)—Mo(2)—O(11)	89.2 (5)	O(8$^{\rm iii}$)—P(2)—O(10)	106.5 (7)
O(7)—Mo(2)—O(8)	89.9 (6)	O(9$^{\rm i}$)—P(2)—O(10)	109.7 (7)

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

Table 3. *Refined displacement parameters* (Å^2)

The form of the anisotropic displacement parameter is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl]$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo(1)	0.0013 (1)	0.0017 (1)	0.0021 (1)	0.0004 (2)	0.0004 (2)	−0.0001 (3)
Mo(2)	0.0016 (1)	0.0016 (1)	0.0022 (1)	0.0003 (2)	0.0006 (2)	0.0004 (3)
P(1)	0.0019 (4)	0.0022 (4)	0.0027 (4)	−0.0001 (6)	0.0023 (7)	−0.0002 (7)
P(2)	0.0026 (3)	0.0012 (3)	0.0017 (4)	−0.0006 (6)	0.0016 (6)	0.0001 (7)
Tl(1)	0.00373 (8)	0.00425 (9)	0.00384 (9)	−0.0010 (2)	−0.0009 (2)	−0.0016 (2)
Tl(2)	0.00808 (9)	0.00564 (8)	0.0144 (2)	−0.0024 (2)	0.0038 (2)	−0.0025 (2)
Tl(3)	0.0153 (3)	0.0162 (3)	0.0050 (2)	0.0206 (4)	0.0078 (4)	0.0067 (5)

Calculations were performed using a MicroVAX II computer with the *SDP* system (Frenz, 1982). Drawings were prepared using *STRUPLO*84 (Fisher, 1985).

Lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71289 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1040]

References

Fisher, R. X. (1985). *J. Appl. Cryst.* **18**, 258–262.
B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package*. College Station Texas, USA.
Gueho, C., Borel, M. M., Grandin, A., Leclaire, A. & Raveau, B. (1993). *J. Solid State Chem.* **104**, 202–208.

Acta Cryst. (1993). C49, 1879–1881

Structure of $Sr_3V_{10}O_{28}.22H_2O$

J. M. Nieto, P. Salagre, F. Medina and
J. E. Sueiras

Departament de Química, Facultad de Química,
Universitat Rovira i Virgili, Pza. Imperial Tarraco, 1.
E-43005 Tarragona, Spain

X. Solans

Departament de Cristal.lografia,
Mineralogia i Dipòsits Minerals,
Universitat de Barcelona, Martí i Franquès s/n,
E-08028 Barcelona, Spain

(*Received* 14 *October* 1992; *accepted* 2 *April* 1993)

Abstract

The crystal structure of hydrated strontium decavanadate, $Sr_3V_{10}O_{28}.22H_2O$, has been determined. It contains two types of strontium ions: the first is coordinated to seven water molecules which define a pentagonal bipyramid; the second bonds to five molecules of water and three O atoms of different decavanadate ions, thereby bridging between decavanadate ions to produce layers of formula $[Sr_2V_{10}O_{38}H_{20}]_n$ on the crystallographic plane (100). The heptacoordinated Sr and the five molecules of water associated with it are located between the different layers.

Comment

The complicated chemistry which arises from the condensation reactions of aqueous vanadate ions and other transition-metal oxo anions depends on the pH of the solution in which the reactions occur. The more acidic the solution the greater is the molecular weight of the isopolynuclear complex anions formed. The following vanadates may be obtained: orthovanadates (pH > 12.6), pyrovanadates (pH 12.6–9.6), metavanadates (pH 9.6–6.5), polyvanadates (pH 6.5–2) and pervanadyl (pH < 0.8). Among the polyvanadates, the decavanadates (salts of the $V_{10}O_{28}^{6-}$ ion) have particular mining and chemical interest as they are found in uranium ores.

Norblad (1875) described $Ba_3V_{10}O_{28}.19H_2O$ among other polyvanadates. The crystal structures of synthetic and natural forms of pascoite $Ca_3V_{10}O_{28}.17H_2O$ have been determined (Marvin & Magin, 1959; Swallow, Ahmed & Barnes, 1966), as have those of double salts like $K_2Mg_2V_{10}O_{28}.16H_2O$, $Cs_2Mg_2V_{10}O_{28}.16H_2O$ and $K_2Zn_2V_{10}O_{28}.16H_2O$ (Weeks, Cisney & Sherwood, 1951; Evans, 1966) and that of the protonated species $[(C_6H_5)_4P]_3H_3V_{10}O_{28}.4CH_3CN$ (Day, Klemperer & Maltbie, 1987).

The title compound was prepared by the addition of excess vanadium pentoxide to a suspension of strontium hydroxide in boiling water, adjusting the pH to about 5 with acetic acid, until the solution became ochre in colour, followed by immediate filtration and crystallization of the filtrate. Crystals were obtained from solutions in distilled water and dried in air at room temperature. Vanadium content was determined spectrophotometrically by the peroxide method (Sandell, 1950) and the strontium content was determined by microgravimetry, giving a chemical analysis and atomic ratio V:Sr = 9.9:3.0. The empirical formula is also consistent with elemental analyses for O and H.

The Sr(1) atom has a B_{eq} value double that of Sr(2). Refinement of its occupancy factor led to a value of 1; slight disorder of this atom around the twofold axis may explain its relatively large B_{eq} value. As only five H atoms were observed in difference syntheses, it was difficult to distinguish between oxide and water O atoms. The presence of

Acta Crystallographica Section C
ISSN 0108-2701 ©1993