absent reflections were eliminated, and symmetry-equivalent reflections were averaged to yield a set of unique reflections. Only those data with $F_o > 3\sigma(F_o)$ were used in least-squares refinement.[31]

A Patterson synthesis yielded the positions of the molybdenum atoms. All other non-hydrogen atoms were located with use of successive difference-Fourier syntheses. Positional and thermal parameters (anisotropic for Mo, P, F, O, and C) were refined by full-matrix least squares.[32] Hydrogen atoms were not located and were placed in idealized fixed positions for the final cycles of refinement. Final refinement parameters are given in Table I. A final difference-Fourier map was essentially featureless; the largest peak, 0.97 e Å^{-3}, was located near the CF_3 group of the monodentate carboxylate.

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Registry No. **1a**, 72509-74-1; **1b**, 72509-73-0; **1c**, 98064-56-3; $Mo_2(TFA)_4{\cdot}PEt_3{\cdot}PBu_3$, 98064-57-4; $Mo_2(TFA)_4$, 36608-07-8; Mo, 7439-98-7.

Supplementary Material Available: Tables of calculated hydrogen atom positions, selected bond distances and angles (Table III), anisotropic thermal parameters, and structure factors for **1c** (9 pages). Ordering information is given on any current masthead page.

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Contribution from the Department of Chemistry,
Georgetown University, Washington, D.C. 20057

Characterization of an Unsymmetrical Oxo-Sulfido Complex, $[(n\text{-Bu})_4N]_2[syn\text{-}(S_2)OMo(\mu\text{-}S)_2MoS(S_2)]$

XINQUAN XIN, NANCY L. MORRIS, GEOFFREY B. JAMESON,* and MICHAEL T. POPE*

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The complex $[(n\text{-Bu})_4N]_2[Mo_2S_7O]$ crystallizes in the orthorhombic space group *Pbca* in a unit cell with dimensions $a = 18.026$ (3) Å, $b = 16.085$ (2) Å, $c = 31.883$ (4) Å, and $Z = 8$. The structure, described by 396 variable parameters, was refined on F by full-matrix least-squares methods using 2628 reflections with $I > 3\sigma(I)$ in the range 0.0308 Å^{-1} < $(\sin\theta)/\lambda$ < 0.4239 Å^{-1} to final values of R and R_w of 0.083 and 0.106. The structure of the $Mo_2S_7O^{2-}$ unit is similar to that of the $Mo_2S_8^{2-}$ anion, except that the apical positions are disordered between O and S. The average Mo-S/O separation of 1.965 (6) Å is in close accord with the electron-weighted average of 1.97 Å from analogous $Mo_2S_8^{2-}$ and $Mo_2S_6O_2^{2-}$ ions. Spectroscopic evidence suggests that the compound is pure and not a solid solution of the above two ions.

Introduction

Molybdenum-sulfur compounds are of importance in the chemistry of several enzymes, such as xanthine oxidase, sulfite oxidase, and aldehyde reductase, and a molybdenum-iron-sulfur cluster is found in nitrogenase.[1] In addition hydrogenation, hydrogendenitrogenation, and hydrodesulfurization are catalyzed heterogeneously by molybdenum-sulfide species.[2] Finally, the study of the condensation of MoS_4^{2-} is an obvious elaboration of the extensive chemistry of polyoxoanions.[3-5] Mononuclear $MoS_nO_{4-n}^{2-}$, n = 0-14, species have been known for some time. Recently, there have been characterized a number of symmetrical di- and trinuclear complexes, such as $Mo_2S_6O_2^{2-}$ and $Mo_2S_8^{2-}$, and one probably unsymmetrical species, $Mo_2OS(\mu\text{-}S_2)(\text{Et-dtc})_2$ (Et-dtc = *N,N'*-diethyldithiocarbamate).[8] We report here the complete characterization of what we believe to be an unsymmetrical compound $[(n\text{-Bu})_4N]_2[(S_2)OMo(\mu\text{-}S)_2MoS(S_2)]$ of the non-carbonaceous molybdenum-sulfur cluster compounds.

Experimental Section

Preparation of the Compound. The title compound was prepared by dissolving 0.65 g of $(NH_4)_2MoS_4$[9-11] in 15 mL of water to which 0.6 mL of 2 N HCl was added with vigorous stirring. The solution turned a dark red color. The complex was crystallized by the addition of 1.8 g of $(n\text{-Bu})_4NBr$ in 10 mL of water (yield 1.3 g) and recrystallized by permitting a hot (60 °C) methanol solution to cool and evaporate over 2 days (yield 0.65 g or 56%) to give dark red daggers with distinctively curved edges, soluble in acetone, acetonitrile, and dimethyl sulfoxide and slightly soluble in methanol. The crystals are weakly deliquescent.

Anal. Found (calcd for $[(n\text{-Bu})_4N]_2[Mo_2S_7O]$): C, 42.38 (41.90); H, 8.28 (7.91); N, 3.12 (3.05); S, 24.96 (24.47); Mo, 21.30 (20.92); O, 1.56 (1.74).

UV-visible spectrum ($(n\text{-Bu})_4N^+$ salt dissolved in CH_3CN): 273 nm (ϵ 1.51 × 10^4 cm^{-1} M^{-1}), 233 nm (2.38 × 10^4), shoulder at 305 nm and

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(11) For comparison with other tetra-*n*-butylammonium salts the $[(n\text{-Bu})_4N]_2MoS_4$ salt was prepared by addition of an $(n\text{-Bu})_4NBr$ solution to an aqueous solution of $(NH_4)_2MoS_4$ without vigorous stirring. Anal. Found (calcd for $C_{32}H_{72}N_2MoS_4$): C, 54.23 (54.20); H, 10.31 (10.23); N, 3.90 (3.95); Mo, 13.43 (13.53); S, 18.23 (18.08). Red needles; λ_{max}(acetonitrile) 467, 326, 244 nm; λ_{max}(water) 467, 317, 242 nm.

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Table I. Positional Parameters and Their Estimated Standard Deviations[a]

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
Mo1	0.62722 (9)	0.1303 (1)	0.66227 (6)	4.17 (4)	C133	0.228 (2)	0.878 (1)	0.5124 (8)	9.2 (8)
Mo2	0.53126 (9)	0.1229 (1)	0.59286 (5)	4.27 (4)	C134	0.222 (2)	0.909 (2)	0.555 (1)	12 (1)
S1	0.5614 (3)	0.0160 (4)	0.6381 (2)	5.1 (1)	C141	0.253 (1)	0.666 (1)	0.4040 (7)	5.5 (6)
S2	0.5662 (3)	0.2397 (4)	0.6306 (2)	4.7 (1)	C142	0.258 (1)	0.578 (1)	0.3858 (8)	6.6 (7)
S11	0.6229 (4)	0.2020 (5)	0.7274 (2)	7.0 (2)	C143	0.284 (1)	0.578 (2)	0.3395 (8)	8.6 (7)
S12	0.6225 (4)	0.0731 (4)	0.7315 (2)	7.3 (2)	C144	0.229 (2)	0.629 (2)	0.3142 (9)	10.1 (9)
S21	0.4118 (3)	0.0624 (4)	0.5797 (2)	6.2 (2)	C211	0.137 (2)	0.657 (2)	0.7722 (7)	11 (1)
S22	0.4170 (3)	0.1900 (4)	0.5767 (2)	5.7 (2)	C212	0.054 (2)	0.652 (2)	0.790 (1)	11 (1)
SO13	0.7282 (6)	0.1264 (6)	0.6408 (3)	7.8 (3)	C213	0.069 (3)	0.662 (2)	0.839 (1)	14 (1)
SO23	0.5946 (5)	0.1063 (7)	0.5425 (3)	7.3 (3)	C214B	0.019 (4)	0.642 (4)	0.861 (2)	11 (2)*
N1	0.2350 (9)	0.6745 (9)	0.4512 (5)	4.3 (4)	C214A	0.060 (4)	0.581 (5)	0.862 (2)	12 (2)*
N2	0.136 (1)	0.651 (1)	0.7234 (6)	7.1 (6)	C221	0.102 (1)	0.566 (1)	0.7100 (7)	7.4 (7)
C111	0.292 (1)	0.629 (1)	0.4756 (6)	5.6 (6)	C222	0.136 (2)	0.490 (2)	0.728 (1)	16 (1)
C112	0.373 (1)	0.654 (1)	0.4695 (8)	6.6 (7)	C223	0.103 (4)	0.417 (3)	0.708 (1)	29 (3)
C113	0.425 (2)	0.605 (2)	0.494 (1)	13 (1)	C224	0.124 (3)	0.393 (3)	0.679 (2)	27 (2)
C114	0.505 (2)	0.620 (2)	0.487 (1)	14 (1)	C231	0.216 (1)	0.655 (1)	0.7099 (9)	7.6 (7)
C121	0.162 (1)	0.635 (1)	0.4617 (6)	5.1 (5)	C232	0.230 (1)	0.649 (2)	0.6643 (9)	12 (1)
C122	0.097 (1)	0.672 (1)	0.4399 (7)	6.1 (6)	C233	0.305 (2)	0.652 (2)	0.6537 (9)	11 (1)
C123	0.024 (1)	0.637 (1)	0.4554 (7)	5.9 (6)	C234	0.322 (2)	0.641 (2)	0.609 (1)	13 (1)
C124	−0.042 (1)	0.668 (2)	0.435 (1)	10.0 (9)	C241	0.090 (1)	0.722 (2)	0.7059 (8)	7.7 (7)
C131	0.240 (1)	0.766 (1)	0.4596 (6)	4.4 (5)	C242	0.113 (2)	0.808 (3)	0.719 (1)	16 (1)
C132	0.231 (1)	0.788 (1)	0.5048 (7)	6.2 (6)	C243	0.071 (4)	0.869 (2)	0.698 (1)	21 (2)
					C244	0.095 (2)	0.891 (3)	0.663 (2)	19 (2)

[a] Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as follows: $^4/_3[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

weak shoulders at 390 and 475 nm (Figure 1).

Infrared spectrum (KBr pellet): 940 (s, (Mo═O)), 518 (m), 460 (m) 350 (m) cm^{-1}; cation bands at 720 and 865 cm^{-1}.

Cyclic voltammetry (1 mM acetonitrile solution, pyrolytic graphite electrode with 0.1 M $[(n\text{-Bu})_4N][ClO_4]$ supporting electrolyte): irreversible reduction at 1.8 V; no other redox features between 0 and −2.0 V vs. Ag/AgCl.

Crystal Structure Determination

The title complex crystallizes in the uniquely determinable space group *Pbca* in a unit cell with dimensions a = 18.026 (3), b = 16.085 (2), and c = 31.883 (4) Å, derived from least-squares refinement of the setting angles of 17 reflections in the range 0.3119 Å^{-1} < (sin θ)/λ < 0.4239 Å^{-1}. The calculated and observed densities are 1.307 (Z = 8) and 1.305 (5) g cm^{-3}. Eventually a marginally suitable crystal was located, and data were collected at 22 °C by using ω scans with on-line profile analysis on a Picker FACS-I diffractomer running under NRC(Canada) software. Graphite-monochromated Mo Kα radiation (λ = 0.7107 Å) was used; no overlap of data was detected despite a broad scan range of 0.7° to either side of the calculated peak position dictated by the poor mosaicity of the crystal. Direct methods (MULTAN80), as incorporated in the Enraf-Nonius SDP, were used to solve the structure. Atomic scattering factors and anomalous dispersion corrections were taken from ref 27. Values of R and R_w of 0.098 and 0.109, respectively, were obtained for a model without hydrogen atoms included, with one carbon atom disordered, and with only Mo and S atoms anisotropically refined. Of the 4414 unique data measured (+h+k+l), the 2628 data with $I > 3\sigma(I)$ in the range 0.0308 Å^{-1} < (sin θ)/λ < 0.5394 Å^{-1} were corrected for absorption by using Gaussian integration (μ(Mo Kα) = 8.586 cm^{-1}, dagger-shaped crystal, 0.30 × 0.20 × 0.08 mm, maximum and minimum transmission factors of 0.916 and 0.701), and the weighting scheme was adjusted, $w = 1/\sigma^2$, where $\sigma^2 = \sigma^2_{\text{counting}} + (0.05F_o^2)^2$. Hydrogen atoms were included, where calculable, as a fixed contribution to F_c, and refinements converged at values for R and R_w of 0.083 and 0.106 for a model in which all nondisordered atoms (except H) were refined anisotropically. No evidence for extinction was apparent. Tables of anisotropic thermal parameters, hydrogen atom parameters, and $10F_c$ vs. $10F_3$ have been deposited as supplementary material. The final positional parameters with equivalent isotropic thermal parameters are given in Table I.

Description and Discussion

The dianion $Mo_2S_7O^{2-}$ is illustrated in Figure 2, along with selected metrical details (more are given in Table II). The structure of the basal moiety $Mo_2(S_2)_2(\mu\text{-}S)_2$ is very similar to those for other *syn*-$Mo_2(S_2)_2(\mu\text{-}S)_2X_2$ (X = O, S) structures. The dihedral angle between the two basal planes is 142.9°, and the Mo(V)···Mo(V) separation is 2.811 (1) Å, a separation common to other Mo(V)···Mo(V) dimers and indicative of a single Mo–Mo bond. The dihedral angle between the two MoS_2(bridging) triangles is 28.3°. The Mo atoms are displaced 0.780 (2) (Mo2) and 0.785 (2) (Mo1) Å from the basal plane. Dimensions in-

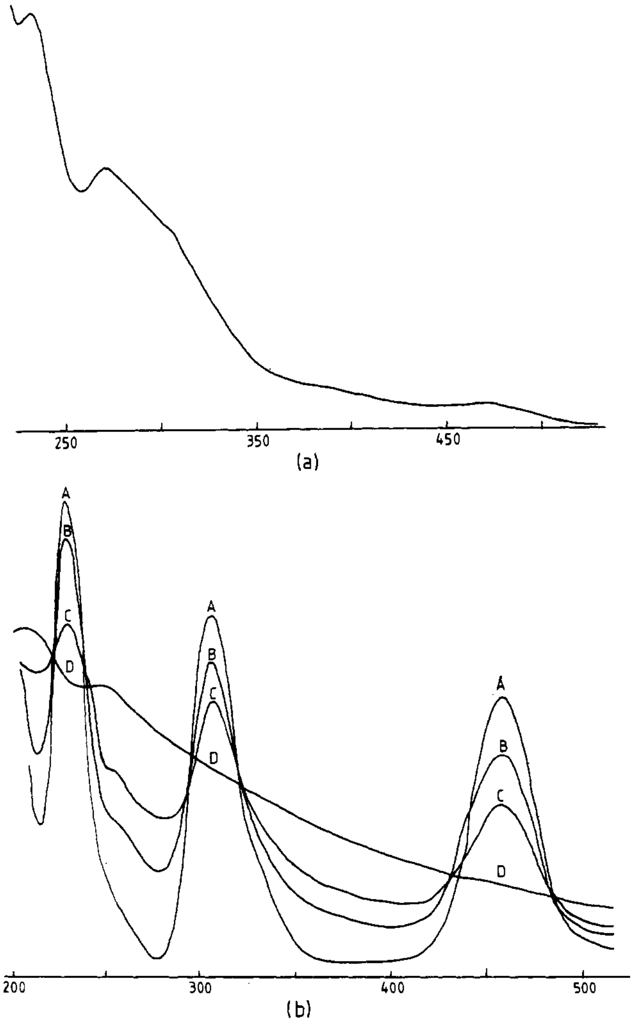

Figure 1. UV–visible spectra for (a) $Mo_2S_7O^{2-}$ in acetonitrile solution and (b) aqueous solution of $(NH_4)_2MoS_4$ (0.026 g/50 mL) to which HCl had been added with vigorous stirring. Curves A, B, C, and D are spectra recorded within 30 min of the addition of 0, 1, 2, and 4 drops of 3 M HCl, respectively.

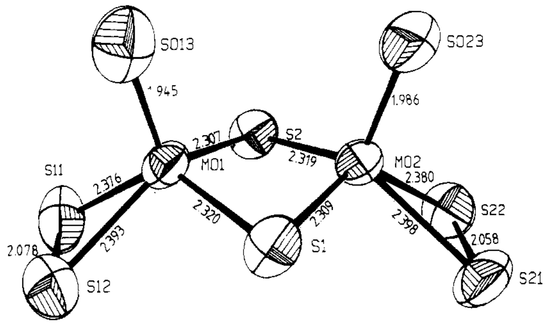

Figure 2. ORTEP diagram of the $Mo_2S_7O^{2-}$ anion, with selected metrical details. Thermal ellipsoids are drawn at the 50% probability level.

Table II. Selected Bond Distances and Angles for the Anion

	Distances, Å		
Mo1–S1	2.320 (4)	Mo1–S2	2.307 (3)
Mo2–S1	2.309 (4)	Mo2–S2	2.319 (4)
Mo1–S11	2.376 (4)	Mo1–S12	2.393 (4)
Mo2–S21	2.398 (4)	Mo2–S22	2.380 (4)
Mo1–SO13	1.945 (6)	Mo2–SO23	1.986 (6)
S11–S12	2.078 (6)	S21–S22	2.058 (5)
	Angles, deg		
S1–Mo1–S2	102.5 (1)	S1–Mo2–S2	102.4 (1)
S1–Mo1–S11	131.1 (2)	S1–Mo2–S22	132.5 (1)
S2–Mo1–S12	132.8 (2)	S2–Mo2–S21	131.6 (1)
S1–Mo1–S12	89.1 (1)	S1–Mo2–S21	91.0 (1)
S2–Mo1–S11	89.8 (1)	S2–Mo2–S22	88.8 (1)
S1–Mo1–SO13	109.7 (2)	S1–Mo2–SO23	105.6 (2)
S2–Mo1–SO13	108.4 (2)	S2–Mo2–SO23	111.9 (2)
S11–Mo1–S12	51.6 (2)	S21–Mo2–S22	51.0 (1)
S11–Mo1–SO13	110.7 (2)	S21–Mo2–SO23	108.6 (2)
S12–Mo1–SO13	110.2 (2)	S22–Mo2–SO23	112.5 (2)
Mo1–S1–Mo2	74.8 (1)	Mo1–S2–Mo2	74.8 (1)
Mo1–S11–S12	64.6 (2)	Mo2–S21–S22	64.0 (2)
Mo1–S12–S11	63.8 (2)	Mo2–S22–S21	64.9 (2)

Table III. Mo=O and Mo=S (Å) Separations for Mo_2S_6XY Complexes (X, Y = O, S)[a]

complex	Mo=O	Mo=S	ref
anti-$(\eta^5\text{-}C_5H_5)_2Mo_2S_2O_2$	1.679 (6)		17
anti-$(\eta^5\text{-}C_5H_5)_2Mo_2S_4$		2.141	18
syn-$Mo_2S_6O_2^{2-}$	1.683		12
syn-$Mo_2S_6O_2^{2-}$	1.675		13
syn-$Mo_2S_2O_2$(cys-Me)$_2$	1.71 (3)		20
syn-$Mo_2S_2O_2$(cys)$_2^{2-}$	1.62 (3)		21
syn-$Mo_2S_2O_2$(his)$_2^{2-}$	1.71 (2)		22
syn-$Mo_2S_2O_2$(EDTA)	1.683 (6)		23
syn-$Mo_2S_2O_2$(*i*-mnt)$_2^{2-}$	1.664 (7)		24
syn-Mo_2S_4(*n*-Budtc)$_2$	1.937 (6)		16[b]
syn-Mo_2S_3O(Et-dtc)$_2$	1.82 (12)	1.98 (1)	8[c]
syn-$Mo_2S_7O^{2-}$	1.965 (6)		*d*
syn-Mo_2S_4(dme)$_2^{2-}$		2.100 (4)	15
anti-Mo_2S_4(dme)$_2^{2-}$		2.129 (3)	15
syn-$Mo_2S_8^{2-}$		2.118 (1)	25
syn-Mo_2S_4(Et-dtc)$_2$		2.092	14

[a] Abbreviations: Et-dtc, *N,N'*-diethyldithiocarbamate; *n*-Budtc, *N,N'*-di-*n*-butyldithiocarbamate; dme, dimercaptoethane; *i*-mnt, isomaleonitriledithiolate; cys, cysteine; cys-Me, cysteine methyl ester; his, histidine; EDTA, ethylenediaminetetraacetic acid. [b] See text. [c] Terminal oxo and sulfido moieties are disordered. [d] This work.

volving the apical Mo=X moiety are however different. The five precisely determined structures containing the $Mo_2S_6O_2^{2-}$ unit (i.e. with esd <0.01 Å) yield an average Mo=O separation of 1.678 Å; similarly the average Mo=S separation in $Mo_2S_8^{2-}$ structures is 2.111 Å. (see Table III[8,12-25]) The electron-weighted average of the mean Mo=S and Mo=O separations is 1.97 Å, a value that corresponds closely with the average value of 1.965 (6) Å observed herein $Mo_2S_7O^{2-}$. A rather similar value was reported for a complex of purported composition Mo_2S_4(*n*-Budtc)$_2$.[16] Thus, we concur with the earlier reservations[14] about the structure and with the suggestion[15] that that material contained oxygen as well as sulfur in the apical positions.

Density measurements, infrared spetroscopy, microanalysis, and the crystal structure determination all support unequivocally an empirical formulation of the anion as $Mo_2S_7O^{2-}$. The X-ray data, however, do not distinguish between a solid solution of the two already well-characterized compounds $Mo_2S_6O_2^{2-}$ [12,13] and $Mo_2S_8^{2-}$ [25] and discrete $Mo_2S_7O^{2-}$ ions. Spectroscopic data support the latter, although not unequivocally. With the exception of the ν(Mo=O) stretch at 940 cm^{-1}, the infrared spectra of $Mo_2S_6O_2^{2-}$ and $Mo_2S_8^{2-}$ (as the tetraethylammonium salts) are very similar.[26] Not surprisingly the spectrum of $Mo_2S_7O^{2-}$ (as the tetrabutylammonium salt) is very similar to these two, with one significant exception: the peak at 460 cm^{-1} has a higher integrated intensity than the peak at 518 cm^{-1}, whereas for both pure $Mo_2S_8^{2-}$ and $Mo_2S_6O_2^{2-}$ the reverse is true. Second, the UV–visible spectrum of $Mo_2S_7O^{2-}$ is not a simple summation of those for $Mo_2S_6O_2^{2-}$ (resolved peaks at 275, 310, 440 nm in CH_3CN) and $Mo_2S_8^{2-}$ (resolved peaks at 295, 467, 573 nm in CH_3CN). Third, the cyclic voltammogram shows only one peak. Finally the density measurement (by flotation) indicates that individual crystals have identical density, consistent with a pure compound. Thus, although we support the formulation of this material as pure $Mo_2S_7O^{2-}$, we remain cognizant of solid-state effects on IR spectra and solvent-media effects on UV–visible spectra. Similar conclusions were reached for the *N,N'*-diethyldithiocarbamate analogue.[8]

The formation of $Mo_2S_7O^{2-}$ was accompanied by the evolution of H_2S. At this stage we have not distinguished between two plausible redox-condensation reaction schemes:

$$2MoS_4^{2-} + {}^1/_2O_2 + H_3O^+ \rightarrow Mo_2S_7O^{2-} + H_2S + OH^-$$

$$2MoS_4^{2-} + 2H_3O^+ \rightarrow Mo_2S_7O^{2-} + H_2S + H_2 + H_2O$$

The first reaction is formally similar to that proposed for the formation of $Mo_2S_8^{2-}$:[24]

$$2MoS_4^{2-} + RSSR \rightarrow Mo_2S_8^{2-} + 2RS^-$$

The course of the reaction as HCl is added is shown in Figure 1. It is clear from the imperfect isosbestic points, especially at ~440 nm, that the reaction does not involve only MoS_4^{2-} and $Mo_2S_7O^{2-}$ species. Thus the first step of the redox–condensation

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reaction, by analogy to the formation of MoS_nO_{4-n} species is very likely

$$MoS_4^{2-} + H_2O \rightarrow MoS_3O^{2-} + H_2S$$

followed by reaction of the intermediate

$$MoS_3O^{2-} + MoS_4^{2-} + {}^1/_2O_2 + H^+ \rightarrow Mo_2S_7O^{2-} + OH^-$$

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Registry No. $[(n\text{-Bu})_4N]_2[Mo_2S_7O]$, 97689-85-5; $(NH_4)_2MoS_4$, 15060-55-6.

Supplementary Material Available: Listings of bond distances and angles of the cation, anisotropic thermal parameters, hydrogen atom parameters, and structure factor amplitudes (22 pages). Ordering information is given on any current masthead page.

Notes

Contribution from the ISSECC, CNR, and Department
of Chemistry, University of Florence, Florence, Italy,
and Institut für Physikalische Chemie,
Technische Hochschule, Darmstadt, FRG

Anisotropic Exchange in Transition-Metal Dinuclear Complexes. 7.[1] Bis[μ-2-(diethylamino)ethanolato]dibromodicopper(II)

A. Bencini,[2a] D. Gatteschi,*[2b] C. Zanchini,[2b] and W. Haase[2c]

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In this series of papers the **g** and **D** tensors of dinuclear copper(II) complexes were measured, showing that in any case a dominant contribution[1,3–8] of the exchange interaction to the zero field splitting was operative. For all the studied complexes in which the bridging ligands were oxygen atoms, either hydroxo or alkoxo in nature, the coordination environment around the copper ions was tetragonally distorted with the bridging ligands occupying two adjacent equatorial positions. The value of D was found to be uncorrelated to the isotropic exchange constant, J, but a smooth tendency to a decrease in D_{zz} as the copper–copper, r, distance increases was evident.[3]

Recently the crystal structure[9,10] and the temperature dependence of the magnetic susceptibility[11] of bis[μ-2-(diethylamino)ethanolato]dibromodicopper(II) was reported. The complex is dinuclear with each copper ion coordinated to a bromine, to a nitrogen atom of the aminoalcohol, and to the two bridging oxygen atoms. The coordination environment is close to square planar, with a significant tetrahedral distortion. The magnetic susceptibility data showed that the two metal ions are coupled in an antiferromagnetic way, with a singlet–triplet splitting of 817 cm^{-1}.

Since the reported copper–copper distance,[9] 3.033 (5) Å, falls in a region of our D_{zz} vs. r plot where only one data point is available,[3] we decided to measure the single-crystal EPR spectra of the compound, in order to obtain D_{zz} and see if it confirms the relation with the copper–copper distance.

Single crystals of the compound prepared as previously described[9] were grown by slow evaporation of ethanol solutions. They have a complex shape with $(10\bar{1})$ and $(\bar{1}01)$ best developed faces. Polycrystalline powder EPR spectra, recorded at room temperature at the Q-band frequency, are shown in Figure 1. They are similar to those of the other members of the series, being broad and showing no resolved copper hyperfine splitting. Single-crystal EPR spectra were recorded at the Q-band frequency by rotating, with a one-circle goniometer, around b, the y laboratory axis, x, which is orthogonal to b in the (101) face, and $z = x \times y$. An analysis of the data, performed with the procedure previously described,[4] yielded the **g** and **D** tensors. They are shown in Table I, the principal directions being expressed as direction cosines referred to the laboratory frame. As in the other members of the series, the largest D component, $D_{zz} = -0.372$ (7) cm^{-1}, is found roughly parallel to the direction of the largest g value, while the smallest D component, $D_{xx} = 0.140$ (6) cm^{-1}, is found parallel to the copper–copper direction. The negative sign of D_{zz} is assigned on the basis of considerations developed elsewhere.[3]

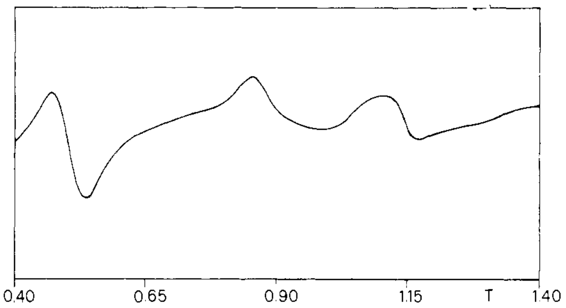

Figure 1. Polycrystalline powder EPR spectrum at the Q-band frequency and room temperature of bis(μ-(2-diethylamino)ethanolato)dibromodicopper(II).

Table I. Principal Values and Directions of **g** and **D** Tensors[a] for Bis[μ-2-(diethylamino)ethanolato]dibromodicopper(II)

g_{xx}	g_{yy}	g_{zz}
2.08 (1)	1.98 (2)	2.19 (2)
0.7 (1)	−0.58 (8)	0.4 (1)
−0.07 (9)	0.51 (5)	0.86 (2)
−0.7 (1)	−0.63 (8)	0.3 (1)
D_{xx}[b]	D_{yy}[b]	D_{zz}[b]
0.140 (6)	0.231 (8)	−0.372 (7)
0.46 (4)	−0.73 (2)	0.499 (9)
0.27 (4)	0.65 (1)	0.708 (5)
−0.84 (1)	−0.19 (5)	0.500 (8)

[a] The directions are given by the cosines referred to the laboratory frame specified in the text. [b] In cm^{-1}.

The **g** tensor is determined only with moderate accuracy, and experimental errors must be the explanation for the low value of $g_y = 1.98$ (2).

The observed $|D_{zz}|$ value is smaller than that previously reported[3] for bis(μ-hydroxo)bis[bis(2-methylimidazole)copper(II)] diperchlorate dihydrate, in which the copper–copper distance is 2.993

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