Binding of Thioether Sulfur to Trivalent Chromium. A Family of $CrS_2N_2O_2$ Complexes Derived from Acyclic Ligands

Sujay Pattanayak, Diganta Kumar Das, Partha Chakraborty, and Animesh Chakravorty*

Department of Inorganic Chemistry, Indian Association for the Cultivation of Science, Calcutta 700 032, India

Received May 5, 1995

Introduction

We are interested in thioether binding of 3d-ions.[1–5] The thioether group is a poor ligand, and stable binding is best achieved with the help of macrocyclic ligands.[6,7] The problem of thioether coordination is especially acute for the hard trivalent ions, and very few authentic complexes of manganese(III)[2,8] and iron(III)[4,9] have been described. The chromium(III) situation is even worse,[10] and only very recently were two complexes, both macrocyclic, structurally characterized.[11,12]

Herein we describe a family of stable chromium complexes of coordination type $Cr^{III}S_2N_2O_2$ derived from an acyclic hexadentate ligand. A representative X-ray structure is presented along with spectra and the electrochemistry of the family.

Results and Discussion

Synthesis. The ligand system used is **1**, abbreviated H_2RxL. It was chosen because of its proven affinity for manganese(III)[2] and iron(III).[4] When stoichiometric quantities of H_2RxL and chromium(III) chloride tetrahydrate were heated in a dichloromethane–methanol mixture, the solution color changed from light green to deep brown, and upon addition of sodium perchlorate the dark-colored complex $[Cr(RxL)]ClO_4$ separated in very good yield. In acetonitrile solution the complexes behave as 1:1 electrolytes ($\Lambda = 115–125\ \Omega^{-1}\ cm^2\ M^{-1}$).

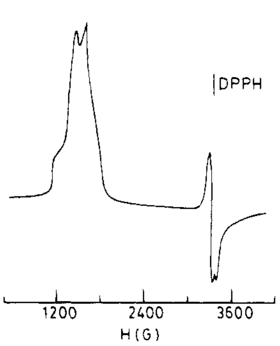
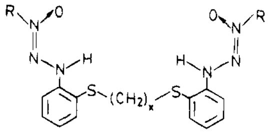


Figure 1. (a) EPR spectrum at X-band of $[Cr(Me3L)]ClO_4$ in dichloromethane–toluene (1:1) at 77 K. Instrument settings: power, 30 dB; modulation, 100 kHz; sweep center, 3000 G; sweep width, 8000 G; sweep time, 240 s, microwave frequency, 9.10 GHz.



H_2RxL

R=Me, Ph ; x = 2,3

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Magnetism, Spectra, and Electrochemistry. Selected characterization data are listed in Table 1. The magnetic moment of the complexes corresponds to the $S = {}^3/_2$ ground state. Their EPR spectra were examined in frozen (77 K) 1:1 dichloromethane–toluene solution. Two main resonance domains are $g = 5.6–3.5$ and $g \sim 2$, the former being more intense, corresponding to strong rhombic distortion.[13] The representative case of $[Cr(Me3L)]ClO_4$ is displayed in Figure 1.

Several electronic bands are observed in the form of shoulders in the visible region. The bands at ~850 and ~650 nm are assigned to an octahedral ν_1 transition split by the low symmetry of ligand field.[14] The average D_q value is computed to be 1360 cm^{-1}. A similar splitting has been recorded in the case of $Ni^{II}(RxL)$,[3d] for which the average D_q value is 860 cm^{-1}.

All the complexes exhibit a one-electron quasireversible cyclic voltammogram near −0.1 V versus SCE, corresponding to the metal redox potential. The $E_{1/2}$ values of the $M^{III}(RxL)^+–M^{II}(RxL)$ couple for M = Mn[2] and M = Fe[4] (both oxidation states high-spin) are known, and we can now observe the $E_{1/2}$ order Mn > Fe > Cr. Interestingly, there is a correlation between the $E_{1/2}$ values of the $M^{III}(RxL)^+–M^{II}(RxL)$ and $M^{III}(H_2O)_6{}^{3+}–M^{II}(H_2O)_6{}^{2+}$ couples.[15,16]

Structure. The structure of $[Cr(Me3L)]ClO_4\cdot{}^1/_2CH_2Cl_2$ has been determined. A view of the cations is shown in Figure 2. The asymmetric unit contains two independent but metrically similar molecules which differ in their configurations. The two thioether sites in a given molecule have the same chiral configuration, which is enantiomeric to that in the other molecule. The configurations of the chromium sites are also mutually enantiomeric. The asymmetric unit is thus constituted

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* Author to whom correspondence should be addressed. Fax: +91-33-473-2805. E-mail: icac@iacs.ernet.in.

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Table 1. Characterization Data

compound	UV−vis data[a] λ_{max}, nm (ϵ, M^{-1} cm^{-1})	$E_{1/2}$,[a−c] V (ΔE_p,[d] mV)	μ_{eff},[e] μ_B	EPR g[f] values
[Cr(Me3L)]ClO$_4$	840[g] (64), 650[g] (181), 430[g] (640), 375[g] (1200)	−0.12 (120)	3.93	5.54,[h] 4.61,[i] 4.26,[i] 1.99,[i] 1.97,[h] 1.95[h]
[Cr(Me2L)]ClO$_4$	830[g] (87), 660[g] (125), 450[g] (565), 375[g] (1636)	−0.15 (100)	3.91	4.27,[h] 3.81,[i] 3.56,[i] 2.09,[h] 2.06,[i] 1.99[i]
[Cr(Ph2L)]ClO$_4$	850[g] (107), 650[g] (267), 425[g] (857), 360[g] (1857)	−0.07 (140)	3.95	5.60,[h] 4.72,[i] 4.29,[h] 2.05,[i] 2.01[h]

[a] In acetonitrile at 298 K. [b] At a platinum electrode; the supporting electrolyte is tetraethylammonium perchlorate (TEAP, 0.1 M); scan rate, 50 mV s^{-1}; reference electrode, SCE; solute concentration, $\sim 10^{-3}$ M. [c] $E_{1/2}$ is calculated as the average of the anodic (E_{pa}) and cathodic (E_{pc}) peak potentials. [d] $\Delta E_p = E_{pa} - E_{pc}$. [e] In the solid state at 298 K. [f] Dichloromethane−toluene (1:1) at 77 K. [g] Shoulder. [h] Weak resonance. [i] Strong resonance.

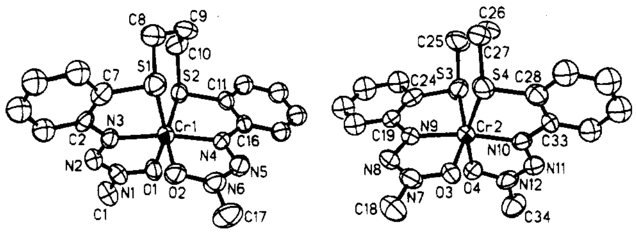

Figure 2. ORTEP plot and labeling scheme for $[Cr(Me3L)]^+$ in [Cr(Me3L)]ClO$_4$·$^1/_2$CH$_2$Cl$_2$ with all atoms represented by their 50% probability ellipsoids.

Table 2. Selected Bond Distances (Å) and Angles (deg) and Their Estimated Standard Deviations for [Cr(Me3L)]ClO$_4$·$^1/_2$CH$_2$Cl$_2$

	Distances		
Cr(1)−S(1)	2.417(3)	Cr(2)−S(3)	2.419(4)
Cr(1)−S(2)	2.445(3)	Cr(2)−S(4)	2.449(3)
Cr(1)−O(1)	1.951(6)	Cr(2)−O(3)	1.952(7)
Cr(1)−O(2)	1.958(7)	Cr(2)−O(4)	1.947(6)
Cr(1)−N(3)	1.970(6)	Cr(2)−N(9)	1.974(7)
Cr(1)−N(4)	1.984(6)	Cr(2)−N(10)	1.985(6)
O(1)−N(1)	1.338(9)	O(3)−N(7)	1.340(10)
O(2)−N(6)	1.341(8)	O(4)−N(12)	1.338(10)
N(1)−N(2)	1.271(11)	N(7)−N(8)	1.258(10)
N(2)−N(3)	1.333(10)	N(8)−N(9)	1.310(13)
N(4)−N(5)	1.322(10)	N(10)−N(11)	1.313(11)
N(5)−N(6)	1.270(13)	N(11)−N(12)	1.272(12)
	Angles		
O(1)−Cr(1)−O(2)	96.3(3)	O(3)−Cr(2)−O(4)	94.6(3)
O(1)−Cr(1)−N(3)	77.2(3)	O(3)−Cr(2)−N(9)	77.4(3)
O(1)−Cr(1)−N(4)	98.3(3)	O(3)−Cr(2)−N(10)	100.2(3)
O(1)−Cr(1)−S(1)	158.4(2)	O(3)−Cr(2)−S(3)	158.7(2)
O(1)−Cr(1)−S(2)	87.5(2)	O(3)−Cr(2)−S(4)	87.2(2)
O(2)−Cr(1)−N(3)	94.1(3)	O(4)−Cr(2)−N(9)	93.4(3)
O(2)−Cr(1)−N(4)	77.6(3)	O(4)−Cr(2)−N(10)	77.2(3)
O(2)−Cr(1)−S(1)	87.6(2)	O(4)−Cr(2)−S(3)	88.9(2)
O(2)−Cr(1)−S(2)	158.6(2)	O(4)−Cr(2)−S(4)	158.3(2)
N(3)−Cr(1)−N(4)	170.2(3)	N(9)−Cr(2)−N(10)	170.2(3)
N(3)−Cr(1)−S(1)	81.3(2)	N(9)−Cr(2)−S(3)	81.5(2)
N(3)−Cr(1)−S(2)	107.3(2)	N(9)−Cr(2)−S(4)	108.0(2)
N(4)−Cr(1)−S(1)	103.3(2)	N(10)−Cr(2)−S(3)	101.0(2)
N(4)−Cr(1)−S(2)	81.0(2)	N(10)−Cr(2)−S(4)	81.3(2)
S(1)−Cr(1)−S(2)	96.7(1)	S(3)−Cr(2)−S(4)	97.3(1)

of a racemic [Cr(Me3L)]ClO$_4$ pair and a dichloromethane molecule. Selected bond parameters are collected in Table 2.

The ligand binds the metal in hexadentate fashion, each ONS-coordinating half spanning meridionally. The CrS$_2$N$_2$O$_2$ coordination sphere is severely distorted from ideal octahedral geometry, the cis and trans angles in the sphere spanning the ranges 77.2(3)−108.0(2)° and 158.4(2)−170.2(3)°, respectively. The five-membered rings incorporating N,O and N,S chelation are satisfactorily planar (mean deviation 0.02−0.07 Å).

The Cr−S distances are of two types in each molecule, with averages of 2.418(4) and 2.447(3) Å. Interestingly in high-spin [Fe(Me3L)]ClO$_4$^{4}, which has only one molecule in the asymmetric unit, the Fe^{III}−S lengths are 2.557(2) and 2.533(2) Å. Thus the Cr−S length is shorter than the Fe−S length by ~0.1 Å. Part of the reason is no doubt the smaller size of the Cr^{3+} ion (ionic radii:[17] Cr^{3+}, 0.615 Å; Fe^{3+}, 0.645 Å). We also note that Fe^{3+} is significantly harder than Cr^{3+} (η values:[18] Cr^{3+}, 9.1; Fe^{3+}, 12.08), and this may selectively accentuate Cr^{III}−S binding over Fe^{III}−S binding because the thioether sulfur is soft. To our knowledge [Cr(Me3L)]ClO$_4$·$^1/_2$CH$_2$Cl$_2$ represents the first example of a structurally characterized Cr(III) complex derived from acyclic ligand supporting Cr^{III}−S (thioether) binding.

Comparisons. In the two reported structures incorporating Cr^{III}−S (thioether) binding, the coordination spheres are of types CrSN$_5$[11] and cis-CrS$_4$Cl$_2$[12] with Cr−S distances lying in the range 2.39−2.41 Å, which corresponds to the shorter distance in our complex. The D_q values of the above complexes (2120 and 1650 cm^{-1}, respectively) are higher than that in [Cr(RxL)]ClO$_4$ (1360 cm^{-1}). The chromium(III)−chromium(II) reduction potentials of the CrSN$_5$ and CrS$_4$Cl$_2$ species lie near −0.9 V versus SCE. The macrocyclic environment stabilizes (in the redox sense) the trivalent state much better than the S$_2$N$_2$O$_2$ coordination sphere does ($E_{1/2}$, −0.1 V). The EPR spectrum of CrSN$_5$ is not reported[11] while CrS$_4$Cl$_2$ is stated to give rise to a very broad signal near $g = 2$.[12] In contrast [Cr(R*x*L)]ClO$_4$ species display well-resolved resonances consistent with strong rhombic distortions. Thus the [Cr(R*x*L)]ClO$_4$ complexes have certain spectral and redox features that are distinct from those of the known macrocycles.

Concluding Remarks

The hexadentate triazene 1-oxide substituted thioether ligand family (H$_2$R*x*L readily binds chromium(III), affording stable complexes of coordination type CrS$_2$N$_2$O$_2$. The crystal structure of the complex $[Cr(Me3L)]^+$ has been determined. This is the first example of a structurally characterized nonmacrocyclic chromium(III) entity incorporating thioether coordination. Chromium(III) binds thioether sulfur better than high-spin iron(III), and a systematic search for new chromium(III)−thioether species should be rewarding.

Experimental Section

Materials. Purification of solvents and preparation of the supporting electrolyte for the electrochemical work were done as before.[19] All other chemicals and solvents were of analytical grade and were used as received.

Physical Measurements. Solution ($\sim 10^{-3}$ M) electrical conductivities were measured with the help of a Philips PR 9500 bridge. Magnetic susceptibilities of solids were measured by using a PAR-155 vibrating sample magnetometer fitted with a Walker Scientific L75FBAL magnet. Electronic spectra were recorded with a Hitachi 330 spectrophotometer. EPR spectra were collected in the X-band using a Varian 109C spectrometer fitted with a quartz Dewar. Calibration was done with DPPH ($g = 2.0037$). Electrochemical measurements were performed on a PAR Model 370-4 electrochemistry system, as reported earlier.[20] All potentials reported in this work are corrected for the junction

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contribution. A Perkin-Elmer 240C elemental analyzer was used to collect microanalytical data (C, H, N).

Preparation of Complexes. The ligands H_2RxL were prepared following the reported procedure.[3d,21–23]

Synthesis of [1,3-Bis((*o*-(1-methyl-1-oxidotriazen-3-yl)phenyl)thio)propanato-*N*^{3},*S*,*O*]chromium(III) Perchlorate ([Cr(Me3L)]ClO$_4$). [*Caution*! *Perchlorate salts of metal complexes with organic ligands are explosive.*] A methanolic (15 mL) solution of $[Cr(H_2O)_4Cl_2]Cl\cdot 2H_2O$ (0.13 g, 0.49 mmol) was added to a dichloromethane solution (15 mL) of ligand H_2Me3L (0.20 g, 0.49 mmol), and the solution was heated at 70–80 °C for a period of 6–7 h. Addition of 0.069 g (0.49 mmol) of solid $NaClO_4\cdot H_2O$ provided a brown precipitate, which was washed with water and methanol. The product was finally dried in vacuo. The yield was 0.194 g (~71%). Anal. Calcd for $C_{17}H_{20}N_6O_6S_2ClCr$: C, 36.69; H, 3.59; N, 15.11. Found: C, 36.74; H, 3.53; N, 15.16.

The brown chelates [Cr(Me2L)]ClO$_4$ and [Cr(Ph2L)]ClO$_4$ were prepared similarly (yield ~70%). Anal. Calcd for $C_{16}H_{18}N_6O_6S_2ClCr$ ([Cr(Me2L)]ClO$_4$): C, 35.42; H, 3.32; N, 15.49. Found: C, 35.37; H, 3.38; N, 15.53. Calcd for $C_{26}H_{22}N_6O_6S_2ClCr$ ([Cr(Ph2L)]ClO$_4$): C, 46.85; H, 3.30; N, 12.61. Found: C, 46.79; H, 3.35; N, 12.57.

X-ray Structure Determination. Cell parameters of [Cr(Me3L)]ClO$_4\cdot{}^1/_2CH_2Cl_2$ (0.22 × 0.48 × 0.60 mm^3) grown by slow diffusion of *n*-hexane into dichloromethane solution were determined by least-squares fits of 30 machine-centered reflections ($2\theta = 15–30°$). The structure was successfully solved in the space group $P\bar{1}$, and no significant correlations were present. Data were collected by the ω-scan method ($2\theta = 3–45°$) on a Nicolet R3m/V diffractometer with graphite-monochromated Mo Kα (λ = 0.710 73 Å) radiation. Two check reflections measured after every 198 reflections showed no significant intensity reduction during an ~55 h exposure to X-rays. Data were corrected for Lorentz-polarization effects and absorption.[24] Of the 6522 reflections collected, 6495 were unique, of which 3427 satisfying $I > 3\sigma(I)$ were used for structure solution.

The structure was solved by direct methods. All non-hydrogen atoms except C(3)–C(6), C(12)–C(15), C(20)–C(23), and C(29)–C(32) were made anisotropic. Hydrogen atoms were added at calculated positions with fixed U = 0.08 Å^2. All refinements were performed by full-matrix least-squares procedures. The highest residual was 0.58 e/Å^3. All calculations were carried out on a MicroVax II computer with the programs of SHELXTL-PLUS.[25] Significant crystal data are listed in Table 3. Atomic coordinates and isotropic thermal parameters of selected atoms of the structure are given in Table 4.

Acknowledgment. We are thankful to the Department of Science and Technology, New Delhi and the Council of Scientific and Industrial Research, New Delhi for financial support. The Jawaharlal Nehru Centre for Advanced Scientific Research, Bangalore provided a Summer Research Fellowship to D.K.D.

Table 3. Crystallographic Data for [Cr(Me3L)]ClO$_4\cdot{}^1/_2CH_2Cl_2$

chem formula	$C_{17.5}H_{21}N_6O_6S_2Cl_2Cr$
fw	598.4
color; habit	dark prismatic
space group	$P\bar{1}$
a, Å	8.484(4)
b, Å	13.538(5)
c, Å	23.476(12)
α, deg	73.56(3)
β, deg	89.34(4)
γ, deg	73.87(3)
V, Å^3	2477(2)
Z	4
T, °C	23
λ, Å	0.71073
ρ_{calcd}, g cm^{-3}	1.615
hkl range	h, −9 to 9; k, −13 to 14; l, 0 to 25
μ, cm^{-1}	8.94
transm coeff	0.6329–0.7210
R,[a] %	5.62
R_w,[b] %	5.98
GOF[c]	1.32

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$; $w^{-1} = \sigma^2(|F_o|) + g|F_o|^2$; $g = 0.0001$. [c] Goodness of fit is defined as $[w(|F_o| - |F_c|)^2/(n_o - n_v)]^{1/2}$, where n_o and n_v denote the numbers of data and variables, respectively.

Table 4. Selected Atomic Coordinates (×10^4) and Equivalent[a] Isotropic Displacement Coefficients (Å^2 × 10^3) for [Cr(Me3L)]ClO$_4$

	x	y	z	U(eq)
Cr(1)	1719(2)	7971(1)	9490(1)	45(1)
O(1)	−426(7)	8233(4)	9822(3)	55(3)
O(2)	2936(7)	8106(5)	10155(3)	57(3)
N(1)	−1335(9)	9257(6)	9607(3)	53(4)
N(2)	−779(10)	9970(6)	9261(3)	51(3)
N(3)	760(9)	9534(5)	9148(3)	46(3)
N(4)	2667(8)	6451(5)	9968(3)	41(3)
N(5)	3565(9)	6269(6)	10463(3)	51(3)
N(6)	3663(9)	7143(7)	10541(3)	55(4)
S(1)	3918(3)	8348(2)	8881(1)	59(1)
S(2)	649(3)	7100(2)	8864(1)	51(1)
C(2)	1593(11)	10240(6)	8829(3)	43(4)
C(7)	3163(12)	9784(7)	8671(4)	49(4)
C(11)	1480(10)	5753(6)	9329(4)	44(4)
C(16)	2400(9)	5550(6)	9855(4)	40(3)
Cr(2)	625(2)	7862(1)	4474(1)	50(1)
O(3)	2385(7)	8295(5)	4773(3)	57(3)
O(4)	−624(7)	7866(5)	5174(3)	59(3)
N(7)	2100(10)	9364(7)	4588(3)	58(4)
N(8)	826(11)	9983(6)	4273(3)	56(4)
N(9)	−113(9)	9442(5)	4145(3)	51(3)
N(10)	1237(8)	6317(5)	4935(3)	47(3)
N(11)	622(10)	6056(6)	5453(3)	58(4)
N(12)	−315(10)	6884(7)	5563(3)	61(4)
S(3)	−1863(3)	8029(2)	3916(1)	68(1)
S(4)	2586(3)	7146(2)	3815(1)	63(1)
C(19)	−1652(12)	10048(7)	3849(4)	52(4)
C(24)	−2626(12)	9462(8)	3732(4)	59(4)
C(28)	3056(11)	5767(7)	4237(4)	54(4)
C(33)	2373(10)	5494(6)	4768(4)	45(4)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Supporting Information Available: Tables of full listing of atomic coordinates (Table S1), complete bond distances (Table S2), angles (Table S3), anisotropic displacement parameters (Table S4), and hydrogen atom positional parameters (Table S5) (8 pages). Ordering information is given on any current masthead page.

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