Stevens, 1986) and Fe^{II} (Figgis, Kucharski, Reynolds & Tasset, 1989) ammonium Tutton salts measured at helium temperatures by neutron diffraction. Those were precursors, as in this case, to the spin-density measurements by the polarized neutron-diffraction experiment (Deeth, Figgis, Forsyth, Kucharski & Reynolds, 1989; Figgis, Forsyth, Kucharski, Reynolds & Tasset, 1990; Fender *et al.*, 1986; Fender, Figgis & Forsyth, 1986*a*). The chromous Tutton salt structure has been determined at 295 and 84 K by X-ray diffraction (Figgis, Kucharski & Reynolds, 1990). The unit-cell dimensions and CrO_6 octahedron of this structure determination are virtually the same as those found in the 84 K X-ray structure. In the present structure, of course, the hydrogen positions are much better defined. There are sulfate and ammonium ions present, each close to a regular tetrahedron in shape [av. S—O = 1·480 (8) Å, av. O—S—O = 109·5 (12)°, av. N—D = 1·028 (3) Å, av. H—N—H 109·4 (17)°]. There are also $Cr(OD_2)_6^{2+}$ ions, but these show the expected Jahn–Teller distortion from regular octahedral geometry: Cr—O(8), 2·389 Å, is much longer than the other two Cr—O bonds, which average 2·068 (14) Å. There is a complex hydrogen-bonding network which differs only in detail from that seen in the other members of the series. The observation from the 84 K X-ray structure that D(13) is hydrogen bonded only to O(4), and not also to O(3) as it is in the other members in the series is confirmed.

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Structure of $GdMo_5O_8$

By P. Gougeon, P. Gall and M. Sergent

Laboratoire de Chimie Minérale B, Unité de Recherche Associée au CNRS nº 254, *Université de Rennes* I,
Avenue du Général Leclerc, 35042 *Rennes CEDEX*, *France*

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Abstract. Gadolinium pentamolybdate, $GdMo_5O_8$, $M_r = 764·95$, monoclinic, $P2_1/c$, $a = 7·559$ (3), $b = 8·9926$ (9), $c = 9·923$ (3) Å, $\beta = 109·81$ (2)°, $V = 634·6$ (3) Å^3, $Z = 4$, $D_x = 8·006$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 197·4$ cm^{-1}, $F(000) = 1352$, $T = 298$ K, $R = 0·028$ for 2098 observed reflections. The title compound is isostructural with $LaMo_5O_8$ and contains biococtahedral $Mo_{10}O_{18}$ cluster units strongly linked by Mo(1)—Mo(1) intercluster bonds [2·674 (1) Å] to form infinite molybdenum oxide chains. These are interconnected through Mo—O—Mo bonds to create channels where the Gd^{3+} ions are located. The Mo—Mo distances in the Mo_{10} cluster lie between 2·598 (1) and 2·832 (1) Å (average 2·746 Å). The Mo—O distances range between 1·994 (5) and 2·142 (5) Å (average 2·074 Å). The Gd^{3+} ions are ten coordinated to O atoms with Gd—O distances ranging from 2·315 (5) to 2·959 (5) Å (average 2·614 Å).

Experimental. Single crystals of $GdMo_5O_8$ were obtained by heating a cold-pressed pellet of a stoichiometric mixture of Gd_2O_3, MoO_3 and Mo in sealed molybdenum crucibles at about 2100 K for 5 min, cooling to 1273 K at a rate of 100 K h^{-1} and finally furnace cooling to room temperature.

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Table 1. *Positional parameters and equivalent isotropic thermal parameters*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
Gd	0·26917 (4)	0·50355 (4)	0·53030 (3)	0·531 (4)
Mo(1)	0·60399 (7)	−0·11587 (6)	0·48718 (5)	0·297 (7)
Mo(2)	0·38505 (7)	−0·11800 (6)	0·67761 (5)	0·287 (7)
Mo(3)	0·17146 (7)	−0·12714 (6)	0·38177 (5)	0·238 (7)
Mo(4)	0·80645 (7)	−0·13121 (6)	0·29839 (5)	0·223 (7)
Mo(5)	−0·00141 (7)	−0·12404 (6)	0·58669 (5)	0·240 (7)
O(1)	0·4016 (6)	0·0064 (6)	0·8516 (5)	0·53 (7)
O(2)	−0·0002 (6)	−0·2192 (5)	0·9342 (5)	0·40 (7)
O(3)	−0·0186 (6)	0·0031 (6)	0·2299 (5)	0·41 (7)
O(4)	0·3896 (6)	−0·2456 (6)	0·0134 (5)	0·48 (7)
O(5)	0·7962 (6)	−0·2388 (6)	0·1128 (5)	0·42 (7)
O(6)	0·2018 (6)	−0·2380 (6)	0·2084 (5)	0·61 (8)
O(7)	0·6091 (6)	−0·2302 (6)	0·8321 (5)	0·42 (7)
O(8)	0·3828 (6)	−0·0070 (6)	0·3276 (5)	0·40 (7)

Table 2. *Selected interatomic distances* (Å)

Mo(1)—Mo(2)	2·682 (1)	Mo(1)—O(5)	2·036 (5)
Mo(3)	2·802 (1)	O(7)	2·080 (5)
Mo(4)	2·795 (1)	O(8)	2·113 (5)
Mo(5)	2·807 (1)	O(8)	2·118 (5)
Mo(1)*	2·674 (1)	O(4)	2·129 (5)
Mo(2)*	2·904 (1)		
Mo(3)*	3·078 (1)	Mo(2)—O(6)	1·994 (5)
		O(1)	2·025 (5)
Mo(2)—Mo(3)	2·832 (1)	O(4)	2·050 (5)
Mo(4)	2·722 (1)	O(8)	2·100 (5)
Mo(5)	2·750 (1)	O(7)	2·116 (5)
Mo(3)—Mo(4)	2·598 (1)	Mo(3)—O(3)	2·059 (5)
Mo(5)	2·669 (1)	O(6)	2·067 (5)
Mo(5)	2·765 (1)	O(4)	2·068 (5)
		O(2)	2·079 (5)
Mo(4)—Mo(5)	2·735 (1)	O(8)	2·142 (5)
Mo(5)	2·757 (1)		
		Mo(4)—O(7)	2·056 (5)
Mo(5)—Mo(5)	2·822 (1)	O(5)	2·059 (5)
		O(3)	2·068 (5)
Gd—O(1)	2·315 (5)	O(1)	2·089 (5)
O(1)	2·364 (5)	O(2)	2·104 (5)
O(4)	2·387 (5)		
O(7)	2·506 (5)	Mo(5)—O(6)	2·025 (5)
O(5)	2·556 (5)	O(5)	2·049 (5)
O(3)	2·634 (5)	O(2)	2·070 (5)
O(2)	2·739 (5)	O(3)	2·081 (5)
O(8)	2·779 (5)		
O(6)	2·905 (5)		
O(2)	2·959 (5)		

* Intercluster distance.

A crystal fragment with dimensions 0·1 × 0·1 × 0·11 mm was selected for the intensity data collection and this was carried out on an Enraf–Nonius CAD-4 diffractometer equipped with graphite-monochromatized Mo *Kα* radiation. Accurate cell parameters were obtained by a least-squares refinement of the setting angles of 25 reflections with $7 \leq 2\theta \leq 24°$. 2704 reflections were recorded in the range $2 \leq 2\theta \leq 70°$ with h 0→12, k 0→14, l −16→16; ω–2θ mode was used with scan width $\Delta\omega = (1 + 0{\cdot}35\tan\theta)°$ and counter aperture $\Delta l = (2 + 0{\cdot}5\tan\theta)$ mm. Three orientation and three intensity control reflections were checked every 250 reflections and every hour, respectively, and showed no significant variation. Data were corrected for Lorentz–polarization and an empirical absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied to isotropically refined data. The minimum and maximum correction factors were 0·859 and 1·216. After averaging of symmetry-related reflections (R_{int} = 0·030), 2566 unique reflections of which 2098 had $I \geq 2\sigma(I)$ were used to solve the structure.

The atomic coordinates of $LaMo_5O_8$ (Hibble, Cheetham, Bogle, Wakerley & Cox, 1988) were used as initial values in the first stage of the structure refinement. Subsequently, isotropic and anisotropic thermal parameters for all atoms as well as the extinction coefficient were introduced in the full-matrix least-squares refinement on *F*. The final

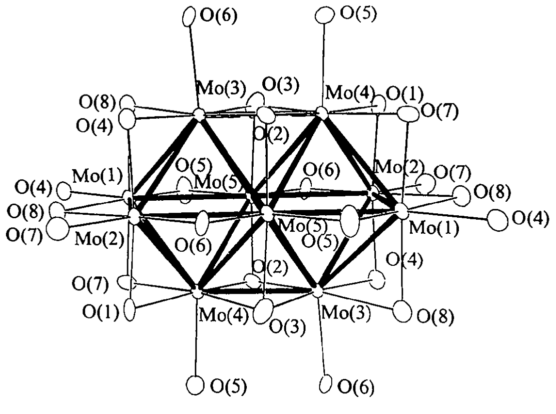

Fig. 1. The $Mo_{10}O_{18}$ cluster unit shown with its eight additional corner-linked O atoms. Inversion center at the middle of the Mo(5)—Mo(5) bonds.

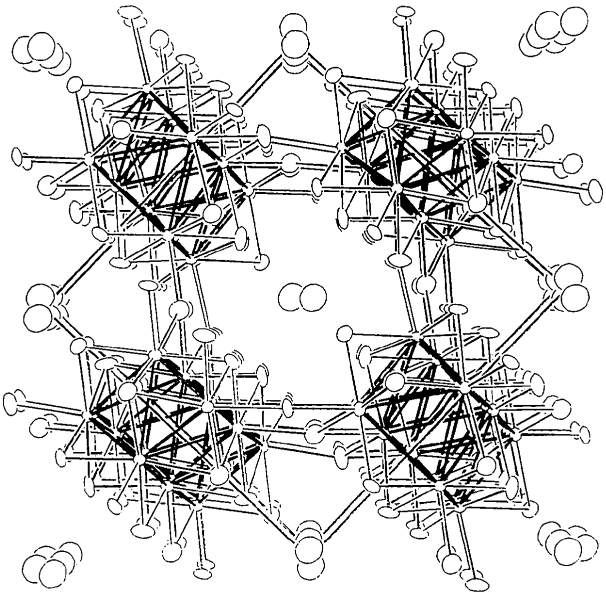

Fig. 2. View of the structure down the *a* axis. The unconnected open ellipsoids represent the Gd atoms.

values of R and wR were 0·028 and 0·037, respectively, where $w = 4F_o^2/[\sigma^2(F_o^2) + (0{\cdot}02F_o^2)^2]$ and $S = 0{\cdot}937$ using 2098 reflections with $I \geq 2\sigma(I)$ and 128 variables. The largest shift/e.s.d. was < 0·01 and the largest residuals on final difference map 1·85 and 2·46 e Å^{-3}; final value of $g = 5{\cdot}064 \times 10^{-7}$ (Stout & Jensen, 1968). Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV). All the calculations were performed on a PDP 11/60 with the *SDP* programs (B. A. Frenz & Associates, Inc., 1982). Final atomic coordinates and equivalent isotropic thermal parameters with their e.s.d.'s are given in Table 1* and selected interatomic distances in Table 2. Fig. 1 shows the Mo_{10} moiety with its oxygen environment and Fig. 2 the structure viewed down the a axis.

Related literature. The structure of the prototype compound $LaMo_5O_8$ was reported by Hibble *et al.* (1988) from X-ray and neutron powder diffraction data. More recently, single-crystal X-ray diffraction analyses were made on the isostructural phases $SnMo_5O_8$ (Gougeon, Potel & Sergent, 1990) and $PbMo_5O_8$ (Dronskowski & Simon, 1989). Analogous compounds are also formed with Ca, Sr, Ce, Pr, Nd, Sm and Eu as a ternary element (Gougeon, 1989).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53444 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of $YbMnO_3$

By M. Isobe, N. Kimizuka, M. Nakamura and T. Mohri

National Institute for Research in Inorganic Materials, Namiki 1-1, *Tsukuba, Ibaraki* 305, *Japan*

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Abstract. $M_r = 275{\cdot}98$, hexagonal, $P6_3cm$, $a = 6{\cdot}0433$ (1), $c = 11{\cdot}5575$ (5) Å, $V = 365{\cdot}54$ (2) Å^3, $Z = 6$, $D_x = 7{\cdot}52$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 42{\cdot}75$ mm^{-1}, $F(000) = 714$, $T = 295$ K, $R = 0{\cdot}036$, 742 unique observed reflections. The structure is of the $LuMnO_3$ type and described as a slightly distorted close packing of O atoms with Lu atoms in octahedral voids and Mn atoms in tetrahedral voids. The Yb atom is surrounded by seven and the Mn atom by five O atoms.

Experimental. The single crystals were grown through solid-state reaction between Yb_2O_3 and Mn_2O_3 powders in a Pt tube at 1823 K. For intensity measurements, a specimen was roughly ground into a sphere with average radius 0·06 mm. Intensity data were collected on an Enraf–Nonius CAD-4 single-crystal diffractometer with graphite-monochromated Mo $K\alpha$ radiation by the ω–2θ technique. The lattice parameters were derived from a least-squares fit of 25 reflections, in the range $41 < \theta < 43°$. Intensities of three standard reflections, monitored throughout the data collection, were constant within 3% of their respective mean intensities. A total of 1034 intensities were measured to $\theta_{\max} = 60°$, in the index range $0 \leq h(=k) \leq 12$, $0 \leq l \leq 28$. The intensities were corrected for Lorentz, polarization and absorption factors giving minimum and maximum transmission factors 0·036 and 0·109, respectively. The structure was refined by least squares by assuming isotropic secondary extinction. The calculation was inititated with the atomic parameters of $LuMnO_3$ given by Yakel, Koehler, Bertaut & Forrat (1963). Unit weight was given to all the observed reflections. The atomic scattering factors for neutral atoms and the real part of the dispersion correction factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final refinement on F values, based on 31 variables and 742 unique observations, with $I > 1{\cdot}5\sigma(I)$, resulted in $R = 0{\cdot}036$, $wR = 0{\cdot}053$, $S = 4{\cdot}3$, with Δ/σ not exceeding 0·01, including refinement of an isotropic extinction

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