

Generally, the Mg<sup>II</sup> ion prefers octahedral coordination. In MgCl<sub>2</sub>·6H<sub>2</sub>O, the Mg<sup>II</sup> ion forms an almost regular octahedron (Agron & Busing, 1985). However, the Mg—O bond lengths in the double salts of MgCl<sub>2</sub>·RbCl·6H<sub>2</sub>O and MgCl<sub>2</sub>·NH<sub>4</sub>Cl·6H<sub>2</sub>O vary widely due to the effect of the surrounding Cl<sup>-</sup> ions. The different bond lengths in the coordination sphere of the Mg<sup>II</sup> ion may be responsible for the dehydration process of the double salts.

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## Structure of a Neutral *N*-Alkylated Diaminedithiol (dadt) <sup>99</sup>Tc<sup>V</sup> Complex: *syn*[<sup>99</sup>TcO(NEt-tmdadt)]

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**Abstract.** *syn*-(4-Ethyl-2,9-dimethyl-4,7-diaza-2,9-decanedithiolato)oxo(<sup>99</sup>Tc)technetium(V), *syn*-[<sup>99</sup>TcO(NEt-tmdadt)], [Tc(O)(C<sub>12</sub>H<sub>25</sub>N<sub>2</sub>S<sub>2</sub>)], *M<sub>r</sub>* = 375, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 9.638 (2), *b* = 14.371 (5), *c* = 11.893 (3) Å, β = 100.79 (2)°, *V* = 1618.15 (75) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.54 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 11.0 cm<sup>-1</sup>, *F*(000) = 776, *T* = 293 K, *R* = 0.028, *wR* = 0.042 for 2788 unique observed reflections with *I* > σ(*I*). This study is the

first which conclusively establishes the structure of the major complex derived from the reaction of an *N*-substituted diaminedithiol ligand with reduced pertechnetate. An intramolecular repulsion exists between the ethyl substituent and the oxo-metal core, resulting in a larger-than-expected angle for N(2)—Tc—O, 104.7 (1)°.

**Introduction.** The metastable isotope of technetium, technetium-99m (<sup>99m</sup>Tc, γ = 140 keV, *t*<sub>1/2</sub> = 6.02 h), is the radionuclide chosen for performing the majority of diagnostic nuclear medicine procedures

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(Pinkerton, Dislets, Hock, Mikelson & Wilson, 1985). Due to the high specific radioactivity and short half life of  $^{99m}\text{Tc}$ , structural analyses of complexes containing this isotope are precluded. Technetium-99 ( $^{99}\text{Tc}$ ,  $\beta = 0.292$  MeV,  $t_{1/2} = 2.1 \times 10^5$  year), however, is available on the gram scale, and characterization of  $^{99}\text{Tc}$  complexes by standard analytical techniques provides the chemical foundation needed for the interpretation of the *in vivo* behavior of the corresponding  $^{99m}\text{Tc}$  complexes.

In recent studies, we have found a well defined structure – *in vivo* biodistribution relationship for the lung uptake of a series of neutral, lipophilic  $^{99m}\text{Tc}$  complexes derived from diaminedithiol (dadt) ligands (Lever *et al.*, 1988). As [ $^{99m}\text{TcO}(\text{NET-tmdadt})$ ] showed the highest pulmonary accumulation in rodents, the corresponding  $^{99}\text{Tc}$  analog of the major isomer was prepared and analyzed by single-crystal X-ray crystallography.

**Experimental.** Complexation reactions and subsequent manipulations were carried out in laboratories approved for low-level radioactivity. The ligand, 4-ethyl-2,9-dimethyl-4,7-diaza-2,9-decanedithiol dihydrochloride (NET-tmdadt.2HCl), was synthesized as previously reported (Baidoo, 1988) and characterized by  $^1\text{H}$  NMR, IR and elemental analysis.

In a typical synthesis, aqueous  $\text{NH}_4^{99}\text{TcO}_4$  was reduced with sodium dithionite in a basic medium in the presence of the ligand. Two lipophilic components were separated and purified by preparative thin-layer silica-gel chromatography (benzene, methylene chloride, acetonitrile 70:25:5):  $R_f = 0.40$  (brown) and 0.32 (yellow) in a 84:16 ratio. Each of these products was crystallized by slow diffusion of pentane into a methylene chloride solution of the complex. The crystals were washed several times with cold pentane and dried *in vacuo*. Both complexes gave the same elemental analysis which is indicative of structural isomerism. The most likely configurational difference is derived from a *syn/anti* relationship of the ethyl side chain with respect to the oxo-metal core.

A pale-brown needle-shaped crystal of the major isomer,  $0.75 \times 0.35 \times 0.15$  mm, was selected and mounted on a glass fiber with epoxy cement. Nicolet  $R3m$  diffractometer, graphite monochromator; unit-cell parameters by least-squares refinement of 25 reflections ( $24 < 2\theta < 34^\circ$ );  $\theta$ – $2\theta$  scans at variable rates,  $2\theta_{\text{max}} = 50^\circ$  for the range  $0 < h < 12$ ,  $0 < k < 18$ ,  $-15 < l < 15$ ; three reflections monitored every 97 reflections with negligible change in intensity over the course of data collection ( $< 1\%$ ); 3185 measured unique intensities, 2788 unique observed reflections with  $I > \sigma(I)$  used for refinement. An empirical absorption correction was made due to the shape of the crystal. Empirical absorption corrections were

Table 1. *Atomic coordinates* ( $\times 10^4$ ) *and temperature factors* ( $\text{\AA}^2 \times 10^3$ )

$U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	x	y	z	$U_{\text{eq}}$
Tc	–516 (1)	3105 (1)	1912 (1)	35 (1)
S(1)	1614 (1)	2540 (1)	1697 (1)	48 (1)
C(1)	1031 (3)	1425 (2)	987 (2)	47 (1)
C(2)	767 (3)	714 (2)	1886 (2)	54 (1)
C(3)	2198 (3)	1072 (2)	373 (3)	66 (1)
C(4)	–311 (3)	1642 (2)	152 (2)	49 (1)
N(1)	–1201 (2)	2285 (2)	656 (2)	43 (1)
C(5)	–2602 (3)	2442 (2)	–41 (2)	51 (1)
C(6)	–2821 (3)	3473 (2)	–80 (2)	49 (1)
N(2)	–2468 (2)	3855 (1)	1105 (2)	40 (1)
C(7)	–3585 (2)	3581 (2)	1786 (2)	50 (1)
C(8)	–5108 (3)	3806 (3)	1244 (3)	68 (1)
C(9)	–2313 (3)	4895 (2)	1058 (2)	47 (1)
C(10)	–1110 (3)	5298 (2)	1937 (2)	48 (1)
C(11)	–1409 (3)	5337 (2)	3149 (2)	61 (1)
C(12)	–800 (4)	6283 (2)	1537 (3)	65 (1)
S(2)	458 (1)	4565 (1)	1901 (1)	49 (1)
O	–838 (2)	2741 (1)	3181 (1)	51 (1)

calculated on the basis of azimuthal scan data for eight reflections, and applied to all intensities; calculated transmission factors ranged from 0.583 to 0.763. Structure determination by direct methods: ten of the 25 H atoms found on difference map; structure refinement: all C–H bond lengths fixed at 0.96 Å and refined with ideal geometry; anisotropic thermal parameters for all non-H atoms and fixed isotropic thermal parameters for H atoms (20% greater than that of carrying atom). Refined by cascade block-diagonal least squares on  $F$  with max.  $(\sin\theta)/\lambda = 0.59 \text{ \AA}^{-1}$ ; refinement of 163 parameters converged to  $R = 0.028$ ,  $wR = 0.042$ ;  $w = [\sigma^2(F) + 0.0008F^2]^{-1}$ , where  $\sigma^2(F)$  is derived from counting statistics; goodness of fit = 1.235;  $(\Delta/\sigma)_{\text{max}} = -0.147$  in final cycle; highest peak in final difference map is 0.36, deepest hole  $-0.52 e \text{ \AA}^{-3}$ ; atomic scattering factors and anomalous-dispersion corrections were from *International Tables for X-ray Crystallography* (1974, Vol. IV); all calculations were performed on a Data General Eclipse S140 computer using the *SHELXTL* program package (Sheldrick, 1984).

A crystal of the minor isomer was prepared and mounted as described above. However, decomposition prior to completion of data collection precluded structural elucidation.

**Discussion.** Fractional atomic coordinates and thermal parameters for non-H atoms are given in Table 1.\* The molecule has a distorted square pyramidal arrangement around the Tc atom (Fig. 1) with the

\* Anisotropic thermal parameters and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53453 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

oxo group occupying the apical position. Upon coordination, the ethyl substituent adopts the *syn* configuration with respect to the oxo-metal group, and the unsubstituted nitrogen is deprotonated resulting in a neutral molecule. There is considerable puckering of the ligand so that the four basal atoms do not lie in a plane; hence, an idealized plane was calculated. S(1) and N(2) lie 0.1168 and 0.1274 Å above and S(2) and N(1) lie 0.1058 and 0.1384 Å below the calculated plane. Distortion in the square pyramidal geometry is further borne out by the torsion angles in the three basal five-membered rings: S(1)—C(1)—C(4)—N(1) (−40.9°), N(1)—C(5)—C(6)—N(2) (−49.1°) and N(2)—C(9)—C(10)—S(2) (46.4°). The Tc atom lies above the mean calculated plane, which is similar to the displacement observed for [TcO(SCHCH<sub>2</sub>S)] (Davison, Orvig, Trop, Sohn, DePamphilis & Jones, 1980), [TcO(SCH<sub>2</sub>CON—CH<sub>2</sub>—)<sub>2</sub>] (Jones *et al.*, 1982) and [TcO(NH-hmdadt)] (Epps, 1984; Watson, Tulip & Roe, 1986) (hmdadt = 2,5,5,9-tetramethyl-4,7-diaza-2,9-decanedithiol).

Selected bond lengths and angles are given in Table 2. The Tc—S bond lengths are similar to those observed for a number of Tc-thiolate complexes (Davison *et al.*, 1980; DePamphilis, Jones, Davis & Davison, 1978). However, there is a notable difference between the two Tc—S bonds. The Tc—S(1) bond *trans* to the substituted quaternary nitrogen is shorter [2.265 (1) Å] than the Tc—S(2) [2.300 (1) Å] bond. Further asymmetry was found for the Tc—N bond lengths which differ by 0.303 (4) Å. The shorter Tc—N(1) bond length [1.921 (2) Å] is comparable to those previously observed for Tc—N bonds, whereas the Tc—N(2) bond [2.224 (2) Å] is significantly longer than the average reported Tc—N bond length (2.07 Å) (Bandoli, Mazzi, Roncari & Deutsch, 1982). The differences in these bond lengths are consistent with *sp*<sup>2</sup> hybridization of N(1) and *sp*<sup>3</sup> hybridization of

Table 2. Bond lengths (Å) and bond angles (°)

Tc—S(1)	2.265 (1)	Tc—N(1)	1.921 (2)
Tc—N(2)	2.224 (2)	Tc—S(2)	2.300 (1)
Tc—O	1.681 (2)	S(1)—C(1)	1.850 (3)
C(1)—C(2)	1.534 (4)	C(1)—C(3)	1.537 (4)
C(1)—C(4)	1.508 (3)	C(4)—N(1)	1.463 (4)
N(1)—C(5)	1.464 (3)	C(5)—C(6)	1.495 (4)
C(6)—N(2)	1.492 (3)	N(2)—C(7)	1.515 (3)
N(2)—C(9)	1.504 (3)	C(7)—C(8)	1.524 (3)
C(9)—C(10)	1.523 (3)	C(10)—C(11)	1.522 (4)
C(10)—C(12)	1.540 (4)	C(10)—S(2)	1.849 (3)
S(1)—Tc—N(1)	83.1 (1)	S(1)—Tc—N(2)	146.7 (1)
N(1)—Tc—N(2)	79.7 (1)	S(1)—Tc—S(2)	87.0 (1)
S(2)—Tc—N(1)	129.6 (1)	N(2)—Tc—S(2)	82.5 (1)
S(1)—Tc—O	108.3 (1)	N(1)—Tc—O	114.4 (1)
N(2)—Tc—O	104.7 (1)	S(2)—Tc—O	115.6 (1)
Tc—S(1)—C(1)	99.1 (1)	S(1)—C(1)—C(2)	109.5 (2)
S(1)—C(1)—C(3)	108.4 (2)	C(2)—C(1)—C(3)	110.1 (2)
S(1)—C(1)—C(4)	105.7 (2)	C(2)—C(1)—C(4)	111.3 (2)
C(3)—C(1)—C(4)	111.6 (2)	C(1)—C(4)—N(1)	110.9 (2)
Tc—N(1)—C(4)	124.3 (2)	Tc—N(1)—C(5)	119.5 (2)
C(4)—N(1)—C(5)	114.5 (2)	N(1)—C(5)—C(6)	106.3 (2)
C(5)—C(6)—N(2)	109.2 (2)	Tc—N(2)—C(6)	104.7 (1)
Tc—N(2)—C(7)	105.8 (1)	C(6)—N(2)—C(7)	110.6 (2)
Tc—N(2)—C(9)	114.5 (1)	C(6)—N(2)—C(9)	109.7 (2)
C(7)—N(2)—C(9)	111.3 (2)	N(2)—C(7)—C(8)	116.3 (2)
N(2)—C(9)—C(10)	114.9 (2)	C(9)—C(10)—C(11)	114.1 (2)
C(9)—C(10)—C(12)	107.7 (2)	C(11)—C(10)—C(12)	109.9 (2)
C(9)—C(10)—S(2)	106.7 (2)	C(11)—C(10)—S(2)	110.1 (2)
C(12)—C(10)—S(2)	108.1 (2)	Tc—S(2)—C(10)	100.6 (1)

N(2). Similar differences in Tc—S and Tc—N bond lengths have been observed in the unsubstituted analog [TcO(NH-tmdadt)] (Epps, 1984) in which one nitrogen is deprotonated and the other nitrogen bears a hydrogen in the *syn* configuration.

Interestingly, there is a small but significant intramolecular repulsion between the oxo-metal group and the ethyl substituent. A comparison of the N(2)—Tc—O angles in [TcO(NEt-tmdadt)] and the unsubstituted complex [TcO(NH-tmdadt)] (Epps, 1984) shows an increase of 3.6 (4)° (Table 3). The N(2)—C(9)—C(10)—S(2) torsion angle decreases by 7.2° upon nitrogen substitution (Table 3).

The factors which contributed to the inability to obtain crystal data on the minor complex, presumably the *anti* isomer, are not clear at this time. The minor complex is stable for long periods of time in solution or in solid form, which suggests that decomposition of the minor crystal may be due primarily to experimental artifacts.

Of note, the major complex in the present study had the *N*-ethyl substituent *syn* with respect to the oxo-metal core, and in the case of the unsubstituted [TcO(NH-tmdadt)], the N—H proton is also *syn* (Epps, 1984). Taken together, these data suggest that the *syn* configuration may be thermodynamically more stable than the corresponding *anti* configuration. As this is the first complete crystallographic analysis of any technetium complex derived from an *N*-substituted dadt ligand, whether or not these observations are representative of a general trend remains to be established.

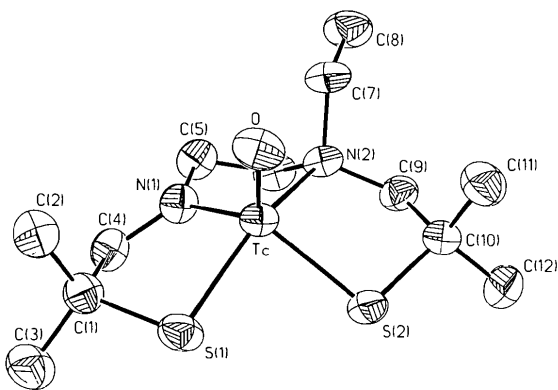


Fig. 1. ORTEP drawing of *syn*-(4-ethyl-2,9-dimethyl-4,7-diaza-2,9-decanedithiolato)oxo(<sup>99</sup>Tc)technetium, *syn*-[<sup>99</sup>TcO(NEt-tmdadt)]. Probability ellipsoids are shown at the 50% level. H atoms have been eliminated for clarity.

Table 3. Comparison of bond and torsion angles ( $^{\circ}$ ) for [TcO(NEt-tmdadt)] and [TcO(NH-tmdadt)]

Bond angles	[TcO(NEt-tmdadt)]	[TcO(NH-tmdadt)]*
S(1)—Tc—O	108.3 (1)	109.3 (2)
N(2)—Tc—O	104.7 (1)	101.1 (3)
S(1)—Tc—N(2)	146.7 (1)	149.3 (3)
S(2)—Tc—O	115.6 (1)	117.0 (3)
N(1)—Tc—O	114.4 (1)	117.8 (4)
S(2)—Tc—N(1)	129.6 (1)	124.4 (3)
Torsion angles†		
S(1)C(1)C(4)N(1)	N(1)C(5)C(6)N(2)	N(2)C(9)C(10)S(2)
[TcO(NEt-tmdadt)]	-40.9	46.4
[TcO(NH-tmdadt)]*	-41.1	53.6

\* Epps (1984).

† Since a glide plane exists, there are mirror image molecules which have these angles with the signs reversed.

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## Structure of Dicarboxyl( $\eta$ -cyclopentadienyl)( $N,N'$ -diphenylacetamidinato)-molybdenum(II), [Mo(C<sub>5</sub>H<sub>5</sub>)(C<sub>14</sub>H<sub>13</sub>N<sub>2</sub>)(CO)<sub>2</sub>]

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**Abstract.** [C<sub>5</sub>H<sub>5</sub>Mo(CO)<sub>2</sub>{C<sub>6</sub>H<sub>5</sub>NC(CH<sub>3</sub>)NC<sub>6</sub>H<sub>5</sub>}], C<sub>21</sub>H<sub>18</sub>MoN<sub>2</sub>O<sub>2</sub>,  $M_r = 426.33$ , triclinic,  $P\bar{1}$ ,  $a = 9.683$  (4),  $b = 10.873$  (5),  $c = 11.161$  (5) Å,  $\alpha = 94.87$  (3),  $\beta = 123.56$  (4),  $\gamma = 102.10$  (3) $^{\circ}$ ,  $V = 928$  (1) Å<sup>3</sup>,  $D_m = 1.51$  (1) Mg m<sup>-3</sup>,  $Z = 2$ ,  $D_x = 1.526$  (2) Mg m<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 0.707$  mm<sup>-1</sup>,  $F(000) = 432$ ,  $T = 293$  K,  $R = 0.032$  for 3067 unique observed reflections. The amidino group bonds to Mo in a symmetrical bidentate mode, with the metal lying in the ligand skeletal NCN plane. The Mo atom is coordinated to 2N at 2.17–2.18 Å, to 2C (carbonyl) at 1.94–1.95 Å, and 5C (Cp) at 2.28–2.41 Å. Skeletal C–N distances are short (1.32 Å). Crystal packing is layered, with adjacent molecules within the layers having opposite orientations.

**Introduction.** Complexes of the type [C<sub>5</sub>H<sub>5</sub>M(CO)<sub>2</sub>{R'NC(R)NR'}] ( $M = \text{Mo, W}$ ,  $R' = \text{aryl}$ ,  $R = \text{Ph, H, CH}_3$ ) were first synthesized from C<sub>5</sub>H<sub>5</sub>M(CO)<sub>3</sub>X ( $X = \text{halide}$ ) using amidines or their metallo-derivatives (Inglis & Kilner, 1975; Gaylani & Kilner, 1976; de Roode & Vrieze, 1978). The amidinato-group here acts as a 3-electron ligand, which may be accomplished by  $N,N'$ -chelation, by bridging between two metals in a binuclear or larger unit, or by C,N-chelation by an *ortho*-metallated group (Barker, Cameron, Kilner, Mahmoud & Wallwork, 1986; Cotton, Inglis, Kilner & Webb, 1975; Barker, Kilner, Mahmoud & Wallwork, 1989). The structures of a series of chiral benzamidinato-complexes [R''C<sub>5</sub>H<sub>4</sub>Mo(CO)<sub>2</sub>{R'NC(R)NR'}] [ $R = \text{C}_6\text{H}_5$ ,  $R' = \text{CH}(\text{C}_6\text{H}_5)Y$ , where  $Y = \text{H, CH}_3$ ,  $R'' = \text{H}$ ,

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