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Crystal Structure and Magnetic Susceptibility of $[(CH_3)_3NH]_3Mn_2Cl_7$[1]

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The crystal structure of $[(CH_3)_3NH]_3Mn_2Cl_7$ has been determined and the magnetic susceptibility of the compound measured. The salt belongs to the polar hexagonal space group $P6_3mc$ with $a = 14.509$ (19) and $c = 6.415$ (7) Å, $Z = 2$. The structure consists of linear chains of face-shared $MnCl_6$ octahedra and discrete $MnCl_4^{2-}$ tetrahedra which are separated by the trimethylammonium ions. The susceptibility data can be explained by a model consisting of the susceptibility of a system of linear chains together with the susceptibility of the discrete $MnCl_4^{2-}$ tetrahedra.

Introduction

As part of a recent study in this laboratory of one-dimensional and psuedo-one-dimensional magnetic systems, the salt $[(CH_3)_3NH]_3Mn_2Cl_7$ ($T_3Mn_2Cl_7$) was prepared. Preliminary x-ray analysis indicated an interesting relationship to the structure of the one-dimensional antiferromagnet $(CH_3)_4NMnCl_3$, (TMMC)[2]—both salts are hexagonal with $c \approx 6.4$ Å—and so a structural and magnetic investigation of the salt was undertaken. As the analysis of the data was under way, we became aware of the more complete magnetic investigation in progress by Dr. McElearney, which is reported in the following paper in this issue.

Structual Data and Solution

The compound was prepared by combining 3:2 ratios of $(CH_3)_3NHCl$ and $MnCl_2$ in absolute ethanol. Pale pink needles were obtained by slow cooling of a hot, saturated solution. Anal. Calcd: C, 20.08; N, 7.80; H, 5.62. Found: C, 19.93; N, 7.74; H, 5.56.

Preliminary x-ray analysis revealed that the crystals were hexagonal with $6/mmm$ Laue symmetry. Systematic extinctions ($l = 2n + 1$ for hhl) indicated the possible space groups as $P6_3/mmc$ (centric), $P\bar{6}2c$ (acentric), or $P6_3mc$ (acentric). Subsequent structural analysis indicated that the third choice represented the correct space group. The lattice constants, determined from least-squares refinement of the reciprocal lattice coordinates of 12 accurately centered reflections ($\lambda_{MoK\alpha} = 0.71069$ Å), are $a = 14.509$ (19) and $c = 6.415$ (7) Å. The calculated density for two formula units per unit cell is 1.53 g/cm^3, in good agreement with the observed value of 1.53 g/cm^3.

Intensity data were collected on an automated Picker full-circle diffractometer with Zr-filtered Mo Kα radiation. The crystal was a hexagonal prism of approximately 0.2-mm cross section × 0.36-mm length mounted along the c axis. A total of 692 unique reflections were collected in the range $5° \leq 2\theta \leq 50°$ using a θ–2θ scan with a scan width of 2.2° with 20 steps/deg and 2 s/step. The standard deviation of each reflection was calculated by $\sigma^2(I) = TC + BG + 0.03^2I^2$, where TC is total counts, BG is background counts, and I = TC − BG. The intensities of three reflections were monitored every 20 reflections to check for decomposition, etc. No deviation from the statistical average was observed during the course of the data collection.

Structure Solution and Refinement

Statistical analysis of the structural data indicated the structure was acentric, thus eliminating the choice of $P6_3/mmc$ for the space group. The structure was refined in the space group $P6_3mc$ since, with only two molecules per unit cell, the choice of $P\bar{6}2c$ would have required both the $MnCl_4^{2-}$ tetrahedra and the cations to be disordered. Successful refinement in the former plus lack of satisfactory structural models in the latter (as judged by least-squares analysis) confirms $P6_3mc$ as the correct choice of space group.

Data were corrected for absorption ($\mu = 19.2$ cm^{-1}, transmission factors ranged from 0.70 to 0.72) and positions of the manganese and chlorine atoms were deduced from a Patterson synthesis. Carbon and nitrogen positions were determined from a subsequent difference map, and positional and thermal parameters were refined for these atoms. Hydrogen positions were located from another difference map, and refinement of all parameters except hydrogen thermal parameters, which were fixed at 4.0, converged to R $[=\sum||F_o| - |F_c||/\sum|F_o|]$ of 6.2% and R_w $[=(\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2)^{1/2}]$ of 3.9% for all reflections. The goodness of fit was 1.27 with $w = 1/\sigma^2(F)$. Maximum parameter shifts on the last cycle (some of the hydrogen positional parameters) were about half of their estimated errors. Refinement of the structure with the opposite polarity resulted in a higher value of $R_w = 4.0\%$, indicating the reported structure has the correct polarity. Scattering factor tables for Mn^{2+}, Cl^-, N, C, and H were taken from ref 3. Computer programs used are part of a local library.[4] Final parameters are listed in Table I and bond distances and angles are given in Table II.

Crystal Structure

This is a chemically most interesting structure since it contains Mn(II) ions in both tetrahedral and octahedral coordinate ion sites. In the hexagonal lattice, chains of face-sharing $MnCl_6$ octahedra run parallel to the c direction. Discrete $MnCl_4^{2-}$ ions and trimethylammonium ions are located between the chain, as shown in Figures 1 and 2. .

Although the entire structure is not centric, the chain itself is within experimental error of being so. (The Cl(3)–Mn–Cl(3) bond angle is 179.9 (5)° instead of 180°, as would be required if the Mn were at a center of inversion.) Thus, while the crystallographic symmetry of the chains is C_{3v}, for all practical purposes, it is D_{3h}. A comparison of the intrachain bond distances and bond angles for this compound in Table II with those for TMMC in Table III shows that the chain structures are quite similar. In TMMC, the $MnCl_6$ octahedron is distorted by a lengthening along the chain axis, as if the chain had been pulled. In $T_3Mn_2Cl_7$, this distortion is less, with the result that the Cl–Mn–Cl angles are closer to 90° and the intrachain Mn–Mn distance is shorter (3.208 Å compared to 3.2470 Å).

The $MnCl_4^{2-}$ anions sit on sites of C_{3v} symmetry also, with significant distortion from T_d symmetry. The bond distance to the chlorine atom on the threefold axis, Cl(1), is longer than the bond distances to three equivalent chlorine atoms, Cl(2), off the axis (2.402 (6) vs. 2.368 (3)Å). Each $(CH_3)_3NH^+$ cation sits on a mirror plane and forms a weak nonlinear hydrogen bond to the Cl atom of the $MnCl_4^{2-}$ which lies on the threefold axis. The Cl(1)–N distance is 3.304 Å, and the N–H–Cl(1) bond angle is 147°. The existence of three such hydrogen bonds to each Cl(1) atom, weak though they are, accounts for the lengthening observed for the Mn–Cl(1) bond. The nonlinearity is forced upon the system by the steric interaction between the methyl groups and the chlorine atoms in the chains. This steric interaction forces the cations to be ordered while the tetramethylammonium ions are disordered in TMMC. It is these steric factors which force the salt into the polar space group also.

Magnetic Suceptibility

The magnetic susceptibility of $T_3Mn_2Cl_7$, measured via a mutual inductance technique[5] over the temperature range 1.2–150 K, is shown in Figure 3. In addition, $\chi_m(T - \Theta)$ is plotted vs. T in Figure 4 to show how the system deviates from Curie–Weiss behavior. A Curie constant of 8.76 was used

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Table I

a. Positional and Thermal Parameters for the Nonhydrogen Atoms in $[(CH_3)_3NH]_3Mn_2Cl_7$[a,b]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mn(1)	0.0	0.0	0.0	0.003 92 (5)	0.003 92	0.007 92 (25)	0.001 96	0.0	0.0
Mn(2)	0.333 33	0.666 67	0.143 13 (45)	0.005 24 (8)	0.005 24	0.016 98 (48)	0.002 62	0.0	0.0
Cl(1)	0.333 33	0.666 67	0.517 97 (70)	0.005 11 (11)	0.005 11	0.015 66 (65)	0.002 55	0.0	0.0
Cl(2)	0.422 84 (7)	−0.422 84	0.027 16 (51)	0.006 62 (9)	0.006 62	0.028 09 (51)	0.003 94 (11)	0.001 04 (13)	−0.001 04
Cl(3)	0.079 58 (5)	−0.079 58	0.251 19 (46)	0.005 27 (7)	0.005 27	0.011 51 (24)	0.003 70 (8)	0.000 05 (10)	−0.000 05
N	0.791 70 (23)	−0.791 70	0.180 81 (98)	0.006 15 (31)	0.006 15	0.022 93 (156)	0.004 58 (37)	0.000 27 (31)	−0.000 27
C(1)	0.825 27 (33)	−0.825 27	0.374 10 (140)	0.008 81 (54)	0.008 81	0.024 86 (254)	0.005 91 (61)	−0.002 00 (50)	0.002 00
C(2)	0.401 49 (56)	0.114 53 (64)	0.056 10 (115)	0.006 59 (54)	0.008 06 (53)	0.032 81 (260)	0.002 76 (44)	−0.000 87 (81)	−0.004 85 (87)

b. Positional Parameters for Hydrogen Atoms in $[(CH_3)_3NH]_3Mn_2Cl_7$[a,c]

Atom	x	y	z	Atom	x	y	z
NH	0.758 (4)	−0.758	0.196 (8)	CH(2,1)	0.462 (6)	0.145 (5)	0.8885 (7)
CH(1,1)	0.854 (4)	−0.854	0.3034 (7)	CH(2,2)	0.403 (8)	0.066 (8)	0.1798 (8)
CH(1,2)	0.364 (5)	0.113 (5)	0.4426 (8)	CH(2,3)	0.335 (5)	0.079 (5)	1.0137 (3)

[a] Estimated standard deviation in the last digit is given in parentheses. [b] β_{ij} defined by $T = \exp(\beta_{11}h^2 + \ldots + 2\beta_{12}hk + \ldots)$. [c] Isotropic thermal parameters fixed at 4.0.

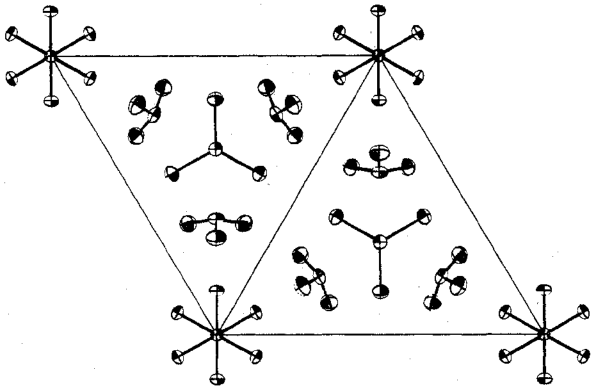

Figure 1. Structure of $[(CH_3)_3NH]_3Mn_2Cl_7$ as viewed from the c direction.

since there are two Mn(II) ions per mole of $T_3Mn_2Cl_7$ and the Weiss constant of −43 K was obtained from a least-squares fit of the data from 70 to 150 K to the Curie–Weiss law. The susceptibility below 2 K reaches a maximum and then decreases as the temperature decreases.

Since the structure of $T_3Mn_2Cl_7$ indicates that it can be treated as two sets of different magnetic species, the magnetic susceptibility was considered to be a combination of the susceptibilities from linear chains and discrete $MnCl_4^{2-}$ ions.

Table II. Bond Distances (Å) and Angles (deg) for $[(CH_3)_3NH]_3Mn_2Cl_7$[a]

Distances

Mn(1)–Cl(3)	2.559 (3)	C(1)–CH(1,2)	1.11 (9)
Mn(1)–Mn(1)	3.208 (3)	C(2)–CH(2,1)	1.32 (17)
Mn(2)–Cl(1)	2.405 (6)	C(2)–CH(2,2)	1.07 (14)
Mn(2)–Cl(2)	2.369 (3)	C(2)–CH(2,3)	0.88 (6)
N–C(1)	1.502 (10)	Cl(1)–N	3.308 (5)
N–C(2)	1.497 (10)	Cl(1)–NH	2.54 (6)
N–NH	0.88 (4)	Cl(2)–NH	3.09 (6)
C(1)–CH(1,1)	0.86 (7)	Cl(2)–NH	3.11 (7)

Angles

Cl(3)–Mn(1)–Cl(3)	85.20 (9)	Mn(1)–Cl(3)–Mn(1)	77.46 (8)
Cl(3)–Mn(1)–Cl(3)	95.00 (9)	NH–N–C(2)	100. (3)
Cl(3)–Mn(1)–Cl(3)	179.73 (10)	NH–N–C(1)	118. (5)
Cl(2)–Mn(2)–Cl(2)	110.62 (13)	C(2)–N–C(2)	113.3 (8)
Cl(2)–Mn(2)–Cl(1)	108.30 (10)	C(2)–N–C(1)	111.6 (3)

[a] Uncertainty in last digit given in parentheses.

On this basis, the magnetic susceptibility data of $T_3Mn_2Cl_7$ were fit to the expression

$$\chi_m = \frac{C}{T - \Theta} + \chi_F$$

In this expression, the molar susceptibility is assumed to be produced by a system of weakly interacting paramagnets and a system of noninteracting linear chains, where χ_F is the Fisher

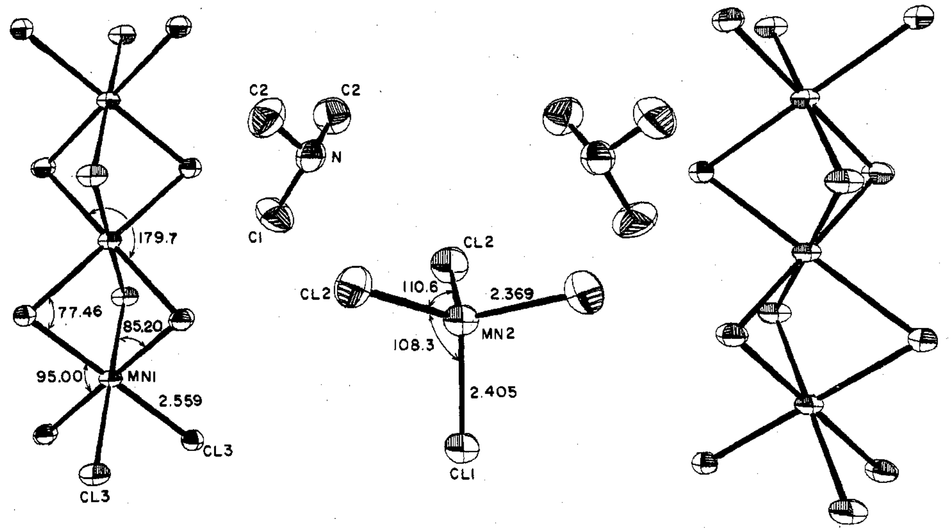

Figure 2. View of $[(CH_3)_3NH]_3Mn_2Cl_7$ along the 110 direction. Linear chains of face-sharing $MnCl_6$ octahedra are separated by $MnCl_4^{2-}$ and $(CH_3)_3NH^+$ ions.

Table III. Comparison of Chains in $(CH_3)_4NMnCl_3$ and $[(CH_3)_3NH]_3Mn_2Cl_7$

Compd	TMMC	$T_3Mn_2Cl_7$
Space group	$P6_3/m$	$P6_3mc$
a, Å	9.151 (1)	14.509 (19)
c, Å	6.494 (1)	6.415 (7)
Mn-Cl-Mn, deg	78.69	77.46
J/k, K	6.3	~8
Mn-Cl, Å	2.560	2.559 (3)
Mn-Mn, Å	3.247	3.208 (3)
Cl-Mn-Cl, deg	84.09	85.02 (9)

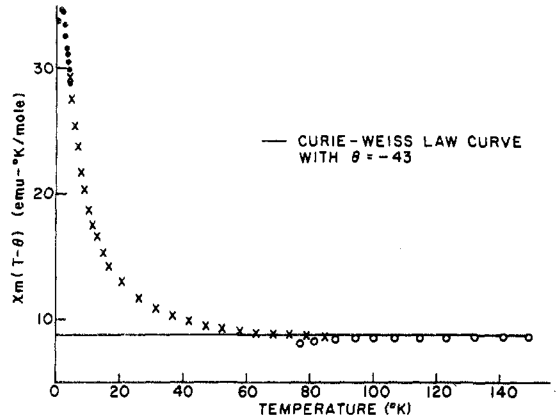

Figure 3. Magnetic susceptibility behavior of $[(CH_3)_3NH]_3Mn_2Cl_7$ compared to Curie-Weiss law behavior. Different symbols represent data taken during different runs.

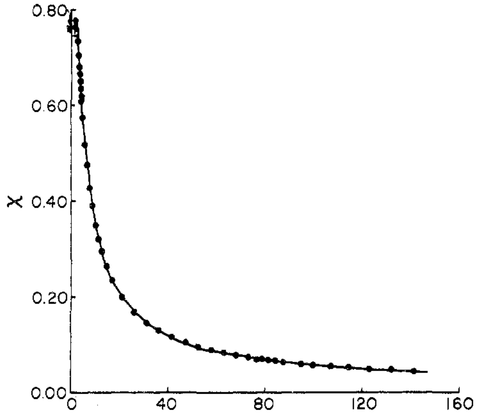

Figure 4. Magnetic susceptibility of $[(CH_3)_3NH]_3Mn_2Cl_7$ showing the fit obtained by assuming a system of two linear chains with $J/k = -8$ K and $J'/k = -0.196$ K.

molar susceptibility for these chains.[6] The Weiss constant, Θ, can be related to the coupling constant between magnetic species, J'/k, as in

$$\Theta = 2S(S+1)zJ'/3k$$

The least-squares fit to the data gives $J/k = -6$ K and $J'/k = -0.2$ K. The J/k value is very reasonable in view of the similar value for TMMC (6.3 K). It is clear that, to first order, it is legitimate to treat the magnetic system in $T_3Mn_2Cl_7$ as a set of TMMC-like linear chains and weakly coupled $MnCl_4^{2-}$ anions. Nevertheless, this mean field model for the coupling experienced by the $MnCl_4^{2-}$ anions is not completely satisfactory since it fails to account for the maximum in the susceptibility below 2 K.

As pointed out by McElearney in the accompanying paper, the behavior at low temperature can be successfully accounted for if one assumes that the coupling, J', experienced by the $MnCl_4^{2-}$ tetrahedra, is between adjacent ions along the *c* axis. In this model, the magnetic system consists of two independent sets of linear chains: the TMMC-like chains and the chains of tetrahedra. As seen in Figure 4, this model gives an excellent fit of the data over the complete temperature range with $J/k = -8$ (1) K and $J'/k = -0.196$ (3) K. This model is structurally valid, since the Cl-Cl contacts between adjacent tetrahedra along the *c* direction is 3.966 Å, while the closest contact between anions in adjacent chains of tetrahedra is 5.033 Å. It is also evident from Figure 1 that coupling between the two types of chains is likely to be negligible because of the intervening trimethylammonium ions. A list of observed and calculated susceptibility values is given in Table IV.

Table IV. Observed and Calculated Magnetic Susceptibilities for $[(CH_3)_3NH]_3Mn_2Cl_7$

	χ, emu/mol			χ, emu/mol	
T, K	Obsd	Calcd	*T*, K	Obsd	Calcd
3.97	0.6360	0.6369	31.44	0.1460	0.1455
3.78	0.6504	0.6507	36.57	0.1299	0.1290
3.56	0.6666	0.6670	41.83	0.1168	0.1160
3.36	0.6816	0.6817	47.51	0.1058	0.1051
3.06	0.7048	0.7042	52.66	0.0975	0.0972
2.64	0.7346	0.7339	58.15	0.0907	0.0916
2.17	0.7635	0.7618	63.25	0.0851	0.0846
1.87	0.7732	0.7732	68.40	0.0800	0.0798
1.61	0.7757	0.7765	73.60	0.0757	0.0756
1.21	0.7641	0.7643	78.90	0.0720	0.0718
4.18	0.6201	0.6221	84.50	0.0682	0.0661
4.89	0.5747	0.5746	77.06	0.0707	0.0753
5.85	0.5180	0.5188	81.60	0.0684	0.0700
6.79	0.4755	0.4726	88.00	0.0649	0.0662
7.94	0.4273	0.4257	94.60	0.0621	0.0628
8.99	0.3904	0.3900	99.90	0.0598	0.0602
10.37	0.3507	0.3514	106.6	0.0572	0.0574
11.60	0.3224	0.3229	114.4	0.0547	0.0544
12.95	0.2969	0.2967	123.2	0.0518	0.0514
14.73	0.2657	0.2681	132.0	0.0493	0.0488
17.23	0.2359	0.2369	141.2	0.0472	0.0463
21.04	0.2007	0.2014	149.2	0.0452	0.0444
26.39	0.1698	0.1676			

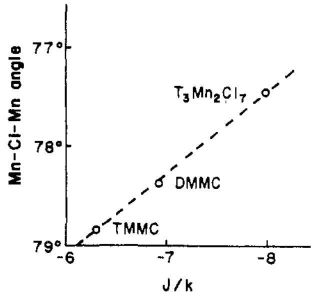

Figure 5. Plot of exchange coupling parameter vs. average Mn-Cl-Mn bond angle for TMMC-like salts.

Discussion

$T_3Mn_2Cl_7$ represents an intriguing magnetic system with a number of potentially fruitful investigations. Clay et al. have shown that the analogous Cu(II) salts[7] form mixed crystals with Co(II). With the two different coordinator sites, one can visualize that in $T_3MM'Cl_7$ salts, all of the octahedral sites will be occupied by one type of metal ion and all of the tetrahedral sites by the second type. Thus it would seem reasonable, for example, that in the $T_3MnZnCl_7$ salt, the Zn(II) ions should preferentially occupy the tetrahedral sites, making it possible to study solely the interaction in the TMMC-like chains. With the increased separation between chains, the system should behave like a true one-dimensional antiferromagnet at temperatures well below the ordering temperature of TMMC. Conversely in $T_3MnMgCl_7$ or $T_3MnCdCl_7$ salts, the Mn(II) ions should occupy the tetrahedral sites and thus the only exchange observed would be along the chains of tetrahedra.

Figure 5 shows a plot of J/k vs. the bridging Mn–Cl–Mn angle for TMMC-like chains in three salts: TMMC, DMMC [$\equiv(CH_3)_2NH_2MnCl_3$][8], and $T_3Mn_2Cl_7$. This shows exactly what is expected from superexchange arguments, the antiferromagnetic coupling getting stronger as the bridging angle deviates further from 90°. The apparent linear relation must be regarded as tenuous, due to the insensitivity of the $T_3Mn_2Cl_7$ data to changes in J/k; nevertheless the trend must be correct since McElearney's data indicate that J/k is closer to -11. Further studies such as those proposed above on a salt like $T_3MnZnCl_7$ would be useful in helping to establish quantitatively the variation of J with bridging angle in these salts.

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Registry No. $[(CH_3)_3NH]_3Mn_2Cl_7$, 58150-41-7.

Supplementary Material Available: Listing of structure factor amplitudes (5 pages). Ordering information is given on any current masthead page.

References and Notes

(1) Work sponsored by NSF Grant MPS-74-23207 and the Ames Laboratory of the U.S. Atomic Energy Commission.

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Two Independent Heisenberg Linear-Chain Species in Tris(trimethylammonium) *catena*-Tri-μ-chloro-manganate(II) Tetrachloromanganate(II), $[(CH_3)_3NH]_3Mn_2Cl_7$

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Magnetic susceptibility measurements on a single crystal of $[(CH_3)_3NH]_3Mn_2Cl_7$ parallel and perpendicular to the crystal's hexagonal c axis have been made between 1.5 and 30 K. The results show the presence of anisotropy over the entire temperature region. A quantitative fit to the parallel susceptibility has been achieved using a model in which the compound is considered to be composed of two different types of Heisenberg linear chains: one with intrachain exchange of -11 ± 6 K and one with intrachain exchange of -0.20 ± 0.01 K. The model also qualitatively explains the anisotropy observed.

Recently there has been increased interest in compounds which display pseudo-one-dimensional magnetic behavior.[1] Such materials are of interest since many of their properties can be described by relatively simple theories and thus they serve as important testing grounds for various aspects of the theories. In the course of an attempt to obtain a nonhydrated analog of the widely studied linear-chain series[2] $[(CH_3)_3NH]MX_3\cdot 2H_2O$ (M = transition metal; X = halide) single crystals of $[(CH_3)_3NH]_3Mn_2Cl_7$ were obtained. The stoichiometry, growth habit, and optical behavior of this material suggested that it probably was analogous to, if not isomorphous with, the corresponding Cu(II) compound, $[(CH_3)_3NH]_3Cu_2Cl_7$. The structure of that material has been reported[3] and is unusual in that it contains two types of copper anions: infinite $(CuCl_3)_n^{n-}$ chains and isolated $(CuCl_4)^{2-}$ tetrahedra. Since they are surrounded by trimethylammonium cations, the infinite $(CuCl_3)_n^{n-}$ chains are effectively magnetically insulated from each other as well as from the $(CuCl_4)^{2-}$ tetrahedra. Since one of the more widely studied one-dimensional materials,[4-7] $[(CH_3)_4N]MnCl_3$ (TMMC), contains similar infinite $(MnCl_3)_n^{n-}$ chains, studies of $[(CH_3)_3NH]_3Mn_2Cl_7$ are deemed important. Thus, single-crystal magnetic susceptibility measurements from 1.5 to 30 K were undertaken on this material. Upon completion of this work it was learned that similar powder susceptibility measurements, as well as a complete structure determination, had been performed elsewhere.[8] The structural results indicate that, indeed, $[(CH_3)_3NH]_3Mn_2Cl_7$ is essentially isostructural with its copper analogue (although the two materials are not strictly isomorphic). Although the single-crystal susceptibility results presented here are not in contradiction to the powder results, a novel interpretation is given to them.

Experimental Section

Single crystals of $[(CH_3)_3NH]_3Mn_2Cl_7$ were obtained by slow evaporation of a methanolic solution of equimolar amounts of $(CH_3)_3N\cdot HCl$ and $MnCl_2$. The red-orange crystals grow as long thin needles, becoming thick only after long periods of time. The crystal used in the present study was grown over a period of 1 year and weighed 0.1 g. Anal. Calcd for $[(CH_3)_3NH]_3Mn_2Cl_7$: C, 20.08; H, 5.62; N, 7.80; Mn, 20.41; Cl, 46.09. Found (commercially): C, 20.06; H, 6.04; N, 7.77; Mn, 20.08; Cl, 45.62. The agreement is excellent.

Isothermal magnetic susceptibilities parallel and perpendicular to the crystallographic c axis were measured using apparatus that has been described elsewhere.[9] The most important point about the method is that it involves a low-frequency mutual-inductance technique applied in near-zero field (less than 5 Oe). The calibration of the system is considered to be accurate within 2–3%. Temperatures were measured with a commerically calibrated germanium resistance thermometer and generally were stable to within 0.02 K for each data point measured.

Results and Analysis

The results are shown in Figure 1. Three important features are to be noted: (1) the relatively reduced magnitude of the susceptibility, (2) the rounded maximum near 2 K observed in both data sets, (3) the anisotropy between the