91·8 (2)° and 0·978 (3) Å in $[FeCo_3(\mu_3\text{-}H)(CO)_9\{P(OMe)_3\}_3]$ (Teller, Wilson, McMullan, Koetzle & Bau, 1978), 93·9 (3)° and 0·907 (6) Å in $[Ni_4(\mu_3\text{-}H)_3(\eta\text{-}C_5H_5)_4]$ (Koetzle, Müller, Tipton, Hart & Bau, 1979) and 95·7° and 0·957 (6) Å for $[Rh_4(\mu_3\text{-}H)_4(\eta\text{-}C_5Me_5)]^{2+}$ (Ricci, Koetzle, Goodfellow, Espinet & Maitlis, 1974). Therefore, the hydride lies closer to the trimetal plane here than in the other examples although the difference is not significant in the case of the Ni_4 cluster. In $[Os_6(\mu_3\text{-}H)(CO)_{18}]^-$ the Os—Os distances in the bridged triangle (mean 2·968 Å) are notably longer than the other Os—Os lengths which may be separated into two classes, distinguishable owing to the near-C_{3v} symmetry of the cluster: those in the Os(1) Os(2) Os(3) triangle (mean 2·881 Å), and those connecting the two triangles (mean 2·863 Å). These values are in good agreement with those obtained at 200 K by X-ray diffraction in this work (2·963, 2·881 and 2·868 Å) and those reported for the $(PPh_3)_2N^+$ salt by McPartlin *et al.* (1976) (2·973, 2·872 and 2·863 Å respectively). These distances reflect the distortions in the Os_6 framework that occur on protonation of $[Os_6(CO)_{18}]^{2-}$ [*cf.* mean Os—Os in the approximate D_3 symmetry dianion 2·863 Å (McPartlin *et al.*, 1976)]. These distortions and those in the surrounding carbonyl coordination sphere (note large Os—Os—C angles involving carbonyls *cis* to the hydride) were the basis for the assignment of the μ_3 site for the hydride in $[Os_6(\mu_3\text{-}H)(CO)_{18}]^-$ (McPartlin *et al.*, 1976; Orpen, 1980). That assignment is fully confirmed by the present study.

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Synthesis and Molecular Structure of Tetrabutylammonium Tetrachloro(pyrimidine-2-thiolato)technetate(IV)

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Abstract. $[N(C_4H_9)_4][Tc(C_4H_3N_2S)Cl_4]$, $M_r = 594·3$, triclinic, $P\bar{1}$, $a = 11·182$ (6), $b = 11·505$ (4), $c = 12·235$ (4) Å, $\alpha = 64·53$ (4), $\beta = 76·81$ (3), $\gamma = 84·10$ (5)°, $V = 1383·5$ Å^3, $Z = 2$, $D_x = 1·427$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·97$ mm^{-1}, $F(000) = 614$, $T = 297$ K, $R = 0·058$ for 3670 unique observed reflections. The coordination about Tc is distorted octahedral with Tc—N 2·087 (6), Tc—S 2·429 (2) and Tc—Cl 2·346 (2)–2·396 (2) Å. The N—Tc—S angle is 67·1 (2)°, constituting the major angular distortion from ideal octahedral.

Introduction. The γ-emitting radionuclide ^{99m}Tc is widely used as an imaging agent in diagnostic nuclear medicine. The biodistribution of ^{99m}Tc following administration to the patient may be controlled by incorporating it in a coordination or organometallic compound. Both the nature of the ligand and the Tc oxidation state are important in determining the physical properties of the product complex and thus its

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biodistribution. The demonstration that lipophilic cationic complexes of ^{99m}Tc can localize in the myocardium has stimulated interest in cationic compounds (Deutsch, Libson & Jurisson, 1985; Jones, Abrams, Davison, Brodack, Toothaker, Adelstein & Kassis, 1984).

In an attempt to prepare and characterize examples of such complexes we have been investigating the reactions of $[TcOCl_4]^-$ with bidentate ligands containing a neutral nitrogen donor atom and an ionizable thiol group. In this way we hoped to prepare compounds of formula $[TcOL_2]^+$ containing the $\{TcON_2S_2\}^+$ coordination shell. The reaction between $[TcOCl_4]^-$ and 2-mercaptopyrimidine afforded a brown crystalline product in low yield. Infra-red spectral data indicated the absence of a band attributable to Tc=O but did indicate the presence of the thiolate ligand. A single-crystal X-ray diffraction study was undertaken to fully characterize this compound.

Experimental. Reagents were used as received and solvents were dried by standard techniques before use. $[Bu_4N]^+[^{99}TcOCl_4]^-$ was prepared according to a previously described method but using Bu_4NBr in place of Bu_4NCl (Davison, Orvig, Trop, Sohn, DePamphilis & Jones, 1980). Infra-red spectra were recorded on a PE 297 instrument.

$Bu_4^nN[TcCl_4(SC_4N_2H_3)]$

To a solution of $[Bu_4N]^+[TcOCl_4]^-$ (100 mg, 0·2 mmol) in dry methanol (15 ml) was added 2-mercaptopyrimidine (56 mg, 0·5 mmol). A brown solution formed and the mixture was stirred at room temperature for 18 h. After this time the mixture was filtered to remove a black solid and the filtrate volume reduced by evaporation (to *ca* 10 ml). Diethyl ether (6 ml) was then added and the mixture cooled to 273–278 K for 72 h. The red–brown solid which separated was recrystallized from dichloromethane/diethyl ether mixtures (16 mg, 13%). A crystal of size 0·3 × 0·4 × 0·4 mm was selected from this material for X-ray analysis.

Measurements were made on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation. Lattice parameters were determined from the setting angles of 25 reflections (θ 12–18°). Intensity data were measured with ω–2θ scans in the range $2 < \theta < 24°$, index range $h \pm 12$, k −11 to 13, l 0 to 13. Two standard reflections measured every 2 h showed no significant variation over the period of data collection. 5451 reflections were scanned of which 4319 were unique, $R_{int} = 0{\cdot}019$, and 3670 were considered observed $[F > 5\sigma(F)]$ and were used in the analysis. No absorption correction was applied. The structure was solved by Patterson and Fourier methods. The H atoms of the pyrimidine ring were located in a difference Fourier map and included in the refinement at fixed positions. The H atoms of the cation were placed in calculated positions 'riding' on their bonded C atoms. Coordinates were refined for the non-H atoms using full-matrix least squares on F values with weights $w = 1/\sigma^2(F)$ from counting statistics. The atoms of the complex anion were assigned anisotropic thermal parameters; those of the cation were refined isotropically. The H atoms of the anion and those of the cation were each assigned one overall isotropic temperature factor (0·07 and 0·08 Å^2 respectively). The refinement was terminated when all shift/e.s.d. ratios were less than 0·01 and $R = 0{\cdot}058$, $wR = 0{\cdot}083$ for the 3670 observed reflections. The residual electron density

Table 1. *Fractional atomic coordinates* (× 10^4) *with e.s.d.'s in parentheses and isotropic temperature factors* (Å^2 × 10^3)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{23}\cos\alpha + 2U_{13}\cos\beta + 2U_{12}\cos\gamma).$$

	x	y	z	U_{eq}/U
Tc	1904 (1)	1871 (1)	1954 (1)	33
Cl(1)	2998 (2)	241 (2)	3359 (2)	38
Cl(2)	727 (2)	3542 (2)	721 (2)	38
Cl(3)	534 (1)	302 (2)	2128 (2)	31
Cl(4)	3365 (2)	1901 (2)	229 (2)	40
S	2763 (2)	3463 (2)	2364 (2)	42
N(1)	825 (5)	2171 (6)	3445 (6)	35
C(2)	1421 (7)	3141 (7)	3451 (7)	36
N(3)	1020 (7)	3740 (7)	4188 (7)	47
C(4)	−72 (9)	3323 (9)	4981 (9)	52
C(5)	−743 (8)	2358 (9)	5035 (8)	50
C(6)	−281 (8)	1768 (8)	4239 (8)	45
N(11)	7328 (5)	2279 (6)	−83 (5)	36 (1)*
C(11)	6708 (7)	1002 (7)	815 (7)	43 (2)*
C(12)	7099 (8)	338 (8)	2061 (8)	53 (2)*
C(13)	6440 (9)	−879 (9)	2835 (9)	64 (3)*
C(14)	6686 (11)	−1509 (12)	4112 (11)	91 (3)*
C(15)	8680 (7)	1993 (7)	−529 (7)	42 (2)*
C(16)	9416 (7)	3135 (7)	−1528 (7)	42 (2)*
C(17)	10736 (7)	2720 (8)	−1865 (8)	50 (2)*
C(18)	11580 (9)	3847 (9)	−2747 (9)	66 (3)*
C(19)	6664 (7)	2926 (7)	−1130 (7)	39 (2)*
C(20)	6695 (8)	2235 (8)	−1965 (8)	54 (2)*
C(21)	5940 (9)	2987 (9)	−2953 (9)	59 (2)*
C(22)	6443 (9)	4271 (10)	−3868 (9)	71 (3)*
C(23)	7314 (7)	3187 (7)	552 (7)	41 (2)*
C(24)	6051 (8)	3496 (9)	1119 (8)	57 (2)*
C(25)	6185 (9)	4213 (9)	1914 (9)	67 (3)*
C(26)	6602 (12)	3371 (13)	3089 (12)	103 (4)*

* Refined isotropic temperature factor.

Table 2. *Bond lengths* (Å) *and angles* (°) *in the complex anion*

Tc–Cl(1)	2·384 (2)	N(1)–C(2)	1·360 (10)
Tc–Cl(2)	2·366 (2)	C(2)–N(3)	1·333 (10)
Tc–Cl(3)	2·396 (2)	N(3)–C(4)	1·357 (11)
Tc–Cl(4)	2·346 (2)	C(4)–C(5)	1·373 (13)
Tc–N(1)	2·087 (6)	C(5)–C(6)	1·393 (12)
Tc–S	2·429 (2)	C(6)–N(1)	1·368 (10)
S–C(2)	1·713 (8)		
Cl(1)–Tc–Cl(2)	174·4 (1)	Cl(4)–Tc–S	100·6 (1)
Cl(1)–Tc–Cl(3)	91·0 (1)	N(1)–Tc–S	67·1 (2)
Cl(1)–Tc–Cl(4)	92·3 (1)	Tc–S–C(2)	80·5 (3)
Cl(1)–Tc–N(1)	88·8 (2)	Tc–N(1)–C(2)	102·9 (5)
Cl(1)–Tc–S	88·9 (1)	Tc–N(1)–C(6)	137·8 (6)
Cl(2)–Tc–Cl(3)	90·2 (1)	C(6)–N(1)–C(2)	118·9 (7)
Cl(2)–Tc–Cl(4)	92·8 (1)	N(1)–C(2)–N(3)	125·1 (7)
Cl(2)–Tc–N(1)	85·8 (2)	C(2)–N(3)–C(4)	115·5 (7)
Cl(2)–Tc–S	88·1 (1)	N(3)–C(4)–C(5)	123·5 (8)
Cl(3)–Tc–Cl(4)	98·8 (1)	C(4)–C(5)–C(6)	118·6 (8)
Cl(3)–Tc–N(1)	93·4 (2)	C(5)–C(6)–N(1)	118·4 (8)
Cl(3)–Tc–S	160·6 (1)	S–C(2)–N(1)	109·1 (6)
Cl(4)–Tc–N(1)	167·7 (2)	S–C(2)–N(3)	125·7 (6)

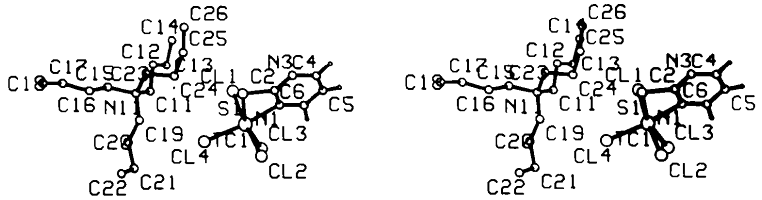

Fig. 1. Stereoscopic view of the anion and cation as they occur in the crystal, showing the atom-numbering scheme. The H atoms of the cation have been omitted.

in a final difference map was within ±1·15 e Å^{-3} with all main peaks close to Cl atoms. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974); computations were carried out with *SHELX* (Sheldrick, 1978) and *PLUTO*78 (Motherwell & Clegg, 1978).

Discussion. The product from the reaction between $[\mathrm{Bu_4N}]^+[\mathrm{TcOCl_4}]^-$ and 2-mercaptopyrimidine was expected to be $[\mathrm{TcO(SC_4N_2H_3)_2}]^+$ in which the four chloride ligands had been displaced by two thiolate ligands. However, the X-ray diffraction study revealed that the product isolated was the Tc$^{\mathrm{IV}}$ complex $[\mathrm{Bu}_4^n\mathrm{N}]^+[\mathrm{TcCl_4(SC_4N_2H_3)}]^-$, not a complex of Tc$^{\mathrm{V}}$. Atomic coordinates are given in Table 1* and bond lengths and angles for the complex anion are in Table 2. The structure of the complex is illustrated in Fig. 1.

Formation of such a Tc$^{\mathrm{IV}}$ complex from a Tc$^{\mathrm{V}}$ precursor by one-electron reduction and oxygen abstraction is unusual, although such a reduction has been reported to occur in the reaction between $[\mathrm{TcOCl_4}]^-$ and a dithiocarbamate ligand (duPreez, Gerber & Knoesen, 1985). In this case a neutral Tc$^{\mathrm{IV}}$ tetrakisdithiocarbamate complex was formed in 87% yield. A more likely explanation for the formation of the title compound would be the presence of $[\mathrm{TcCl_6}]^{2-}$ as an impurity in the $[\mathrm{TcOCl_4}]^-$ used. Direct chloride substitution by the thiolate ligand would then lead to the observed Tc$^{\mathrm{IV}}$ complex. A Tc analysis of the $[\mathrm{Bu}_4^n\mathrm{N}][\mathrm{TcOCl_4}]$ used was carried out by scintillation methods. The values of 17·9 and 18·5% obtained are consistent with the presence of *ca* 20% $[\mathrm{Bu}_4^n\mathrm{N}]_2[\mathrm{TcCl_6}]$ in the $[\mathrm{Bu}_4^n\mathrm{N}][\mathrm{TcOCl_4}]$ precursor. This would in turn be in accord with the low yield of crystalline product obtained from the reaction.

The essentially octahedral coordination about Tc is distorted by the small N—Tc—S angle (see Table 2) of the four-membered chelate ring. The Cl—Tc—Cl angle *trans* to this angle is enlarged to 98·8 (1)°. Despite the angular distortions, the atoms Tc, Cl(3), Cl(4), S, N(1), forming the basal plane of the octahedron, are coplanar to within ±0·01 (1) Å. The four-membered ring deviates by up to ±0·04 (1) Å from planarity. Formation of the chelate ring imposes considerable angular strain at N(1) and C(2), the exocyclic angles being greater by 34·9° at N(1) and by 16·6° at C(2), than the corresponding ring angle. The pyrimidine ring is planar to within the limits of experimental error. Excluding the two axial Cl atoms, Cl(1) and Cl(2), the remaining ten non-H atoms of the anion are coplanar to within ±0·10 (1) Å.

In the structure of the analogous tetrachloro(salicylaldehydato)technetate(IV) in crystals of the tetraphenylphosphonium salt (Mazzi, Roncari, Bandoli & Clemente, 1982) the octahedral coordination geometry about Tc is less distorted. Here chelation involves a six-membered ring and the maximum deviation of an angle at Tc from 90 or 180° is only 6·5°. The Tc—Cl bond lengths in this anion range from 2·31 to 2·36 Å, mean 2·33 Å, somewhat shorter than the mean Tc—Cl length of 2·373 Å in the title compound. This is in accord with the more basic S and N donor atoms of the mercaptopyrimidine producing higher electron density on Tc than the more electronegative O donor atoms of the salicylaldehyde ligand.

The tetrabutylammonium counter ion has two of the butylammonium chains in the extended antiperiplanar–antiperiplanar conformation, with N—C—C—C and C—C—C—C torsion angles all within 7 (1)° of 180°. The other two butylammonium chains have antiperiplanar–synclinal conformations [torsion angles 178 (1), 66 (1) and 170 (1), −72 (1)°]. Bond lengths in the cation are normal, C—N 1·50 (1)–1·54 (1) Å and C—C 1·49 (1)–1·56 (1) Å.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles for the cation have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44056 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetraphenylphosphonium (Bipyridine)tetrachloromolybdate(III) $[PPh_4][MoCl_4(bpy)]$

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Abstract. $[P(C_6H_5)_4][MoCl_4(C_{10}H_8N_2)]$, $M_r = 733{\cdot}34$, triclinic, $P\bar{1}$, $a = 9{\cdot}211\,(1)$, $b = 12{\cdot}840\,(1)$, $c = 14{\cdot}441\,(2)$ Å, $\alpha = 104{\cdot}31\,(1)$, $\beta = 101{\cdot}77\,(2)$, $\gamma = 90{\cdot}91\,(1)°$, $V = 1616{\cdot}1\,(8)$ Å^3, $Z = 2$, $D_x = 1{\cdot}51$ Mg m^{-3}, D_m not measured, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 8{\cdot}04$ cm^{-1}, $F(000) = 742$, $T = 272$ K, $R = 0{\cdot}035$ for 4891 reflexions with $I \geq 3\sigma(I)$. Reaction of $[MoCl_4(bpy)]$ with Li_2S_2 in tetrahydrofuran gives $Li[MoCl_4(bpy)]$. Metathesis using PPh_4Br forms the deep purple title complex, which has been structurally characterized by X-ray crystallography. The Mo atom is surrounded by four Cl ions and a bipyridine ligand in a distorted octahedral arrangement. Average Mo—Cl distance of 2·43 Å is consistent with values found in other Mo^{III} complexes. Two C—C distances of 3·37 and 3·38 Å occur between bipyridine ligands across the centre of symmetry.

Introduction. The EXAFS data on nitrogenase and other molybdoenzymes (Cramer, Hodgson, Gillum & Mortenson, 1978; Cramer *et al.*, 1978) has stimulated intense research interest in the preparation of Mo–S complexes. We have studied the reaction of molydenum complexes with various sulfur-containing reagents (Povey & Richards, 1984; Povey, Richards & Shortman, 1986) and in continuation of this work lithium sulfide was investigated as a potential source of sulfur ligation.

With the aim of metathesising chloride for sulfide, the complex $[MoCl_4(bpy)]$ was treated with Li_2S_2, but reduction of the Mo centre occurred with the elimination of S to yield the red moisture-sensitive complex $Li[MoCl_4(bpy)]$. Subsequent treatment by PPh_4Br in methanol converted the lithium complex to the deep purple tetraphenylphosphonium analogue which is the subject of this structural investigation.

Experimental. Accurate unit-cell parameters were measured on a CAD-4 diffractometer using 25 accurately centred reflexions ($21 \leq \theta \leq 23°$) from a crystal of dimensions 0·3 × 0·02 × 0·15 mm. A full hemisphere of reciprocal space was measured ($0 \leq h \leq 11$, $-15 \leq k \leq 15$, $-17 \leq l \leq 17$), θ limit 26°, $\omega/2\theta$ scan, scan speed 3·3° min^{-1}, 002 reflexion monitored hourly. Variation of its intensity was insignificant during the data-collection period and after data reduction from a total of 6343 unique reflexions 4891 had $I \geq 3\sigma(I)$. Intensity statistics indicated a centrosymmetric distribution and space group $P\bar{1}$ was assumed.

The solution to the structure was not straightforward. Routine application of the direct-methods program *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) failed to produce a refinable structure in the first six solutions although all the heavy-atom peaks satisfied the vector distribution in the Patterson map. Reducing the space group to $P1$, *MULTAN* then produced the correct structure from which the centre of symmetry was deduced. The correct solution in space group $P\bar{1}$ proved to be the eighth solution.

From an electron density map phased upon Mo, four Cl and P the coordinates of the four phenyl groups were obtained and refinement of structure amplitudes converged at $R = 0{\cdot}105$ with H atoms at calculated positions ($d_H = 1{\cdot}0$ Å). An absorption correction by *DIFABS* (Walker & Stuart, 1983) reduced R to 0·076 with minimum and maximum corrections 0·661 and 1·215 respectively. Full-matrix anisotropic refinement

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