



Journal of Coordination Chemistry

ISSN: 0095-8972 (Print) 1029-0389 (Online) Journal homepage: https://www.tandfonline.com/loi/gcoo20

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To cite this article: Kun Lu, Zihan Zhu, Lin Peng, Hai Fu, Xinlong Wang & Enbo Wang (2020) The ionothermal synthesis of a new 3-D framework based on classic β -[Mo₈O₂₆]⁴⁻ anions, Journal of Coordination Chemistry, 73:2, 255-265, DOI: 10.1080/00958972.2020.1728745

To link to this article: https://doi.org/10.1080/00958972.2020.1728745

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Published online: 26 Mar 2020.

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The ionothermal synthesis of a new 3-D framework based on classic β -[Mo₈O₂₆]⁴⁻ anions

Kun Lu^a, Zihan Zhu^a, Lin Peng^a, Hai Fu^{a,b}, Xinlong Wang^b and Enbo Wang^b

^aCollege of Chemical Engineering, School of Chemistry and Life Science, Changchun University of Technology, Changchun, P.R. China; ^bKey Laboratory of Polyoxometalate Science of Ministry of Education, Institute of Polyoxometalate Chemistry, Department of Chemistry, Northeast Normal University, Changchun, P.R. China

ABSTRACT

An unusual metal-organic framework, $\{H[Ag(I)L][Mo(VI)_4O_{13}]\}_n$ (1) (L=(4-((1H-1,2,4-triazol-1-yl)methyl)phenyl)methanol)), has been obtained under ionothermal conditions. According to the single-crystal X-ray diffraction analysis, 1 is a new 3-D framework based on β -[Mo₈O₂₆]⁴⁻ type polymolybdate anions with two kinds of channels (A and B) along the *b*-axes and *c*-axes in the framework. The neighboring inorganic Mo₈-Ag chains are linked by the organic ligands through the Ag centers resulting in 2-D layers. These adjacent 2-D layers extend into a 3-D framework through Ag1 nodes and β -[Mo₈O₂₆]⁴⁻ anions, respectively, resulting in a topological symbol ($6^4 \cdot 8^2$)₂($6^4 \cdot 8^2$). In addition, the gas adsorption, proton conduction and luminescent properties of 1 are also discussed in detail. Moreover, the electrochemical impedance spectroscopy (EIS) measurements show a conductivity $(1.9 \times 10^{-4} \text{ S cm}^{-1} \text{ at})$ 65 °C and 95% relative humidity (RH)), with an activation energy of 0.41 eV for 1. The mechanism of proton conduction for 1 is proved to be the vehicular mechanism.

ARTICLE HISTORY

Received 15 September 2018 Accepted 14 November 2019

KEYWORDS

lonothermal synthesis; 3-D framework; adsorption; proton conduction; luminescence



CONTACT Hai Fu 🔯 mr.fuhai@163.com 🝙 College of Chemical Engineering, School of Chemistry and Life Science, Changchun University of Technology, Changchun, Jilin 130012, P.R. China

Supplemental data for this article is available online at https://doi.org/10.1080/00958972.2020.1728745.

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1. Introduction

In recent years, polyoxometalate(POM)-based metal-organic frameworks (PMOFs) have been of great interest for their potential applications in fields such as electrochemistry, catalysis, photochemistry, gas storage and photophysics [1–6]. PMOFs with pores combine the unique properties of organic and inorganic substituents through the interactions between POMs and metal-organic fragments. With their fascinating structural features, including discrete, mobile ionic structures and tunable electronic band structures, POMs have been regarded as attractive candidates for proton conduction and gas adsorption, which are critical for addressing the global energy shortage and environmental pollution [7–10].

Based on different [MoO₄], [MoO₅] and [MoO₆] polyhedra, eight different types of isomeric octa-molybdates (α , β , γ , δ , ε , ζ , η and θ isomers) have been reported [9–15]. Until now, a series of polymers based on octa-molybdates have already been synthesized under hydrothermal conditions [9–11, 16–19]. However, three-dimensional β -isomer octa-molybdate-based PMOFs possessing two different kinds of channels are scarce to date. Hence, the synthesis of such materials with both gas sorption and high proton conductivity is one of the most challenging issues in inorganic synthetic chemistry. With such purposes, we pursued ionothermal techniques to overcome the challenges, as it is regarded as a more efficient and environmentally friendly synthetic method. Under ionothermal conditions, modern room temperature ionic liquids (RTILs) can play the roles of solvents or templates in preparing a large variety of materials. The two main reasons are as follows: (1) RTILs are liquid below a certain temperature (below about 200 °C); (2) Due to the high solubility for most inorganic/organic reactants, tunable pH values, and lower vapor pressure, RTILs can be used as good solvents to prepare the hybrids. Recently, 3-D frameworks have become one of the most interesting and important developed materials. However, many polymers synthesized in ILs show 2-D/3-D structures, revealing ionothermal techniques have great potential applications in the field of synthetic chemistry. The changing of the solvent (from traditional water or organic solvent to RTILs) leads to great successes in preparing 3-D hybrids [20-22].

In order to build new 3-D PMOFs with channels, we chose the ionic liquid, 1-ethyl-3-methylimidazolium bromide ([Emim]Br), as the solvent [23, 24]. A new three-dimensional (3-D) polymolybdate-based MOF {H[AgL][Mo₄O₁₃]}_n (**1**, L=(4-((1H-1,2,4-triazol-1-yl)methyl)-phenyl)methanol), with two different kinds of channels has been successfully synthesized under ionothermal conditions. To the best of our knowledge, it is the first time that a 3-D classic β -[Mo₈O₂₆]⁴⁻ anion-based PMOF has been synthesized in ILs. It exhibits an unprecedented (6⁴ · 8²)₂(6⁴ · 8²) topology for 4,4,4-connected 3-D framework. Moreover, the adsorption, proton conduction and luminescent properties of **1** were also studied in detail. Electrochemical impedance spectroscopy (EIS) measurements also show conductivity (1.9×10^{-4} S cm⁻¹ at 65 °C and 95% RH relative humidity), with an activation energy of 0.41 eV for **1**. The mechanism of proton conduction for this molybdate material is proved to be vehicular mechanism.

2. Experimental

2.1. Materials and methods

All reagents and solvents were purchased from Aladdin and used without purification. An ASAP 2020 HD88 BET surface analyzer was used to measure gas adsorption, and the photoluminescence (PL) spectra were measured on a Perkin Elmer LS55 spectrophotometer at room temperature. Impedance measurements of **1** were performed on a PARSTAT 2273 advanced electrochemical impedance analyzer. The IR spectra were measured on a Thermofisher IS50. The TGA curve was measured on a TA Q50. The Rigaku Smartlab was used to carry out the PXRD for **1**.

2.2. Synthesis of $(Bu_4N)_4Mo(VI)_8O_{26} \cdot nH_2O$

 $Na_2Mo(VI)O_4 \cdot 2H_2O$ (5.00 g, 20.70 mmol) was added into 12 mL water, then acidified with 6.0 M HCL (5.17 mL, 31.00 mmol) in a 50 mL Erlenmeyer flask with vigorous stirring about 1–2 min at room temperature. A solution of tetrabutylammonium bromide (3.34 g, 10.40 mmol) in 10 mL water was then added to the above mixture stirring for 10 min. Then the precipitate was collected on a medium-porosity filter, washed with water, ethanol, acetone and ethyl ether (20 mL four times), successively. The crudes were dissolved in acetonitrile (30 mL) and stored for 24 h at -10 °C. Then block-shaped crystals were collected by suction filtration and dried in vacuum, yielding 3.58 g (1.66 mol), 64% based on Mo.

2.3. Synthesis of 1-ethyl-3-methylimidazolium bromide

Bromoethane 10.83 g (13.80 mL, 80.00 mmol) was dropwise added into N-methyl imidazole 8.21 g (100.00 mmol) over 1 h while stirring magnetically and refluxed for another 2 h. The turbidity of the solution was removed by a separatory funnel, washing with ethyl acetate. The white product was dried in a vacuum at 50 °C, yielding 67%. ¹H NMR (CD₃OD): δ 8.999 (1H), 7.675 (1H), 7.599 (1H), 4.289 (2H), 3.336 (3H), 1.576 (3H).

2.4. Preparation of (4-((1H-1,2,4-triazol-1-yl)methyl)phenyl)methanol

1,2,4-Triazole (0.69 g, 9.00 mmol), PEG-400 (2.04 g, 5.10 mmol), K₂CO₃ (5.06 g, 37.00 mmol) and KI (0.55 g, 3.30 mmol) were dissolved in acetone (30 mL, 410.00 mmol) stirring for 30 min. Then α, α' -dichloro-p-xylene (1.75 g, 10.00 mmol) was added drop-wise to the above solution. The mixture was vigorously stirred and refluxed for 10 h. White residue was obtained after filtering and distilling off the filtrate. The crude material was purified by flash chromatography using a CH₂Cl₂/CH₃COOCH₂CH₃ [1:1] mixture as eluent to obtain white pure product; yield about 45%. ¹H NMR (D₂O): δ 3.33 (1H), 5.27 (2H), 5.42(2H), 7.29 (2H), 7.97(2H), 8.59 (1H) and 8.65(1H).

2.5. Synthesis of 1

A mixture of [Emim]Br (2.02 g, 4.60 mmol), $(Bu_4N)_4Mo(VI)_8O_{26} \cdot nH_2O$ (0.130 g), Ag(I)₂SO₄ (1.56 g, 5.00 mmol) and (4-((1H-1,2,4-triazol-1-yl)methyl)phenyl)methanol (0.120 g, 0.600 mmol) was stirred for 30 min at room temperature. The pH value of the mixture was adjusted to about 3.50 using HCl (conc.) and NaOH (solid). Then the suspension was sealed in a 23 mL stainless steel Teflon-lined autoclave at 160 °C for

Empirical formula	C ₁₀ H ₁₂ AgMo ₄ N ₃ O ₁₄
Formula weight	889.85
Т (К)	293(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	P2/c
a (Å)	10.657(2)
b (Å)	10.398(2)
c (Å)	21.301(6)
α (°)	90
β (°)	119.84(2)
γ (°)	90
V (Å ³)	2047.4(8)
Z	4
D_{calc} (g/cm ³)	2.884
μ (mm ⁻¹)	3.399
F(000)	1676
heta range (°)	3.51-25.00
Rint	0.0465
Data/restraints/parameters	3609/2/295
GOF on F^2	1.066
Final R_1^{a} , wR_2^{b} [I > 2 σ (I)]	$R_1^a = 0.0351, w R_2^b = 0.0841$

 Table 1. Crystal data and structure refinements of 1.

5 days. After cooling to room temperature at the rate of 10 °C h⁻¹, brown block-shaped crystals of **1** were filtered off, washed with acetone and dried in air (yield 65% based on Mo). In order to investigate the importance of the ILs in the system, we use water or ethyl alcohol/water instead of the ILs as solvent. Under the same conditions, we could not obtain **1**. Anal. Calcd for **1** (%): C, 13.50; H, 1.36; N, 4.72; Mo, 43.13; Ag, 12.12. Found: C, 13.21; H, 1.63; N, 4.56; Mo, 43.82; Ag, 12.96. The IR spectra of **1** are shown in Figure S7 in supporting information. In the spectra, characteristic bands at 629, 754, 882 and 944 cm⁻¹ are attributed to the ν (Mo = O_t) and ν (Mo–O–Mo), and the bands observed in the region of 1109–1620 cm⁻¹ for **1** are due to the organic ligand.

2.6. Single-crystal X-ray crystallography

The diffraction data were collected on a Bruker Apex II CCD using the ω -2 θ scan method. Single crystals were mounted on a glass fiber, and the crystallographic data were collected at 293 (K) for **1** on a BRUKER D8 Venture Single-Crystal Diffractometer using graphite monochromatic Mo-K α radiation ($\lambda = 0.71073$ Å). The structure of **1** was solved by the direct method and refined by the full-matrix least-squares method on F^2 using the SHELXTL-2018 crystallographic software package [25–27]. All non-hydrogen atoms were refined with anisotropic thermal parameters. The H atoms on the C atoms were fixed in the calculated positions. Detailed crystal data and structure refinements for **1** are summarized in Table 1.

3. Results and discussion

3.1. Crystal structure

The single-crystal X-ray data reveal that **1** crystallized in the monoclinic space group P2/c. **1** consists of two [AgL]⁺ metal-organic cation fragments, one classical β -[Mo₈O₂₆]⁴⁻ anion



Figure 1. (a) The ball-stick and polyhedron representation of the channel along the *a*-axis in **1**. (b) The ball-stick and polyhedron representation of the channel A along the *b*-axis in **1**. (c) The ball-stick and polyhedron representation of the channel B along the *c*-axis in **1**. All hydrogens are omitted for clarity (the green balls symbolize Ag ions, the small dark blue balls symbolize N atoms, the red polyhedrons symbolize MOO_6 octahedrons, and the red balls symbolize O ions).

and two dissociative H⁺ (supporting information Figure S1a). According to the Bond valence sum calculations, all eight Mo ions show + VI oxidation states (Calc BVS (JRC-LLB, version: March-2005)), shown in supporting information Table S3. The Ag(I) ions can be divided into two groups (Ag1 and Ag2). The Ag1 centers show six-coordinate octahedral geometry, defined by four terminal O ions from two $[Mo_8O_{26}]^{4-}$ anions and two N atoms from two organic ligands (supporting information Figure S1b) (with the bond distance Aq1-O11 2.4533(55), Aq1-O12 2.6096(58) and Ag1-N1 2.3730(62)). Ag2 centers represent four-coordinate plane quadrangle geometry defined by two terminal O ions from two [Mo₈O₂₆]⁴⁻ anions and two N atoms from two organic ligands (with the bond distance Aq2-O9 2.8622(55) and Aq2-N2 2.0357(99)) (supporting information Figure S1c). The organic ligand donates two N atoms to coordinate with the Ag1 and Ag2 ions, respectively (supporting information Figure S1d). Furthermore, the [Mo₈O₂₆]⁴⁻ anions are fourcoordinate by two pairs of crystallographically independent Ag1 and Ag2 ions through the O11/O12 and O9 ions (supporting information Figure S1e). The neighboring $[Mo_8O_{26}]^{4-}$ anions were connected through Aq1-O9, resulting in 1-D infinite chains as shown in supporting information Figure S2. The adjacent 1-D chains were linked through Ag1-L-Ag2 connections resulting in 2-D layers as shown in supporting information



Figure 2. The 3-D ball and stick representation of the two different kinds of channels in 1. All hydrogens are omitted for clarity (the green balls symbolize Ag ions, the small dark blue balls symbolize N atoms, the light gray ball symbolize C atoms, the red and dark blue channels symbolize the two different kinds of channel in 1 and the red balls symbolize O ions).

Figure S3. The organic ligand L acted as a bidentate ligand in the compound. Assuming the $[Mo_8O_{26}]^{4-}$ anions and the Ag1 ions as 4 and 2 connected nodes and linkers, respectively, the 2-D layer can be symbolized as a net with a (6,4) topology layer as shown in supporting information Figure S4. The adjacent 2-D layers were connected by Ag1 ions through O11 and O12, resulting in a 3-D framework (Figure 1). If the $[Mo_8O_{26}]^{4-}$ anions and Ag centers are regarded as four-connected nodes and linkers, the structure of **1** can be symbolized as a net with $(6^4 \cdot 8^2)_2(6^4 \cdot 8^2)$ topology analyzed by the TOPOS program [28–30] (supporting information Figure S5). To our knowledge, such a network is exotic and has not yet been reported [30–32]. The channels of **1** along the *a*-axis are blocked by part of the benzene ring from the organic ligands as shown in Figure 1(a). Two different kinds of open channels A and B with sizes of 2.9846(101) and 4.7012(75) Å, respectively, are shown in Figures 1(b,c) and 2. Some other Ag-Mo₈O₂₆ polymers with conductivity were also been reported [31]. However, compounds with high-dimensional structures are scarce.

3.2. Adsorption properties

The properties of architectural stability and permanent porosity are of great importance for applications in environmental pollution. N_2 adsorption experiments were carried out for the fully activated samples. The N_2 adsorption isotherm at 77 K reveals characteristic type II behavior for **1**, which indicates the microporous nature in



Figure 3. N_2 sorption isotherm of activated 1 at 77 K.

accordance with the structural feature. The maximum N₂ uptake (at standard temperature and pressure, STP) was reached $163 \text{ cm}^3 \text{ g}^{-1}$ at 1 atm (Figure 3). The Brunauer Emmett Teller (BET) surface areas were $287.9 \text{ m}^2 \text{ g}^{-1}$ based on the N₂ adsorption isotherm for **1** at P/P₀=0.99. Before N₂ adsorption, the thermal stability and the purification of the crystals used in the measurements were investigated by TGA and X-ray powder diffraction as shown in supporting information Figures S10 and S11. After the N₂ adsorption experiment, the stability of **1** was also confirmed by the XRPD in supporting information Figure S11. According to the result of the N₂ adsorption and TG curve, it is shown that the maximum N₂ uptake and the BET surface areas are much lower than those of famous MOFs compounds (such as MIL-101, MOF-5 and UIO-66, etc.) [33]. In fact, the N₂ adsorption result of **1** is similar with some classical PMOFs [24]. However, the architectural stability and permanent porosity of **1** were confirmed by the result, and it could potentially be used as a proton conductivity material [34, 35].

3.3. Proton conductivity

Based on the structural properties, stability and the N₂ adsorption results above, **1** was a potential candidate for a proton-conductor. Because of poor hydrophilicity and water retention of the molybdates-based proton-conductors, water molecules are easily desorbed from these conductors at low humidity, resulting in disconnected proton conducting pathways. Retaining efficient proton transport and avoiding the desorption of the materials at low humidity are of great significance for practical application. Highly humid conditions (commonly near 100% RH) are often reported in water-mediated molybdate-based proton-conductors [34].

According to the relation $\sigma = L/(S \times R)$ (*R* the resistance, *L* the width and *S* the area of the sample plate), Nyquist plots under different humidity conditions were used to determine the proton conductivity of **1** (Figure 4). When the temperature increases, the electro-conductibility increases under 45% RH, 70% RH and 95% RH, respectively.

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Figure 4. Nyquist plot of proton conduction for 1 with 65 °C condition (a) at 45% RH, (b) at 70% RH and (c) at 95% RH. (d) Arrhenius plots of proton conductivities for 1 at 45% RH, 70% RH and 95% RH, respectively.

The value of conductivity is the largest at 95% RH. The increasing of the RH makes the absorption of water molecules in the 1-D hydrophilic channels easier, facilitating the proton transportation to larger proton conductivities. The activation energy of 1 is 0.36, 0.39, 0.41 eV under different humidity (45% RH, 70% RH, 95% RH), respectively, calculated by the Arrhenius equation. The EIS measurements show the conductivity $(1.9 \times 10^{-4} \text{ S cm}^{-1} \text{ at } 65 \,^{\circ}\text{C} \text{ and } 95\% \text{ RH})$ and an activation energy of 0.41 eV for **1**.

The vehicular mechanism of 1 is dominant because of the proton conduction under 45% RH, 70% RH and 95% RH. According to the reduced activation energy and the increased proton conductivity, it is suggested that 1 possesses continuous H-bonds among the organic ligands. In an appropriate range, the number of water molecules in the channels increases along with the increasing of humidity. Owing to the open channels, the conductivity of the material increases with the increasing of free protons. Because of the architectural feature and the limited water molecules in the channels as vehicles, the vehicular transfer mechanism is predominant [35]. The additional protons with water molecules lost H-bonds from the networks can directly diffuse in the channels or the layers in the frameworks.

Although the result is lower than those of Im@MOF-808 (8.89 imes 10⁻³ S cm⁻¹ at 288 K, 2.04×10^{-2} S cm⁻¹ at 313 K, and 3.45×10^{-2} S cm⁻¹ at 338 K), it is still consistent with SA-EIMS@MIL-101, MSA-EIMS@MIL-101 and PTSA-EIMS@MIL-101 (1.89 \times 10⁻³ S cm⁻¹,



Figure 5. Emission spectra of 1 (red) and L (black) in solid state at room temperature.

 1.02×10^{-4} S cm⁻¹ and 2.78×10^{-4} S cm⁻¹). Compared with some open-frameworks Ni-POM-EN and Cu₃Mo₅P₂ synthesized by Banerjee *et al.* (39 °C, 59 °C and 98% RH are 2.09×10^{-5} , 1.69×10^{-5} S cm⁻¹ and 2.2×10^{-5} S cm⁻¹ at 28 °C and 98% RH, respectively), our result(s) is(are) much higher [36, 37]. Also, the proton conductivity of **1** at 65 °C, 95% RH is comparable with some PMOFs NENU-3 and HKUST-1 (3.79×10^{-4} S cm⁻¹ at 40 °C reported by Liu *et al.*) [38] and much higher than some MIL-53-based MOFs (*ca.* 10^{-6} to 10^{-9} S cm⁻¹ reported by Kitagawa *et al.* at 25 °C, 95% RH) [39] and a zinc-phosphonate MOF (*ca.* 10^{-5} S cm⁻¹ reported by Shimizu *et al.* at 25 °C, 98% RH) [40].

3.4. Photoluminescent properties

Luminescent compounds are attracting interest due to their various applications in chemical sensors, photochemistry and electroluminescent (EL) displays [41]. Ag(I) complexes commonly show photoluminescent properties at low temperature. However, examples reported at room temperature are scarce [42-44]. In this article, the emission spectra of 1 and the organic ligand L in solid state were investigated at room temperature upon excitation at 255 nm and 330 nm, respectively. The emission spectra of 1 at 394, 420, 446 and 460 nm, which are similar to the ligand, can be attributable to an intra-ligand transition [43]. In addition, 1 still shows weak emission spectra at 414 nm, ascribed to the Ag(I)...O interactions (shown in Figure 5) [41]. Noting that the Aq(I) ions are in d¹⁰ configuration and they are hard to oxidize or reduce. Hence, the luminescence in 1 may neither be attributed to metal-to-ligand charge transfer (MLCT) nor ligand-to-metal transfer (LMCT). It is well known that intermolecular interactions, such as Aq...O interactions, play an important role in the formation of photoluminescent properties of d¹⁰ metal complexes [44, 45]. They can effectively increase the rigidity of the ligand, reduce the loss of energy by non-radiation decay, and decrease the energy gaps of HOMO-LUMO. All these effects support that coordination enhances the emission absorptions [46, 47]. The different emission bands of silver compounds may be attributed to their different 3-D architectures featuring different interactions (such 264 🕢 K. LU ET AL.

as π - π and Ag...Ag interactions), which probably leads to different HOMO-LUMO gaps. The Ag...Ag distance is below the sum of van der Waals radii of the two Ag ions (3.44 \oplus), indicating a weak Ag...Ag interaction. However, in **1**, there is no π - π or Ag...Ag weak interaction in an appropriate range. The resemblance of the emission spectrum of **1** with free ligand points to ligand-based emission [41, 42].

4. Conclusion

A new 3-D polymolybdate-based MOF constructed by classical secondary building unit β -[Mo₈O₂₆]⁴⁻ anions has been isolated and characterized under ionothermal conditions, displaying a new structure with two different kinds of channels (A and B). The N₂ sorption isotherm at 77 K reveals a characteristic type II behavior for **1** and its microporous nature. The experimental results prove that **1** possesses proton conductivity $(1.9 \times 10^{-4} \text{ S cm}^{-1} \text{ at } 65 \,^{\circ}\text{C}$ and 95% RH) and an activation energy (0.41 eV). In addition, **1** still shows a characteristic band at 414 nm, which is ascribed to the Ag(I)---O interactions. Argentophilicity is significant for the formation of luminescent species and was confirmed experimentally and theoretically. The successful preparation of **1** not only exploits a new approach to construct novel high-dimensional 3-D polymolybdate-based MOFs, but also provides a new idea in preparing compounds with different kinds of channels.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was financially supported by the National Nature Science Foundation of China (Nos. 21301019 and 21503022), the Nature Science Foundation of the Technology Department of Jilin Province, China (Nos. 20160101299JC and 20160520136JH), the National Science Foundation for Post-doctoral Scientists of China (No. 2013M530135), China Scholarship Council (No. 201708220042) and Science Research Project of the Education Department of Jilin Province, China (No. jjkh20170546kj).

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