Synthesis and Structural Characterization of the Novel Cluster Compound $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4\,H_2O$ (dtp = $S_2P(OC_2H_5)_2^-$)

Jin Chen, Shao-Fang Lu,* Zi-Xiang Huang, Rong-Min Yu, and Qiang-Jin Wu[a]

Abstract: Reaction of $[Mo_3Y(\mu\text{-}S)_3(dtp)_4(H_2O)]$ (Y = O, S; dtp = $S_2P(OC_2H_5)_2^-$) with HgI_2 gave the novel compound $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4\,H_2O$ (**1**), which contains a $\{[Mo_3S_7(dtp)_3]_4\cdot I\}$ tetramer and $(HgI_3)^-$. Compound **1** has been characterized by IR, Raman, UV/Vis, and NMR spectroscopy and single-crystal X-ray diffraction analysis. It is shown that this formation process can be referred to as a new cluster reaction. The structure and spectroscopic data of the tetramer is also compared with that of the related discrete cluster $[Mo_3S_7(dtp)_3\cdot I]$. Crystal data: space group $F23$, $a = 26.786(3)$ Å, $V = 19218.7(4)$ Å^3, $Z = 4$, $R = 0.059$.

Keywords: cluster compounds · mercury · molybdenum · polymerization · structure elucidation

Introduction

The structural chemistry of $[Mo_3YS_3]$ and $[Mo_3Y(S_2)_3]$ (Y = O, S) clusters has been extensively studied.[1–5] Herein we focus on the reaction of $[Mo_3YS_3]$ with another metal M′ or metallic complex. It is anticipated that this study will stimulate some new reactions and provide information on specific physicochemical properties for such clusters. To date, many cubane-type clusters (sandwich cubanes and double cubanes) with a $[Mo_3YS_3M']$ core have been generated by the incorporation of a metal M′ in $[Mo_3YS_3]$.[6–8] Most of the metals M′ are transition metals and main group metals. As far as the metals of Group 12 (Zn, Cd, Hg) with a filled d^{10} shell are concerned, although the crystal structures of the sandwich cubane-type clusters $[(H_2O)_9Mo_3S_4HgS_4Mo_3(H_2O)_9](CH_3C_6H_5SO_3)_8\cdot 20\,H_2O$ and $[Mo_3S_4CdS_4Mo_3]^{8+}$ have been reported by Shibahara et al.,[9, 10] and the ionic clusters $[Mo_3S_4(Et_2PS_2)_3(py)_3][ZnI_3py]$ and $[Mo_3OS_3(dtp)_3(py)_3][CdI(dtp)_2]$ (dtp = $S_2P(OC_2H_5)_2^-$; py = pyridine) have been published by Keck et al.[11] and ourselves,[12] respectively, there are still relatively few structurally characterized examples of such clusters. Herein we report on the syntheis and structure of the novel network-like cluster $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4\,H_2O$ (**1**), which represents the first cluster containing a $\{[Mo_3S_7(dtp)_3]_4\cdot I\}$ tetramer and $(HgI_3)^-$.

Results and Discussion

Synthesis: The reaction of $[Mo_3(\mu_3\text{-}O,S)(\mu\text{-}S)_3(dtp)_4(H_2O)]$ with HgI_2 and Bu_4NI in a mixture of CH_2Cl_2 and CH_3CN gave $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4\,H_2O$ (**1**). Compound **1** is also formed in an analogous reaction mixture to which KBF_4 was added.

Evidently, during the reaction the cluster core $[Mo_3(\mu_3\text{-}O,S)(\mu\text{-}S)_3]^{4+}$ (Y = O, S) in the starting cluster is transformed to $[Mo_3(\mu_3\text{-}S)(\mu\text{-}S_2)_3]^{4+}$ in **1**. Such a conversion has been observed previously in our reaction systems.[13] However, a further tetramerization of $[Mo_3(\mu_3\text{-}S)(\mu\text{-}S_2)_3]$ as occurs in the present case is noted for the first time. It is estimated that the participation of HgI_2 in the reaction causes the elimination of the dtp bridging ligand from the starting $\{Mo_3\}$ cluster. The dtp ligands decompose to provide the sulfur atoms for the sulfur-addition reaction. Although the mechanism of this tetramerization is not yet clear, the reaction evidently involves the recombination and polymerization of molybdenum clusters. This reaction can therefore be referred to as a recombination–polymerization reaction.

Structural features: The compound crystallizes in the highly symmetrical cubic space group $F23$ (no. 196). The crystal structure consists of $[Mo_3S_7(dtp)_3]^+$, I^-, and $(HgI_3)^-$ ions and water of crstallization.

The ORTEP drawing of the $[Mo_3S_7(dtp)_3\cdot I_{1/4}]^{3/4+}$ fragment and the structure of the tetramer $\{[Mo_3S_7(dtp)_3]_4\cdot I\}^{3+}$ are shown in Figure 1 and Figure 2, respectively. It can be seen from Figure 1 that the $[Mo_3S_7(dtp)_3\cdot I_{1/4}]^{3/4+}$ unit contains a typical $[Mo_3(\mu_3\text{-}S)(\mu\text{-}S_2)_3]$ cluster core,[14–16] in which the μ_3-S atom is located on the threefold axis, thus the three Mo atoms form an equilateral triangle. The Mo–Mo bond length of

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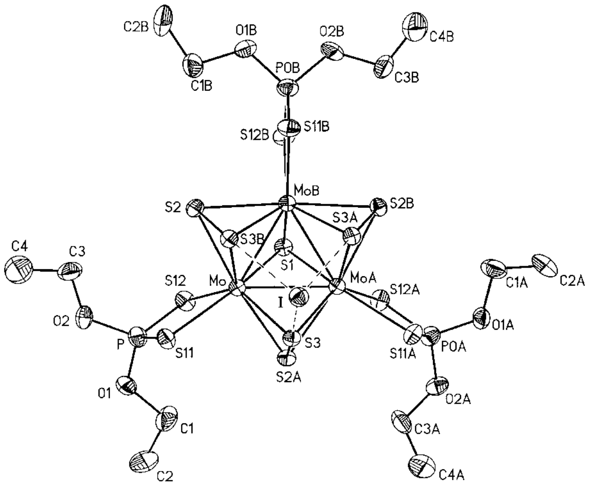

Figure 1. Structure of $[\mathrm{Mo_3S_7(dtp)_3}\cdot\mathrm{I_{1/4}}]^{+3/4}$ (ORTEP diagram with thermal ellipsoids at 20% probability).

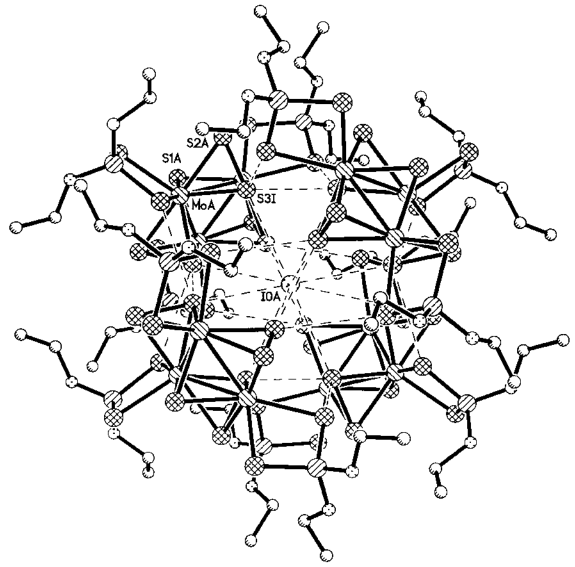

Figure 2. The structure of the tetramer $\{[\mathrm{Mo_3S_7(dtp)_3}]_4\cdot\mathrm{I}\}^{3+}$.

2.734(2) Å is slightly longer than that in the discrete $[\mathrm{Mo_3S_7(dtp)_3I}](\mathrm{CH_3C_6H_5})$ molecule (2.723(1) Å).[15d]The bridging $\mathrm{S_2}^{2-}$ group can generally be described by $\mathrm{S_{eq}}$–$\mathrm{S_{ax}}$, where in this case $\mathrm{S_{eq}}$ refers to the S atom lying in the $\{\mathrm{Mo_3}\}$ plane (denoted S2), whereas $\mathrm{S_{ax}}$ refers to the S atom lying out of the $\{\mathrm{Mo_3}\}$ plane (denoted S3). The $\mathrm{S_{eq}}$–$\mathrm{S_{ax}}$ bond length is 2.036(6) Å, which is slightly shorter than that in the discrete $[\mathrm{Mo_3S_7(dtp)_3I}]\cdot(\mathrm{CH_3C_6H_5})$ molecule (2.055 Å).

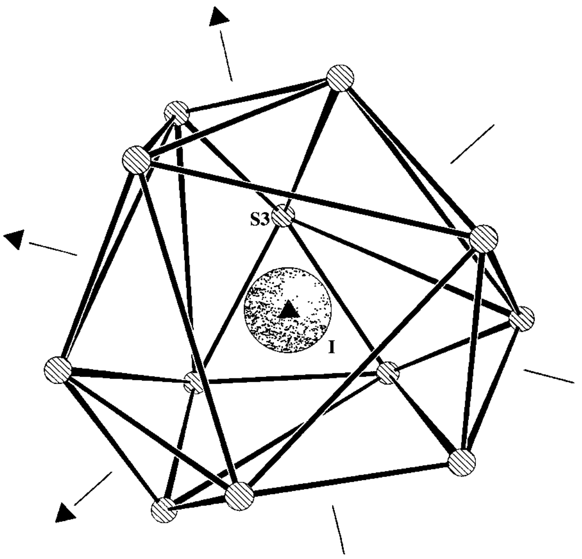

Figure 3. The $\mathrm{S_{12}}$ icosahedron formed from 12 symmetry-equivalent S3 atoms about a central I atom. The I ··· S3 distance is 3.590(4) Å, and the S3 ··· S3 distance is 3.197(8) Å.

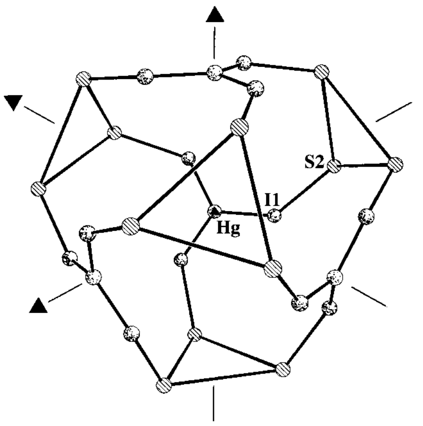

Figure 4. The $\mathrm{S_{12}}$ icosahedron formed from 12 symmetry-equivalent S2 atoms connected with $\mathrm{HgI_3}^-$. The I1 ··· S2distance is 3.405(5) Å, the Hg–I bond length is 2.645(2) Å, and the I-Hg-I bond angle is 119.98(1)°.

The first striking structural feature of the present molecule is that four $[\mathrm{Mo_3S_7(dtp)_3}]^+$ ions are tetramerized about a common I atom; the I ··· $\mathrm{S_{ax}}$ distance of 3.590(4) Å is significantly longer than the value of 3.172 Å found in the discrete $[\mathrm{Mo_3S_7(dtp)_3I}]\cdot(\mathrm{CH_3C_6H_5})$ molecule. This is attributed to the increase in the coordination number of the I atom (12-coordinate, whereas the I atom is only three-coordinate in the discrete cluster). As a result, a distorted $\mathrm{S_{12}}$ icosahedron is formed by 12 symmetry-equivalent S3 atoms around the central I^- ion, which lies on a special position of the *F*23 space group (Figure 3). This chiral polyhedron has true crystallographic *T* symmetry, which is an esthetically pleasing geometrical feature.

Interestingly, if one $[\mathrm{Mo_3S_7(dtp)_3}]^+$ unit is taken from each of the four $\{[\mathrm{Mo_3S_7(dtp)_3}]_4\cdot\mathrm{I}\}$ tetramers, then the 12 symmetry-equivalent S2 atoms from these four $[\mathrm{Mo_3S_7(dtp)_3}]^+$ units can form a much larger distorted $\mathrm{S_{12}}$ icosahedron in a tetrahedral cavity (Figure 4). Three $\mathrm{HgI_3}^-$ units are statistically located on three of the four faces of the tetrahedron and form a weak interaction with $[\mathrm{Mo_3S_7(dtp)_3}]^+$ through a I ··· $\mathrm{S_{eq}}$ contact (3.405(5) Å). The Hg atom also lies on the threefold axis and its displacement from the $\{\mathrm{I_3}\}$ plane is 0.0389 Å. Thus, Hg is almost coplanar with the $\{\mathrm{I_3}\}$ plane; the Hg–I bond length is 2.645(2) Å and the I-Hg-I bond angle is 119.98(1)°. In addition, four oxygen atoms of the water of crystallization are situated on four threefold axes of this tetrahedron.

All the I ··· S distances are much longer than a I–S covalent bond (~2.5 Å[17]), but much shorter than the sum of the corresponding I$\cdots$S van der Waals radii (~4.0 Å[18]). The $[Mo_3S_7(dtp)_3]^+$ units form a three-dimensional network with I^-, $(HgI_3)^-$ ions, and molecules of water of crystallization; the tetrahedral meshes are held together by weak I$\cdots$S interactions as shown in Figure 5.

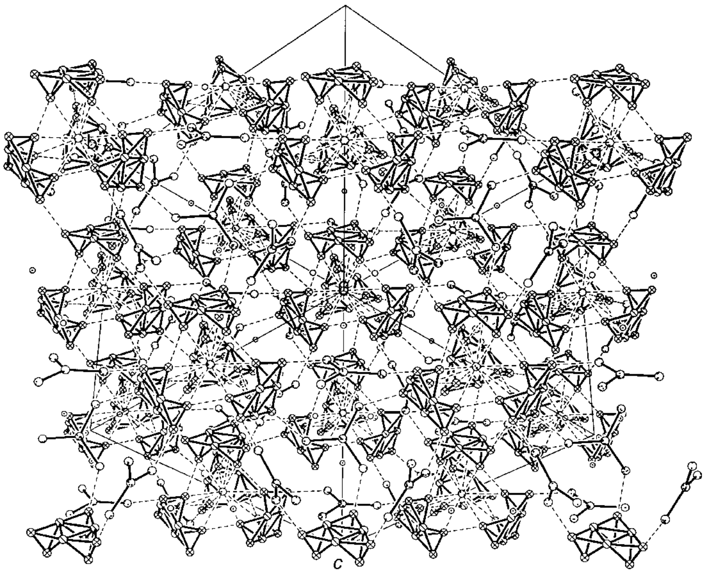

Figure 5. The network-like packing structure of $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4H_2O$ (**1**).

To date, only the $[Mo_3S_7]$ tetramer $[Mo_3S_7(dtc)_3]_4\cdot(SO_4)_2\cdot 3THF$ has been reported in the literature (dtc = diethyldithiocarbamate).[19] The present crystal structure, however, contains another metal compound $(HgI_3)^-$ and exhibits the uncommon interaction between the $\{[Mo_3S_7(dtp)_3]_4\cdot I\}$ tetramer and $(HgI_3)^-$.

Spectroscopic characterization and I$\cdots$S charge transfer:[20] IR and Raman spectra reveal that the vibration band of ν(S–S) appears at 532–545 cm^{-1} and 541–544 cm^{-1}, respectively, slightly higher than those (530–544 cm^{-1} and 537cm^{-1}) in the discrete cluster $[Mo_3S_7(dtp)_3\cdot I]$.[21] This indicates the effects of the interaction of S_2^{2-} with I^- and $(HgI_3)^-$. The I$\cdots$S charge transfer leads to the increase of S_{eq}–S_{ax} force constant, that is, the ν(S–S) frequency. This is consistent with the conclusion by Hegetschweiler et al. that the force constants of S_{eq}–S_{ax} bonds are rather sensitive to the nature of the anion and correlates with the S$\cdots$X distance (X = halogen).[19] The other bands for the $[Mo_3(\mu_3\text{-}S)(\mu\text{-}S_2)_3]$ core are assigned as follows: ν(Mo-μ_3-S) 447cm^{-1}, ν(Mo–S_{ax}) 397cm^{-1}, ν(Mo–S_{eq}) 297cm^{-1}, which is in agreement with the assignment by Zimmermann et al.[15a] and Müller et al.[22] In addition, the ν(Mo–Mo) band appears at 174 cm^{-1} also in accordance with that assigned by Saito et al.(170–200 cm^{-1}).[4] The absorption band at about 3450 cm^{-1} is consistent with the presence of the water of crystallization in the structure.

The electronic absorption spectra exhibit a high-energy absorption at 242 nm ($\varepsilon = 2.2\times10^5\,M^{-1}cm^{-1}$) and a shoulder at 352 nm ($\varepsilon = 5.5\times10^4\,M^{-1}cm^{-1}$). The former can be assigned as an intraligand transition of dtp, while the latter is likely to be characteristic of the $[Mo_3(\mu_3\text{-}S)\,(\mu\text{-}S_2)_3]$ core.[15a, 15c] The latter is red-shifted from that of the discrete cluster (≈322 nm), which is also due to the effect of I$\cdots$S charge transfer.

In the ^{95}Mo NMR spectrum, there is a sharp peak at $\delta = -511.5$ with a linewidth of 681 Hz; the chemical shift is thus 9 ppm less than that for $\{Mo_3S_7[S_2P(OC_3H_7)_2]_3\cdot I\}$ ($\delta = -502.3$).[23] This is presumably due to the change of the Mo–Mo bonding on tetramerization of the $\{Mo_3S_7\}$ core.[24] The ^{31}P NMR spectrum is also very simple; a single resonance is observed at $\delta = 96.19$ which is higher than that in $\{Mo_3S_7[S_2P(OC_3H_7)_2]_3\cdot I\}$($\delta = 93.46$).[23]

Conclusion

A novel cluster compound $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4H_2O$ has been obtained through a recombination–polymerization reaction. Although the cluster cannot be considered to be an addition cluster in a strict sense, the synthetic route is apparently a new one. The I$\cdots$S interaction between $\{[Mo_3S_7(dtp)_3]_4\cdot I\}$ and $(HgI_3)^-$ is of great significance in both structurally and in terms of studies on the electromagnetic properties. Efforts are currently underway to increase the yield of the reaction and to study the structure–property relationships.

Experimental Section

General procedure: All reactions were carried out in air. Reagents were commercially available and used without further purification. C, H elemental analyses were performed on an EA1110 CHNS-0 CE instrument; Mo was analyzed with spectrophotometric determination; Hg, S analyses were carried out with a Bruker SRS 3400 fluorescence spectrometer. IR spectra were recorded on a Nicolet Magna 750 FTIR spectrometer with KBr discs (4000–600 cm^{-1}) and CsI discs (600–100 cm^{-1}). Raman spectra were measured on a 910 laser Raman FT Spectrometer with SP grade KBr. UV/Vis spectra were obtained on a Shimadzu UV-3000 spectrometer. NMR were measured on a Varian Unity 500 spectrometer at ambient temperature (20 °C). The samples were dissolved in CH_2Cl_2, the chemical shifts were referenced to an external standard of Na_2MoO_4 (2 M) in D_2O for the ^{95}Mo NMR spectra and 80 % H_3PO_4 for the ^{31}P NMR spectra. ^{13}C NMR spectra were measured in $CDCl_3/CH_2Cl_2$, and ^1H NMR spectra were measured in $CDCl_3$.

Synthesis of 1: A mixture of $[Mo_3(\mu_3\text{-}O)S_3(dtp)_4(H_2O)]$ and $[Mo_3(\mu_3\text{-}S)S_3(dtp)_4(H_2O)]$ was prepared as previously reported.[25] This mixture (0.116 g, ca. 0.1 mmol), HgI_2 (0.182 g, 0.4 mmol), and Bu_4NI (0.103 g, 0.28 mmol) were added to a solvent mixture of CH_2Cl_2 (20 mL) and CH_3CN (20 mL). After the mixture had been stirred under reflux for about one hour, the remaining solid was removed by filtration, and the filtrate was allowed to evaporate at room temperature in air. Dark red-black crystals formed over about one and half months. These were filtered off, washed with ethanol and petroleum (60–90 °C), and dried to give **1** (0.030 g, 19.3 %). A subsequent X-ray structure analysis confirmed that the formula was $\{[Mo_3S_7(dtp)_3]_4\cdot I\}\{(HgI_3)_3\}\cdot 4H_2O$. IR (KBr): $\tilde{\nu} = 532$ (S_{aq}–S_{ex}), 447 (Mo-μ_3-S), 397 (Mo–S_{ax}), 297 (Mo–S_{eq}), 174 cm^{-1} (Mo–Mo); Raman (CsI): $\tilde{\nu} = 541\,cm^{-1}$ (S_{aq}–S_{ex}); ^1H NMR (499.8 MHz, $CDCl_3$, 20 °C, TMS): $\delta = 1.4$ (t, ^3J(H,H) = 7.0 Hz, 3H; CH_3), 4.20 (q, ^3J(H,H) = 7.0 Hz, 2H; CH_2); ^{13}C NMR (125.7 MHz, $CDCl_3/CH_2Cl_2$, decoupled, 20 °C): $\delta = 15.8$ (CH_3), 65.2 (CH_2); ^{31}P NMR (202.4 MHz, CH_2Cl_2, 20 °C, 80 % H_3PO_4): $\delta = 96.19$; ^{95}Mo NMR (32.56 Mhz, CH_2Cl_2, 20 °C, 2 M Na_2MoO_4): $\delta = -511.5$; UV/Vis (CH_2Cl_2): $\lambda_{max}(\varepsilon) = 242$ nm ($2.2\times10^5\,M^{-1}cm^{-1}$), 352 nm ($5.5\times10^4\,M^{-1}cm^{-1}$); elemental analysis calcd (%) for $C_{48}H_{128}Hg_3I_{10}Mo_{12}O_{28}P_{12}S_{52}$: C 9.28, H 2.08, Hg 9.68, Mo 18.53, S 26.83; found: C 9.20, H 2.07, Hg 10.26, Mo 18.5, S 24.35.

Crystal structural analysis: A crystal of the dimensions 0.25 × 0.22 × 0.19 mm was attached to the end of a glass fiber by using neutral jelly and then mounted on a Siemens SMART CCD diffractometer. The structure was solved by direct methods by using $Mo_K\alpha$ radiation ($\lambda = 0.71073$ Å) with the SHELXTL-5 program.[26]The non-hydrogen atoms were located by successive difference Fourier syntheses. The structure was then refined by full-matrix least-squares on F^2. Anisotropic thermal factors were applied for all the non-hydrogen atoms. The idealized positions of the hydrogen atoms were located by using a riding model. The hydrogen atoms were included in the structure factor calculations without refinement. Details of structure analysis are summarized in Table 1. The final atomic fractional coordinates and U_{eq} of all non-hydrogen atoms are listed in Table 2 and selected bond lengths and angles are given in Table 3. Notably, when the occupation probability of the Hg atom located in threefold axis and the I(1) atom in a general position in the $(HgI_3)^-$ ions were 0.333 and 1, respectively, the temperature factors were unexpectedly high. According to the electrovalent balance principle, therefore, their occupancy factors were taken to be only three quarters of their original values. Thus, $(HgI_3)^-$ occupies statistically only three of the four possible positions. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-149896. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: (+44) 1223-336033; e-mail: deposit@ccdc.cam.ac.uk).

Table 1. Summary of the crystallographic data and data collection parameters for the structure determination of $\{[Mo_3S_7(dtp)_3]_4 \cdot I\} \cdot (HgI_3)_3 \cdot 4H_2O$ (**1**).

empirical formula	$C_{48}H_{128}Hg_3I_{10}Mo_{12}O_{28}P_{12}S_{52}$
formula weight	6214.31
crystal dimensions [mm]	0.25 × 0.22 × 0.19
crystal system	cubic
space group	$F23$ (no. 196)
a [Å]	26.7860(3)
V [Å^3]	19218.7(4)
Z	4
ρ_{calcd} [g cm^{-1}]	2.148
$\mu(Mo_{K\alpha})$ [mm^{-1}]	5.454
radiation, λ [Å]	0.71073
scan mode	$\omega - 2\theta$
$F(000)$	11704
temperature of measurement [K]	293(2)
$2\theta_{max}$ [°]	50.04
no. of measured and independent reflections	19353, 2846
no. of reflections included in refinement	2846
method of refinement	full-matrix least-squares on F^2
no. of refined parameters	129
min./max. transmission	0.7408/1.0000
$R(wR)[I > 2\sigma(I)]$[a]	0.0593 (0.1484)
goodness of fit indicator, S[b]	0.858
$\Delta\rho$(max, min)[e Å^{-3}]	1.111 (1.46 Å from Hg) – 0.666 (1.46 Å from S(3))
Δ/σ	0
Flack parameter	– 0.02(2)

[a] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. $wR = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$. $w = [\sigma^2(F_o^2) + (0.1350P)^2 + 180.2218P]^{-1}$. $P = (F_o^2 + 2F_c^2)/3$. [b] $S = [\Sigma w(|F_o| - |F_c|)^2/(N_{obs} - N_{param})]^{1/2}$.

Table 3. Selected bond lengths [Å] and angles [°] for **1**.[a]

Mo–MoA$^{\#4}$	2.734(2)	Mo–S1	2.378(5)	Mo–S2	2.504(4)
Mo–S3	2.400(4)	Mo–S2A$^{\#4}$	2.492(4)	Mo–S3B$^{\#3}$	2.377(4)
Mo–S11	2.550(5)	Mo–S12	2.499(5)	S2–S3B$^{\#3}$	2.036(6)
S12–P	2.003(8)	S11–P	1.995(7)	Hg–I1	2.645(2)
I···S3	3.590(4)	S3···S3$^{\#5}$	3.197(8)	I1···S2	3.405(5)
S1-Mo-S2	85.97(11)	S1-Mo-S3	109.15(13)	S1-Mo-S11	160.56(16)
S1-Mo-S12	82.73(16)	S2-Mo-S2A$^{\#4}$	172.04(16)	S2-Mo-S11	94.50(16)
S2-Mo-S12	89.75(17)	S2-Mo-MoB$^{\#3}$	56.62(12)	S3-Mo-S2	132.80(15)
S3B$^{\#3}$-Mo-S2	49.21(13)	S3-Mo-S11	84.73(15)	S3B$^{\#3}$-Mo-S3	83.9(2)
S12-Mo-S11	77.84(16)	S3-Mo-S12	135.31(18)		
MoB$^{\#3}$-Mo-MoA$^{\#4}$	60.0				
S3B$^{\#3}$-S2-Mo	62.13(16)	MoB$^{\#3}$-S2-Mo	66.35(12)	MoB$^{\#3}$-S1-Mo	70.15(17)
S2A$^{\#4}$-S3-Mo	67.79(17)	MoA$^{\#4}$-S3-Mo	69.82(13)	P-S11-Mo	87.9(2)
P-S12-Mo	97.1(8)	I1-Hg-I1$^{\#2}$	119.98(1)		

[a] Symmetry transformations: #1: $z + 1/2, -x + 1/2, -y$; #2: $-y + 1/2, -z, x - 1/2$; #3: $y + 1/2$, z, $x - 1/2$; #4 $z + 1/2, x - 1/2, y$; #5: $-x + 3/2, y, -z + 1/2$.

Table 2. Atomic coordinated and equivalent isotropic temperature factors for non-hydrogen atoms of cluster **1**.

Atom	x	y	Z	$U_{(eq)}$[a]	Occupancy factor
Hg	0.6197(1)	– 0.1197(1)	0.1197(1)	0.110(1)	0.25
I	0.7500	0.2500	0.2500	0.055(1)	0.0833
I(1)	0.5523(1)	– 0.1129(2)	0.1915(1)	0.175(2)	0.75
Mo	0.6371(1)	0.1129(1)	0.1941(1)	0.047(1)	1
S(1)	0.6097(1)	0.1097(1)	0.1097(1)	0.053(2)	0.3333
S(2)	0.5618(2)	0.1661(2)	0.2096(2)	0.056(1)	1
S(3)	0.7250(1)	0.1300(2)	0.1958(2)	0.050(1)	1
S(11)	0.6487(2)	0.0896(2)	0.2856(2)	0.067(1)	1
S(12)	0.5842(2)	0.0365(2)	0.2018(2)	0.070(1)	1
P	0.6020(2)	0.0328(2)	0.2753(2)	0.072(1)	1
O(1)	0.6231(5)	– 0.0191(5)	0.2899(5)	0.083(4)	1
O(2)	0.5563(5)	0.0333(5)	0.3106(5)	0.081(4)	1
O(3)	0.6946(15)	– 0.1946(15)	0.1946(15)	0.210(20)	0.3333
C(1)	0.6689(11)	– 0.0383(9)	0.2649(12)	0.122(10)	1
C(2)	0.6830(13)	– 0.0825(14)	0.2926(13)	0.164(15)	1
C(3)	0.5241(9)	0.0766(9)	0.3165(12)	0.108(9)	1
C(4)	0.5006(14)	0.0761(13)	0.3627(12)	0.152(12)	1

[a] Equivalent isotropic temperature factor U_{eq} [Å^2] is defined as one third of the trace of the orthogonalized U_{ij} tensor.

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