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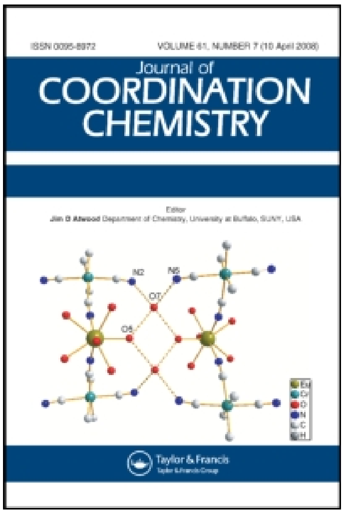

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A new organic-inorganic heteropolyoxometalate compound [Co(2,2'-bipy)$_3$]H[Al(OH)$_6$(Mo$_6$O$_{18}$)]·17H$_2$O based on B-Anderson-Evans type polyanion (2,2'-bipy=2,2'-bipyridine)

Jing-Ping Wang[a]; Su-Zhi Li[a]; Jing-Yang Niu[a]
[a] School of Chemistry and Chemical Engineering, University of Henan, Institute of Molecular and Crystal Engineering, Kaifeng 475001, China

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A new organic–inorganic heteropolyoxometalate compound $[Co(2,2'\text{-bipy})_3]H[Al(OH)_6(Mo_6O_{18})]\cdot 17H_2O$ based on B-Anderson–Evans type polyanion (2,2′-bipy = 2,2′-bipyridine)

JING-PING WANG, SU-ZHI LI and JING-YANG NIU*

Institute of Molecular and Crystal Engineering, School of Chemistry and Chemical Engineering, University of Henan, Minglun Street, Kaifeng 475001, China

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A new organic–inorganic polyoxometalate compound $[Co(2,2'\text{-bipy})_3]H[Al(OH)_6(Mo_6O_{18})]\cdot 17H_2O$ has been synthesized and characterized by elemental analysis, IR, UV spectra, thermogravimetry and differential thermal analysis (TG-DTA) and single-crystal X-ray diffraction analysis. The title compound crystallizes in a monoclinic, $C2/c$ space group, with $a = 37.292(8)$, $b = 14.214(3)$, $c = 25.050(5)$ Å, $\beta = 120.62(3)$, $V = 11426(4)$ Å^3, $Z = 8$, $F(000) = 7200$, $D_c = 2.116\ \mathrm{Mg\,m^{-3}}$, $R = 0.0748$, $wR_2 = 0.1853$. Crystal structural analysis indicates that the asymmetric unit in the crystal structure of the title compound consists of one B-Anderson–Evans type heteropolyoxoanion, one octahedral $[Co(2,2'\text{-bipy})_3]^{2+}$ coordination cation, seventeen water molecules of crystallization and a proton based on charge balance. The curves of TG-DTA indicate that weight loss of the title compound is divided into three stages. The third weight loss reveals that the backbone of heteropolyoxoanion decomposes at 744°C.

Keywords: B-Anderson–Evans structure; Polyoxometalates; Supramolecular

1. Introduction

It has been recognized that polyoxometalate species exhibit unique topological structures and properties that make them useful in catalysis, material science, medicine, photochemistry, electrochemistry and magnetism [1–8]. This extensive contemporary interest in the topological structures, properties and applications of this class of metal-oxygen clusters has spurred significant activity in the design of synthetic phases [9–11]. Recently, structurally characterized organic–inorganic hybrid materials related to the classic Keggin and Wells–Dawson structures have been studied [12–19]. In the construction of these organic–inorganic polyoxometalate compounds, organic ligands with oxygen-donor atoms are employed. However, N-donor organic ligands have been little used [20–23], because coordination ability of N-donor ligands is lower than that of O-donor ligands in the presence of polyoxometalates under ambient aqueous conditions. Anderson–Evans type polyoxometalates containing N-donor

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*Corresponding author. Tel.: +86-378-2192443. Fax: +86-378-2853650. Email: jyniu@henu.edu.cn

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ligands are very limited [24]. In the present article, we report the synthesis, characterization and crystal structure of a new organic-inorganic B-Anderson–Evans type heteropolyoxometalate compound $[Co(2,2'\text{-bipy})_3]H[Al(OH)_6(Mo_6O_{18})] \cdot 17H_2O$ based on N-donor ligands.

2. Experimental

All chemicals used for synthesis were reagent grade, purchased from commercial sources and used without further purification.

2.1. *Synthesis of* $[Co(2,2'\text{-bipy})_3]H[Al(OH)_6(Mo_6O_{18})] \cdot 17H_2O$

A 50 mL aqueous solution containing 1.75 g (7.23 mmol) $Na_2MoO_4 \cdot 2H_2O$ was added to a 25 mL aqueous solution containing 0.74 g (3.11 mmol) $CoCl_2 \cdot 6H_2O$, and then a solution of glacial acetic acid was added slowly under vigorous stirring until the pH of the mixed solution was adjusted to 4.0. A 20 mL methanol–water (1 : 1, volume ratio) mixed solution containing 0.1 g (0.64 mmol) 2,2′-bipyridine and 0.75 g (3.11 mmol) $AlCl_3 \cdot 6H_2O$ was then added dropwise. The pH of the resulting solution was adjusted to 2.6 with concentrated HCl solution. The obtained solution was filtered and slowly evaporated at ambient temperature. One week later, pink block crystals suitable for X-ray diffraction were collected. Elemental analysis, found (%): C, 19.72; H, 3.01; N, 4.58. Calcd: C, 19.65; H, 3.09; N, 4.61.

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2.2. *Physical measurements*

C, H, N elemental analyses were performed on a Perkin-Elmer 2400 II elemental analyzer. The IR spectrum (KBr pellets) was recorded on a Nicolet AVATAR 360 FT-IR spectrometer (4000–400 cm^1). UV spectrum was obtained on a Unican UV-500 spectrometer (distilled water as solvent) in the range of 400–190 nm. TG-DTA measurements were carried out on a Exstar 6000 thermal analysis system in flowing N_2 with a heating rate of $10^\circ C\,min^{-1}$.

2.3. *Crystal structure determination*

A pink single crystal of the title compound was mounted on a glass fiber capillary. Intensity data were collected at 293(2) K with a Rigaka RAXIS-IV diffractometer using Mo-Kα radiation ($\lambda = 0.71073$ Å) in the range of $1.99 < \theta < 25.00^\circ$. Data reduction was performed with SAINT software. Intensity data were corrected for Lorentz and polarization effects, and an empirical absorption correction based on symmetry-equivalent reflections was applied by using the SADABS program [25]. The structure was solved by direct methods and refined by full-matrix least-squares based on F^2, using the SHELXTL-97 software package [26]. Crystallographic data are summarized in table 1. The relevant bond distances and bond angles are listed in table 2.

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Table 1. Crystallographic data and structure refinement for the title compound.

Formula	$C_{30}H_{58}AlCoMo_6N_6O_{41}$
Formula weight	1820.37
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$C2/c$
Unit cell dimensions (Å, °)	
a	37.292(8)
b	14.214(3)
c	25.050(5)
α	90
β	120.62(3)
γ	90
Volume (Å^3)	11426(4)
Z	8
Density (calculated) (Mg m^{-3})	2.116
Absorption coefficient (mm^{-1})	1.685
$F(000)$	7200
Crystal size (mm^3)	$0.23 \times 0.15 \times 0.12$
θ range for data collection (°)	1.99–25.00
Limiting indices	$0 \le h \le 44$, $0 \le k \le 16$, $-29 \le l \le 25$
Reflections collected	9124
Independent reflections	9124 ($R_{\text{int}} = 0.0000$)
Completeness to $\theta = 25.00°$ (%)	90.7
Absorption correction	Empirical
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	9124/0/768
Goodness-of-fit on F^2	1.124
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0748$, $wR_2 = 0.1853$
Largest diffraction peak and hole (e Å^{-3})	1.547 and −1.001

$R_1 = \sum\|F_o| - |F_c\|/\sum|F_o\|$, $wR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$.

Table 2. Relevant bond distances (Å) and angles (°).

Co(1)–N(2)	1.915(9)	Co(1)–N(2′)	1.923(9)
Co(1)–N(3)	1.924(9)	Co(1)–N(3′)	1.931(9)
Co(1)–N(1)	1.934(10)	Co(1)–N(1′)	1.952(10)
Al(1)–O(19)	1.882(8)	Al(1)–O(21)	1.883(8)
Al(1)–O(23)	1.894(8)	Al(1)–O(24)	1.898(8)
Al(1)–O(22)	1.913(8)	Al(1)–O(20)	1.925(8)
Mo(1)–O(1)	1.687(8)	Mo(1)–O(2)	1.688(8)
Mo(1)–O(14)	1.929(8)	Mo(1)–O(13)	1.937(7)
Mo(1)–O(19)	2.297(7)	Mo(1)–O(20)	2.291(7)
Mo(2)–O(3)	1.690(8)	Mo(2)–O(4)	1.715(8)
Mo(2)–O(14)	1.918(7)	Mo(2)–O(15)	1.950(8)
Mo(2)–O(20)	2.295(7)	Mo(2)–O(21)	2.289(7)
Mo(3)–O(6)	1.695(8)	Mo(3)–O(16)	1.908(8)
Mo(3)–O(21)	2.277(7)	Mo(4)–O(8)	1.740(8)
Mo(4)–O(17)	1.932(8)	Mo(4)–O(22)	2.311(7)
Mo(5)–O(9)	1.695(8)	Mo(5)–O(17)	1.927(7)
Mo(5)–O(23)	2.300(7)	Mo(6)–O(12)	1.704(8)
Mo(6)–O(13)	1.933(8)	Mo(6)–O(24)	2.294(7)
O(19)–Al(1)–O(23)	97.2(3)	O(23)–Al(1)–O(20)	177.4(4)
O(1)–Mo(1)–O(2)	106.0(4)	O(1)–Mo(1)–O(20)	162.1(4)
N(2′)–Co(1)–N(3)	88.1(4)	N(2)–Co(1)–N(3′)	174.4(4)
N(2)–Co(1)–N(1)	96.0(4)	N(3)–Co(1)–N(3′)	83.2(4)
N(3)–Co(1)–N(1)	94.5(4)	N(2′)–Co(1)–N(1)	177.3(4)
N(3)–Co(1)–N(1′)	178.1(4)	N(2′)–Co(1)–N(1′)	93.5(4)
N(1)–Co(1)–N(1′)	83.9(4)	N(3′)–Co(1)–N(1′)	95.7(4)

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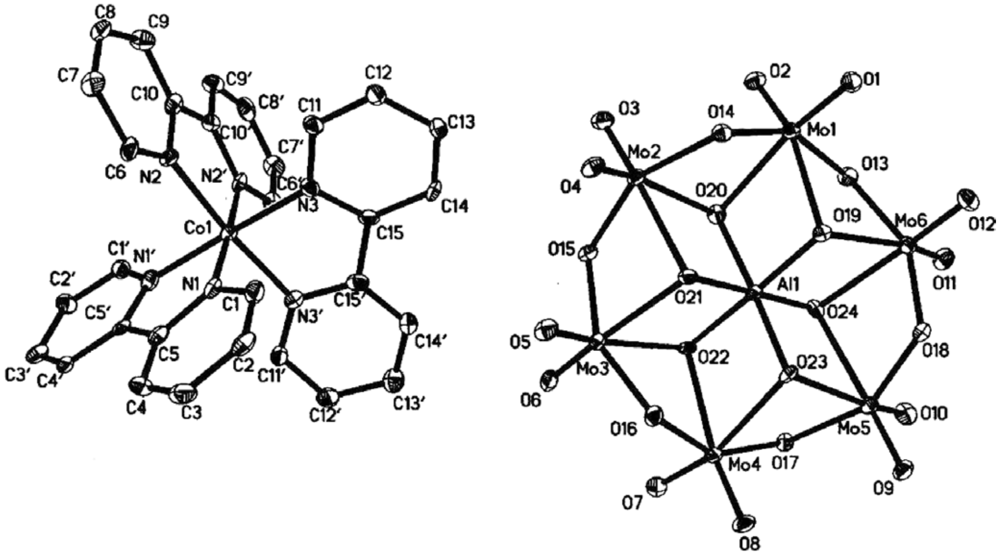

Figure 1. View of the molecular unit of the title compound. All hydrogen atoms are omitted for clarity. Displacement ellipsoids are scaled to enclose 30% probability.

3. Results and discussion

3.1. *Crystal structure of the title compound*

A ball-and-stick representation of the title compound is shown in figure 1. The asymmetric unit of the title compound consists of one B-Anderson–Evans type heteropolyoxoanion which is close to the D_{3h} point symmetry, one octahedral $[Co(2,2'\text{-bipy})_3]^{2+}$ coordination cation, seventeen water molecules of crystallization and a proton based on charge balance. Anderson-type heteropolyoxoanions can be divided into two groups according to the different valence and ionic radius of the central atom X, and the presence of protonated O atoms. The first type with general formula $[X^{n+}M_6O_{24}]^{(12-n)-}$ ($X = Te^{6+}$, I^{7+}, etc., $M = Mo^{6+}$, W^{6+}) is described as an A-type polyanion, whereas the second type $[X^{n+}M_6O_{24}H_6]^{(6-n)-}$ ($X = Cr^{3+}$, Co^{3+}, etc., $M = Mo^{6+}$, W^{6+}) is referred to as a B-type anion [27]. Based on the bond valence model [28], the bond valence sum (BVS) of all O (19–24) atoms by means of equation (1) (where V_i represents the oxidation state of cation i, S_{ij} stands for the valence between the cation i and the anion j; r'_0 (the value is 1.651(2) for Al–O and 1.907(2) for Mo–O) and B (the value is 0.37) are the empirically determined parameters; r_{ij} is the observed bond length) range from 1.16 to 1.25, suggesting that every O(19–24) atom is additionally bonded to a proton which confirms that the anion

$$V_i = \sum_j S_{ij} = \sum_j \exp\left(\frac{r'_0 - r_{ij}}{B}\right) \tag{1}$$

of (I) belongs to the B-type.

In the heteropolyoxoanion, each metal atom is coordinated by six O atoms to form a distorted octahedron. The six different MoO_6 octahedra share edges with each other

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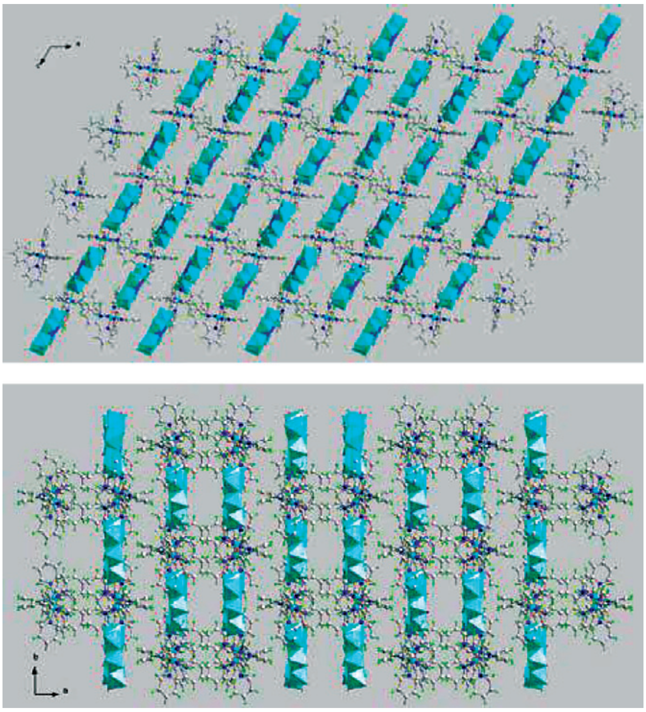

Figure 2. Perspective arrangement of the title compound viewed down *b* and *c* axis. Solvent molecules are omitted for clarity. Displacement ellipsoids are scaled to enclose 30% probability.

and are arranged around the central AlO_6 octahedron with which they likewise share edges. According to the different oxygen coordination, three kinds of O atoms exist in the heteropolyoxoanion: terminal atoms O_t (O1–O12) with bonds to one Mo atom, bridging atoms O_b (O13–O18) with bonds to two Mo atoms, and central atoms O_c (O19–O24) that have bonds to the central Al atom and two Mo atoms. The Mo–O_t, Mo–O_b and Mo–O_c distances vary from 1.687(8) to 1.715(8) Å, 1.908(8) to 1.950(8) Å and 2.277(7) to 2.329(7) Å respectively, very similar to corresponding ones in $Na_3[CrMo_6O_{24}H_6] \cdot 8H_2O$ whose average bond distances are 1.707, 1.939 and 2.292 Å for Mo–O_t, Mo–O_b and Mo–O_c, respectively [29]. In the central AlO_6 octahedron, the Al–O distances range from 1.882(8) to 1.925(8) Å, similar to the corresponding Al–O distances of 1.895(3)–1.920(3) in $[GaO_4Al_{12}(OH)_{24}(H_2O)_{12}][Al(OH)_6Mo_6O_{18}]_2(OH) \cdot 30H_2O$ [30].

Figure 1 also demonstrates the structure of the Co^{2+} coordination cation. The central Co^{2+} ion is located in a distorted octahedral environment of N6 constituted by six pyridine nitrogen atoms from three 2,2′-bipyridine ligands. In $[Co(2,2'\text{-bipy})_3]^{2+}$ the Co–N distances vary from 1.915(9) to 1.952(10) Å and the N–Co–N angles deviate from 90° or 180°, indicating that Co^{2+} has a distorted octahedral structure.

In addition, there are electrostatic and short contact interactions among water molecules, octahedral $[Co(2,2'\text{-bipy})]^{2+}$ coordination cations and the B-Anderson–Evans type heteropolyoxoanions. The most interesting structural feature is that all the heteropolyoxoanions $[Al(OH)_6(Mo_6O_{18})]^{3-}$ are interconnected by means of water molecules and metal-organic cations through the electrostatic and short contact interactions, generating a 3D supramolecular structure (figure 2). Intermolecular electrostatic and short contact interactions play key roles in formation of the supramolecular framework in the solid state.

3.2. *IR and UV spectra*

In the IR spectrum of the title compound, there exist three strong stretching vibrational bands derived from the $[Al(OH)_6(Mo_6O_{18})]^{3-}$ heteropolyoxoanion. The characteristic stretching vibrational regions appearing at 941, 915 cm^{-1}; 836, 665, 645 cm^{-1} and 578 cm^{-1} are attributed to $\nu_{as(Mo\text{–}O_t)}$, $\nu_{as(Mo\text{–}O_b)}$ and $\nu_{as(Al\text{–}O_a)}$, respectively, showing that the title heteropolyoxoanion retains a B-Anderson–Evans type structure [27]. Furthermore, the presence of organic groups is suggested by the occurrence of a series of vibrational bands centered at 1449, 1276 and 1008 cm^{-1} resulting from the 2,2′-bipyridine ligand. In addition, in comparison with the free 2,2′-bipyridine, the resonance at 1449 cm^{-1} attributed to the ν_{as}(C–N of 2,2′-bipyridine) in the title compound has a red-shift of 34 cm^{-1}, indicating that the 2,2′-bipyridine coordinates to the Co^{2+} by means of the N atom.

The UV spectrum of the title compound in aqueous solution displays two strong absorption peaks at 208 and 240 nm. The higher energy spectral band can be assigned to the charge transfer transition of the $O_t \rightarrow Mo$ band, whereas the lower can be attributed to that of $O_b \rightarrow Mo$ band, suggesting a B-Anderson–Evans heteropolyoxoanion. The lower spectral band has a blue-shift of 20 nm as compared to the characteristic absorption band at 260 nm of $O_{b,c} \rightarrow Mo$ of the MoO_6 octahedron only with one terminal oxygen atom [31], the major reason of which may be that each MoO_6 octahedron in the title compound owns two terminal oxygen atoms, and the exclusion interaction between the two terminal oxygen atoms is stronger, leading to the energy gap of the pπ–dπ charge transition of $O_b \rightarrow Mo$ band becoming larger.

3.3. *Thermal property*

The TG curve of the title compound shows three-step weight loss in the range 25–900°C (figure 3), the first and second steps giving a total loss of 44.10% in the range of 0–694°C, correspond to the release of seventeen water molecules of crystallization and three 2,2′-bipyridine molecules (42.55% Calcd) per formula unit of the title compound. The first weight loss of 17.74% occurs between 15 and 276°C (one endothermal peak observed at 341°C in the corresponding DTA curve), corresponding to the removal of seventeen water molecules of crystallization (Calcd 16.81%). The second weight loss of 26.36% between 276 and 694°C corresponds to the loss of three 2,2′-bipyridine molecules (Calcd 25.74%). Three exothermal peaks observed at 464, 546 and 621°C respectively in the corresponding DTA curve arise from combustion of the remaining 2,2′-bipyridine ligands. The third weight loss reveals that the heteropolyoxoanion decomposes at 744°C.

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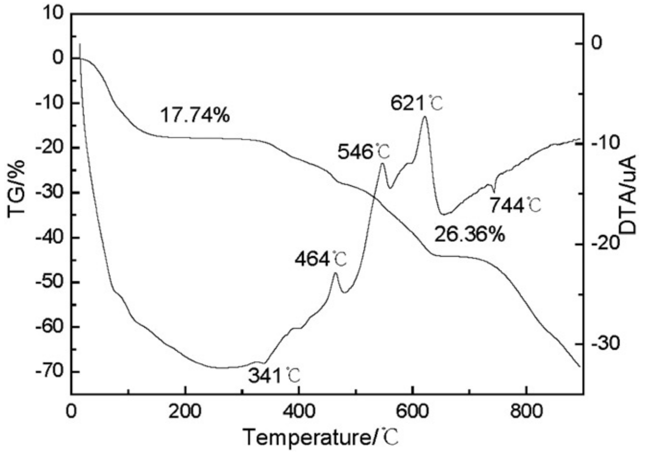

Figure 3. The TG-DTA curves of the title compound at temperatures from 0 to 900°C.

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Supplementary data

Crystallographic data for the structural analyses reported in this article have been deposited with the Cambridge Crystallographic Data Centre with CCDC numbers 615071. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; Email: deposit@ccdc.cam.ac.uk).

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