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Structural Studies of Technetium Complexes. VII.* Structure of Tetraphenylarsonium Hexachlorotechnetate(IV), $[As(C_6H_5)_4]_2[TcCl_6]$

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Abstract. $M_r = 1078{\cdot}32$, triclinic, $P\bar{1}$, $a = 10{\cdot}111$ (4), $b = 12{\cdot}165$ (3), $c = 10{\cdot}263$ (5) Å, $\alpha = 93{\cdot}86$ (3), $\beta = 114{\cdot}51$ (4), $\gamma = 99{\cdot}08$ (3)°, $U = 1122$ (2) Å^3, $Z = 1$, $D_x = 1{\cdot}596$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7{\cdot}477$ mm^{-1}, $F(000) = 539$, $T = 294$ (1) K. Final $R = 0{\cdot}025$ for 3590 observed reflections. The $[TcCl_6]^{2-}$ ion shows small but significant deviations from ideal octahedral geometry, with Tc—Cl bond distances 2·3441 (7), 2·3635 (7) and 2·3649 (7) Å, and Cl—Tc—Cl angles 88·63 (3), 89·95 (3) and 90·26 (3)°. The small tetragonal distortion in the anion seems to be caused by the crystal-packing forces arising from the presence of the bulky $[AsPh_4]^+$ ions.

Introduction. Technetium in the form of the short-lived isomer technetium-99m ($t_{1/2} = 6$ h) is widely used in diagnostic nuclear medicine. However, the chemistry of technetium in radiopharmaceuticals is poorly understood due to the very low molar concentrations (typically *ca* 10^{-8} mol dm^{-3}) of the metal. By use of the long-lived technetium-99 nuclide ($t_{1/2} = 2{\cdot}12 \times 10^5$ years), it is possible to isolate technetium compounds which can be characterized by conventional chemical and spectroscopic techniques.

We have in hand a study of the coordination chemistry of technetium by techniques including that of single-crystal X-ray diffraction analysis. The results are being used in a research programme aimed at the design of new organ-specific radiopharmaceuticals.

A number of complex anions of technetium have recently been prepared and isolated as the tetraphenylarsonium salts, and the crystal structure of tetraphenylarsonium tetrachloronitridotechnetate(VI), $[AsPh_4][TcCl_4N]$, has been determined (Baldas, Boas, Bonnyman & Williams, 1984). In the course of this work, we isolated crystals of tetraphenylarsonium hexachlorotechnetate(IV), $[AsPh_4]_2[TcCl_6]$, for which preliminary photographs showed the crystal system to be triclinic. The structures of a number of salts of $[TcCl_6]^{2-}$ are known, but apart from that of $H_2[TcCl_6].9H_2O$ which is triclinic and of space group $P1$ (Koz'min, Novitskaya & Kuzina, 1972; Koz'min & Novitskaya, 1975), the others for which Tc—Cl distances are available are cubic.

The structure of $K_2[TcCl_6]$ is cubic, of space group $Fm3m$, with Tc—Cl 2·35 (1) Å (Elder, Fergusson, Gainsford, Hickford & Penfold, 1967), and that of $(NH_4)_2[TcCl_6]$ has the same space group with Tc—Cl 2·353 (1) Å (Elder, Estes & Deutsch, 1979). In both structures, the point symmetry of technetium is $m3m$ and the chlorine ligands are required to form a regular octahedral coordination environment. In contrast, there are no symmetry constraints on the positions of the six chlorine ligands in $H_2[TcCl_6]$ and the coordination environment is considerably distorted from regular octahedral, with Tc—Cl distances ranging from 2·22 (3) to 2·51 (3) Å and Cl—Tc—Cl angles from 81·8 (2·5) to 97·4 (2·5)°. The lack of precision in the structural parameters in this case arises from the use of film data in the structure analysis.

We have therefore determined the structure of $[AsPh_4]_2[TcCl_6]$ to accurately define the $[TcCl_6]^{2-}$ structural parameters in a situation where symmetry constraints do not dictate the coordination geometry, and to add to the data on Tc—Cl bonding.

Experimental. Ammonium [^{99}Tc]pertechnetate was supplied by Amersham International p.l.c. Ammonium pertechnetate (31·5 mg) and one equivalent of ammonia were added to conc. hydrochloric acid (20 ml). The mixture was refluxed for 1 h, allowed to cool, and then tetraphenylarsonium chloride (160 mg) in water (2 ml) was added. The yellow precipitate was collected and dried, and recrystallized from acetonitrile–ethanol (1:1 v/v) solution. The IR spectrum of the compound, over the region 600–4000 cm^{-1}, showed only peaks attributable to the tetraphenylarsonium cation. The only other reported preparation of this compound was by a route involving triphenylphosphonium hexachlorotechnetate (Bandoli, Clemente, Mazzi & Roncari, 1982).

Single crystals of $[AsPh_4]_2[TcCl_6]$ suitable for X-ray diffraction studies were grown by slow evaporation at room temperature of an acetonitrile–ethanol (1:1 v/v) solution. Oscillation and Weissenberg photographs showed the crystals to be triclinic. No alternative unit

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* Part VI: Baldas, Boas, Bonnyman & Williams (1984).

cell of higher symmetry was compatible with the derived cell parameters. Unit-cell parameters, together with their e.s.d.'s, were derived by a least-squares analysis (Grant, Gabe & Le Page, 1978) of the setting angles, determined on a diffractometer at 293 K with Cu $K\alpha$ radiation, for 16 angularly well separated reflections with $2\theta > 40°$. Intensity data were recorded on an automatic Siemens AED diffractometer with nickel-filtered Cu $K\alpha$ radiation. The crystal had six well defined faces of type (100), (010), and (001) with perpendicular distances between parallel faces of 0·168, 0·116, and 0·214 mm, respectively. The crystal was aligned with the c axis approximately parallel to the diffractometer φ axis. Intensities were measured by the 'five-values' θ:2θ scan procedure detailed by Hoppe (1965), with a 2θ scan rate of 10° min^{-1}. A reference reflection, monitored every 20 reflections, showed no significant variation in intensity during data collection. A unique hemisphere of 4510 data was collected within the limit $(\sin\theta)/\lambda \le 0{\cdot}609$ Å^{-1}, range of hkl: h $-12\rightarrow10$, k $-14\rightarrow14$, l $0\rightarrow12$; of these data, 3590 reflections were considered observed $[I > 3\sigma(I)]$ and used for structure analysis. The integrated intensities were corrected for Lorentz and polarization effects, and for absorption (Cromer & Liberman, 1970).

Attempts to refine the structure in the space group $P1$ were unsuccessful, and large correlations were apparent in the least-squares procedure. Accordingly, the space group was assumed to be $P\bar{1}$ and the Tc atom was placed at the special position (0,0,0). Difference Fourier syntheses revealed the positions of all non-H atoms and full-matrix least-squares refinement with data uncorrected for absorption and with anisotropic temperature factors assigned to all atoms converged (259 variables) with $R = 0{\cdot}041$; the function minimized was $\sum w(\Delta F)^2$ where $w = [\sigma^2(F_o) + 0{\cdot}0001F_o^2]^{-1}$ is the weight assigned to the $|F_o|$ values. After absorption corrections were applied, with transmission factors ranging between 0·31 and 0·51, the same refinement converged with $R = 0{\cdot}039$. H atoms were included in the scattering model, in idealized positions with C—H 1·00 Å. These were not refined, with the exception of an overall isotropic temperature factor which at convergence had the value 0·064 (3) Å^2. Refinement (260 variables) converged with $R = 0{\cdot}028$. An examination of $|F_o|$ and $|F_c|$ values at this stage indicated that a small extinction effect was apparent in the very intense data. An empirical isotropic extinction correction (Sheldrick, 1976) was included in the model and refinement (261 variables, 3590 data) converged with $R = 0{\cdot}025$, $R_w = 0{\cdot}030$, $S = 1{\cdot}89$; $(\Delta/\sigma)_{\max} = 0{\cdot}06$; largest peaks on final difference synthesis 0·53 and $-0{\cdot}38$ e Å^{-3}. Neutral-atom scattering-factor curves for C and Cl from Cromer & Mann (1968), those for neutral As and Tc from *International Tables for X-ray Crystallography* (1974), and that for H from Stewart, Davidson & Simpson (1965); real and imaginary anomalous-dispersion corrections applied to non-H atoms (Cromer & Liberman, 1970). Structure determination and refinement performed with *SHELX*76 (Sheldrick, 1976) on the Commonwealth Department of Health IBM 370/168 computer.

Table 1. *Atomic positional coordinates, with e.s.d.'s in parentheses, and equivalent isotropic thermal parameters for* $[AsPh_4]_2[TcCl_6]$

	x	y	z	B_{eq}*(Å^2)
Tc	0·0	0·0	0·0	2·75
Cl(1)	0·08143 (8)	−0·05763 (6)	−0·17171 (8)	3·74
Cl(2)	0·01029 (9)	−0·17905 (6)	0·07544 (9)	4·01
Cl(3)	0·24892 (8)	0·06810 (6)	0·17179 (9)	4·10
As	0·17844 (3)	0·31098 (2)	0·61056 (3)	2·67
C(1)	0·2347 (3)	0·3813 (2)	0·4757 (3)	3·03
C(2)	0·2828 (3)	0·4983 (2)	0·4975 (3)	3·61
C(3)	0·3296 (3)	0·5471 (3)	0·4023 (4)	4·46
C(4)	0·3297 (4)	0·4812 (3)	0·2884 (4)	4·95
C(5)	0·2798 (4)	0·3665 (3)	0·2646 (4)	4·82
C(6)	0·2309 (3)	0·3154 (3)	0·3581 (3)	3·94
C(7)	0·1838 (3)	0·4262 (2)	0·7502 (3)	2·77
C(8)	0·3174 (3)	0·4988 (2)	0·8389 (3)	3·58
C(9)	0·3208 (3)	0·5853 (3)	0·9343 (3)	4·11
C(10)	0·1915 (4)	0·5995 (3)	0·9430 (4)	4·28
C(11)	0·0603 (3)	0·5258 (3)	0·8585 (4)	4·22
C(12)	0·0550 (3)	0·4384 (2)	0·7610 (3)	3·46
C(13)	−0·0199 (3)	0·2248 (2)	0·5142 (3)	2·84
C(14)	−0·0605 (3)	0·1297 (2)	0·5658 (3)	3·85
C(15)	−0·2091 (4)	0·0766 (3)	0·5050 (4)	4·86
C(16)	−0·3130 (4)	0·1181 (3)	0·3940 (4)	4·77
C(17)	−0·2700 (3)	0·2105 (3)	0·3412 (4)	4·50
C(18)	−0·1224 (3)	0·2651 (2)	0·4014 (3)	3·70
C(19)	0·3244 (3)	0·2280 (2)	0·7158 (3)	2·94
C(20)	0·3225 (3)	0·1898 (2)	0·8398 (3)	3·26
C(21)	0·4355 (3)	0·1380 (2)	0·9236 (4)	3·95
C(22)	0·5497 (3)	0·1271 (2)	0·8863 (4)	4·18
C(23)	0·5503 (3)	0·1637 (2)	0·7632 (4)	4·03
C(24)	0·4375 (3)	0·2144 (2)	0·6760 (4)	3·56

* $B_{eq} = \frac{8}{3}\pi^2\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Discussion. Final atomic parameters are listed in Table 1.* The geometry and atom numbering of the $[TcCl_6]^{2-}$ ion are shown in Fig. 1. Interatomic bond distances and angles, with e.s.d.'s derived from the least-squares refinement, are given in Table 2. The contents of the unit cell are shown in Fig. 2.

Tc—Cl bond distances have been tabulated for 22 compounds (Bandoli, Mazzi, Roncari & Deutsch, 1982), and lie between 2·24 and 2·68 Å for a range of technetium oxidation states (II to V), coordination numbers (5 to 8), and *trans*-situated groups. In $[AsPh_4]_2[TcCl_6]$, there are two distinct Tc^{IV}—Cl bond lengths, 2·344 (1) and 2·364 (1) Å. The Tc^{IV}—Cl distances in the regular octahedral $[TcCl_6]^{2-}$ ions of $(NH_4)_2[TcCl_6]$ [2·3531 (5) Å; Elder *et al.*, 1979] and of $K_2[TcCl_6]$ [2·35 (1) Å; Elder *et al.*, 1967] lie between these values.

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* Tables of structure factors, anisotropic thermal parameters and intra- and interionic contact distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39395 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic bond distances* (Å) *and angles* (°) *in* $[AsPh_4]_2[TcCl_6]$

Tc—Cl(1)	2·3441 (7)	C(9)—C(10)	1·383 (4)
Tc—Cl(2)	2·3635 (7)	C(10)—C(11)	1·371 (4)
Tc—Cl(3)	2·3649 (7)	C(11)—C(12)	1·389 (4)
As—C(1)	1·905 (3)	C(13)—C(14)	1·378 (4)
As—C(7)	1·915 (3)	C(13)—C(18)	1·377 (4)
As—C(13)	1·911 (3)	C(14)—C(15)	1·388 (4)
As—C(19)	1·913 (2)	C(15)—C(16)	1·381 (5)
C(1)—C(2)	1·400 (4)	C(16)—C(17)	1·367 (5)
C(1)—C(6)	1·386 (4)	C(17)—C(18)	1·385 (4)
C(2)—C(3)	1·380 (4)	C(19)—C(20)	1·390 (4)
C(3)—C(4)	1·372 (5)	C(19)—C(24)	1·394 (4)
C(4)—C(5)	1·376 (5)	C(20)—C(21)	1·388 (4)
C(5)—C(6)	1·388 (4)	C(21)—C(22)	1·380 (4)
C(7)—C(8)	1·388 (4)	C(22)—C(23)	1·369 (5)
C(7)—C(12)	1·381 (4)	C(23)—C(24)	1·389 (4)
C(8)—C(9)	1·375 (4)		

Cl(1)—Tc—Cl(2)	88·63 (3)	C(8)—C(9)—C(10)	120·0 (3)
Cl(1)—Tc—Cl(3)	90·26 (3)	C(9)—C(10)—C(11)	120·0 (3)
Cl(2)—Tc—Cl(3)	89·95 (3)	C(10)—C(11)—C(12)	120·6 (3)
C(1)—As—C(7)	108·3 (1)	C(11)—C(12)—C(7)	119·2 (3)
C(1)—As—C(13)	110·3 (1)	As—C(13)—C(14)	119·8 (2)
C(1)—As—C(19)	109·1 (1)	As—C(13)—C(18)	118·5 (2)
C(7)—As—C(13)	108·1 (1)	C(14)—C(13)—C(18)	121·4 (3)
C(7)—As—C(19)	106·0 (1)	C(13)—C(14)—C(15)	118·6 (3)
C(13)—As—C(19)	114·8 (1)	C(14)—C(15)—C(16)	120·2 (3)
As—C(1)—C(2)	120·1 (2)	C(15)—C(16)—C(17)	120·4 (3)
As—C(1)—C(6)	119·5 (2)	C(16)—C(17)—C(18)	120·2 (3)
C(2)—C(1)—C(6)	120·4 (3)	C(17)—C(18)—C(13)	119·1 (3)
C(1)—C(2)—C(3)	119·2 (3)	As—C(19)—C(20)	118·6 (2)
C(2)—C(3)—C(4)	120·1 (3)	As—C(19)—C(24)	120·6 (2)
C(3)—C(4)—C(5)	121·1 (3)	C(20)—C(19)—C(24)	120·7 (2)
C(4)—C(5)—C(6)	119·9 (3)	C(19)—C(20)—C(21)	118·9 (3)
C(5)—C(6)—C(1)	119·2 (3)	C(20)—C(21)—C(22)	120·5 (3)
As—C(7)—C(8)	119·6 (2)	C(21)—C(22)—C(23)	120·4 (3)
As—C(7)—C(12)	120·2 (2)	C(22)—C(23)—C(24)	120·4 (3)
C(8)—C(7)—C(12)	120·2 (3)	C(23)—C(24)—C(19)	119·1 (3)
C(7)—C(8)—C(9)	119·9 (3)		

Two other six-coordinated complexes of technetium(IV) with Tc—Cl bonds are $[TcCl_5(PPh_3)]^-$ (Bandoli, Clemente, Mazzi & Roncari, 1982) and $[TcCl_4(\text{salicylaldehydato})]^-$ (Mazzi, Roncari, Bandoli & Clemente, 1982). In the $[TcCl_5(PPh_3)]^-$ anion, which has approximate octahedral symmetry, Tc—Cl distances range between 2·32 (1) and 2·36 (1) Å, the longest bond being *trans* to the coordinated triphenylphosphine group. Similar Tc^{IV}—Cl distances [2·31 (1)–2·36 (1) Å] are found in the $[TcCl_4(\text{salicylaldehydato})]^-$ anion, and it therefore appears that in the absence of factors such as large *trans* effects or steric crowding due, for example, to bulky ligands or coordination numbers greater than six, 'normal' Tc^{IV}—Cl bond distances will lie within the narrow range of *ca* 2·32 to 2·36 Å.

It has been claimed, from a survey of a number of technetium complexes with oxidation states ranging from (II) to (V) (Bandoli, Mazzi, Roncari & Deutsch, 1982), that the oxidation state of technetium has little effect on the Tc—Cl bond length. In support of this contention is the first report of a Tc^{VI}—Cl bond distance, in the square-pyramidal $[TcCl_4N]^-$ anion (Baldas *et al.*, 1984). The Tc^{VI}—Cl bond length of 2·3220 (9) Å is only slightly shorter than the Tc^{IV}—Cl distances of 2·3441 (7) to 2·3649 (7) Å observed in $[AsPh_4]_2[TcCl_6]$.

A large range is found for Tc^{IV}—Cl bond distances in the polymeric structure of $TcCl_4$ (Elder & Penfold, 1966). The distorted $TcCl_6$ units possess three pairs of chemically distinct Tc—Cl bonds, two involving bridging chlorine atoms [2·383 (5), 2·492 (5) Å], and a third which is quite short [2·242 (5) Å] to terminal chlorine atoms. Clearly in this case the gross effects caused by the sharing of four chlorine atoms with neighbouring $TcCl_6$ units is concomitant with the degree of distortion from ideal octahedral geometry and with the variation in Tc—Cl bond lengths. It is of interest that there are small but significant distortions from ideal octahedral geometry in the $[TcCl_6]^{2-}$ anion of $[AsPh_4]_2[TcCl_6]$, especially in view of the regular geometry of this anion in $K_2[TcCl_6]$ and $(NH_4)_2[TcCl_6]$. These distortions are manifested by the 0·021 (1) Å variation in Tc—Cl bond lengths, and by the 1·37 (3), 0·26 (3) and 0·05 (3)°

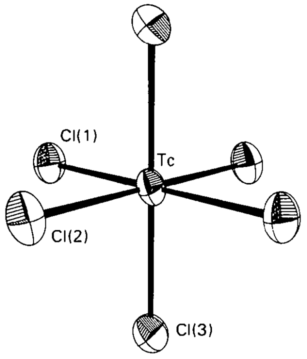

Fig. 1. An *ORTEP* drawing (Johnson, 1976) of the $[TcCl_6]^{2-}$ anion in $[AsPh_4]_2[TcCl_6]$. Thermal ellipsoids are drawn at the 40% probability level.

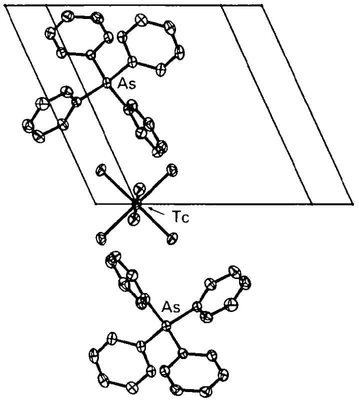

Fig. 2. The unit cell of $[AsPh_4]_2[TcCl_6]$ and its contents. Only one formula unit is shown for clarity; others are related to this by unit translations, with $[TcCl_6]^{2-}$ anions sited at each corner of the unit cell. The Tc atom is at the origin. Thermal ellipsoids are drawn at the 30% probability level.

differences from 90° in the Cl—Tc—Cl bond angles. It is reasonable to relate these effects to the presence of the $[AsPh_4]^+$ cations in the crystal lattice, and indeed close Cl···H contacts of *ca* 2·8 Å, and Cl···C contacts of *ca* 3·5 Å, occur with each chlorine atom. The sum of van der Waals radii for chlorine and hydrogen is 3·0 Å (Pauling, 1960). It seems likely, therefore, that crystal-packing forces resulting from the presence of the bulky $[AsPh_4]^+$ cations, rather than electronic effects, are the cause of the small tetragonal distortion in the $[TcCl_6]^{2-}$ anion in this compound.

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Structure of Tris(cyclohexylammonium) Chloride Tetrachlorocobaltate(II), $(C_6H_{14}N)_3Cl(CoCl_4)$

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Abstract. $M_r = 536·75$, monoclinic, $P2_1/c$, $a = 15·365$ (6), $b = 10·180$ (5), $c = 22·042$ (9) Å, $\beta = 127·84$ (2)°, $V = 2722·8$ Å^3, $Z = 4$, $D_x = 1·309$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 1·15$ mm^{-1}, $F(000) = 1147$, $T = 293$ (2) K, wR (R) = 0·056 (0·070) for 1376 observed reflections. The structure consists of slightly distorted $CoCl_4^{2-}$ tetrahedra, connected by hydrogen bonds through cyclohexylammonium groups into chains along **c**, and *via* two amine nitrogen atoms and an uncoordinated chloride along **b**. All cyclohexyl rings are in the chair conformation.

Introduction. In recent years we have been successful in synthesizing materials with ferromagnetic interactions predominantly in one direction (Willett, Gaura & Landee, 1983; Willett & Landee, 1981). In particular, the compound cyclohexylammonium trichlorocuprate(II), for short CHAC (Groenendijk, Blöte, Van Duyneveldt, Gaura, Landee & Willett, 1981), attracted quite some interest in the solid-state physics community (Kopinga, Tinus & de Jonge, 1982; Poertadji, Ablart, Pescia, Clément & Cheik-Rouhou, 1983). We therefore tried to synthesize the corresponding cobalt(II) salt. In the course of these investigations we obtained the compound tris(cyclohexylammonium) chloride tetrachlorocobaltate(II), CHCO, which promises to have interesting magnetic properties, even though its structure is not related to CHAC.

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