Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ta1	0	0	0	0.0050 (4)
Ta2	0.37078 (8)	0.31212 (8)	0	0.0066 (3)
Sb3	0.29588 (13)	0.05161 (13)	0	0.0066 (4)

The crystal structure was solved by direct methods (*SHELXS*97; Sheldrick, 1990). The space groups *I*4, *I*$\bar{4}$ and *I*4/*m* were allowed based upon the observed systematic absences. The space group *I*4/*m* was selected for initial refinements, and this group was confirmed by comparing the refinement results using the other two groups, which were identical with *I*4/*m* within three s.u.'s. The Ta and Sb atoms were readily located from the *E* map and refined with anisotropic displacement parameters. The reflection (110) with $2\theta = 5.62°$ was omitted from the refinement due to the close proximity and possible interference from the beam stop of the X-ray diffractometer. The largest residuals in the final difference map were 4.49 e Å^{-3} at a distance of 1.82 Å from Ta1 and −4.60 e Å^{-3} on Ta1.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1990). Program(s) used to solve structure: *SHELXS*97. Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1226). Services for accessing these data are described at the back of the journal.

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Strontium tetramolybdate dihydrate, $SrMo_4O_{13}\cdot 2H_2O$

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Abstract

$SrMo_4O_{13}\cdot 2H_2O$ contains infinite sheets of distorted MoO_6 and MoO_5 moieties sharing edges and vertices. Inter-layer nine-coordinate Sr^{2+} cations [d_{av}(Sr—O) = 2.678 (3) Å] and water molecules complete the structure, which is isostructural with that of $BaMo_4O_{13}\cdot 2H_2O$.

Comment

$SrMo_4O_{13}\cdot 2H_2O$ (Fig. 1) is confirmed to be isostructural with $BaMo_4O_{13}\cdot 2H_2O$ (Harrison *et al.*, 1995). The polyhedral connectivity of the Mo/O groups results in buckled anionic sheets of stoichiometry $[Mo_4O_{13}]^{2-}$, which propagate normal to [100]. Both vertex-sharing and edge-sharing of the MoO_6 and MoO_5 groups occurs in this phase, which has essentially the same sheet structure as $BaMo_4O_{13}\cdot 2H_2O$.

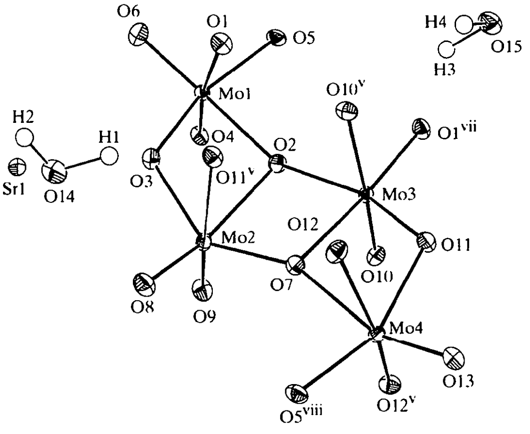

Fig. 1. Fragment of the $SrMo_4O_{13}\cdot 2H_2O$ structure, drawn with 50% displacement ellipsoids. The very long Mo2—O11^{v} bond is indicated by a thin line (see text). Symmetry codes are as in Table 1.

The Sr1 atom is nine-coordinate to O atoms [d_{av}(Sr1—O) = 2.678 (3) Å] in irregular coordination (Fig. 2). Four of these neighbours are the O atoms of

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water molecules (2 × O14 and 2 × O15). The bond valence sum (BVS) for Sr of 2.04 (expected: 2.00; Brown, 1996) indicates that this cation is well suited to its site in terms of satisfying its valence requirements, compared with the 'overbonded' Ba^{2+} cation (BVS = 2.36) in $BaMo_4O_{13}{\cdot}2H_2O$.

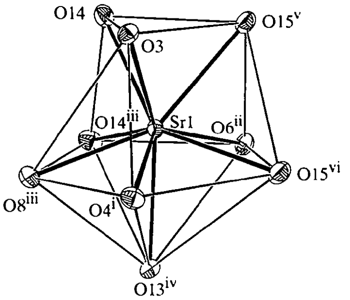

Fig. 2. Detail of the SrO_9 polyhedron in $SrMo_4O_{13}{\cdot}2H_2O$, with non-bonding O···O contacts shorter than 4 Å indicated by thin lines. Symmetry codes are as in Table 1.

The $Mo1O_6$, $Mo3O_6$, and $Mo4O_6$ octahedra in $SrMo_4O_{13}{\cdot}2H_2O$ are highly distorted, with the Mo atoms displaced from the best centres (Balić Žunić & Makovicky, 1996) of their octahedra by 0.42, 0.41, and 0.37 Å, for Mo1, Mo3, and Mo4, respectively. The Mo2 atom has a less common coordination of five near-neighbour O atoms, in an approximate square-based pyramidal arrangement; Mo2 is displaced by 0.40 Å towards O9 in the apical position from the best least-squares plane formed by O2, O3, O7, and O8. A very long contact to O11 [2.835 (3) Å], *trans* to O9, completes what could be regarded as a grossly distorted octahedron for this species. The corresponding Mo atom in $BaMo_4O_{13}{\cdot}2H_2O$ possesses an even longer sixth bond [Mo—O 2.949 (9) Å]. Bond valence sums for the molybdenum species [BVS(Mo1) = 6.01, BVS(Mo2) = 5.94, BVS(Mo3) = 6.03 and BVS(Mo4) = 6.10] are consistent with the sole presence of Mo^{VI} in $SrMo_4O_{13}{\cdot}2H_2O$.

The water molecules, O14 and O15, occupy the inter-layer region. Based on the riding model for the H atoms, each molecule makes two O—H···O hydrogen bonds, as O14—H1···O11 [H···O 1.81 and O···O 2.778 (5) Å], O14—H2···O9 [H···O 1.99 and O···O 2.809 (6) Å], O15—H3···O10 [H···O 2.19 and O···O 3.021 (5) Å] and O15—H4···O9 [H···O 1.89 and O···O 2.832 (5) Å]. These hydrogen bonds do not participate in any inter-sheet linkages. The acceptor O9 species is the apical atom of the $Mo2O_5$ square pyramid; O10 and O11 form Mo—O—Mo links. Both O14 and O15 bond to two distinct Sr^{2+} cations.

Experimental

Small plates and shards of $SrMo_4O_{13}{\cdot}2H_2O$ of poor to average visual quality were prepared from $SrCO_3$ (0.308 g), MoO_3 (0.60 g), SeO_2 (0.463 g) and H_2O (10 ml). These components were sealed in a 23 ml Teflon-lined hydrothermal bomb and heated to 453 K for 4 d, followed by cooling to ambient temperature over several hours. The solid product was recovered by vacuum filtration and washing with water.

Crystal data

$SrMo_4O_{13}{\cdot}2H_2O$
$M_r = 715.40$
Orthorhombic
Pbcn
$a = 19.3111\ (12)$ Å
$b = 7.5851\ (4)$ Å
$c = 15.5140\ (8)$ Å
$V = 2272.4\ (4)$ Å^3
$Z = 8$
$D_x = 4.18$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6651 reflections
$\theta = 1.0$–$27.5°$
$\mu = 9.04$ mm^{-1}
$T = 298$ K
Shard
0.090 × 0.005 × 0.004 mm
Colourless

Data collection

Siemens SMART area-detector diffractometer
Peak integration from area-detector frames (*SAINT*; Siemens, 1997)
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.494$, $T_{max} = 0.695$

24 914 measured reflections
2938 independent reflections
2742 reflections with $I > 0.5\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 26$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 21$
Intensity decay: none

Refinement

Refinement on F
$R = 0.031$
$wR = 0.022$
$S = 1.049$
2742 reflections
183 parameters
Only H-atom U's refined
Chebychev polynomial (Carruthers & Watkin, 1979) with 3 parameters 0.736, 0.980 and 0.461

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.08$ e Å^{-3}
$\Delta\rho_{min} = -0.93$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Sr1—O3	2.573 (4)	Mo2—O7	1.886 (3)
Sr1—$O4^{i}$	2.737 (4)	Mo2—O8	1.709 (4)
Sr1—$O6^{ii}$	2.669 (4)	Mo2—O9	1.710 (4)
Sr1—$O8^{iii}$	2.590 (4)	Mo2—$O11^{v}$	2.835 (3)
Sr1—$O13^{iv}$	2.714 (3)	Mo3—$O1^{vii}$	1.731 (3)
Sr1—O14	2.850 (4)	Mo3—O2	1.863 (3)
Sr1—$O14^{iii}$	2.611 (4)	Mo3—O7	2.198 (3)
Sr1—$O15^{v}$	2.777 (4)	Mo3—O10	1.741 (3)
Sr1—$O15^{vi}$	2.583 (4)	Mo3—$O10^{v}$	2.358 (3)
Mo1—O1	2.354 (3)	Mo3—O11	1.917 (3)
Mo1—O2	2.158 (3)	Mo4—$O5^{viii}$	1.875 (3)
Mo1—O3	2.010 (4)	Mo4—O7	2.129 (3)
Mo1—O4	1.700 (4)	Mo4—O11	2.034 (3)
Mo1—O5	1.891 (3)	Mo4—O12	1.740 (3)
Mo1—O6	1.722 (4)	Mo4—$O12^{v}$	2.230 (3)
Mo2—O2	2.237 (3)	Mo4—O13	1.698 (4)
Mo2—O3	1.915 (4)		
Mo1—O1—$Mo3^{v}$	139.4 (2)	Mo2—O7—Mo3	106.17 (15)
Mo1—O2—Mo2	97.31 (13)	Mo2—O7—Mo4	154.41 (19)

Mo1—O2—Mo3	156.37 (19)	Mo3—O7—Mo4	98.59 (14)
Mo2—O2—Mo3	105.45 (15)	Mo3—O10—Mo3^{vii}	135.17 (18)
Mo1—O3—Mo2	114.46 (18)	Mo3—O11—Mo4	112.23 (16)
Mo1—O5—Mo4^{ix}	148.4 (2)	Mo4—O12—Mo4^{vii}	145.74 (19)

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x, 1-y, -z$; (iii) $-x, y, \frac{1}{2}-z$; (iv) $x-\frac{1}{2}, \frac{1}{2}+y, \frac{1}{2}-z$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, z$; (vi) $x-\frac{1}{2}, \frac{1}{2}-y, -z$; (vii) $\frac{1}{2}-x, y-\frac{1}{2}, z$; (viii) $x, -y, \frac{1}{2}+z$; (ix) $x, -y, z-\frac{1}{2}$.

The highest difference peak is 0.45 Å from Sr1. Crystal decay was monitored by collecting duplicate frames at the end of the experiment, and was found to be negligible.

Data collection: *SMART* (Siemens, 1997). Cell refinement: *SMART*. Data reduction: *SMART*. Program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1997). Molecular graphics: *ORTEP*-3 (Farrugia, 1997). Software used to prepare material for publication: *CRYSTALS*.

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