

Discussion. The framework of RbMoP_2O_7 is similar to those observed for KMoP_2O_7 and CsMoP_2O_7 . It is built up from corner-sharing MoO_6 octahedra and P_2O_7 groups (Fig. 1).

The $\text{Mo}^{\text{VI}}\text{O}_6$ octahedra are characterized by three short Mo—O distances and three longer ones (Table 2) as in other Mo^{VI} compounds such as $\text{MoP}_3\text{SiO}_{11}$ (Leclaire & Raveau, 1987), NaMoP_2O_7 (Leclaire *et al.*, 1988a), CsMoP_2O_7 (Lii & Haushalter, 1987), KMoP_2O_7 (Leclaire *et al.*, 1989), or $\text{Mo}_4\text{P}_6\text{Si}_2\text{O}_{25}$ (Leclaire, Lamire & Raveau, 1988).

The PO_4 tetrahedra present the features usually observed in the diphosphate groups: one long distance corresponding to the bridging oxygen of the P_2O_7 group and three shorter bonds corresponding to the O atoms shared with the MoO_6 octahedra (Table 2).

As in other alkaline molybdenum diphosphates, one observes $\text{MoP}_2\text{O}_{11}$ units where a diphosphate group shares two of its corners with the same MoO_6 octahedron. These units form layers with heptagonal windows (Fig. 1). The stacking along [001] of these layers alternately with their enantiophorph creates two

types of tunnels connecting cages where the Rb atoms are located and realize a tenfold coordination (Table 2).

References

- B. A. FRENZ & ASSOCIATES, INC. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.
- HOK-NAM, N. G. & CALVO, C. (1973). *Can. J. Chem.* **51**, 261–262.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- LECLAIRE, A., BOREL, M. M., GRANDIN, A. & RAVEAU, B. (1988a). *J. Solid State Chem.* **76**, 131–135.
- LECLAIRE, A., BOREL, M. M., GRANDIN, A. & RAVEAU, B. (1988b). *Z. Kristallogr.* **184**, 247–255.
- LECLAIRE, A., BOREL, M. M., GRANDIN, A. & RAVEAU, B. (1989). *J. Solid State Chem.* **78**, 220–226.
- LECLAIRE, A., LAMIRE, M. & RAVEAU, B. (1988). *Acta Cryst.* **C44**, 1181–1184.
- LECLAIRE, A. & RAVEAU, B. (1987). *J. Solid State Chem.* **71**, 283–290.
- LIU, K. H. & HAUSHALTER, R. (1987). *Acta Cryst.* **C43**, 2036–2038.
- RIOU, D., LABBÉ, PH. & GOREAUD, M. (1988). *Eur. J. Solid State Inorg. Chem.* **25**, 215–229.

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Structure of $\text{Mo}_8\text{Ga}_{41}\text{S}$

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Abstract. $M_r = 3658.1$, tetragonal, $I4/m$, $a = 12.861$ (3), $c = 5.284$ (1) Å, $V = 873.94$ Å³, $Z = 1$, $D_m = 6.96$ (1), $D_x = 6.950$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 353.46$ cm⁻¹, $F(000) = 1623$, $T = 298$ K, $R = 0.045$ for 1012 unique observed [$|F_o| > 3\sigma(F_o)$] reflections. The structure can be described by two types of coordination polyhedra: MoGa_{10} and $(\text{S,Ga})\text{Ga}_{12}$. The twofold position 0,0,0.5 is statistically occupied by S and Ga atoms.

Introduction. Two intermetallic phases exist in a Mo—Ga phase diagram: cubic Mo_3Ga and monoclinic $\text{Mo}_6\text{Ga}_{31}$ (Bornand, Siemens & Oden, 1973; Wood, Compton, Matthias & Corenzwit, 1958; Matthias, Wood, Corenzwit & Bala, 1956; Yvon, 1974). Gallium-rich alloys are, however, sensitive to the introduction of small amounts of elements from the oxygen group of the periodic table and the rhombohedral phase $\text{Mo}_8\text{Ga}_{41}$ reported by Yvon (1975) was shown by

Horyń & Andruszkiewicz (1985) to contain oxygen. The introduction of sulfur results in a new gallium-rich tetragonal phase (Andruszkiewicz & Horyń, 1988). Isomorphic phases containing selenium and tellurium have been reported by Horyń & Andruszkiewicz (1982).

This paper presents results of the structure analysis performed on a single crystal with nominal composition $\text{Mo}_8\text{Ga}_{41}\text{S}$.

Experimental. Molybdenum (purity 99.95%), gallium (99.999%) and sulfur (99.8%) taken as starting materials for single-crystal growing. A mixture of MoS_2 and Mo in the proportion 1:15 was placed as a pellet in the hot end of a boat containing gallium. Crystallization occurred in an evacuated and sealed-off quartz ampoule during 5 d. Single crystals were grown in a colder area of a boat at a temperature of 973 K, separated from gallium by dissolving the excess gallium

in a mixture of methanol and hydrochloric acid, and rinsed in methanol. Density measured by pycnometric method.

Black, metallic single crystals, polygonal-prism shaped, 11 side faces parallel to the *c* axis, an average base diameter of 0.08 mm and a height of 0.17 mm. Weissenberg photographs showed systematic absences $h + k + l \neq 2n$ and limited possible space groups to *I4/m*, $\bar{I}4$ and *I4*. Intensity data collected on Stoe-Siemens AED 2 diffractometer, graphite monochromator, lattice parameters determined by least squares from 43 automatically centered reflections, $15 < 2\theta < 69^\circ$; 4156 measured reflections, $3 < 2\theta < 70^\circ$, ω - θ scan mode, $-21 \leq h, k \leq 21, 0 \leq l \leq 9$; three standard reflections monitored every hour of exposure, random fluctuations during data collection $< 2\%$; analytical absorption correction, transmission factors between 0.01 and 0.07; 1041 unique reflections with $R_{\text{int}} = 0.047$, 29 unobserved with $|F_o| < 3\sigma(F_o)$, 1012 corrected for Lp used in the refinement. Structure solved by Patterson method; least-squares refinement in space group *I4/m* based on *F* with all atoms anisotropic and occupancy factors free; occupancy and temperature factors of S and Ga(5) atoms refined separately at an early stage of refinement in order to avoid correlation, full-matrix refinement at the final stage; $R = 0.045$, $wR = 0.059$, $w = 7.5827[\sigma^2(F_o) + 2.77 \times 10^{-4}|F_o|^2]^{-1}$, $\Delta_{\text{max}}/\sigma = 0.04$; the strongest correlations (correlation matrix elements greater than 0.5) occur between: $U_{11} = U_{22}$ of Ga(5) and occupancy of S (-0.55), $U_{11} = U_{22}$ and occupancy of Ga(5) (0.76), occupancy of S and Ga(5) (-0.76); final difference Fourier map peaks between -2.8 and $3.4 \text{ e } \text{\AA}^{-3}$, empirical secondary-extinction correction based on $F_c' = F_c(1 - 8.7 \times 10^{-8}F_c^2/\sin\theta)$; atomic scattering factors and f' , f'' values from *International Tables for X-ray Crystallography* (1974). Calculations performed with *SHELXS86* and *SHELX76* (Sheldrick, 1986, 1976), molecular plots obtained with *PLUTO* (Motherwell & Clegg, 1978).

Discussion. The projection of Mo₈Ga₄₁S along the *c* axis is shown in Fig. 1. The atomic parameters,

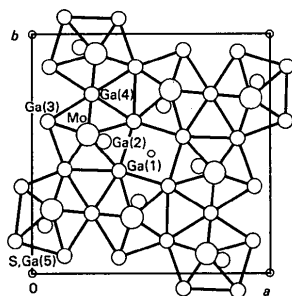


Fig. 1. The projection of Mo₈Ga₄₁S along the *c* axis. Bonds to Mo and [S,Ga(5)] atoms are not plotted.

Table 1. Atomic positions, occupancy and equivalent isotropic temperature factors (\AA^2)

Occupancy factor *p* modifies the structure factor according to the formula: $F(\mathbf{H}) = \sum_k p_k f_k \exp i 2\pi(\mathbf{r}_k \cdot \mathbf{H})$, summation over independent atoms. $U_{\text{eq}} = \frac{1}{3}(\text{trace of the } U_{ij} \text{ matrix})$.

	<i>x</i>	<i>y</i>	<i>z</i>	Occupancy	U_{eq}
Mo	0.7335 (0)	0.0805 (0)	0	1.009 (5)	0.0063 (2)
Ga(1)	0.1357 (1)	0.0727 (1)	0	1.000 (6)	0.0203 (5)
Ga(2)	0.5537 (1)	0.7006 (1)	0	0.988 (6)	0.0152 (4)
Ga(3)	0.0657 (0)	0.6364 (1)	0.2504 (1)	1.002 (4)	0.0154 (3)
Ga(4)	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	1.008 (6)	0.0164 (4)
S	0	0	0	0.43 (4)	0.070 (6)
Ga(5)	0	0	$\frac{1}{2}$	0.49 (2)	0.009 (1)

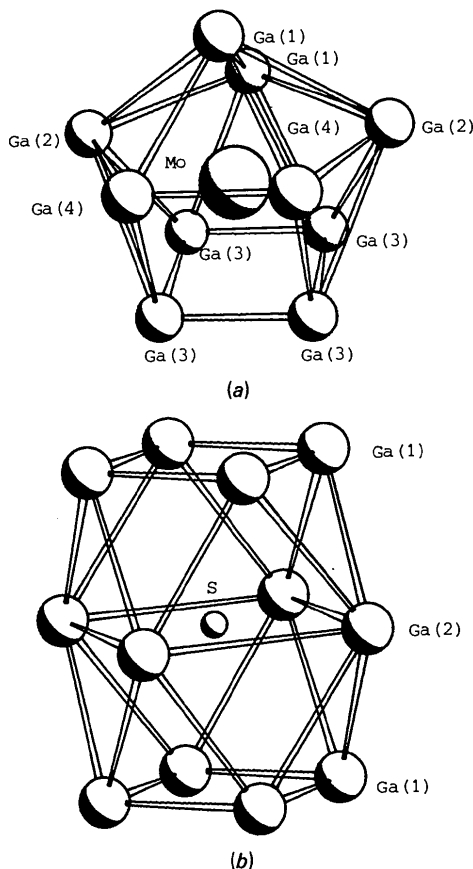


Fig. 2. (a) MoGa₁₀ coordination polyhedron. (b) (S,Ga)Ga₁₂ coordination cuboctahedron.

occupancy and temperature factors are collected in Table 1.*

In comparison with the work of Andruszkiewicz & Horyn (1988), who recognized a new Mo₈Ga₄₀S

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51702 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å)

MoGa ₁₀ polyhedron		(S,Ga)Ga ₁₂ cuboctahedron	
Mo—Ga(1)	2.591 (1)	S,Ga(5)—Ga(1)	3.302 (1)
Ga(2)	2.796 (1)	Ga(2)	2.671 (1)
Ga(3)	2.617 (1)	Ga(1)—Ga(1)	2.801 (1)
Ga(3)	2.629 (1)	Ga(2)	3.212 (1)
Ga(4)	2.558 (0)	Ga(2)	3.286 (1)
Ga(1)—Ga(1)	2.801 (1)		
Ga(2)	3.212 (1)		
Ga(2)	3.286 (1)		
Ga(3)	3.216 (1)		
Ga(4)	3.017 (1)		
Ga(2)—Ga(3)	2.914 (1)		
Ga(3)	3.451 (1)		
Ga(4)	2.920 (1)		
Ga(3)—Ga(3)	2.638 (1)		
Ga(3)	2.754 (1)		
Ga(4)	2.784 (1)		
Ga(4)—Ga(4)	2.642 (0)		

compound (*via* analysis of the Mo—Ga—S phase diagram at 1073 K), the present work presents single crystals of a compound richer in gallium by means of the partial occupation of a twofold site by one additional Ga atom, resulting in an increased density. Both the S and Ga(5) atoms are statistically disordered and have occupation factors a little smaller than 0.5. The occupancies of other atoms do not differ significantly from 1. They were refined, however, in order to check the possibility of the existence of defects at Mo and Ga sites, as was found in Mo₆Ga₃₁ and Mo₈Ga₄₁ (Yvon, 1974, 1975), but the refinement did not confirm such effects. Attempts to establish a model at lower symmetry, in which the S and Ga sites were ordered, were unsuccessful.

Like the V₈Ga₄₁-type structures (Girgis, Petter & Pupp, 1975; Yvon, 1975) Mo₈Ga₄₁S can be described by two types of coordination polyhedra: MoGa₁₀ (Fig. 2*a*) and (S,Ga)Ga₁₂ (Fig. 2*b*). The bond lengths (Table 2) within the MoGa₁₀ polyhedron are typical, whereas (S,Ga)—Ga distances in the (S,Ga)Ga₁₂ cuboctahedron correspond well to usual Ga—Ga bonds; there are,

however, longer than typical S—Ga bond lengths (Goodyear & Steigmann, 1963; Kuhn, Chevy & Chevalier, 1976), consistent with the high temperature factor of sulfur.

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References

- ANDRUSZKIEWICZ, R. & HORYŃ, R. (1988). *J. Less-Common Met.* **138**, 87–93.
- BORNAND, J. D., SIEMENS, R. E. & ODEN, L. L. (1973). *J. Less-Common Met.* **30**, 205–209.
- GIRGIS, K., PETTER, W. & PUPP, G. (1975). *Acta Cryst.* **B31**, 113–116.
- GOODYEAR, J. & STEIGMANN, G. A. (1963). *Acta Cryst.* **16**, 946–949.
- HORYŃ, R. & ANDRUSZKIEWICZ, R. (1982). *J. Appl. Cryst.* **15**, 248.
- HORYŃ, R. & ANDRUSZKIEWICZ, R. (1985). *J. Less-Common Met.* **114**, 355–359.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- KUHN, A., CHEVY, A. & CHEVALIER, R. (1976). *Acta Cryst.* **B32**, 983–984.
- MATTHIAS, B. T., WOOD, E. A., CORENZWIT, E. & BALA, B. (1956). *J. Phys. Chem. Solids*, **1**, 188–192.
- MOTHERWELL, W. D. S. & CLEGG, B. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1986). *SHELX86*. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.
- WOOD, E. A., COMPTON, V. B., MATTHIAS, B. T. & CORENZWIT, E. (1958). *Acta Cryst.* **11**, 604–606.
- YVON, K. (1974). *Acta Cryst.* **B30**, 853–861.
- YVON, K. (1975). *Acta Cryst.* **B31**, 117–120.

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(1,3-Diamino-2-propanol-*N,N'*)(malonato-*O,O'*)platinum(II) Dihydrate

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Abstract. [Pt(C₃H₇O₄)(C₃H₁₀N₂O)].2H₂O, $M_r = 423.2$, triclinic, *P*1, $a = 7.693$ (2), $b = 8.399$ (2), $c = 11.495$ (2) Å, $\alpha = 108.17$ (2), $\beta = 104.31$ (2), $\gamma = 116.67$ (2)°, $V = 561.2$ (2) Å³, $Z = 2$, $D_m = 2.50$, $D_x = 2.52$ (3) g cm⁻³, graphite-monochromatized Mo *K* α radiation, $\lambda = 0.71069$ Å, $\mu = 131.8$ cm⁻¹, $F(000) =$

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