anomalous-dispersion correction from *International Tables for X-ray Crystallography* (1974). All the calculations were performed on a PDP 11/60 using the *SDP* programs (B. A. Frenz & Associates Inc., 1985). Final atomic coordinates and equivalent isotropic thermal parameters with their e.s.d.'s are given in Table 1 and selected interatomic distances in Table 2; Fig. 1 shows the numbering scheme of the $Mo_{15}Se_{17}$ cluster unit and Fig. 2 the packing arrangement.*

Related literature. $Cs_3Mo_{15}Se_{17}$ belongs to a large family of condensed Mo cluster compounds of general formula $M_{n-2}Mo_{3n}X_{3n+2}$ (M = Rb, Cs; X = S, Se; n = 2, 3, 4, 5, 6, 7, 8, 10 and ∞). These series can be subdivided into two groups depending on the parity of n. The even members crystallize in the rhombohedral trigonal space group $R\bar{3}$ and have a structure closely related to the ternary chalcogenides MMo_6X_8 (Gougeon, 1984; Gougeon, Potel, Padiou & Sergent, 1987, 1988). The odd members as well as the limiting compound MMo_3X_3 ($n = \infty$) crystallize in the hexagonal space group $P6_3/m$ (Potel, Chevrel & Sergent, 1980).

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51921 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

B. A. Frenz & Associates Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Gougeon, P. (1984). Thesis, Univ. of Rennes, France.

Gougeon, P., Potel, M., Padiou, J. & Sergent, M. (1987). *Mater. Res. Bull.* **22**, 1087–1093.

Gougeon, P., Potel, M., Padiou, J. & Sergent, M. (1988). *Mater. Res. Bull.* **23**, 453–460.

Gougeon, P., Potel, M. & Sergent, M. (1989). *Acta Cryst.* **C45**, 182–185.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Potel, M., Chevrel, R. & Sergent, M. (1980). *Acta Cryst.* **B36**, 1545–1548.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1989). **C45**, 1415–1417

Structure of a Linear-Chain Chloro-Bridged Binuclear Platinum Complex, $(NH_4)_4[Pt_2Cl(P_2O_5H_2)_4]$

By Songchun Jin* and Tasuku Ito

Department of Chemistry, Faculty of Science, Tohoku University, Sendai 980, *Japan*

Koshiro Toriumi†

Institute for Molecular Science, Okazaki National Research Institutes, Okazaki 444, *Japan*

and Masahiro Yamashita

Department of Chemistry, College of General Education, Nagoya University, Chikusa-ku, Nagoya 464, *Japan*

(*Received* 13 *March* 1989; *accepted* 26 *April* 1989)

Abstract. Ammonium *catena*-μ-chloro-tetrakis(μ-diphosphonato-P,P)-diplatinum(4−), $(NH_4)_4[Pt_2Cl(P_2O_5H_2)_4]$, M_r = 1073·61, orthorhombic, $Pnnm$, a = 9·660 (5), b = 14·959 (7), c = 8·215 (4) Å, V = 1187 (1) Å^3, Z = 2, D_x = 3·004 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 13·172 mm^{-1}, $F(000)$ = 1010, T = 296 K, R = 0·025 for 1507 reflections [$|F_o| > 3\sigma(F_o)$]. The structure consists of a linear chain with a $\cdots Pt^{II}—Pt^{II}\cdots Cl—Pt^{III}—Pt^{III}—Cl\cdots$ repeating unit along the c axis. Two Pt atoms are bridged by four pyrophosphato ligands ($P_2O_5H_2^{2-}$) and the Pt—Pt separation is 2·830 (1) Å. The bridging Cl atom is disordered over two sites, giving short Pt—Cl [2·363 (4) Å] and long Pt—Cl [3·022 (4) Å] distances in the chain. The Pt dimers lie on $2/m$ sites. The bridging O atoms of the $P_2O_5H_2^{2-}$ ligands deviate 0·298 (6) and 0·548 (5) Å from the planes defined by the two Pt atoms and the two P atoms of the ligand, respectively. No diffuse scattering could be observed on the X-ray photographs.

---

* On leave from Changchun Institute of Applied Chemistry, Changchun, China.

† To whom all correspondence should be addressed.

0108-2701/89/091415-03$03.00

Table 1. *Fractional coordinates* ($\times 10^5$ *for* Pt; $\times 10^4$ *for others*) *and equivalent isotropic thermal parameters* (Å^2) *for the non*-H *atoms*

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

	x	y	z	B_{eq}
Pt	0	0	32774 (2)	1·1
Cl	0	0	402 (4)	3·9
P(1)	2391 (1)	−232 (1)	3202 (1)	1·5
P(2)	465 (1)	1521 (1)	3245 (1)	1·5
O(1)	3064 (6)	−96 (4)	5000	3·0
O(2)	3235 (4)	401 (3)	2183 (5)	3·0
O(3)	2860 (4)	−1198 (2)	2745 (5)	3·3
O(4)	1133 (4)	1813 (3)	5000	1·8
O(5)	−716 (4)	2165 (2)	2919 (4)	2·5
O(6)	1663 (4)	1801 (2)	2071 (4)	2·6
N(1)	135 (5)	3817 (4)	5000	1·9
N(2)	−340 (7)	3205 (5)	0	2·3

Table 2. *Relevant interatomic distances* (Å) *and angles* (°), *and hydrogen-bond distances* (Å)

Pt—Pt^{i}	2·830 (1)	P(1)—O(2)	1·504 (4)
Pt—Cl	2·363 (4)	P(1)—O(3)	1·560 (4)*
Pt···Cl^{ii}	3·022 (4)	P(2)—O(4)	1·639 (2)
Pt—P(1)	2·337 (2)	P(2)—O(5)	1·518 (4)
Pt—P(2)	2·319 (2)	P(2)—O(6)	1·564 (4)*
P(1)—O(1)	1·627 (3)	Cl···Cl^{ii}	0·660 (5)
Cl—Pt—P(1)	88·48 (3)	O(1)—P(1)—O(2)	102·1 (2)
Cl—Pt—P(2)	89·34 (3)	O(1)—P(1)—O(3)	102·6 (3)
P(1)—Pt—P(2)	87·35 (5)	O(2)—P(1)—O(3)	107·0 (2)
P(1)—Pt—P(1^{iii})	176·96 (4)	O(4)—P(2)—O(5)	106·4 (2)
P(2)—Pt—P(2^{iii})	178·68 (4)	O(4)—P(2)—O(6)	100·4 (2)
Pt—P(1)—O(1)	110·6 (2)	O(5)—P(2)—O(6)	106·1 (2)
Pt—P(1)—O(2)	117·2 (2)	P(1)—O(1)—P(1^{ii})	130·5 (4)
Pt—P(1)—O(3)	115·5 (1)	P(2)—O(4)—P(2^{ii})	123·2 (3)
Pt—P(2)—O(4)	109·1 (2)		
Pt—P(2)—O(5)	118·6 (2)		
Pt—P(2)—O(6)	114·4 (1)		
O(2)···N(1)	2·821 (6)	O(2)···O(6)	2·588 (5)
O(3)···N(1^{iv})	2·973 (5)	O(3)···O(5^{iii})	2·530 (5)
O(5)···N(2)	2·882 (5)		

Key to symmetry operations: (i) $x, y, 1-z$; (ii) $x, y, -z$; (iii) $-x, -y, z$; (iv) $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

* An H atom is attached to the O atom.

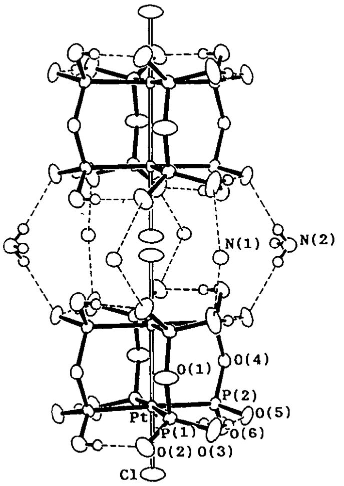

Fig. 1. *ORTEP* drawing of a portion of the linear chain along the *c* axis, showing the atomic numbering scheme; thermal ellipsoids at the 50% probability level, hydrogen bonds by broken lines and the binuclear complex lying on a 2/*m* site. The hydrogen-bond network extended over the chains [O(2)···H—N(1)—H···O(3)] constructs a layer structure parallel to the *ac* plane. Two H atoms of NH_4^+ located by difference maps are shown.

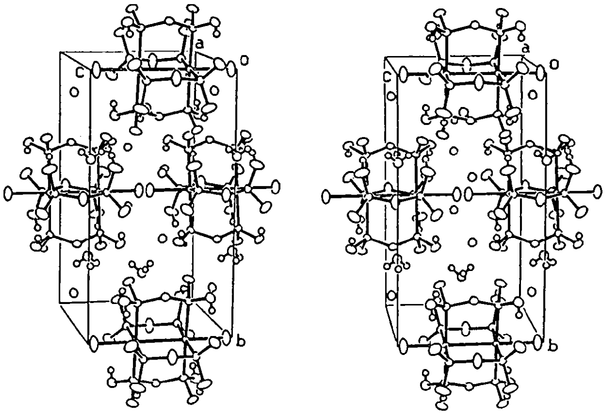

Fig. 2. A stereoscopic view of the crystal structure.

Experimental. The compound was prepared by the slow addition of excess aqueous NH_4Cl to an aqueous solution containing equimolar amounts of $K_4[Pt_2(P_2O_5H_2)_4]$ and $K_4[Pt_2Cl_2(P_2O_5H_2)_4]$ (Butler, Zietlow, Che, Schaefer, Sridhar, Grunthaner, Swanson, Clark & Gray, 1988). A red brown prismatic crystal with dimensions 0·28 × 0·24 × 0·16 mm and faces (010), (101) and (10$\bar{1}$) was used for the X-ray diffractometry. Rigaku AFC-5R diffractometer operating at 50 kV 120 mA, graphite-monochromated Mo $K\alpha$ radiation. Accurate cell parameters by least-squares fit for 31 reflections with $25 < 2\theta < 30°$. Intensity data collected by θ–2θ scan, scan rate 5° min^{-1}, scan range (°) $1{\cdot}4 + 0{\cdot}5 \times \tan\theta$, $2\theta_{max} = 60°$, h −14 to 14, k 0 to 22, l 0 to 12. Three standard reflections monitored every 100 reflections showed no crystal movement or decay. Intensity corrected by numerical absorption method based on Gaussian integration, transmission factor range = 0·192–0·050; 3822 reflections measured, 1507 reflections with $|F_o| > 3\sigma(F_o)$ observed; $R_{int} = 0{\cdot}0261$ for 1312 symmetry-related pairs.

The structure was solved by conventional heavy-atom method, refined on F by block-diagonal least squares. All the non-H atoms refined anisotropically. H atoms of the $P_2O_5H_2^{2-}$ ligand and those in one of the two NH_4^+ ions were located by difference synthesis and refined isotropically. Weighting scheme $w = [\sigma_c^2 + (0{\cdot}020 \times |F_o|)^2]^{-1}$; no extinction correction; $R(F) = 0{\cdot}025$, $wR(F) = 0{\cdot}034$, $S = 1{\cdot}69$ for 1507 independent reflections, 104 parameters refined. Maximum and minimum peak heights in difference Fourier map were 0·92 and $-1·94\ e\ \text{Å}^{-3}$, ratio of max. (shift/σ) = 0·35. Atomic scattering factors and anomalous-scattering corrections from *International Tables for X-ray Crystallography* (1974). All the calculations were made by *UNICS*III (Sakurai & Kobayashi, 1979) and *ORTEP* (Johnson, 1965). Deviations from the least-squares plane and their e.s.d.'s were calculated by the procedure proposed by Ito (1981).

Atomic parameters are given in Table 1.* Relevant interatomic distances and angles and hydrogen-bond distances are presented in Table 2. Fig. 1 shows the linear-chain structure and the atomic numbering scheme. A stereoscopic view of the crystal structure is shown in Fig. 2.

Related literature. This compound is one of a series of one-dimensional halogen-bridged mixed valence compounds which are of interest due to the strong electron–lattice interaction. Crystal structures and solid state properties of the related compounds, $K_4[Pt_2(P_2O_5H_2)_4X].nH_2O$ (X = Cl, Br and I), were reported (Butler *et al.*, 1988; Clark, Kurmoo, Dawes & Hursthouse, 1986; Che, Herbstein, Schaefer, Marsh & Gray, 1983). The Pt—Pt distance of 2·830 (1) Å is intermediate between the Pt^{II}—Pt^{II} distance [2·925 (1) Å] observed for $K_4[Pt_2(P_2O_5H_2)_4].2H_2O$ (Marsh & Herbstein, 1983; Che *et al.*, 1983) and the Pt^{III}—Pt^{III} distance [2·695 (1) Å] for $K_4[Pt_2(P_2O_5H_2)Cl_2].2H_2O$ (Che *et al.*, 1983).

---

* Lists of structure factors, anisotropic thermal parameters, and positional and isotropic thermal parameters of H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51917 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Butler, L. G., Zietlow, M. H., Che, C.-M., Schaefer, W. P., Sridhar, S., Grunthaner, P. J., Swanson, B. I., Clark, R. J. H. & Gray, H. B. (1988). *J. Am. Chem. Soc.* **110**, 1155–1162.

Che, C.-M., Herbstein, F. H., Schaefer, W. P., Marsh, R. E. & Gray, H. B. (1983). *J. Am. Chem. Soc.* **105**, 4604–4607.

Clark, R. J. H., Kurmoo, M., Dawes, H. M. & Hursthouse, M. B. (1986). *Inorg. Chem.* **25**, 409–412.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–98, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Ito, T. (1981). *Acta Cryst.* **A37**, 621–624.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Marsh, R. E. & Herbstein, F. H. (1983). *Acta Cryst.* **B39**, 280–287.

Sakurai, T. & Kobayashi, K. (1979). *Rikagaku Kenkyusho Hokoku* (*Rep. Inst. Phys. Chem. Res.*), **55**, 69–77.

Acta Cryst. (1989). **C45**, 1417–1418

Structure of $NaVP_2O_7$

By Y. P. Wang and K. H. Lii*

Institute of Chemistry, Academia Sinica, Taipei, Taiwan

and S. L. Wang*

Department of Chemistry, National Tsing Hua University, Hsinchu, Taiwan

(*Received* 27 *December* 1988; *accepted* 13 *March* 1989)

Abstract. Sodium vanadium pyrophosphate, M_r = 247·874, monoclinic, $P2_1/c$, a = 7·324 (5), b = 7·930 (4), c = 9·586 (6) Å, β = 111·96 (4)°, V = 516·3 (5) Å^3, Z = 4, D_x = 3·189 g cm^{-3}, Mo $K\alpha$, λ = 0·71073 Å, μ = 2·53 mm^{-1}, $F(000)$ = 480, T = 296 K, R = 0·020, wR = 0·022 for 793 reflections with $I > 3·0\sigma(I)$. The compound, which forms a tunnel structure along the [101] direction, is isostructural with $NaMoP_2O_7$ [Leclaire, Borel, Grandin & Raveau (1988). *J. Solid State Chem.* **76**, 131–135]. The Na^+ cations are located in the tunnels formed by pyrophosphate groups and $V^{3+}O_6$ octahedra.

Experimental. The title compound $NaVP_2O_7$ was obtained as yellowish green crystals in an attempt to prepare a sodium analogue of $Cs_2V_3P_4O_{17}$ (Lii, Wang & Wang, 1989) by heating a mixture of $Na_4V_2O_7$, V, VO_2 and P_2O_5 at 1170 K in a sealed quartz tube. Nicolet $R3/V$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation; ω–2θ scan technique. Cell parameters on crystal 0·18 × 0·10 × 0·10 mm from least-squares procedure on 10 reflec-

---

* To whom correspondence should be addressed.

0108-2701/89/091417-02$03.00