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Mechanochemical Reactions of Molybdenum and Tungsten Chalcohalides with $(Bu_4N)_2[Zn(Dmit)_2]$. Crystal Structure of $(Bu_4N)_2[Mo_3Se_{5.88}S_{1.12}(Dmit)_3] \cdot 1.15CH_2Cl_2$

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Abstract—The mechanochemical reactions of cluster chalcohalides $M_3Q_7X_4$ (M = Mo, W; Q = S, Se, Te; X = Br, I) with $(Bu_4N)_2[Zn(Dmit)_2]$ (Dmit is 1,3-dithia-2-thionedithiolate, $C_3S_5^{2-}$) are studied. It is shown that the reaction of $Mo_3Se_7Br_4$ with $(Bu_4N)_2[Zn(Dmit)_2]$ followed by crystallization from CH_2Cl_2 results in the formation of a new compound of the composition $(Bu_4N)_2[Mo_3Se_{5.88}S_{1.12}(Dmit)_3] \cdot 1.15CH_2Cl_2$ (**I**) characterized by IR spectroscopy, electrospray mass spectrometry, and X-ray structure analysis. Under similar conditions, $W_3Q_7Br_4$ and $Mo_3Te_7I_4$ form binuclear complexes $[M_2O_2Q_2(Dmit)_2]^{2-}$ identified by electrospray mass spectrometry. Various types of nonvalent contacts Q···Q are observed in the crystal structure of compound **I**.

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Interest in transition metal dithiolate complexes for a prolonged time is due to their importance in bioinorganic processes and design of molecular materials based on these complexes. In the recent time these complexes are intensively studied as blocks of molecular conductors with semiconducting, metallic, and even superconducting properties [1, 2].

The study of the molybdenum sulfide clusters with dithiolate ligands resulted in the discovery of the first one-component molecular conductors based on $[Mo_3S_7(dithiolate)_3]$ [3–5] synthesized by the two-electron oxidation of the corresponding anionic complexes $[Mo_3S_7(dithiolate)_3]^{2-}$. It is shown that specific nonvalent interactions S···S between the clusters play an important role in electron transport in these conductors. The selenide clusters $[Mo_3Se_7(dithiolate)_3]^{2-}$ synthesized in 2010 [6] are also precursors of molecular conductors. Unlike the sulfide analogs, the crystal structures of the selenium-containing clusters include nonvalent equatorial contacts Se(eq.)···Q that, along with the axial contacts Se(ax.)···Q, result in the formation of nonvalent-bonded agglomerates of different dimensionality.

In this work we systematically studied the mechanochemical reactions of cluster chalcohalides $M_3Q_7X_4$ (M = Mo, W; Q = S, Se, Te; X = Br, I) with $(Bu_4N)_2[Zn(Dmit)_2]$ (Dmit is isotrithionedithiolate, $C_3S_5^{2-}$), According to the data of electrospray mass spectrometry, these reactions most frequently result in the formation of the known binuclear complexes $[M_2O_2Q_2(Dmit)_2]^{2-}$. Only the reaction of $Mo_3Se_7Br_4$ with $(Bu_4N)_2[Zn(Dmit)_2]$ afforded a new trinuclear dithiolate complex $[Mo_3Se_{5.88}S_{1.12}(Dmit)_3]^{2-}$ containing the cluster core mixed in chalcogen in which each molybdenum atom is coordinated by the isotrithionedithiolate ligand through the bidentate mode. The complex was isolated as a tetrabutylammonium salt $(Bu_4N)_2[Mo_3Se_{5.88}S_{1.12}(Dmit)_3] \cdot 1.15CH_2Cl_2$ (**I**) characterized by IR spectroscopy, electrospray mass spectrometry, and X-ray structure analysis.

EXPERIMENTAL

Chalcohalides $M_3Q_7X_4$ (M = Mo, W; Q = S, Se, Te) were synthesized using the known procedures by the interaction of stoichiometric amounts of metal or metal chalcogenide, chalcogen, and halogen in an evacuated glass ampule at a specified temperature [7, 8]. Compound $(Bu_4N)_2[Zn(Dmit)_2]$ was obtained by the reduction of carbon disulfide with sodium in dimethylformamide according to a described procedure [9]. The solvents were purified by standard procedures.

Vibrational spectra in the range from 4000 to 400 cm^{-1} were recorded in KBr pellets on a Scimitar FTS 2000 instrument with a resolution of 1 cm^{-1}. The electrospray mass spectrum (ESI-MS) was recorded on a Quattro LC mass spectrometer (quadrupole–hexapole–quadrupole; Micromass, Manchester, UK) [10].

The mechanochemical reactions were carried out in a vibrational mill, whose structure was described in detail [11]. A titanium cylindrical reactor (volume 100 mL, height 50 mm) and balls of tungsten carbide 10 mm in diameter (weight 300 g) were used. The degree of filling of the reactor with balls was 65%. Mechanical activation was carried out subjecting the reactor to vibration during 12 h with a frequency of 25 Hz and an amplitude of 1 cm.

Synthesis of compound I. After the mechanochemical activation of a mixture of $Mo_3Se_7Br_4$ (1.0 g, 0.9 mmol) and $(Bu_4N)_2[Zn(Dmit)_2]$ (1.0 g, 1.1 mmol), the reaction product was extracted with CH_2Cl_2 (30 mL) and filtered off. After complete evaporation, the solid product was washed with a large amount of methanol to remove $(Bu_4N)_2[Zn(Dmit)_2]$ (stirring several times for many hours) and ether. Single crystals of compound **I** were obtained as a solvate with methylene chloride by the diffusion of ether to a solution of CH_2Cl_2. The yield of **I** · $1.15CH_2Cl_2$ was 0.38 g (23%).

IR (ν, cm^{-1}): 2955 w, 2868 w, 1477 w, 1447 m ν(C=C); 1372 w, 1298 w, 1169 w, 1051 s ν(C=S); 1019 c ν(C–S); 895 m, 736 w, 562 w, 516 m, 466 m, 445 w.

Mass spectrum (CH_2Cl_2; *m/z*): $[Mo_3Se_7(C_3S_5)_3]^{2-}$ (715), $[Mo_3Se_6S(C_3S_5)_3]^{2-}$ (691), $[Mo_3Se_5S_2(C_3S_5)_3]^{2-}$ (668).

X-ray structure analysis was carried out according to a standard procedure on a Bruker-Nonius X8Apex automated four-circle diffractometer equipped with a two-coordinate CCD detector (MoK_α radiation, $\lambda = 0.71073$ Å, graphite monochromator). Reflection intensities were measured in the φ- and ω scan modes for narrow (0.5°) frames to $2\theta = 55°$ at $T = 150$ K. An absorption correction was empirically applied using the SADABS program [12]. The structure was solved by a direct method and refined by full-matrix least squares for F^2 in the anisotropic approximation for non-hydrogen atoms using the SHELXTL program package [13]. Hydrogen atoms were placed in the geometrically calculated positions and refined in the rigid body approximation. The sulfur content in the positions of selenium in the cluster core was refined in the isotropic model at fixed thermal parameters. A sulfur admixture was found only in the equatorial positions in which the sulfur content was refined as 0.20, 0.50, and 0.42 for the positions S(2), S(4), and S(6), respectively. The obtained values of multiplicities of filling the positions of sulfur and selenium were fixed and refined in the anisotropic approximation with the equalized coordinates and thermal parameters. We failed to separate the positions of sulfur and selenium. The solvate molecules of methylene chloride partially occupy their positions. One of the molecules is disordered over two similar positions. The crystallographic characteristics of complex **I** and details of diffraction experiment are given in Table 1. The geometric parameters of the cluster core are listed in Table 2. The coordinates of atoms and thermal parameters were deposited with the Cambridge Crystallographic Data Centre (no. 865209; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Table 1. Main crystallographic characteristics and details of X-ray structure experiment for compound **I**

Empirical formula	$C_9Mo_3S_{16.12}Se_{5.88} \cdot 2(C_{16}H_{36}N) \cdot 1.15(CH_2Cl_2)$
FW	1959.58
Temperature, K	150.0(2)
Crystal system	Triclinic
Space group	$P\bar{1}$
a, Å	12.7889 (4)
b, Å	17.5464 (9)
c, Å	18.9053 (14)
α, deg	115.045 (2)
β, deg	95.952 (2)
γ, deg	106.534 (1)
V, $Å^3$	3559.7 (3)
Z	2
ρ_{calcd}, g/cm^3	1.828
μ, mm^{-1}	4.12
F(000)	1928
Crystal size, mm	0.23 × 0.08 × 0.03
θ Range, deg	1.12–27.5
Reflection index ranges	$-15 \le h \le 14$, $-22 \le k \le 22$, $-24 \le l \le 24$
Measured reflections	32237
Independent reflections	15544 ($R_{int} = 0.060$)
Reflections with $I \ge 2\sigma(I)$	8363
Number of refined parameters	686
Goodness-of-fit	0.875
R_1 ($I > 2\sigma(I)$)	0.0273
wR_2 (all reflections)	0.0534
Residual electron density (min/max), *e* $Å^{-3}$	1.99/–1.95

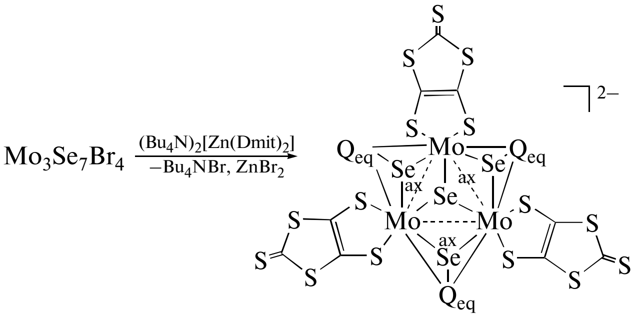
RESULTS AND DISCUSSION

Mechanochemical reactions represent a convenient method for the synthesis of molybdenum and tungsten clusters with the island structure of one-dimensional coordination polymers $M_3Q_7X_4$ (M = Mo, W; Q = S, Se, Te) [14]. The halide [15], oxalate [16], acetylacetonate [17], and dithiophosphate complexes [18] containing the cluster core $\{M_3Q_7\}^{4+}$ were thus obtained. We studied the mechanochemical reactions of $M_3Q_7X_4$ with $(Bu_4N)_2[Zn(Dmit)_2]$ in order to obtain new triangular complexes with the dithiolate

Table 2. Selected geometric parameters of the cluster core $\{Mo_3Se_4Q_3\}^{4+}$ in compound **I**

Bond	*d*, Å	Bond	*d*, Å
Mo(1)–Se(1)	2.5045 (15)	Mo(3)–Se(7)	2.5472 (13)
Mo(1)–Q(2)	2.6356 (15)	Q(2)–Se(3)	2.3041 (16)
Mo(1)–Se(3)	2.5548 (12)	Q(4)–Se(5)	2.2384 (18)
Mo(1)–Q(4)	2.5907 (17)	Q(6)–Se(7)	2.2456 (18)
Mo(1)–Se(5)	2.5513 (13)	Mo(1)–Mo(2)	2.7993 (11)
Mo(2)–Se(1)	2.5018 (14)	Mo(1)–Mo(3)	2.8123 (12)
Mo(2)–Q(4)	2.5893 (19)	Mo(2)–Mo(3)	2.7924 (12)
Mo(2)–Se(5)	2.5350 (14)	Mo(1)–S(11)	2.470 (3)
Mo(2)–Q(6)	2.6084 (18)	Mo(1)–S(12)	2.548 (3)
Mo(2)–Se(7)	2.5459 (13)	Mo(2)–S(21)	2.453 (3)
Mo(3)–Se(1)	2.4872 (14)	Mo(2)–S(22)	2.510 (3)
Mo(3)–Q(2)	2.6162 (14)	Mo(3)–S(31)	2.452 (3)
Mo(3)–Se(3)	2.5342 (13)	Mo(3)–S(32)	2.523 (3)
Mo(3)–Q(6)	2.6271 (16)		
Angle	**ω, deg**	**Angle**	**ω, deg**
Mo(2)Mo(1)Mo(3)	59.69 (3)	Mo(3)Q(2)Mo(1)	64.75 (4)
Mo(3)Mo(2)Mo(1)	60.39 (3)	Mo(3)Se(3)Mo(1)	67.09 (4)
Mo(2)Mo(3)Mo(1)	59.93 (3)	Mo(2)Q(4)Mo(1)	65.42 (5)
Mo(2)Se(1)Mo(1)	67.99 (4)	Mo(2)Se(5)Mo(1)	66.78 (4)
Mo(3)Se(1)Mo(1)	68.58 (4)	Mo(2)Q(6)Mo(3)	64.47 (4)
Mo(3)Se(1)Mo(2)	68.07 (4)	Mo(2)Se(7)Mo(3)	66.50 (4)

ligands. The mechanochemical reaction between $Mo_3Se_7Br_4$ and $(Bu_4N)_2[Zn(Dmit)_2]$ affords the triangle complex $[Mo_3Se_{5.88}S_{1.12}(Dmit)_3]^{2-}$ crystallized from a methylene chloride solution as tetrabutylammonium salt **I**



According to the X-ray structure analysis data for compound **I**, the partial substitution of the selenium atoms in the equatorial positions for sulfur atoms is observed in the cluster anion. The composition observed indicates the cocrystallization of various forms differed only in the content of selenium and sulfur, namely, $[Mo_3Se_7(C_3S_5)_3]^{2-}$, $Mo_3Se_6S(C_3S_5)_3]^{2-}$, $[Mo_3Se_5S_2(C_3S_5)_3]^{2-}$, and $[Mo_3Se_4S_3(C_3S_5)_3]^{2-}$, which are indiscernible from the viewpoint of X-ray structure analysis. According to the data of mass spectrometry, a solution of compound **I** in methylene chloride gives signals of three of the above listed dianionic forms: $[Mo_3Se_7(C_3S_5)_3]^{2-}$, $[Mo_3Se_6S(C_3S_5)_3]^{2-}$, and $[Mo_3Se_5S_2(C_3S_5)_3]^{2-}$. Both the starting $(Bu_4N)_2[Zn(Dmit)_2]$ remained in the reaction mixture after the end of the reaction and the reaction products can serve as sources of sulfur for the substitution of selenium. We have earlier described the individual selenide complex $[Mo_3Se_7(Dmit)_3]^{2-}$ synthesized by the reaction of $[Mo_3Se_7Br_6]^{2-}$ with $(Bu_4N)_2[Zn(Dmit)_2]$ at ambient temperature in a solution of CH_2Cl_2 [6]. The reaction course was monitored by electrospray mass spectrometry. However, it turned out that the subsequent crystallization of $(Bu_4N)_2[Mo_3Se_7(Dmit)_3]$ from solution results in the formation of $(Bu_4N)_2[Mo_3Se_{6.1}S_{0.9}(Dmit)_3]$ in which the partial substitution of selenium for sulfur was observed by X-ray structure analysis. Thus, the isotrithionedithiolate ion (similarly to $NCSe^-$ and PPh_3Se) can act as a source of sulfur (selenium) in the substitution of the selenium atom in the equatorial position of the cluster core.

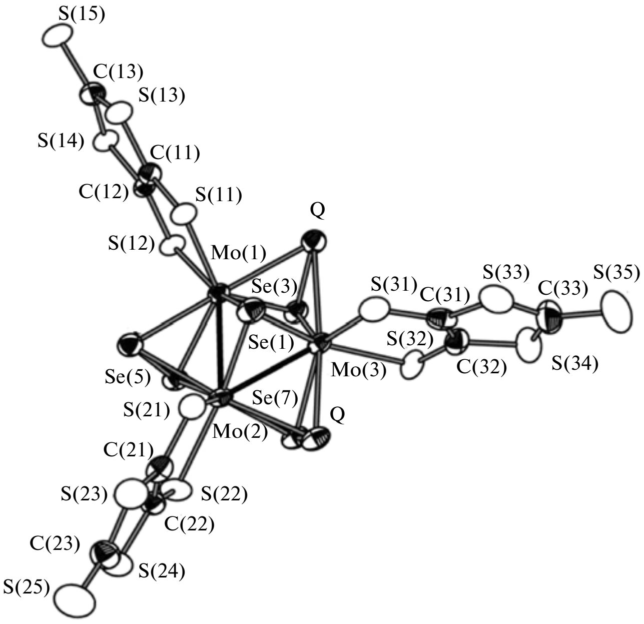


Fig. 1. Structure of the cluster $[Mo_3Se_{5.88}S_{1.12}(Dmit)_3]^{2-}$ (50% probability ellipsoids).

In this work we also studied the mechanochemical reactions of other molybdenum and tungsten chalcohalides with $(Bu_4N)_2[Zn(Dmit)_2]$. It was found using electrospray mass spectrometry that, under similar conditions, $Mo_3Te_7I_4$ and $W_3Q_7Br_4$ (Q = S, Se, Te) form the known binuclear complexes $[M_2^{V}O_2Q_2(Dmit)_2]^{2-}$ (Q = S, Se) due to the oxidative fragmentation of the cluster. As a rule, a mixture of the complexes with different O/Q ratios is formed. For example, $W_3S_7Br_4$ gives a mixture of $[W_2^{V}O_2S_2(Dmit)_2]^{2-}$ ($m/z = 428$), $[W_2^{V}OS_3(Dmit)_2]^{2-}$ ($m/z = 437$), and $[W_2^{V}S_4(Dmit)_2]^{2-}$ ($m/z = 445$) with the predominant content of $[W_2^{V}O_2S_2(Dmit)_2]^{2-}$. A similar fragmentation is observed in the reactions of $M_3Q_7Br_4$ (Q = S, Se) with $[Zn(Dmit)_2]^{2-}$ and PPh_3. Trinuclear complexes $[M_3^{IV}Q_4(Dmit)_3]^{2-}$ formed under these conditions at the intermediate stage are rapidly oxidized in air to form binuclear $[M_2^{V}O_2Q_2(Dmit)_2]^{2-}$, which is observed by electrospray mass spectrometry [19].

Thus, the tungsten clusters manifest a lower stability than the molybdenum analogs, which is due to the general tendency to a lower stability of the low oxidation states of the early 5*d* elements compared to the 4*d* elements.

The crystal structure of compound **I** includes different types of nonvalent interactions Q···Q characteristic of the triangular chalcogenide-bridged clusters $\{M_3Q_7\}^{4+}$ (M = Mo, W, Q = S, Se, Te) [6, 8, 18]. The main building block in crystal structure **I** is the trinuclear cluster anion $[Mo_3(\mu_3\text{-}Se)(\mu_2\text{-}QSe)_3(Dmit)_3]^{2-}$ (Fig. 1). The equatorial positions of the nonsymmetrically coordinated dichalcogenide ligands (QSe) are randomly occupied by the sulfur and selenium atoms. Each molybdenum atom nonsymmetrically coordinates one bidentate ligand $C_3S_5^{2-}$. These ligands are arranged rigidly in the planes formed by μ_3-Se and μ_2-QSe pairs. It is known that the bent conformation of the isotrithionedithiolate ligand can be observed for a similar type of complexes, which is, most likely, due to strong nonvalent inter- and intramolecular interactions of the sulfur atoms of the ligand and the chalcogen atoms of the cluster core [6]. The formation of dimers of the cluster anions (Fig. 2) characteristic of the complexes with the $C_3S_5^{2-}$ ligands is observed in the structure of complex **I** due to the nonvalent contacts S(Dmit)···Se(ax.) (3.233(1)–3.719(1) Å) [4–6]. However, the further formation of agglomerates due to nonvalent interactions can be very diverse. Sulfide complexes $[Mo_3S_7(Dsit)_3]^{2-}$ (Dsit is 1,3-dithia-2-dithione-4,5-diselenolate), $[Mo_3S_7(Dmid)_3]^{2-}$ (Dmid is bis(trifluoromethyl)-1,2-dithiolate), and other dithiolate complexes were described [5]. Additional interactions between the remained ligands Dmid or Dsit of the adjacent clusters are observed along with the formation of dimers on the basis of the S(L)···S(ax.) (L = Dmit, Dsit) interactions with one of the ligands of the adjacent cluster anion in the crystal structures of $(Bu_4N)_2[Mo_3S_7(Dsit)_3]$ and $(PPh_4)_2[Mo_3S_7(Dmid)_3]$. This results in the formation of infinite chains along the crystallographic directions [011] and [101]. In both compounds the third coordinated ligand is not involved in secondary interactions. Dimer formation also occurs in the structure of $(Bu_4N)_2[Mo_3S_7(Dmit)_3]$ [4]. The contacts S(eq.)···S(Dmit) and S(Dmit)···S(Dmit) affords agglomerates of various dimensionality.

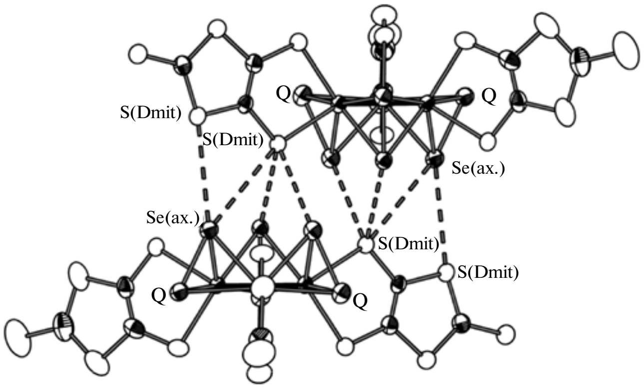


Fig. 2. Formation of the dimers from the cluster anions in structure **I**.

Infinite chains oriented along the crystallographic axis *x* are formed in the structure of complex **I** due to

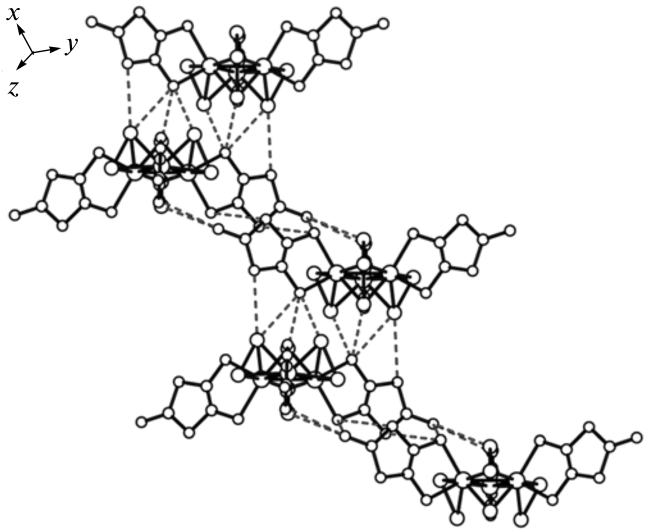


Fig. 3. Joining of the dimers into chains by the nonvalent interactions S(Dmit)···S(Dmit) in structure **I**.

the contacts S(Dmit)···S(Dmit)3.528(1)–3.843(1) Å between the ligands of the adjacent dimers (Fig. 3). Two ligands from each cluster participate in the formation of these chains, and the third ligand is not involved in the binding. Interestingly, only one of the equatorial positions with the occupancy Se/S = 0.8/0.2 is involved in the formation of nonvalent contacts. Thus, there is some dependence of the type of crystalline packing on the occupancy of the equatorial position with the sulfur atom. For example, in the structure of $(Bu_4N)_2[Mo_3Se_{6.1}S_{0.9}(Dmit)_3]$ [6] with a lower sulfur content in the equatorial positions (occupancies 0.4, 0.4, and 0.1) the Q(eq.)···S(Dmit) (Q = S, Se) contacts result in the formation of the network 4^4 along the crystallographic direction [101]. The S(Dmit)···S(Dmit) interactions join these networks along the crystallographic *y* into the three-dimensional structure. In the case of the equatorial positions completely occupied by the sulfur atoms, the nonvalent contacts S(eq.)···X and S(eq.)···S(Dtc) are observed in the structures of the diethyldithiocarbamate complexes $[Mo_3(\mu_3\text{-}S)(\mu_2\text{-}S_2)_3(Dtc)_3]X$ (X = Cl, Br, I, ClO_4, etc.) [20–22].

Thus, the mechanochemical reactions of cluster chalcohalides $M_3Q_7X_4$ (M = Mo, W; Q = S, Se, Te; X = Br, I) with $(Bu_4N)_2[Zn(Dmit)_2]$, as a rule, afford the known binuclear complexes $[M_2O_2Q_2(Dmit)_2]^{2-}$, which are reliably identified by electrospray mass spectrometry. The reaction of $Mo_3Se_7Br_4$ with $(Bu_4N)_2[Zn(Dmit)_2]$ affords a new trinuclear dithiolate complex $[Mo_3Se_{5.88}S_{1.12}(Dmit)_3]^{2-}$ containing the cluster core mixed in chalcogen in which each molybdenum atom is coordinated through the bidentate mode to the isotrithionedithiolate ligand $C_3S_5^{2-}$. Complex **I**, whose crystal structure contains the axial and equatorial nonvalent contacts Q···Q resulting in the formation of nonvalent-bonded chains, was structurally characterized.

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