Note on the Crystal Structure of Dioxomolybdenum(VI)diethyldithiocarbamate

ANN KOPWILLEM and
PEDER KIERKEGAARD

Institute of Inorganic and Physical Chemistry, University of Stockholm, Stockholm, Sweden

The crystal structure of dioxomolybdenum(VI)diethyldithiocarbamate, $MoO_2((C_2H_5)_2NCS_2)_2$, has been determined from three-dimensional single-crystal X-ray film data. The coordinates of the nonhydrogen atoms derived from Patterson and Fourier syntheses were refined by least-squares techniques with isotropic temperature factors.

The following data were obtained for the monoclinic structure:

Unit cell dimensions (from a Guinier-Hägg powder photograph taken with $CuK\alpha_1$ radiation at 25°C): $a = 17.3839 \pm 14$ Å, $b = 8.6656 \pm 7$ Å, $c = 13.5907 \pm 12$ Å, $\beta = 124.66 \pm 0.01°$.

Cell content: 4 $MoO_2((C_2H_5)_2NCS_2)_2$ (observed density 1.65 g/cm³, calculated density 1.67 g/cm³).

Space group: $C2/c$ (No. 15).

Arrangements of atoms (*cf.* Table 1)

4 Mo in 4(*e*): $\pm(0, y, 1/4)$;
$\pm(1/2, 1/2+y, 1/4)$

8 S_1, 8 S_2, 8 O_1, 8 N_1, 8 C_1 to 8 C_5 in 8(*f*):
$\pm(x,y,z)$; $\pm(\bar{x},y,1/2-z)$;
$\pm(1/2+x,1/2+y,z)$;
$\pm(1/2-x,1/2+y,1/2-z)$

The discrepancy factor, R, is 12.9 % (calculated for all 551 reflections, $h0l-h4l$ and $hk0$, registered with $CuK\alpha$ radiation.

The crystals are built up of $MoO_2((C_2H_5)_2NCS_2)_2$ molecules formed by a MoO_2 group with the oxygens in *cis*-position and two $S_2CN(C_2H_5)_2$ groups. The two sulphur atoms of each of these groups are in contact with the molybdenum atom so that a distorted MoO_2S_4 octahedron is formed. No intermolecular contacts likely to correspond to hydrogen bonds are present in the structure so the molecules are hold together by van der Waals interactions.

Intramolecular distances and their standard deviations, σ, (in Å):

Mo	−2 S_1	2.443 ± 8
	−2 S_2	2.629 ± 11
	−2 O_1	1.634 ± 25
S_1	−C_1	1.75 ± 3
S_2	−C_1	1.68 ± 3
N_1	−C_1	1.29 ± 5
	−C_2	1.57 ± 5
	−C_3	1.47 ± 4
C_3	−C_4	1.45 ± 5
C_2	−C_5	1.58 ± 7

A schematic drawing showing a projection of the structure along the b axis is given in Fig. 1.

The *cis*-position of the oxygen atoms are in agreement with the conclusions by Moore and Larson[1] from IR data. Full details of this structure investigation and a discussion of the results will be given elsewhere.

Table 1. Atomic parameters and standard deviations obtained by full matrix least-squares refinement with isotropic temperature factors.

Atom	x	y	z	B (Å²)
Mo	0	0.0541 ± 5	1/4	0.9 ± 0.1
S_1	0.0489 ± 5	0.1300 ± 14	0.4509 ± 7	2.1 ± 0.2
S_2	0.1183 ± 5	0.2862 ± 14	0.3313 ± 7	2.2 ± 0.2
O_1	0.4190 ± 15	0.4514 ± 37	0.2451 ± 19	3.2 ± 0.5
N_1	0.3604 ± 17	0.1065 ± 43	0.4713 ± 23	2.6 ± 0.5
C_1	0.1145 ± 17	0.2845 ± 44	0.4521 ± 22	1.0 ± 0.5
C_2	0.1937 ± 22	0.4681 ± 56	0.0196 ± 29	3.0 ± 0.7
C_3	0.3659 ± 21	0.1155 ± 52	0.3676 ± 27	2.5 ± 0.6
C_4	0.2774 ± 25	0.1502 ± 61	0.2565 ± 33	3.7 ± 0.8
C_5	0.3764 ± 30	0.1714 ± 66	0.0409 ± 40	5.3 ± 1.1

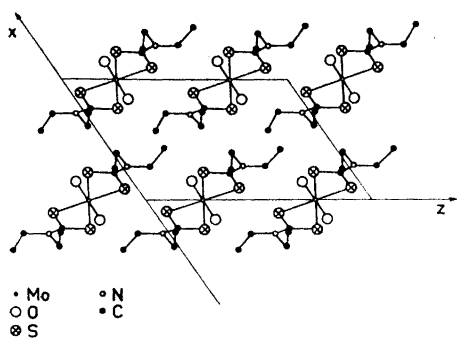

Fig. 1. The structure of $MoO_3((C_2H_5)_2NCS_2)_2$. Schematic drawing showing the *xz* projection.

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1. Moore, F. W. and Larson, M. L. *Inorg. Chem.* **6** (1967) 998.

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An Improved Preparation of Cellulose Layers for the Thin-layer Chromatography of Amino Acids

GUNNAR OGNER

The Forest Soil Fertilization Research Group, The Norwegian Forest Research Institute, Vollebekk, Norway

Among the several thin-layer systems described for the resolution of amino acids, one of the most useful is that described by Jones and Heathcote,[1] in which they demonstrate the two-dimensional resolution of 23 naturally occurring amino acids on cellulose layers. Their procedure has the advantage that both developing solvents are easily prepared single-phase mixtures.

According to our results, using the system referred to above, the separation of leucine and isoleucine is disturbed or even prevented by uneven solvent flow in the second dimension. This is caused by an undulating, yellow-coloured band of impurities which forms along the solvent front in the first dimension. As the movement of leucine and isoleucine is almost as fast as the solvent front, it is difficult to isolate the disturbing band of impurities by breaking the layer between it and the compounds in question. This problem can be solved by making the cellulose layers according to the following procedure.

To a mixture of 50 ml water and 40 ml ethanol is added 15 g of cellulose powder (MN 300), and the slurry stirred vigorously for about 2 min with an electric stirrer designed to exclude air bubbles. The slurry is immediately spread on five 20×20 cm glass plates, previously cleaned with detergent solution, and well rinsed, to a layer of thickness 0.30 mm using Shandon equipment. After air drying overnight the plates were placed in a chromarack and washed by developing them in the first solvent-mixture until the solvent front almost reached the top of the plates. After air drying for about 24 h the plates were ready for use. Treatment of the plates with steam after washing, for faster removal of the washing solvent-mixture, did not improve the separation in question. This method of preparation gives a very pure and extremely strong layer of cellulose which in dry condition is only about 0.05 mm thick.

Sample spots were applied in amounts of 1—10 µl from 0.05 % solutions in isopropanol to which had been added enough 1 N hydrochloric acid to dissolve the compounds. After developing in the first dimension with isopropanol:formic acid:water (40:2:10), (about 4 h for a 12 cm length of run at room temperature), the plates were air dried overnight. The thin yellow line indicating the solvent front was isolated from the chromatogram by scoring the layer just behind it. Development in the second dimension was carried out for about 3.5 h using the system *tert.*-butanol:2-butanone:0.9 1ammonia: wa-