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STRUCTURE OF ORGANIC COMPOUNDS

Hydrothermal Synthesis and Characterization of a New Strandberg-type Diphosphopentamolybdate $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$[1]

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Abstract—An inorganic-organic hybrid material formulated as $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$, has been hydrothermally synthesized and characterized by single crystal X-ray diffraction technique, IR spectrum, thermogravimetric analysis and UV-visible spectroscopy. The structure consists of diphosphopentamolybdate Strandberg type polyoxoanions $[P_2Mo_5O_{23}]^{6-}$, protonated 1-(2-aminoethyl)piperazine cations and six crystallization water molecules. These anions are connected with the organic amine through hydrogen bonding interactions between the hydrogen atoms of the organic molecules and the oxygen atoms of the polyoxoanion and water molecules which generate a three-dimensional network.

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INTRODUCTION

Polyoxometalates (POMs) constitute an immense class of polynuclear oxide clusters of transition metal usually obtained by Mo, W, V, or Nb and mixture of these elements. They have been found to be extremely versatile inorganic building blocks for the construction of supramolecular compounds, having a large variety of properties in numerous disciplines such as in homogenous and heterogeneous catalysis, in medicine as antiviral and anti-tumoral agents and in multifunctional materials and chemical analysis [1–8].

In the last decades, several research strategies have been developed to obtain hybrid organic-inorganic compounds based on polyoxometalates. These POMs can be used as elementary building blocks connected with the organic molecules via van der Waals and/or hydrogen bonding interactions of conventional O–H···O, C–H···O, and N–H···O motifs [9, 10]. They also can be covalently linked through secondary metal coordination groups forming extended structure under hydrothermal condition [11, 12], the self-assembly yields one-, two-, or three-dimensional frameworks.

The molybdenum phosphate clusters, including Strandberg-type structure anion $[P_2Mo_5O_{23}]^{6-}$ have been attracting much attention due to their particular applications. Few of these compounds have been reported in which the inorganic counter ions is Na^+ or NH_4^+ such as in $Na_6[P_2Mo_5O_{23}] \cdot 13H_2O$ [13], $(NH_4)_5[HP_2Mo_5O_{23}] \cdot 3H_2O$ [14], $Na_6[P_2Mo_5O_{23}] \cdot 14H_2O$ [15], and $Na_4[H_2P_2Mo_5O_{23}] \cdot 10H_2O$ [15]. Other researchers are interested in the synthesis of organic-inorganic hybrid materials in which the counter ions are organoamines such as in the compounds: $(C_4N_2H_{12})_3[P_2Mo_5O_{23}] \cdot H_2O$ ($C_4N_2H_{12}$ = piperazine) [16], $(C_3N_2H_{12})_3[P_2Mo_5O_{23}] \cdot 4H_2O$ ($C_3N_2H_{12}$ = 1,2-aminopropane) [16], $(C_5N_2H_7)_5[HP_2Mo_5O_{23}]$ ($C_5N_2H_7$ = 2-aminopyridine) [17], $(bipyH_2)_2[H_2P_2Mo_5O_{23}] \cdot H_2O$ (*bipy* = bipyridine) [18], $(dienH_3)_2[P_2Mo_5O_{23}]$ (*dien* = diethylenetriamine) [19], and $(C_4H_{14}N_2O)_3[P_2Mo_5O_{23}] \cdot 3H_2O$ ($C_4H_{14}N_2O$ = N-(2-hydroxyethyl)-1,2-ethanediaminium) [20]. In addition, a large number of diphosphopentamolybdate Strandberg-type structures containing transition metal complexes (Cu, Co, Ni...) with organic ligands have also been prepared and characterized. The most remarkable structure feature of these compounds is that Strandberg structural unit $[P_2Mo_5O_{23}]^{6-}$ acts as ligand towards the transition metal atom, such as $\{[Cu(en)(Hen)]_2[P_2Mo_5O_{23}]\} \cdot 3H_2O$ (*en* = ethylenediamine) [21], $\{[Cu(2,2'\text{-}bpy)(H_2O)_2]_5[Cu(2,2'\text{-}bpy)(H_2O)]\}[P_2Mo_5O_{23}]_2 \cdot 10H_2O$ (*bpy* = 2,2'-bipyridine) [22], $\{Cu(tpyprz)(H_2O)\}_2\{Mo_5O_{15}(O_3PCH_2\ CH_2PO_3)\}$ (*tpyprz* = tetra-2-pyridylpyrazine) [23], $\{Co_3(tpyprz)_2(H_2O)_3\}\{Mo_5O_{15}(O_3P(CH_2)_3PO_3)\}_2$ [23], and $\{Ni_4(tpyprz)_3\}\ \{Mo_5O_{15}(O_3P(CH_2)_3PO_3\}_2$ [23].

We have synthesized hydrothermally a new diphosphopentamolybdate compound with a Strandberg-type structure $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$,

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[1] The article is published in the original.

Table 1. Crystallographic characteristics and the X-ray-data collection and structure-refinement parameters for $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$

System, sp. gr., Z	Tetragonal, $P4_3$, 4
a, b, c, Å	10.671(1), 10.671(1), 31.113(1)
V, Å^3	3542.8(2)
D_x, g cm^{-3}	2.404
Radiation, λ, Å	MoK_α, 0.71073
μ, mm^{-1}	1.925
T, K	293(2)
Sample size, mm	0.05 × 0.06 × 0.15
Diffractometer	Enraf-Nonius CAD4
Scan mode	$\omega/2\theta$
Absorption correction, $T_{\min}$, $T_{\max}$	Empirical, 0.521, 0.981
$\theta_{\max}$, deg	25.389
h, k, l ranges	$-12 \le h \le 12$, $-12 \le k \le 9$, $-37 \le l \le 33$
Number of reflections: measured/unique ($N1$), $R_{\rm int}$/with $I > 2\sigma(I)$ ($N2$)	6222/6195, 0.029/6160
Refinement method	Full-matrix least-squares on F^2
Number of refined parameters	561
$R1/wR2$ relative to $N1$	0.062/0.1650
$R1/wR2$ relative to $N2$	0.0425/0.1405
S	1.0142
$\Delta\rho_{\max}/\Delta\rho_{\min}$, e/Å^3	2.937/–3.396
Programs	Psi-scans [24], SHELX-97 [25]

where the Strandberg anions and the protonated organic molecules held together into a three-dimensional supramolecular network through hydrogen bonding interactions. To our best knowledge, there is no reports on synthesis of hybrid materials based on diphosphopentamolybdate with the protonated 1-(2-aminoethyl)piperazine as organic cation. In this paper, the hydrothermal synthesis, crystal structure and properties of the titled compound are reported.

EXPERIMENTAL

Table 2. Selected bond lengths d (Å) and angle ω (deg) limits for $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$

Bond	d	Bond	d
Mo1–O1	1.66(3)	Mo4–O13	1.90(2)
Mo1–O2	1.96(2)	Mo4–O14	1.92(3)
Mo1–O3	2.41(2)	Mo4–O15	1.63(2)
Mo1–O4	2.20(4)	Mo4–O16	1.77(2)
Mo1–O5	1.79(2)	Mo4–O17	2.39(2)
Mo1–O6	1.61(3)	Mo4–O18	2.40(2)
Mo2–O2	1.93(2)	Mo5–O5	1.99(2)
Mo2–O4	2.12(3)	Mo5–O16	2.12(3)
Mo2–O7	1.68(3)	Mo5–O18	2.35(2)
Mo2–O8	1.67(3)	Mo5–O19	2.33(2)
Mo2–O9	1.86(2)	Mo5–O20	1.63(2)
Mo2–O10	2.28(3)	Mo5–O21	1.58(2)
Mo3–O9	1.89(2)	P1–O4	1.59(4)
Mo3–O10	2.31(2)	P1–O17	1.53(2)
Mo3–O11	1.62(3)	P1–O19	1.48(2)
Mo3–O12	1.61(3)	P1–O23	1.40(4)
Mo3–O13	1.94(2)	P2–O3	1.58(3)
Mo3–O17	2.29(2)	P2–O10	1.56(3)
		P2–O18	1.39(2)
		P2–O22	1.53(2)
Polyhedron	ω limits	Polyhedron	ω limits
Mo1O$_6$	66.8(1)–149.5(1)	Mo2O$_6$	68.9(1)–162.0(1)
Mo3O$_6$	69.0(1)–158.0(1)	Mo4O$_6$	70.5(9)–160.3(1)
Mo5O$_6$	68.2(8)–168.1(1)	P1O$_4$	102.4(1)–124.4(2)
P2O$_4$	103.1(1)–117.9(2)		

Synthesis. The compound $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$ was obtained by combining ammonium molybdate hydrate $(NH_4)_6Mo_7O_{23} \cdot 4H_2O$ (1.2 g), 1-(2-aminoethyl)piperazine $C_6N_3H_{15}$ (0.4 mL) and H_2O (20 mL), the pH value was adjusted to pH = 4 by the addition of 0.4 mL of phosphoric acid H_3PO_4 (85%). The resulting mixture was homogenized at room temperature for about 2 h, then transferred and sealed in a 25 mL Teflon-lined autoclave, which was heated to 120°C for 60 h. After slow cooling to room temperature, the crystalline product was filtered, washed with distilled water and dried at ambient temperature.

Single-crystal X-ray diffraction. A summary of the crystal data and structure refinement for title compound is given in Table 1. The structure was solved by direct methods and refined by full-matrix least-squares. All non-hydrogen atoms were located from difference Fourier maps and refined with anisotropic

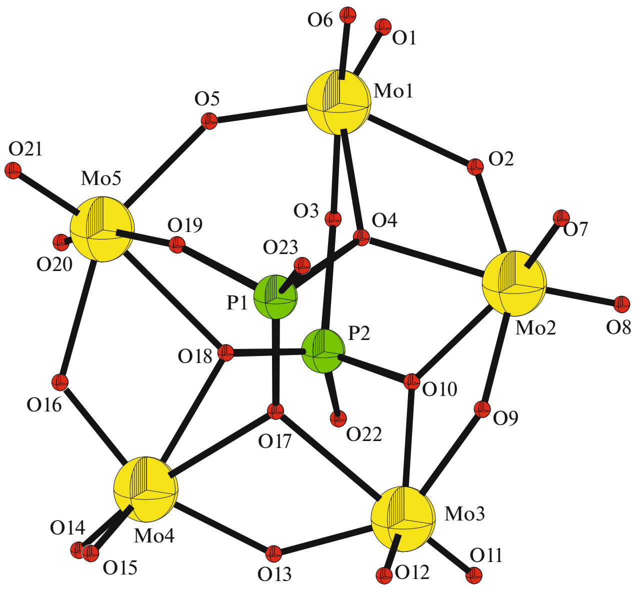

Fig. 1. Ball-and-stick representation of the diphosphopentamolybdate $[P_2Mo_5O_{23}]^{6-}$ cluster anion.

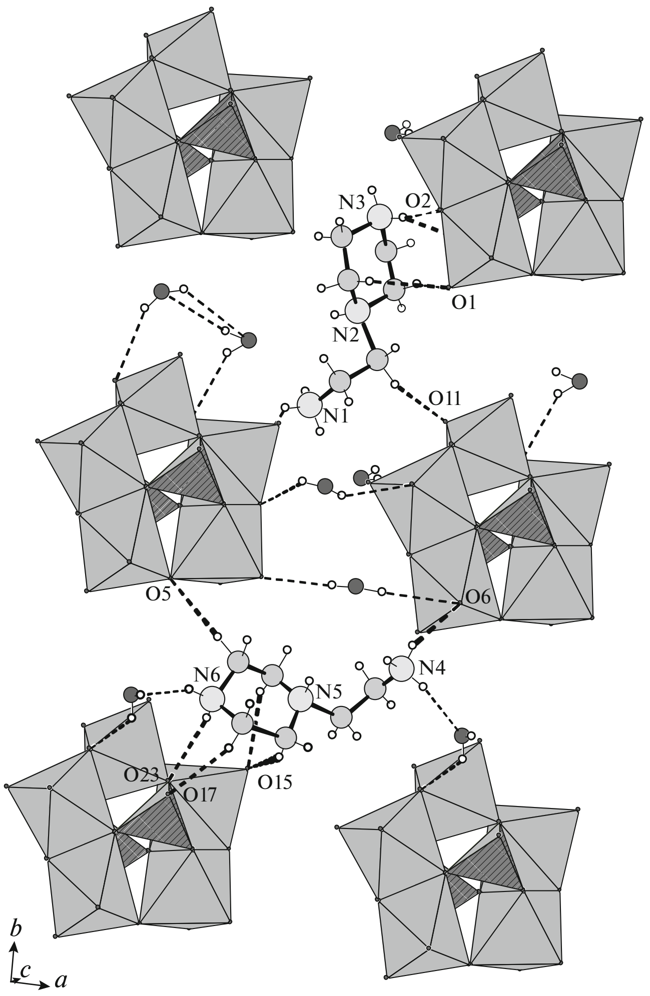

Fig. 2. A polyhedral representation of two-dimensional layer structure of $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$ visualizing H-bonds between the organic molecules and the $[P_2Mo_5O_{23}]$ cluster.

displacement parameter. Hydrogen atoms were located from difference Fourier maps calculated from successive least-squares refinements. These atoms were added in idealized positions and refined in subsequent refinement cycles with an isotropic displacement parameters fixed at 1.2 U_{eq} (Å^2) of the parent atoms.

The crystallographic data were deposited with the Cambridge Crystallographic Data centre: No. CCDC 1441376. These data can be obtained via http//www.ccdc.cam.ac.uk.

RESULTS AND DISCUSSION

Crystal structure. The asymmetric unit of $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$ contains two protonated 1-(2-aminoethyl)piperazine cations, diphosphopentamolybdate $[P_2Mo_5O_{23}]^{6-}$ cluster anion and six lattice water molecules. The cluster $[P_2Mo_5O_{23}]^{6-}$ is of a Strandberg-type polyxometalate anion, which was first crystallographically characterized by Strandberg [13]. It can be described as a pentagonal ring formed by five distorted edge and corner sharing MoO_6 octahedra with two PO_4 tetrahedra capped on each side via sharing three oxygen atoms (Fig. 1). Two of the molybdenum octahedra $Mo1O_6$ and $Mo5O_6$ share a common oxygen atom O5, while each of the other three MoO_6 octahedra shares an edge with adjacent octahedron. In the title compound, each MoO_6 octahedron is bonded to two terminal oxygen atoms with Mo–O distances in the range of 1.58(2)–1.92(3) Å. The bridging oxygen atoms can be divided into three types: the first class contains five doubly bridging oxygen atom O2, O5, O9, O13, and O16 bonded to two Mo atoms, bond length vary from 1.77(2) to 2.12(3) Å; the second class is made up by two doubly bridging oxygen atoms O3 and O19 bonded to one Mo atom and one P atom with Mo–O bond lengths range from 2.33(2) to 2.41(2) Å and the third kind formed by four triply bridging oxygen atoms O4, O10, O17, O18 each shared between two Mo atoms and one P atom with bond lengths from 2.12(4) to 2.40(2) Å. Each P atom is bonded to one terminal oxygen atom, with P–O distances of 1.40(4) and 1.53(2) Å, and three bridging oxygen atoms with P–O distances varied from 1.49(2) to 1.59(4) Å.

The crystal packing demonstrates an extensive hydrogen bonding interactions between N–H and C–H groups of the amine cation with oxygen atoms of the cluster (Table 3, Figs. 2, 3). There are also hydrogen bonds between N—H and C—H groups of the amine with oxygen atoms of the water molecules and between O(water)—H groups and oxygen atoms of cluster anion.

Table 3. Hydrogen bonds and short intermolecular contacts in $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$ crystal

D—H···A	D—H, Å	H···A, Å	D···A, Å	D—H···A, deg
O29—H42···O9	0.819(2)	2.343(2)	2.99(4)	137(3)
O27—H48···O10	0.818(1)	2.439(2)	3.21(5)	148(8)
O26—H46···O11	0.823(2)	2.534(3)	3.39(5)	158(8)
N1—H25···O14	0.888(1)	2.396(2)	3.06(5)	130(5)
C9—H18···O15	0.913(3)	2.485(3)	3.28(5)	142(3)
C11—H23···O15	0.884(2)	2.481(3)	3.23(5)	147(3)
O25—H43···O16	0.822(2)	2.484(3)	3.19(5)	141(6)
N6—H35···O17	0.890(3)	2.329(2)	3.10(5)	143(3)
C9—H1···O21	0.974(3)	2.410(2)	3.11(5)	128(2)
C12—H21···O23	0.937(1)	2.351(2)	3.19(5)	149(4)
C6—H9···O19^{i}	0.978(3)	2.275(2)	3.14(5)	142(3)
N1—H27···O23^{i}	0.890(2)	2.090(2)	2.87(5)	142(5)
N2—H28···O23^{i}	0.885(2)	1.870(2)	2.72(4)	168(3)
C1—H2···O24^{i}	0.952(3)	2.467(3)	3.28(5)	140(3)
O26—H45···O24^{i}	0.820(2)	2.589(3)	3.09(5)	121(6)
C6—H9···O25^{i}	0.979(1)	2.447(2)	3.15(5)	130(2)
O25—H43···O7^{ii}	0.822(2)	2.224(2)	2.92(4)	137(6)
C2—H3···O11^{ii}	1.027(3)	1.951(2)	2.83(4)	137(3)
C7—H14···O28^{ii}	0.970(3)	2.486(3)	3.19(5)	132(2)
C3—H5···O1^{iii}	0.932(1)	2.488(3)	3.29(5)	147(2)
C5—H8···O1^{iii}	0.951(3)	2.519(3)	3.37(5)	152(3)
N3—H30···O2^{iii}	0.890(1)	2.067(2)	2.89(4)	156(4)
C5—H8···O3^{iii}	0.947(3)	2.171(2)	3.01(5)	140(2)
N3—H30···O3^{iii}	0.891(2)	2.391(3)	3.09(5)	133(3)
N4—H31···O6^{iii}	0.891(1)	2.119(2)	3.01(5)	157(6)
C10—H19···O5^{iv}	1.123(3)	2.576(3)	3.72(5)	176(2)
O28—H37···O6^{iv}	0.822(2)	2.492(3)	3.29(5)	167(2)
C11—H24···O14^{v}	0.953(3)	2.401(3)	3.35(5)	165(5)
C12—H22···O20^{v}	0.898(3)	2.220(2)	2.81(5)	127(3)
N4—H32···O22^{v}	0.891(1)	1.891(2)	2.76(4)	161(5)
N5—H34···O22^{v}	0.892(3)	1.802(2)	2.62(4)	159(4)
O24—H40···O13^{vi}	0.822(2)	2.342(3)	3.10(5)	152(8)
O24—H39···O24^{vi}	0.822(2)	2.343(3)	3.12(5)	157(8)
O28—H38···O21^{vii}	0.821(1)	2.149(2)	2.98(5)	167(8)

Symmetry codes: (i) $-y + 1, x, z - 1/4$; (ii) $x, y - 1, z$; (iii) $x + 1, y - 1, z$; (iv) $x + 1, y, z$; (v) $y, -x, z + 1/4$; (vi) $y, -x + 1, z + 1/4$; (vii) $x + 1, y + 1, z$.

In this structure, there are two crystallographically independent protonated 1-(2-aminoethyl)piperazine molecules (named as M1 and M2), the first one (M1) is interlinked with three neighboring clusters forming hydrogen bonds with terminal O atoms of POM: C2—H3···O11 with the first POM, N1—H25···O14 with the second cluster, and C3—H5···O1 and C5—H8···O1 with the third POM. In hydrogen bonds N3—H30···O2 and N3—H30···O3, bridging O atoms take part.

The second protonated amine molecule (M2) connect three clusters anions. C10—H19 group forms hydrogen bond with bridging O5 atom of the first POM and N4—H31 group interacts with terminal O6 atom of the second POM. The third POM is connected to the M2 molecule by three oxygen atoms: the terminal oxygen atom O15 is hydrogen bonded with C11—H23 and C9—H18 groups; terminal oxygen atom O23 of the PO4 tetrahedron is bonded to the C12—H21 group; and the threefold coordinated oxygen atom O17 is bonded to the N6—H35 group. This hydrogen bond system leads to the formation of two-dimensional layer structure in the form of layer (Fig. 2).

In addition, hydrogen bonds also exist between the layers forming three dimensional structure (Fig. 3). The amine molecule M1 forms hydrogen bonds N1—H27···O23 and N2—H28···O23 with the terminal oxygen atom O23 of PO_4 group and hydrogen bond C6—H9···O19 with POM. The M2 molecule forms hydrogen bonds N4—H32···O22 and N5—H34···O22 with the terminal oxygen atom O22 of the PO_4 tetrahedron as well as C11—H24···O14 and C12—H22···O20 with two terminal oxygen atoms of the cluster anions.

IR and thermal analysis. The IR spectrum of the title compound shows the characteristic vibrational peaks similar to those in the known diphosphopentamolybdate structure $[P_2Mo_5O_{23}]^{6-}$. The series of bands in range of 559–420 cm^{-1} are attributed to the (Mo—O—Mo) vibrations. Those at 984, 932, 810, and 757 cm^{-1} are assigned to the terminal Mo=O stretching. There are some characteristic bands for the organic molecules at 1667 cm^{-1} which can be ascribed to δ_{as}(N—H) vibration, 1492 cm^{-1} to δ_{as}(C—H), 1448 cm^{-1} to δ_{s}(C—H), 1421 cm^{-1} to ν_{s}(C—C), 1240 cm^{-1} to ν_{as}(C—N), and 1202 cm^{-1} to ν_{s}(C—N). In addition, the presence of lattice water molecules is confirmed by the broad band at 3000 cm^{-1}. The characteristic absorption bands of P—O stretching appear at 1029 cm^{-1}.

The TG and DTA analysis was studied under dynamic N_2 atmosphere from ambient temperature to 500°C at a heating rate of 10°C min^{-1}. The TG curve can be divided into two stages (Fig. 4). The DTA curve shows that the two stages are endothermic. The first

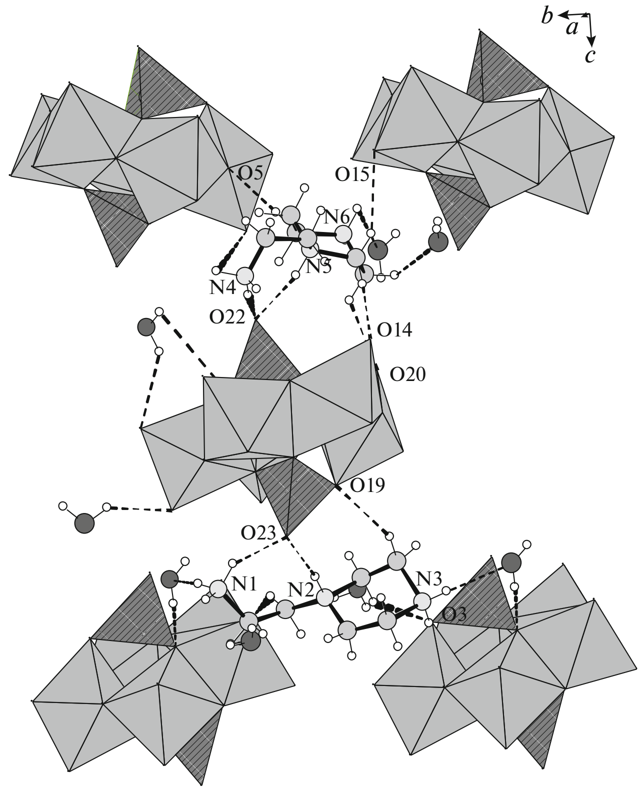

Fig. 3. A view of hydrogen bonding interactions between the two two-dimensional layers in the structure of $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$.

display a weight loss of 8.7% (calculated value: 8.4%) occurring from 90 to 230°C corresponding to the loss of six crystallization water molecules. From about 230 to 500°C, the second weight loss is 21.0%, in good accordance with the calculated value (20.6%), is ascribed to the degradation of the two organic molecules. The whole weight losses of 29.7% were in agreement with the calculated value of 29%.

UV-visible absorption spectrum. The UV-visible absorption behavior of the title compound was analyzed in aqueous solution (4×10^{-3} M). The UV spectrum exhibits two absorption bands centered at 215 and 265 nm (Fig. 5). The strong higher energy band can be attributed to the ligand-to-metal charge transfer from terminal oxygen atom to the molybdenum center ($O_t \rightarrow Mo$), where the nonbonding electrons localized over the oxygen atoms in the highest occupied molecular orbital (HOMO) are transferred to the lowest unoccupied molecular orbital (LUMO) of the molybdenum atoms. The broad lower energy band is attributed to the electron charge transfer from the bridging oxygen atoms to the molybdenum center ($O_b \rightarrow Mo$), where the electrons of oxygen atoms in 2*p* orbital (HOMO) are promoted to the unoccupied *d* orbitals of molybdenum atoms (LUMO) [26–28].

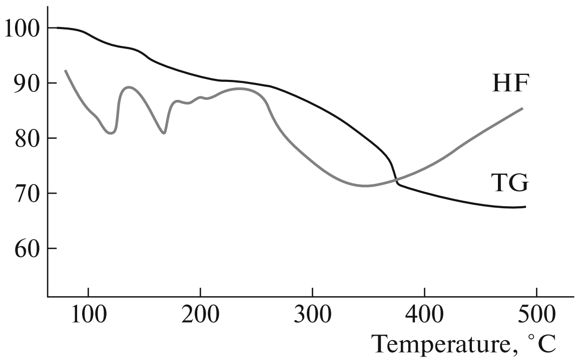

Fig. 4. TGA and DTA curves of $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$.

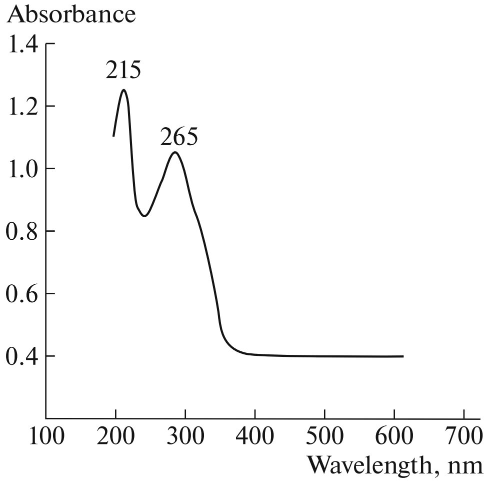

Fig. 5. UV-visible absorption spectrum of $(C_6N_3H_{18})_2[P_2Mo_5O_{23}] \cdot 6H_2O$.

CONCLUSIONS

The hydrothermal syntheses of a new hybrid organic-inorganic compound based on diphosphopentamolybdate cluster anion with the Strandberg-type structure and organic cations have been presented. The organic amine has been used in its triprotonated form to counter balance the negative charge of the POM. The three-dimensional structure of the compound consists of diphosphopentamolybdate anions $[P_2Mo_5O_{23}]^{6-}$ linked by protonated 1-(2-aminoethyl)piperazine $(C_6N_3H_{18})^{3+}$ cation via extensive hydrogen-bonding interactions. The successful syntheses of the title compound show that hydrothermal method can be effective to synthesize some novel hybrid materials.

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