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Synthesis and X-ray crystallographic characterization of two different inorganic–organic hybrid isopolyoxomolybdates with α-dipropylammonium *N*,*N*-diisobutylacetamide by varying reaction conditions

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ABSTRACT

Two new polyoxomolybdate based inorganic–organic hybrid compounds have been obtained from same ammonium ion ligand **1** and molybdate source just by altering reaction conditions. The new hybrid polyoxomolybdate complexes $(LH)_2[Mo_6O_{19}]$ **3** and $(LH)_4[Mo_8O_{26}]\ (MeCN)_2$ **4** have distinct structural features revealed by X-ray crystallographic studies. The complex **3** contains two ammonium ligands to balance the charge on hexamolybdate dianion. The most striking feature of $(LH)_2[Mo_6O_{19}]$ is the presence of inter-ligand hydrogen bonding (N—H···O=C) between two ligands arranged in an antiparallel fashion that favors the formation of a net like structure which embeds the Mo_6O_{19} dianion. The complex **4** contains four ammonium ligands to balance the charge on β-octamolybdate tetraanion. Interestingly, complex **4** exhibits strong hydrogen-bonding interactions between ammonium ligand and octamolybdate anion (N—H···O—Mo) and no inter-ligand H-bonding interaction. The different structural features of hybrid compounds **3** and **4** suggest that different polyoxomolybdate clusters play a key role in the process of assembling of ligands.

1. Introduction

Molybdenum compounds are popular target for studies because of their importance in biological systems [1–3], interesting structural features [3,4] and extensive applications in various fields of sciences [5]. Besides normal compounds and complexes, Mo forms extensive range of oxometalate clusters consists of isopolyacids or heteropolyacids [3–6]. The Lindqvist anionic hexamolybdate $[Mo_6O_{19}]^{2-}$ has been structurally characterized as isolated salt in solid state [7]. The interesting aspects of structure chemistry of the octamolybdate anion, $[Mo_8O_{26}]^{4-}$ is seen in varied structural patterns in the solid state [8]. Seven isomeric forms viz. α–η of octamolybdates, $[Mo_8O_{26}]^{4-}$ are known [8–10]. The structures differ in the types of polyhedron that fuse to form the cluster and in the linkage between polyhedra.

Several research attempts have been made to functionalize the polyoxomolybdates (POMs) with an organic component which gives rise to the formation of inorganic–organic hybrid materials where POMs work as building blocks. As the POM anions contain many oxygen atoms which act as good hydrogen bond acceptors, fictionalization of these POMs with organic molecules having many hydrogen atoms give rise to interesting supramolecular chemistry [11–13]. The supramolecular interactions are mediated by various type of hydrogen bonds like O—H···O, N—H···O, C—H···O, O—H···N, etc. [11,12].

Quaternary ammonium salt such as Aliquat and tertiary amine like Alamines are generally used for extraction of Mo from weakly acidic aqueous medium [14–16]. Crystal structure of POM anion with Bu_4N^+ as a mixed $[Mo_6O_{19}]^{2-}$ and $[Mo_8O_{26}]^{4-}$, i.e., $[n\text{-}Bu_4N]_8[Mo_6O_{19}]_2[\alpha\text{-}(Mo_8O_{26})]$ [17] has been reported recently. Crystals of $[Mo_6O_{19}]^{2-}$ and $[Mo_8O_{26}]^{4-}$ have also been made using different organic ligands [12,18–20]. Complexes of POM with organic amino type ligands associated with transition metals such as Cu or Co have been prepared and characterized by X-ray crystallography [21–24]. But, individual complexes of $[Mo_6O_{19}]^{2-}$ and $[Mo_8O_{26}]^{4-}$ with the same ligand are not known. Recently, we have developed α-dipropylamino *N*,*N*-diisobutylacetamide (L) **1** (Scheme 1) as an

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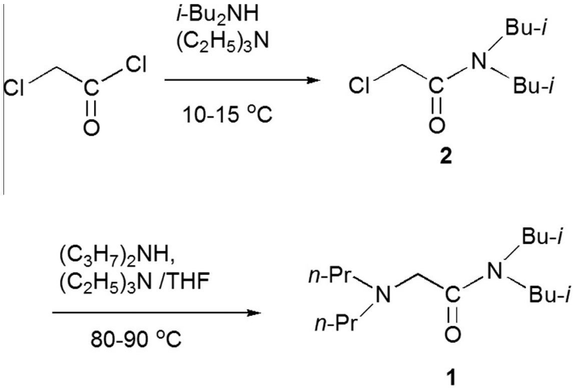
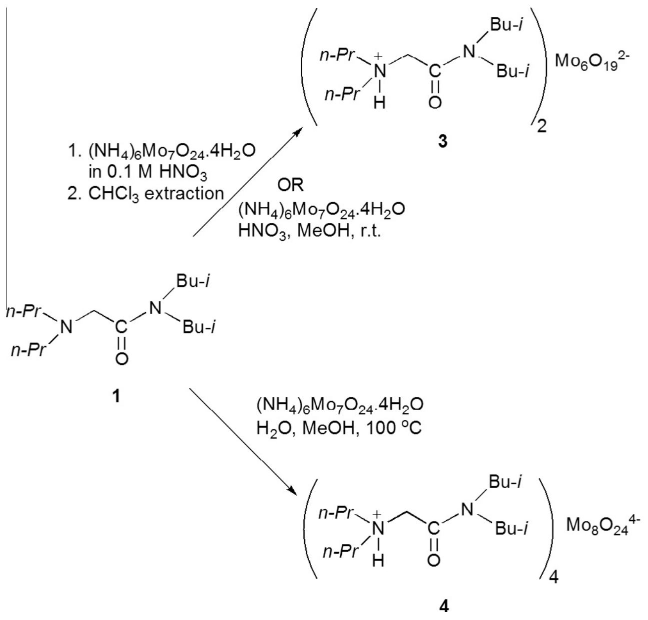


Scheme 1. Synthesis of α-dipropylamino *N*,*N*-diisobutylacetamide **1**.



Scheme 2. Synthesis of metal–ligand complexes **3** and **4**.

amine based extractant for Mo with an aim for its recovery from spent nuclear fuel dissolved in nitric acid where other amine based extractants did not work [25]. Considering the great importance of inorganic–organic hybrid materials of polyoxomolybdates, we herein report the condition dependent crystal engineered syntheses of $(LH^+)_2[Mo_6O_{19}]^{2-}$ and $(LH^+)_4[Mo_8O_{26}]^{4-}$ complexes and their X-ray crystallographic characterization.

2. Results and discussion

α-Dipropylamino *N*,*N*-diisobutylacetamide **1** was synthesized following our reported procedure [25] from the commercially available starting materials as described in Scheme 1. For this, chloroacetyl chloride was reacted with diisobutylamine in the presence of triethylamine to give the amide **2** which was subsequently reacted with diisopropylamine to give the desired α-dipropylamino *N*,*N*-diisobutylacetamide **1** in very good overall yield.

When the chloroform solution of ligand **1** was repeatedly contacted with fresh solution of $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ in 0.1 M aqueous HNO_3 and the resultant organic phase was concentrated, a yellow precipitate was obtained which on crystallization from acetonitrile provided a yellow complex $(LH)_2[Mo_6O_{19}]$ **3** (Scheme 2). The same complex **3** was achieved when a 0.5 M nitric acid solution of $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ and a methanolic solution of ligand **1** were mixed and stirred at room temperature. Interestingly, when $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ was dissolved in water, mixed with a methanolic solution of ligand **1** and heated at 100 °C, a colorless complex $(LH)_4[Mo_8O_{26}]$ **4** was formed.

Table 1
Crystallographic data and details of structure refinement for complexes **3** and **4**.

	3	4
Empirical formula	Mo_6O_{19}, $2(C_{16}H_{35}N_2O)$	Mo_8O_{26}, $4(C_{16}H_{35}N_2O)$, $2(C_2H_3N)$
Formula weight	1422.56	2351.46
T (K)	293(2)	293(2)
λ (Å)	1.5418	1.5418
Crystal system	monoclinic	triclinic
Space group	*I*2/*a*	$P\bar{1}$
a (Å)	24.8703(11)	11.8091(11)
b (Å)	11.1774(3)	12.1612(11)
c (Å)	20.4442(9)	18.2009(13)
α (°)	90	77.755(7)
β (°)	119.195(6)	81.247(7)
γ (°)	90	87.642(7)
V ($Å^3$)	4961.2(4)	2524.6(4)
Z	4	1
D_{calc} (g/cm^3)	1.905	1.547
μ (mm^{-1})	12.706	8.471
F(000)	2840.0	1200.0
Crystal size (mm^3)	0.4585 × 0.288 × 0.1217	0.5939 × 0.3818 × 0.1877
θ max (°)	69.865	70.096
Reflections collected/unique	8318/4616	16607/9390
R_{int}	0.0621	0.0809
Data/restraints/parameters	4616/48/332	9390/24/535
Goodness-of-fit on F^2	1.010	1.133
R_1/wR_2 $[I \geqslant 2\sigma(I)]$	0.0943/0.2491	0.1100/0.2873
R_1/wR_2 (all data)	0.1161/0.2800	0.1319/0.3204
Residuals (e $Å^{-3}$)	1.92, −3.29	3.48, −2.54

2.1. Synthesis of complex $(LH)_2[Mo_6O_{19}]$ 3 (route A)

A solution of acetamide **1** (271 mg, 1 mmol) in chloroform (5 mL) was repeatedly contacted with fresh solution of $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ (46 mg, 0.037 mmol) in 0.1 M aqueous HNO_3 (5 mL). The chloroform extract was concentrated under reduced pressure to give a yellow precipitate of metal ligand complex. The precipitate was triturated with 10% ethyl acetate–hexane solution to remove excess ligand **1**. The solid residue was then crystallized from acetonitrile to obtain the yellow crystalline complex **3** (603 mg, 85%). IR (KBr): $\bar{\nu}$ 3450, 3109, 2960, 2877, 1643 (C=O), 1464, 1387, 1257, 1157, 953 (Mo=O), 800, 592 (Mo—O—Mo), 436 cm^{-1}.

2.2. Synthesis of Mo crystal $(LH)_2[Mo_6O_{19}]$ 3 (route B)

A solution of $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ (84 mg, 0.068 mmol) in 0.5 M HNO_3 (3 mL) was added to a solution of acetamide **1** (55 mg, 0.204 mmol) in MeOH (2 mL) and the reaction mixture was stirred at room temperature for 5 h. The reaction mixture was filtered and the light yellow precipitate was washed with 10% ethyl acetate–hexane solution to remove the excess ligand **1**. The yellow powder was crystallized from acetonitrile to give yellow crystals of complex **3** (79 mg, 81%).

2.3. Synthesis of Mo crystal $(LH)_4[Mo_8O_{26}]$ $(MeCN)_2$ 4

A solution of $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ (42 mg, 0.034 mmol) in water (2 mL) was added to a solution of acetamide **1** (64 mg, 0.238 mmol) in MeOH (1 mL). The reaction mixture was stirred at 100 °C for 3 h, brought to room temperature and concentrated under reduced pressure. The solid residue was washed with 10% ethyl acetate–hexane solution to remove the excess ligand **1**. The

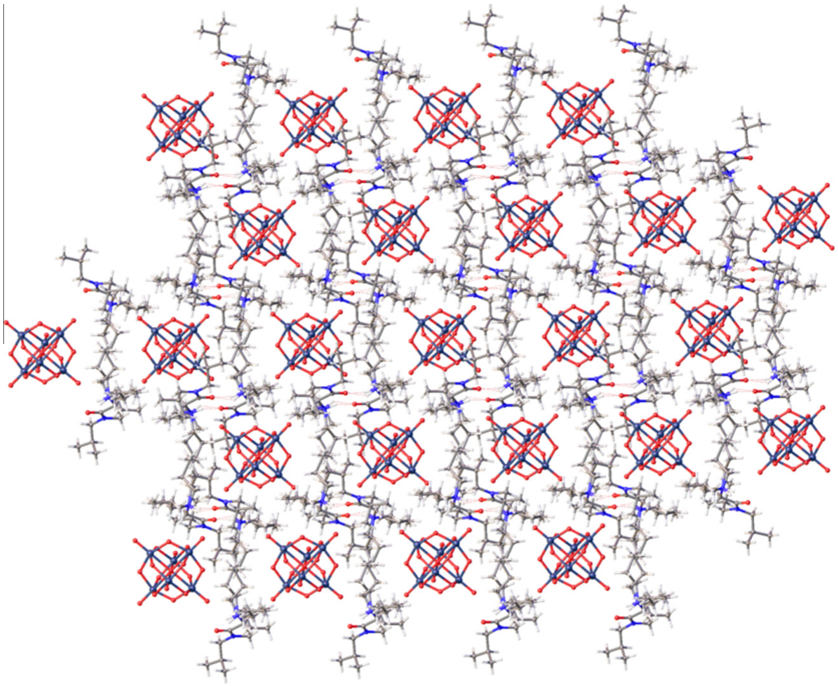


Fig. 1. Cluster packing of complex **3**, $(LH)_2[Mo_6O_{19}]$, in the crystal. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

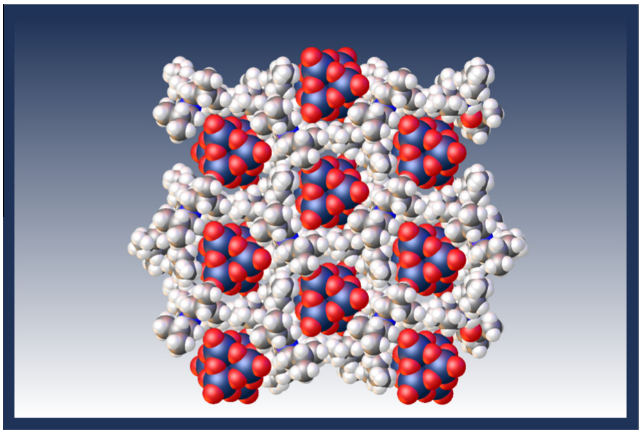


Fig. 2. Space filled cluster packing of complex **3**, $(LH)_2[Mo_6O_{19}]$, in the crystal. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

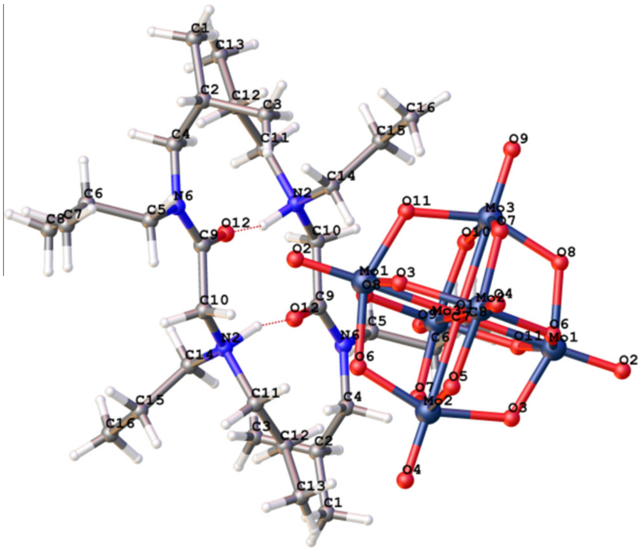


Fig. 3. The hexamolybdate anion with a pair of anti-parallel cationic ligand in complex **3**, $(LH)_2[Mo_6O_{19}]$. The inter ligand hydrogen bonds (N—H···O=C) are shown as red dotted lines. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

white powder was crystallized from acetonitrile to give colorless crystals of complex **4** (59 mg, 74%). IR (KBr): $\bar{\nu}$ 3467, 2964, 2939, 2875, 1647 (C=O), 1471, 1431, 1369, 944, 913 (Mo=O), 848, 714, 664 (Mo—O—Mo), 557, 525 cm^{-1}.

*2.4. Crystallographic characterization of complexes **3** and **4***

Single crystal X-ray diffraction data were collected on Agilent Supernova system equipped with a microfocus Cu-source (λ = 1.5418 Å) and a Titan CCD detector. The crystals were separated, coated with paraffin oil and mounted on a loop for X-ray diffraction data collection at room temperature (293 K). The data reduction and analysis were carried out with CrysAlisPro software suit. Analytical absorption correction using a multifaceted crystal model based on expressions derived by Clark and Reid [26] and as implemented in the CrysAlisPro software suit was carried out for both the crystals. The structures were solved by direct method using SHELXS and refined using SHELXL softwares [27] using Olex2 interface [28]. All the nonhydrogen atoms were refined anisotropically and hydrogens were generated at their idealized positions and refined isotropically according to riding model.

The complex **3** crystallizes in the monoclinic space group $I2/a$ while complex **4** crystallizes in the triclinic space group $P\bar{1}$. The crystal data and the refinement statistics is shown in Table 1.

The crystal structure of complex **3** shows that the protonated ligand (LH$^+$) **1** forms a cage-like structure in which the Mo_6O_{19} fits in the cavity (Figs. 1 and 2). The counterions (LH$^+$)-**1** regularly distribute in the interspaces formed by the polyanions. A pair of (LH$^+$)-**1** ligand molecules orient in anti-parallel fashion with formation of two inter-ligand hydrogen bonds between amide oxygen atom of one ligand molecule and the quaternary nitrogen

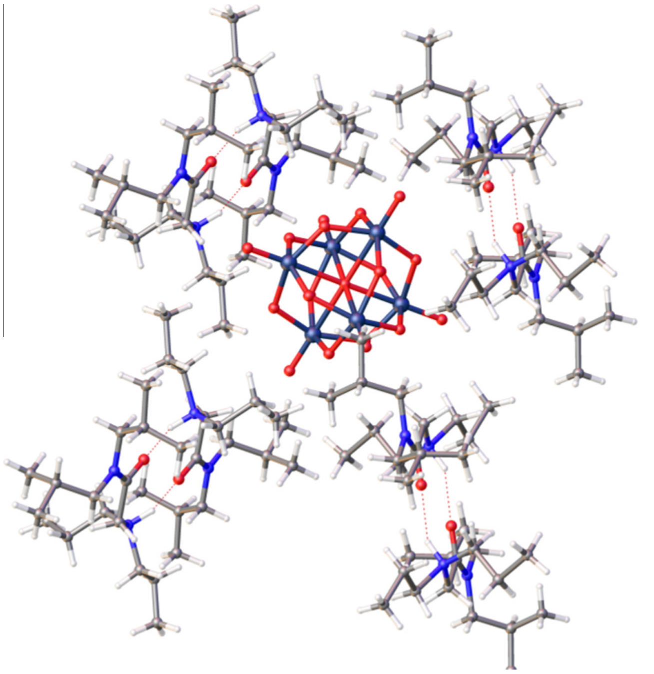


Fig. 4. The hexamolybdate anion surrounded by four anti-parallel dimeric units of ligand in the complex **3**, $(LH)_2[Mo_6O_{19}]$. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

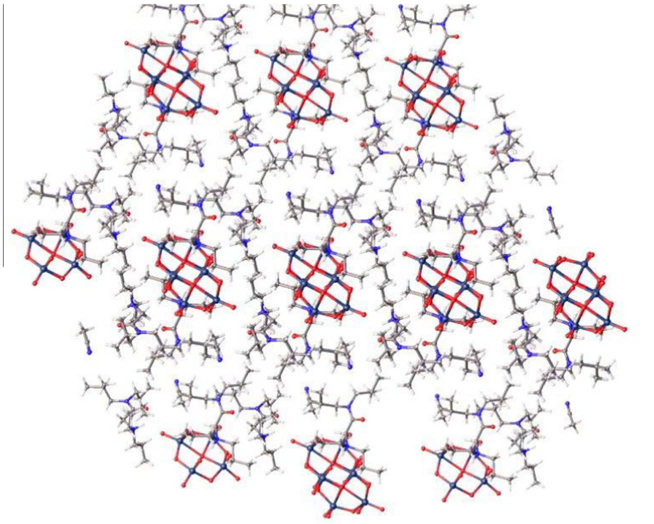


Fig. 5. Cluster packing of complex **4**, $(LH)_4[Mo_8O_{26}]$ $(MeCN)_2$. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

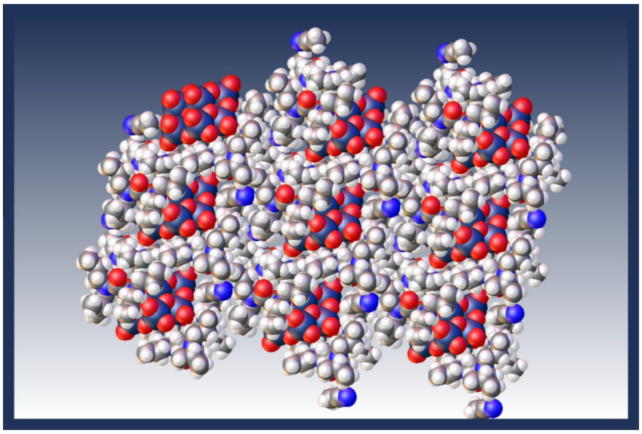


Fig. 6. Space filled cluster packing of complex **4**, $(LH)_4[Mo_8O_{26}]$ $(MeCN)_2$. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

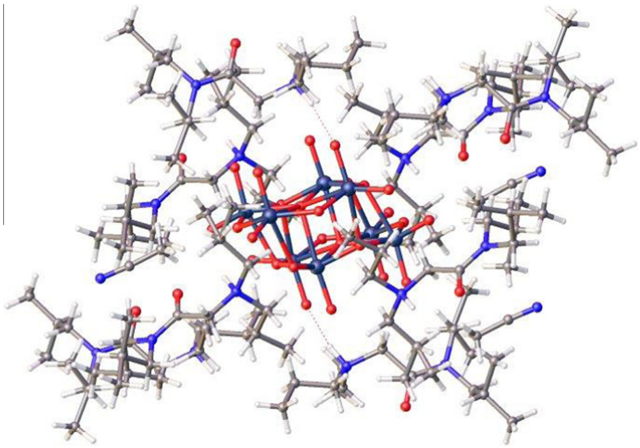


Fig. 7. The octamolybdate anion surrounded by eight ligands in complex **4**, $(LH)_4[Mo_8O_{26}]$ $(MeCN)_2$. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

atom of other ligand molecule (Fig. 3). The inter ligand N—H···O=C H-bond distance is 2.92 Å. Each $[Mo_6O_{19}]^{2-}$ anion is surrounded by six (LH^+)-**1** cations but its surrounding is not uniform. Each cluster anion is surrounded by two H-bonded dimeric (LH^+)-**1** cations and two (LH^+)-**1** cations not H-bonded to each other but to other protonated ligand molecules. The cage-like structure is then formed by hydrophobic interactions among intermolecular hydrogen bonded ligand dimers surrounding the central metal cluster (Fig. 4). The distances of quaternary N in (LH^+)-**1** cations and the nearest Mo in adjacent polyanions range from 4.77 to 5.31 Å. Three kinds of interactions viz. the Van der Waals forces, electrostatic attractions between (LH^+)-**1** cations and polyanions $[Mo_6O_{19}]^{2-}$ and several hydrogen bonds between oxygen atoms of the polyanions and the hydrogens attached to carbon atoms of (LH^+)-**1** have been observed between (LH^+)-**1** cations and polyanion layers. The shortest C—H/O distance observed was 3.41 Å [between C(16) and O(10)]. The structure of $[Mo_6O_{19}]^{2-}$ anion in complex **3** shows slightly distorted cage of six molybdenum atoms located octahedrally around a central oxygen atom, with 12 oxygen atoms disposed in Mo—O—Mo units and one terminal oxygen attached to each molybdenum. Three distinct types of Mo—O bond lengths are observed. The terminal Mo—O bond lengths are shorter with an average bond length of 1.66 Å and the bridging Mo—O bonds have an average bond length of 1.91 Å. The central Mo—O bond lengths are the longest with an average value of 2.32 Å. The exact values are presented in Table 2.

The crystal structure for complex **4** shows that octamolybdate anion is present in β-form and the protonated ligand (LH^+) **1** forms a different cage structure (Figs. 5 and 6) to accommodate the polyanion in the cavity. Each β-$[Mo_8O_{26}]^{4-}$ anion is surrounded by eight (LH^+)-**1** cationic ligands (Fig. 7). Two MeCN molecules per cluster anion are incorporated into the interstitial positions held by hydrophobic interaction with the ligands and a weak C—H···O hydrogen bonding interaction (C···O distance 3.1 Å) with β-$[Mo_8O_{26}]^{4-}$ anion (Fig. 8). The size of the cavity in this cage-structure is bigger than that in complex **3**. The clustering of

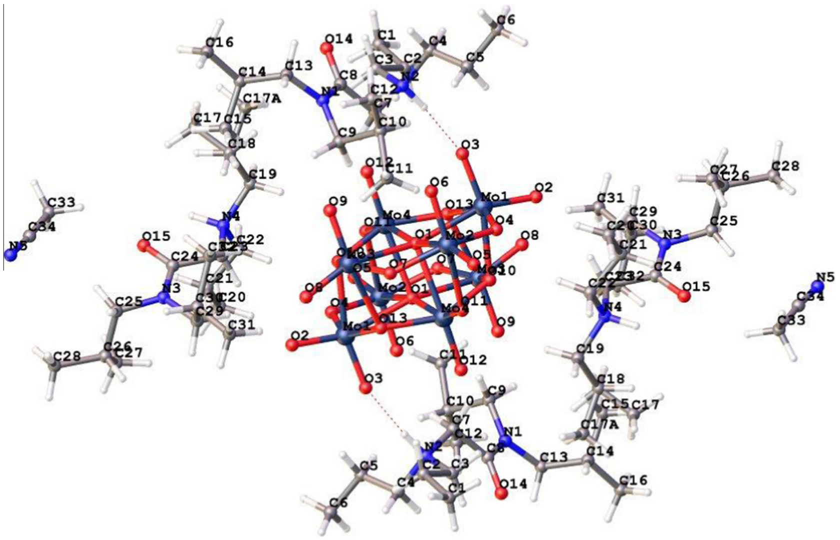


Fig. 8. The hydrogen bonding interactions in complex **4**, $(LH)_4[Mo_8O_{26}]$ $(MeCN)_2$. The ligand N—H···O—Mo bondings are shown as dotted lines. The dark blue balls are Mo; red balls: O; light blue balls: N; gray balls: C; white balls: H. (Color online.)

Table 2
Bond lengths (Å) for complex **3** $(LH)_2[Mo_6O_{19}]$.

Mo3—O10	1.919(5)	O12—C9	1.224(12)
Mo3—O8	1.898(8)	C10—N2	1.476(12)
Mo3—O7	1.910(7)	C10—C9	1.555(14)
Mo3—O9	1.654(7)	N2—C11	1.64(3)
Mo3—O11	1.945(9)	N2—C14	1.38(3)
Mo3—O1	2.329(7)	N2—C11A	1.29(4)
Mo2—O4	1.664(7)	N2—C14A	1.66(4)
Mo2—O7	1.920(7)	C9—N6	1.317(12)
Mo2—O5	1.914(5)	C2—C1	1.517(17)
Mo2—O6	1.898(8)	C2—C3	1.490(18)
Mo2—O1	2.308(7)	C2—C4	1.501(15)
Mo2—O3	1.934(8)	C11—C12	1.52(3)
O10—Mo3[#1]	1.919(5)	C6—C5	1.487(17)
O8—Mo1[#1]	1.937(7)	C6—C7	1.502(17)
O2—Mo1	1.665(7)	C6—C8	1.52(2)
Mo1—O8[#1]	1.937(7)	C5—N6	1.484(13)
Mo1—O11	1.882(9)	C4—N6	1.462(13)
Mo1—O6[#1]	1.951(8)	C15—C16	1.67(2)
Mo1—O1	2.3183(8)	C15—C14	1.58(4)
Mo1—O3	1.893(8)	C12—C13	1.54(2)
O5—Mo2[#1]	1.914(5)	C13—C12A	1.81(4)
O6—Mo1[#1]	1.951(8)	C16—C15A	1.53(3)
O1—Mo3[#1]	2.329(7)	C15A—C14A	1.52(4)
O1—Mo2[#1]	2.308(7)	C12A—C11A	1.50(6)
O1—Mo1[#1]	2.3182(8)		

[#1] Atoms generated by symmetry operation $(-1/2 - X, +Y, -Z)$.

Table 3
Bond lengths (Å) for complex **4**, $(LH)_4[Mo_8O_{26}]$ $(MeCN)_2$.

C1—C2	1.529(16)	C30—C31	1.500(17)
C2—C3	1.510(16)	C30—C32	1.58(2)
C3—N2	1.495(14)	Mo1—O1	2.351(7)
C4—C5	1.460(17)	Mo1—O2	1.729(6)
C4—N2	1.481(14)	Mo1—O3	1.721(7)
C5—C6	1.54(2)	Mo1—O4	1.884(6)
C7—C8	1.518(16)	Mo1—O10[#1]	2.316(7)
C7—N2	1.512(13)	Mo1—O13	1.989(7)
C8—N1	1.346(14)	Mo2—O1	2.482(7)
C8—O14	1.244(13)	Mo2—O4	1.917(7)
C9—C10	1.59(3)	Mo2—O5	1.711(6)
C9—N1	1.484(14)	Mo2—O6	1.719(7)
C10—C11	1.57(3)	Mo2—O7	1.917(8)
C10—C12	1.32(3)	Mo2—O11[#1]	2.271(7)
C13—C14	1.540(19)	Mo3—O1	2.308(7)
C13—N1	1.462(15)	Mo3—O7	1.884(7)
C14—C15	1.47(2)	Mo3—O8	1.695(7)
C14—C16	1.50(2)	Mo3—O9	1.706(7)
C17—C18	1.29(4)	Mo3—O10	1.998(6)
C19—N4	1.510(15)	Mo3—O13[#1]	2.357(7)
C19—C18	1.493(19)	Mo4—O1	2.134(6)
C20—C21	1.519(19)	Mo4—O1[#1]	2.388(7)
C21—C22	1.514(17)	Mo4—O10	1.962(7)
C22—N4	1.510(13)	Mo4—O11	1.753(6)
C23—C24	1.520(14)	Mo4—O12	1.715(7)
C23—N4	1.487(11)	Mo4—O13	1.932(6)
C24—N3	1.351(13)	O1—Mo4[#1]	2.387(7)
C24—O15	1.212(12)	O10—Mo1[#1]	2.316(7)
C25—C26	1.52(2)	O11—Mo2[#1]	2.271(7)
C25—N3	1.464(14)	O13—Mo3[#1]	2.357(7)
C26—C27	1.45(3)	C34—N5	1.19(3)
C26—C28	1.56(2)	C34—C33	1.36(3)
C29—C30	1.486(17)	C17A—C18	1.43(3)
C29—N3	1.476(12)		

[#1] Atoms generated by symmetry operation $(-X, 1 - Y, 2 - Z)$.

ligand here is different and there is no inter-ligand hydrogen bond present as in complex **3**. The quaternary ammonium N atom in each ligand molecule, however, makes hydrogen bond with one of the oxygen atoms of metal cluster as shown in Figs. 7 and 8. Each anionic cluster β-$[Mo_8O_{26}]^{4-}$ is strongly H-bonded to two (LH^+)-**1** and weakly H-bonded to another two (LH^+)-**1** (N—H···O—Mo) with an average bond length of 2.82 and 2.9 Å, respectively. Remaining four (LH^+)-**1** ligands are not H-bonded to the cluster but H-bonded to adjacent cluster anions. Besides H-bonding interactions, there exist three weak kinds of interactions between (LH^+)-**1** cations and inorganic cluster anions: the Van der Waals force, electrostatic attractions between cations (LH^+)-**1** and polyanions β-$[Mo_8O_{26}]^{4-}$ and several H-bond between oxygen atoms of the polyanions and the C—H of (LH^+)-**1** ligands. The shortest C—H/O distance observed was 3.08 Å [between C(7) and O(6)]. The distances of quaternary N in (LH^+)-**1** cations and the nearest Mo in adjacent polyanions range from 4.48 to 4.54 Å. The β-isomeric octamolybdate cluster anion β-$[Mo_8O_{26}]^{4-}$ is made up of Mo_6O_{24} ring capped on opposite faces by two MoO_6 octahedra with overall eight edge-sharing MoO_6 octahedra. There are two classes of Mo—O bonds, the terminal Mo—O with a bond length between 1.70 and 1.72 Å and three types of bridging Mo—O bonds. The doubly bridged Mo—O(μ_2), triply bridged Mo—O(μ_3) and quintly bridged Mo—O(μ_5) having bond length in the range of 1.884(6)–1.917(7) Å, 1.932(6)–2.357(7) Å and 2.134(6)–2.482(7) Å, respectively. In each type of bonds, the *trans* Mo—O bonds to terminal oxygen atoms are considerably long compared to the ones of trans to the bridging oxygen atoms, which can be related to *trans* effect of terminal oxygen atom on the Mo—O bonds. The details of Mo—O bonds are presented in Table 3.

3. Conclusions

In conclusion, individual complexes of $[Mo_6O_{19}]^{2-}$ and $[Mo_8O_{26}]^{4-}$ with the same ligand are not known and we have provided two new homopolyoxomolybdate complexes of the same ligand from ammonium molybdate and acetamide **1** just by changing the reaction conditions. Based on the single-crystal analysis of these inorganic–organic hybrid compounds, the functionalized ammonium cation of ligand **1** has been incorporated for charge balance. In complex **3**, only weak (C—H···O—Mo) H-bonding interaction between ligand **1** and hexamolybdate anion has been observed. The hexamolybdate anion is embedded in the cage formed by inter ligand H-bondings with antiparallel arrangement of two ligand molecules. Interestingly, complex **4** exhibits strong hydrogen-bonding between ligand **1** and β-octamolybdate anion (N—H···O—Mo). Different reaction conditions may be developed using the ligand for the synthesis of other inorganic–organic hybrid polyoxomolybdates with interesting structural features and molecular properties.

Appendix A. Supplementary data

CCDC 1401083 and 1401084 contains the supplementary crystallographic data for **3** and **4**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk.

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