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Structure of $Gd_4Mo_{18}O_{32}$

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Abstract. $M_r = 2867{\cdot}90$, monoclinic, $P2/c$, $a = 9{\cdot}847\,(4)$, $b = 5{\cdot}6635\,(7)$, $c = 24{\cdot}116\,(11)$ Å, $\beta = 109{\cdot}74\,(2)°$, $V = 1265{\cdot}9\,(8)$ Å^3, $Z = 2$, $D_x = 7{\cdot}524$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 188{\cdot}71$ cm^{-1}, $F(000) = 2536$, $T = 295$ K, $R = 0{\cdot}035$ for 2452 reflections with $I > 2\sigma(I)$. $Gd_4Mo_{18}O_{32}$ is isostructural with $Ca_{5\cdot45}Mo_{18}O_{32}$ and thus contains three kinds of infinite Mo chains: linear chains with alternate short and long Mo—Mo distances, chains of *trans*-edge-shared Mo_4 rhomboidal clusters and finally chains of *trans*-edge-shared Mo_6 octahedral clusters. While the Ca ions are delocalized in tunnels running along the *b* axis, the Gd ions show an order which induces a doubling of the *b* parameter and a changing of the space group from $C2/m$ to $P2/c$.

Introduction. The structures of the reduced molybdenum oxides are generally characterized by the formation of Mo—Mo bonds giving rise to molybdenum clusters of various sizes. Thus triangular Mo_3 clusters are observed in the compounds $M_2Mo_3O_8$ (M = Mg, Mn, Fe, Co, Ni, Zn, Cd) (McCarroll, Katz & Ward, 1957), $LiRMo_3O_8$ (R = Sc, Y, In, Sm, Gd, Tb, Dy, Ho, Er, Yb) (McCarroll, 1977), $LiZn_2Mo_3O_8$ (Torardi & McCarley, 1985), $ScZnMo_3O_8$ and $Zn_3Mo_3O_8$ (Torardi, Fecketter, McCarroll & Di Salvo, 1985) and $Na_2In_2Mo_5O_{16}$ (Collins, Fine, Potenza, Tsai & Greenblatt, 1989). Rhomboidal Mo_4 clusters occur in $Ba_{1\cdot14}Mo_8O_{16}$ (Torardi & McCarley, 1981) and $K_2Mo_8O_{16}$ (Torardi & Calabrese, 1984). Octahedral Mo_6 clusters as well as chain fragments of two, three, four and five *trans*-edge-shared Mo_6 octahedra are present in $Ca_{16\cdot5}Mo_{13\cdot5}O_{40}$ (Lindblom & Strandberg, 1989), MMo_5O_8 (M = Ca, Sr, Sn, Pb, La→Gd) (Hibble, Cheetham, Bogle, Wakerley & Cox, 1988; Gougeon, Potel & Sergent, 1990; Gougeon, Gall & Sergent, 1991), $Tl_{0\cdot8}Sn_{0\cdot6}Mo_7O_{11}$ (Dronskowski & Simon, 1989) and $In_{11}Mo_{40}O_{62}$ (Mattausch, Simon & Peters, 1986) respectively. In addition, Mo_8 clusters formed by capping two faces of an Mo_6 octahedron have been recently obtained in the MMo_8O_{14} (M = La, Ce, Pr and Nd) series (Leligny, Ledesert, Labbe, Raveau & McCarroll, 1990; Gougeon & McCarley, 1991). Among these clusters, the Mo_4 and Mo_6 can condense *via* edge sharing to form infinite chains which are observed in $Na_{0\cdot9}Mo_2O_4$ (McCarley, Lii, Edwards & Brough, 1985) for the former type and in $M_xMo_4O_6$ (M = Na, K, Rb, Sr, Ba, Sn, Pb, In) (Torardi & McCarley, 1979; McCarley, 1986), $Sc_{0\cdot75}Zn_{1\cdot25}Mo_4O_7$ (McCarley, 1982), $Mn_{1\cdot5}Mo_8O_{11}$ (Carlson, Brough, Edwards & McCarley, 1989), MMo_8O_{10} (M = Li, Zn) (Lii, McCarley, Kim & Jacobson, 1986) and $M_4Mo_4O_{11}$ (M = Y, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu) (Gougeon, Gall & McCarley, 1991) for the latter. Moreover, infinite linear chains of single Mo atoms occur typically in MoO_2 (Brant & Skapski, 1967) and $M_5Mo_2O_{12}$ (M = Y, Gd) (Torardi *et al.*, 1985). These three types of infinite chains are observed together in the unique compound $Ca_{5\cdot45}Mo_{18}O_{32}$ (McCarley *et al.*, 1985) in which they extend parallel to the *b* axis of the monoclinic unit cell. The Ca ions are delocalized in channels which parallel the chains. We present here the crystal structure of an isostructural compound, $Gd_4Mo_{18}O_{32}$, in which the Gd ions are ordered. This cationic ordering induces a doubling of the *b* parameter and a changing of the space group from $C2/m$ to $P2/c$.

Experimental. Single crystals were obtained by heating a stoichiometric mixture of Gd_2O_3, MoO_3 and Mo in a sealed molybdenum crucible at ~2170 K for 10 min, cooling it at 600 K h^{-1} to 1773 K, then at 100 K h^{-1} to 1273 K and finally furnace cooling to room temperature.

Crystal with dimensions 0·02 × 0·02 × 0·36 mm was selected for data collection on an Enraf–Nonius CAD-4 diffractometer equipped with graphite-monochromatized Mo $K\alpha$ radiation. Accurate cell

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parameters were obtained by a least-squares refinement of the setting angles of 25 reflections with $13 \leq 2\theta \leq 60°$. The systematic absence ($h0l$, $l = 2n + 1$) allows the space group to be $P2/c$ or Pc; the former was assumed and confirmed by the successful analysis. 4194 reflections were recorded in the range $2 \leq 2\theta \leq 60°$ with h 0→13, k 0→7, l −33→33; ω–2θ mode was used with scan width $\Delta\omega = (1{\cdot}20 + 0{\cdot}35\tan\theta)°$ 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* (Walker & Stuart, 1983) procedure was applied to isotropically refined data. Min. and max. correction factors were 0·841 and 1·271. After averaging ($R_{\rm int} = 0{\cdot}031$), 3236 unique data were used to solve the structure with the aid of *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and subsequent difference Fourier synthesis. The function minimized in a least-squares program was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/[\sigma^2(F_o) + (0{\cdot}015F_o)^2 + 0{\cdot}25]$ (Killean & Lawrence, 1969). The refinement of all Mo and Gd atoms anisotropically and O atoms isotropically along with the extinction coefficient (166 variables) converged to $R = 0{\cdot}035$, $wR = 0{\cdot}042$, $S = 0{\cdot}888$ for 2452 reflections with $F_o^2 > 2\sigma(F_o^2)$. The largest shift/e.s.d. was $< 0{\cdot}01$, max. and min. electron densities in final difference Fourier map were $+3{\cdot}54$ and $-2{\cdot}42$ e Å^{-3} respectively; the final value of g was $4{\cdot}08 \times 10^{-8}$ (Stout & Jensen, 1968). A refinement of the occupancy factors for the Gd sites confirmed that they are fully occupied. Scattering factors for neutral atoms and f', f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed with *SDP* (Frenz, 1985) programs on a Digital MicroVAX 3100. Final atomic coordinates and $B_{\rm eq}$ are reported in Table 1 and selected interatomic distances in Table 2.*

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

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

	x	y	z	$B_{\rm eq}$ (Å^2)
Gd(1)	−0·06768 (6)	0·3602 (1)	−0·13122 (2)	0·389 (9)
Gd(2)	−0·26577 (6)	0·1309 (1)	0·12978 (2)	0·410 (9)
Mo(1)	0·5663 (1)	0·3841 (2)	−0·16426 (4)	0·26 (2)
Mo(2)	0·5751 (1)	0·1563 (2)	0·33495 (4)	0·16 (2)
Mo(3)	0·3663 (1)	0·8862 (2)	0·25007 (4)	0·23 (2)
Mo(4)	0·3564 (1)	0·3870 (2)	0·25051 (4)	0·22 (2)
Mo(5)	0·3654 (1)	0·3600 (2)	0·00555 (4)	0·27 (2)
Mo(6)	−0·1426 (1)	0·1419 (2)	0·49503 (4)	0·28 (2)
Mo(7)	0·6365 (1)	0·1099 (2)	−0·01139 (4)	0·28 (2)
Mo(8)	−0·1481 (1)	0·3873 (2)	−0·00228 (4)	0·29 (2)
Mo(9)	0·000	0·3405 (3)	−0·250	0·55 (3)
Mo(10)	0·000	0·1137 (3)	0·250	0·50 (2)
O(1)	0·2642 (8)	0·387 (2)	0·1606 (3)	0·3 (1)*
O(2)	0·4132 (8)	0·386 (2)	0·3412 (3)	0·3 (1)*
O(3)	0·2806 (9)	0·124 (2)	0·6598 (4)	0·4 (1)*
O(4)	0·5698 (9)	0·107 (2)	0·6603 (4)	0·6 (1)*
O(5)	0·3542 (9)	0·383 (2)	0·5676 (4)	0·4 (1)*
O(6)	0·3462 (8)	0·141 (2)	0·0709 (4)	0·3 (1)*
O(7)	0·2100 (9)	0·125 (2)	0·2577 (4)	0·5 (1)*
O(8)	0·8035 (9)	0·355 (2)	0·7572 (4)	0·4 (1)*
O(9)	0·5902 (9)	0·365 (2)	0·0432 (4)	0·4 (1)*
O(10)	0·5904 (9)	0·111 (2)	0·5490 (4)	0·5 (1)*
O(11)	0·8345 (8)	0·371 (2)	0·5581 (3)	0·3 (1)*
O(12)	0·8361 (9)	0·120 (2)	0·0527 (4)	0·7 (1)*
O(13)	0·9294 (9)	0·386 (2)	0·4478 (4)	0·4 (1)*
O(14)	0·9254 (9)	0·131 (2)	0·9519 (4)	0·4 (1)*
O(15)	0·9413 (9)	0·111 (2)	0·6798 (4)	0·5 (1)*
O(16)	0·9489 (9)	0·386 (2)	0·1869 (4)	0·6 (1)*

* Refined isotropically.

Discussion. The crystal structure of $Gd_4Mo_{18}O_{32}$ contains three types of infinite molybdenum oxide chains, all of which run parallel to the b axis. The first one is composed of distorted edge-shared MoO_6 octahedra with alternating short and long Mo—Mo distances as in the distorted-rutile structure of MoO_2 (Fig. 1a). The second type consists of *trans* edge-shared Mo_4 rhomboidal clusters identical with those found in $NaMo_2O_4$ and can also be described as a double string of edge-shared MoO_6 octahedra, each sharing four edges with four adjacent octahedra (Fig. 1b). Moreover, two other edges are shared with neighbouring chains to form layers parallel to the ab plane. The third type of infinite chain consists of *trans*-edge-shared Mo_6 octahedra with O atoms above all free edges and corners and is similar to that observed in $NaMo_4O_6$ (Fig. 1c). The interconnection of these cluster chains shown in Fig. 2 is basically the same as described for the $Ca_{5{\cdot}45}Mo_{18}O_{32}$ compound. Thus, in the **a** direction the octahedral cluster chains are connected with the single Mo atom chains through O atoms to form layers in the ab plane. These layers are next linked with the neighbouring layers formed by the rhomboidal chains mainly through O—Gd—O bonding. The most obvious structural changes induced by the ordering of the cations between the two analogous compounds were the distortions which occur in the Mo_4 and Mo_6 chains owing to the doubling of their repeat unit. Thus, within the Mo_4 chains, the Mo(5)—Mo(7) and Mo(6)—Mo(8) bonds parallel to the b axis are alternately short [2·665 (2) and 2·668 (2) Å] and long [3·006 (2) and 2·999 (2) Å] while they are identical and equal to 2·8475 (7) Å in the Ca compound. In the same way, the unique transversal bond of 2·546 (2) Å in $Ca_{5{\cdot}45}Mo_{18}O_{32}$ gives rise to four different Mo—Mo bonds of 2·513 (2), 2·553 (2), 2·562 (2) and 2·588 (2) Å (mean value 2·554 Å). The Mo_6

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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 54222 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond distances* (Å)

Mo(1)—Mo(2)	2·605 (2)	Mo(1)—O(2)	2·030 (10)
Mo(1)—Mo(4)	2·703 (2)	Mo(1)—O(4)	2·084 (10)
Mo(1)—Mo(4)	2·767 (2)	Mo(1)—O(3)	2·085 (10)
Mo(1)—Mo(3)	2·783 (2)	Mo(1)—O(1)	2·090 (10)
Mo(1)—Mo(3)	2·827 (2)	Mo(1)—O(5)	2·193 (10)
Mo(1)—Mo(2)	3·062 (2)	Mo(2)—O(1)	2·029 (10)
		Mo(2)—O(4)	2·095 (11)
Mo(2)—Mo(4)	2·711 (2)	Mo(2)—O(2)	2·102 (10)
Mo(2)—Mo(4)	2·744 (2)	Mo(2)—O(3)	2·104 (11)
Mo(2)—Mo(3)	2·770 (2)	Mo(2)—O(6)	2·138 (9)
Mo(2)—Mo(3)	2·813 (2)	Mo(3)—O(4)	2·036 (10)
		Mo(3)—O(3)	2·053 (10)
Mo(3)—Mo(3)	2·634 (2)	Mo(3)—O(7)	2·101 (10)
Mo(3)—Mo(4)	2·829 (2)	Mo(3)—O(8)	2·119 (10)
Mo(3)—Mo(4)	2·838 (2)	Mo(4)—O(1)	2·048 (9)
		Mo(4)—O(2)	2·066 (9)
Mo(4)—Mo(4)	2·836 (2)	Mo(4)—O(8)	2·111 (10)
		Mo(4)—O(7)	2·116 (10)
Mo(5)—Mo(6)	2·513 (2)	Mo(5)—O(11)	2·048 (9)
Mo(5)—Mo(8)	2·553 (2)	Mo(5)—O(6)	2·064 (10)
Mo(5)—Mo(7)	2·665 (2)	Mo(5)—O(10)	2·071 (10)
Mo(5)—Mo(7)	3·006 (2)	Mo(5)—O(9)	2·086 (10)
Mo(5)—Mo(7)	3·168 (2)*	Mo(5)—O(9)	2·090 (9)
Mo(5)—Mo(5)	3·177 (2)*	Mo(5)—O(5)	2·119 (10)
		Mo(6)—O(13)	2·064 (10)
Mo(6)—Mo(7)	2·562 (2)	Mo(6)—O(11)	2·068 (10)
Mo(6)—Mo(8)	2·668 (2)	Mo(6)—O(14)	2·090 (9)
Mo(6)—Mo(8)	2·999 (2)	Mo(6)—O(12)	2·093 (11)
Mo(6)—Mo(8)	3·134 (2)*	Mo(6)—O(14)	2·097 (10)
Mo(6)—Mo(6)	3·174 (2)*	Mo(6)—O(6)	2·097 (9)
		Mo(7)—O(12)	2·050 (10)
Mo(7)—Mo(8)	2·588 (2)	Mo(7)—O(6)	2·068 (10)
Mo(7)—Mo(7)	3·175 (2)*	Mo(7)—O(5)	2·077 (10)
		Mo(7)—O(10)	2·084 (10)
Mo(8)—Mo(8)	3·152 (2)*	Mo(7)—O(9)	2·105 (10)
		Mo(7)—O(10)	2·109 (9)
Mo(9)—Mo(10)	2·572 (3)	Mo(8)—O(11)	2·049 (10)
Mo(9)—Mo(10)	3·091 (3)	Mo(8)—O(12)	2·052 (11)
		Mo(8)—O(13)	2·074 (10)
Gd(1)—O(1)	2·316 (10)	Mo(8)—O(14)	2·095 (10)
Gd(1)—O(11)	2·377 (9)	Mo(8)—O(5)	2·108 (10)
Gd(1)—O(3)	2·386 (10)	Mo(8)—O(13)	2·110 (9)
Gd(1)—O(13)	2·394 (10)	Mo(9)—O(8)	2·002 (9) (× 2)
Gd(1)—O(14)	2·407 (10)	Mo(9)—O(15)	2·056 (10) (× 2)
Gd(1)—O(15)	2·427 (10)	Mo(9)—O(16)	2·108 (11) (× 2)
Gd(1)—O(16)	2·508 (10)	Mo(10)—O(7)	2·014 (9) (× 2)
Gd(1)—O(8)	2·559 (9)	Mo(10)—O(15)	2·041 (11) (× 2)
		Mo(10)—O(16)	2·107 (11) (× 2)
Gd(2)—O(2)	2·317 (9)		
Gd(2)—O(12)	2·392 (10)		
Gd(2)—O(4)	2·407 (10)		
Gd(2)—O(10)	2·412 (10)		
Gd(2)—O(15)	2·415 (10)		
Gd(2)—O(9)	2·475 (10)		
Gd(2)—O(16)	2·548 (10)		
Gd(2)—O(7)	2·581 (10)		

* Interchain distance.

chains distort in the same manner as in $ZnMo_8O_{10}$ (Lii *et al.*, 1986) or $Gd_4Mo_4O_{11}$ (Gougeon *et al.*, 1991) and thus, they exhibit a marked pairing between the apical Mo(1) and Mo(2) atoms which results in alternately short and long distances of 2·605 (2) and 3·062 (2) Å between these atoms as well as between those in the shared edges of the octahedra [Mo(3)—Mo(3) 2·634 (2) and Mo(4)—Mo(4) 2·836 (2) Å]. Consequently, the repeat unit comprises two Mo_6 clusters instead of one in the Ca-containing compound. The average of the Mo—Mo distances within the Mo_8O_{16} repeat unit is 2·784 Å which is comparable to that observed in $Ca_{5·45}Mo_{18}O_{32}$ (2·785 Å) and a little shorter than those in compounds only containing infinite Mo_6 chains (*ca* 2·80 Å). The single Mo atom chains are quite similar in the two related compounds, but while they are formed of two independent Mo atoms in $Gd_4Mo_{18}O_{32}$, they result from the positional disorder of one Mo atom in $Ca_{5·45}Mo_{18}O_{32}$. The spacing between Mo atoms along the chain is alternately 2·572 (3) and 3·091 (3) Å [2·560 (9) and 3·135 (9) Å in $Ca_{5·45}Mo_{18}O_{32}$]. The shortest Mo—Mo interchain distance is 3·134 (2) Å and occurs between the Mo_4 chains. The remaining interchain distances are

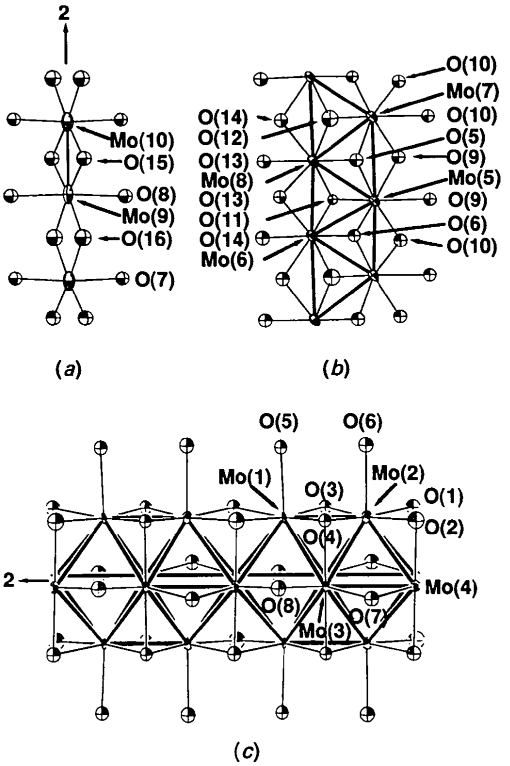

Fig. 1. The sections of the molybdenum-oxide cluster chains.

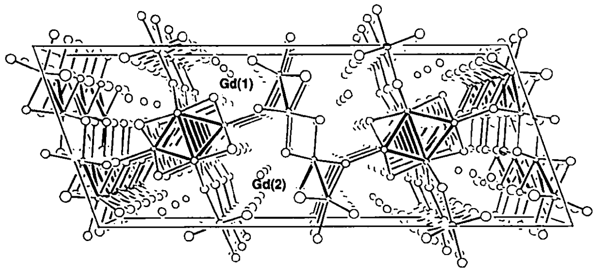

Fig. 2. The structure of $Gd_4Mo_{18}O_{32}$ as viewed down the *b* axis parallel to the direction of chain growth. Mo—Mo bonds in the cluster chains are shown by thick lines, Mo—O bonds by thin lines. The unconnected open ellipsoids represent the Gd cations.

greater than 3·80 Å and, consequently, are essentially non-bonding. The range of the Mo—O distances is 2·002 (9)–2·108 (11) (av. 2·055 Å), 2·048 (9)–2·119 (10) (av. 2·082 Å) and 2·030 (10)–2·193 (10) (av. 2·089 Å) in the linear, rhomboidal and octahedral Mo chains, respectively.

The two crystallographically inequivalent Gd cations are located in tunnels formed along the **b** direction. Their environments are similar and consist of eight O atoms placed at the vertices of a distorted bicapped trigonal prism. The average Gd—O distances are 2·421 and 2·443 Å for the Gd(1) and Gd(2) sites respectively. The shortest distance between Gd atoms is 4·294 (1) Å [Gd(1)—Gd(2)].

By using the bond-length–bond-strength formula (Brown & Wu, 1976) for the Mo—O bonds [$s = (d_{\rm Mo—O}/1{\cdot}882)^{-6{\cdot}0}$], the valence of each independent Mo atom was determined as follows: Mo(1) +2·63, Mo(2) +2·64, Mo(3) +2·21, Mo(4) +2·16, Mo(5) +3·28, Mo(6) +3·23, Mo(7) +3·26, Mo(8) +3·27, Mo(9) +3·55 and Mo(10) +3·55. From these values, we can deduce an average Mo oxidation state in the infinite Mo_2, Mo_4 and Mo_6 chains of +3·55, +3·26 and +2·41 respectively (+3·76, +3·38 and +2·34 in $Ca_{5{\cdot}45}Mo_{18}O_{32}$). The calculated valence of the molybdenum in $Gd_4Mo_{18}O_{32}$ is +2·91 which is in good agreement with that based on the stoichiometry, +2·89.

Isostructural compounds are also formed with Nd, Sm, Gd, Tb, Dy, Er, Tm, Yb, Lu and Y as the ternary element (Gougeon & McCarley, 1985).

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Mesitylbis[2-pyridinethiolato(1 −)]antimony(III)

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Abstract. [$Sb(C_{19}H_{19}N_2S_2)$], $M_r = 461{\cdot}24$, triclinic, $P\bar{1}$, $a = 8{\cdot}168\ (21)$, $b = 10{\cdot}015\ (8)$, $c = 12{\cdot}964\ (12)$ Å, $\alpha = 76{\cdot}74\ (8)$, $\beta = 85{\cdot}73\ (12)$, $\gamma = 69{\cdot}22\ (7)°$, $V = 965\ (3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}587$ Mg m^{-3}, λ(Ag $K\alpha$) = 0·56087 Å, $\mu = 0{\cdot}86$ mm^{-1}, $F(000) = 460$, $T = 291\ (1)$ K, final $R = 0{\cdot}040$ for 4227 unique observed

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