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Phosphine-Substituted Cubane Clusters with the Mo_3S_4Ga Core

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Abstract—The reaction of cationic clusters $[Mo_3S_4(Dppet)_3X_3]X$ (**I**; Dppet = *cis*-1,2-bis(diphenylphosphino)ethylene; X = Cl, Br) with gallium metal in tetrahydrofuran (THF) affords cubane-like clusters $[Mo_3S_4(GaX)(Dppet)_3X_3]$ (**IIa**, X = Cl; **IIb**, X = Br). In the synthesis of **IIb**, the crystals of the cationic cluster $[Mo_3S_4(Dppet)_3Br_3](GaBr_4)$ (**III**) were isolated from the mother liquor. The structures of complexes **IIa** · 1.5THF, **IIb** · 1.5THF, **III** · 1.625THF, and $[Mo_3S_4(Dppe)_3Br_3](GaBr_4)$ · 2THF (**IV** · 2THF; Dppe = 1,2-bis(diphenylphosphino)ethane) were established by X-ray diffraction (CIF files CCDC № 1851341–1851344).

Keywords: clusters, molybdenum, gallium, phosphines, X-ay diffraction

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INTRODUCTION

Clusters predominate among complexes of Mo and W in intermediate oxidation states, especially those containing chalcogenide ions [1]. One type of stable clusters with abundant chemistry are complexes composed of the six-electron cluster core $[M_3Q_4]^{4+}$ (M = Mo, W; Q = S, Se) and various terminal ligands, including mono- and diphosphines. A benefit of phosphine ligands is that they can be functionalized and thus acquire either additional properties (e.g., chirality or redox activity) or additional donor atoms [2–7]. Relatively recently, it was found that the six-electron cluster core $[M_3S_4]^{4+}$ stabilized by the coordination of bulky diphosphine ligands can be converted to the seven-electron core $[M_3S_4]^{3+}$ [8–10]. The reduction may be accompanied by the formation of heterocubane derivatives $[M_3S_4M']^{n+}$ or (for molybdenum clusters) by transformation of the $\{Mo_3Q_4\}$ core into $\{Mo_3Q_5\}$ [11, 12]. Heterocubane derivatives of the Mo and W clusters are known for a broad range of Group 6–15 elements. The compounds with aqua-, cyclopentadienyl-, and phosphine-substituted cluster cores are most abundant [13–22]. Nevertheless, for phosphine-substituted clusters, heterocubane derivatives containing a post-transition element have been obtained only in our recent study [23, 24]. It was found that the outcome of reaction of diphosphine clusters $[Mo_3Q_4(Diphos)_3X_3]X$ (Diphos = diphosphine) with gallium depends not only on the chalcogen and the halogen atoms, but also on the phosphine. In the case of 1,2-bis(diphenylphosphino)ethane (Dppe), it is possible to isolate both the reduction product $[Mo_3Q_4(Diphos)_3X_3]X$ and the cubane cluster $[Mo_3S_4(GaBr)(Dppe)_3Br_3]$, whereas in the case of aliphatic 1,2-bis(dimethylphosphino)ethane (Dmpe), only the cubane can be isolated [23]. Meanwhile, the reaction of the chloride cluster $[Mo_3S_4(Dppe)_3Cl_3]Cl$ with Ga gives only the reduction product $[Mo_3S_4(Dppe)_3Cl_3]$.

This study addresses the reactions between Ga and the cationic clusters $[Mo_3S_4(Dppet)_3X_3]X$ (X = Cl, Br) containing coordinated *cis*-1,2-bis(diphenylphosphino)ethylene (Dppet).

EXPERIMENTAL

All operations involved in the synthesis, purification, and characterization of the complexes were carried out under inert atmosphere using standard Schlenk technique. The solvents for the synthesis were dried and degassed by refluxing and distillation in an inert gas atmosphere using appropriate drying agents [25]. The initial complexes $[Mo_3S_4(Dppet)_3X_3]X$ (X = Cl (**Ia**), Br (**Ib**)) were prepared from $(Et_4N)_2Mo_3S_7X_6$ and Dppet similarly to Dppe-substituted clusters [26] and purified on a SiO_2 column (elution with $CHCl_3$–methanol (**Ia**) or acetone–methanol (**Ib**)). NMR spectra were recorded on a Bruker Avance III 500 spectrometer operating at 500.03 MHz for ^1H and 202.46 MHz for ^{31}P. IR spectra were measured on a SCIMITAR FTS 2000 instrument (KBr pellets).

Synthesis of $[Mo_3S_4(GaCl)(Dppet)_3Cl_3]$ (IIa). Solid $Mo_3S_4(Dppet)_3Cl_4$ (63 mg, 0.036 mmol) and Ga (109 mg, 1.581 mmol) were placed into a Schlenk ves-

sel, cooled to –196°C, and evacuated; then ~15 mL of THF was condensed into the vessel. The temperature of the mixture was brought to room temperature, and the suspension thus formed was refluxed with intense stirring for 14 days and allowed to settle. The brown-green solution was carefully filtered through a glass filter (G4) into an H-shaped Schlenk vessel, and ~20 mL of Et_2O was placed into the other leg. After complete mixing of the solvents, dark crystals of **IIa** · $1.5C_4H_8O$ suitable for X-ray diffraction were formed in a yield of 15 mg (22%).

For $C_{82}H_{74}OP_6S_4Cl_4GaMo_3$

Anal. calcd., %	C, 52.14	H, 3.95	S 6.79
Found, %	C, 51.85	H, 3.70	S, 6.85

IR (KBr; ν, cm^{-1}): 3049 m, 3001 m, 1618 m, 1585 m, 1483 m, 1433 s, 1308 m, 1189 m, 1158 m, 1096 m, 1026 m, 998 m, 912 m, 839 m, 738 s, 691 s, 552 s, 516 s, 489 m, 442 w, 423 w.

Synthesis of $[Mo_3S_4(GaBr)(Dppet)_3Br_3]$ (IIb) was carried out in a similar way from $Mo_3S_4(Dppet)_3Br_4$ (111 mg, 0.057 mmol) and Ga (128 mg, 1.875 mmol). The yield was 35 mg (30%).

For $C_{82}H_{74}OP_6S_4Br_4GaMo_3$

Anal. calcd., %	C, 47.65	H, 3.61	S, 6.21
Found, %	C, 47.35	H, 3.50	S 6.45

IR (KBr; ν, cm^{-1}): 3051 m, 1622 w, 1583 w, 1572 w, 1481 m, 1433 s, 1332 w, 1309 w, 1273 w, 1190 m, 1159 m, 1095 s, 1028 m, 999 m, 918 w, 843 w, 739 s, 692 s, 551 s, 517 s, 490 m, 443 w.

The mother liquor after the synthesis of **IIb** was transferred into another Schlenk vessel via a Teflon capillary in an argon flow. After some time, dark crystals of $[Mo_3S_4(Dppet)_3Br_3](GaBr_4)$ · $1.625C_4H_8O$ (**III** · $1.625C_4H_8O$) suitable for X-ray diffraction were formed in a yield of 15 mg (11%).

For $C_{84}H_{78}O_{1.5}P_6S_4Br_7GaMo_3$

Anal. calcd., %	C, 43.07	H, 3.35	S, 5.48
Found, %	C, 42.80	H, 3.60	S, 5.75

IR (KBr; ν, cm^{-1}): 3049 m, 3000 m, 1619 w, 1584 w, 1571 w, 1482 m, 1433 s, 1332 w, 1306 w, 1273 w, 1190 m, 1158 m, 1131 w, 1095 s, 1027 m, 998 m, 916 w, 838 w, 792 w, 737 s, 690 s, 616 w, 597 w, 552 s, 516 s, 488 w, 474 w, 441 w, 420 w. ^1H NMR (CD_3CN; δ, ppm): 6.24 (t, J = 7 Hz, 1H, =CH–), 6.52 (t, J = 7 Hz, 1H, =CH–), 6.90–8.05 (m, 20H, 4Ph). $^{31}P\{^1H\}$ NMR (CD_3CN; δ, ppm): 30.2 (d, J = 7 Hz), 40.4 (d, J = 7 Hz).

X-ray diffraction. All measurements were carried out by a standard procedure on a Bruker-Nonius X8 APEX four-circle automated diffractometer (CCD array detector, λ = 0.71073 Å, graphite monochromator). The reflection intensities were measured in the φ-scan mode for narrow (0.5°) frames. Absorption corrections were applied empirically (SADABS) [27]. The structures were solved by the direct method and refined by full-matrix least squares method in the anisotopic approximation for non-hydrogen atoms using the SHELXTL program package [28] and OLEX2 software [29]. The hydrogen atoms were located geometrically and refined in the rigid body approximation. In structures **IIa** · 1.5THF, **IIb** · 1.5THF, and **III** · 1.625THF, some THF molecules of solvation were highly disordered and could not be adequately modeled. The contribution of these molecules to the electron density was eliminated by the PLATON/SQUEEZE procedure [30]. The potential void volume was ~500 $Å^3$ for **IIa** · 1.5THF and **IIb** · 1.5THF and ~480 $Å^3$ for **III** · 1.625THF. The numbers of electrons per unit cell were 105, 117, and 40, respectively. This corresponds to three THF molecules per cell and 0.5 THF molecules per formula unit for **IIa** · 1.5THF and **IIb** · 1.5THF or one THF molecule per cell (partly occupying four equivalent positions) and 0.125 THF molecules per formula unit for **III** · 1.625THF. The crystal structure characteristics of the complex and X-ray diffraction experiment details are summarized in Table 1.

The crystallographic data for complexes **I**–**IV** are deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1851341–1851344; http://www.ccdc.cam.ac.uk/conts/retrieving.html).

RESULTS AND DISCUSSION

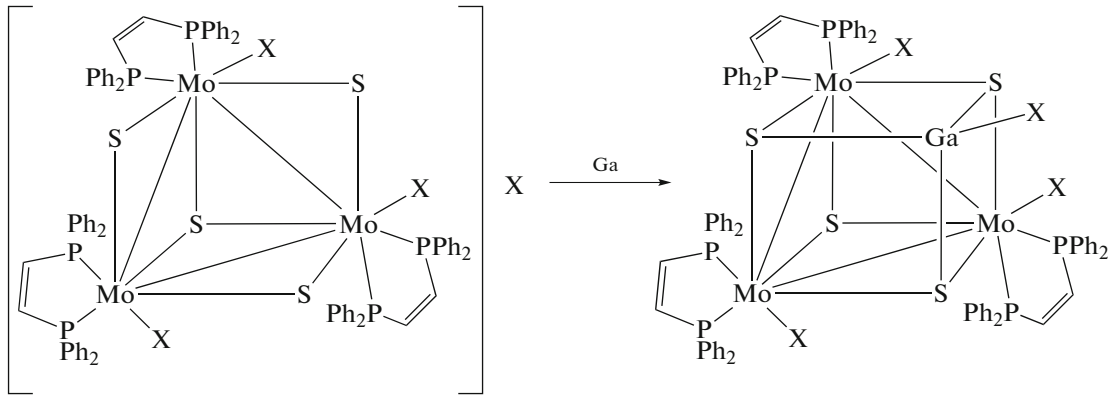
The Dppet cluster complexes, like Dppe analogues, were synthesized by treatment of the thiohalide complex $(Et_4N)_2[Mo_3S_7X_6]$ with phosphine [25]. Also, complex **Ia** can be prepared by replacing the monodentate phosphine by a bidentate one in the reaction of $[Mo_3S_4Cl_4(PPh_3)_3]$ with 3 equiv. of Dppet [31]. Previously, we observed a partial replacement of bromide ions in $[Mo_3S_4(Dppe)_3Br_3]Br$ on dissolution in CH_2Cl_2 or in CH_2Cl_2-containing solvent mixtures [8]. Therefore, chlorine-containing solvents were not used for the synthesis of **Ib**.

Heating of a suspension of **Ia** or **Ib** in THF with gallium metal results in gradual disappearance of the suspension being replaced by a brown solution; gradual diffusion of diethyl ether into the solution induces crystallization of cubane clusters **IIa** and **IIb** (Scheme 1). The yield of chloride-containing product is lower than that of the bromide analogue, which may be related to lower solubility of the starting cluster **Ia** in THF and, hence, longer time needed for completion of the reaction. Previously, it was noted that in the case of reduced $[Mo_3S_4(Dppe)_3X_3]$ clusters, the solubility

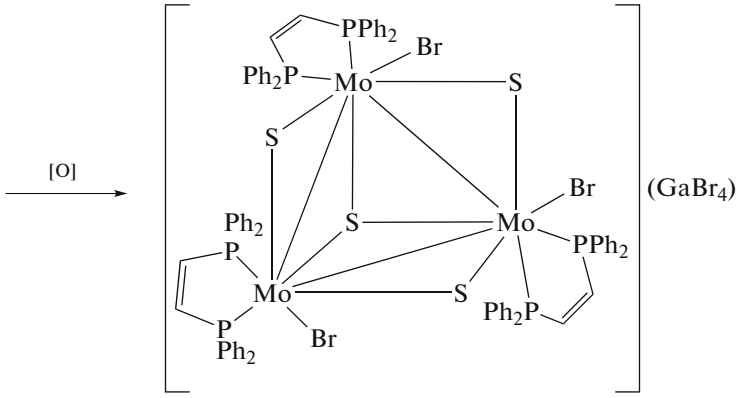
Table 1. Crystal structure characteristics and refinement parameters for **IIa** · 1.5THF, **IIb** · 1.5THF, **III** · 1.625THF, and **IV** · 2THF

Parameter	Value			
	IIa · 1.5THF	**IIb** · 1.5THF	**III** · 1.625THF	**IV** · 2THF
Molecular formula	$C_{84}H_{78}O_{1.5}S_4P_6Cl_4GaMo_3$	$C_{84}H_{78}O_{1.5}S_4P_6Br_4GaMo_3$	$C_{84.5}H_{79}O_{1.625}S_4P_6Br_7GaMo_3$	$C_{86}H_{88}O_2S_4P_6Br_7GaMo_3$
M	1924.98	2102.78	2351.51	2384.53
System, space group	Trigonal, $R\bar{3}$	Trigonal, $R\bar{3}$	Monoclinic, $C2/c$	Monoclinic, $P2_1/n$
Temperature, K	150	150	150	150
a, Å	15.256(3)	15.2151(7)	29.7194(9)	18.7589(4)
b, Å	15.256(3)	15.2151(7)	26.6232(8)	22.7059(5)
c, Å	59.471(11)	60.056(3)	25.5155(5)	21.4442(5)
β, deg	90	90	115.665(1)	103.104(1)
V, Å^3	11987(5)	12040.2(12)	18196.7(9)	8896.0(3)
Z	6	6	8	4
μ, mm^{-1}	1.197	3.050	4.01	4.11
Crystal size, mm	0.35 × 0.28 × 0.13	0.32 × 0.20 × 0.18	0.25 × 0.12 × 0.10	0.25 × 0.15 × 0.08
F(000)	5718	6150	9200	4704
Data collection range for θ, deg	1.579–25.655	1.688–26.368	1.168–26.372	1.431–25.026
Range of indices h, k, l	$-12 \le h \le 18$, $-17 \le k \le 7$, $-71 \le l \le 71$	$-18 \le h \le 13$, $-19 \le k \le 17$, $-71 \le l \le 75$	$-35 \le h \le 37$, $-33 \le k \le 33$, $-31 \le l \le 16$	$-18 \le h \le 22$, $-21 \le k \le 27$, $-25 \le l \le 25$
Number of measured, unique, and observed ($I > 2\sigma(I)$) reflections	15189, 4919, 3816	20157, 5474, 4699	62702, 18263, 13188	52629, 15708, 11208
R_{int}	0.0495	0.0309	0.0327	0.047
R_1, wR_2 ($I > 2\sigma(I)$)	0.1095, 0.2359	0.0652, 0.1790	0.0538, 0.0958	0.0493, 0.1327
R_1, wR_2 (all reflections)	0.1261, 0.2419	0.0737, 0.1838	0.0895, 0.1058	0.0819, 0.1436
GOOF	1.088	1.083	1.067	1.072
Number of refined parameters	309	309	1006	974
Number of restraints	82	10	72	50
$\Delta\rho_{max}/\rho_{min}$, e Å^{-3}	3.47/–2.51	1.65/–4.15	1.56/–1.14	1.86/–1.55

is also lower for the chloride derivative than for the bromide one [10]. Note also that the reaction of $[Mo_3S_4(Dppe)_3Cl_3]$ with gallium metal does not generate the cubane cluster; it was prepared by the reaction of $[Mo_3S_4(Dppe)_3Cl_3]$ with an excess of $[GaCp^*]_6$ [24].



(Ia), **(Ib)** **(IIa)**, **(IIb)**



(III)

Scheme 1.

The isostuctural phases **IIa** · 1.5THF (Fig. 1) and **IIb** · 1.5THF crystallize in the trigonal system, with a threefold inversion axis passing through the S(1) and Ga(1) atoms and the Ga-coordinated halogen atom. Therefore, the Mo-atom triangle is equilateral. In the gallium-containing cubanes crystallizing in the monoclinic system, the cluster core is substantially distorted, resulting in shortening of one Mo–Mo bond and deviation of the SGaX angle from 180°; the most pronounced distortion was found for $[Mo_3S_4(GaCl)(Dppe)_3Cl_3]$ (Table 2). The Mo–Mo distances in the $[Mo_3GaS_4(H_2O)_{12}]^{5+}$ aqua ion [32] are markedly shorter than those in phosphine clusters, whereas the Mo–Ga distances are, conversely, markedly longer. This may be due to both different numbers of cluster electrons in these clusters and greater coordination number of the Ga atom.

Owing to the presence of Ga in a low formal oxidation state, phosphine-substituted cubane clusters are extremely sensitive to oxygen and air moisture. The static magnetic susceptibility data for polycrystalline samples of **IIb** give much lower μ_{eff} values compared with that expected for one unpaired electron (1.73 μ_B). Yet another case of exceptional sensitivity of these complexes is the formation of the six-electron cationic cluster $[Mo_3S_4(Dppet)_3Br_3](GaBr_4)$ (**III**) upon contact of the mother liquor of crystallization of **IIb** with O_2 impurity present in argon.

The cluster cation in the crystal structure of **III** · 1.625THF retains the geometric characteristics inherent in this type of clusters with coordinated diphosphine ligands. The Mo atoms form a nearly equilateral triangle with the Mo–Mo bond lengths of 2.7754(6)–2.7915(6) Å (Fig. 2). These values are close to those present in $[Mo_3S_4(Dppet)_3Cl_3](PF_6)$ (2.779(1) Å [31])

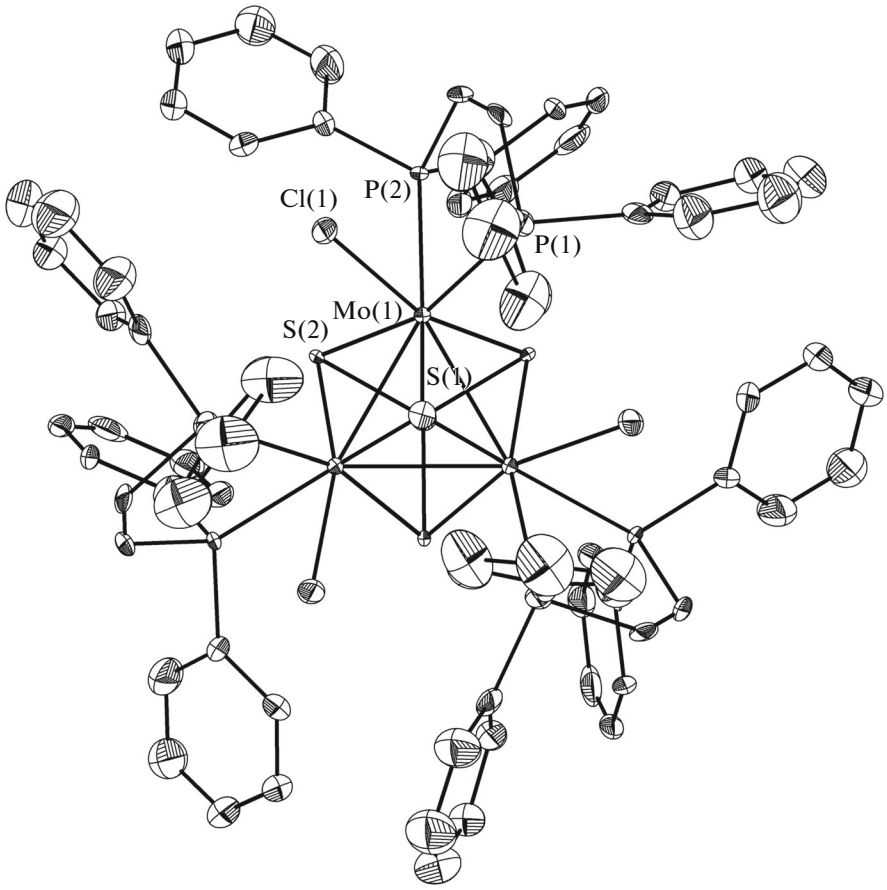


Fig. 1. Structure of complex **IIa**. The thermal ellipsoids are given at 30% probability level. The H atoms are omitted.

and $[Mo_3S_4(Dppe)_3Br_{0.75}Cl_{2.25}](BF_4)_{0.5}Cl_{0.5}$ (on average, 2.777 [6] Å) [33], which confirms the cationic structure of the cluster. The $GaBr_4$ counter-ions are disordered over two close crystallographic positions with virtually equal occupancies. The structure also incorporates THF molecules of solvation. The cationic state of the cluster is also confirmed by the $^{31}P\{^1H\}$ NMR spectrum, which exhibits two signals at 30.2 and 40.4 ppm corresponding to the P atoms located on different sides of the Mo_3 plane.

The single crystals of $[Mo_3S_4(Dppe)_3Br_3](GaBr_4) \cdot 2THF$ (**IV** · 2THF) were isolated in a moderate yield on attempted crystallization of the cubane cluster $[Mo_3S_4(GaBr)(Dppe)_3Br_3]$ [23], apparently, as a

Table 2. Geometric characteristics of the cubane clusters **IIa**, **IIb**, and their analogues

Compound	Distance, Å		SGaX angle, deg
	M–M	M–Ga	
IIa*	2.7874(17)	3.299(2)	180
IIb*	2.8023(10)	3.266(2)	180
$[Mo_3S_4(GaBr)(Dppe)_3Br_3]$ [23]	2.795 [18]	3.33 [4]	175.52(5)
$[Mo_3S_4(GaBr)(Dmpe)_3Br_3]$ [23]	2.7784(6)	3.2704(8)	180
$[Mo_3S_4(GaCl)(Dppe)_3Cl_3]$ [24]	2.78 [6]	3.32 [6]	169.23(3)
$[W_3S_4(GaBr)_{0.81}(Dppe)_3Br_3]$ [24]	2.787 [18]	3.41 [4]	175.13(6)
$[Mo_3GaS_4(H_2O)_{12}]^{5+}$ [32]	2.713 [3]	3.52 [2]	

* This work.

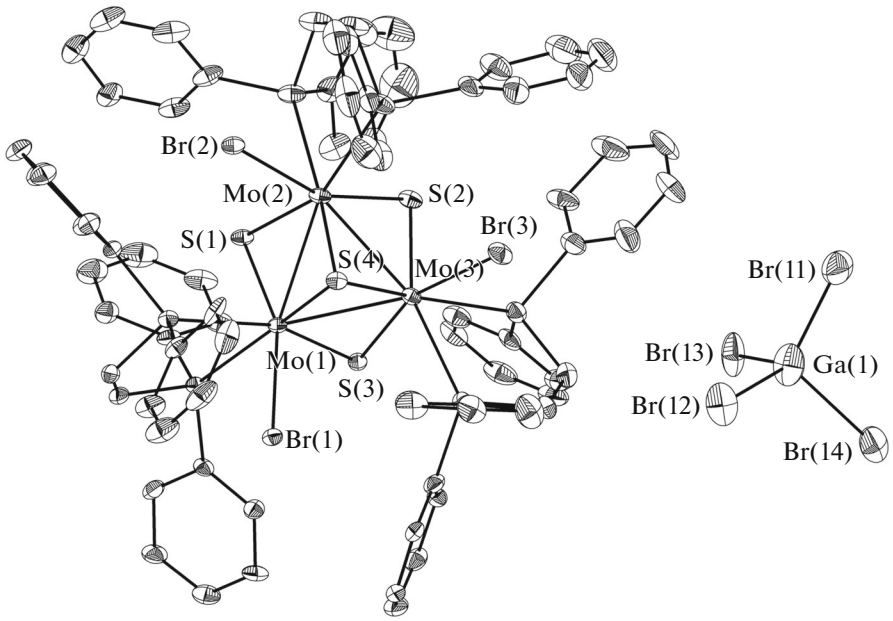


Fig. 2. Structure of complex **III**. The thermal ellipsoids are given at 30% probability level. One of the two disordered $GaBr_4^-$; anions is shown. The H atoms are omitted.

result of slow diffusion of air into the reaction mixture. Presumably, the formation of a stable salt of the cluster cation with GaX_4^- is a typical oxidation pathway of cubane clusters with the $\{M_3Q_4Ga\}$ core. The Mo–Mo distances in complex **IV** · 2THF (2.7811(7)–2.7944(7) Å) and other geometric parameters of the cluster core are close to those of **III** · 1.625THF and other cationic Mo clusters with the coordinated diphosphine ligands.

Thus, cubane-like clusters $[Mo_3S_4(GaX)(Dppet)_3X_3]$ **IIa** and **IIb** were isolated upon the reactions of the clusters $[Mo_3S_4(Dppet)_3X_3]X$ (X = Cl, Br) with elemental Ge. The products were characterized by X-ray diffraction as solvates **IIa** · 1.5THF and **IIb** · 1.5THF. The oxidation of the bromide cluster with the traces of oxygen leads to the recovery of the initial cationic cluster core giving $[Mo_3S_4(Dppet)_3Br_3](GaBr_4)$.

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