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The unit cell and space group of potassium molybdenum dioxyoxalate, K[MoO₂(C₂O₄), H₂O].

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It has been suggested that the apparent failure of a number of molybdenum complexes to show a temperature-dependent paramagnetism, despite the presence of unpaired 4d-electrons, may be due to the existence of these compounds in the dimeric state (French & Garside, 1962). Since this suggestion could not be tested by measuring the molecular weight of the substance by orthodox chemical methods (French & Garside, 1962), an attempt has been made to find supporting evidence by X-ray diffraction.

Single crystals of the complex were grown by dissolving some of the powder in distilled water at 60 °C and cooling down the saturated solution to 20 °C over a period of 12 days. The single crystals were reddish brown, measured a few tenths of a millimetre across, and formed spherulite-type aggregates. Since the complex is known to crystallize with different amounts of crystal water (Spittle & Wardlaw, 1928), the carbon and hydrogen contents of the single crystals were checked by chemical microanalysis. The results agreed well with the chemical formula given above.

Weissenberg and precession X-ray photographs were taken from three selected single-crystal samples, mounted along the three crystallographic axes, using filtered copper radiation. The photographs showed monoclinic symmetry and from the observed systematic absences the space group was determined unambiguously as $P2_1/c$ with Z=4 formula units in the unit cell. The lattice parameters were determined by recording a few selected high-angle Bragg reflexions with the Weissenberg camera and superimposing on them the powder patterns of a gold and

an aluminum wire. Both wires were of 0.25 mm diameter and their (spectrographically determined) total metallic impurity content did not exceed 5 and 50 p.p.m. respectively. The lattice parameters obtained by interpolation using the two standard powder patterns were found to be:

$$a = 7.508 \pm 0.002$$
, $b = 14.235 \pm 0.003$, $c = 6.602 + 0.003$ Å; $\beta = 94^{\circ} 33' + 2'$.

The density calculated from the lattice parameters, $\varrho_c = 2 \cdot 57_9$ g.cm⁻³ agrees well with the measured density of the single crystals, $\varrho_m = 2 \cdot 58_9$ g.cm⁻³. The latter value was determined by the flotation method with a mixture of bromoform and ethyl iodide maintained at 20 °C.

Though the above results do not exclude the possibility of dimerization, they restrict it to the case where the two halves of the dimer are related to each other by a centre of symmetry coinciding with a centre of symmetry of the crystal.

No further work is contemplated on this compound.

The powder was kindly supplied by the late Dr C. M. French, Chemistry Dept., Q.M.C. and the microanalysis was carried out by Dr G. Weiler, Oxford.

References

French, C. M. & Garside, J. H. (1962). J. Chem. Soc. p. 2006.

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Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the General Secretary of the International Union of Crystallography (D. W. Smits, Mathematisch Instituut, University of Groningen, Reitdiepskade 4, Groningen, The Netherlands). Publication of an item in a particular issue cannot be guaranteed unless the draft is received 8 weeks before the date of publication.

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Crystallographic Data for Various Polymers. IV.

A complete revision of the table listing currently available crystallographic data for various crystallizable polymers* has been completed. Copies are available free of charge from the author who would be pleased to receive further data or references for inclusion in future revisions and to receive any comments or suggestions for its improvement. Address: R. L. Miller, Chemstrand Research Center, Inc., P. O. Box 731, Durham, North Carolina, U.S.A.

^{*} MILLER, R. L. & NIELSEN, L. E. (1960). J. Polymer Sci. 44, 391.

MILLER, R. L. & NIELSEN, L. E. (1961). J. Polymer Sci. 55, 643.