Title
CRYSTAL AND MOLECULAR STRUCTURE OF [Nd(tren)2(CH3CN)] (C104)3

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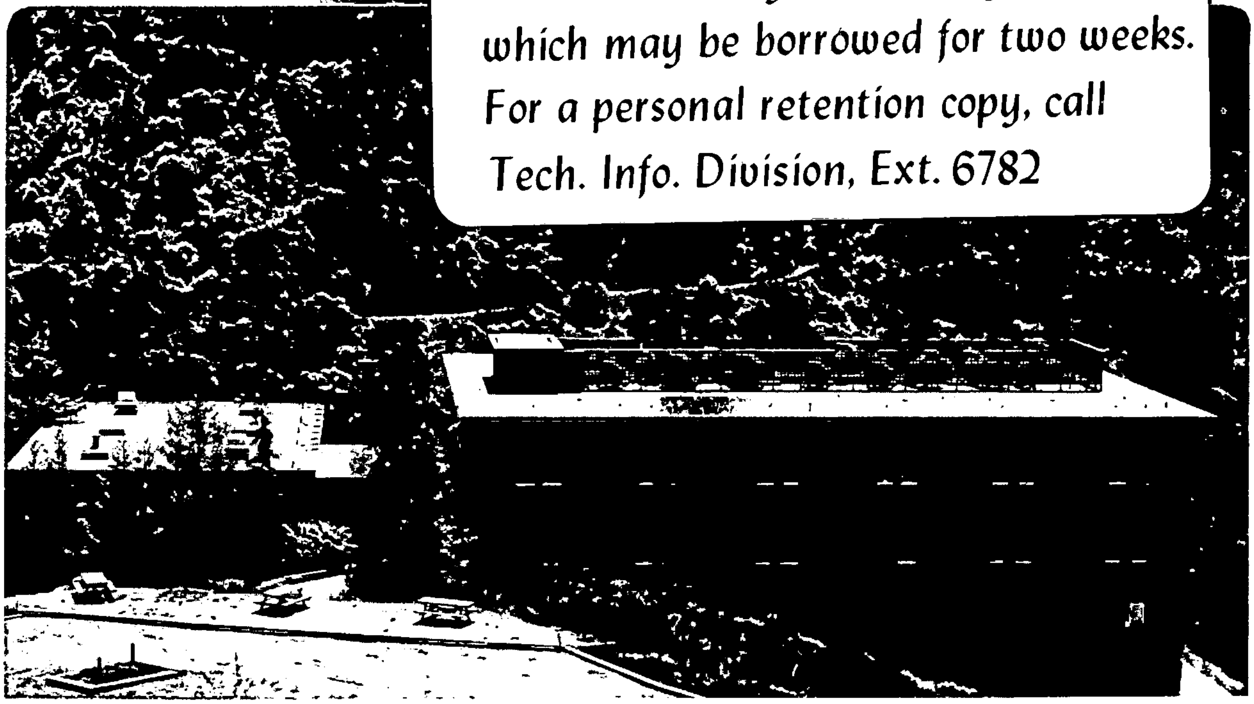

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Crystal and Molecular Structure of $[Nd(tren)_2(CH_3CN)](ClO_4)_3$

By

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Abstract

The crystal and molecular structure of the title compound [tren = $N(CH_2CH_2NH_2)_3$] has been determined by single crystal X-ray diffraction. The structure consists of a three-dimensional hydrogen-bonding network of perchlorate anions and $[Nd(tren)_2(CH_3CN)]^{3+}$ cations at general positions in the unit cell. The coordination of the Nd^{3+} ion is by two tetradentate tren ligands and the nitrogen atom from an acetonitrile molecule, for a total coordination number of 9. The coordination polyhedron is a tri-capped trigonal prism. Pink crystals from acetonitrile/benzene conform to space group Cc with a = 15.044(1), b = 17.729(1), c = 11.088(1) Å, β = 95.079(5)°, and four formula weights per unit cell. For the 3553 independent reflections with $F^2 > 3\sigma(F^2)$, the final weighted and unweighted R factors are 3.19% and 2.94%, respectively. A similar complex of 1,5,9,13-tetraazacyclohexadiene, $[Nd([16]aneN_4)][ClO_4]_3$, has been prepared and its structure partially characterized.

Introduction

Lanthanide coordination chemistry is dominated by oxygen donor ligands, especially by chelate ligands such as β-diketonates or EDTA. However, recently several nitrogen-bonded complexes have been well characterized. As with oxygen donor ligands, amine complexes are generally more stable for chelating ligands. For example, pyridine complexes exist only in solution, while complexes with ethylenediamine or 1,10-phenanthroline can be isolated.[1] In addition, Forsberg and co-workers have recently added compounds of the type $Ln(tren)X_3$ [tren = $N(CH_2CH_2NH_2)_3$] and $Ln(tren)_2X_3$ to the list of lanthanide amine complexes.[2,3] We are interested in the structures of these compounds and their possible use as template compounds in macrocycle synthesis. There has been a continuing interest in the use of lanthanide ions as shift reagents in nmr spectroscopy. During the last decade,[4] lanthanide shift reagents have been actively investigated and utilized in simplifying complex nmr spectra. The utility of these compounds has been hampered to some extent by the simplifying assumptions that must be made — including the geometries. It would be extremely interesting to have a substitutionally inert lanthanide complex formed by an amine macrocycle, such as can be envisioned from $Ln(tren)_2$ as a precursor. This paper reports the structural characterization of $[Nd(tren)_2(CH_3CN)](ClO_4)_3$, as the first step in this project.

Experimental

Manipulation of moisture-sensitive materials was accomplished with Schlenk techniques and the use of a Vacuum Atmospheres HE-93-A glove box with recirculating moisture-free argon atmosphere. Elemental analyses were performed by the Microanalytical Laboratory, University of California, Berkeley. Infrared spectra were obtained on a Perkin-Elmer 597 spectrophotometer (Nujol mulls, reported in cm^{-1}).

Materials

Acetonitrile, CH_3CN, was distilled from P_2O_5, benzene from potassium benzophenone ketyl. The tren $[N(CH_2CH_2NH_2)_3]$ was extracted from crude triethylenetetraamine.[2] Crystalline 16-cyclam was a generous gift from William Smith.[5]

Neodymium perchlorate, $Nd(ClO_3)_3$, was prepared by addition of excess Nd_2O_3 to 70 percent $HClO_4$[6]. The excess Nd_2O_3 was removed by filtration and the solution evaporated to dryness. Residual H_2O was removed by heating to ∿ 250°C under vacuum for three days. Some reversion to the oxide was evidenced by the presence of blue amongst the pink perchlorate. This contaminant was conveniently left behind during an extraction into acetonitrile. Evaporation of the solution left a compound of formula $Nd(ClO_4)_3(CH_3CN)_4$.

Anal. Calcd for $Nd(ClO_4)_3(CH_3CN)_4$: C, 15.83; H, 1.98; N, 9.27; Nd, 23.78. Found: C, 16.23; H, 2.26; N, 9.23; Nd, 24.88.

$[Nd(16\text{-}aneN_4)][ClO_4]_3$. The combination of $Nd(ClO_4)_3(CH_3CN)_4$ and 16-cyclam (16-aneN_4) in a 1:2 molar ratio leads to an intractable blue solid that was not characterized. The similar reaction in a 1:1 molar ratio leads to the formation of an acetonitrile-soluble material of formula $[Nd(16\text{-}cyclam)](ClO_4)_3$.

Anal. Calcd for $NdC_{12}H_{24}Cl_3O_{12}N_4$: C, 21.47; H, 4.17; N, 8.35; Nd, 21.51. Found: C, 21.71; H, 4.23; N, 8.80; Nd, 21.11.

The 5355 single crystal X-ray diffraction data were collected between 4° and 55° 2θ in space group $Pn2_1a$ with a = 18.192(2), b = 15.762(2), c = 8.096(1) Å and Z = 4. Refinements in this and the centric space group Pnma have not yielded a totally satisfactory solution. We are able to report that the Nd^{3+} ion is seven or eight coordinate with coordination by four 16-cyclam nitrogen atoms, one oxygen atom from each of two of the perchlorate anions, and one or two oxygen atoms from the remaining perchlorate anion. The Nd-N and Nd-O bond lengths are about 2.5 Å.

$Nd(tren)_2(ClO_4)_3$. This complex was prepared after Forsberg.[3] Crystals suitable for diffraction were obtained by the addition of benzene to a concentrated acetonitrile solution. After standing at room temperature for two days, the clear pink solution yielded several large, well-formed crystals. Infrared spectroscopy revealed the presence of acetonitrile (ν_{CN} = 2262 cm^{-1}).

Data Collection, Solution, and Refinement[7]

The absences identified with the precession camera (hkℓ, h+k odd; h0ℓ, ℓ odd) are consistent with the space groups Cc and C2/c. The cell parameters were determined using 25 automatically centered reflections with 2θ between 27 and 43°. They are a = 15.0442(11), b = 17.7290(14), c = 11.0880(6) Å, β = 95.079(5)°. These data, in conjuction with the measured density (1.751), yield Z = 4 (d_{calc} = 1.750 g cm^{-3}).

The initial Patterson map confirmed the non-centric space group Cc. The structure was then solved using heavy atom techniques. The model refined to weighted and unweighted R factors of 3.19 and 2.94%, respectively.[9] (The initial polarity refined to 3.58 and 3.24%.) Final positional and thermal parameters appear in Table I. Pertinent bond distances and angles appear in Table II.

Discussion

The crystal structure consists of discrete, mononuclear cations at general positions in the unit cell (Figure 1) engaged in a three dimensional network of hydrogen bonds. The structure of the molecular cation (Figure 2) consists of Nd^{3+} coordinated by eight tren nitrogens and an acetonitrile nitrogen, to form a tri-capped trigonal prismatic coordination geometry. The inclusion of two tetradentate tren ligands in the coordination sphere of one neodymium ion is a reflection of the lanthanide's relatively large size. As in other tren structures (vide infra), the intraligand bond angles indicate the ligand molecule is essentially unstrained.[11] The capping nitrogens are the two tertiary tren nitrogens and that of the acetonitrile. Data on the various planes and their interrelationship can be found in Table III. An idealized tri-capped trigonal prism exhibits angles of 0° between end faces, 60° between rectangular faces, and 90° between the two sets of faces. The data in Table III reveal a close resemblance to the idealized polyhedron (see Figure 2) and suggest a molecular (non-crystallographic) twofold axis along the acetonitrile (Figure 3). A twofold axis was then defined as the vector from Nd to a point whose coordinates were obtained by the summation of all pseudo-twofold related atoms (C's and N's). Rotation by 180° about this axis and calculation of the difference between the new atom positions and the old ones quantitates the quality of the twofold[11] axis. The average difference is 0.21 Å with a standard deviation of 0.16 Å. The major deviations from the twofold symmetry are the carbon atoms of the acetonitrile; the remaining atoms have an average difference of 0.16 Å with a standard deviation of 0.06 Å. When considering only the nitrogen atoms that constitute the coordination sphere, the average difference is 0.11 Å, with a standard deviation of 0.03 Å.

Figure 4 illustrates the hydrogen-bonding around a cation, between perchlorate oxygens and some of the primary amine nitrogens. There are eight such bonds within 3.25 Å, ranging from 3.11 to 3.25 Å.[11] Among the other metal tren structures published is another example of a perchlorate salt, $[Co(tren)(glycinato)]Cl(ClO_4)$, where a more extensive hydrogen bonding network was found.[12] Of the remaining tren structures that have been reported,[14-16] the ligands in each case are tetradentate.

The compound $[Eu(terpy)_3](ClO_4)_3$[17] is the only other lanthanide structure determined to date in which all coordination sites are occupied by nitrogen atoms. In this and other mixed lanthanide amine complexes, the M-N bond distances are in general agreement with those predicted from the metal ionic radii. The M-N distances in $[Eu(terpy)_3]$-$(ClO_4)_3$ are about 0.05 Å shorter than those found in the present compound. This difference is about what one would expect for the difference between an sp^3 and an sp^2 hybridized nitrogen atom (0.08 Å) and the difference in ionic radii of Nd^{3+} and Eu^{3+} ion (0.04 Å).[18]

The crystal structure was carried out in part to determine the appropriate number of carbon atoms needed to bridge across the tren ligands in order to encapsulate the metal ion. The proposed encapsulation would bridge the closest nitrogens (Table IV): 3 and 7, 6 and 4, and 2 and 8. In the structure of 16-aneN_4,[5] non-bonded nitrogens are separated by 2.9 Å and bridged by propyl chains. This appears to be the appropriate length for an encapsulating reaction.

Acknowledgment

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Supplementary Material

Ordering information is given on any current masthead page.

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6. Forsberg, J. H.; Moeller, T. *Inorg. Chem.* **1969**, *8*, 883.
7. A total of 3819 $+h$, $+k$, $\pm\ell$ data were collected between 4 and 56° in 2θ, monitoring three intensity standards every 7200 seconds of X-ray exposure, and checking three orientation standards every 250 reflections. Azimuthal scans were collected on four reflections with 2θ between 11 and 33°. Data reduction and processing were carried out as described elsewhere.[8] The intensity standards underwent decay of 14, 14, and 17% during data collection. A decay correction of 14% was applied. The six crystal faces were identified with the help of the diffractometer and their dimensions measured at 7X under a binocular microscope. The absorption correction ($\mu = 21.09$ cm^{-1}) applied ranged between 1.212 and 1.498. The data were then averaged (R = 2.6%) to yield the 3553 independent reflections used in refinement.
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9. After a difference Fourier revealed most of the hydrogens, their positions were calculated with a C-H distance of .95 Å and an N-H distance of .87 Å.[10] The temperature factors of all atoms were refined anisotropically except for those of the hydrogens, where the isotropics B's were set at 7.0 $\AA^2$ and not refined. On the final least-squares cycle, all parameters shifted by less than 0.06 σ. In the final difference Fourier, the largest peak at a grid point was .55 e^{-} $\AA^3$ and the most negative electron density was -.38 e^{-} $\AA^3$. The residuals showed no abnormalities.

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11. Supplementary material includes tabulations of the difference between new and old atom positions after rotation by 180° about the pseudo twofold axis, the intraligand bond angles, and the hydrogen bonding distances.

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Table Ia. Positional and thermal parameters (x 10^4) for $[Nd(tren)_2(CH_3CN)](ClO_4)_3$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Nd	-.250000	-.235763(17)	0.	31.49(13)	22.12(8)	60.9(2)	-3.1(3)	11.22(12)	-.6(4)
Cl(1)	-.16340(17)	-.36018(14)	-.5761(2)	66.7(14)	42.9(9)	98.(2)	-9.7(9)	-4.2(13)	16.7(11)
Cl(2)	-.05706(16)	-.07716(15)	-.3868(3)	50.7(12)	49.4(10)	147.(3)	8.2(8)	27.7(15)	27.8(14)
Cl(3)	-.38879(16)	-.15158(13)	-.4311(2)	57.3(12)	39.9(9)	98.(2)	-6.2(8)	-.5(13)	8.8(10)
N(1)	-.2693(4)	-.3781(3)	-.0893(6)	45.(3)	25.(2)	84.(6)	-2.(2)	9.(3)	-8.(3)
N(2)	-.4074(5)	-.2625(4)	-.1225(6)	43.(3)	40.(3)	97.(6)	2.(2)	1.(4)	4.(4)
N(3)	-.2009(5)	-.2504(4)	-.2184(6)	51.(4)	43.(3)	81.(6)	-6.(2)	20.(4)	-3.(3)
N(4)	-.1339(5)	-.3341(4)	.0931(6)	54.(4)	36.(3)	84.(6)	7.(2)	10.(4)	4.(3)
N(5)	-.3521(4)	-.1357(3)	.1090(5)	53.(4)	25.(2)	80.(6)	2.(2)	23.(4)	2.(3)
N(6)	-.1767(5)	-.1829(5)	.2069(7)	67.(5)	42.(3)	109.(8)	-7.(3)	-6.(5)	-15.(4)
N(7)	-.2668(5)	-.1045(4)	-.1092(6)	61.(4)	34.(3)	77.(6)	-2.(2)	18.(4)	7.(3)
N(8)	-.3470(5)	-.2967(4)	.1601(6)	47.(4)	32.(3)	83.(6)	-6.(3)	18.(4)	6.(3)
N(9)	-.0869(5)	-.1767(4)	-.0183(7)	44.(4)	39.(3)	109.(7)	-12.(2)	13.(4)	5.(3)
O(1)	-.1006(6)	-.3106(5)	-.6216(8)	119.(7)	68.(4)	171.(10)	-40.(4)	5.(6)	29.(5)
O(2)	-.2261(8)	-.3140(10)	-.5198(17)	92.(9)	191.(12)	435.(29)	29.(8)	-8.(13)	-159.(17)
O(3)	-.2144(7)	-.3973(5)	-.6718(7)	119.(6)	69.(4)	129.(8)	-26.(4)	-20.(6)	15.(4)
O(4)	-.1247(7)	-.4066(5)	-.4888(8)	149.(9)	58.(4)	164.(10)	-19.(5)	-51.(8)	29.(5)
O(5)	-.0450(8)	-.1462(8)	-.4427(13)	87.(7)	68.(5)	384.(25)	0.(5)	32.(10)	-75.(10)
O(6)	-.1082(6)	-.0882(6)	-.2891(7)	92.(5)	101.(5)	151.(9)	-25.(4)	63.(6)	13.(5)
O(7)	-.1003(6)	-.0294(6)	-.4732(8)	89.(6)	83.(5)	191.(11)	10.(4)	-16.(6)	55.(6)
O(8)	.0299(5)	-.0471(5)	-.3472(8)	68.(4)	67.(4)	172.(9)	-13.(3)	11.(5)	21.(5)
O(9)	-.4388(8)	-.1258(10)	-.5321(8)	94.(7)	174.(12)	74.(7)	30.(7)	15.(5)	24.(7)
O(10)	-.4169(9)	-.1141(9)	-.3288(8)	190.(12)	154.(9)	125.(10)	65.(8)	-35.(9)	-47.(8)
O(11)	-.4016(12)	-.2248(6)	-.4150(13)	272.(18)	53.(5)	309.(21)	-46.(7)	-43.(15)	47.(8)
O(12)	-.3019(7)	-.1364(10)	-.4381(16)	61.(6)	161.(10)	483.(29)	-14.(6)	0.(11)	127.(14)
C(1)	-.2191(6)	-.3863(5)	-.1983(8)	64.(5)	38.(3)	92.(8)	2.(3)	22.(5)	-11.(4)
C(2)	-.1531(6)	-.3235(6)	-.2122(9)	52.(5)	50.(5)	97.(9)	3.(4)	33.(5)	-9.(5)
C(3)	-.3655(7)	-.3960(6)	-.1191(9)	55.(5)	33.(4)	108.(9)	-10.(3)	2.(6)	-4.(5)
C(4)	-.4132(6)	-.3352(6)	-.1904(8)	51.(5)	44.(4)	113.(9)	-14.(3)	-11.(5)	-12.(5)
C(5)	-.2343(8)	-.4307(4)	.0066(14)	54.(7)	25.(2)	119.(8)	9.(3)	9.(6)	-2.(6)
C(6)	-.1427(7)	-.4139(6)	.0534(10)	68.(6)	32.(3)	127.(11)	8.(3)	-2.(7)	2.(5)
C(7)	-.3000(7)	-.0938(5)	.2073(8)	85.(7)	30.(3)	96.(8)	-4.(3)	26.(6)	-14.(4)
C(8)	-.2025(10)	-.1043(7)	.2124(12)	114.(10)	51.(6)	161.(15)	-27.(6)	-1.(9)	-16.(7)
C(9)	-.3890(6)	-.0829(5)	.0147(8)	57.(5)	34.(3)	106.(8)	13.(3)	26.(5)	9.(4)
C(10)	-.3206(7)	-.0465(5)	-.0537(9)	91.(7)	26.(3)	111.(9)	3.(3)	27.(6)	10.(4)
C(11)	-.4256(6)	-.1766(5)	.1614(8)	49.(4)	37.(3)	113.(8)	8.(3)	39.(5)	7.(4)
C(12)	-.3950(7)	-.2431(5)	.2334(8)	67.(5)	39.(3)	98.(8)	0.(3)	46.(5)	9.(4)
C(13)	-.0226(6)	-.1446(5)	-.0283(7)	49.(4)	31.(3)	93.(8)	-2.(3)	11.(5)	0.(3)
C(14)	.0592(7)	-.1033(6)	-.0405(10)	53.(5)	47.(4)	132.(11)	-14.(4)	19.(6)	-7.(5)

Table Ib. Calculated hydrogen atom positions[a] for $[Nd(tren)_2(CH_3CN(](ClO_4)_3$

	x	y	z
H(1A)	-.18764	-.43271	-.19302
H(1B)	-.26082	-.38673	-.26808
H(2A)	-.12453	-.33067	-.28414
H(2B)	-.10985	-.32322	-.14437
H(3A)	-.36991	-.44139	-.16444
H(3B)	-.39258	-.40258	-.04553
H(4A)	-.47405	-.34907	-.20644
H(4B)	-.38697	-.32913	-.26465
H(5A)	-.27123	-.42764	.07190
H(5B)	-.23648	-.48039	-.02523
H(6A)	-.10406	-.42249	-.00907
H(6B)	-.12572	-.44603	.11988
H(7A)	-.31226	-.04152	.19618
H(7B)	-.31943	-.10983	.28227
H(8A)	-.17636	-.08342	.28591
H(8B)	-.18079	-.07860	.14579
H(9A)	-.42098	-.04450	.05243
H(9B)	-.42897	-.10993	-.04082
H(10A)	-.28247	-.01641	-.00033
H(10B)	-.34882	-.01550	-.11570
H(11A)	-.45424	-.14289	.21231
H(11B)	-.46725	-.19275	.09709
H(12A)	-.44527	-.26793	.26153
H(12B)	-.35608	-.22720	.30063
H(13A)	-.44792	-.26290	-.07102
H(13B)	-.41836	-.22602	-.17401
H(14A)	-.24704	-.25210	-.27184
H(14B)	-.16592	-.21391	-.23618
H(15A)	-.08156	-.31863	.07690
H(15B)	-.13723	-.33375	.17109
H(16A)	-.19633	-.20769	.26701
H(16B)	-.11905	-.18683	.20995
H(17A)	-.21347	-.08624	-.11276
H(17B)	-.29123	-.11255	-.18214
H(18A)	-.38656	-.32568	.12132
H(18B)	-.31194	-.32363	.20926

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[a] Isotropic thermal parameters equal 7.0 $e^-/\AA^3$.

Table II. Pertinent bond distances (Å) for $[Nd(tren)_2(CH_3CN)](ClO_4)_3$

Nd-N(1)	2.717(6)	Nd-N(6)	2.629(7)
Nd-N(2)	2.666(7)	Nd-N(7)	2.625(7)
Nd-N(3)	2.607(6)	Nd-N(8)	2.628(7)
Nd-N(4)	2.615(7)	Nd-N(9)	2.692(7)
Nd-N(5)	2.701(6)	Nd-N(ave)	2.65(4)
N(1)-C(1)	1.49(1)	N(5)-C(7)	1.49(1)
C(1)-C(2)	1.51(1)	C(7)-C(8)	1.47(2)
C(2)-N(3)	1.48(1)	C(8)-N(6)	1.45(1)
N(1)-C(3)	1.49(1)	N(5)-C(9)	1.47(1)
C(3)-C(4)	1.48(1)	C(9)-C(10)	1.48(1)
C(4)-N(2)	1.49(1)	C(10)-N(7)	1.48(1)
N(1)-C(5)	1.48(1)	N(5)-C(11)	1.48(1)
C(5)-C(6)	1.46(2)	C(11)-C(12)	1.48(1)
C(6)-N(4)	1.48(1)	C(12)-N(8)	1.48(1)
N(9)-C(13)	1.14(1)	C(13)-C(14)	1.45(1)
Cl(1)-O(1)	1.416(8)	Cl(2)-O(5)	1.391(11)
Cl(1)-O(2)	1.432(12)	Cl(2)-O(6)	1.396(7)
Cl(1)-O(3)	1.416(8)	Cl(2)-O(7)	1.396(8)
Cl(1)-O(4)	1.361(8)	Cl(2)-O(8)	1.444(8)
Cl(3)-O(9)	1.371(10)	Cl(3)-O(10)	1.412(10)
Cl(3)-O(11)	1.327(10)	Cl(3)-O(12)	1.344(10)

Table III. Relationships between planes for $[Nd(tren)_2(CH_3CN)](ClO_4)_3$

Plane No.	N's	Ave. dev. (Å)	d-Nd (Å)	d-cap (Å)	Angle with planes 1, 2, 3, 4
1	2, 3, 7	-	1.83	-	-
2	4, 6, 8	-	1.80	-	12.58
3	2, 3, 4, 8	.18	.95	1.75	84.79
					91.91
4	3, 4, 6, 7	.09	.90	1.79	84.27
					96.75
					62.68
5	2, 7, 6, 8	.16	.96	1.72	89.63
					93.86
					54.44
					62.88

Table IV. Interatomic distances (Å) for $[Nd(tren)_2(CH_3CN)](ClO_4)_3$

Nitrogens	Distance
3, 7	3.06
6, 4	3.06
2, 8	3.24
7, 2	3.51
6, 8	3.27
2, 3	3.38
4, 8	3.42
3, 4	3.81
6, 7	3.90

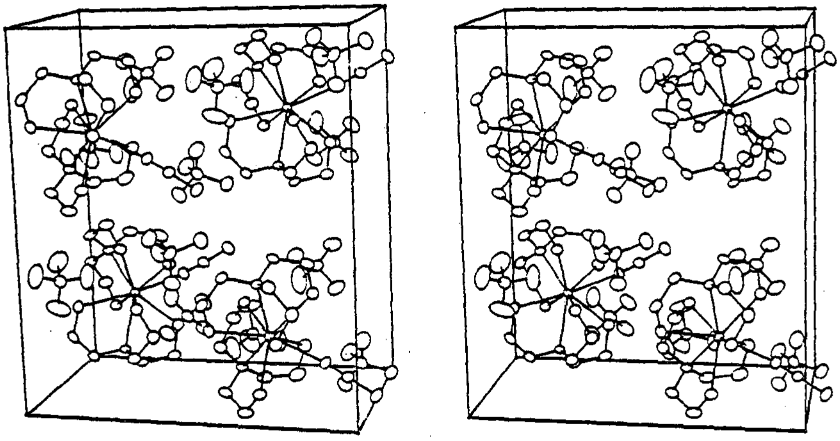
Figure Captions

Figure 1. Stereoscopic drawing of the unit cell of $[Nd(tren)_2(CH_3CN)](ClO_4)_3$.

Figure 2. ORTEP drawing of the molecular cation of $[Nd(tren)_2(CH_3CN)](ClO_4)_3$ emphasizing the pseudo-threefold symmetry. The nitrogen atoms are drawn at the 50% contour. For clarity, the carbon atoms are drawn at the 10% contour.

Figure 3. ORTEP drawing of the molecular cation of $[Nd(tren)_2(CH_3CN)](ClO_4)_3$ emphasizing the pseudo-twofold symmetry. The nitrogen atoms are drawn at the 50% contour. For clarity, the carbon atoms are drawn at the 10% contour.

Figure 4. ORTEP drawing of the hydrogen-bonding experienced by one molecular cation of $[Nd(tren)_2(CH_3CN)](ClO_4)_3$.



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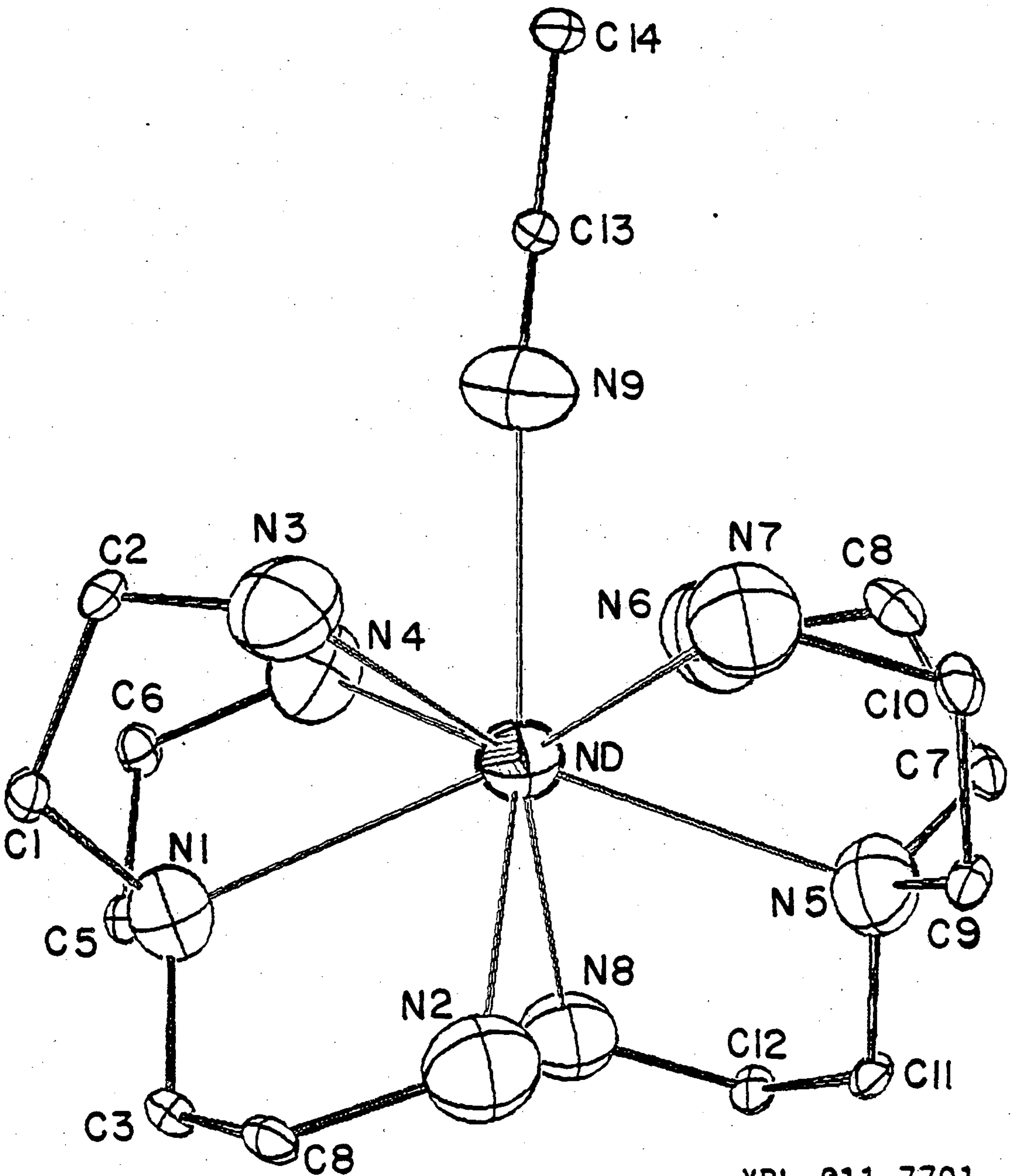
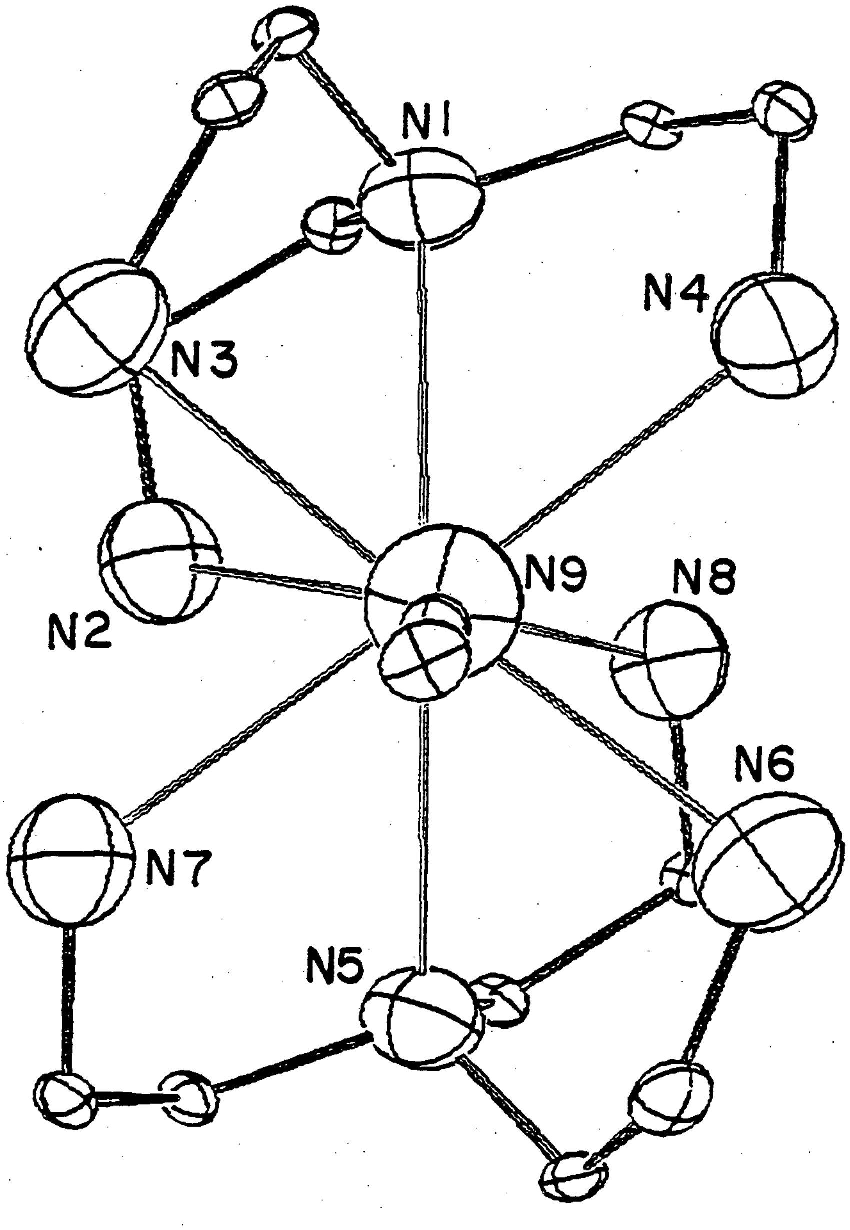

C14
C13
N9
N3
C2
N4
N7
C8
N6
C6
C10
ND
C7
C1
N1
N5
C5
C9
N8
N2
C12
C11
C3
C8

XBL 811-7791



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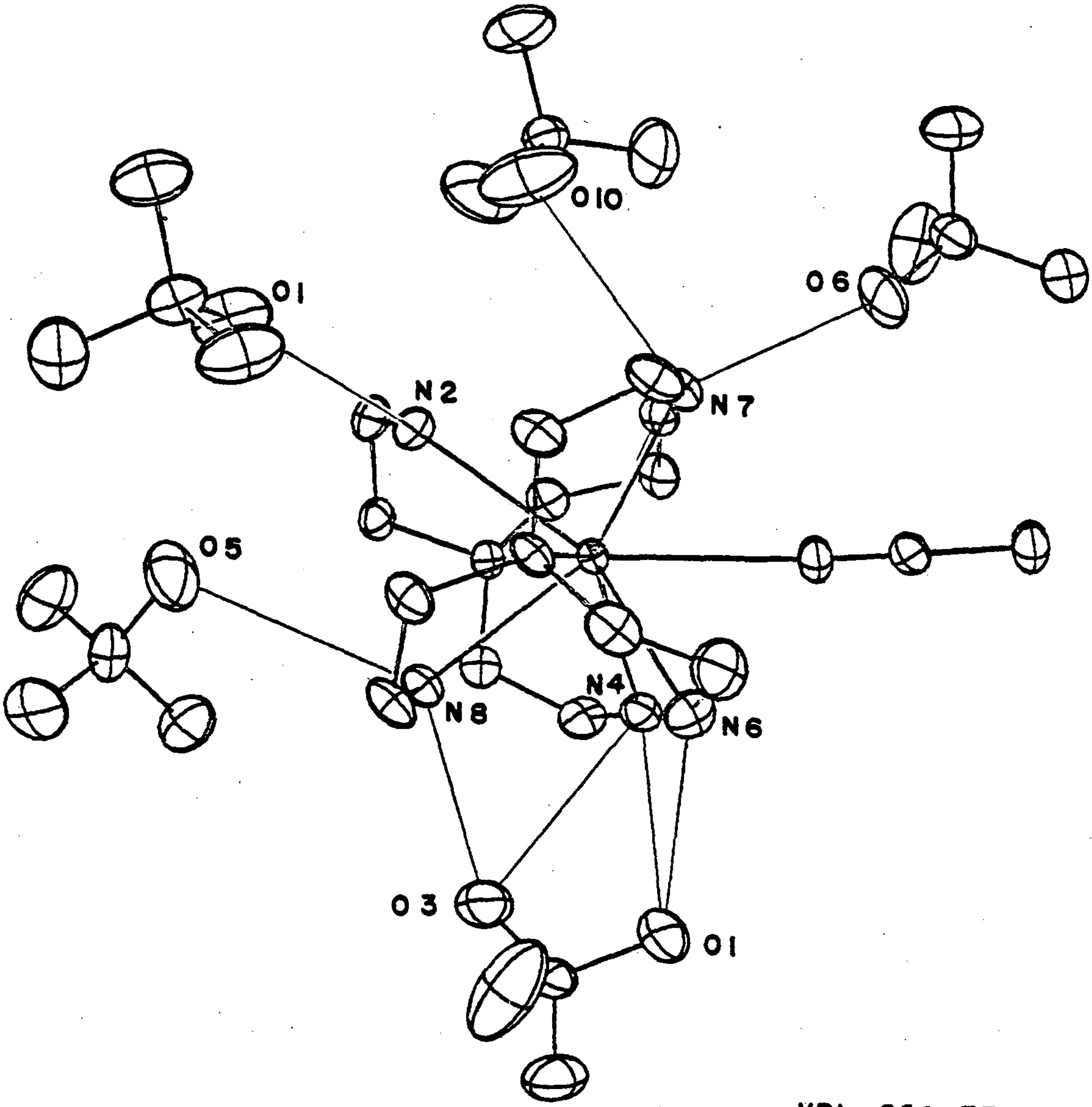

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SUPPLEMENTARY MATERIAL

Ligand bond angles for [Nd(tren)$_2$(CH$_3$CN)](ClO$_4$)$_3$ (± .2°)

C(1)-N(1)-C(3)	110.6	C(7)-N(5)-C(9)	110.1
C(3)-N(1)-C(5)	107.6	C(9)-N(5)-C(11)	109.9
C(5)-N(1)-C(1)	110.4	C(7)-N(5)-C(11)	108.7
N(1)-C(1)-C(2)	113.4	N(5)-C(7)-C(8)	115.1
C(1)-C(2)-N(3)	109.2	C(7)-C(8)-N(6)	112.9
N(1)-C(3)-C(4)	112.2	N(5)-C(9)-C(10)	113.9
C(3)-C(4)-N(2)	110.8	C(9)-C(10)-N(7)	110.0
N(1)-C(5)-C(6)	113.1	N(5)-C(11)-C(12)	113.3
C(5)-C(6)-N(4)	110.9	C(11)-C(12)-N(8)	111.1

Results of a twofold rotation for $[Nd(tren)_2(CH_3CN)](ClO_4)_3$

Nd , Nd	.000
N(1) , N(5)	.156
N(2) , N(8)	.113
N(3) , N(6)	.105
N(4) , N(7)	.129
N(9) , N(9)	.064
Average	.11
Std. Dev.	.03
X , X	.000
C(1) , C(7)	.152
C(2) , C(8)	.242
C(3) , C(11)	.173
C(4) , C(12)	.122
C(5) , C(9)	.242
C(6) , C(10)	.265
Average	.16
Std. Dev.	.06
C(13) , C(13)	.304
C(14) , C(14)	.687
Average	.21
Std. Dev.	.16

Hydrogen bond lengths for $[Nd(tren)_2(CH_3CN)](ClO_4)_3$ (Å)

N(2)-O(1)	3.18(1)	N(7)-O(6)	3.25(1)
N(4)-O(1)	3.19(1)	N(7)-O(10)	3.17(1)
N(4)-O(3)	3.17(1)	N(8)-O(3)	3.16(1)
N(6)-O(1)	3.11(1)	N(8)-O(5)	3.25(1)

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