

*Crystallography* (Hahn, 1983). Subsequent  $\Delta F$  synthesis yielded the positions of the O and H atoms. The positions of the latter could be refined isotropically without any constraints. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The program used was *SHELX76* (Sheldrick, 1978). Atomic parameters are given in Table 2,\* bond lengths and some important bond angles in Table 3. A projection plot of the isotypic gypsum-type structure is given elsewhere (Cole & Lancucki, 1974).

**Related literature.** A discussion of the structural relationships between some phases with gypsum-type structure like  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (Cole & Lancucki, 1974),  $\text{CaSeO}_4 \cdot 2\text{H}_2\text{O}$  (present work) and  $\text{Y}_{1-x}(\text{Gd}, \text{Dy}, \text{Er})_x\text{PO}_4 \cdot 2\text{H}_2\text{O}$  (Seidel, Sowa, Reithmayer, Schulz, Krüger & Abriel, 1991) will be published in due course (Krüger & Abriel, 1991).

Financial support by the Deutsche Forschungsgemeinschaft (SFB 173/A1) and the Fonds der Chemischen Industrie is gratefully acknowledged.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54045 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Bond lengths (Å) and bond angles (°)

Ca—O(W)	2 × 2.358 (2)	O(2)—O(1)	2.573 (2)
—O(2)	2 × 2.365 (2)	—O(2)	2.695 (2)
—O(2)	2 × 2.565 (2)	—O(2)	2.703 (2)
—O(1)	2 × 2.643 (1)	—O(1)	2.718 (2)
Se—O(2)	2 × 1.634 (1)	O(W)—H(2)	0.73 (4)
—O(1)	2 × 1.635 (2)	—H(1)	0.75 (2)
O(1)—O(2)	2.573 (2)	H(1)—H(2)	1.16 (4)
—O(2)	2.718 (2)	—O(1)	2.11 (2)
—O(1)	2.727 (2)	H(2)—O(1)	2.12 (4)
—O(W)	2.844 (2)		
—O(W)	2.846 (3)		
O(2)—Se—O(2)	111.59 (8)	H(2)—O(W)—H(1)	103 (3)
O(2)—Se—O(1)	2 × 112.49 (6)		
O(2)—Se—O(1)	2 × 103.79 (7)		
O(1)—Se—O(1)	112.96 (9)		

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*Acta Cryst.* (1991). **C47**, 1959–1961

## Structure of Tripotassium Hexahydrogenhexamolybdoaluminate(III) Heptahydrate

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(Received 28 January 1991; accepted 15 February 1991)

**Abstract.**  $\text{K}_3\text{H}_6[\text{AlMo}_6\text{O}_{24}] \cdot 7\text{H}_2\text{O}$ ,  $M_r = 1236.06$ , monoclinic,  $P2_1/a$ ,  $a = 11.392$  (1),  $b = 11.273$  (1),  $c = 22.354$  (2) Å,  $\beta = 94.69$  (1)°,  $V = 2861.0$  (8) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.87$  g cm<sup>-3</sup>, Mo  $K\alpha$  radiation (graphite monochromator,  $\lambda = 0.71069$  Å),  $\mu = 30.7$  m<sup>-1</sup>,  $F(000) = 2360$ ,  $T = 296$  K,  $R = 0.068$  ( $wR = 0.068$ ) for 3642 reflections with  $I_o > 4.00\sigma(I_o)$ . The  $\text{H}_6[\text{AlMo}_6\text{O}_{24}]^{3-}$  polyanion is a typical Anderson-type heteropolyanion. The Mo—O distances range

from 1.67 to 2.32 Å. The  $\text{AlO}_6$  octahedron is almost regular and mean Al—O bond distances range from 1.89 to 1.92 Å.

**Experimental.** Colorless, monoclinic crystals of  $\text{K}_3\text{H}_6[\text{AlMo}_6\text{O}_{24}] \cdot 7\text{H}_2\text{O}$  were obtained by mixing  $\text{AlCl}_3$  and  $\text{K}_2\text{MoO}_4$  solution in the molar ratio Al:Mo = 1:6, by adjusting the pH to 3–4 with dilute HCl solution (Hall, 1907; Baker, Foster, Tan,

Table 1. Positional and equivalent isotropic thermal parameters

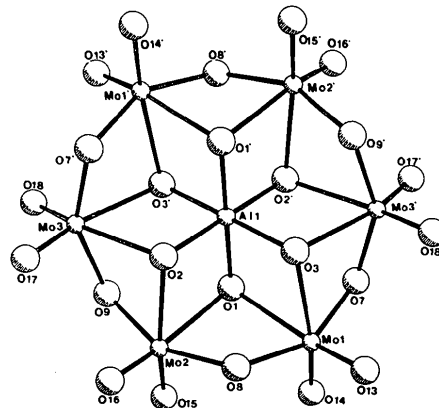
$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B <sub>eq</sub> (Å <sup>2</sup> )
Mo1	0.9285 (1)	0.2341 (1)	1.0807 (1)	1.61 (6)
Mo2	0.8942 (1)	0.2395 (1)	0.9317 (1)	1.56 (6)
Mo3	0.9686 (1)	0.0095 (1)	0.8522 (1)	1.64 (6)
Mo4	0.4775 (1)	0.5000 (1)	0.3514 (1)	1.73 (6)
Mo5	0.7206 (1)	0.4024 (1)	0.4263 (1)	1.67 (6)
Mo6	0.7439 (1)	0.4059 (1)	0.5748 (1)	1.63 (6)
K1	1.1323 (4)	0.4663 (5)	0.8366 (2)	3.8 (2)
K2	0.7315 (4)	0.1951 (4)	1.2073 (2)	3.3 (2)
K3	1.0440 (4)	0.3465 (4)	0.6457 (2)	3.0 (2)
Al1	1.00000	0	1.00000	1.3 (3)
Al2	1/2	1/2	1/2	1.6 (3)
O1	0.870 (1)	0.104 (1)	1.0041 (5)	1.3 (4)
O2	1.045 (1)	0.102 (1)	0.9387 (5)	1.0 (4)
O3	1.072 (1)	0.096 (1)	1.0639 (5)	1.3 (4)
O4	0.590 (1)	0.556 (1)	0.4380 (5)	1.3 (4)
O5	0.597 (1)	0.362 (1)	0.5007 (5)	1.4 (4)
O6	0.393 (1)	0.447 (1)	0.436 (1)	1.4 (4)
O7	0.903 (1)	0.097 (1)	1.130 (1)	1.8 (5)
O8	0.974 (1)	0.300 (1)	1.006 (1)	2.2 (5)
O9	0.846 (1)	0.112 (1)	0.879 (1)	2.4 (5)
O10	0.379 (1)	0.628 (1)	0.375 (1)	1.9 (5)
O11	0.583 (1)	0.369 (1)	0.374 (1)	2.2 (5)
O12	0.793 (1)	0.468 (1)	0.499 (1)	1.7 (5)
O13	1.019 (1)	0.315 (1)	1.127 (1)	2.7 (6)
O14	0.794 (1)	0.303 (1)	1.083 (1)	2.6 (6)
O15	0.759 (1)	0.305 (1)	0.934 (1)	2.6 (6)
O16	0.968 (1)	0.327 (1)	0.887 (1)	2.7 (6)
O17	1.040 (1)	0.108 (1)	0.810 (1)	2.4 (5)
O18	0.877 (1)	-0.071 (1)	0.806 (1)	2.0 (5)
O19	0.384 (1)	0.427 (1)	0.301 (1)	2.7 (6)
O20	0.564 (1)	0.585 (1)	0.311 (1)	2.6 (6)
O21	0.800 (1)	0.479 (1)	0.378 (1)	2.8 (6)
O22	0.779 (1)	0.266 (1)	0.429 (1)	3.1 (6)
O23	0.802 (1)	0.269 (1)	0.572 (1)	2.9 (6)
O24	0.839 (1)	0.487 (1)	0.621 (1)	2.6 (6)
Aq1	1.146 (1)	0.461 (1)	0.984 (1)	3.7 (7)
Aq2	1.038 (1)	0.358 (1)	0.515 (1)	2.7 (6)
Aq3	0.593 (1)	0.175 (1)	1.302 (1)	3.1 (6)
Aq4	1.130 (1)	0.622 (1)	0.742 (1)	4.1 (7)
Aq5	0.963 (1)	0.337 (1)	0.753 (1)	4.5 (8)
Aq6	1.227 (1)	0.331 (2)	0.737 (1)	4.6 (8)
Aq7	0.721 (2)	-0.057 (2)	1.198 (1)	7 (1)

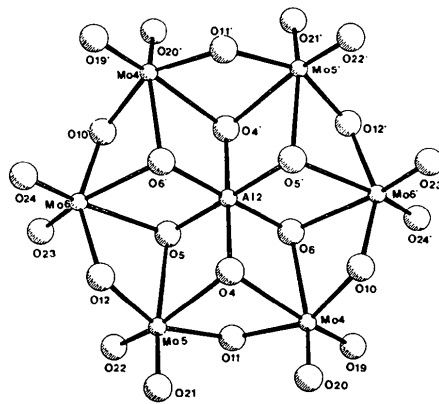
Table 2. Selected interatomic distances (Å)

Al1—Mo1	3.336 (2)	Al1—Mo2	3.282 (2)
Al1—Mo3	3.295 (2)	Al2—Mo4	3.310 (2)
Al2—Mo5	3.304 (2)	Al2—Mo6	3.298 (2)
Mo1—Mo2	3.321 (2)	Mo1—Mo3 <sup>i</sup>	3.299 (2)
Mo2—Mo3	3.293 (2)	Mo4—Mo5	3.306 (2)
Mo5—Mo6	3.307 (2)	Mo4—Mo6 <sup>ii</sup>	3.300 (2)
Mo1—O1	2.31 (1)	Mo1—O3	2.31 (1)
Mo2—O1	2.26 (1)	Mo2—O2	2.31 (1)
Mo3—O2	2.30 (1)	Mo3—O3 <sup>i</sup>	2.30 (1)
Mo4—O4	2.32 (1)	Mo4—O6	2.27 (1)
Mo5—O4	2.31 (1)	Mo5—O5	2.31 (1)
Mo6—O5	2.31 (1)	Mo6—O6 <sup>ii</sup>	2.28 (1)
Mo1—O7	1.93 (1)	Mo1—O8	1.94 (1)
Mo2—O8	1.94 (1)	Mo2—O9	1.91 (1)
Mo3—O7 <sup>i</sup>	1.90 (1)	Mo3—O9	1.95 (1)
Mo4—O10	1.92 (1)	Mo4—O11	1.95 (1)
Mo5—O11	1.91 (1)	Mo5—O12	1.91 (1)
Mo6—O10 <sup>ii</sup>	1.91 (1)	Mo6—O12	1.95 (1)
Mo1—O13	1.67 (1)	Mo1—O14	1.72 (1)
Mo2—O15	1.72 (1)	Mo2—O16	1.68 (1)
Mo3—O17	1.72 (1)	Mo3—O18	1.68 (1)
Mo4—O19	1.71 (1)	Mo4—O20	1.69 (1)
Mo5—O21	1.69 (1)	Mo5—O22	1.68 (1)
Mo6—O23	1.69 (1)	Mo6—O24	1.70 (1)
Al1—O1	1.90 (1)	Al1—O2	1.89 (1)
Al1—O3	1.92 (1)	Al2—O4	1.90 (1)
Al2—O5	1.91 (1)	Al2—O6	1.90 (1)

Symmetry operations: (i) 2 + x, y, 2 + z; (ii) 1 - x, 1 - y, 1 - z.



Poly anion A



Poly anion B

Fig. 1. Two poly anion structures in K<sub>3</sub>H<sub>6</sub>[AlMo<sub>6</sub>O<sub>24</sub>].7H<sub>2</sub>O. [Primes refer to the 2 + x, y, 2 + z (poly anion A) and 1 - x, 1 - y, 1 - z (poly anion B) symmetry operations.]

Scholnick & McCutcheon, 1954). A single crystal of dimensions 0.1 × 0.1 × 0.08 mm, Rigaku AFC-5R diffractometer, graphite monochromator, cell parameters refined by least-squares method on the basis of 25 independent indices (20 < 2θ < 25°), Mo Kα radiation (λ = 0.71069 Å); intensity measurement performed up to 2θ = 55.2° (0 ≤ h ≤ 14, 0 ≤ k ≤ 14, -28 ≤ l ≤ 28); ω-2θ scan mode, scan speed 8° min<sup>-1</sup>(θ); three standard reflections monitored every 150 reflections found to be constant; 7430 independent reflections measured, 3642 intensities with I<sub>o</sub> > 4.00σ(I<sub>o</sub>) used for the structure determination; correction for absorption (max. 1.00, min. 0.84); structure solved by Patterson and Fourier method, full-matrix least-squares refinement on F, H atom not located. All atoms refined anisotropically for a total of 374 parameters. Final refinement parameters R = 0.068, wR = 0.067, w<sup>-1</sup> = σ<sup>2</sup>(|F<sub>o</sub>| + (0.03|F<sub>o</sub>|)<sup>2</sup>), S = 2.68, (Δ/σ)<sub>max</sub> = 0.07, Δρ<sub>max</sub> = 2.18 e Å<sup>-3</sup>. All calculations performed using TEXSAN (Swepston, 1986) with scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are listed in

Table 1\* and interatomic distances are given in Table 2. An ORTEP (Johnson, 1976) drawing of the two crystallographically independent anions is shown in Fig. 1.

**Related literature.** X-ray structures of other *B*-type Anderson-structure heteropolyanions (heteroatom of low oxidation state, protonated polyanion) such as  $H_6[X^{n+}Mo_6O_{24}]^{(6-n)-}$  (*X*: heteroatom) have been reported:  $Cr^{3+}$  (Perloff, 1970),  $Cu^{2+}$  (Ito, Ozeki, Ichida & Sasaki, 1989) and  $Co^{3+}$  (Nagano, Lee, Ichida & Sasaki, 1990). *A*-type structures (heteroatom of high oxidation state, nonprotonated polyanion) such as  $[X^{n+}Mo_6O_{24}]^{(12-n)-}$  have been

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reported:  $Te^{5+}$  (Evans, 1974) and  $I^{7+}$  (Kondo, Kobayashi & Sasaki, 1980).

We thank the Korea Science and Engineering Foundation for financial support (891-0305-046-1).

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*Acta Cryst.* (1991). **C47**, 1961–1963

## Structure of Carbonyl(2-pyridinethiolato *N*-oxide- $\kappa$ O, $\kappa$ S)(triphenylphosphine)rhodium(I)

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(Received 17 December 1990; accepted 22 February 1991)

**Abstract.**  $[Rh(C_5H_4NOS)(CO)\{P(C_6H_5)_3\}]$ ,  $M_r = 519.4$ , triclinic,  $P\bar{1}$ ,  $a = 9.127$  (1),  $b = 9.371$  (1),  $c = 14.893$  (1) Å,  $\alpha = 85.665$  (6),  $\beta = 79.203$  (5),  $\gamma = 62.109$  (7)°,  $V = 1105.8$  (2) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.53$  (1),  $D_x = 1.56$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71073$  Å,  $\mu = 9.41$  cm<sup>-1</sup>,  $T = 295$  K,  $F(000) = 524$ , final  $R = 0.0299$  for 5841 observed reflections. The complex has square-planar geometry about the Rh atom. The S atom of the five-membered cyclic thiohydroxamate ring exerts a smaller *trans* influence on the Rh—P bond compared to the six-membered thioacetylacetonate ring in a similar complex [Botha, Basson & Leipoldt (1987). *Inorg. Chim. Acta*, **126**, 25–28]. Bond distances are Rh—O = 2.031 (2), Rh—S = 2.311 (1), Rh—P = 2.278 (1) and Rh—C = 1.800 (3) Å.

**Experimental.** The title complex was obtained by mixing equimolar amounts of  $PPh_3$  and  $[Rh(C_5H_4NOS)(CO)_2]$  in acetone. Suitable yellow needle-like crystals were obtained by slow evapor-

ation of an acetone solution at 263 K. The density was determined by flotation in sodium iodide solution. A crystal of size 0.25 × 0.25 × 0.45 mm was used for data collection on an Enraf-Nonius CAD-4F diffractometer with graphite-monochromatized Mo  $K\alpha$  radiation. The  $\omega/2\theta$ -scan technique was used with variable scan width  $\Delta\omega = (0.42 + 0.34\tan\theta)$ °, a scan speed maximum of 5.49° min<sup>-1</sup> in  $\omega$  and a maximum scan time of 60 s per reflection. The unit-cell parameters were determined with a least-squares fit of 25 reflections with  $23 < \theta < 24$ °. The intensity measuring range was  $3 < \theta < 30$ °. Empirical absorption correction (North, Phillips & Mathews, 1968) was made with minimum correction factor 0.95 and maximum 0.99. The mean intensity of three standard reflections, measured every 3600 s of X-ray exposure, varied from the initial value by -0.7%. All possible reflections with  $(\sin\theta)/\lambda < 0.71$  Å<sup>-1</sup> in the index ranges  $0 < h < 12$ ,  $-13 < k < 13$ ,  $-20 < l < 20$  gave 6226 unique reflections of which 5841 were considered observed with  $I >$