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Note

Reactions of the tridentate and tetradentate amine ligands di-(2-picolyl)(*N*-ethyl)amine and 2,5-bis-(2-pyridylmethyl)-2,5 diazohexane with technetium nitrosyl complexes

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ABSTRACT

The reaction of the Tc(II) nitrosyl complex $(Bu_4N)[Tc(NO)Cl_4]$ with di-(2-picolyl)(*N*Et)amine in methanol yields the neutral complex [Tc(NO)Cl(py-*N*(Et)-py)]. The reaction of the Tc(I) nitrosyl complex $[Tc(NO)Cl_2(HOMe)(PPh_3)_2]$ with this tridentate ligand yields cationic $[Tc(NO)Cl(py\text{-}N(Et)\text{-}py)(PPh_3)]Cl$. These two complexes have been structurally characterized. The reaction of $[Tc(NO)Cl_2(HOMe)(PPh_3)_2]$ with the tetradentate ligand 1,4-bis-(2-pyridylmethyl)-1,4-diazobutane yields a mixture of products including cationic [Tc(NO)Cl(py-NH-NH-py)]Cl and cationic $[Tc(NO)Cl(PPh_3)(py\text{-}NH\text{-}NH{\sim}py)]Cl$, with a pyridyl terminus left dangling.

1. Introduction

We recently began studies of Tc-nitrosyl chemistry which represents a new and novel core for radiopharmaceutical development. To gain a better understanding of the nature of the ligand donor-set and coordination geometries necessary for the synthesis of ^{99m}Tc-nitrosyl complexes, the long-lived macroscopic $^{99}Tc{=}N{=}O$ core was studied, starting initially with the redox active Tc(II) precursor $(Bu_4N)[^{99}Tc(NO)Cl_4]$. Previous results from the reaction with the neutral, tridentate ligand $\{Ph_2PCH_2CH_2N(R)CH_2CH_2PPh_2\}$, (PNP) highlight the many hurdles encountered when employing this highly reactive Tc(II)-nitrosyl precursor. This reaction yielded both the *mer-* and *fac-*structural isomers of $[Tc(NO)Cl_2(PNP)]$, which were isolated and structurally characterized. Both of these species are present in equilibrium during the course of the reaction, as determined by infrared spectroscopy [1]. Herein we describe studies with the Tc(I) nitrosyl precursor $[Tc(NO)Cl_2(HOMe)(PPh_3)_2]$ and its reactions with various ligands and their coordination geometries.

2. Experimental

ESI Mass Spectrometry was performed on a Bruker Daltonics APEX IV FT–ICR–MS (Fourier Transform–Ion Cyclotron Resonance–Mass Spectrometer) with ESI (Electoral Spray Ionization) or DART (Direct Analysis in Real Time – made by IonSense) ion source. ESI condition: Drying gas (N_2) at 30 psi, heated to 230 °C; Nebulizing gas (N_2) at 40 psi, with no heat. Voltage applied to Capillary at −3724 V, End-plate at −3281 V, and CapExit at 112.4 V. DART condition: Drying gas (N_2) at 10 psi; reaction gas (He) at 2.2 L/min and heated up to 200–500 °C, depending on the sample's molecular weight (higher temperature for higher MW). Sample solution: Most samples were dissolved in 50–100 μL of dichloromethane, then methanol added.

2.1. $[Tc(NO)Cl_2(pyN(Et)py)]$ complex (**1**)

To a solution of $(Bu_4N)[Tc(NO)Cl_4]$ [2] (45 mg, 0.09 mmole) in 45 mL of CH_2Cl_2 was added di-(2-picolyl)(*N*-ethyl)amine (20 mg, 0.092 mmole), which was stirred overnight at room temperature. The resulting orange solution was concentrated under vacuum and chromatographed, yielding 2 yellow bands in addition to an insoluble residue. The major band was evaporated to dryness, redissolved in dichloromethane and layered with methanol and ether to give a deep orange crystalline product. Yield: 23%. ESI-MS(+) (*m*/*z*): 426 $[Tc(NO)Cl_2(pyN(Et)py)]^+$, 391 $[Tc(NO)Cl(pyN(Et)py)]^+$. IR (neat): $\nu(N{=}O)$ 1788 (vs).

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Table 1
Selected bond lengths (Å) and angles (°) for [Tc(NO)Cl_2(py*N*(Et)py)].

Bond lengths (Å)		Bond angles (°)			
Tc–N1	1.744(4)	Tc1–N1–O1	172.5(4)	N4–Tc1–Cl2	168.85(12)
N1–O1	1.187(6)	N1–Tc1–N4	94.60(18)	N2–Tc1–Cl2	89.78(11)
Tc–$N3_{(ali)}$	2.233(4)	N1–Tc1–N2	92.56(18)	N3–Tc1–Cl2	90.73(11)
Tc–$N2_{(py)}$	2.109(4)	N4–Tc1–N2	91.79(16)	N1–Tc1–Cl1	97.90(14)
Tc–$N4_{(py)}$	2.097(4)	N1–Tc1–N3	166.23(17)	N4–Tc1–Cl1	86.28(12)
Tc–Cl1	2.4404(12)	N4–Tc1–N3	78.98(16)	N2–Tc1–Cl1	169.48(12)
Tc–Cl2	2.4349(12)	N2–Tc1–N3	75.62(15)	N2–Tc1–Cl1	90.17(4)
		N1–Tc1–Cl2	96.36(14)	N3–Tc1–Cl1	93.86(11)

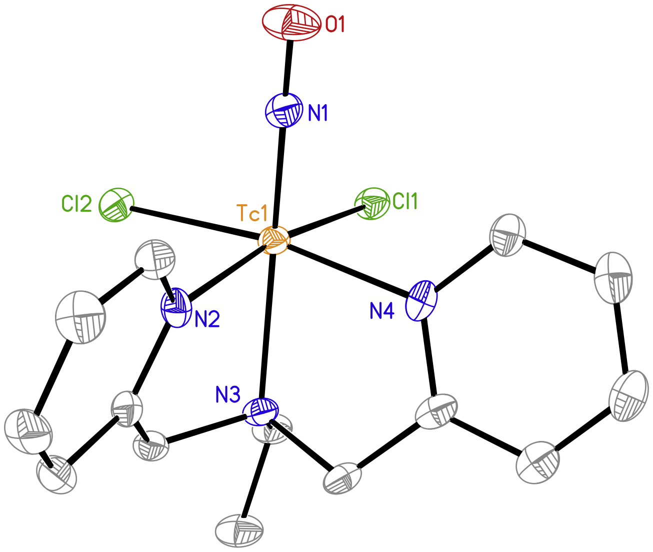


Fig. 1. ORTEP diagram of neutral complex (**1**) [Tc(NO)Cl_2(py*N*(Et)py)].

Table 2
Selected bond lengths (Å) and angles (°) for [Tc(NO)Cl(py*N*(Et)py)(PPh_3)]Cl.

Bond lengths (Å)		Bond angles (°)			
Tc–N1	1.796(3)	O1–N1–Tc1	178.4(2)	N4–Tc1–Cl1	159.77(6)
N1–O1	1.088(3)	N1–Tc1–N4	96.94(9)	N3–Tc1–Cl1	89.65(6)
Tc–$N3_{(ali)}$	2.215(2)	N1–Tc1–N3	94.08(9)	N2–Tc1–Cl1	85.31(6)
Tc–$N2_{(py)}$	2.221(2)	N4–Tc1–N3	80.19(9)	N1–Tc1–P1	87.20(7)
Tc–$N4_{(py)}$	2.138(2)	N1–Tc1–N2	168.57(9)	N4–Tc1–P1	103.08(6)
Tc–P1	2.4133(7)	N4–Tc1–N2	75.35(8)	N3–Tc1–P1	176.33(7)
Tc–Cl1	2.3984(8)	N3–Tc1–N2	76.46(8)	N2–Tc1–P1	102.65(6)
		N1–Tc1–Cl1	101.22(8)	Cl1–Tc1–P1	86.72(2)

2.2. [Tc(NO)Cl(pyN(Et)py)(PPh_3)]Cl complex (**2**)

To a solution of [Tc(NO)Cl_2(HOMe)(PPh_3)$_2$] [3] (55 mg, 0.072 mmole) in 60 mL of CH_2Cl_2 was added di-(2-picolyl)(*N*-ethyl)amine (17 mg, 0.075 mmole), which was stirred overnight at room temperature. The resulting dark orange solution was concentrated under vacuum and washed with ether. The residue was dissolved in dichloromethane and layered with methanol and ether to give a red crystalline product. Yield: 47%. ESI-MS(+) (*m*/*z*): 653 [Tc(NO)Cl(py*N*(Et)py)(PPh_3)]$^+$. IR (neat): ν(N═O) 1786 (vs).

2.3. [Tc(NO)Cl(pyNH-NHpy)]Cl complex (**3**)

To a solution of [Tc(NO)Cl_2(HOMe)(PPh_3)$_2$] (68 mg, 0.090 mmole) in 55 mL of CH_2Cl_2 was added 2,5-bis-(2-pyridylmethyl)-2,5 diazohexane (23 mg, 0.095 mmole) which was stirred overnight at room temperature. The resulting orange solution was concentrated and chromatographed on silica gel, yielding two distinctly separate yellow bands in addition to a significant immobile residue. The major band was evaporated to dryness, redissolved in dichloromethane and layered with methanol and ether to give orange crystalline product. Yield 18%. ESI-MS(+) (*m*/*z*): 406 [Tc(NO)Cl(pyNH-NHpy)]$^+$, 370 [Tc(NO)(pyNH-NHpy)]$^+$. IR (neat): ν(N═O) 1710 (vs).

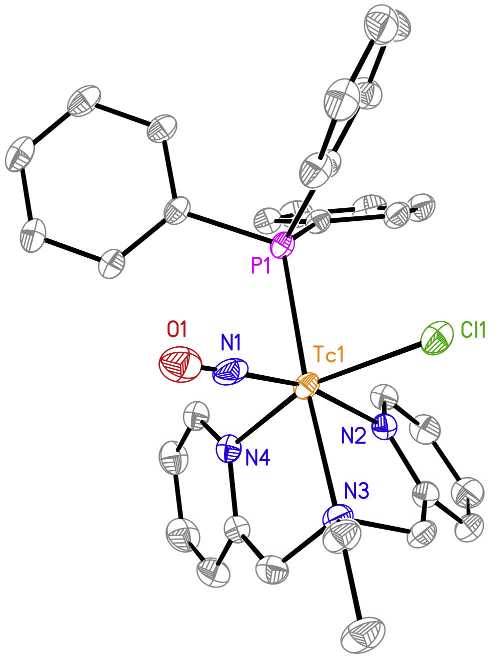


Fig. 2. ORTEP diagram of cationic complex (**2**) [Tc(NO)Cl(pyN(Et)py)(PPh_3)]Cl. The chloride counter ion has been omitted.

2.4. [Tc(NO)Cl(pyNH-NH–py)(PPh_3)]Cl complex (**4**)

From the preparation of complex (**3**), the second yellow band from the chromatographic plate was evaporated to dryness, dissolved in methanol and layered with ether. Upon sitting for 7 days, small green crystals of complex (**4**) appeared from the yellow solution. Yield 7%. ESI-MS(+) (*m*/*z*): 668 [Tc(NO)Cl(PPh_3)(pyNH-NH-py)] 632 [Tc(NO)(PPh_3)(pyNH-NH-py)]$^+$, 406 [Tc(NO)Cl(pyNH-NH-py)]$^+$, 370 [Tc(NO)(pyNH-NH-py)]$^+$. IR (neat): ν(N═O) 1727 (vs).

3. Results and discussion

We recently reported *fac*- and *mer*-isomers of the Tc(I) complex [TcCl_2NO(PNP)], which were isolated and structurally characterized from the reaction of the Tc(II) species (Bu_4N)[TcNOCl_4] and the neutral tridentate phosphine-amine ligand PNP [1]. This product displays oxidation state Tc(I) which suggests there are redox reactions competing with the desired substitution chemistry when employing this Tc(II) precursor.

Similarly, the reaction of the Tc(II) complex (Bu_4N)[TcNOCl_4] with the neutral, tridentate ligand *N*-ethyl-bis(2-picolyl)amine in dichloromethane yields a mixture of yellow-brown products which are evident in the infrared spectrum of the crude product as multiple nitrosyl absorptions in the 1700–1900 cm^{-1} region. Chromatography of this crude reaction mixture on silica produced a prominent yellow band, in addition to a large fraction of insoluble, intractable material. This yellow complex yielded X-ray quality crystals when layered with methanol and diethylether. The single

Table 3
Selected bond lengths (Å) and angles (°) for [Tc(NO)Cl(pyNH-NHpy)]Cl.

Bond lengths (Å)		Bond angles (°)			
Tc–N1	1.751(3)	O1–N1–Tc1	172.3(3)	N4–Tc1–N3	79.75(10)
N1–O1	1.189(3)	N2–Tc1–N1	95.83(11)	N4–Tc1–N5	79.15(10)
$Tc–N3_{(ali)}$	2.098(2)	N1–Tc1–N3	171.11(11)	N1–Tc1–Cl1	97.06(9)
$Tc–N4_{(ali)}$	2.151(3)	N5–Tc1–N1	94.43(11)	N2–Tc1–Cl1	88.30(7)
$Tc–N2_{(py)}$	2.078(3)	N2–Tc1–N5	99.07(10)	N3–Tc1–Cl1	86.74(7)
$Tc–N5_{(py)}$	2.113(2)	N3–Tc1–N5	83.14(10)	N5–Tc1–Cl1	165.66(7)
Tc–Cl1	2.4183(8)	N1–Tc1–N4	108.27(12)	N4–Tc1–Cl1	89.08(7)
		N2–Tc1–N4	155.90(10)	N3–Tc1–N2	76.19(10)

crystal structural characterization established this complex as the neutral [Tc(NO)Cl_2(py*N*(Et)py)], a Tc(I) species. The complex mixture of products generated in this reaction and separated chromatographically, some of which showed no nitrosyl absorption in their infrared spectra, illustrates the advantage of employing a pre-reduced synthetic precursor. The infrared spectrum of yellow complex (**1**) displays the absorption from *ν*(N═O) at 1788 cm^{-1}. The ESI(+) mass spectrum displays the parent ion [Tc(NO)Cl_2(py*N*(Et)py)]$^+$ of 426 *m*/*z* and the ion generated from the loss of a chloride [Tc(NO)Cl(pyN(Et)py)]$^+$ at 391 *m*/*z*.

The single crystal X-ray structure of [Tc(NO)Cl_2(py*N*(Et)py)] displays multiple bonding throughout the linear nitrosyl unit, with Tc═N═O bond angle of 172.5(4)°. The facial arrangement of the tridentate ligand with its small bite angle results in deviation from ideal octahedral geometry. Bond lengths are unexceptional. Table 1 lists selected bond lengths and angles. And Fig. 1 displays an ORTEP diagram for [Tc(NO)Cl_2(py*N*(Et)py)].

The reaction of the Tc(I) complex [Tc(NO)Cl_2(HOMe)$(PPh_3)_2$] with this same tridentate ligand in dichloromethane yields the cationic species [Tc(NO)Cl(pyN(Et)py)(PPh_3)]Cl, complex (**2**). The linear nitrosyl ligand displays a Tc═N═O bond angle of 178.4(2)° and multiple bonding as well, with the Tc–N bond of 1.796(3) Å and the N–O bond of 1.088(3) Å, all within expected parameters. Trans to the nitrosyl atom is a pyridyl terminus from the tridentate ligand. The central amine nitrogen atom of this ligand is coordinated trans to the phosphine, and the remaining pyridyl nitrogen atom is bound trans to the chloride ligand. The coordination geometry is a distorted octahedral with a large bite angle of 101.22(8)°. Table 2 lists selected bond lengths and angles for [Tc(NO)Cl(pyN(Et)py)(PPh_3)]Cl and Fig. 2 displays an ORTEP diagram.

The reaction of the Tc(I) complex [Tc(NO)Cl_2(HOMe)$(PPh_3)_2$] with one equivalent of 2,5-bis-(2-pyridylmethyl)-2,5 diazohexane in dichloromethane yields an orange solution which displayed multiple absorptions in the 1600–1800 cm^{-1} region of the IR spectrum. Thin layer chromatography of this crude mixture, eluting with MeOH and dichloromethane, yielded two yellow bands.

One yellow component was methanol soluble which upon layering with ether yielded orange crystals of the cationic complex [Tc(NO)Cl(pyNH-NHpy)]Cl, which contains the tetradentate ligand occupying four coordination sites. The Tc–N and N–O bond lengths display the expected multiple bonding and a near linear bond angle of 172.3(3)°. Three of the four coordination sites occupied by the tetradentate ligand form a meridional band with one pyridine terminus trans to the chloride ligand. The nitrosyl ligand sits trans to one of the internal aliphatic amine nitrogens. Overall coordination geometry is distorted octahedral, with significant deviation imposed by the small bite angles of the five-membered chelate rings of the polydentate ligand. Table 3 lists selected bond lengths and angles for [Tc(NO)Cl(pyNH-NHpy)]Cl and Fig. 3 displays an ORTEP diagram with the chloride counter ion omitted.

The second yellow band isolated from the chromatographic plate was dichloromethane soluble but methanol insoluble. Upon sitting at room temperature the yellow-orange solution very gradually deposited a few green crystals. This green complex was determined to have only three of the four amine groups from the

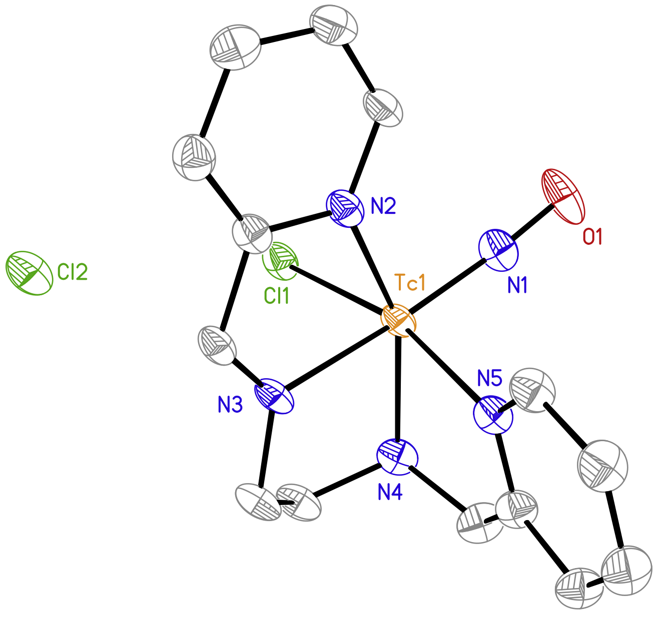


Fig. 3. ORTEP diagram of cationic complex (**3**) [Tc(NO)Cl(pyNH-NHpy)]Cl.

Table 4
Selected bond lengths (Å) and angles (°) for [Tc(NO)Cl(pyNH-NH–py)(PPh_3)]Cl.

Bond lengths (Å)		Bond angles (°)			
Tc–N1	1.723(7)	O1–N1–Tc1	176.3(5)	N2–Tc1–Cl1	89.38(12)
N1–O1	1.216(9)	N1–Tc1–N3	96.2(2)	N4–Tc1–Cl1	89.93(12)
$Tc–N3_{(ali)}$	2.158(4)	N2–Tc1–N3	74.04(16)	N1–Tc1–Cl1	97.05(7)
$Tc–N4_{(ali)}$	2.213(4)	N1–Tc1–N4	91.5(2)	N1–Tc1–P1	89.01(17)
$Tc–N2_{(py)}$	2.183(4)	N4–Tc1–N3	79.49(15)	N3–Tc1–P1	100.33(11)
Tc–P1	2.4157(13)	N2–Tc1–N4	88.59(15)	N2–Tc1–P1	90.88(11)
Tc–Cl1	2.402(2)	N5–Tc1–Cl1	100.51(17)	N4–Tc1–P1	179.47(12)
		N1–Tc1–Cl1	160.41(13)	P1–Tc1–Cl1	90.09(6)

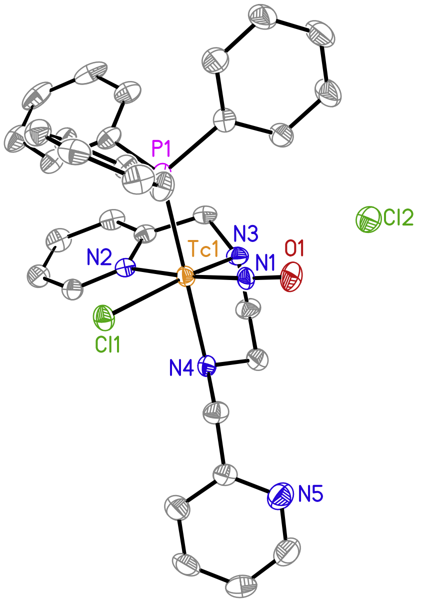
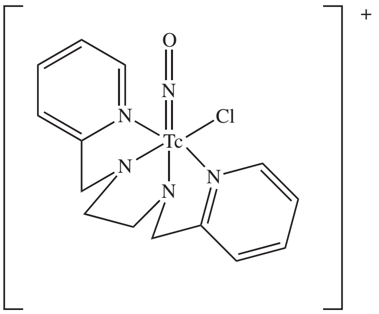


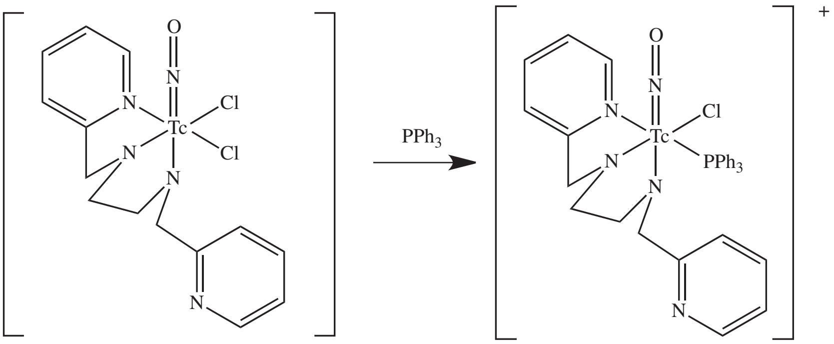
Fig. 4. ORTEP diagram of cationic complex (**4**) [Tc(NO)Cl(pyNH-NH–py)(PPh_3)]Cl.

tetradentate ligand coordinated, with one pyridyl terminus left dangling. The nitrosyl ligand is again coordinated in a linear manner, with multiple bonding throughout. The Tc=N=O bond angle is 176.3(5)° and Tc–N and N–O bonds are 1.723(7) Å and 1.216(9)°, respectively. Trans to the nitrosyl ligand is the coordinated pyridyl moiety. This cationic product has a triphenylphosphine ligand occupying the site to one of the aliphatic amine groups of the tridentate ligand. The chloride ligand is cis to the nitrosyl ligand and trans to the second aliphatic amine nitrogen. Table 4 lists selected bond lengths and angles for [Tc(NO)Cl(pyNH-NH–py)(PPh_3)]Cl, and Fig. 4 displays an ORTEP diagram.

One plausible explanation for this color change from yellow chromatography band to green crystals is that the yellow species, upon sitting in solution, selectively replaces a chloride ligand with a triphenylphosphine, which co-elutes with the complex. Excess triphenylphosphine is present in the precursor [Tc(NO)Cl_2(HOMe)$(PPh_3)_2$], and the substitution reaction itself releases additional phosphine to the solution. We hypothesize that the observed color change upon sitting results from a triphenylphosphine replacing a coordinated terminal pyridyl unit from the tetradentate ligand. See Fig. 5 for a diagram displaying the proposed mechanism.



MeOH soluble yellow species



CH_2Cl_2 soluble yellow species

Green crystals upon sitting at RT

Fig. 5. Proposed mechanism for the formation of complex (**4**).

Table 5
Nitrosyl ligand bonding parameters and technetium oxidation states for various structurally characterized complexes with *trans*-ligand bond lengths.

Complex #	Tc–N (Å)	N–O (Å)	Tc–N—O (°)	Tc–$L_{(trans)}$ (Å)	Reference
[Tc^I(NO)Cl_2(pyN(Et)py)]	1.744(4)	1.187(6)	172.5(4)	2.233(4) aliphatic amine	This work
[Tc^I(NO)Cl(pyN(Et)py)(PPh_3)]Cl	1.796(3)	1.088(3)	178.4(2)	2.221(2) aromatic amine	This work
[Tc^I(NO)Cl(pyNH-NHpy)]Cl	1.751(3)	1.189(3)	172.3(3)	2.098(2) aliphatic amine	This work
[Tc^I(NO)Cl(pyNH-NH~py)(PPh_3)]Cl	1.723(7)	1.216(9)	176.3(5)	2.183(4) aliphatic amine	This work
[Tc^{II}(NO)Cl_3(bipy)]	1.759(4)	1.178(4)	176.9(4)	2.150(3) aromatic amine	[4]
[Tc^I(NO)Cl_2(PNP)] *fac*	1.751(5)	1.163(6)	177.9(4)	2.364(4) aliphatic amine	[1]
[Tc^I(NO)Cl_2(PNP)] *mer*	1.758(4)	1.184(6)	173.0(4)	2.4197(15) chloride	[1]
[Tc^I(NO)Cl_2(pyPPh$_2$-P,N)(pyPPh$_2$-P)	1.743(5)	1.170(6)	177.2(5)	2.441(2) chloride	[5]
[Tc^{II}(NO)Cl_2(DPPE)](PF_6)	1.717(3)	1.145(3)	179.0(2)	2.3262(7) chloride	[6]
[Tc^I(NO)Cl_2(PPh_3)(HN=Npy)]	1.752(4)	1.180(5)	175.1(4)	2.153(4) aromatic amine	[7]
[[Tc^I(NO)$Cl_2$$(py)_3$]	1.781(5)	1.192(5)	176.8(7)	2.367(2) chloride	[3]
[Tc^{III}(NO)Cl$(SAr)_3$]	1.767(6)	1.150(7)	176.8(6)	2.366(2) chloride	[8]
[Tc^I(NO)Cl(Spy)$(PPh_3)_2$]	1.748(4)	1.196(5)	177.7(3)	2.173(4) aromatic amine	[9]
[Tc^I(NO)$(Spy)_2$(PPh_3)]	1.7497(12)	1.1941(17)	173.35(12)	2.1771(12) aromatic amine	[9]
[Tc^I(NO)$Br_2$$(CN\text{-}R)_3$]	1.726(15)	1.136(17)	175.9(1.6)	2.137(22) isonitrile	[10]
$[Tc(NO)(CO)_2Cl_2]_2$ (Cl-bridged)	1.749(14)	1.16(2)	177.4(8)	2.375(5) chloride	[11]
$[Tc(NO)(CO)_2Cl_2]_2$ (Cl-bridged)	1.794(14)	1.13(2)	174.3(9)	2.380(5) chloride	[11]
[Tc(NO)(NH_3)$(phen)_2$]Cl_2	1.739(9)	1.160(9)	171.9(8)	2.16(1) aromatic amine	[12]

4. Conclusion

The Tc(II) complex $(Bu_4N)[TcNOCl_4]$ was shown to be both overly reactive and redox unstable in reactions with polydentate ligands. Redox chemistry greatly diminishes yields and a complex mixture of products are formed which required chromatographic separation. The Tc(I) complex $[Tc(NO)Cl_2(HOMe)(PPh_3)_2]$ is shown to be much more selective in analogous reactions with polydentate amine ligands.

Synthesis and characterization of four new Tc(I) nitrosyl complexes are reported above. These mixed ligand complexes, with a variety of donor sets coordinated, further our aim of establishing plausible bonding orientations for utilization in the design of more stable, polydentate ligands to be employed in radiopharmaceutical design. The reactions employing the Tc(I) precursor show better yields and products are more stable in solution than those which employ the Tc(II) precursor $[TcNOCl_4]^+$.

From the structural data of these four nitrosyl–polyamine complexes, complex (**2**) is unique in having an aromatic amine in the position *trans*- to the nitrosyl ligand. The different electronic character of this ligand compared to the aliphatic amines in the *trans*-positions of the other three complexes results in an elongation of the Tc═N bond and a contraction of the N═O bond. The *trans*-influence of the ligands opposite to these multiply bonded cores is well established in the literature [13]. Table 5 lists a selection of structurally characterized technetium nitrosyl complexes and their bonding parameters, including the bond lengths to the ligands *trans* to the nitrosyl unit.

The cationic Tc(I) nitrosyl complex *fac*-$[TcCl_3(CO)_2(NO)]^+$ has received considerable attention in the literature in recent years. This modified tricarbonyl species can be expected to show completely different reaction properties from the Tc(I) *mer*-$[TcNOCl_2(HOMe)(PPh_3)_2]$ species employed here. The nitrosyl, dicarbonyl, histidine complex $[Tc(HIS)(CO)_2(NO)][BF_4]$ has been reported, synthesized from the reaction of $[NEt_4][TcCl_3(CO)_2(NO)]$ with the tridentate amino acid in methanol. No structural data was reported for direct comparison [14].

Acknowledgements

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2011.04.002.

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