

complex. This is comparable to the seven-coordinate, and sterically crowded,  $[\text{W}(\text{CH}_2\text{PMe}_3)(\text{CO})_2\text{Cl}(\text{PMe}_3)_3]^+\cdot\text{CF}_3\text{SO}_3^-$  (Churchill & Wasserman, 1982) which shows  $126.4(4)^\circ$  for the analogous W—C—P bond angle. In contrast,  $[\text{Cp}^*\text{Fe}(\text{CO})_2(\text{CH}_2\text{PPh}_3)]^+\cdot\text{BF}_4^-$  (Guerchais, Astruc, Nunn & Cowley, 1990), a sterically less demanding complex, has an Fe—C—P angle of only  $118.1(7)^\circ$ . The steric crowding is also manifested in distortions of the bonding of ligands about the metal. The Cp ring is tilted with respect to the Mo center having Mo—C distances ranging from 2.318 (2) to 2.327 (2) Å for C(6), C(7), C(3) to 2.364 (2) and 2.344 (2) Å for C(4) and C(5), respectively. Further, the Mo—P(1)—O angles of the phosphite ligand are found to vary from  $111.35(7)$  to  $122.82(6)^\circ$  and the O—P(1)—O angles range from  $95.91(9)$  to  $104.84(9)^\circ$ .

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## Structure of Bis(4-nitro-1,2-catecholato)oxotechnetium(V)

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**Abstract.** Tetra-*n*-butylammonium bis(4-nitro-1,2-benzenediolato)oxotechnetate(V),  $[\text{N}(\text{C}_4\text{H}_9)_4][\text{Tc}(\text{C}_6\text{H}_3\text{NO}_4)_2(\text{O})]$ ,  $M_r = 663.57$ , orthorhombic,  $P2_12_12$ ,  $a = 14.457(4)$ ,  $b = 21.380(6)$ ,  $c = 10.209(3)$  Å,  $V = 3155(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.39(1)$ ,  $D_x = 1.392$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.49$  mm<sup>-1</sup>,  $F(000) = 1384$ ,  $T = 295$  K and  $R = 0.065$  for 2658 observed reflections. The geometry around the Tc atom is square pyramidal with a short Tc—O(oxo) bond [ $1.634(4)$  Å] in the apical position. The Tc—O bonds with the diolato ligands are longer with distances of  $1.952(4)$  to  $1.979(5)$  Å. The Tc atom lies  $0.6947(5)$  Å out of the plane of the four diolato O atoms. The nitro groups are in the same planes as the catecholato ligands. The nitro group of one ligand is disordered.

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**Introduction.** The chemistry of Tc has recently become very important especially in relation to the use of the isotope <sup>99m</sup>Tc in nuclear medicine as an organ-imaging agent. The element has been combined with different types of ligands to produce very specific radiopharmaceuticals for imaging the bones, the heart, the brain, the kidneys, the liver and other organs, and also tumor tissue (Clarke & Podbielski, 1987; Nicolini, Bandoli & Mazzi, 1990).

Several of the <sup>99m</sup>Tc radiopharmaceuticals contain purely O donor ligands [see Davison, DePamphilis, Jones, Franklin & Lock (1987) and references therein] but few have been characterized. Recently, oxotechnetium(V) complexes of ethyleneglycol and catechol were reported and the crystal structure of the catechol compound was determined (Davison *et*

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*al.*, 1987). These compounds were synthesized from the reaction of  $[\text{Tc}^{\text{V}}\text{OCl}_4]^-$  with the ligand. The chloro ligands in  $[\text{Tc}^{\text{V}}\text{OCl}_4]^-$  are labile (Davison, Orvig, Trop, Sohn, DePamphilis & Jones, 1980), and the compound is a good versatile intermediate for the synthesis of several oxo- $\text{Tc}^{\text{V}}$  compounds (Davison & Jones, 1982). We have recently started a research project on the synthesis of Tc complexes with O donor ligands. In order to facilitate the deprotonation of the catechol ligand, we have chosen a ligand containing a nitro group. The reaction of  $[\text{Tc}^{\text{V}}\text{OCl}_4]^-$  with 4-nitro-1,2-catechol was studied. We have isolated  $[\text{Tc}^{\text{V}}(\text{O})(\text{nitrocatecholato})_2]^-$  with a large cation,  $[\text{N}(n\text{-butyl})_4]^+$  and its crystal structure was determined. The results are reported below.

**Experimental.** Ammonium pertechnetate ( $\text{NH}_4^{99}\text{TcO}_4$ ) was obtained from Oak Ridge National Laboratory. It was recrystallized from nitric acid (caution: ammonium pertechnetate in acid medium will produce radioactive volatile compounds) and dissolved in water. A 0.286 M solution was prepared. All manipulations were made in a laboratory approved for low-level radioactive material ( $^{99}\text{Tc}$  is a  $\beta$ -emitter with a particle energy of 0.292 MeV and a half-life of  $2.13 \times 10^5$  years). The ligand 4-nitro-1,2-catechol was purchased from Aldrich and used without further purification.

The infrared spectra were measured in the solid state as KBr pellets on a Perkin-Elmer 783 spectrometer.

The compound  $[\text{N}(n\text{-C}_4\text{H}_9)_4][\text{Tc}(\text{O})(\text{C}_6\text{H}_3\text{NO}_4)_2]$  was synthesized by a method similar to the one described by Davison *et al.* (1987), from the reaction of  $[\text{N}(n\text{-C}_4\text{H}_9)_4][\text{Tc}^{\text{V}}\text{OCl}_4]$  with 4-nitrocatechol in the presence of NaOH in methanol, but using a smaller quantity of NaOH.  $[\text{N}(\text{C}_4\text{H}_9)_4][\text{Tc}^{\text{V}}\text{OCl}_4]$  was synthesized as described by Davison *et al.* (1980). Brownish-red crystals of  $[\text{N}(n\text{-C}_4\text{H}_9)_4][\text{Tc}(\text{O})(\text{C}_6\text{H}_3\text{NO}_4)_2]$ , suitable for X-ray diffraction methods, were obtained by slow recrystallization from a water-acetone mixture. Yield ~40%; m.p. 411–414 K, dec. at 448–473 K. IR ( $\text{cm}^{-1}$ ):  $\nu(\text{Tc}-\text{O})$ , 983; main other bands: 2960 $m$ , 2865 $m$ , 1579 $m$ , 1540 $s$ , 1465 $s$ , 1420 $m$ , 1338 $s$ , 1248 $vs$ , 1228 $s$ , 1113 $m$ , 1062 $m$ , 939 $m$ , 872 $m$ , 807 $s$ , 748 $m$ , 740 $w$ , 728 $m$ , 704 $w$ , 664 $s$ , 610 $m$ , 545 $s$ , 450 $w$  and 320 $w$ .

**Crystallographic measurement and structure resolution.** The reddish square plate (0.4 × 0.4 × 0.18 mm) of  $[\text{N}(\text{C}_4\text{H}_9)_4][\text{Tc}(\text{O})(\text{C}_6\text{H}_3\text{NO}_4)_2]$  was selected after examination under a polarizing microscope for homogeneity. The unit-cell parameters were obtained by least-squares refinement of the angles  $2\theta$  (10–24°),  $\omega$  and  $\chi$  for 15 well centered reflections on a Syntex P1 diffractometer using graphite-monochromated Mo  $K\alpha$  radiation.  $D_m = 1.39$  (1)  $\text{Mg m}^{-3}$  in  $\text{CCl}_4$ -cyclohexane by flotation method; maximum  $2\theta$  60°;

$h$ , 0 → 20;  $k$ , 0 → 30;  $l$ , 0 → 14; scan technique  $2\theta/\theta$ ; standard reflections (variation < 1.7%): 620, 390, 204; number of independent reflections: 5155; number of observed reflections: 2658 with  $I_{\text{net}} > 2.5\sigma$ ; scan rates between 1 and 24°  $\text{min}^{-1}$ . Corrections were made for Lorentz-polarization effects and the anomalous-dispersion terms of Tc were included in the calculations (Cromer, 1965).

The coordinates of the Tc atom were determined from the three-dimensional Patterson map and the positions of all the other non-H atoms were found by the usual Fourier methods. The  $\text{NO}_2$  group in one ligand was found to be disordered on C(10) and C(11). The occupancy factors were refined keeping the isotropic thermal factors constant, and then fixed at 0.5 for each group. The positions of one O in each of the two groups were coincident. Therefore the occupancy factor of O(7) is 1. The thermal factors of a few terminal C atoms [especially C(28)] in the  $n$ -butyl chains were found to be quite high. It was not possible to resolve the disorder around C(28).

The H atoms of the methyl groups could not be located. The coordinates of the other H atoms were fixed at their calculated positions with C—H = 0.95 Å and isotropic  $U_{\text{eq}} = 0.076$  Å<sup>2</sup>. Individual weights  $w = 1/\sigma^2(F)$  were applied. The refinement of the scale factor, coordinates and anisotropic temperature factors of all the non-H atoms (389 parameters) was performed using block-diagonal matrices and converged to  $R = 0.065$  and  $wR = 0.060$ . There were a few residual peaks (< 0.46 e Å<sup>-3</sup>) in the close environment of the Tc atom. The maximum shift/e.s.d. was 0.5 [for C(28)] and the standard deviation (unit weight) = 1.96.

The scattering curves of Cromer & Waber (1965) were used except for the H atoms (Stewart, Davidson & Simpson, 1965). The calculations were performed on a Cyber 830 computer with programs already described (Melanson & Rochon, 1975).

**Discussion.** The refined atomic parameters of the structure are listed in Table 1.\* A labelled diagram of the molecule is shown in Fig. 1. The geometry of the complex is square pyramidal with the oxo ligand in the apical position. The Tc atom is 0.6947 (5) Å out of the plane calculated through the four diolato O atoms. Since the diolato O atoms are coplanar with their benzene rings, the two rings are bent away from the Tc atom with a dihedral angle of 145° between the two rings.

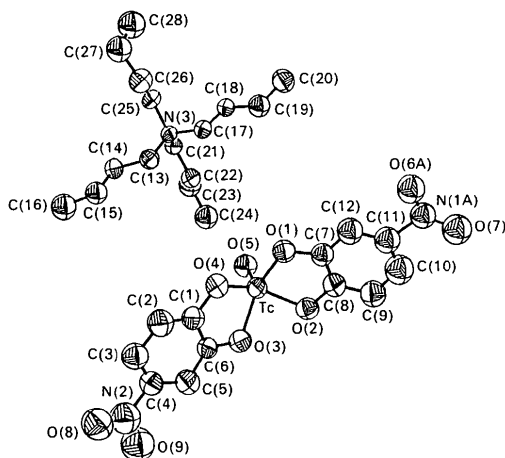
\* Lists of structure factors, anisotropic thermal parameters, weighted best planes, bond distances and angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54735 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0058]

Table 1. Positional parameters ( $\times 10^4$ ) with e.s.d.'s in parentheses and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^4$ )
$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
Tc	152.3 (4)	2157.9 (3)	2237.8 (5)	557
O(1)	-153 (3)	1898 (2)	4019 (4)	647
O(2)	-529 (3)	1384 (2)	1828 (5)	685
O(3)	-443 (3)	2508 (2)	654 (4)	670
O(4)	-148 (3)	2992 (2)	2906 (4)	723
O(5)	1263 (3)	2083 (2)	1982 (4)	741
C(1)	-440 (5)	3398 (3)	1985 (8)	790
C(2)	-608 (7)	4018 (4)	2116 (10)	1084
C(3)	-865 (7)	4397 (4)	1142 (9)	1011
C(4)	-1003 (6)	4130 (3)	26 (7)	771
C(5)	-878 (5)	3530 (4)	-271 (8)	833
C(6)	-591 (4)	3128 (3)	785 (7)	626
C(7)	-493 (5)	1348 (4)	4097 (7)	725
C(8)	-689 (4)	1041 (3)	2884 (9)	775
C(9)	-1073 (5)	443 (4)	2820 (11)	1038
C(10)	-1294 (8)	58 (6)	3866 (12)	1530
C(11)	-1069 (5)	338 (3)	4967 (11)	1247
C(12)	-703 (5)	996 (5)	5216 (10)	1082
C(13)	6151 (4)	2811 (3)	2130 (7)	636
C(14)	6586 (6)	3363 (4)	1444 (8)	799
C(15)	5905 (6)	3867 (4)	1055 (9)	898
C(16)	6327 (8)	4359 (4)	204 (11)	1324
C(17)	6164 (4)	1813 (3)	3258 (6)	524
C(18)	6632 (5)	1257 (3)	3800 (7)	675
C(19)	5923 (6)	794 (4)	4459 (10)	1021
C(20)	6319 (6)	223 (3)	5114 (9)	967
C(21)	7349 (4)	2030 (3)	1502 (7)	605
C(22)	6815 (5)	1734 (3)	402 (7)	686
C(23)	7525 (7)	1490 (5)	-666 (9)	1208
C(24)	7091 (8)	1182 (6)	-1830 (10)	1470
C(25)	7507 (5)	2546 (4)	3589 (7)	673
C(26)	7093 (5)	2863 (4)	4835 (7)	856
C(27)	7915 (8)	3177 (6)	5616 (10)	1555
C(28)	8563 (9)	2887 (10)	6100 (13)	2845
N(2)	-1248 (7)	4519 (4)	-1002 (9)	1551
N(3)	6782 (3)	2296 (2)	2620 (5)	505
O(8)	-1351 (6)	5095 (4)	-903 (7)	1548
O(9)	-1351 (7)	4322 (3)	-2119 (7)	1787
O(7)	-1610 (5)	-588 (3)	5788 (8)	1461
N(1A)	-1291 (9)	-72 (6)	5988 (11)	865
N(1B)	-1472 (11)	-432 (6)	4462 (16)	716
O(6A)	-1089 (8)	174 (5)	7043 (10)	1148
O(6B)	-1564 (11)	-716 (8)	3460 (18)	1267

Table 2. Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Tc—O(1)	1.952 (4)	Tc—O(2)	1.970 (4)
Tc—O(4)	1.958 (4)	Tc—O(5)	1.634 (4)
O(2)—C(8)	1.325 (10)	O(3)—C(6)	1.350 (8)
C(1)—C(2)	1.36 (1)	C(2)—C(3)	1.33 (1)
C(4)—C(5)	1.33 (1)	C(5)—C(6)	1.44 (1)
C(7)—C(8)	1.43 (1)	C(8)—C(9)	1.39 (1)
C(10)—C(11)	1.31 (2)	C(11)—C(12)	1.52 (1)
C(4)—N(2)	1.39 (1)	N(2)—O(8)	1.25 (1)
N(3)—C(av.)	1.521 (8)	C—C(butyl)	1.50 (1)
Tc—O(3)	1.979 (5)	C(6)—C(1)	1.37 (1)
O(1)—C(7)	1.277 (8)	C(9)—C(10)	1.39 (1)
O(4)—C(1)	1.346 (9)	C(12)—C(7)	1.40 (1)
C(3)—C(4)	1.29 (1)	N(2)—O(9)	1.23 (1)
O(5)—Tc—O(1)	110.0 (2)	O(5)—Tc—O(2)	112.0 (2)
O(5)—Tc—O(4)	111.2 (2)	O(1)—Tc—O(2)	81.1 (2)
O(1)—Tc—O(4)	83.4 (2)	O(2)—Tc—O(3)	85.8 (2)
O(2)—Tc—O(4)	136.7 (2)	Tc—O(1)—C(7)	114.1 (4)
Tc—O(3)—C(6)	111.1 (4)	Tc—O(4)—C(1)	114.4 (4)
O(1)—C(7)—C(12)	128.9 (7)	O(2)—C(8)—C(9)	122.7 (7)
O(4)—C(6)—C(1)	118.4 (6)	O(4)—C(1)—C(2)	128.2 (7)
C(1)—C(2)—C(3)	124.8 (9)	C(2)—C(3)—C(4)	115.6 (9)
C(4)—C(5)—C(6)	116.4 (7)	C(5)—C(6)—C(1)	116.7 (7)
C(7)—C(8)—C(9)	122.7 (7)	C(8)—C(9)—C(10)	126.9 (9)
C(10)—C(11)—C(12)	130.4 (9)	C(11)—C(12)—C(7)	115.8 (8)
C(3)—C(4)—N(2)	116.3 (8)	C(5)—C(4)—N(2)	116.1 (8)
C(4)—N(2)—O(8)	124.3 (9)	C(4)—N(2)—O(9)	122.1 (9)
O(5)—Tc—O(3)	109.5 (2)	O(4)—C(1)—C(6)	113.8 (6)
O(3)—Tc—O(4)	81.0 (2)	C(3)—C(4)—C(5)	127.4 (8)
O(1)—Tc—O(3)	140.4 (2)	C(6)—C(1)—C(2)	118.1 (7)
Tc—O(2)—C(8)	112.4 (4)	C(9)—C(10)—C(11)	109.3 (10)
O(1)—C(7)—C(8)	116.5 (6)	C(12)—C(7)—C(8)	114.6 (7)
O(3)—C(6)—C(5)	123.9 (6)	O(8)—N(2)—O(9)	113.6 (9)
C—N(3)—C(av.)	109.5 (5)	C—C—C(butyl, av.)	113.8 (8)
N(3)—C—C(av.)	116.2 (5)		

Fig. 1. Labelled diagram of  $[\text{N}(\text{C}_4\text{H}_9)_4][\text{Tc}(\text{O})(\text{C}_6\text{H}_3\text{NO}_2)_2]$  [the  $\text{NO}_2$  shown in position C(11) is disordered equally on positions C(10) and C(11)].

The bond distances and angles are shown in Table 2. The Tc—O(5)(oxo) distance is 1.634 (4)  $\text{\AA}$ , much shorter than the other four Tc—O(diolato) bonds

which vary from 1.952 (4) to 1.979 (5)  $\text{\AA}$ . These values are close to those observed by Davison *et al.* (1987) in the complexed anion  $[\text{Tc}(\text{O})(\text{C}_6\text{H}_4\text{O}_2)_2]^-$ . The angles around Tc are close to the expected square pyramidal values. The O(oxo)—Tc—O(diolato) angles vary from 109.5 (2) to 112.0 (2) $^\circ$ , while the O(diolato)—Tc—O(diolato) angles are 81.0 (2) and 81.1 (2) $^\circ$  for the chelate angles, 83.4 (2) and 85.8 (2) $^\circ$  for the other *cis* bonds, and 136.7 (2) and 140.4 (2) $^\circ$  for the *trans* bonds.

The bond distances and angles of the two nitro-catecholato ligands are normal. The C—O bonds vary from 1.277 (9) to 1.350 (8)  $\text{\AA}$  while the average C—C distance in the phenyl rings is 1.380 (7)  $\text{\AA}$ . The Tc—O—C angles are slightly higher than the tetrahedral value and vary from 111.1 (4) to 114.4 (4) $^\circ$ . The nitro group was found to be disordered on one of the ligands. In the non-disordered ligand, the N(2)—O bonds are 1.23 (1) and 1.25 (1)  $\text{\AA}$ , while the angle O—N(2)—O is 113.6 (9) $^\circ$ . On the disordered ligand, the nitro group was placed equally on C(10) and C(11), with O(7) being coincident. Fig. 1 shows only one of the two positions. The atoms of the  $\text{NO}_2$  groups are located in the plane of the catecholato ligands. When the second ligand enters the coordinating sphere of Tc, it can bind the metal in two ways, leading to two different isomers. The crystal studied was a mixture of the two isomers in about the same proportion. The bond distances and angles of the disordered atoms are given in the supplementary material.

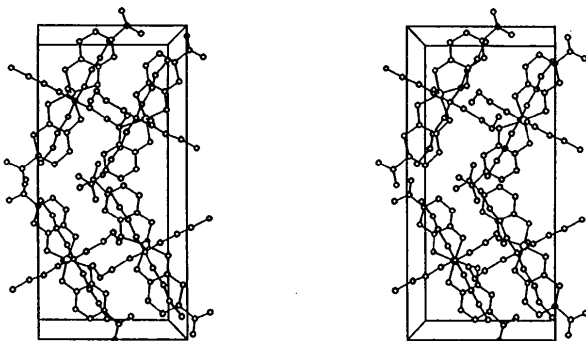


Fig. 2. Stereoscopic view of the unit cell in the crystal  $[\text{N}(\text{C}_4\text{H}_9)_4][\text{Tc}(\text{O})(\text{C}_6\text{H}_3\text{NO}_4)_2]$  ( $b$  axis vertical,  $c$  axis horizontal).

The average value of the N(3)—C bonds in the cation is 1.521 (8) Å, and the average angle around N(3) is 109.5 (5)°. All the values are shown in the supplementary material. The terminal C atoms have very high thermal factors, especially C(28), resulting in high standard deviations. The average C—C distance is 1.50 (1) Å, and the  $n$ -butyl chains are flattened as shown by the large angular values. The average angle N(3)—C—C is 116.2 (5)° and C—C—C of the  $n$ -butyl chains is 113.8 (8)°.

The packing of the molecules in the crystal is shown in Fig. 2. No hydrogen bonding is expected in this crystal. The  $n$ -butyl groups of the cations are arranged in a flat cross (except one terminal C atom), with the cross parallel to the base of the square pyramid.

The infrared absorption band at 983  $\text{cm}^{-1}$  was assigned to the stretching  $\nu(\text{Tc}=\text{O})$ . This value can be compared to the values obtained in a series of  $[\text{TcOX}_4]^-$  ions as compiled by Davison *et al.* (1987). The authors have found a slight correlation between the Tc=O bond distances and the absorption band

$\nu(\text{Tc}=\text{O})$ . As the bond distance increases, the absorption band decreases in energy. When  $X$  is Cl, the Tc=O bond is 1.610 (4) Å with  $\nu(\text{Tc}=\text{O})$  at 1016  $\text{cm}^{-1}$  (Cotton, Davison, Day, Gage & Trop, 1979), while when  $X$  is S, the bond is longer (~1.67 Å) (Davison *et al.*, 1987; Rochon, Melanson & Kong 1992) and the vibration was found around 950  $\text{cm}^{-1}$ . There is only one example where  $X$  is O and the  $\nu(\text{Tc}=\text{O})$  was reported at 970  $\text{cm}^{-1}$  with a Tc=O bond distance of 1.648 (5) Å (Davison *et al.*, 1987). We have found a bond length of 1.634 (4) Å and  $\nu(\text{Tc}=\text{O})$  at 983  $\text{cm}^{-1}$ , which is in good agreement with these authors.

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## Structure of Ni<sup>II</sup>, Pd<sup>II</sup> and Pt<sup>II</sup> Complexes with the Bidentate Ligand 2,11-Bis(diethylphosphinomethyl)benzo[*c*]phenanthrene

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**Abstract.** {2,11-Bis(diethylphosphinomethyl)benzo[*c*]phenanthrene-*P,P'*}dichloronickel(II),  $[\text{Ni}(\text{C}_{28}\text{H}_{34}\text{P}_2)\text{Cl}_2]$ ,  $M_r = 562.14$ , orthorhombic, *Pbcn*,  $a =$

16.197 (6),  $b = 16.886$  (4),  $c = 9.693$  (2) Å,  $V = 2651$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.408$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 10.7$  cm<sup>-1</sup>,  $F(000) = 1176$ ,  $T =$