



Soviet-era science, translated into English

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1965

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Abstract

Full Text

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MIXED MOLYBDENUM CHALCOGENIDES

(Presented by Academician I. V. Tananaev, January 16, 1965)

Molybdenum chalcogenides are represented by the following types of compounds: Mo_2X_3 , MoX_2 , Mo_2X_5 , and MoX_3 (where X is sulfur, selenium, or tellurium) (¹⁻⁴). The most stable compounds are the dichalcogenides. Their synthesis is usually carried out by the reaction of interaction between the elements (⁵⁻⁸). In addition, it is known that under certain conditions the interaction of a sesquichalcogenide with an excess of chalcogen leads to the formation of a dichalcogenide (⁹). This latter circumstance served as the basis for developing a method for the synthesis of new molybdenum compounds—mixed chalcogenides.

The present article describes methods of synthesis and X-ray studies of mixed molybdenum chalcogenides.

Experimental Part

As starting products we used molybdenum sesquisulfide, selenide, and telluride, as well as elemental sulfur, selenium, and tellurium. Molybdenum sesquisulfide was obtained by the method of direct synthesis from the elements (¹⁰) and was identified by X-ray diffraction.

This very simple and convenient procedure was used by us to develop a new method for the synthesis of molybdenum sesquiselenide and sesquitelluride, since the methods currently known (^{3, 4}) are very laborious and do not always ensure reliable results.

Thus, molybdenum sesquiselenide and sesquitelluride were also obtained by the method of direct synthesis from the elements. The substances obtained were identified by X-ray diffraction.

The conditions for the synthesis of molybdenum sesquichalcogenides were as follows:

Compound	Temperature	Time	Temperature	Time	Temperature	Time
Mo_2S_3^*	500°	6 hr	900°	14 hr	1400°	0.25 hr
$\text{Mo}_2\text{Se}_3^{**}$	700°	10 hr	1000°	15 hr	1450°	0.5 hr
$\text{Mo}_2\text{Te}_3^{**}$	700°	10 hr	1000°	15 hr	1450°	0.5 hr

In the case of molybdenum sesquisulfide, the X-ray data obtained proved identical with those known from the literature (¹⁰). The X-ray diffraction patterns of

molybdenum sesquitelluride obtained by the method we propose coincide with the X-ray diffraction patterns of Mo_2Te_3 obtained by Moretto's method⁽⁴⁾. Molybdenum sesquiselenide is isostructural with the sesquitelluride.

The synthesis of mixed molybdenum chalcogenides was carried out according to the reaction: $\text{Mo}_2\text{X}_3 + \text{X}' = \text{Mo}_2\text{X}_3\text{X}'$, where X and X' are different chalcogens.

The starting substances, in stoichiometric amounts with a total weighed portion of about 3–5 g, were placed in a quartz ampoule, which was connected to a vacuum system and evacuated to a residual pressure of $1 \cdot 10^{-5}$ mm Hg, after which the ampoule was sealed. The ampoule was immersed in a massive metal block (to improve heat exchange), which

* Synthesized according to data from⁽¹⁰⁾.

** Our data.

in turn, was placed in an electric furnace. The temperature was raised at a rate of 1.5–2.0 deg/min to 1000°, after which the substance was held at this temperature for 15 h.

The products obtained were slightly sintered gray crystalline substances with a strong metallic luster, which were readily ground to a powder.

By the method described, the following mixed molybdenum chalcogenides were synthesized for the first time: $\text{Mo}_2\text{S}_3\text{Se}$, $\text{Mo}_2\text{S}_3\text{Te}$, $\text{Mo}_2\text{Se}_3\text{S}$, $\text{Mo}_2\text{Se}_3\text{Te}$, $\text{Mo}_2\text{Te}_3\text{S}$, and $\text{Mo}_2\text{Te}_3\text{Se}$.

Table 1

Cell parameters of mixed molybdenum chalcogenides

	a, Å	c, Å	c/a		a, Å	c, Å	c/a
MoS_2	3.16	12.32	3.899	$\text{Mo}_2\text{Se}_3\text{Te}$	3.32	13.30	4.006
$\text{Mo}_2\text{S}_3\text{Se}$	3.22	12.50	3.882	MoTe_2	3.52	13.96	3.966
$\text{Mo}_2\text{S}_3\text{Te}$	3.25	13.10	4.031	$\text{Mo}_2\text{Te}_3\text{S}$	3.48	13.70	3.937
MoSe_2	3.29	12.90	3.921	$\text{Mo}_2\text{Te}_3\text{Se}$	3.50	13.82	3.949
$\text{Mo}_2\text{Se}_3\text{S}$	3.25	12.75	3.923				

X-ray diffraction study of powders of the substances obtained showed (Table 1) that the mixed molybdenum chalcogenides crystallize in the hexagonal system and are isostructural with the corresponding dichalcogenides. The small deviations in the lattice parameters are naturally explained by the increase in the size of the chalcogen in the series $\text{S} \rightarrow \text{Se} \rightarrow \text{Te}$.

Thus, partial replacement of one chalcogen by another in molybdenum dichalcogenide has proved possible, and such replacement does not lead to profound structural changes.

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Received
15 I 1965

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Note: Figure translations are in progress. See original paper for figures.

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