discussions with Harry Gray. This work was supported, in part, by the National Science Foundation, the National Institutes of Health, the International Copper Research Association, and the Biomedical Research Support Program (Grant No. RR07003).

Registry No. **1**, 61128-83-4; **2**, 61114-07-6; **11**, 67814-42-0; $Cu(LBF_2)(ClO_4)$, 64783-10-4; $Zn(LBF_2)(ClO_4)$, 67761-33-5.

Supplementary Material Available: A listing of structure factor amplitudes (11 pages). Ordering information is given on any current masthead page.

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Preparation, Properties, and Crystal Structure of Dichloro(1,3-dibenzoyl-2-azapropenato)(ethanol)iron(III)

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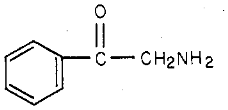
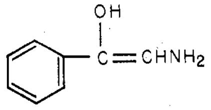
The compound dichloro(1,3-dibenzoyl-2-azapropenato)(ethanol)iron(III), $Fe(C_{16}H_{12}NO_2)Cl_2(C_2H_5OH)$, has been prepared, and its structure has been determined crystallographically. The compound was prepared by the reaction of iron(II) chloride with α-aminoacetophenone in the presence of air. The structure consists of octahedral iron(III) coordinated by two chlorides, an ethanol, and a tridentate ligand derived from two molecules of α-aminoacetophenone by oxidation and condensation. The two five-membered rings of the ligand are virtually identical. The compound crystallizes in monoclinic space group $C2/c$ with eight formula units in a unit cell of dimensions $a = 29.606$ (8) Å, $b = 6.689$ (1) Å, $c = 20.627$ (6) Å, $\beta = 114.70$ (2)°, $\rho_{calcd} = 1.51$ g/cm^3, and $\rho_{obsd} = 1.51$ g/cm^3. The structure was solved by Patterson methods, and least-squares refinement converged at final values of $R = 0.066$ and $R_w = 0.045$ for 1729 reflections with $I \geq 2\sigma(I)$.

Introduction

As part of a continuing investigation of transition-metal complexes of amino alcohols and related compounds, we became interested in the possibility of utilizing α-aminoacetophenone, I, as a ligand. Although it is not an amino alcohol, there is the possibility of an enol form, II. The reaction of α-aminoacetophenone hydrochloride with most metal chlorides produced only salts of the tetrachlorometalate ions, and attempts to neutralize the hydrochloride to carry out reactions of the amine led to organic condensation products. The reaction of iron(II) chloride with the hydrochloride under nitrogen gave the tetrachloroferrate(II) ion, but the same reaction in air produced an iron(III) complex with an unusual ligand; the ligand is an anion formed from α-aminoacetophenone by condensation and oxidation. In this paper we report the preparation, properties, and crystal structure of this



I



II

0020-1669/78/1317-3571$01.00/0

iron(III) compound.

Experimental Section

Preparation. Iron(II) chloride tetrahydrate (1.99 g, 10 mmol) was dissolved in approximately 50 mL of dry and deaerated ethanol, and solid α-aminoacetophenone hydrochloride (3.43 g, 20 mmol) was added to the solution under a nitrogen atmosphere. The orange solution was refluxed for 20 min; pale yellow crystals separated on cooling. Anal. Calcd for $(C_8H_{10}NO)_2FeCl_4\cdot 2H_2O$: C, 38.14; H, 4.80; N, 5.62; Cl, 28.09. Found: C, 37.98; H, 4.78; N, 5.54; Cl, 28.02.

The orange solution was exposed to air to give a red solution. After several hours, green hexagonal platelets formed and were filtered. Anal. Calcd for $FeC_{18}H_{18}NO_3Cl_2$: C, 51.10; H, 4.29; N, 3.31; Cl, 16.76; Fe, 13.20. Found: C, 51.34; H, 4.17; N, 3.28; Cl, 16.83; Fe, 13.55.

Magnetic Susceptibilities. The magnetic susceptibilities of the compounds were determined by the Faraday method using $HgCo(NCS)_4$ as calibrant. Diamagnetic corrections were applied using tabulated values.[1] The experimental moments per gram-atom of iron for the yellow compound and the green compound are 5.50 and 5.71 μ_B, respectively.

Collection of X-ray Diffraction Data. A dark green, hexagonal-shaped platelet with approximate dimensions 0.1 × 0.4 × 0.4 mm was mounted on a glass fiber using epoxy cement such that one of the longer crystal dimensions (b^*) was approximately parallel to the fiber axis.

Unit cell parameters and the orientation matrix were determined on a Syntex $P2_1$ four-circle diffractometer equipped with a graphite monochromator (Bragg 2θ angle = 12.2°) using Mo Kα radiation at a takeoff angle of 6.75°. Fifteen reflections whose 2θ values ranged from 4.4 to 19.9° were machine-centered and used in least-squares refinement of the lattice parameters and the orientation matrix. Unit cell parameters obtained are[2] $a = 29.606$ (8) Å, $b = 6.689$ (1) Å, $c = 20.627$ (6) Å, $\beta = 114.70$ (2)°, and $V = 3711$ (2) Å^3. The calculated density of 1.51 g/cm^3 for eight formula units per unit cell agrees with the experimental density of 1.51 g/cm^3 measured by the flotation method using a mixture of carbon tetrachloride and hexane. ω scans of several low 2θ angle reflections gave peak widths at half-height of less than 0.25°, indicating a satisfactory mosaic spread for the crystal.

Axial photographs indicated that the crystal belonged to the monoclinic system. Intensity data for zero and upper levels were collected at a rapid scan rate, and the intensities were examined for systematic absences. The absence of $l = 2n + 1$ reflections for $h0l$ and $h + k = 2n + 1$ reflections for hkl is consistent with space group Cc (No. 9[3]) or $C2/c$ (No. 15). Space group $C2/c$ was assumed, and the successful refinement of the structure has verified this assumption.

Intensity data were collected using θ–2θ scans with X-ray source and monochromator settings identical with those used for determination of the unit cell parameters. A variable scan rate from 7.3 to 29.3°/min was used, and a scan width of 2° was sufficient to collect all of the peak intensity. Stationary-background counts were measured at the beginning (bgd1) and at the end (bgd2) of each scan with a total background to scan time ratio of 1.0. No significant fluctuations were observed in the intensity of a standard reflection ($\bar{2}\bar{2}0$) monitored every 50 reflections. Intensities were calculated from the total scan (CT) and background counts by the relationship

$$I = \mathrm{CT} - (\mathrm{bgd1} + \mathrm{bgd2})$$

The intensities were assigned standard deviations according to the formula

$$\sigma(I) = [\mathrm{CT} + (\mathrm{bgd1} + \mathrm{bgd2})]^{1/2}$$

From a total of 2813 reflections collected in a complete quadrant (k, $l \geq 0$) of data out to $2\theta = 50°$, 1729 were accepted as statistically above background on the basis that $I \geq 2\sigma(I)$. Lorentz and polarization corrections were made in the usual way. Absorption corrections were not applied.

Solution and Refinement of the Structure. Computations were performed using standard programs;[4] all computations were carried out on the CDC Cyber 74 system. For structure factor calculations the scattering factors were taken from Cromer and Waber's tabulation[5] for all atoms except hydrogen; Stewart's hydrogen atom scattering factors[6] were used. The scattering factors for iron and chlorine were corrected for the real and imaginary anomalous dispersion components using the dispersion factors given by Cromer.[7] The agreement factors are defined in the usual way as

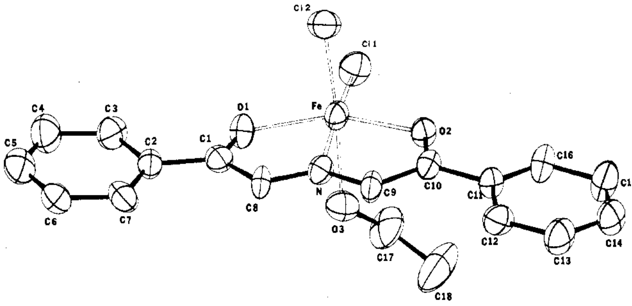


Figure 1. Molecular structure and atom labeling of $FeLCl_2(EtOH)$.

$$R = (\sum||F_o| - |F_c||)/\sum|F_o|$$

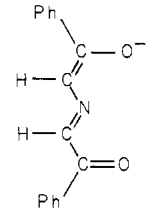
$$R_w = [\sum w(|F_o| - |F_c|)^2/\sum w(|F_o|)^2]^{1/2}$$

In all least-squares refinements, the quantity minimized was $w(|F_o| - |F_c|)^2$. A weighting scheme based on counting statistics ($w = 4I/\sigma^2(I)$) was employed in the calculation of R_w and in least-squares refinement.

Atomic coordinates for the iron atoms were obtained from a three-dimensional Patterson synthesis, and the remaining nonhydrogen atoms were located by mean of successive Fourier syntheses. A least-squares refinement using isotropic temperature factors for all nonhydrogen atoms converged with $R = 0.098$ and $R_w = 0.082$. Anisotropic thermal parameters were introduced, and further refinement gave $R = 0.073$ and $R_w = 0.053$. The 18 hydrogen atoms were located as the principal features on a difference electron density map. Subsequent refinements, varying coordinates of hydrogen atoms but maintaining hydrogen thermal parameters at 3.0, converged to give $R = 0.066$ and $R_w = 0.045$ (225 variables). In the final cycle of refinement, the maximum parameter shift was 0.8σ (B_{22} for C17). The major feature on a final difference Fourier map was a peak of 1.0 e/Å^3 approximately 1.1 Å from iron; this compares to values of approximately 4.0 for typical carbon atoms in this structure. Final positional and thermal parameters are given in Table I. A list of calculated and observed structure factors is available.[8]

Discussion

The analysis of the green compound corresponds to $FeLCl_2(EtOH)$ where L represents the anion III. The formula and the magnetic moment, 5.71 μ_B, indicate an iron(III) complex.



III

The structure, Figure 1, consists of an iron atom with distorted octahedral coordination. The two oxygen atoms and the nitrogen atom of the tridentate ligand occupy three coplanar coordination sites, the ethanol oxygen occupies a position cis to the nitrogen, and the two chlorine atoms occupy the remaining two corners of the octahedron. The O–Fe–N angles within the five-membered chelate rings, 76.0 (2) and 75.0 (2)°, are considerably smaller than the 90° of a regular octahedron but are similar to the angle in five-membered rings of other iron(III) structures.[9-11]

The two Fe–Cl distances, 2.216 (2) and 2.352 (4) Å, are not equivalent, and the chlorine in the plane of the ligand, Cl1, shows the shorter distance. The alcohol oxygen, O3, is weakly coordinated with an Fe–O distance, 2.157 (7) Å, that is significantly longer than the Fe–O distances for ligand oxygens, 2.021 (4) and 2.037 (5) Å. The coordination, thus, corresponds to a tetragonally distorted octahedron with the axial ligands,

Table I. Final Atomic Parameters for $FeLCl_2(EtOH)$[a]

atom	*x*	*y*	*z*	B_{11} or *B* ($Å^2$)	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Fe	0.12822 (4)	±0.20274 (17)	−0.00540 (5)	3.03 (6)	3.48 (5)	1.87 (4)	0.29 (5)	0.97 (4)	0.00 (2)
Cl(1)	0.0652 (1)	0.2797 (3)	0.0212 (1)	3.5 (3)	5.3 (1)	3.4 (1)	0.22 (7)	1.8 (2)	−0.20 (2)
Cl(2)	0.1679 (1)	0.5159 (3)	0.0215 (1)	4.3 (3)	3.15 (9)	3.9 (1)	0.07 (7)	1.8 (2)	−0.09 (2)
O(1)	0.1002 (2)	0.2499 (6)	−0.1118 (2)	2.9 (3)	3.3 (3)	2.5 (1)	0.8 (2)	1.4 (2)	0.04 (5)
O(2)	0.1807 (2)	0.0824 (8)	0.0856 (2)	2.9 (3)	4.6 (3)	2.4 (1)	0.1 (2)	1.2 (2)	0.21 (7)
O(3)	0.1005 (2)	−0.0987 (8)	−0.0308 (3)	4.31 (3)	2.6 (2)	5.1 (3)	0.1 (2)	1.2 (2)	−0.05 (9)
N	0.1851 (2)	0.0962 (8)	−0.0348 (3)	3.2 (3)	3.2 (3)	2.0 (3)	−0.1 (3)	1.2 (2)	0.09 (7)
C(1)	0.1267 (3)	0.1980 (12)	−0.1441 (3)	2.9 (3)	3.0 (3)	2.4 (3)	−0.5 (4)	0.8 (2)	0.04 (10)
C(2)	0.1068 (2)	0.2156 (11)	−0.2222 (3)	2.3 (3)	4.2 (3)	1.9 (3)	0.4 (4)	1.2 (2)	−0.04 (10)
C(3)	0.0592 (3)	0.2799 (12)	−0.2583 (4)	3.6 (2)					
C(4)	0.0370 (3)	0.2946 (13)	−0.3328 (4)	4.5 (2)					
C(5)	0.0643 (3)	0.2405 (11)	−0.3698 (4)	3.7 (2)					
C(6)	0.1108 (3)	0.1712 (13)	−0.3351 (4)	4.5 (2)					
C(7)	0.1334 (3)	0.1548 (11)	−0.2605 (4)	3.5 (2)					
C(8)	0.1751 (3)	0.1158 (11)	−0.1026 (4)	2.3 (6)	3.7 (4)	2.9 (4)	0.4 (4)	1.4 (2)	0.05 (10)
C(9)	0.2250 (3)	0.0259 (12)	0.0177 (3)	2.9 (3)	4.2 (4)	2.0 (3)	0.4 (4)	0.8 (2)	0.02 (10)
C(10)	0.2215 (3)	0.0222 (11)	0.0840 (3)	3.2 (6)	2.8 (4)	2.5 (3)	−0.6 (4)	1.2 (2)	0.00 (9)
C(11)	0.2624 (3)	−0.0435 (10)	0.1511 (3)	2.7 (1)					
C(12)	0.3055 (3)	−0.1182 (11)	0.1519 (4)	3.6 (2)					
C(13)	0.3456 (3)	−0.1704 (13)	0.2176 (4)	4.7 (2)					
C(14)	0.3401 (3)	−0.1418 (12)	0.2784 (5)	4.3 (2)					
C(15)	0.2984 (4)	−0.0704 (13)	0.2797 (5)	4.7 (2)					
C(16)	0.2577 (3)	−0.0196 (11)	0.2147 (3)	3.9 (2)					
C(17)	0.0504 (4)	−0.1708 (16)	−0.0704 (5)	4.9 (6)	4.1 (5)	5.5 (6)	−0.4 (4)	0.8 (4)	−0.3 (1)
C(18)	0.0404 (6)	−0.2229 (20)	−0.1442 (7)	10 (1)	7.1 (8)	5.6 (6)	−0.5 (8)	0.8 (6)	−0.6 (3)

atom	*x*	*y*	*z*	atom	*x*	*y*	*z*
HC(3)	0.040 (2)	0.339 (9)	−0.239 (3)	HC(14)	0.370 (2)	−0.170 (9)	0.324 (3)
HC(4)	−0.003 (3)	0.326 (9)	−0.350 (3)	HC(15)	0.283 (2)	−0.031 (9)	0.305 (3)
HC(5)	0.042 (2)	0.242 (8)	−0.432 (3)	HC(16)	0.224 (3)	0.042 (9)	0.217 (3)
HC(6)	0.134 (3)	0.133 (9)	−0.345 (3)	HAC(17)	0.050 (2)	−0.299 (11)	−0.047 (3)
HC(7)	0.168 (3)	0.125 (9)	−0.235 (3)	HBC(17)	0.032 (3)	−0.077 (11)	−0.061 (3)
HC(8)	0.196 (3)	0.082 (10)	−0.118 (4)	HAC(18)	0.003 (3)	−0.248 (10)	−0.171 (3)
HC(9)	0.252 (3)	−0.042 (10)	0.006 (3)	HBC(18)	0.033 (3)	−0.114 (12)	−0.153 (4)
HC(12)	0.314 (2)	−0.141 (9)	0.117 (3)	HCC(18)	0.057 (3)	−0.327 (11)	−0.145 (4)
HC(13)	0.369 (2)	−0.198 (10)	0.207 (3)	HO(3)	0.115 (2)	−0.218 (10)	−0.024 (3)

[a] For all hydrogen atoms $B = 3.0$ $Å^2$.

Table II. Interatomic Distances (Å) and Angles (deg) in $FeLCl_2(EtOH)$

Distances

Fe–Cl(1)	2.216 (2)	Fe–O(1)	2.021 (4)
Fe–Cl(2)	2.352 (4)	Fe–O(2)	2.037 (5)
Fe–N	2.136 (6)	Fe–O(3)	2.157 (7)
O(1)–C(1)	1.270 (8)	O(2)–C(10)	1.286 (8)
C(1)–C(8)	1.437 (10)	C(10)–C(9)	1.415 (10)
C(8)–N	1.309 (9)	C(9)–N	1.312 (8)
C(1)–C(2)	1.473 (9)	C(10)–C(11)	1.474 (9)
C(2)–C(3)	1.36 (1)	C(11)–C(12)	1.36 (1)
C(3)–C(4)	1.40 (1)	C(12)–C(13)	1.42 (1)
C(4)–C(5)	1.37 (1)	C(13)–C(14)	1.34 (1)
C(5)–C(6)	1.34 (1)	C(14)–C(15)	1.33 (1)
C(6)–C(7)	1.40 (1)	C(15)–C(16)	1.42 (1)
C(7)–C(2)	1.39 (1)	C(16)–C(11)	1.39 (1)

Angles

Cl(1)–Fe–Cl(2)	97.1 (1)	Cl(2)–Fe–N	89.3 (2)
Cl(1)–Fe–O(1)	103.1 (2)	O(1)–Fe–O(3)	86.6 (2)
Cl(1)–Fe–O(2)	105.1 (2)	O(1)–Fe–N	76.0 (2)
Cl(1)–Fe–O(3)	90.2 (2)	O(2)–Fe–O(3)	85.4 (2)
Cl(2)–Fe–O(1)	93.2 (2)	O(2)–Fe–N	75.0 (2)
Cl(2)–Fe–O(2)	91.2 (2)	O(3)–Fe–N	83.3 (2)
Fe–O(1)–C(1)	117.6 (4)	Fe–O(2)–C(10)	117.6 (4)
O(1)–C(1)–C(2)	120.1 (6)	O(2)–C(10)–C(11)	118.8 (6)
O(1)–C(1)–C(8)	118.2 (6)	O(2)–C(10)–C(9)	118.0 (7)
C(2)–C(1)–C(8)	121.7 (8)	C(9)–C(10)–C(11)	123.2 (8)
C(1)–C(8)–N	113.5 (7)	C(10)–C(9)–N	113.5 (8)
C(8)–N–Fe	114.6 (5)	C(9)–N–Fe	115.5 (5)
C(8)–N–C(9)	129.9 (7)	Fe–O(3)–C(17)	130.2 (5)
C(2)–C(3)–C(4)	121.8 (8)	C(11)–C(12)–C(13)	120.4 (8)
C(3)–C(4)–C(5)	118.4 (8)	C(12)–C(13)–C(14)	118.4 (9)
C(4)–C(5)–C(6)	120.5 (8)	C(13)–C(14)–C(15)	123.0 (9)
C(5)–C(6)–C(7)	121.7 (9)	C(14)–C(15)–C(16)	119.5 (9)
C(6)–C(7)–C(2)	118.7 (8)	C(15)–C(16)–C(11)	119.1 (9)
C(7)–C(2)–C(3)	119.1 (6)	C(16)–C(11)–C(12)	119.5 (7)

Table III. Equations of Least-Squares Planes[a] and Distances of Atoms from the Planes

Equations of Planes

plane a: coordinating rings (O(1), O(2), N, Cl, C(8), C(9), C(10))

$$0.343x + 0.919y + 0.195z = 2.434$$

plane b: ligand, L

$$0.371x + 0.920y + 0.130z = 2.584$$

Deviations of Atoms from Planes (Å)

atom	plane a	plane b	atom	plane a	plane b
Fe	0.110	0.075	C(6)	−0.494	−0.057
Cl(1)	−0.038	−0.164	C(7)	−0.313	0.033
Cl(2)	2.456	2.417	C(8)	−0.017	0.130
O(1)	0.040	0.139	C(9)	0.022	0.033
O(2)	−0.033	−0.159	C(10)	0.010	−0.080
O(3)	−2.042	−2.064	C(11)	0.069	−0.086
N	0.012	0.067	C(12)	0.048	−0.073
C(1)	−0.033	0.136	C(13)	0.181	−0.004
C(2)	−0.182	0.086	C(14)	0.343	0.065
C(3)	−0.295	−0.014	C(15)	0.360	0.045
C(4)	−0.483	−0.110	C(16)	0.213	−0.039
C(5)	−0.564	−0.115			

[a] Direction cosines of the plane refer to the orthogonal axis system *a, b, c**.

Cl2 and O3, more weakly bound.

The two five-membered rings are virtually identical with Fe–O distances of 2.021 (4) and 2.037 (5) Å, C–O distances of 1.270 (8) and 1.286 (8) Å, C–C distances of 1.437 (10) and 1.445 (10) Å, and C–N distances of 1.309 (9) and 1.312 (8) Å. The least-squares plane calculated for the ligand atoms comprising the two five-membered rings, Table III, shows these atoms to be coplanar, and the iron is 0.110 Å out of this plane. The two phenyl substituents are on opposite sides of this plane with the C_{11}–C_{16} ring on the same side as the iron atom. The negative charge and the double bonds do not appear to be localized as in III; there is instead extensive delocalization of the ligand π system. This delocalization is not typical of iron(III)–imine complexes as there are usually[9-13] distinct carbon–nitrogen single bonds and carbon–nitrogen double bonds. This type of delocalization is postulated for the intermediate in transamination and other reactions of metal complexes of the imines of pyridoxal and α-amino acids.[14] The delocalization in the iron compound may be enhanced by the phenyl substituents since the phenyl rings are aligned with the chelate rings.

The phenyl groups have the expected parameters. The carbon–carbon bond distances vary from 1.33 to 1.42 Å with an average of 1.38 (3) Å; the angles vary from 118 to 123° with an average of 120 (1)°.

The mechanism of formation of the tridentate ligand is of considerable interest. The dependence on metal ion and molecular oxygen suggests an oxidative deamination process, as observed with vitamin B_6 complexes[14] followed by condensation with a second molecule of α-aminoacetophenone.

In the absence of a metal ion, the α-aminoacetophenone hydrochloride seems stable in solution. However upon neutralization of the hydrochloride salt self-condensation takes place to yield a cyclic dimer, $C_{16}H_{14}N_2$. With iron(II) chloride in the absence of oxygen, the α-aminoacetophenone hydrochloride reacts to form a salt, $(C_8H_{10}NO)_2FeCl_4$. An orange solution of this salt in ethanol upon exposure to air gives a red solution from which green crystals of the iron(III) compound separate. With an excess of α-aminoacetophenone hydrochloride in the absence of molecular oxygen, the color of the solution changes to yellow and the iron is apparently reduced. Addition of oxygen restores the red color of the iron(III) compound.

Registry No. $FeLCl_2(EtOH)$, 68070-71-3; I·HCl, 5468-37-1; $(C_8H_{10}NO)_2FeCl_4$, 68036-76-0.

Supplementary Material Available: Table of observed and calculated structure factors for $FeLCl_2(EtOH)$ (9 pages). Ordering information is given on any current masthead page.

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Structural Chemistry of Molybdenum Complexes of Cyclic Polythia Ethers. Crystal and Molecular Structure of (Hydrosulfido)(oxo)(1,5,9,13-tetrathiacyclohexadecane)molybdenum(IV) Trifluoromethanesulfonate

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The crystal and molecular structures of (hydrosulfido)(oxo)(1,5,9,13- tetrathiacyclohexadecane)molybdenum(IV) trifluoromethanesulfonate are reported. The complex crystallizes in the monoclinic space group $P2_1/c$ with four molecules per unit cell of dimensions $a = 7.494$ (3) Å, $b = 20.500$ (6) Å, $c = 14.902$ (5) Å, $\beta = 101.09$ (3)°, and $V = 2248$ (1) Å^3. Full-matrix least-squares refinement gave final discrepancy factors of $R_1 = 0.035$ and $R_2 = 0.047$ for 2509 data for which $F^2 > 2.5\sigma(F^2)$. The macrocyclic ring is coordinated to molybdenum in the conventional planar tetradentate manner, with oxo and hydrosulfido groups occupying trans positions along the symmetry axis of the monomeric cation. A comparison of bonding parameters in the title complex with those in the sulfur-bridged dimeric precursor complex is given.

Introduction

In our investigation of molybdenum complexes of cyclic polythia ethers, we have studied in detail the reaction of the binuclear quadruply bonded dimolybdenum trifluoromethanesulfonate $[Mo^{II}_2(H_2O)_4(CF_3SO_3)_2](CF_3SO_3)_2$ with the 16-membered macrocyclic polythia ether 1,5,9,13-tetrathiacyclohexadecane.[1] The reaction yields several products, three of which have been structurally characterized. This is the last of three reports on those complexes.[1,2]

Previous structural work on complexes of cyclic polythia ethers has revealed interesting features, dictated in part by the constraints imposed by the macrocycle. These have included the exo or "inside out" conformation found in the complex $(NbCl_5)_2(14\text{-ane}[S_4])$[3,4] and the unique bridging nature of tetradentate 16-ane$[S_4]$ in the complex $[Mo_2(SH)_2(16\text{-ane}[S_4])](CF_3SO_3)_2{\cdot}2H_2O$ from which the title compound is derived.[1] This is the first mononuclear molybdenum complex of a cyclic polythia ether for which the structure has been determined. The most notable feature is the terminal SH group in the lower axial coordination site. Bridging SH groups or SR groups are not uncommon in Mo chemistry, but terminal sulfur ligands are usually the doubly bonded sulfide.

Experimental Section

The synthesis of the title compound has been reported previously.[1] Crystals were isolated from the reaction vessel with only a wash of warm hexane to remove surface organic contaminants. The crystals were found to be air stable but were nonetheless sealed in thin-walled capillaries as a precaution.

A suitable crystal measuring approximately 0.38 × 0.19 × 0.11 mm^3 was mounted on a Syntex $P2_1$ four-circle diffractometer. Oscillation photographs, together with a small set of counter data,

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