

In the structure of anhydrite there are two crystallographically distinct sulphate oxygen atoms. The sulphur–oxygen distances are practically identical, with a mean value of 1.475 Å. This should be compared with the mean S–O distance 1.473 Å in sulphate structures reported by Baur (1970) and also with 1.473 Å for an isolated sulphate anion (McGinnety, 1972) derived from the S–O bond lengths in K₂SO₄. In gypsum, CaSO₄·2H₂O (Cole & Lancucki, 1974), the S–O distances, 1.457 and 1.461 Å, are a little shorter than the present values. Ferraris & Catti (1973) have recently deduced an empirical relationship between predictive length (L) and bond strength (p) of the form:

$$(L - L_m)/L_m = k(p - 2)$$

where L_m is the mean bond length. For the S–O bonds in anhydrite, $k = 0.082$, $L_m = 1.473$ Å and $p = 2$ v.u. The predictive value is equal to the mean value of 1.473 Å.

The point symmetry of the sulphate ion in anhydrite is very close to sphenoidal $\bar{4}2m$ although the exact point symmetry belongs to $mm2$. The sulphate ion in anhydrite is more regular than that in gypsum. The lowering of the symmetry of the sulphate ion in gypsum is explained by the formation of hydrogen bonds (Cole & Lancucki, 1974).

The Ca atom in anhydrite is surrounded by eight oxygen atoms with an average distance of 2.468 Å, which can be compared with an average distance of 2.458 Å in gypsum. Four symmetry-related O(1) atoms make a planar trapezoid around the Ca atom and the

O(2) atoms also make a planar trapezoid normal to that of the O(1) atoms.

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Bis-(*N*-methylsalicylideneiminato)dioxomolybdenum(VI)

BY KOJI TSUKUMA,* TAKEMI KAWAGUCHI† AND TOKUNOSUKÉ WATANABÉ‡

Faculty of Science, Kwansei Gakuin University, Uegahara, Nishinomiya, Japan

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Abstract. C₁₆H₁₆MoN₂O₄, orthorhombic, $P2_12_12_1$, $a = 12.840(1)$, $b = 19.496(1)$, $c = 6.6346(5)$ Å, $V = 1661.02$ Å³, $Z = 4$, $D_m(\text{floatation}) = 1.602$, $D_x = 1.585$ g cm⁻³. The molybdenum atom is coordinated by two nitrogen and two oxygen atoms in chelate rings and two oxo (terminal) oxygen atoms, forming a distorted octa-

hedron. The two terminal oxygen atoms are *cis* to each other.

Introduction. Crystals made available to us by Professor S. Yamada (Yamanouchi & Yamada, 1974) are thin yellow parallelepipeds. The cell dimensions were determined by least-squares refinement of 20 and 30 2θ values respectively obtained from the $h0l$ and $0kl$ Weissenberg photographs. The crystal used has the dimensions 0.12 × 0.08 × 0.60 mm. 1824 independent non-zero reflexions ($0kl \sim 7kl$, $hk0 \sim hk4$) were collected on equi-inclination Weissenberg photographs taken

* Present address: Scientific and Industrial Research Institute, Osaka University, Suita, Osaka, Japan.

† Present address: Research division, Nippon Crucible Co. Ltd, 925 Inada, Higashi-Osaka 577, Japan.

‡ Present address: Kanazawa Medical University, Uchinada, Ishikawa-ken, Japan.

Table 1. *Bijvoet inequalities*

<i>h</i>	<i>k</i>	<i>l</i>	$ F_c(hkl) $	$ F_c(hkl) $	Obs.
1	4	3	39.26	38.83	<
1	8	2	42.66	49.31	<
1	9	2	41.97	36.15	>
1	15	2	30.83	40.99	<
1	16	3	31.95	33.76	<
2	10	3	30.73	26.85	>
2	14	4	18.61	21.50	<
2	15	4	54.39	50.54	>
2	5	4	42.76	47.48	<
3	1	2	12.91	18.00	<
3	3	3	36.70	41.91	<
4	1	2	39.43	33.48	>
9	2	3	37.43	39.07	<
11	1	2	47.51	46.73	>
13	4	3	34.78	34.01	>

with Cu $K\alpha$ radiation, and their intensities were estimated visually with a calibration wedge. Lorentz and polarization corrections were applied but no absorption

Table 3. *Bond distances (Å) and angles (°) around the molybdenum atom*

Figures in parentheses are standard deviations.			
Mo–O(11)	1.682 (8)	Mo–O(21)	1.688 (7)
Mo–O(12)	1.950 (8)	Mo–O(22)	1.979 (7)
Mo–N(11)	2.345 (8)	Mo–N(21)	2.337 (9)
O(11)–Mo–O(21)	107.7 (4)	O(11)–Mo–N(11)	86.6 (4)
O(11)–Mo–O(12)	98.1 (4)	O(22)–Mo–N(11)	83.5 (3)
O(21)–Mo–O(12)	96.0 (4)	O(12)–Mo–N(12)	81.9 (3)
O(21)–Mo–O(22)	95.7 (3)	O(13)–Mo–N(11)	80.7 (3)
O(11)–Mo–O(22)	95.6 (4)	O(22)–Mo–N(21)	80.2 (3)
O(21)–Mo–N(21)	89.2 (3)	N(11)–Mo–N(21)	76.5 (3)

Table 2. *Final positional and thermal parameters ($\times 10^4$) with standard deviations in parentheses*

$$B = \exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{23}kl + B_{31}lh)]$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{31}
Mo	−982 (1)	−1816 (1)	−1305 (1)	33 (1)	17 (1)	75 (1)	3 (1)	3 (1)	15 (2)
O(11)	156 (6)	−1468 (4)	−2037 (12)	37 (5)	23 (12)	112 (17)	−9 (6)	10 (11)	−8 (18)
O(12)	−1619 (7)	−992 (4)	−185 (13)	46 (5)	15 (2)	166 (20)	5 (6)	−12 (11)	−22 (19)
N(11)	−272 (7)	−1853 (5)	1950 (12)	55 (6)	18 (2)	67 (16)	9 (8)	16 (13)	−42 (19)
C(11)	−1262 (8)	−502 (5)	1051 (19)	33 (6)	20 (3)	170 (28)	5 (6)	−52 (16)	−17 (25)
C(12)	−1661 (9)	166 (6)	753 (19)	46 (7)	15 (3)	197 (32)	−1 (6)	1 (16)	35 (8)
C(13)	−1321 (11)	684 (7)	2010 (23)	68 (9)	24 (3)	221 (37)	10 (9)	−23 (19)	31 (32)
C(14)	−625 (11)	565 (6)	3529 (24)	70 (10)	27 (3)	227 (38)	−15 (9)	−62 (22)	70 (39)
C(15)	−266 (10)	−95 (6)	3870 (21)	58 (9)	23 (3)	148 (28)	−10 (9)	−31 (20)	−9 (35)
C(16)	−574 (9)	−630 (5)	2620 (17)	44 (7)	18 (3)	96 (23)	11 (7)	3 (14)	−28 (25)
C(17)	−169 (9)	−1329 (6)	3077 (16)	48 (7)	15 (3)	101 (22)	−6 (8)	1 (13)	−34 (24)
C(18)	958 (13)	−2512 (7)	2828 (20)	108 (13)	16 (3)	138 (29)	34 (11)	15 (17)	−124 (36)
O(21)	−1760 (6)	−1865 (5)	−3352 (11)	48 (5)	34 (3)	82 (17)	11 (7)	−6 (13)	−65 (17)
O(22)	−496 (5)	−2778 (4)	−1429 (13)	32 (4)	19 (2)	138 (18)	1 (5)	27 (12)	9 (20)
N(21)	−2285 (7)	−2346 (5)	599 (15)	24 (6)	23 (3)	149 (23)	−4 (6)	−23 (14)	19 (20)
C(21)	−1096 (9)	−3327 (5)	−1920 (16)	41 (7)	13 (2)	145 (24)	−1 (7)	−14 (12)	−37 (26)
C(22)	−615 (11)	−3826 (6)	−3188 (19)	51 (10)	19 (3)	164 (30)	7 (9)	−8 (16)	29 (29)
C(23)	−1199 (12)	−4400 (7)	−3707 (28)	74 (13)	35 (4)	257 (40)	−4 (11)	−89 (26)	52 (50)
C(24)	−2184 (13)	−4542 (7)	−2866 (25)	85 (12)	25 (4)	284 (45)	−18 (11)	−46 (23)	−10 (41)
C(25)	−2605 (10)	−4054 (7)	−1602 (28)	54 (8)	22 (4)	285 (50)	−17 (9)	25 (25)	−2 (40)
C(26)	−2057 (9)	−3447 (6)	−1083 (22)	48 (7)	19 (3)	173 (31)	−5 (8)	1 (18)	40 (33)
C(27)	−2578 (9)	−2979 (6)	339 (19)	30 (6)	19 (3)	200 (27)	1 (7)	−19 (16)	−32 (25)
C(28)	−2870 (10)	−1951 (7)	2090 (19)	53 (8)	32 (5)	180 (28)	0 (11)	−60 (20)	119 (29)

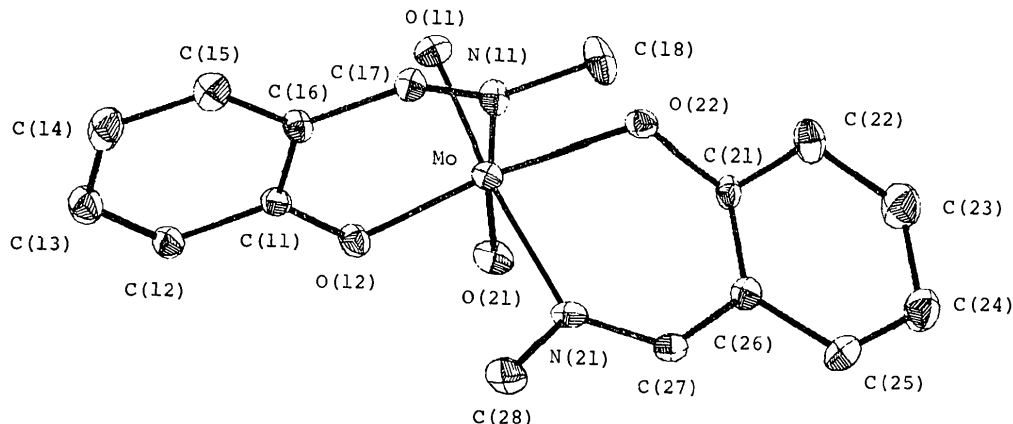
Fig. 1. A *DEAM* drawing of the title compound showing the atomic numbering and thermal ellipsoids.

Table 4. Bond distances (Å) and angles (°) in *N*-methylsalicylaldiminato groups

Figures in the parentheses are standard deviations.

O(12)–C(11)	1.339 (14)	O(22)–C(31)	1.358 (12)
N(11)–C(17)	1.274 (14)	N(21)–C(27)	1.302 (15)
N(11)–C(18)	1.488 (16)	N(21)–C(28)	1.462 (16)
C(11)–C(12)	1.413 (16)	C(21)–C(22)	1.427 (16)
C(12)–C(13)	1.380 (18)	C(22)–C(23)	1.390 (19)
C(13)–C(14)	1.366 (21)	C(23)–C(24)	1.411 (23)
C(14)–C(15)	1.386 (18)	C(24)–C(25)	1.378 (22)
C(15)–C(16)	1.390 (17)	C(25)–C(26)	1.419 (18)
C(16)–C(11)	1.389 (16)	C(26)–C(21)	1.373 (17)

suggested by Yamanouchi & Yamada (1974) from an infrared study is thus established. The two terminal Mo–O bonds, Mo–O(11) and Mo–O(21), are much shorter than the other Mo–O bonds in the same molecule, but their mean value, 1.684 Å, can be compared with corresponding values reported in other complexes containing the *cis*-MoO₂ group: 1.69 Å in K₂[{MoO₂(C₂O₄)(H₂O)}₂O] (Cotton, Morehouse & Wood, 1964), 1.65 Å in Mo₂O₃(S₂COC₂H₅)₄ (Blake, Cotton & Wood, 1964), 1.63 Å in MoO₂[(C₂H₅)₂NCS₂]₂ (Kopwille & Kierkegaard, 1969), 1.68 Å in

Table 4 (cont.)

Mo—N(11)–C(17)	123.8 (8)	Mo—N(21)–C(27)	123.7 (8)
Mo—N(11)–C(18)	120.7 (7)	Mo—N(21)–C(28)	120.1 (8)
C(18)–N(11)–C(17)	115.5 (9)	C(28)–N(21)–C(27)	116.1 (10)
N(11)–C(17)–C(16)	125.4 (10)	N(21)–C(27)–C(26)	122.7 (11)
C(17)–C(16)–C(15)	117.8 (10)	C(27)–C(26)–C(25)	116.5 (11)
C(17)–C(16)–C(11)	122.7 (10)	C(27)–C(26)–C(21)	124.2 (10)
C(11)–C(12)–C(13)	118.3 (11)	C(21)–C(22)–C(23)	117.5 (12)
C(12)–C(13)–C(14)	122.0 (13)	C(22)–C(23)–C(24)	122.9 (14)
C(13)–C(14)–C(15)	119.6 (13)	C(23)–C(24)–C(25)	117.2 (14)
C(14)–C(15)–C(16)	120.4 (11)	C(24)–C(25)–C(26)	121.9 (13)
C(15)–C(16)–C(11)	119.5 (10)	C(25)–C(26)–C(21)	119.3 (12)

corrections were made. Approximate coordinates of all the atoms except hydrogen were derived from a three-dimensional Patterson synthesis and a Fourier synthesis. The structure was refined by the block-diagonal approximation least-squares method. The absolute configuration of the molecular structure was determined by the Bijvoet relation. The dispersing atom is Mo for Cu *K*α, and the results are given in Table 1. The final atomic parameters listed in Table 2, expressed in terms of a right-handed set of axes, give the actual configuration of the molecule. The final *R* value was 0.07.*

Discussion. A perspective view of the molecular structure along the *a* axis is given in Fig. 1. The molecule contains two *N*-methylsalicylaldiminato groups and two terminal oxygen atoms, and the corresponding bond distances and angles in the two halves of the molecule are identical within the limits of significance, as can be seen in Tables 3 and 4. The *cis* configuration of the two terminal oxygen atoms, O(11) and O(21),

NH₄MoO₂F₃ (Atovmyan, Krasochka & Rahlin, 1971) The shortening of these terminal Mo–O bonds can be interpreted by the multiple π bonds between the molybdenum and oxygen atoms. Slight elongations of the Mo–N bonds are observed as compared with the corresponding Mo–N bonds in MoO₃.dien (Cotton & Elder, 1963) which may be caused by the higher multiplicity of the terminal Mo–O bonds in the *trans* position.

The two 'best' planes of the benzene rings make an angle of 75.4°. The nitrogen atoms show slight shifts from these planes [0.12 Å for N(11) and 0.25 Å for N(21)]. No intermolecular contacts corresponding to hydrogen bonds are observed.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30949 (20 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.