ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2015, Vol. 41, No. 11, pp. 759–764. © Pleiades Publishing, Ltd., 2015.
Original Russian Text © D.S. Ogienko, A.I. Smolentsev, D.A. Bashirov, M.Yu. Afonin, P.A. Petrov, S.N. Konchenko, 2015, published in Koordinatsionnaya Khimiya, 2015, Vol. 41, No. 11, pp. 687–692.

Syntheses and Structures of Complexes $\{Mo_2S_2O_2\}^{2+}$ with Labile Cl^- and DMF Ligands

D. S. Ogienko[a], A. I. Smolentsev[a, b], D. A. Bashirov[a, b], M. Yu. Afonin[a], P. A. Petrov[a, b], and S. N. Konchenko[a, b, *]

[a] *Nikolaev Institute of Inorganic Chemistry, Siberian Branch, Russian Academy of Sciences, pr. Akademika Lavrent'eva 3, Novosibirsk, 630090 Russia*

[b] *Novosibirsk State University, ul. Pirogova 2, Novosibirsk, 630090 Russia*

**e-mail: konch@niic.nsc.ru*

Received April 22, 2015

Abstract—Complex $(Me_4N)_2[Mo_2S_2O_2Cl_4]$ ($(Me_4N)_2$[**II**]) is synthesized by passing gaseous HCl through a solution of $(Me_4N)_2[Mo_2O_2S_8]$ ($(Me_4N)_2$[**I**]). The recrystallization of $(Me_4N)_2$[**II**] from *N*,*N*-dimethylformamide (DMF) gives compound $(Me_4N)[Mo_2S_2O_2Cl_3(DMF)_3]$ ((Me_4N)[**III**]). The oxidation of $(Et_4N)_2$[**I**] with iodine in DMF affords the earlier described complex $[Mo_2S_2O_2(DMF)_6]I_2$ ([**IV**]I_2). However, this complex was not structurally characterized. The structures of the synthesized compounds are determined by X-ray diffraction analyses (CIF files CCDC nos. 1059924–1059926). Thus, several complexes in which the binuclear fragment $\{Mo_2S_2O_2\}^{2+}$ has a labile coordination environment were synthesized and characterized.

DOI: 10.1134/S1070328415110056

INTRODUCTION

The chemistry of chalcogen oxide clusters of molybdenum is an area formed at the turn of the chemistry of well studied polyoxometallates and the chemistry of chalcogenide clusters of these metals. The latter two classes of compounds were well studied and recommended themselves as promising objects for a whole number of fields of practical application: from bioinorganic chemistry to materials science [1–5]. Interest in chalcogenide oxide clusters is stimulated by the potential possibility of successful combining the properties of polyoxometallates and chalcogenide clusters with the appearance of a synergistic effect.

In the recent two decades, a representative series of chalcogen oxide clusters was obtained by the pH-controlled condensation of the aqua complexes of $\{Mo_2O_2S_2\}^{2+}$ and $\{Mo_3S_4\}^{2+}$ cations and by their cocondensation with organic acids or polyoxometallates, which showed rich possibilities of synthetic approaches based on the use of the "building blocks" $\{Mo_2O_2S_2\}$ and $\{Mo_3S_4\}$ [5].

At the same time, it is interesting that almost the whole chemistry of chalcogen oxide clusters based on the $\{Mo_2O_2S_2\}$ fragments represents nowadays predominantly the chemistry of aqueous solutions. The possibility of using organic media for the synthesis of chalcogen oxide clusters can substantially enrich their chemistry and open a way to the synthesis of basically novel structures of the chalcogen oxide clusters themselves, as well as related coordination polymers and supramolecular compounds.

The "assembling" of chalcogen oxide clusters in non-aqueous media needs synthetically convenient compounds being sources of the $\{Mo_2O_2S_2\}$ and $\{Mo_3S_4\}$ fragments, i.e., complexes of cations $\{Mo_2O_2S_2\}^{2+}$ and $\{Mo_3S_4\}^{2+}$ with a sufficiently labile ligand environment. One of the earlier known suitable complexes is $[Mo_2S_2O_2(DMF)_6]^{2+}$ ([**IV**]$^{2+}$) generated in situ by the oxidation of $[Mo_2O_2S_8]^{2-}$ ([**I**]$^{2-}$) with iodine in a DMF solution [6, 7]. The in situ generation of this compound has a series of drawbacks in respect of the further synthetic use: an inaccurate composition of the used solution and the presence of colloidal sulfur in this solution. In addition, the structure of [**IV**]$^{2+}$ has not be established up to now.

In this work, we proposed the procedure of synthesis and isolation of [**IV**]I_2 in the crystalline state, which made it possible to determine the structure by X-ray diffraction analysis. In addition, the following novel complexes were synthesized and structurally characterized: $(Me_4N)_2[Mo_2S_2O_2Cl_4]$ ($(Me_4N)_2$[**II**]) and $(Me_4N)[Mo_2S_2O_2Cl_3(DMF)_3]$ ((Me_4N)[**III**]). The synthesized compounds can be convenient sources of the $\{Mo_2O_2S_2\}$ fragment in the synthesis of new chalcogen oxide clusters in non-aqueous media.

EXPERIMENTAL

All procedures on the synthesis and isolation of products and the preparation of samples for recording and elemental analyses were carried out in air. The solvents were purified using standard procedures [8]. The starting compounds were synthesized using known procedures: [9] for $(Et_4N)_2$[**I**], and [10] for $[(Me_4N)_2$[**I**]. All other reagents were used in the commercially available form.

Elemental analyses of the samples to C, H, and N were carried out at the Laboratory of Microanalysis of the Nikolaev Institute of Inorganic Chemistry (Siberian Branch, Russian Academy of Sciences) on a Euro EA 3000 analyzer. IR spectra were recorded on a Scimitar FTS 2000 instrument.

Synthesis of $(Me_4N)_2$[II]. A suspension of $(Me_4N)_2$[**I**] (0.828 g, 1.32 mmol) in CH_2Cl_2 (50 mL) was purged for 40 min with gaseous HCl, resulting in a change in its color from orange to dark brown. The reaction mixture was filtered. A precipitate was washed with CS_2 (15 mL) to remove S_8 and then extracted with two portions of a CH_3CN–CH_2Cl_2 (1 : 1) mixture (60 mL). A dark green solution was evaporated to dryness on a rotary evaporator to give a green crystalline product $(Me_4N)_2$[**II**] contaminated with S_8. A sulfur impurity was removed by heating of the solid residue to a constant weight in vacuo (5×10^{-2} mm Hg) at 120°C. The yield of $(Me_4N)_2$[**II**] 0.482 was 0.482 g (63%). The crystals suitable for X-ray diffraction analysis were obtained by the slow evaporation of a solution of the complex in a CH_2Cl_2–CH_3CN (20 : 1) mixture.

IR (KBr; ν, cm^{-1}): 1611 m, 1484 s, 948 s, 467 m.

For $C_8H_{24}N_2O_2S_2Cl_4Mo_2$

anal. calcd., %:	C, 16.6;	H, 4.2;	N, 4.8.
Found, %:	C, 16.6;	H, 4.5;	N, 4.7.

Synthesis of (Me_4N)[III]. Dimethylformamide (10 mL) was added to a solid sample of $(Me_4N)_2$[**II**] (0.686 g, 1.19 mmol). The obtained suspension was stirred for 12 h at room temperature, and the solution was filtered. Orange crystals of compound (Me_4N)[**III**] were obtained by the slow diffusion of diethyl ether vapors to a solution of the complex in DMF at room temperature. The yield of (Me_4N)[**III**] was 0.213 g (26%).

IR (KBr; ν, cm^{-1}): 1640 s, 1484 m, 1363 m, 936 s, 682 m, 461 m. No satisfactory data on the elemental analysis of (Me_4N)[**III**] were obtained, because the complex decomposed with the loss of one DMF molecule already at room temperature. The storage of (Me_4N)[**III**] in air at room temperature for several days afforded $(Me_4N)[Mo_2S_2O_2Cl_3(DMF)_2]$, and a more prolonged storage of the complex in air did not result in any changes.

For $C_{10}H_{26}N_3O_4S_2Cl_3Mo_2$

anal. calcd., %:	C, 19.5;	H, 4.3;	N, 6.8;	S, 10.4.
Found, %:	C, 19.6;	H, 4.7;	N, 6.7;	S, 10.2.

Synthesis of $[Mo_2O_2S_2(DMF)_6]I_2 \cdot$ DMF ([IV]$I_2 \cdot$ DMF). The synthesis was carried out using a modified procedure described previously [7]. A solution of I_2 (1.38 g, 5.4 mmol) in DMF (8.5 mL) was added to a solution of $(Et_4N)_2$[**I**] (2 g, 2.7 mmol) in DMF (30 mL). The reaction mixture was stirred for 15 min, and diethyl ether (Et_2O, 70 mL) was added. In 10 min, the reaction mixture was filtered through a paper filter. Another portion (70 mL) of Et_2O was added to a dark red solution. The obtained mixture was kept at –15°C for 20 h, which led to the precipitation of colorless crystals of $(Et_4N)I$ and the formation of a dark red film on the bottom of the vessel. The film was dissolved in DMF (40 mL) cooled to –15°C, the solution was rapidly decanted, and Et_2O (130 mL) was added. The storage of the mixture at –15°C for 4 days resulted in the crystallization of [**IV**]$I_2 \cdot$ DMF in the form of orange needle-like crystals suitable for X-ray diffraction analysis. The yield was 1.28 g (45%).

IR (KBr; ν, cm^{-1}): 2925 w, 1645 s, 1491 w, 1429 m, 1363 s, 947 s, 928 m, 473 m.

For $C_{21}H_{49}N_7O_9S_2I_2Mo_2$

anal. calcd., %:	C, 23.9;	H, 4.7;	N, 9.3;	S, 6.1.
Found, %:	C, 23.8;	H, 4.7;	N, 9.1;	S, 5.9.

X-ray diffraction analyses of compounds $(Me_4N)_2$[**II**], (Me_4N)[**III**], and [**IV**]$I_2 \cdot$ DMF were carried out using a standard procedure on a Bruker-Nonius X8 Apex automated four-circle diffractometer equipped with a 4K two-coordinate CCD detector (MoK_α radiation, $\lambda = 0.71073$ Å, graphite monochromator). An absorption correction was applied empirically (by intensities of equivalent reflections) using the SADABS program [11]. The structures were solved using a direct method and refined by full-matrix least squares for F^2 in the anisotropic approximation for all non-hydrogen atoms (SHELXTL) [11]. Hydrogen atoms were calculated geometrically and refined by the riding model. The crystallographic data, conditions for diffraction experiments, and results for structure refinement are presented in Table 1. The coordinates of atoms and thermal parameters were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC 1059924–1059926; deposit@ccdc.cam.ac.uk or http://www. ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The synthesis of ([**IV**]I_2) was carried out using a modified known procedure [7]. The procedure was changed in the part of product isolation, which made it possible to obtain crystals suitable for X-ray diffraction analysis. The interaction of $(Et_4N)_2$[**I**] with two equivalents of I_2 in DMF results in the oxidation of the polysulfide ligands, the precipitation of sulfur, and the formation of complex $[\mathbf{IV}]^{2+}$ in the solution (Scheme). The complex can be precipitated in the form of salt [**IV**]I_2 by the addition of Et_2O. However, $(Et_4N)I$ also

Table 1. Crystallographic data, details of diffraction experiments, and refinement results for structures $(Me_4N)_2$[**II**], $(Me_4N)_2$[**III**], and [**IV**]$I_2 \cdot$ DMF

Parameter	Value		
	$(Me_4N)_2$[**II**]	(Me_4N)[**III**]	[**IV**]$I_2 \cdot$ DMF
FW	578.11	687.8	1053.48
Temperature, K	150(2)	296(2)	296(2)
Crystal system	Orthorhombic	Triclinic	Monoclinic
Space group	*Ibam*	$P\bar{1}$	$C2/c$
a, Å	16.4447(13)	8.7303(3)	33.097(3)
b, Å	10.9050(9)	11.7152(5)	14.7383(13)
c, Å	11.8661(11)	14.1880(6)	16.8993(12)
α, deg		72.519(2)	
β, deg		76.6750(10)	105.951(2)
γ, deg		77.2770(10)	
V, Å^3	2127.9(3)	1328.89(9)	7925.9(11)
Z	4	2	8
ρ_{calcd}, g/cm^{-3}	1.804	1.719	1.766
μ_{MoK_α}, mm^{-1}	1.877	1.431	2.344
$F(000)$	1144	692	4144
Crystal size, mm	0.16 × 0.08 × 0.02		
θ Range for data collection, deg	3.43–26.37	2.43–27.51	1.52–25.68
Ranges of h, k, l	$-12 \le h \le 20$, $-13 \le k \le 13$, $-14 \le l \le 13$	$-11 \le h \le 11$, $-14 \le k \le 15$, $-18 \le l \le 14$	$-40 \le h \le 40$, $-17 \le k \le 17$, $-13 \le l \le 20$
Number of measured reflections	3285	9024	25785
Number of independent reflections (R_{int})	1151 (0.0490)	5899 (0.0201)	7451 (0.0465)
Number of observed reflections ($I > 2\sigma(I)$)	901	5399	6071
Completeness of data collection to 2θ = 50°, %	99.5	97.7	98.8
Number of refined parameters	62	272	402
R factor ($I > 2\sigma(I)$)	$R_1 = 0.0640$, $wR_2 = 0.1521$	$R_1 = 0.0222$, $wR_2 = 0.0581$	$R_1 = 0.0752$, $wR_2 = 0.2096$
R factor (all data)	$R_1 = 0.0829$, $wR_2 = 0.1601$	$R_1 = 0,0252$, $wR_2 = 0.0597$	$R_1 = 0,0888$, $wR_2 = 0.2157$
Goodness-of-fit	1.067	1.070	1.047
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	1.72/–0.75	0.47/–0.49	3.90/–0.94

precipitates from the solution together with [**IV**]I_2 precipitated as a dark red film on the walls of the vessel. Complex [**IV**]I_2 can be separated from the major mass of $(Et_4N)I$ due to the high dissolution rate of the complex in cooled DMF from which [**IV**]I_2 crystallizes as a solvate with DMF upon the addition of Et_2O and storage at –15°C.

Compound $(Me_4N)_2$[**II**] was obtained by passing dry HCl through a suspension of $(Me_4N)_2$[**I**] in CH_2Cl_2 (Scheme). The formed $(Me_4N)_2$[**II**] complex precipitates together with sulfur, which can be removed by heating in a high vacuum at 120°C followed by crystallization. Compound $(Me_4N)_2$[**II**] is well soluble in polar organic solvents, for example, in the N-donor solvent acetonitrile from which the solid phase of the same composition crystallizes upon evaporation. However, the solution gains an orange color upon the dissolution of green crystals of $(Me_4N)_2$[**II**} in the O-donor solvent DMF. The slow diffusion of diethyl ether vapors in this solution affords orange crystals of (Me_4N)[**III**]. In this case, one chloride ligand in complex $(Me_4N)_2$[**II**] is replaced by a DMF molecule, and two additional DMF molecules add to the both Mo atoms (Scheme). In the absence of a mother liquor or DMF vapors, the crystals of

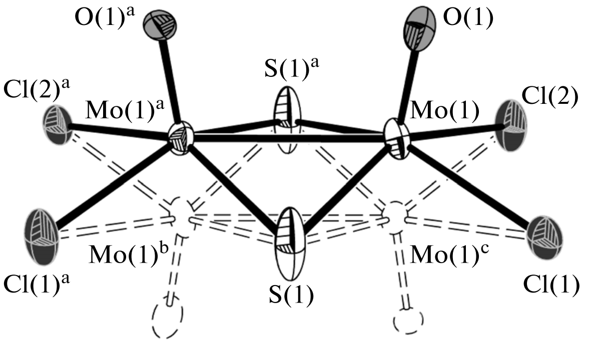


Fig. 1. Structure of the $[Mo_2S_2O_2Cl_4]^{2-}$ anion in compound $(Me_4N)_2$[**II**] (thermal ellipsoids of 50% probability). An alternative orientation of the anion is shown by dashed lines. Symmetry procedures: (a) $-x, 1 - y, z$; (b) $-x, 1 - y, -z$; (c) $x, y, -z$.

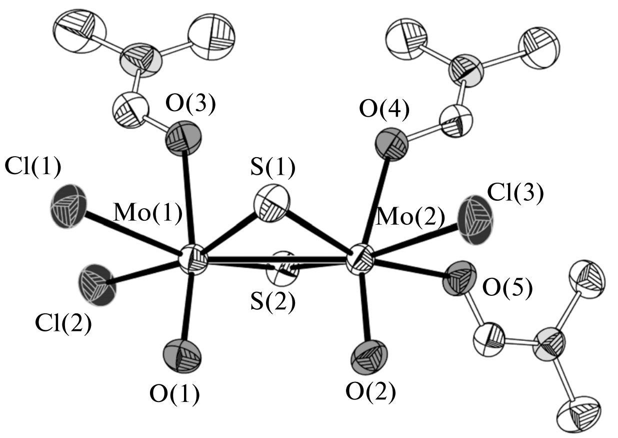
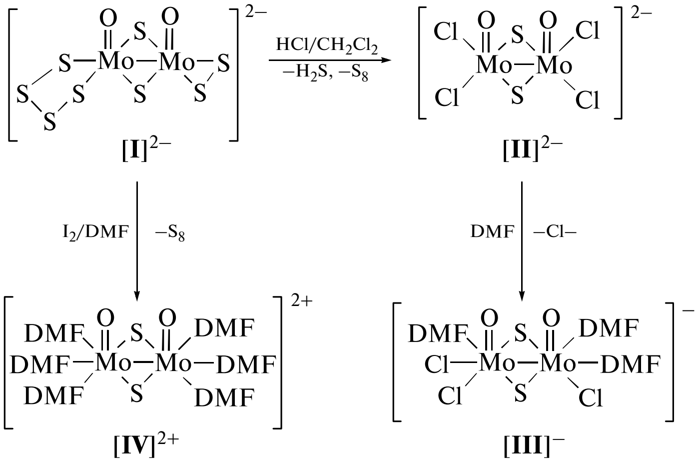


Fig. 2. Structure of the $[Mo_2S_2O_2Cl_3(DMF)_3]^-$ anion in compound $(Me_4N)_2$[**III**] (thermal ellipsoids of 50% probability). Hydrogen atoms of the DMF ligands are omitted.

(Me_4N)[**III**] are gradually dissolved to transform into a finely dispersed powder. According to the elemental analysis data, one DMF molecule is removed and complex $(Me_4N)[Mo_2S_2O_2Cl_3(DMF)_2]$ is formed.



Scheme.

The structures of the synthesized compounds were determined by X-ray diffraction analyses of single crystals.

The crystal structure of $(Me_4N)_2$[**II**] formed by the cluster anions $[Mo_2S_2O_2Cl_4]^{2-}$ (Fig. 1) with the positional symmetry C_2 and tetramethylammonium cations is characterized by two equally probable orientations of the anion connected to each other by the mirror reflection plane passing through the S and Cl atoms. The crystal structure of (Me_4N)[**III**] is formed by the cluster anions $[Mo_2S_2O_2Cl_3(DMF)_3]^-$ (Fig. 2) and tetramethylammonium cations. The independent part of the unit cell of compound [**IV**]I_2 includes the cluster cation $[Mo_2S_2O_2(DMF)_6]^{2+}$ (Fig. 3), two iodide anions, and a solvate DMF molecule.

The common structural fragment of all three compounds is $\{Mo_2(\mu\text{-}S)_2O_2\}$ in which two molybdenum atoms are remote at a distance assuming the existence of an ordinary bond. They are linked by the bridging sulfide ligands, and the terminal ligand O^{2-} is coordinated to each molybdenum atom. In anion $[\mathbf{II}]^{2-}$, the square pyramidal (ignoring the Mo–Mo bond) environment of each Mo atom is completed by two Cl^-. The Mo–Cl bond lengths (2.438(2)–2.473(3) Å) are close to the values of those in $Cs_3[Mo_2O_2S_2Cl_4(H_2O)_2]Cl$ [12] and $[Mo_2S_2O_2Cl_2(Py)_4]$ [13]. In anion $[\mathbf{III}]^-$ and cation $[\mathbf{IV}]^{2+}$, the coordination polyhedron of the molybdenum atoms (ignoring the Mo–Mo bond) is a distorted octahedron. In addition to the oxo and sulfide ligands in $[\mathbf{III}]^-$, two chloride ions and one DMF molecule are coordinated to one of the molybdenum atoms, and two DMF molecules and one chloride ligand are coordinated to another molybdenum atom. In **[IV]**$^{2+}$, three DMF molecules are coordinated to each Mo atom through the oxygen atoms.

In all cases, the $\{Mo_2S_2\}$ fragment is nonplanar. The dihedral angle between the planes (Mo(1)S(1)S(1)^a and Mo(1)^{a}S(1)S(1)^a (Fig. 1) or Mo(1)S(1)S(2) and Mo(2)S(1)S(2) (Figs. 2 and 3) is 134.59(11)° in **[II]**$^{2-}$, 149.86(3)° in **[III]**$^-$, and 154.65(15)° in **[IV]**$^{2+}$. The Mo–Mo and Mo–S distances in all three complexes (Table 2) have values typical of compounds containing the $\{Mo_2(\mu\text{-}S)_2O_2\}^{2+}$ cluster core [14]. This results in a regular shortening of the Mo=O bond and an elongation of the Mo–Mo distance with a change in the coordination number of molybdenum. In complex **[II]**$^{2-}$, where Mo has a coordination number of 5, the Mo–Mo distance is by ~0.03 and 0.05 Å longer and Mo=O is by ~0.07 Å shorter than those in **[III]**$^-$ and **[IV]**$^{2+}$, where the coordination number is 6.

The Mo–O(DMF) bond lengths in **[III]**$^-$ and **[IV]**$^{2+}$ vary in fairly wide ranges, and the maximum difference (up to 0.1 Å) is observed in cation **[IV]**$^{2+}$. In spite of this, the obtained values are consistent with the corresponding values in the earlier described compound $[Mo_2S_2O_2(S_2)(DMF)_3]$ [9]. In cation **[IV]**$^{2+}$, the shortest Mo–O(DMF) bonds correspond to four equatorial DMF ligands, while the longest bonds correspond to two apical DMF ligands. In anion **[III]**$^-$, the maximum length of the Mo–O(DMF) bond is observed for the DMF molecule coordinated to the Mo(1) atom with two chloride ligands. This bond is by ~0.03–0.04 Å longer than the bonds with the oxygen atoms of two other DMF ligands and by 0.02–0.05 Å longer than the corresponding bonds in **[IV]**$^{2+}$. It is most likely that the substantial difference in Mo–O(DMF) bond lengths compared to those in "symmetrical" **[IV]**$^{2+}$ is a consequence of the steric effect of the Cl$^-$ ions. The Mo–Cl bonds in **[III]**$^-$ are also somewhat longer than those in **[II]**$^{2-}$.

Probably, the size of the Cl$^-$ ions explains the fact of **[II]**$^{2-}$ formation under the conditions of the above described reaction of $(Me_4N)_2$**[I]** with HCl in CH_2Cl_2. Since the single ligand can be only Cl$^-$ in this case but this ion has no site in coordination with the Mo atom, the latter remains in the pentacoordinate environment. The potentially vacant sixth coordination site can be occupied by a ligand with a smaller donor atom. However, a steric hindrance can be surmounted for this purpose, which is impossible in the case of the N-donor ligand CH_3CN but occurs in the case of O-donor DMF, and the latter displaces the chloride ion from the coordination sphere of molybdenum due to a high energy of the Mo–O bond. The fact that one of the DMF molecules easily leaves the coordination sphere of the complex already at room temperature is probably related to the steric influence of Cl$^-$ ions. It is most likely that the DMF molecule linked to Mo(1) having two chloride ligands is displaced from the coordination sphere in **[III]**$^-$ (Fig. 2).

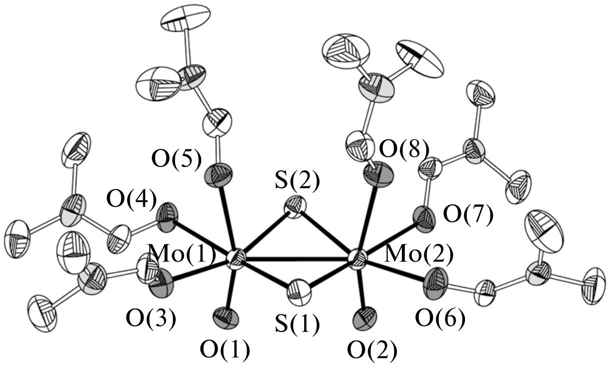


Fig. 3. Structure of the $[Mo_2S_2O_2(DMF)_6]^{2+}$ cation in compound **[IV]**I_2 · DMF (thermal ellipsoids of 50% probability). Hydrogen atoms of the DMF ligands are omitted.

Thus, the new complex $(Me_4N)_2$**[II]** was obtained in this work, The complex can be a convenient source of the $\{Mo_2(\mu\text{-}S)_2O_2\}$ fragment for "assembling" of chalcogen oxide clusters in non-aqueous media. The easy substitution of the chloride ligands in the complex is demonstrated by the reaction with DMF leading to (Me_4N)**[III]**. The comparison of structural features of these new compounds and **[IV]**I_2 suggests a noticeable steric influence of the chloride ions on the structures and properties of complexes $\{Mo_2(\mu\text{-}S)_2O_2\}$.

ACKNOWLEDGMENTS

This work was supported by the Russian Foundation for Basic Research (project nos. 14-03-31268, 13-03-01088, and 12-03-33028) and the Min-

Table 2. Selected bond lengths (Å) in structures $(Me_4N)_2$**[II]**, $(Me_4N)_2$**[III]**, and **[IV]**I_2 · DMF

Bond	$(Me_4N)_2$**[II]**	(Me_4N)**[III]**	**[IV]**I_2 · DMF
Mo–Mo	2.847(2)	2.8206(2)	2.7993(11)
Mo–(μ-S)	2.348(3), 2.357(3)	2.2962(5)–2.3181(5)	2.305(2)–2.312(2)
Mo=O	1.614(7)	1.6819(14), 1.6824(14)	1.683(7), 1.692(7)
Mo–O_{DMF}		2.2363(14)–2.2798(14)	2.156(7)–2.258(7)
Mo–Cl	2.477(2), 2.415(2)	2.4941(6)–2.5347(6)	

istry of Education and Science of the Russian Federation.

REFERENCES

1. Spiro, T.G., *Molybdenum Enzymes*, New York: Wiley, 1985, p. 611.
2. Müller, A., Diemann, E., Jostes, R., and Bögge, H., *Angew. Chem. Int. Ed.*, 1981, vol. 20, p. 934.
3. Müller, A., *Polyhedron*, 1986, vol. 5, p. 323.
4. DuBois, M., *Chem. Rev.*, 1989, vol. 89, p. 1.
5. Cadot, E., Sokolov, M.N., Fedin, V.P., et al., *Chem. Soc. Rev.*, 2012, vol. 41, p. 7335.
6. Gushchin, A.L., Laricheva, Yu.A., Alferova, N.I., et al., *Zh. Strukt. Khim.*, 2013, vol. 54, no. 4, p. 707.
7. Coucouvanis, D., Toupadakis, A., Lane, J.D., et al., *J. Am. Chem. Soc.*, 1991, vol. 113, p. 5271.
8. Gordon, A. and Ford, R., *The Chemist's Companion: A Handbook of Practical Data, Techniques, and References*, New York: Wiley, 1972.
9. Coucouvanis, D., Toupadakis, A., and Hadjikyriacou, A., *Inorg. Chem.*, 1988, vol. 27, p. 3272.
10. Bhattacharyya, R., Chakrabarty, P.K., Ghosh, P.N., et al., *Inorg. Chem.*, 1991, vol. 30, p. 3948.
11. *APEX2 (version 1.08), SAINT (version 7.03), SADABS (version 2.11),* Madison (WI, USA): Bruker AXS Inc., 2004.
12. Sinkevich, P.L., Korenev, V.S., Abramov, P.A., et al., *Russ. J. Coord. Chem.,* 2014, vol. 40, no. 4, p. 195.
13. Mizutani, J., Imoto, H., and Saito, T., *Acta Crystallogr., Sect. C: Cryst. Struct. Commun.*, 1997, vol. 53, p. 47.
14. Allen, F.H., *Acta Crystallogr., Sect. B: Struct. Sci.*, 2002, vol. 58, p. 380.

Translated by E. Yablonskaya