Crystal structure of tristrontium [tetranitridomolybdate(VI)], $\text{Sr}_3[\text{MoN}_4]$

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Discussion

The crystal structures of several ternary alkaline earth nitridomolybdates and -tungstates with $\text{A}_3[\text{MN}_4]$ composition are known up to now: the isotypes LT- $\text{Ba}_3[\text{MoN}_4]$ [1] and LT- $\text{Ba}_3[\text{WN}_4]$ [1], the isotypes HT- $\text{Ba}_3[\text{MoN}_4]$ [2, 3] and HT- $\text{Ba}_3[\text{WN}_4]$ [2, 4], and $\text{Ca}_2\text{Sr}[\text{WN}_4]$ [5]. Predominant structural features of these compounds are isolated complex tetrahedral nitridomolybdate and nitridotungstate anions $[\text{MN}_4]^{6-}$. The above mentioned crystal structures are related to the Na_3As -type structure with the complex anions in a hexagonal close packing arrangement. The tetrahedral holes are occupied by 2/3 of the alkaline earth cations, the remaining alkaline earth cations are in a trigonal planar coordination by the complex anions. The crystal structures of the isotypes $\text{Sr}_3[\text{MoN}_4]$ and $\text{Sr}_3[\text{WN}_4]$ [2] are also members of this structure family; the most obvious differences between the different crystal structures refer to the orientations of the complex anions and the resulting differences in nitrogen coordination polyhedra around the alkaline earth cations. Mo is tetrahedrally coordinated by N ($d(\text{Mo}-\text{N}) = 1.867 \text{ \AA}$), the coordination polyhedra of Sr are irregular (CN: 5–7; $d(\text{Sr}-\text{N}) = 2.757 \text{ \AA}$). Both values are in good agreement with data from literature: $d(\text{Mo}-\text{N}) = 1.867 \text{ \AA}$ (LT- $\text{Ba}_3[\text{MoN}_4]$ [1], $1.863 \text{ \AA}$ (HT- $\text{Ba}_3[\text{MoN}_4]$ [3]); $d(\text{Sr}-\text{N}) = 2.733 \text{ \AA}$ ($\text{Ca}_2\text{Sr}[\text{WN}_4]$ [5]).

Abstract

MoN_4Sr_3 , monoclinic, $C12/c1$ (No. 15), $a = 17.793(1) \text{ \AA}$, $b = 10.295(1) \text{ \AA}$, $c = 9.891(1) \text{ \AA}$, $\beta = 97.75(2)^\circ$, $V = 1795.3 \text{ \AA}^3$, $Z = 12$, $R_{\text{gt}}(F) = 0.045$, $wR_{\text{ref}}(F^2) = 0.112$, $T = 293 \text{ K}$.

Source of material

Yellow single crystals were obtained by reaction of pellets of Sr_2N and Mo (molar ratio 4:1). The reaction was carried out under N_2 at ambient pressure in Mo crucibles. The mixtures were heated to 1323 K (100 K/h). After a period of 36 h the products were cooled down (20 K/h) to ambient temperature. Starting materials and products are sensitive to moisture and air.

Table 1. Data collection and handling.

Crystal:	yellow, parallelepiped, size $0.4 \times 0.2 \times 0.1 \text{ mm}$
Wavelength:	Mo K_α radiation ($0.71070 \text{ \AA}$)
μ :	285.27 cm^{-1}
Diffractometer, scan mode:	Philips PW 1100, $\omega/2\theta$
$2\theta_{\text{max}}$:	59.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4440, 2169
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1993
$N(\text{param})_{\text{refined}}$:	111
Programs:	SHELXS-97 [6], SHELXL-97 [6], DIAMOND [7]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sr(1)	4e	1/2	0.4910(1)	1/4	0.0056(5)	0.0102(5)	0.0172(6)	0	0.0023(5)	0
Sr(2)	8f	0.02302(5)	0.33899(8)	0.08772(9)	0.0089(4)	0.0108(4)	0.0065(4)	−0.0023(3)	0.0017(3)	0.0002(3)
Sr(3)	8f	0.34665(5)	0.26625(7)	0.40018(8)	0.0063(4)	0.0069(3)	0.0057(4)	0.0002(3)	0.0009(3)	−0.0004(3)
Sr(4)	8f	0.32278(5)	0.37283(7)	0.04567(8)	0.0066(4)	0.0071(3)	0.0053(4)	0.0003(3)	0.0007(3)	0.0000(3)
Sr(5)	8f	0.18546(5)	0.46737(8)	0.29985(9)	0.0076(4)	0.0073(4)	0.0131(4)	−0.0004(3)	−0.0010(3)	0.0010(3)
Mo(1)	4e	1/2	0.16572(9)	1/4	0.0038(5)	0.0056(4)	0.0057(5)	0	0.0002(4)	0
Mo(2)	8f	0.17573(4)	0.14820(7)	0.28766(7)	0.0037(3)	0.0048(3)	0.0040(3)	0.0001(2)	0.0009(3)	−0.0001(2)

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Table 2. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	8f	0.2407(4)	0.0959(7)	0.4432(8)	0.005(3)	0.012(3)	0.007(3)	−0.002(3)	0.005(3)	0.003(3)
N(2)	8f	0.1399(5)	0.0031(7)	0.1907(8)	0.017(4)	0.008(3)	0.007(4)	−0.002(3)	0.001(3)	−0.005(3)
N(3)	8f	0.2336(5)	0.2478(7)	0.1836(8)	0.015(4)	0.009(3)	0.007(3)	−0.005(3)	0.007(3)	0.001(3)
N(4)	8f	0.0966(5)	0.2519(8)	0.3360(8)	0.011(4)	0.014(4)	0.013(4)	0.004(3)	−0.004(3)	−0.001(3)
N(5)	8f	0.4348(5)	0.0665(7)	0.3458(8)	0.017(4)	0.008(3)	0.014(4)	−0.002(3)	0.006(3)	0.002(3)
N(6)	8f	0.4491(5)	0.2789(7)	0.1239(8)	0.018(4)	0.011(4)	0.013(4)	0.006(3)	0.002(3)	−0.003(3)

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