tween M–C(CO) and M–C(CS) distances. There appear to be only a few cases in which M–C(CO) and M–C(CS) distances can be directly compared. A comparison[20–23] of these few structures in which a carbonyl or a thiocarbonyl ligand is found in very similar chemical environments suggests that the M–C(CS) distance will be only slightly shorter ($\Delta \approx$ 0.0–0.10 Å). It would thus appear that the Fe–C(CO) distance in a five-coordinate Fe(P)(CO) complex would be within the range 1.66–1.70 Å. The axial bond distance in the isoelectronic complex Mn(TTP)(NO)[24] is 1.641 (2) Å.

The displacement of a metal atom from the N_4 porphinato plane is a function of the spin state of the metal, the number of axial ligands (one or two), and their relative bonding

strengths. In Fe(OEP)(CS), the low-spin iron(II) atom is displaced 0.23 Å from the mean plane of the core (Figure 2) and 0.22 Å from the N_4 mean plane. These displacements are quite comparable to those observed (0.21 Å) for Fe(TPP)(NO).[2] Is is thus reasonable to expect that a similar displacement of the iron atom would be found for an Fe(P)(CO) complex. With these data, the effect of various sixth ligands on the displacement of the iron atom can be examined. The coordination of a sixth ligand (pyridine) to Fe(TPP)(CS) leads to[16] virtual centering of the iron atom. A similar lack of iron displacement is also seen[9] in Fe(TPP)(CO)(py). Replacement of pyridine with the weak-field and weakly binding ligand tetrahydrofuran leads to[10] a displacement of the iron atom of 0.10 Å toward the CO ligand. Thus even one of the weakest possible sixth-ligand interactions decreases the iron atom displacement by about half compared to the displacement expected for the case of no sixth-ligand interaction.

Registry No. Fe(OEP)(CS)·C_7H_8, 80327-76-0.

Supplementary Material Available: Table III (anisotropic temperature factors), Table VI (rigid-group and derived atomic coordinates for the disordered toluene molecule), Table VII (calculated hydrogen atom positions), Table VIII (additional bond distances and angles), and a listing of the structure factor amplitudes ($\times 10$) (29 pages). Ordering information is given on any current masthead page.

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(21) (PPh$_3$)$_2$(Cl)Rh(CS) and (PPh$_3$)$_2$(Cl)Rh(CO): de Boer, J. L.; Rogers, D.; Skapski, A. C.; Troughton, P. G. H. *J. Chem. Soc., Chem. Commun.* **1966**, 756–757. Δ = 0.07 Å.

(22) (η^6-tetralone)Cr(CO)(CS)(PPh$_3$): Korp, J. D.; Bernal, I. *Cryst. Struct. Commun.* **1980**, *9*, 821–828. ($\eta^6\text{-}C_6H_6$)Cr(CO)$_2$(PPh$_3$): Andrianov, V. G.; Struchkov, Y. T.; Baranetzkaya, N. K.; Setkina, V. N.; Kursanov, D. N. *J. Organomet. Chem.* **1975**, *101*, 209–214. Δ = 0.07 Å.

(23) [Ir(CO)$_2$(CS)(PPh$_3$)$_2$]PF$_6$·(CH$_3$)$_2$CO: Field, J. S.; Wheatley, P. J. *J. Chem. Soc., Dalton Trans.* **1972**, 2269–2273. [Ir(CO)$_3$(P-(CH$_3$)$_2$Ph)$_2$]ClO$_4$: Raper, G.; McDonald, W. S. *Acta Crystallogr., Sect. B* **1973**, *B29*, 2013–2015. Δ = 0.01 Å.

(24) Scheidt, W. R.; Hatano, K.; Rupprecht, G. A.; Piciulo, P. L. *Inorg. Chem.* **1979**, *18*, 292–299.

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The University of Nebraska, Lincoln, Nebraska 68588,
and Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

Structure of Yttrium Octachloroditechnetate Nonahydrate

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The crystal structure of the title compound, YTc$_2$Cl$_8$·9H$_2$O, has been determined, and from the structure of the [Tc$_2$Cl$_8$]$^{3-}$ anion some hypotheses concerning the factors determining metal–metal multiple-bond lengths may be suggested. The compound crystallizes in space group $P42_12$, with cell dimensions of a = 11.712 (2) Å, c = 7.661 (2) Å, V = 1050.9 (4) Å^3, and Z = 4. The structure was refined to R_1 = 0.031 and R_2 = 0.039 with 621 reflections with $I > 3\sigma(I)$ and 51 parameters. The correct enantiomer was chosen. The crystal consists of equal numbers of [Tc$_2$Cl$_8$]$^{3-}$ ions at sites of C_4 symmetry, slightly distorted square antiprismatic [Y(H$_2$O)$_8$]$^{3+}$ ions on sites of D_2 symmetry, and H$_2$O molecules on sites of C_2 symmetry. The Tc–Tc distance is 2.105 (1) Å; the Tc–Cl distances average 2.364 (2) Å, and the Tc–Tc–Cl angles average 104.34 (6)°. The ion has symmetry approximating closely to D_{4h}. The independent Y–O distances are 2.346 (5) and 2.373 (5) Å. The shortness of the Tc–Tc bond compared to that in the [Tc$_2$Cl$_8$]$^{2-}$ ion where the formal bond order is higher is attributed mainly to the decreased effective positive charge on the metal atoms in the [Tc$_2$Cl$_8$]$^{3-}$ ion, which leads to stronger π bonding.

Introduction

In a recent paper[2] the structure of (N-*n*-Bu$_4$)$_2$Tc$_2$Cl$_8$ was reported, providing for the first time a structural characterization of [Tc$_2$Cl$_8$]$^{2-}$. Previously, there had been two X-ray crystallographic studies[4,5] of compounds containing the [Tc$_2$Cl$_8$]$^{3-}$ ion; the results of these two are not entirely independent since the two compounds (NH$_4$)$_3$[Tc$_2$Cl$_8$]·2H$_2$O[3] and K$_3$[Tc$_2$Cl$_8$]·*n*H$_2$O[4] are isotypic. The structural data provided by these investigations gave rise to an apparent inconsistency. In [Tc$_2$Cl$_8$]$^{3-}$, where the Tc–Tc bond order is believed[5,6] to be

3.5, the metal–metal bond length is 2.117 (2) Å, while in [Tc$_2$Cl$_8$]$^{2-}$, where the Tc–Tc bond order should be 4.0, the distance is 2.151 (1) Å.

One of several possible explanations for this unexpected result might have been that the above comparison of Tc–Tc bond lengths is invalid, either because one (or both) structures are crystallographically in error or because these bonds are subject to the influences of drastically different surroundings (hard and highly polar in the case of [Tc$_2$Cl$_8$]$^{3-}$ but soft and diffuse for [Tc$_2$Cl$_8$]$^{2-}$) in the two cases. To determine whether either of these factors is pertinent, it is obviously necessary to carry out additional structural studies, preferably on substances in which the surroundings of the anions are changed in nature. Efforts to prepare suitable compounds are under way, but there are difficulties because of the lability of both

(1) (a) Texas A&M University. (b) Massachusetts Institute of Technology. (c) The University of Nebraska.

(2) Cotton, F. A.; Daniels, L.; Davison, A.; Orvig, C., *Inorg. Chem.* **1981**, *20*, 3051.

(3) Cotton, F. A.; Bratton, W. K. *J. Am. Chem. Soc.* **1965**, *87*, 921. Bratton, W. K.; Cotton, F. A. *Inorg. Chem.* **1980**, *9*, 789.

(4) Cotton, F. A.; Shive, L. W. *Inorg. Chem.* **1975**, *14*, 2032.

(5) Cotton, F. A.; Pedersen, E. *Inorg. Chem.* **1975**, *14*, 383.

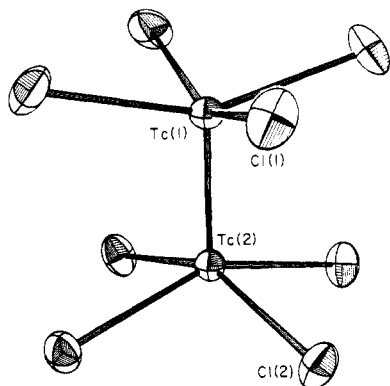(6) Cotton, F. A.; Fanwick, P. E.; Gage, L. D.; Kalbacher, B.; Martin, D. S. *J. Am. Chem. Soc.* **1977**, *99*, 5642.

Figure 1. ORTEP drawing of the $Tc_2Cl_8^{3-}$ anion tilted 20° out of the plane of the paper.

$[Tc_2Cl_8]^{2-}$ and $[Tc_2Cl_8]^{3-}$ in solution.

Thus far we have succeeded in obtaining good crystals of only one additional compound, $Y[Tc_2Cl_8] \cdot 9H_2O.$[7] The surroundings of the $[Tc_2Cl_8]^{3-}$ ion in the yttrium compound are not different in character from those in the previously studied ammonium and potassium compounds. However, the structure of this compound can afford a wholly independent crystallographic investigation in which any unrecognized difficulties in the earlier ones (if such there were) should not recur. This is also the compound used to measure the EPR data[5] for the $[Tc_2Cl_8]^{3-}$ ion. The structure of $YTc_2Cl_8 \cdot 9H_2O$ has therefore been determined accurately and is reported and discussed in this paper.

Procedures

The compound was prepared by the method reported in the literature.[5,7] Suitable crystals were obtained straightforwardly, with no special effort, directly from the preparative reaction.

Several crystals were sealed in thin-walled glass capillaries with epoxy glue and examined on a Syntex P1 automated diffractometer. A crystal having approximately a spherical shape with poor face development, but which scattered X-rays well, was chosen. Its approximate mean diameter was 0.3 mm.

Fifteen intense reflections in the range $20° < 2\theta < 35°$ were employed, with standard software, to establish that the unit cell is tetragonal with $a = 11.712$ (2) Å and $c = 7.661$ (2) Å. With the formula $YTc_2Cl_8 \cdot 9H_2O$ ($M_r = 730.67$) and $Z = 4$, the density is calculated to be 2.31 g cm⁻³, in excellent agreement with the measured density (by flotation in a benzene/bromoform mixture) of 2.31 ± 0.02 g cm⁻³. Data were then collected on the autodiffractometer at 22 ± 2°, using Mo Kα radiation and the $\theta/2\theta$ scanning technique with a scan range from 1.0° below $K\alpha_1$ to 1.0° above $K\alpha_2$ and a ratio of back-ground counting time to peak counting time of 1.0. Variable-scan rates were used, and $2\theta_{max}$ was 55.0°. A total of 660 independent reflections were measured. Three check reflections, remeasured frequently, showed no significant variation. In the final cycles of refinement, 621 reflections with intensity I greater than $3\sigma(I)$ were used. These intensities were corrected for absorption ($\mu = 51.1$ cm⁻¹) with the results of ψ scans near $\chi = 90°$ on seven reflections.

Systematic absences were consistent with either of the space groups $P42_12$ (No. 90) or $P\bar{4}2_1m$ (No. 113). The Patterson function was complex, and therefore we had early recourse to direct methods. Parallel calculations were made for both space groups, first with the MULTAN program.[8] No refineable solution was obtained although a plausible looking Tc_2Cl_8 unit was produced; it was suspected that there was an error in the z coordinate of the origin given by MULTAN. The SHELXTL program[9] was then applied, and positions for the Tc and Y atoms were obtained with different z coordinates from those previously used. These heavy-atom positions refined equally well in

(7) Eakens, J. D.; Humphreys, D. G.; Mellish, C. E. *J. Chem. Soc.* **1963**, 6012.
(8) Two versions of MULTAN were used. The first version was part of the Enraf-Nonius structure determination package. The second was Peter Main's MULTAN78, distributed by the National Resource for Computation in Chemistry.
(9) Nicolet XRD Corp.'s version of George Sheldrick's SHELX.

atom	x	y	z
Tc(1)	0.0000	0.5000	0.2598 (1)
Tc(2)	0.0000	0.5000	0.5346 (1)
Y	0.5000	0.5000	0.0000
Cl(1)	0.1388 (2)	0.3621 (2)	0.1826 (2)
Cl(2)	0.1701 (2)	0.5960 (2)	0.6102 (3)
O(1)	0.4700 (5)	0.3110 (4)	0.0901 (7)
O(2)	0.3865 (5)	0.5321 (4)	0.2519 (5)
O(3)	0.3650 (5)	0.3650 (5)	0.5000

^{a} Estimated standard deviations in the last significant digits are shown in parentheses. Figures without esds are symmetry-determined special positions.

Table II. Interatomic Distances and Angles in $Tc_2Cl_8^{3-}$ ^{a}

atoms	length, Å	atoms	angle, deg
Tc(1)-Tc(2)	2.105 (1)	Tc(2)-Tc(1)-Cl(1)	104.48 (5)
Tc(1)-Cl(1)	2.367 (2)	Tc(1)-Tc(2)-Cl(2)	104.20 (6)
Tc(2)-Cl(2)	2.360 (2)	Cl(1)-Tc(1)-Cl(1')	86.41 (7)
Cl(1)-Cl(1')	3.241 (3)	Cl(2)-Tc(2)-Cl(2')	86.55 (8)
Cl(2)-Cl(2')	3.235 (3)	Cl(1)-Tc(1)-Cl(1'')	151.03 (7)
Cl(1)-Cl(2)	3.335 (3)	Cl(2)-Tc(2)-Cl(2'')	151.60 (8)

^{a} The numbers in parentheses in this and Table III are the estimated standard deviations in the least significant digits.
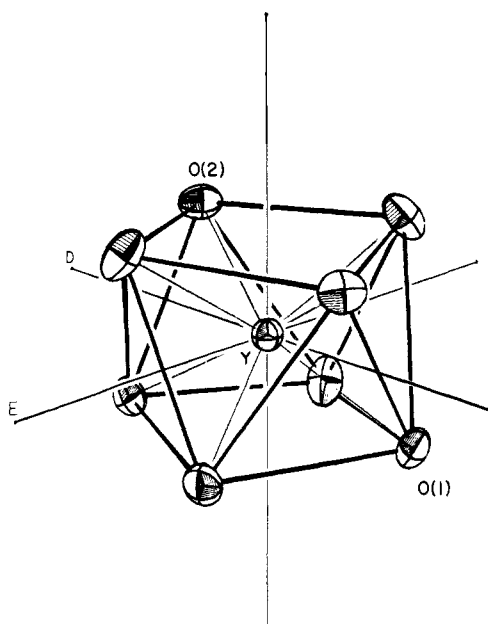

Figure 2. ORTEP drawing of the $Y(H_2O)_8$ coordination polyhedron. The twofold axes are included and are labeled as found in ref 22.

either space group, but the process of locating and refining the Cl and O atoms proceeded successfully only in $P42_12$, and this space group proved satisfactory for the complete refinement of the structure. Anomalous dispersion corrections were made, but extinction effects were neglected throughout.

The other (and presumably correct) enantiomer, selected by comparing the results of refining both, gave appreciably better residuals than the one we had been using. Refinement of this enantiomer, using an empirical weighting scheme,[10] converged with $R_1 = 0.031$, $R_2 = 0.039$, and the esd of an observation of unit weight equal to 1.314. Details and definitions pertaining to many other aspects of the refinement will be found in the supplementary material.

Results

The unit cell contains $[Tc_2Cl_8]^{3-}$ anions, $[Y(H_2O)_8]^{3+}$ cations, and lattice water molecules. Coordinates for all atoms are listed in Table I. Figures 1 and 2 show the anion and

(10) Empirical weights ($w = 1/\sigma^2$) were calculated from $\sigma = \sum a_n F_o^n = 0.985 + 0.0103F_o - (0.192 \times 10^{-3})F_o^2 + (0.204 \times 10^{-5})F_o^3$ where a_n was derived from the least-squares fitting of the curve $||F_o| - |F_c|| = \sum a_n |F_o|^n$.

Table III. Interatomic Distances and Angles in $Y(H_2O)_8^{3+}$

atoms[a]	length, Å	atoms[a]	length, Å
Y-O(1)	2.346 (5)	Y-O(2)	2.373 (5)
O(1)-O(2)	3.033 (7)	O(1)-O(1b)	2.973 (7)
O(1)-O(2a)	2.782 (7)	O(1)-O(2b)	2.860 (7)
O(2)-O(2a)	2.763 (7)	O(1)-O(1c)	3.881 (8)
O(2)-O(2c)	4.088 (8)		

atoms[a]	angle, deg	atoms[a]	angle, deg
O(2a)-O(1)-O(2b)	92.9 (3)	O(1a)-O(2)-O(1b)	87.0 (3)

[a] Lower case letters following the atom numbers in parentheses indicate the following symmetry related position: a, $1-x$, $1-y$, z; b, y, x, $-z$; c, $1-y$, $1-x$, z.
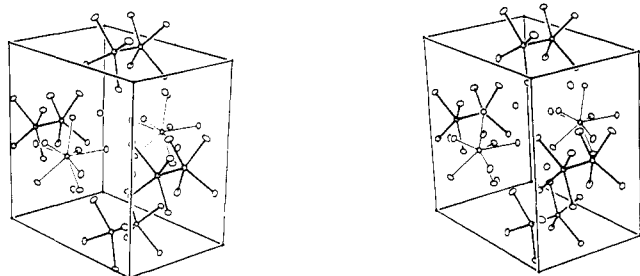
Figure 3. Stereoscopic view of the unit cell of $YTc_2Cl_8 \cdot 9H_2O$.

cation, respectively, and the dimensions of these ions are given in Tables II and III. A stereoview of the unit cell and its contents is shown in Figure 3. The $[Tc_2Cl_8]^{3-}$ anion possesses rigorous crystallographic C_4 symmetry, the fourfold axis being coincident with the metal–metal bond. The Tc–Tc bond distance is 2.105 (1) Å while the average Tc–Cl distance is 2.364 (2) Å. These distances are in good agreement with those found in $K_3Tc_2Cl_8 \cdot nH_2O^4$. The mean Cl–Tc–Cl' and Cl–Tc–Cl" angles are 86.48 (8) and 151.32 (8)°, respectively, with a mean Tc–Tc–Cl angle of 104.34 (6)°. The torsional angle between the Cl(1)–Tc(1)–Tc(2) and Tc(1)–Tc(2)–Cl(2) planes is 15.8°.

The yttrium cation occupies a crystallographic 222 (b) site. The coordination polyhedron of eight water molecules thus possesses rigorous D_2 symmetry. The polyhedron can best be described as a square antiprism slightly compressed along an axis perpendicular to the square faces, as shown in Figure 2 where the three mutually perpendicular twofold axes are included. The oxygen atoms making up the square faces are coplanar within 0.3 Å. The distance between the two least-square planes is 2.5 Å. Contact distances between oxygen atoms and chlorine atoms of the anion, averaging 3.190 (5) Å, suggest the presence of hydrogen bonds between these atoms. However, attempts to refine possible hydrogen atoms were unsuccessful. The ninth water molecule was located lying on a twofold axis. This water molecule may be associated with two symmetry-related O(2) atoms of the yttrium coordination polyhedron through hydrogen bonds, having a contact distance of 2.740 (5) Å. The hydrogen atom, however, was not located.

Discussion

The principal result of this study has been to verify the previously reported Tc–Tc distance in the $[Tc_2Cl_8]^{3-}$ ion. The value found here, 2.105 (1) Å, is in fact slightly shorter than that previously found in the potassium salt, 2.117 (2) Å, by 0.012 (2) Å, which is statistically real. This still leaves unanswered the question of whether a significantly longer distance might be seen in a compound with large organic cations such as NR_4^+ or $Ph_2PNPPh_2^+$. We do not wish to discount this possibility entirely, but in view of the fact that, as the following figures[11] show, the Mo–Mo distance in $[Mo_2Cl_8]^{4-}$

compounds shows no suggestion of such a pattern, we think it is unlikely:

$K_4[Mo_2Cl_8] \cdot 2H_2O$	2.139 (4) Å
$(NH_4)_5[Mo_2Cl_8]Cl \cdot H_2O$	2.150 (5) Å
$(H_3NC_2H_4NH_3)_2[Mo_2Cl_8] \cdot 2H_2O$	2.134 (1) Å

We accept the fact that the Tc–Tc distance in the $[Tc_2Cl_8]^{3-}$ ion, 2.110 (5) Å, is truly about 0.041 (5) Å shorter than that in the $[Tc_2Cl_8]^{2-}$ ion, 2.151 (1) Å,[2] for reasons that are intrinsic to the electronic structures of the ions themselves and not simply as a result of external electrostatic forces.

Earlier, we presented arguments to suggest that the unexpected order of the $[Tc_2Cl_8]^{2-}$ and $[Tc_2Cl_8]^{3-}$ distances might be attributable more to the former being "too long" than to the latter being "too short", in comparison to values that might be extrapolated from related systems. Since we have confirmed the correctness of the result for $[Tc_2Cl_8]^{3-}$, we continue to favor this viewpoint.

It may be noted that a comparison of ν_{Tc-Tc} frequencies in the $[Tc_2Cl_8]^{3-}$ and $[Tc_2Cl_8]^{2-}$ ions is also consistent with the former having a stronger Tc–Tc bond than the latter. For the ground state of $[Tc_2Cl_8]^{2-}$, a value of 307 cm^{-1} has been reported,[12] and in the excited state resulting from the $\delta \rightarrow \delta^*$ transition, a value of 280 cm^{-1} is inferred from vibrational structure on the electronic transition.[12,13] For $[Tc_2Cl_8]^{3-}$ the ground[14] and excited-state[6] values are 370 and 320 cm^{-1}, respectively. For both the ground and excited states, the $[Tc_2Cl_8]^{3-}$ ion has higher ν_{Tc-Tc} values (by 63 and 43 cm^{-1}, respectively) than the $[Tc_2Cl_8]^{2-}$ ion. This is consistent with the shorter Tc–Tc bond in the $[Tc_2Cl_8]^{3-}$ ion and provides independent evidence that the structural results are valid.

We believe that the only tenable explanation for this result must rest on the fact that as the oxidation state of the technetium atoms is changed from +3 in $[Tc_2Cl_8]^{2-}$ to +2.5 in $[Tc_2Cl_8]^{3-}$ the strengths of *all* components, σ, π, and δ, of the Tc–Tc bond must change. One effect of lowering the formal charge on the metal atoms is to allow a slight expansion of the 4d orbitals that are principally responsible for Tc–Tc bonding. Another effect will be to increase the role of the 5s or 5p orbitals in σ bonding. The expansion of the d orbitals may lead to either an increase or a decrease in the strengths of the σ, π, and δ bonds depending on whether they were initially so contracted that optimum overlap was not attained or whether overlap was already optimum so that further expansion would cause a decrease in overlap.

We suggest that when the initial formal charges on the metal atoms are 3+, a moderate decrease to 2.5+ is likely to improve some or all of the d–d overlaps. Some numerical calculations by Dr. B. E. Bursten on the Tc_2^{q+} system,[15] with q varying from 6 to 2, indicate that the $d\pi$–$d\pi$ overlap and the $d\delta$–$d\delta$ overlaps increase significantly from $q = 6$ to $q = 3$ while the $d\sigma$–$d\sigma$ overlap decreases. In this system the charge was varied by introducing electron density into the second set of δ orbitals ($d_{x^2-y^2}$ on each Tc atom). It would be foolish to try to extract too much from this simple model (although it suggests that study of a more sophisticated model would be rewarding), but

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(12) Preetz, W.; Peters, G. *Z. Naturforsch., B: Anorg. Chem., Org. Chem.* **1980**, *35B*, 797.

(13) Martin, D. S., Iowa State University, unpublished observations on (N-n-Bu$_4$)$_2$Tc$_2$Cl$_8$ at 6 K.

(14) Cotton, F. A.; Fanwick, P. E.; Gage, L. D. *J. Am. Chem. Soc.* **1980**, *102*, 1570.

(15) Private communication from Professor Bursten. It may be noted that for the Mo$_2^{q+}$ system[16] for $4 \geq q \geq 1$ the π–π^* separation showed a slight decrease while the σ–σ^* and δ–δ^* separations behaved as in the case of Tc$_2^{q+}$, namely, with the former decreasing and the latter increasing. It looks as though for Mo$_2^{q+}$ with $4 \leq q \leq 6$ the π–π^* separation would decrease from a maximum lying between $q = 3$ and $q = 4$.

(16) Bursten, B. E.; Cotton, F. A. *Faraday Discuss. Chem. Soc.* **1980**, *No. 14*.

it does support the following suggestion.

When the $[Tc_2Cl_8]^{2-}$ ion is reduced to the $[Tc_2Cl_8]^{3-}$ ion, there is a direct small increase in the strengths of the d–d π and δ bonding interactions. The fate of the σ bonding is less clear. The d–d contribution thereto may decrease, but this may be partially mitigated by an increase caused by greater participation of the 5s orbitals. The net result is that increased π and δ bonding, but especially the former, outweighs the antibonding effect of introducing one δ^* electron, thus strengthening and shortening the Tc–Tc bond and raising the stretching force constant.

It is to be noted that an analogous argument could help to explain why the Re–Re bond in $Re_2Cl_4(PEt_3)_4$, 2.232 (6) Å,[17] is not appreciably longer than those in $[Re_2Cl_8]^{2-}$ and $Re_2Cl_8(PEt_3)_2$, 2.222 (3) Å, despite the addition of two δ^* electrons.

Another pertinent comparison is with the bond-length increases observed[18] in the series $[Mo_2(SO_4)_4]^{4-}$, $[Mo_2(SO_4)_4]^{3-}$, and $[Mo_2(HPO_4)_4]^{2-}$, viz., 2.11 to 2.17 to 2.22 Å, as the number of δ electrons decreases from 2 to 1 to 0. While it has been assumed in the past that this series of decreases could be attributed entirely to the loss of δ electrons, we must now recognize that loss of π bonding due to d orbital contraction as the formal charge of the metal atoms increases from 2+ to 2.5+ to 3+ would operate in the same direction and may well play a considerable part in the total observed effect.

The lanthanide elements in oxidation state III generally exhibit coordination numbers of 7, 8, or 9, with 8 being the most common.[19,20] Yttrium, by virtue of its ionic radius and

general chemical properties, is well-known to behave like one of the later lanthanides.[19] The calculations of Kepert,[21] which are particularly relevent here since there is no partially filled inner shell of the metal ion, show that the square antiprism (SAP) and dodecahedral (DD) structures differ little in energy and can interconvert readily. In the present case the arrangement approximates to the SAP but show an appreciable distortion therefrom in that the "square faces" are not truly square. There are two prominent deviations from a square arrangement: (1) the four oxygen atoms are far from coplanar, the perpendicular distance between the two diagonals of the "square" being 0.1278 Å, and (2) the corner angles in the "square" are 92.9° and 87.0°. In view of these distortions, it is not worthwhile to attempt a lengthy examination of the critical geometrical properties of this approximate SAP. Since we were unable to locate the hydrogen atoms reliably, we cannot comment on the role of hydrogen bonding in contributing to the observed distortions except to say that it undoubtedly plays some role.

Registry No. $YTc_2Cl_8 \cdot 9H_2O$, 80327-34-0.

Supplementary Material Available: Tables of final calculated and observed structure factors, thermal parameters, and further details of the experimental and computational work (10 pages). Ordering information is given on any current masthead page.

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