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COORDINATION CHEMISTRY REVIEWS

Technetium 1995

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Contents

Introduction

This review covers the coordination chemistry of technetium published during 1995, and follows the format of the 1994 survey [1]. The literature has been searched using Current Contents and the Cambridge Crystallographic Data Base, implemented through the ETH, Zurich [2]. The article is not fully comprehensive, but provides a representative coverage of the literature, and most organometallic complexes are excluded. Although detailed coverage of medical applications is not given, papers dealing with coordination complexes with potential applications are discussed in the final section of the review. Some references from the tail-end of 1994 which were missed from last year's survey are included here.

Complexes are classified according to the formal oxidation state of the technetium centre, and then subdivided by ligand donor atom. However, within technetium(V) chemistry, the presence of a $Tc{=}O$ or $Tc{\equiv}N$ group has taken priority over the donor atoms possessed by the other ligands in the complexes.

A review (with 294 references) covering details of the structural features of 240

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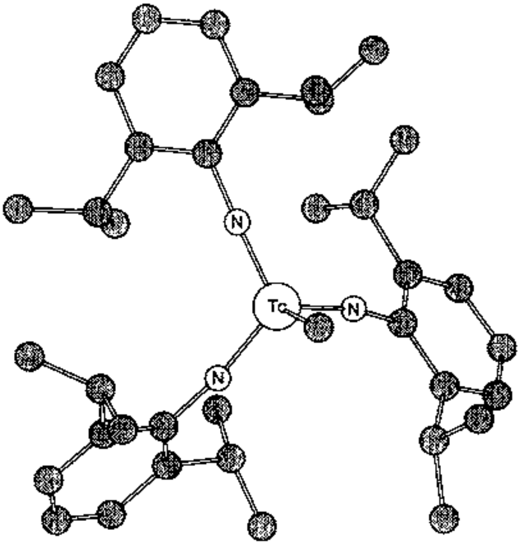
crystallographically characterized technetium complexes has appeared. It deals with the literature during the period 1986–1992, and provides an excellent source of structural information [3]. Another paper that spans various types of technetium complexes concerns X-ray absorption near edge structure spectroscopy; the edge positions span a range of 19.9 eV (Tc metal to $[TcO_4]^-$) and the results show a correlation between chemical shift and formal oxidation state [4].

Caution: ^{99}Tc is a weak β^- emitter ($E=0.292$ MeV, $t_{1/2}=2.12\times10^5$ yr).

1. Technetium(VII)

The preparation of the complex tris(2,6-diisopropylphenylimido) methyltechnetium (1) has been reported. The results of a crystal structure determination of the diethyl ether solvated species reveals that the technetium(VII) centre is in a tetrahedral environment. Each imido nitrogen atom is close to being linear with angles C–N–Tc in the range ≈164–168° [5].



(1)

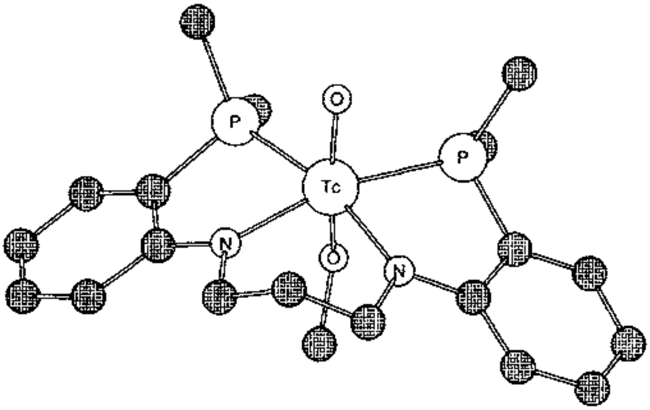
2. Technetium(VI)

The μ-oxo complex [(pic)$_2$(N)Tc–O–Tc(N)(pic)(Hpic)Cl] is one of two products obtained from the reaction of picric acid (Hpic) with $[Bu_4N][TcNCl_4]$. The product has been characterized by visible, IR, ESR and NMR spectroscopic methods [6].

3. Technetium(V)

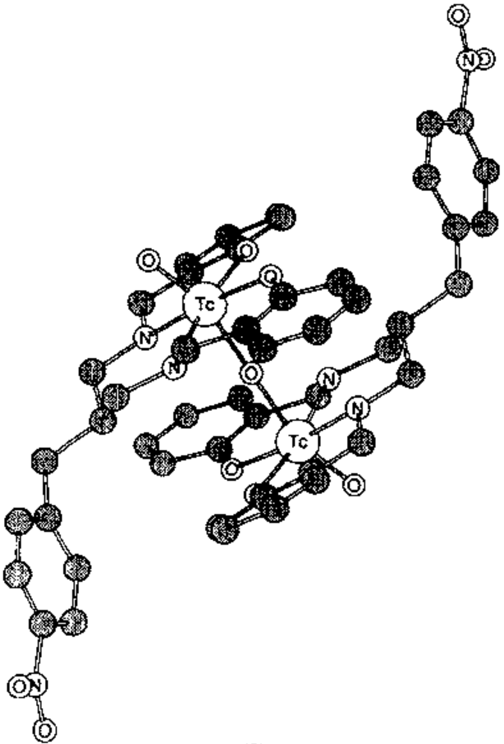
3.1. Oxo complexes

Treatment of $[Bu_4N][TcO_4]$ with *N,N'*-bis(2-(diphenylphosphino)phenyl) propane-1,3-diamine(H_2L) and $[Bu_4N]Cl$ leads to the formation of the complex [Tc(O)LCl]. Members of a series of related species of type [Tc(O)LX] in which X = OH, OMe, OEt or O_2CCF_3 have also been prepared and characterized, and the X-ray crystal structure of the methoxy derivative (**2**) has been determined. The results confirm that the coordination sphere is distorted octahedral and that the methoxy and oxo groups are mutually *trans*. The $Tc–O_{oxo}$ bond distance is 169.1(2) pm and the $Tc–O_{methoxy}$ bond length is 203.0(2) pm [7].



(**2**)

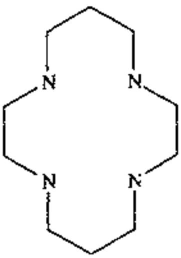
Starting from $[TcOCl_4]^-$ and H_2L {H_2L = *N,N'*-bis(2-hydroxybenzyl)-1,3-diamino-2-(4-nitrobenzyl)propane}, the technetium(V) complex (**3**) has been prepared. Crystallographic data confirm the dinuclear nature of the product and that the ligand coordinates as L^{2-} through the two phenolate oxygen atoms and the two amino groups. Each technetium(V) centre is in a distorted-octahedral environment, with a bridging oxo ligand spanning the two metal centres. The O=Tc-Tc=O unit is close to being linear and important distances are $Tc–O_{terminal}$ = 165.5(7) pm and $Tc–O_{bridge}$ = 190.3(1) pm [8]. An analogous rhenium(V) complex has also been prepared and structurally characterized. A μ-oxo complex has been reported to be one of the products obtained from the reaction of picric acid (Hpic) with $[Bu_4N][TcNCl_4]$ (see also Section 2). When Hpic reacted with $[Bu_4N][TcOCl_4]$, the monomeric complex $[Tc(O)(Pic)_2Cl]$ was isolated. This compound has been characterized by visible, IR, ESR and ^1H and ^{99}Tc NMR spectroscopies [6]. The direct reaction between $[TcO_4]^-$ and alkaline $NaBH_4$ in the presence of *N*-acetylanthranilic acid (H_2L) has been reported to give the complex $[Tc(O)L_2(H_2O)]^-$, characterized by elemental analysis and spectroscopic methods. Reversed phase HPLC was used to isolate the product [9].



(3)

The reactions of an excess of ligand (**4**), (**5**) or (**6**) (L) with $[TcCl_4(PPh_3)_2]$ or $[TcCl_3(MeCN)(PPh_3)_2]$ in acetonitrile provide improved methods of synthesis of the dioxo complexes *trans*-$[Tc(O)_2L]^+$. A similar methodology was also applied to the preparations of *trans*-$[Tc(O)_2L']^+$ where L′ is en, 1,3-pn or 1,5,8,12-tetraazadodecane. Product characterization was by elemental analysis, IR spectroscopy, mass spectrometry and magnetic susceptibility measurements [10].

A new synthesis of (**7**)·HCl has been detailed. This and ligand (**8**) have been



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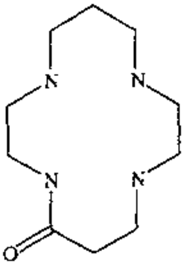

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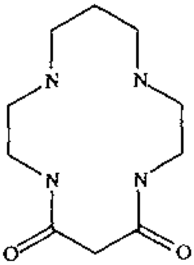

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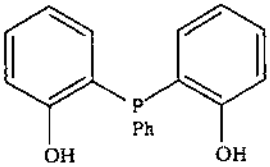

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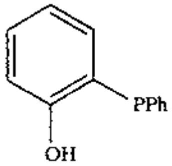
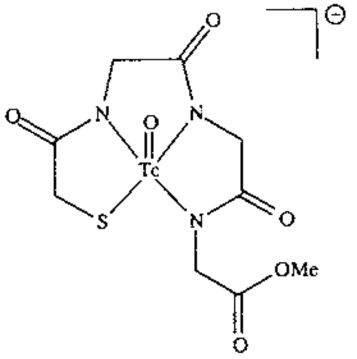


(8)

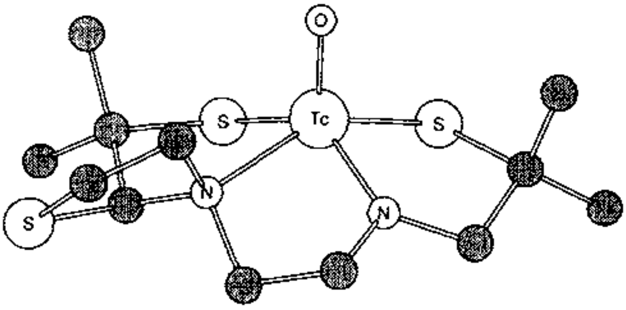
incorporated into the technetium(V) oxo complexes [Tc(O)Cl(**8**–H)$_2$] and [Tc(O)(**8**–2H)(**8**–H)]. Several related rhenium(V) complexes were also described. The products were characterized by IR and ^{1}H NMR spectroscopies and mass spectrometry, and structural data are available for rhenium members of this family of compounds [11].

The reaction between [^{99g}TcOCl$_4$]$^-$ and mercaptoacetylglycylglycylglycine (mag$_3$) leads to the formation of the oxo complex [^{99g}Tc(O)(mag$_3$)]$^-$ which has been characterized by UV–VIS and ^{1}H and ^{13}C NMR spectroscopies, mass spectrometry and an X-ray diffraction study of the [Ph$_4$As]$^+$ salt. The technetium(V) centre is in a square-pyramidal environment with the oxo group in the apical site (Tc–O = 164.7(3) pm). The radiopharmaceutical [^{99m}Tc(O)(mag$_3$)]$^-$ has also been synthesized and proven to be chemically identical to [^{99g}Tc(O)(mag$_3$)]$^-$. A side product of the synthesis of [^{99g}Tc(O)(mag$_3$)] was its methyl ester derivative, (**9**), formed in 14–16% yield. Two polymorphs of the salt [PPh$_4$][(**9**)]·2H$_2$O have been found to exist in the solid state, and X-ray diffraction studies show that they differ in the conformations of the ligand [12]. The technetium(V) complex (**10**) has been prepared and the square-pyramidal structure confirmed by the results of an X-ray diffraction investigation (Tc–O = 166.5(5) pm). This work includes a detailed study of the ligand synthesis and the unexpected formation of the thiazolidine ring [13].



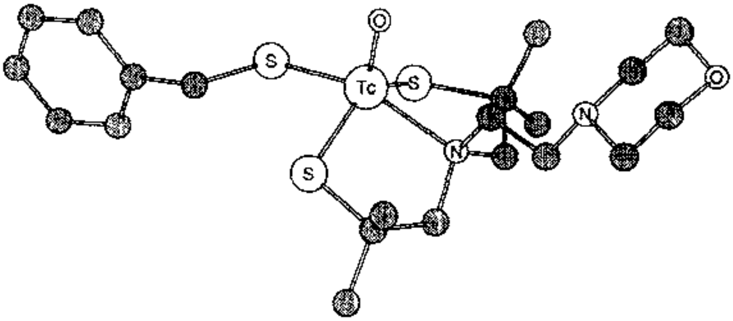
(9)

The syntheses of ligands of the type RCH$_2$CH$_2$N(CH$_2$CMe$_2$SH)$_2$ in which R = NEt$_2$, piperidin-1-yl, pyrrolidin-1-yl or morpholin-4-yl have been described. Each ligand (H$_2$L) reacts with technetium(V) gluconate and benzyl mercaptan (HL′) to give a complex of the form [Tc(O)LL′] and these products have been



(10)

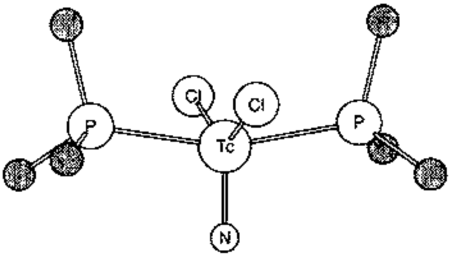
characterized by elemental analysis and ^1H and ^{13}C NMR spectroscopies. For R = morpholin-4-yl, the structure of the product (11) has been confirmed from the results of an X-ray diffraction study. The metal centre is in a distorted square pyramidal environment and the nitrogen-attached RCH_2CH_2 side chain has a *syn* configuration. Results of nOe NMR (nuclear Overhauser effect NMR) spectroscopic studies are consistent with this configuration being retained in solution for each of the complexes [14].



(11)

3.2. *Nitrido complexes*

The reaction between $[Bu_4N][TcOCl_4]$ and $2-NH_2C_6PPh_2(H_2L)$ leads to the formation of the complex $[TcCl_2(L)(HL)]$. Structural data for the rhenium(V) analogue show that the HL^- and $L2^-$ ligands are twisted with respect to each other about the metal centre. When $2\text{-}NH_2C_6H_4PPh_2(H_2L)$ reacts with either $[Bu_4N][TcNCl_4]$ or $[TcNCl_2(PPh_3)_2]$, the technetium(V) nitrido complex $[Tc(N)Cl(H_2L)_2]Cl$ was obtained. The MeCN solvate of this compound has been structurally characterized, and a distorted octahedral environment for the metal centre confirmed; the coordinated chloro ligand is *trans* with respect to the nitrido group, and each H_2L ligand acts as an *N*,*P*-chelate with the nitrogen donors of the two ligands mutually *cis*. The $Tc–N_{nitrido}$ bond distance of 162.7(3) pm compares with values of 217.8(3) and 220.5(4) pm for the $Tc–N_{amino}$ bond lengths [15]. The X-ray crystal structure of $[Tc(N)Cl_2(PPh_3)_2]$ (**12**) has been determined; pertinent angles are P–Tc–P = 162° and Cl–Tc–Cl = 140.6° [16]. The synthesis and structural characterization of the tetraphenylborate salt of $[Tc(N)(H_2O)(HL)]^+$ in which ligand H_2L is 3,3,9,9-tetramethyl-4,8-diazaundecan-2,10-dione dioxime have been described. The technetium(V) centre is in a pseudo-octahedral site with the four nitrogen donor atoms occupying the equatorial plane, such that the aqua and nitrido groups are mutually *trans* [17].

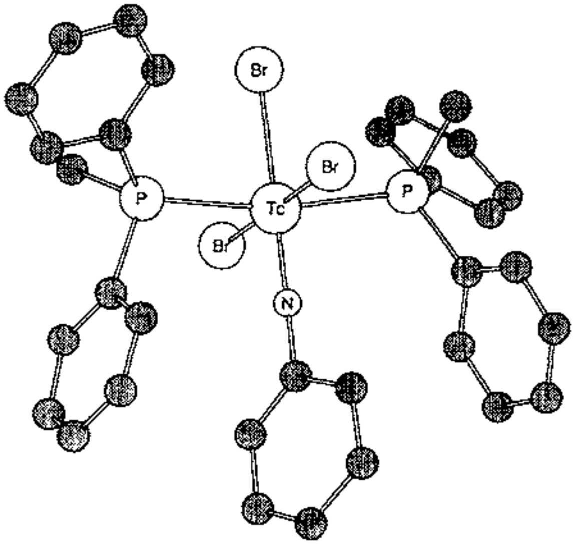


Only the *ipso*-C atoms of the Ph groups are shown

(**12**)

3.3. *Other complexes*

The reaction between $[NH_4][TcO_4]$ and PhNHNHC(O)Me, $PMePh_2$ and HCl or HBr in methanol leads to the formation of the phenylimido complexes $[Tc(NPh)(PMePh_2)_2X_3]$ (X = Cl or Br). Single crystal structural data reveal that the phosphine ligands in both products adopt a *trans*-arrangement. Structure (**13**) shows the tribromo derivative. With the less sterically demanding phosphine, PMe_2Ph, the reaction (using HBr) produces the phenyldiazenido complex $[Tc(NNPh)(PMe_2Ph)_3Br_2]$ and the formation of the phenyldiazenido ligand from the PhNHNHC(O)Me starting material is regarded as being particularly novel. The crystal structure of $[Tc(NNPh)(PMe_2Ph)_3Br_2]$ has been determined; the phosphine ligands are in a *mer*-arrangement. The Tc–N–N and $N–N–C_{Ph}$ bond angles are 172(1)° and 121(1)°, respectively, and the Tc–N and N–N bond lengths are 177.0(11) and 120.8(16) pm. The formal charge of the PhNN unit is considered to be −3, consistent with an oxidation state of +5 for the metal centre [18]. The phenylimido complex $[TcCl_3(NPh)(PPh_3)_2]$ reacts with an excess of PMe_2Ph in methanol at reflux to form $[TcCl_2(NPh)(PMe_2Ph)_3]^+$ which has been isolated as the tetraphenylborate salt. X-ray crystallographic data for the compound have been reported, and significantly, represent the first such report of a *cationic* technetium(V) organoimido species. The Tc centre is in a distorted octahedral environment with the PMe_2Ph ligands mutually *mer*, the Tc–N distance is 171.1(2) pm. The new complex has also been characterized by ^1H NMR and IR spectroscopies [19].



(13)

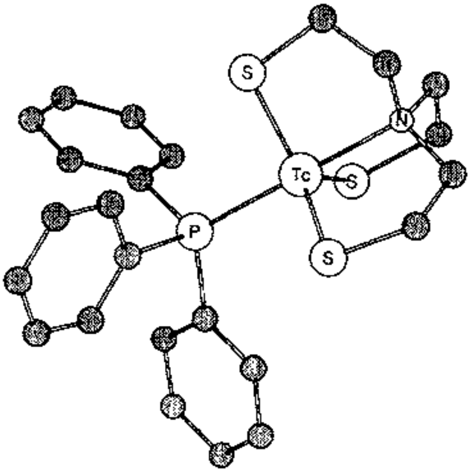
4. Technetium(III)

As part of a study that also incorporates dinuclear species (see below), the compound *trans*-$[TcCl_2(NCMe)_4][BF_4]$ has been prepared and spectroscopically and structurally characterized [20].

The reaction between $K[TcO_4]$ and PPh_3 in the presence of the tripodal ligand $N(CH_2CH_2SH)_3$ (H_3L) leads to the formation of the technetium(III) complex $[TcLPPh_3)]$ (**14**) for which ^1H NMR spectroscopic data have been recorded. Crystallographic data have confirmed that the technetium centre is in a trigonal bipyramidal environment with the three sulfur donor atoms in the equatorial plane. The same product has been obtained from the reaction of H_3L with $[TcCl_3(PPh_3)_2(NCMe)]$. A series of related rhenium complexes has also been prepared and characterized [21]. The single crystal structure of the complex *trans*-$[TcCl_2\{S_2P(OEt)_2\}(PMe_2Ph)_2]$ (**15**) has been elucidated. The technetium(III) centre is octahedrally coordinated with the $S_2P(OEt)_2$ acting as a chelate [22].

5. Technetium(I)

Some overlap with organometallic chemistry is inevitable when discussing technetium(I) species, but the compounds chosen for inclusion in this section should also have appeal to those with a non-organometallic bias. The anions $[Tc(CO)_3X_3]^{2-}$ ($X = Cl$ or Br) have been prepared by the carbonylation (at atmospheric pressure) of $[TcOCl_4]^-$ or $[TcO_4]^-$ in the presence of $BH_3 \cdot THF$ and chloride or bromide ion. The complexes have been isolated as the tetraethylammonium salts



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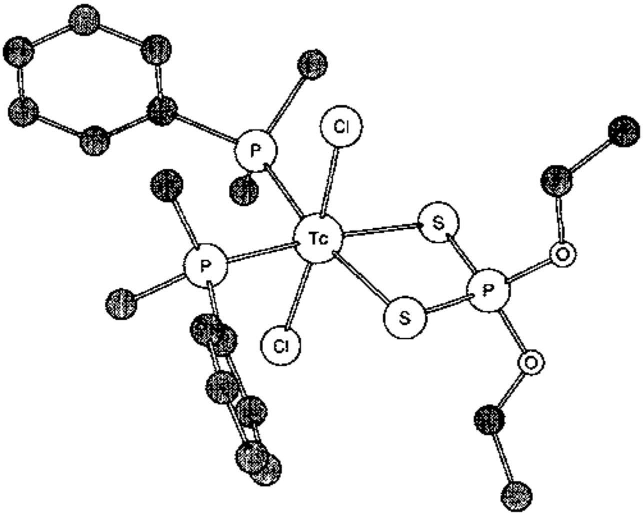
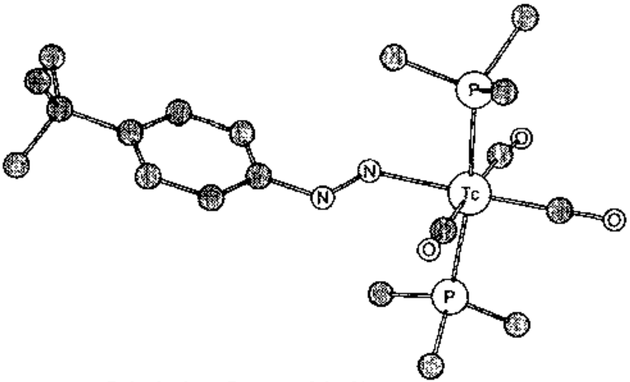


(15)

and have been shown to undergo substitution of the halide ligands, for example to yield $[Tc(CO)_3Cl(CN^tBu)_2]$ and $[Tc(CO)_3(H_2O)_3]^+$. The latter species is particularly novel; it is stable in water for several days [23].

The phosphine complexes $[CpTc(CO)_2(PPh_3)]$ and $[Cp^*Tc(CO)_2(PPh_3)]$ have been synthesized by phosphine-for-carbonyl ligand substitution reactions, initiated by photolysis. Both products have been spectroscopically and crystallographically characterized. A photolytic route was also used to prepare $[(HBpz^*_3)Tc(CO)_2\{P(OMe)_3\}]$ [$HBpz^*_3$ = hydridotris(3,5-dimethylpyrazolyl)borate], the crystal structure of which has been determined. Comparisons have been made with analogous rhenium(I) species [24]. The reaction of $[TcL_6]^{3+}$, where L = thiourea, with ${}^{t}BuNC$ and dppe results in the formation of the mixed ligand complex *trans*-$[Tc(dppe)_2({}^{t}BuNC)_2]^+$ which has been isolated as the hexafluorophosphate salt. An X-ray diffraction study has confirmed that the isocyanide ligands are mutually *trans* within the distorted octahedral coordination shell of the technetium(I) centre. The complex cation undergoes a reversible one-electron oxidation with $E_{1/2}=0.91$ V (versus SCE) [25].

Treatment of the trihydride complex $[TcH_3(PPh_3)_4]$ with CO in benzene is reported to give $[TcH(CO)_3(PPh_3)_2]$. This complex reacts with $[4\text{-}{}^{t}BuC_6H_4N_2][PF_6]$ to generate the salt $[Tc(CO)_2(PPh_3)_2(HNNC_6H_4\text{-}4\text{-}{}^{t}Bu)][PF_6]$ which has been the subject of an X-ray diffraction study (at −72°C). The structure of the cation is shown in (**16**). In the octahedral coordination sphere of the metal centre, the phosphine ligands adopt a *trans*-arrangement. Deprotonation of complex (**16**) can be readily achieved to give $[Tc(CO)_2(PPh_3)_2(NNC_6H_4\text{-}4\text{-}{}^{t}Bu)]$. A wider study of the reactivity of $[TcH_3(PPh_3)_4]$ has also been included in this work, and products include $[Tc(CO)_2(\eta^2\text{-}S_2CH)(PPh_3)_2]$, $[Tc(CO)_2(\eta^2\text{-}N(Me)SCH)(PPh_3)_2]$ and species with Tc–C bonds [26].

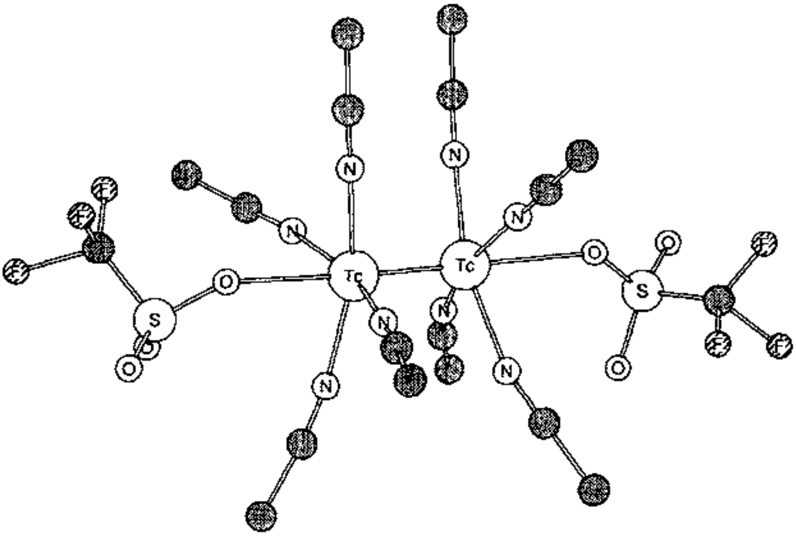


Only the *ipso*-C atoms of the Ph groups are shown

(**16**)

6. Dinuclear and polynuclear complexes

Two oxo-bridged complexes have already been mentioned [6,8]. Reports of ditechnetium complexes with direct metal–metal bonding include that of an EXAFS study of $Tc_2(CO)_{10}$. Results indicate that multiple scattering along the Tc–C vector is dominant over Tc…O scattering. This paper describes a wide-ranging investigation of EXAFS and X-ray absorption near edge structure spectroscopy of technetium-containing compounds [4]. Starting from $[Tc_2Cl_4L_4]$ (L = phosphine) and $HBF_4.Et_2O$ in MeCN and CH_2Cl_2, the complex $[Tc_2(NCMe)_{10}][BF_4]_4$ may be isolated in high yield. A second route to this compound involves the reduction of $[Bu_4N]_2[TcCl_6]$ and subsequent reaction with $HBF_4 \cdot Et_2O$ in MeCN. Treatment of $[Tc_2Cl_8]^{2-}$ with $HBF_4 \cdot Et_2O$ in MeCN and Et_2O generates $[Tc_2(NCMe)_{10}][BF_4]_4$ and $[TcCl_2(NCMe)_4][BF_4]$ (see Section 4). The addition of $Tl[O_3SCF_3]$ to $[Tc_2(NCMe)_{10}][BF_4]_4$ followed by recrystallization from MeCN/Et_2O/hexane permits the isolation of $[Tc_2(NCMe)_8(OSO_2CF_3)_2][BF_4]_2$, containing cation (17). Structural data for the complex $[(17)][BF_4]_2 \cdot MeCN$ reveal that the trifluoromethanesulfonato ligands occupy the two axial sites, with the $Tc_2(NCMe)_8$ unit having a staggered conformation and; the Tc–Tc bond distance is 212.2(1) pm [20].



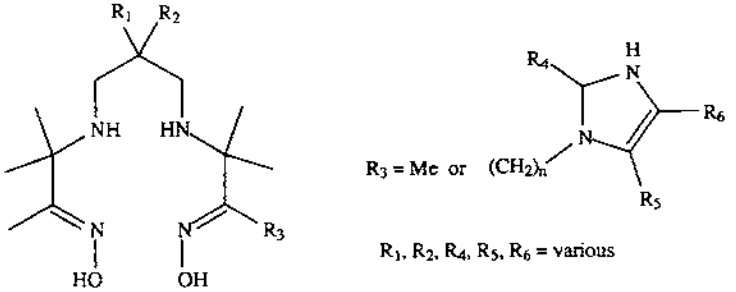
(17)

Two interesting polynuclear technetium complexes were described in 1995. The crystallographic characterization of the complex $[Tc(CO)_3I]_4$ shows that it possesses a cubane-like core with the $Tc(CO)_3$ groups bridged by iodide ligands [27]. The tris(ferrocenium) salt of $[Tc_6I_{14}]^{3-}$ has been prepared and its structure determined by X-ray diffraction studies. The Tc_6-core of the anion is a trigonal prism and possesses six terminal, six edge-bridging and two triply bridging iodo ligands. The ferrocenium cations are disordered [28].

7. ^{99m}Tc labelling studies

This section describes selected studies involving the incorporation of ^{99m}Tc into coordination complexes and their applications in medicine. A review entitled "Technetium radiopharmaceuticals: fundamentals, synthesis, structure and development" provides excellent up-to-date coverage [29], and a second review [30] examines suitable chelating agents for ^{99m}Tc.

Studies related to the radiopharmaceutical $[^{99m}Tc(O)(mag_3)]^-$ were described earlier [12]. The syntheses of phenylethylamine complexes of ^{99m}Tc have been reported, and a study to evaluate the biological significance of the species has been carried out; the ligands involved were 2-(4,5-dimethoxy-2-nitrophenyl)ethylamine, 2-(3,4-dimethoxyphenyl)ethylamine and 2-(2-amino-4,5-dimethoxyphenyl) ethylamine [31]. Amino acid complexes containing ^{99m}Tc multiply bonded to nitrogen have been prepared. One example contains cysteamine, and studies have shown that species containing this ligand are retained in renal tissue [32]. Details of the synthetic routes to prepare compounds (**18**) have been described, the interest in these dioximes being their role as ligands for ^{99m}Tc with a potential for imaging hypoxic tissue [33]. Starting from $[TcO_4]^-$, cationic $[TcL_3]^+$ complexes, in which L is derived from *N*-substituted 2-methyl-3-hydroxy-4-pyridone, may be readily prepared using either ^{99}Tc or ^{99m}Tc. The reducing agent is $Na_2S_2O_5$ or $Na_2S_2O_4$. The method is a rapid means of radiolabelling with high radiochemical purity and yield. Tests in rabbits and mice indicate that the complexes may have applications as kidney imaging agent. An important result of the study is that it confirms the formation of chemically identical $[TcL_3]^+$ species at both the no-carrier added and carrier-added levels [34].



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