

The Crystal Structure of Dioxomolybdenum(VI) Diethyldithiocarbamate

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The crystal structure of dioxomolybdenum(VI) diethyldithiocarbamate, $\text{MoO}_2[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$, has been determined from three-dimensional X-ray film data. The structure is monoclinic, space group $C2/c$. The elementary cell contains four formula units and has the dimensions

$$\begin{aligned} a &= 17.3839 \pm 14 \text{ \AA} \\ b &= 8.6656 \pm 7 \text{ \AA} \\ c &= 13.5907 \pm 12 \text{ \AA} \\ \beta &= 124.66^\circ \pm 1 \end{aligned}$$

The coordination around molybdenum is a distorted octahedron with four sulfur and two oxygen atoms ($\text{Mo}-2\text{S}_1 = 2.44(1) \text{ \AA}$, $\text{Mo}-2\text{S}_2 = 2.63(1) \text{ \AA}$, $\text{Mo}-2\text{O}_1 = 1.63(3) \text{ \AA}$). The oxygens are in a *cis*-position, *i.e.* the distance between the oxygens represents an edge in the polyhedron. The planarity of the NCS_2 group, expected from previous works, has been confirmed. IR spectra and dipole moment measurements reported in literature are in concordance with this structure determination.

Crystals of dioxomolybdenum(VI) diethyldithiocarbamate as well as corresponding butyl compounds were prepared by Moore and Larsson,¹ who concluded from IR spectra that these complexes contain a *cis* O-Mo-O group. In order to confirm this conclusion structure determinations using X-ray methods were suggested by Dr. Moore who also kindly supplied me with crystalline material. This article will describe the investigation of the structure of the ethyl complex. Previous single crystal investigations of the diethyldithiocarbamate group have dealt with $\text{M}[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$, $\text{M} = \text{Ni}^{2+}$,² Cu^{2+} ,³ Zn^{2+} .⁴

CRYSTAL DATA AND X-RAY DATA COLLECTING

The cell dimensions and their errors were calculated by means of the least-squares program PIRUM⁵ from powder photographs taken with monochromatized $\text{CuK}\alpha_1$ radiation ($\lambda = 1.54056 \text{ \AA}$) in a Guinier-Hägg type focusing camera. Potassium chloride ($a = 6.29228 \text{ \AA}$)⁶ was used as an internal standard (see Table 1). The dimensions of the monoclinic unit cell are (25°C):

$$\begin{aligned} a &= 17.3839(14) \text{ \AA} \\ b &= 8.6656(7) \text{ \AA} \\ c &= 13.5907(12) \text{ \AA} \\ \beta &= 124.664(7) \\ V &= 1683.6 \text{ \AA}^3 \end{aligned}$$

The observed density, $\rho = 1.65(3) \text{ g/cm}^3$, was found from the apparent loss of weight in water, giving four formula units in the unit cell ($\rho_{\text{calc}} = 1.67 \text{ g/cm}^3$).

Weissenberg photographs were taken with Ni filtered $\text{CuK}\alpha$ radiation for $h0l - h4l$ and $hk0$ reflections.

The reflections systematically absent are hkl when $h+k$ odd, $h0l$ when l odd and h odd, and $0k0$ when k odd, which gives the choice of the space groups $C2/c$ (No. 15) and Cc (No. 9).

The I_0 values from the films were obtained from intensity measurements with the automatic film scanner of the Abrahamson type according to methods described by Werner.⁷

The crystal used was a hexagonal prism, elongated in the $[101]$ -direction, with the dimensions $0.4 \times 0.08 \times 0.08 \text{ mm}^3$. For the data, absorption correction was made ($\mu = 96.1 \text{ cm}^{-1}$).⁸ In the correction for absorption the transmission factors ranged between 0.18 and 0.53.

STRUCTURE DETERMINATION AND REFINEMENT

The higher symmetry, $C2/c$, was taken as the starting point of the structure determination. The result thus obtained was found to be throughout consistent. It was not significantly improved by lowering the symmetry.

In No. 15 $C2/c$ the following point positions are possible: $(0,0,0; \frac{1}{2}, \frac{1}{2}, 0) +$

$$\begin{aligned} 4 (a): & (0,0,0) (0,0,\frac{1}{2}) \\ 4 (b): & (0,\frac{1}{2},0) (0,\frac{1}{2},\frac{1}{2}) \\ 4 (c): & (\frac{1}{4},\frac{1}{4},0) (\frac{3}{4},\frac{1}{4},\frac{1}{2}) \\ 4 (d): & (\frac{1}{4},\frac{1}{4},\frac{1}{2}) (\frac{3}{4},\frac{1}{4},0) \\ 4 (e): & (0,y,\frac{1}{4}) (0,\bar{y},\frac{3}{4}) \\ 8 (f): & (x,y,z); (\bar{x},\bar{y},\bar{z}); (\bar{x},y,\frac{1}{2}-z); (x,\bar{y},\frac{1}{2}+z) \end{aligned}$$

In the Patterson function $P(uvw)$ three high maxima were found which could be identified as due to Mo-Mo vectors and four lower peaks which regarding their heights and positions could be interpreted as corresponding to Mo-S vectors. Using these sets of vectors the following approximate parameter values were derived for the molybdenum and sulphur atoms:

DIOXOMOLYBDENUM DIETHYLDITHIOCARBAMATE 2943

Table 1. X-Ray powder data of $\text{MoO}_2[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$, $\text{CuK}\alpha_1$ radiation.

hkl	$10^5 \sin^2\theta$ obs	$10^5 \sin^2\theta$ calc	d obs	I obs
1 1 0	1085	1080	7.39	vs
$\bar{1}$ 1 1	1136	1132	7.23	vvs
2 0 0	1160	1160	7.15	vs
$\bar{2}$ 0 2	1373	1371	6.57	vs
0 0 2	1903	1899	5.58	w
$\bar{1}$ 1 2	2146	2135	5.26	vs
$\bar{3}$ 1 1	2615	2610	4.76	vs
3 1 2	2775	2767	4.62	w
0 2 0	3173	3160	4.33	} s
4 0 2		3164		
3 1 0	3387	3402	4.19	vvs
0 2 1	3643	3635	4.04	m
1 1 2	3830	3824	3.94	m
$\bar{2}$ 2 1	3971	3951	3.87	vw
$\bar{1}$ 1 3	4096	4087	3.81	vw
2 2 0	4325	4321	3.70	w
2 2 2	4529	4531	3.62	vw
4 0 0	4631	4643	3.58	vw
0 2 2	5066	5059	3.42	w
3 1 1	5149	5143	3.39	w
2 0 4	5387	5379	3.32	w
4 0 4	5494	5484	3.29	vw
2 2 1	5655	5640	3.24	vw
$\bar{5}$ 1 2	5731	5722	3.22	m
$\bar{3}$ 1 4	5942	5932	3.16	m
$\bar{2}$ 2 3	6064	6061	3.13	w
4 2 2	6330	6325	3.06	w
4 2 1	6597	6589	3.00	s
$\bar{1}$ 1 4	7001	6988	2.91	} m
4 2 3		7010	.	
1 3 0	7395	7401	2.83	w
0 0 4	7596	7597	2.79	m
4 2 0	7810	7804	2.76	m
$\bar{2}$ 2 2	7915	7909	2.74	} s
6 0 4		7910	.	
5 1 0	8054	8045	2.71	w
$\bar{1}$ 3 2	8460	8456	2.65	m
$\bar{2}$ 2 4	8544	8540	2.64	w
$\bar{3}$ 3 1	8933	8931	2.58	} s
$\bar{3}$ 1 5		8938	.	
$\bar{3}$ 3 2	9080	9088	2.56	vw
5 1 5	9359	9359	2.52	vw
3 3 0	9730	9723	2.47	vw
4 2 1	9961	9967	2.44	vw
1 3 2	10135	10145	2.42	vw
1 1 4	10366	10366	2.39	s
$\bar{6}$ 2 2	10446	10440	2.38	} m
6 0 0		10447	.	
0 2 4	10759	10757	2.35	m
$\bar{7}$ 1 2	11012	10998	2.32	m
$\bar{6}$ 2 4	11075	11071	2.31	w
$\bar{4}$ 2 5	11221	11229	2.30	vw

	x	y	z
4 Mo in 4(e):	0	0.053	$\frac{1}{4}$
8 S ₁ in 8(f):	0.05	0.13	0.45
8 S ₂ in 8(f):	0.12	0.29	0.33

Three-dimensional electron densities were then calculated using the signs yielded by the contribution to $F(hkl)$ of the molybdenum and sulfur atoms. The oxygens, the nitrogens and a few of the carbon atoms were easily located. The remaining carbon atoms were found after new Fourier calculations using the signs of $F(hkl)$ from all atoms previously found. A least-squares refinement, using block diagonal approximation, was then performed. In the refinement the $|F_o|$'s were weighted according to Cruickshank's weighting procedure, *i.e.* $\omega = 1/(a + |F_o| + c|F_o|^2)$, using the values $a = 90.0$ and $c = 0.02$. Unobserved reflections were not included in the refinement. The atomic scattering curves for Mo⁶⁺, S⁻, O⁻, N, and C (Refs. 8, 9) were used in the structure factor calculations. When the shifts were less than 10 % of the standard deviations, the discrepancy factor R was 13.0 %.

A full matrix least-squares refinement, based on the 551 independent reflections, with isotropic temperature factors gave the R value 12.9 %. The parameter shifts were then less than 3 % of the standard deviations. The heights of the major peaks observed in the final difference Fourier were less than 50 % of the N and C peaks. The electron density of the major peak is 2.0 e/Å³.

No significant improvement of the structure was obtained by introduction of anisotropic temperature factors for the molybdenum and sulfur atoms.

A weight analysis obtained in the final cycle is given in Table 2. Observed and calculated structure factors are found in Table 3, and atomic parameters with standard deviations and temperature factors in Table 4.

Table 2. Weight analyses obtained in the final cycle of the least-squares refinement. ω = weighting factor, $\Delta = |F_o| - |F_c|$.

$ F_o $ interval	$\omega \Delta^2$	Number of reflections	Interval $\sin \theta$	$\omega \Delta^2$	Number of reflections
0.0 - 45.4	1.0867	55	0.0 - 0.4177	1.5077	103
45.4 - 54.5	0.8335	53	0.4177 - 0.5263	0.8992	94
54.5 - 62.4	1.0992	55	0.5263 - 0.6025	0.8829	81
62.4 - 68.5	0.8975	54	0.6025 - 0.6631	0.7920	62
68.5 - 75.1	1.0175	55	0.6631 - 0.7143	0.7735	54
75.1 - 83.6	0.9430	55	0.7143 - 0.7591	0.7731	48
83.6 - 91.7	0.6986	55	0.7591 - 0.7991	1.1699	33
91.7 - 102.3	1.4272	55	0.7991 - 0.8355	0.7947	25
102.3 - 120.6	0.9901	55	0.8355 - 0.8689	1.5241	26
120.6 - 206.4	0.9987	55	0.8689 - 0.9000	0.5987	16

Table 3. Continued.

F	K	L	FC	FC	F	K	L	FC	FC	H	K	L	FC	FC	H	K	L	FC	FC
-3	3	4	114	126	-5	3	10	40	-38	6	4	-3	74	-76	-4	4	6	28	19
-1	3	4	83	65	1	3	10	39	-45	8	4	-3	90	-91	0	4	6	45	21
1	3	4	28	29	-11	3	11	53	54	1C	4	-3	111	-118	2	4	6	48	-36
-7	3	5	100	-121	-9	3	11	e4	63	12	4	-3	61	-69	4	4	6	68	-52
-5	3	5	108	-106	-7	3	11	81	84	16	4	-3	92	-52	6	4	6	71	-61
-3	3	5	84	-82	-5	3	11	77	89	4	4	-2	58	42	-8	4	7	55	55
-1	3	5	124	-106	-3	3	11	96	101	6	4	-2	73	62	-6	4	7	122	124
1	3	5	154	-144	-1	3	11	58	64	4	4	-1	51	50	-4	4	7	128	135
3	3	5	113	-103	-1	3	12	47	49	6	4	-1	99	108	-2	4	7	73	67
5	3	5	68	-60	-11	3	13	85	-81	8	4	-1	101	117	0	4	7	36	35
7	3	5	58	-53	-7	3	13	47	-53	1C	4	-1	87	100	2	4	7	94	85
9	3	5	47	-50	-5	3	13	62	-66	12	4	-1	66	66	4	4	7	120	109
-7	3	6	62	45	-11	3	14	32	-38	8	4	0	68	56	6	4	7	88	79
-5	3	6	41	-31	-9	3	14	71	-63	10	4	0	89	93	-10	4	8	58	-47
-3	3	6	48	-48	-7	3	14	54	-51	4	4	1	68	-66	-8	4	8	40	-35
-1	3	6	47	-40	12	4	-13	66	37	6	4	1	119	-119	-6	4	8	97	88
3	3	6	67	-62	14	4	-13	35	39	8	4	1	150	-149	-4	4	8	88	77
5	3	6	111	-106	16	4	-13	53	67	10	4	1	92	-73	-2	4	8	38	32
7	3	6	53	-45	16	4	-12	61	46	2	4	2	7C	-48	2	4	8	58	58
-9	3	7	137	144	14	4	-11	5C	-56	4	4	2	31	-23	-8	4	9	120	-115
-7	3	7	160	174	14	4	-10	35	-30	6	4	2	87	-81	-6	4	9	141	-144
-5	3	7	74	65	16	4	-10	33	-35	10	4	2	5E	5C	-4	4	9	64	-67
-3	3	7	43	41	12	4	-9	54	50	C	4	3	91	71	0	4	9	49	-56
-1	3	7	86	85	14	4	-9	102	86	2	4	3	62	53	2	4	9	88	-95
1	3	7	79	69	16	4	-9	66	79	4	4	3	97	92	4	4	9	62	-63
7	3	7	87	80	10	4	-8	64	-47	6	4	3	138	141	-1C	4	10	64	-60
7	3	8	72	56	10	4	-7	61	-63	8	4	3	83	73	-8	4	10	77	-68
-5	3	8	120	105	12	4	-7	65	-82	-6	4	4	71	72	-10	4	11	61	75
-3	3	8	68	61	14	4	-7	70	-88	-4	4	4	85	74	-8	4	11	78	41
-1	3	8	42	45	16	4	-7	53	-59	-2	4	4	67	71	-6	4	11	78	68
1	3	8	78	77	12	4	-6	74	-72	2	4	4	54	-36	C	4	11	53	72
3	3	8	74	60	14	4	-6	65	-71	-6	4	5	1CC	-12C	2	4	11	51	67
-9	3	9	86	-75	8	4	-5	71	73	-4	4	5	122	-126	0	4	12	46	50
-3	3	9	106	-104	10	4	-5	85	90	-2	4	5	106	-115	-10	4	13	64	-65
-1	3	9	76	-77	12	4	-5	109	114	2	4	5	66	-63	-8	4	13	88	-87
1	3	9	41	-38	14	4	-5	62	57	4	4	5	100	-109	-6	4	13	46	-58
3	3	9	60	-61	18	4	-5	62	6C	6	4	5	103	-100	-2	4	13	55	-64
-11	3	1C	47	-47	8	4	-4	42	27	8	4	5	68	-67	-10	4	14	46	-40
-5	3	1C	82	-73	10	4	-4	76	58	-8	4	6	3C	22	-8	4	14	45	-41
-7	3	10	97	-86	14	4	-4	44	-24	-6	4	6	40	29					

Table 4. The structure of $\text{MoO}_2[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$.Space group: $C2/c$.Unit cell dimensions: $a = 17.3839(14) \text{ \AA}$ $b = 8.6656(7) \text{ \AA}$ $c = 13.5907(12) \text{ \AA}$ $\beta = 124.664(7)^\circ$ $V = 1683.6 \text{ \AA}^3$ Cell content: $4\text{MoO}_2[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$ 4Mo in $4(e)$; $\pm(0, y, \frac{1}{2})$; $\pm(\frac{1}{2}, \frac{1}{2} + y, \frac{1}{2})$ 8S_1 , 8S_2 , 8O_1 , 8N_1 , $8\text{C}_1 - 8\text{C}_5$ in $9 \times 8(f)$; $\pm(x, y, z)$; $\pm(\frac{1}{2} + x, \frac{1}{2} + y, z)$; $\pm(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$

Atomic parameters and standard deviations obtained by full matrix least-squares refinement with isotropic temperature factors.

Atom	x	y	z	β
Mo	0	0.0541(5)	$\frac{1}{2}$	0.9(1)
S ₁	0.0489(5)	0.1300(14)	0.4509(7)	2.1(2)
S ₂	0.1183(5)	0.2862(14)	0.3313(7)	2.2(2)
O ₁	0.4190(15)	0.4514(37)	0.2451(19)	3.2(5)
N ₁	0.3604(17)	0.1065(43)	0.4713(23)	2.6(5)
C ₁	0.1145(17)	0.2845(44)	0.4521(22)	1.0(5)
C ₂	0.1937(22)	0.4681(56)	0.0196(29)	3.0(7)
C ₃	0.3659(21)	0.1155(52)	0.3676(27)	2.5(6)
C ₄	0.2774(25)	0.1502(61)	0.2565(33)	3.7(8)
C ₅	0.3764(30)	0.1714(66)	0.0409(40)	5.3(11)

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

A projection in the y -direction of the structure of $\text{Mo}[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$ is given in Fig. 1. The shortest distance between two molecules is $3.33(4)$ Å (O_1-C_2). All other inter-molecular distances are longer than 3.6 Å. Thus, no other bonding except for van der Waals interaction is present between the molecules. The molecule has a twofold axis, through the molybdenum, in the $[010]$ -direction.

Table 5. Bond lengths (Å) and bond angles (deg) around the molybdenum atoms with standard deviations.

Bond lengths	Å	Bond angles	deg
$\text{Mo}-\text{S}_1'=\text{Mo}-\text{S}_1''$	2.443(8)	$\text{S}_1'-\text{Mo}-\text{S}_1''$	148.8(5)
$\text{Mo}-\text{S}_2'=\text{Mo}-\text{S}_2''$	2.629(11)	$\text{S}_2'-\text{Mo}-\text{S}_2''$	80.2(4)
$\text{Mo}-\text{O}_1'=\text{Mo}-\text{O}_1''$	1.634(25)	$\text{O}_1'-\text{Mo}-\text{O}_1''$	114.0(20)
$\text{O}_1'-\text{O}_1''$	2.74 (4)	$\text{S}_1'-\text{Mo}-\text{S}_2'$	68.5(3)
$\text{S}_2'-\text{S}_2''$	3.39 (2)	$\text{S}_1''-\text{Mo}-\text{S}_2''$	87.4(3)
$\text{O}_1'-\text{S}_2'=\text{O}_1''-\text{S}_2''$	3.03 (3)	$\text{S}_1'-\text{Mo}-\text{O}_1'$	110.9(8)
$\text{O}_1'-\text{S}_1'=\text{O}_1''-\text{S}_1''$	3.39 (3)	$\text{S}_1''-\text{Mo}-\text{O}_1''$	86.3(8)
$\text{O}_1''-\text{S}_1'=\text{O}_1'-\text{S}_1''$	2.85 (3)	$\text{S}_2'-\text{Mo}-\text{O}_1'$	87.1(10)
$\text{S}_2'-\text{S}_1'=\text{S}_2''-\text{S}_1''$	2.85 (1)	$\text{S}_2''-\text{Mo}-\text{O}_1''$	152.1(10)
$\text{S}_2''-\text{S}_1'=\text{S}_2'-\text{S}_1''$	3.51 (1)		

Table 5 gives the bond lengths and interbond angles for the bonds of the molybdenum atom. Two oxygens and four sulfurs coordinate the molybdenum atom forming a distorted octahedron (see Figs. 2 and 3). The oxygens form an edge in the polyhedron, *i.e.* they are in a *cis*-position. The two S_2 atoms form the opposing edge. The lengths of the edges in this octahedron vary considerably from $\text{O}_1-\text{O}_1=2.74(4)$ Å to $\text{S}_1-\text{S}_2=3.51(1)$ Å, due to the different ligand atoms. Consequently, the $\text{O}-\text{Mo}-\text{O}$ angle is less than 90° . The $\text{Mo}-\text{O}$ distance, $1.63(3)$ Å, is normal. For comparison the short $\text{Mo}-\text{O}$ distance in

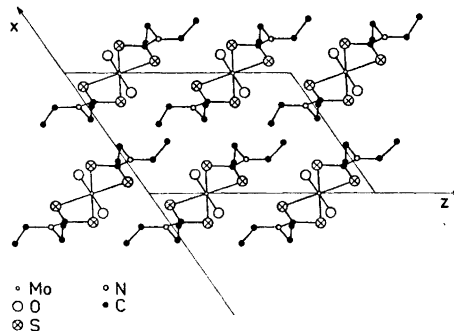


Fig. 1. The structure of $\text{MoO}_2[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$. Schematic drawing showing the xz projection.

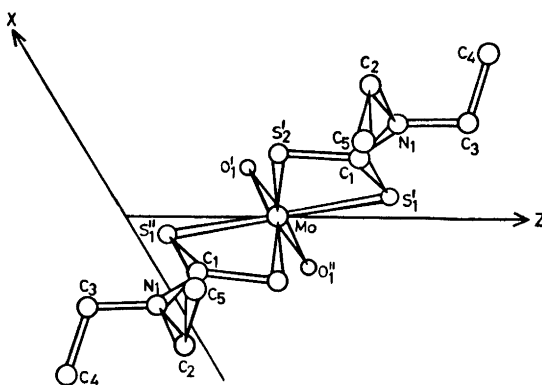


Fig. 2. A perspective view of the molecule along the y -axis. Bond lengths and bond angles are given in Table 5 and 6.

$\text{Mo}_2\text{O}_3(\text{C}_2\text{H}_5\text{OCS}_2)_4$ ¹⁰ is 1.65 Å. In MoO_3 ¹¹ the distances vary from 1.67 to 2.33 Å, and in $\text{K}_2\text{Mo}_3\text{O}_{10}$ ¹² the variation is 1.64–2.14 Å. The Mo–S distances are in the range between Mo–S₁ = 2.44(1) Å to Mo–S₂ = 2.63(1) Å. Worth noticing is, that Mo–S₁ is in a *cis*-position to Mo–O, while Mo–S₂ is in a *trans*-position. The effect of the strong Mo–O bond on S₂ gives a remarkably long Mo–S₂ distance. The variation in Mo–S distances slightly affect the S–C bonds in the NCS₂ group. A long Mo–S bond gives a shorter S–C bond and *vice versa*. A similar variation of bond lengths is found in the pentavalent molybdenum compound $\text{Mo}_2\text{O}_3(\text{C}_2\text{H}_5\text{OCS}_2)_4$ ¹⁰ but for $\text{Ni}((\text{C}_2\text{H}_5)_2\text{NCS}_2)_2$ ² where the metal has four coordinations, the variation for Ni–S is 2.19–2.20 Å.

The structure of the NCS₂ groups is in agreement with previous investigations of the Ni,² Cu,³ and Zn⁴ complexes. The sulfur-carbon bond lengths are, for S₁–C₁ = 1.75(3) Å and for S₂–C₁ = 1.68(3) Å (see Table 6, Figs. 2 and 3), which is the distance of a partial double bond. Previously reported values are S–C = 1.71–1.74 Å. The C₁–N bond = 1.29(5) Å has, as expected, a high double bond character. Bonamico *et al.*^{2–4} have reported C–N distances

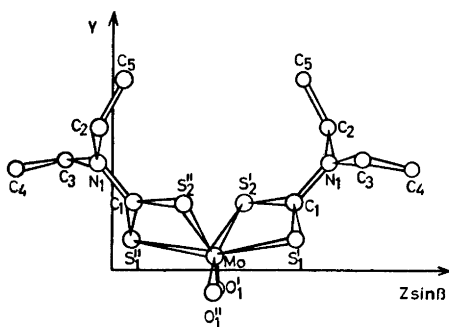


Fig. 3. A perspective view of the molecule along the x -axis. Bond lengths and bond angles are given in Tables 5 and 6.

Table 6. Bond lengths (Å) and bond angles (deg) within the molecule with standard deviations.

Bond lengths	Å	Bond angles	deg
Mo-2S ₁	2.443(8)	S ₁ -Mo-S ₂	68.5(3)
Mo-2S ₂	2.629(11)	Mo-S ₁ -C ₁	91.5(9)
Mo-2O ₁	1.634(25)	Mo-S ₂ -C ₁	87.0(12)
S ₁ -C ₁	1.75 (3)	S ₁ -C ₁ -S ₂	112.6(18)
S ₂ -C ₁	1.68 (3)	S ₁ -C ₁ -N ₁	119.2(21)
C ₁ -N ₁	1.29 (5)	S ₂ -C ₁ -N ₁	127.0(28)
N ₁ -C ₂	1.57 (5)	C ₁ -N ₁ -C ₂	116.1(26)
N ₁ -C ₃	1.47 (4)	C ₁ -N ₁ -C ₃	124.2(33)
C ₃ -C ₄	1.45 (5)	C ₂ -N ₁ -C ₃	119.0(29)
C ₂ -C ₅	1.58 (7)	N ₁ -C ₂ -C ₅	107.9(27)
		N ₁ -C ₃ -C ₅	113.7(27)

ranging from 1.31 to 1.35 Å. In the NCS₂ group the sum of the angles around the carbon atom is 360°, indicating no significant deviation from planarity for this group. Table 7 gives the deviations in Å from a least-squares plane through Mo, S₁, S₂, C₁, and N₁, where all atoms are given the same weight. It shows that all atoms, except for the CH₃ group, form a plane with good approximation.

The remaining intermolecular distances, *i.e.* the bond lengths in the ethyl group and the nitrogen bonding to the ethyl groups, are within standard deviations normal.

The *cis*-position of the oxygens were suggested by Moore and Larsen¹ from IR-spectra, which showed two bands at about 900 cm⁻¹. The large value of the dipole moment, 9.15 D, reported by Moore and Rice,¹³ was another piece of evidence. The dihedral angle between the two diethyldithiocarbamate groups (which are planar except for the CH₃ groups, see Table 8), coordinating the same molybdenum, is 71.2°, which explains the large dipole moment value.

IR-spectra of several dithiocarbamates (Chatt *et al.*¹⁴) show strong absorption bands in the 1500 cm⁻¹ region, which is interpreted as an indication of a strong C-N bond in the NCS₂ group. That is in agreement with the result obtained in this investigation.

Table 7. Departure in Å from the least-squares plane through the Mo, S₁, S₂, C₁, and N₁ atoms.

Atom	Departure in Å	Atom	Departure in Å
Mo	-0.12	C ₂	-0.24
S ₁	0.10	C ₃	-0.19
S ₂	0.08	C ₄	1.09
C ₁	0.09	C ₅	-1.77
N ₁	-0.15		

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