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Structure of $[(C_6H_5)_3PCH_3][TaF_6]$

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Abstract. Methyltriphenylphosphonium hexafluorotantalate, $(C_{19}H_{18}P)[TaF_6]$, $M_r = 572.3$, monoclinic, $P2_1/n$, $a = 10.882\,(2)$, $b = 14.008\,(2)$, $c = 14.147\,(2)$ Å, $\beta = 106.670\,(6)°$, $V = 2066\,(7)$ Å^3, $Z = 4$, $D_x = 1.84$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 53.86$ cm^{-1}, $F(000) = 1096$, $T = 296$ K, $R = 0.064$ for 1860 observed reflections with $I > 3\sigma(I)$. The structure consists of a cation–anion pair. The Ta—F bond lengths are 1.84 (6) Å in an octahedral arrangement.

Experimental. Methylenetriphenylphosphorane (Schmidbaur, Buchner & Scheutzow, 1973) (1 g, 3.62 mmol) was slowly added to a suspension of TaF_5 (Cerac, doubly sublimed) (1 g, 3.62 mmol) in 25 ml of CH_2Cl_2 (distilled from $LiAlH_4$ and TaF_5) at 170 K under argon in glassware with O-ring joints. Crystals were obtained after solvent removal and crystallization from CH_2Cl_2/pentane at 195 K. A white rectangular crystal of dimensions 0.10 × 0.19 × 0.71 mm was sealed in a capillary tube. The unit cell was determined by automatic centering of 27 reflections ($9.72 < 2\theta < 17.88°$). Diffraction data were obtained on a Huber four-circle diffractometer controlled with the Crystal Logic automation system with graphite-monochromated Mo $K\alpha$ radiation. A total of 2940 reflections ($-10 \leq h \leq 11$, $-15 \leq k \leq 0$, $-9 \leq l \leq 15$) were measured in the $\theta/2\theta$ scan mode to a maximum 2θ of 45° at a scan speed of 3° min^{-1}. Equivalent reflections were not measured. Three standard reflections were measured after every 97 reflections and showed no significant variation. The data were corrected for Lorentz and polarization effects and an analytical absorption correction (transmission factors 0.614, 0.349) was applied ($\mu = 53.8$ cm^{-1}, $A_{\max} = 0.783$, $A_{\min} = 0.591$) (Busing & Levy, 1957).

After data reduction there were 1860 observed reflections according to the criterion $I > 3\sigma(I)$ and 1080 unobserved reflections. Systematic absences are $h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$. The Ta- and P-atom positions were located by direct methods (*MULTAN*80; Main *et al.*, 1980). Positions of the non-H atoms were found by successive cycles of full-matrix least-squares refinement and Fourier syntheses.

The TaF_6^- anion was refined as an idealized octahedral rigid group (Strouse, 1970) with F—Ta—F bond angles of 90°, and equivalent Ta—F bond lengths [1.84 (6) Å]. The elements of the group translational vibration tensor, **T**, and the diagonal elements of the group libration tensor, **L**, were refined (Schomaker & Trueblood, 1968). The C- and P-atom positions and anisotropic thermal parameters were refined. H-atom positions were calculated assuming idealized geometries; H atoms were assigned isotropic thermal parameters of 0.062 Å^2 and included as fixed contributors.

The refinement of 197 parameters (data to parameter ratio 9.44) converged at $R(F) = 0.064$ and $wR(F) = 0.075$ [$w = 1/\sigma(F)$]; the goodness of fit was 2.13. The shift to e.s.d. ratio in the final refinement cycle was 0.009. The final difference Fourier synthesis showed excursions of 3.6 and -4.0 e Å^{-3} near the TaF_6^- anion.

The *UCLA Crystallographic Computing Package* (Strouse, 1985) was used throughout. *ORTEP* (Johnson, 1976) was used to draw Fig. 1; atom labels were modified by using *PLOTMD* (Luo, Ammon & Gilliland, 1989). Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 lists fractional atomic coordinates and equivalent isotropic thermal parameters of the non-H atoms of the

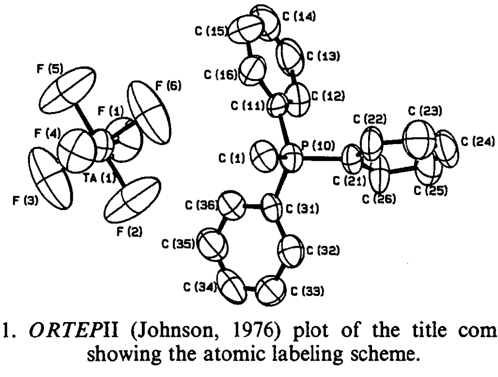

Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title compound showing the atomic labeling scheme.

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^4)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
C(1)	0.6025 (15)	−0.3967 (12)	1.1101 (12)	74 (14)
P(10)	0.4737 (4)	−0.3446 (3)	1.1448 (3)	60 (3)
C(11)	0.4789 (14)	−0.2158 (10)	1.1340 (10)	56 (11)
C(12)	0.3868 (17)	−0.1627 (14)	1.1606 (12)	79 (15)
C(13)	0.4011 (26)	−0.0640 (16)	1.1606 (14)	107 (21)
C(14)	0.4957 (32)	−0.0196 (16)	1.1334 (17)	119 (25)
C(15)	0.5844 (26)	−0.0747 (18)	1.1065 (19)	125 (25)
C(16)	0.5784 (17)	−0.1732 (13)	1.1073 (13)	80 (15)
C(21)	0.4882 (15)	−0.3692 (11)	1.2718 (10)	61 (12)
C(22)	0.6053 (17)	−0.3920 (14)	1.3336 (12)	86 (16)
C(23)	0.6214 (21)	−0.4012 (16)	1.4330 (16)	106 (21)
C(24)	0.5202 (26)	−0.3824 (14)	1.4691 (13)	101 (20)
C(25)	0.4012 (22)	−0.3632 (15)	1.4098 (15)	102 (20)
C(26)	0.3798 (17)	−0.3599 (14)	1.3057 (11)	85 (16)
C(31)	0.3293 (15)	−0.3917 (11)	1.0676 (11)	60 (12)
C(32)	0.2822 (16)	−0.4800 (13)	1.0867 (12)	73 (14)
C(33)	0.1767 (18)	−0.5211 (14)	1.0180 (16)	87 (17)
C(34)	0.1215 (17)	−0.4750 (18)	0.9313 (15)	94 (19)
C(35)	0.1650 (20)	−0.3888 (17)	0.9110 (14)	103 (20)
C(36)	0.2709 (17)	−0.3481 (13)	0.9782 (13)	81 (16)

cation, Table 2 gives selected bond distances and angles.*

Table 2. *Selected bond distances* (Å) *and angles* (°)

P(10)—C(1)	1.77 (2)	P(10)—C(21)	1.79 (1)
P(10)—C(11)	1.81 (1)	P(10)—C(31)	1.76 (2)
C(1)—P(10)—C(11)	109.9 (8)	C(12)—C(11)—P(10)	118.0 (12)
C(1)—P(10)—C(21)	110.5 (8)	C(16)—C(11)—P(10)	119.9 (13)
C(1)—P(10)—C(31)	108.0 (7)	C(22)—C(21)—P(10)	119.1 (13)
C(11)—P(10)—C(21)	106.3 (7)	C(26)—C(21)—P(10)	119.0 (12)
C(11)—P(10)—C(31)	111.3 (7)	C(32)—C(31)—P(10)	121.6 (13)
C(21)—P(10)—C(31)	110.8 (8)	C(36)—C(31)—P(10)	119.6 (13)

Related literature. There are no reported examples of X-ray molecular structures of tetrasubstituted phosphonium salts of TaF_6^-. The closest example is 18-crown-6 bis(methyltriphenylphosphonium) bis(hexafluorophosphate) clathrate.

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References

Busing, W. R. & Levy, H. A. (1957). *Acta Cryst.* **10**, 180–182.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Luo, J., Ammon, H. L. & Gilliland, G. L. (1989). *J. Appl. Cryst.* **22**, 186.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Schmidbaur, H., Buchner, W. & Scheutzow, D. (1973). *Chem. Ber.* **106**, 1251–1255.
Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* B**24**, 63–76.
Strouse, C. E. (1970). *Acta Cryst.* A**26**, 604–608.
Strouse, C. E. (1985). *UCLA Crystallographic Computing Package.* Department of Chemistry, UCLA, Los Angeles, CA 90024, USA.

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* Lists of structure factors, anisotropic thermal parameters, positional parameters for the anion, complete bond distances and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54784 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of [PrCl$_3$(EO4)]$_2$

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Abstract. Di-μ-chloro-bis[dichloro(tetraethylene glycol)praseodymium(III)], $M_r = 882.99$, triclinic, $P\overline{1}$, $a = 8.564$ (5), $b = 9.439$ (9), $c = 9.566$ (5) Å, $\alpha = 86.01$ (9), $\beta = 72.30$ (6), $\gamma = 78.09$ (9)°, $V = 720.8$ Å^3, $Z = 1$, D_m not determined, $D_x = 2.03$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 39.3$ cm^{-1}, $F(000) = 432$, $T = 293$ K, final $R = 0.037$ for 2114 observed [$F_o \geq 5\sigma(F_o)$] reflections. The dimer resides around a crystallographic center of inversion with asymmetric [2.986 (2), 2.910 (2) Å] chloro bridges. Two terminal chloride ions and the pentadentate tetraethylene glycol ligand (EO4) complete the 9-coordinate tricapped trigonal-prismatic Pr coordination sphere. There is one intramolecular hydrogen bond and one

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