

## Supporting information

### **Making one V<sup>IV</sup> substitution for Mo on classical [MoV<sub>2</sub>O<sub>4</sub>]<sup>2+</sup> group: the first heterobimetallic Mo–V subunit in Polyoxomolybdates–Bisphosphonate family**

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## **1 General Methods**

The chemicals are bought on the commercial sources and can be used without further treatment. Powder X-ray diffraction (PXRD) at room temperature was obtained by using Bruker D8 Advance diffractometer with Cu K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). Fourier transform infrared spectrum (FT-IR) was collected with Nicolet IS5 instrument using KBr. Thermogravimetric analysis (TGA) was performed in a nitrogen flow at a heating rate of  $10 \text{ }^\circ\text{C min}^{-1}$  from 25 to  $900 \text{ }^\circ\text{C}$  on an TA Q600 instrument. X-ray photoelectron spectroscopy (XPS) characterization was executed by using an ESCALAB 250Xi with Al K $\alpha$  X-ray source ( $1486.6 \text{ eV}$ ).  $^1\text{H}$  NMR were collected in 400 MHz on Bruker AVANCE III 400.

## 2 Single crystal X-ray diffraction

Single crystal data collection of compounds **1** and **2** was collected at 168K and 298 K using Mo-K $\alpha$  radiation on a Bruker D8 QUEST system. The structures were solved by direct method and refined by full-matrix least-squares on F<sup>2</sup> using the *Olex 2*<sup>1</sup> after being handled by using the *SQUEEZE* routine of *PLATON*.<sup>2</sup> NH<sub>4</sub><sup>+</sup> and H<sub>2</sub>O could not be distinguished based on the observed electron densities, therefore, the amount of water and ammonium is calculated based on TGA to maintain charge balance. Without using lattice water, all non-hydrogen atoms were polished anisotropically. The organic ligand's hydrogen atoms were locked in their predetermined places. The crystal data and structural refinement parameters for the two compounds are given in **Table S1**. The CCDC number of compound **1** and **2** are 2277603-2277604.

## 3 Preparation and Synthesis

### 3.1 Preparation of [Mo<sub>2</sub>O<sub>4</sub>(H<sub>2</sub>O)<sub>6</sub>]<sup>2+</sup> in aqueous HCl

Hydrazine hydrate N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O (500  $\mu$ L, 10.3 mmol) was added to a clear solution of (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O (5.82 g, 4.7 mmol) in aqueous HCl (4M, 160 mL). The mixture solution progressively turned to red after stirring at 60 °C for 3 h, which was allowed to cool to room temperature and then stored in a conical flask for later use.<sup>3</sup>

### 3.2 Synthesis of compound **1**

The  $\text{NH}_4\text{VO}_3$  (0.234 g, 2 mmol) and Risedronic Acid (0.566 g, 2 mmol) were added into 20 mL  $[\text{Mo}_2\text{O}_4(\text{H}_2\text{O})_6]^{2+}$  aqueous acidic media after the pH being adjusted to 2-3 by tetraethylammonium hydroxide aqueous solution (25%). The pH of the mixture was continued to be adjusted to 4-5 by the same solutions. It follows that the turbid brown solution was heated at 60 °C for 3 h. The resulting light-yellow solution was obtained by filtering off dark blue water-insoluble substance, and then left to evaporate at room temperature. The large pale yellow green block crystals were collected by suction filtration after one day (Yield about 31.8% based on Mo). (KBr,  $\text{cm}^{-1}$ ): 3450 (s), 1641 (m), 1573 (m), 1473 (m), 1394 (m), 1311 (w), 1162 (m), 1109 (s), 1066 (s), 1004 (s), 936 (s), 918 (m), 851 (s), 714 (m), 645 (m), 540 (m), 497 (w), 471 (w).

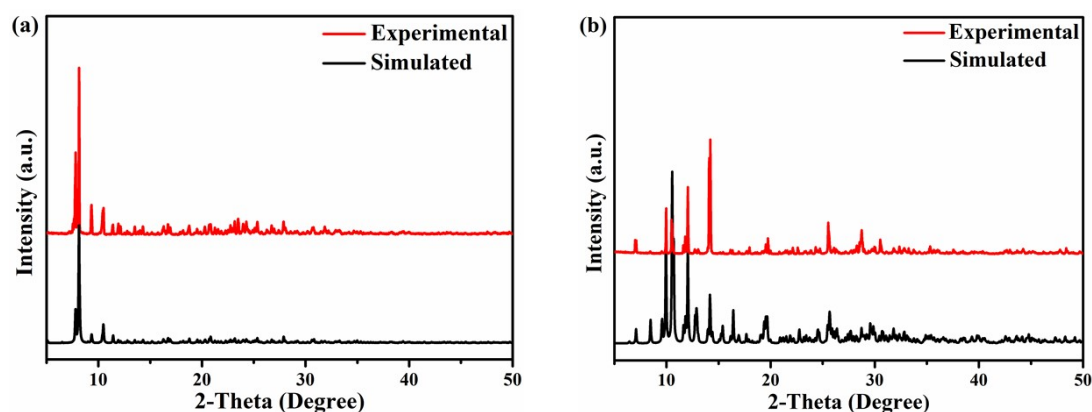
### 3.3 Synthesis of compound 2

Hydrazine hydrate  $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$  (210  $\mu\text{mL}$ , 10.3 mmol) was added to a clear solution of  $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$  (3.40 g, 16.5 mmol) in aqueous HCl (4M, 80 mL). The mixture solution progressively turned to red after stirring at 60 °C for 3 h, which was allowed to cool to room temperature. The  $\text{NaVO}_3$  (0.122 g, 1 mmol) were added into 20 mL  $[\text{Mo}_2\text{O}_4(\text{H}_2\text{O})_6]^{2+}$  aqueous acidic media after the pH being adjusted to 1.5 by  $\text{NH}_3 \cdot \text{H}_2\text{O}$ . Adding risedronic acid (0.566 g, 2 mmol) to the mixture and adjusting the pH to 6 with  $\text{NH}_3 \cdot \text{H}_2\text{O}$ , the mixture solution stirred at 60 °C for 3 h, which was allowed to cool to room temperature, brown block crystals of

compound **2** were obtained one day later (Yield about 20.8% based on Mo). (KBr,  $\text{cm}^{-1}$ ): 3444 (s), 3148 (s), 1628 (m), 1548 (w), 1393 (s), 1135 (m), 1043 (m), 914 (m), 863 (w), 775 (m), 709 (w), 663 (w), 588 (w), 512 (w), 441 (vw).

#### 4 Characterization of compounds

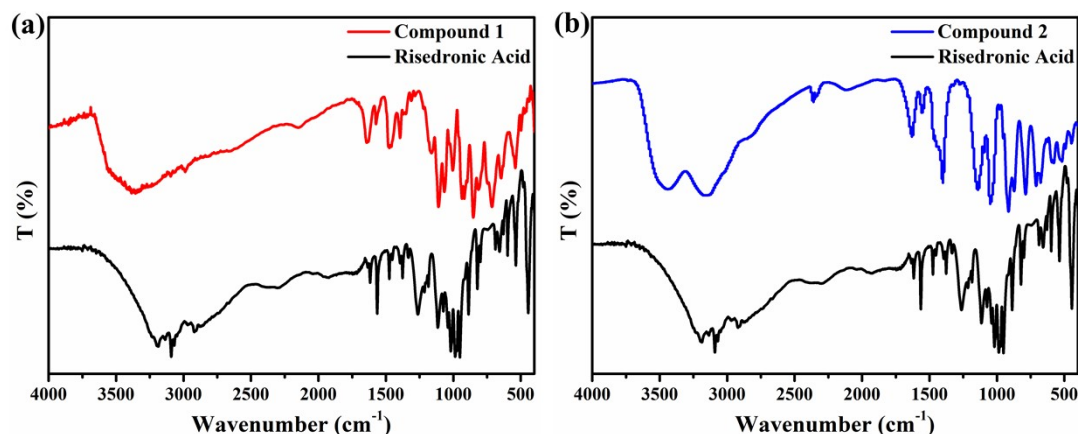
The X-ray diffraction (PXRD) patterns of the compound **1** and **2** is presented in **Fig. S1**. There are no diffraction peaks from other contaminants, and the principal diffraction peaks in the experimental patterns match the calculated results well.



**Fig. S1.** the PXRD pattern of compound **1** and **2**

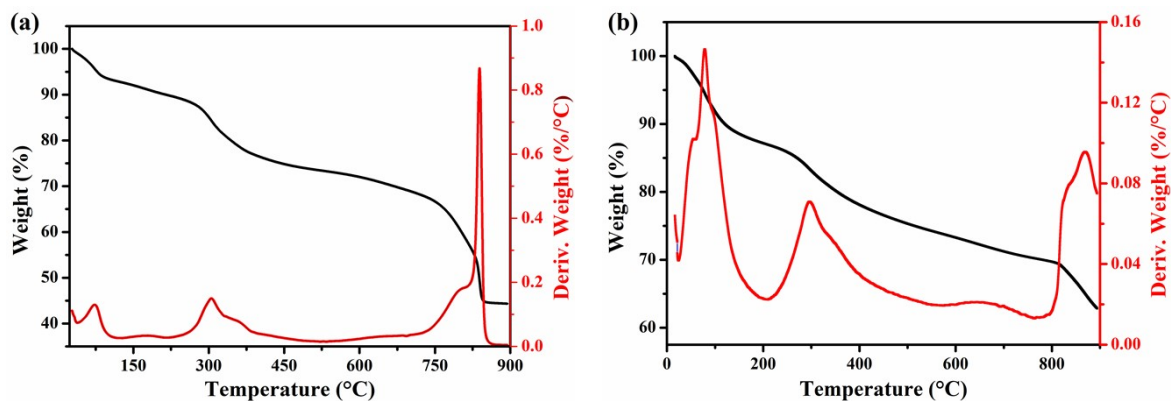
The fourier transform infrared spectrum of the two compounds and Risedronic Acid have similarities. Thus, comparing the peaks in the same position, as shown in **Fig. S2**, it is believed that the  $\nu(-\text{OH})$  attributed to the band at  $3440\text{ cm}^{-1}$ . The peaks at  $1640\text{--}1300\text{ cm}^{-1}$  are assigned to the characteristic vibrations of the same ligand. The vibration peak at  $1109\text{ cm}^{-1}$  corresponds to the  $\nu(\text{P-O})$  bonds in these two compounds. The peaks of the two crystals that appeared at  $950\text{--}700\text{ cm}^{-1}$  can be thought to be

the  $\nu(\text{Mo}=\text{O})$  and  $\nu(\text{M}-\text{O}-\text{M}, \text{M}=\text{Mo} \text{ or } \text{V})$ .<sup>4</sup>



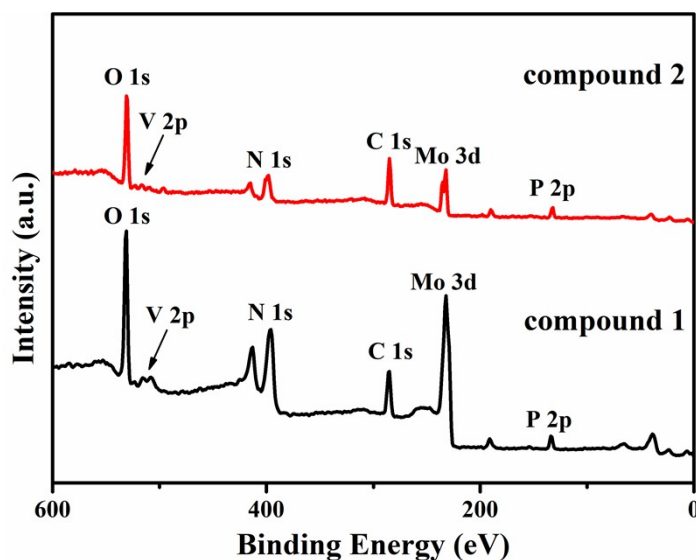
**Fig. S2.** the IR spectra of (a) compound **1** and Risedronic Acid; (b) compound **2** and Risedronic Acid

For compounds **1** and **2**, the weight loss during the first process between 25 and 120 °C is 6.85% and 10.63%, respectively (calcd 7.03% and 11.57%), a result of the two compounds losing their isolated water and coordination water molecules, which is agreed with the one from the SQUEEZE result (compound **1**: 24 H<sub>2</sub>O, 13 NH<sub>4</sub><sup>+</sup>, compound **2**: 5 NH<sub>4</sub><sup>+</sup>, 4 H<sub>2</sub>O). The weight loss of the second process between 210 and 500 °C is 16.83% and 26.92% for compound **1** and **2**, respectively (calcd 17.67% and 27.67%), attributed to the loss of free tetraethylammonium in the two compounds. The weight loss between 700 and 900 °C was thought to be the cause of the collapse of the compound **1** and compound **2** skeletons. The findings of the TG analysis indicated that there were water clusters present and that two compounds had thermal stability. (Fig. S3).



**Fig. S3.** The TGA curve: (a) compound 1; (b) compound 2

The valence state of the main elements in the compound was determined by XPS. In the full-scan spectrum (**Fig. S4**). The elements P, Mo and V are included in the tested sample.



**Fig. S4.** XPS spectra of full-scan spectrum: compound 1 and compound 2

The high-resolution spectrum of Mo 3d for **1** shows two strong peaks located at 235.9 and 232.7 eV, which are regarded as the Mo 3d<sub>3/2</sub> and Mo 3d<sub>5/2</sub> states of the Mo<sup>6+</sup> species, two weak peaks located at 234.5 and 231.6 eV are attributed as the Mo 3d<sub>3/2</sub> and Mo 3d<sub>5/2</sub> states of the Mo<sup>5+</sup> species.<sup>5</sup> The high-resolution spectrum of V 2p shows two peaks located at 523.7 and 515.7 eV, which are regarded as the V 2p<sub>1/2</sub> and V

$2p_{3/2}$  states of the  $V^{4+}$  species (Fig. S5).<sup>6</sup>

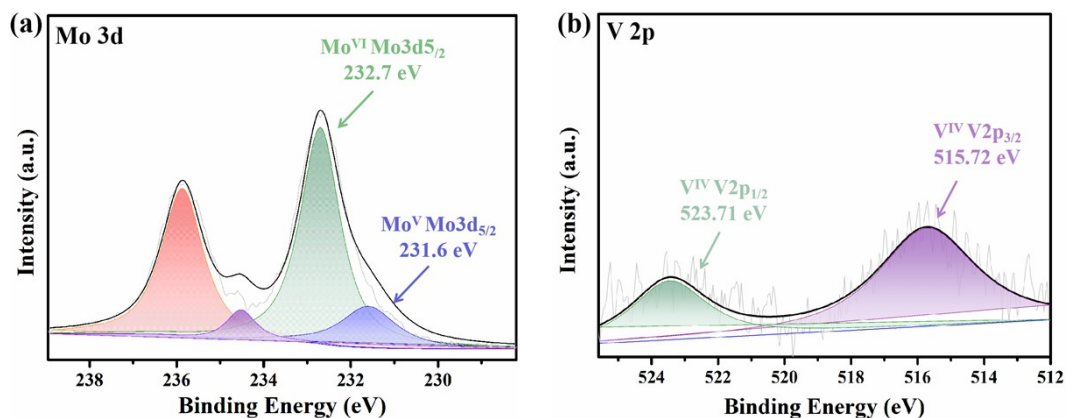


Fig. S5. XPS spectra of compound 1: (a) Mo 3d; (b) V 2p

The high-resolution spectrum of Mo 3d for 2 shows two strong peaks located at 235.43 and 232.27 eV, which are regarded as the  $Mo 3d_{3/2}$  and  $Mo 3d_{5/2}$  states of the  $Mo^{6+}$  species.<sup>5</sup> The high-resolution spectrum of V 2p shows two peaks located at 523.10 and 515.60 eV, which are regarded as the  $V 2p_{1/2}$  and  $V 2p_{3/2}$  states of the  $V^{3+}$  species (Fig. S6).<sup>6</sup>

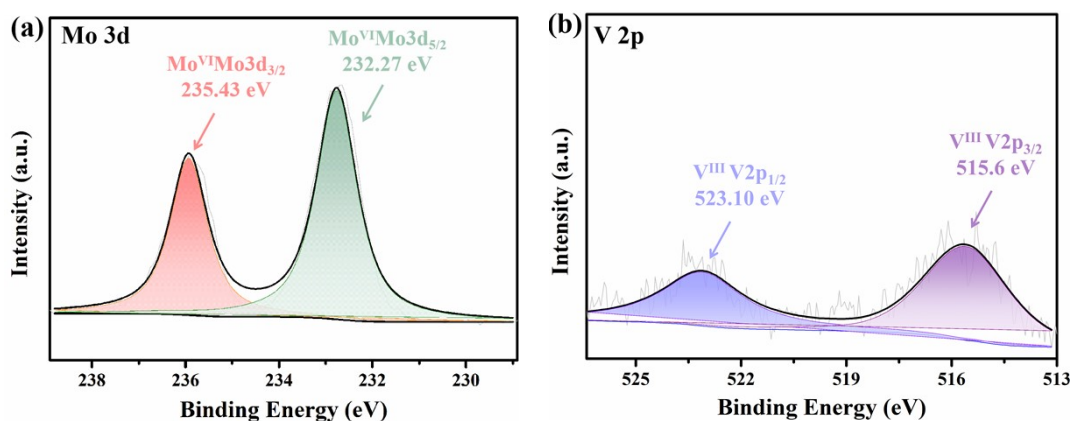


Fig. S6. XPS spectra of compound 2: (a) Mo 3d; (b) V 2p.

**Table S1 Crystallographic data for compounds 1 and 2 (SQUEEZE)**

| Codes                                       | 1  | 2   |
|---|--|---|
| Formula                                     | C <sub>119</sub> H <sub>338</sub> Cl <sub>2</sub> Mo <sub>33</sub> N <sub>16</sub> O <sub>219</sub> P <sub>18</sub> V <sub>3</sub> | C <sub>14</sub> H <sub>28</sub> ClMo <sub>4</sub> N <sub>4</sub> Na <sub>1.5</sub> O <sub>30</sub> P <sub>4</sub> V |
| Mr  | 9415.00  | 1360.92   |
| Crystal system                              | hexagonal  | triclinic   |
| Space group                                 | <i>P</i> 6 <sub>3</sub> / <i>m</i>   | <i>P</i> -1   |
| Temperature (K)                             | 168.90   | 273.15  |
| <i>a</i> (Å)                                | 22.6638(6)   | 10.6878(16)   |
| <i>b</i> (Å)                                | 22.6638(6)   | 15.144(2)   |
| <i>c</i> (Å)                                | 37.7691(10)  | 15.247(2)   |
| $\alpha$ (°)                                | 90   | 66.374(5)   |
| $\beta$ (°)                                 | 90   | 77.188(5)   |
| $\gamma$ (°)                                | 120  | 84.354(5)   |
| Volume (Å <sup>3</sup> )                    | 16800.9(10)  | 2204.5(6)   |
| <i>Z</i>                                    | 2  | 2   |
| <i>D</i> <sub>c</sub> (mg/m <sup>3</sup> )  | 1.861  | 2.050   |
| $\mu$ (mm <sup>-1</sup> )                   | 1.468  | 1.626   |
| Reflections collected                       | 52344  | 28329   |
| Unique data                                 | 10423  | 7748  |
| R <sub>int</sub>                            | 0.0585   | 0.0339  |
| GOF on F <sup>2</sup>                       | 1.084  | 1.038   |
| <sup>a</sup> R <sub>1</sub> [I > 2sigma(I)] | 0.0643   | 0.0327  |
| <sup>b</sup> wR <sub>2</sub>                | 0.1874   | 0.0874  |

$${}^a R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|; {}^b wR_2 = \{ \Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2] \}^{1/2}.$$



**Table S2 Bond valence sums for compounds 1<sup>7</sup>**

| Bond                 | Bond length/Å | Value | Bond                 | Bond length/Å | Value |
|----------------------|---------------|-------|----------------------|---------------|-------|
| Mo1-O1 <sup>i</sup>  | 2.284         | 0.341 | Mo2-O6               | 2.298         | 0.328 |
| Mo1-O8               | 2.095         | 0.568 | Mo2-O9               | 1.715         | 1.588 |
| Mo1-O16              | 2.180         | 0.452 | Mo2-O13              | 1.696         | 1.671 |
| Mo1-O18              | 1.817         | 1.205 | Mo2-O18 <sup>i</sup> | 2.181         | 0.451 |
| Mo1-O19              | 1.706         | 1.627 | Mo2-O26              | 1.857         | 1.082 |
| Mo1-O33              | 1.730         | 1.524 | Mo2-O30              | 2.083         | 0.587 |
| BVS                  |               | 5.717 | BVS                  |               | 5.706 |
| Bond                 | Bond length/Å | Value | Bond                 | Bond length/Å | Value |
| Mo3-O1 <sup>i</sup>  | 2.331         | 0.300 | Mo4-O1W              | 2.348         | 0.287 |
| Mo3-O4               | 2.103         | 0.556 | Mo4-O24              | 2.086         | 0.582 |
| Mo3-O8               | 1.803         | 1.251 | Mo4-O24 <sup>i</sup> | 2.087         | 0.581 |
| Mo3-O20 <sup>i</sup> | 2.078         | 0.595 | Mo4-O25              | 1.723         | 1.554 |
| Mo3-O21              | 1.694         | 1.680 | Mo4-O30              | 1.776         | 1.346 |
| Mo3-O28              | 1.760         | 1.406 | Mo4-O30 <sup>i</sup> | 1.777         | 1.343 |
| BVS                  |               | 5.789 | BVS                  |               | 5.693 |
| Bond                 | Bond length/Å | Value | Bond                 | Bond length/Å | Value |
| Mo5-O3               | 1.697         | 1.667 | Mo6-O7               | 1.699         | 1.658 |
| Mo5-O12              | 2.373         | 0.268 | Mo6-O12              | 2.345         | 0.289 |
| Mo5-O17              | 1.867         | 1.053 | Mo6-O17              | 1.932         | 0.883 |
| Mo5-O23              | 1.692         | 1.689 | Mo6-O26              | 1.918         | 0.915 |
| Mo5-O28 <sup>i</sup> | 2.175         | 0.458 | Mo6-O27              | 1.695         | 1.676 |
| Mo5-O32              | 2.038         | 0.663 | Mo6-O33 <sup>i</sup> | 2.325         | 0.305 |
| BVS                  |               | 5.798 | BVS                  |               | 5.726 |
| Bond                 | Bond length/Å | Value |                      |               |       |
| V1-O5                | 1.584         | 1.807 |                      |               |       |
| V1-O14               | 1.968         | 0.640 |                      |               |       |
| V1-O14 <sup>i</sup>  | 1.968         | 0.640 |                      |               |       |
| V1-O24               | 1.989         | 0.605 |                      |               |       |
| V1-O24 <sup>i</sup>  | 1.989         | 0.605 |                      |               |       |
| BVS                  |               | 4.297 |                      |               |       |

Symmetry transformations used to generate equivalent atoms: i: x, y, 1/2-z.

**Table S3 Bond valence sums for compounds 2 <sup>7</sup>**

| Bond               | Bond length/Å | Value | Bond                 | Bond length/Å | Value |
|--------------------|---------------|-------|----------------------|---------------|-------|
| Mo1-O1             | 2.298         | 0.347 | Mo2-O1               | 2.371         | 0.285 |
| Mo1-O4             | 2.327         | 0.321 | Mo2-O4               | 2.400         | 0.263 |
| Mo1-O8             | 1.802         | 1.328 | Mo2-O7               | 1.943         | 0.907 |
| Mo1-O9             | 1.727         | 1.626 | Mo2-O11              | 1.887         | 1.055 |
| Mo1-O10            | 1.72          | 1.657 | Mo2-O12              | 1.702         | 1.740 |
| Mo1-O11            | 2.025         | 0.726 | Mo2-O13              | 1.704         | 1.730 |
| BVS                |               | 6.008 | BVS                  |               | 5.983 |
| Bond               | Bond length/Å | Value | Bond                 | Bond length/Å | Value |
| Mo3-O14            | 2.386         | 0.274 | Mo4-O14              | 2.332         | 0.317 |
| Mo3-O17            | 2.371         | 0.285 | Mo4-O17              | 2.318         | 0.329 |
| Mo3-O20            | 1.945         | 0.902 | Mo4-O23              | 2.049         | 0.681 |
| Mo3-O21            | 1.722         | 1.648 | Mo4-O24              | 1.711         | 1.698 |
| Mo3-O22            | 1.698         | 1.75  | Mo4-O25              | 1.723         | 1.644 |
| Mo3-O23            | 1.864         | 1.123 | Mo4-O26              | 1.804         | 1.320 |
| BVS                |               | 5.993 | BVS                  |               | 5.991 |
| Bond               | Bond length/Å | Value | Bond                 | Bond length/Å | Value |
| V1-O2              | 2.007         | 0.499 | V2-O15               | 2.047         | 0.439 |
| V1-O2 <sup>i</sup> | 2.007         | 0.499 | V2-O15 <sup>ii</sup> | 2.047         | 0.439 |
| V1-O5              | 2.034         | 0.455 | V2-O18               | 2.021         | 0.471 |
| V1-O5 <sup>i</sup> | 2.034         | 0.455 | V2-O18 <sup>ii</sup> | 2.021         | 0.471 |
| V1-O8              | 1.933         | 0.598 | V2-O26               | 1.941         | 0.585 |
| V1-O8 <sup>i</sup> | 1.933         | 0.598 | V2-O26 <sup>ii</sup> | 1.941         | 0.585 |
| BVS                |               | 3.088 | BVS                  |               | 2.994 |

Symmetry transformations used to generate equivalent atoms: i: 2-x, 1-y, 1-z; ii: 1-x, 2-y, -z.

## Reference

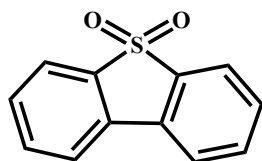
- 1 Bruker, A. X. S. Inc., APEX3 Package, APEX3, SAINT and SADABS, Madison, Wisconsin, USA, 2016.
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- 3 J. D. Compain, A. Dolbecq, J. Marrot, P. Mialane, F. Sécheresse. *C. R., Chimie.* 2010, **13**, 329-335.
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- 6 J. Wang, L. Wang, C. Y. Liu, Y. Wang, F. Ye, W. Yan and B. Liu, Polyoxovanadate ionic crystals with open tunnels stabilized by macrocations for lithium-ion storage, *Nano Res.*, 2023, **16**, 9267-9272.
- 7 N. E. Brese, M. O'Keeffe, *Acta Crystallogr. Sect. B* 1991, **47**, 192-197

## 5 Catalytic Oxidation of Sulfides

In a 10 mL Schlenk tube, the substrate (0.2 mmol) was dissolved in CH<sub>3</sub>CN (2 mL), then catalyst and H<sub>2</sub>O<sub>2</sub> (0.8 mmol, 30% aqueous) were added into the solution at 80 °C for 12 h. After the reaction, separate the catalyst from the reaction mixture by centrifugation. The crude product was purified via silica gel flash column chromatography (petroleum ether /ethyl acetate 3:1-8:1).

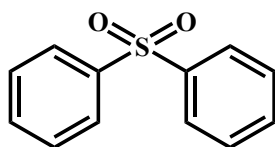
## 6 Characterization of the products

### Dibenzothiophene sulfone (2a)



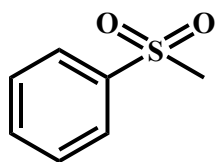
Conversion 95%, Selectivity 99%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.80 (d, J = 7.6 Hz, 2H), 7.76 (d, J = 8.0 Hz, 2H), 7.61 (t, J = 8.0 Hz, 2H), 7.50 (t, J = 8.0 Hz, 2H).

### Diphenyl sulfone (2b)



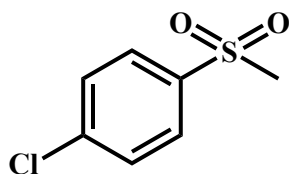
Conversion 86%, Selectivity 99%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.95 (d, J = 8.0 Hz, 4H), 7.56 (t, J = 8.0 Hz, 2H), 7.50 (t, J = 8.0 Hz, 4H).

### Methyl phenyl sulfone (2c)



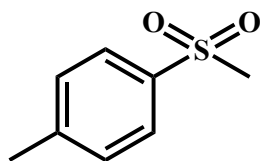
Conversion 99%, Selectivity 95%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.96 (d, J = 7.6 Hz, 2H), 7.67 (t, J = 7.2 Hz, 1H), 7.58 (t, J = 8.0 Hz, 2H), 3.06 (s, 3H).

### 4-Methylsulfonyl chlorobenzene (2d)



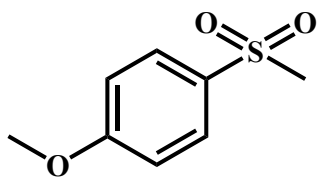
Conversion 99%, Selectivity 87%, off-white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.89 (d, J = 8.4 Hz, 2H), 7.56 (d, J = 8.4 Hz, 1H), 3.06 (s, 3H).

### Methyl p-tolyl sulfone (2e)



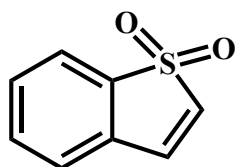
Conversion 70%, Selectivity 97%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.83 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 3.04 (s, 3H), 2.46 (s, 3H).

### (4-Methoxyphenyl)methylsulfone (2f)



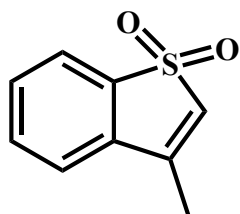
Conversion 82%, Selectivity 99%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.73 (d, J = 8.8 Hz, 2H), 7.03 (d, J = 9.2 Hz, 2H), 3.89 (s, 3H), 3.03 (s, 3H).

### Thianaphthene-1,1-dioxide (2g)



Conversion 79%, Selectivity 91%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.71 (d, J = 7.2 Hz, 1H), 7.56 (t, J = 6.8 Hz, 1H), 7.52 (t, J = 6.4 Hz, 1H), 7.36 (d, J = 6.4 Hz, 1H), 7.22 (d, J = 7.2 Hz, 1H), 6.72 (d, J = 6.8 Hz, 1H).

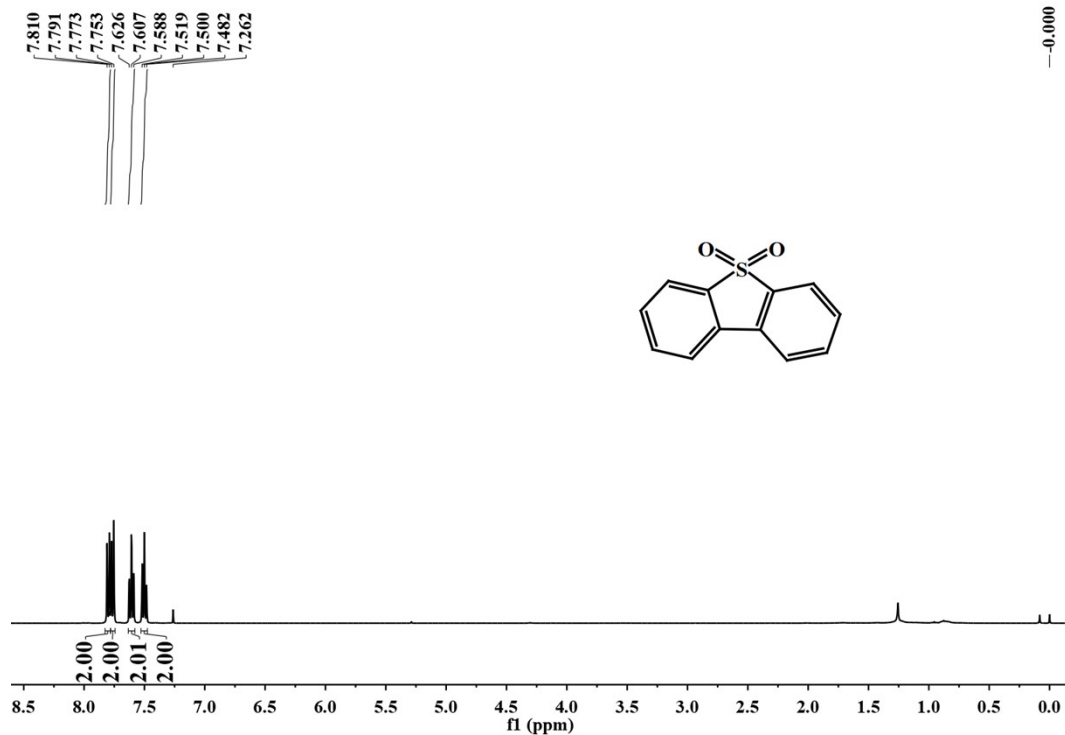
### 3-Methylbenzo[b]thiophene dioxide (2h)



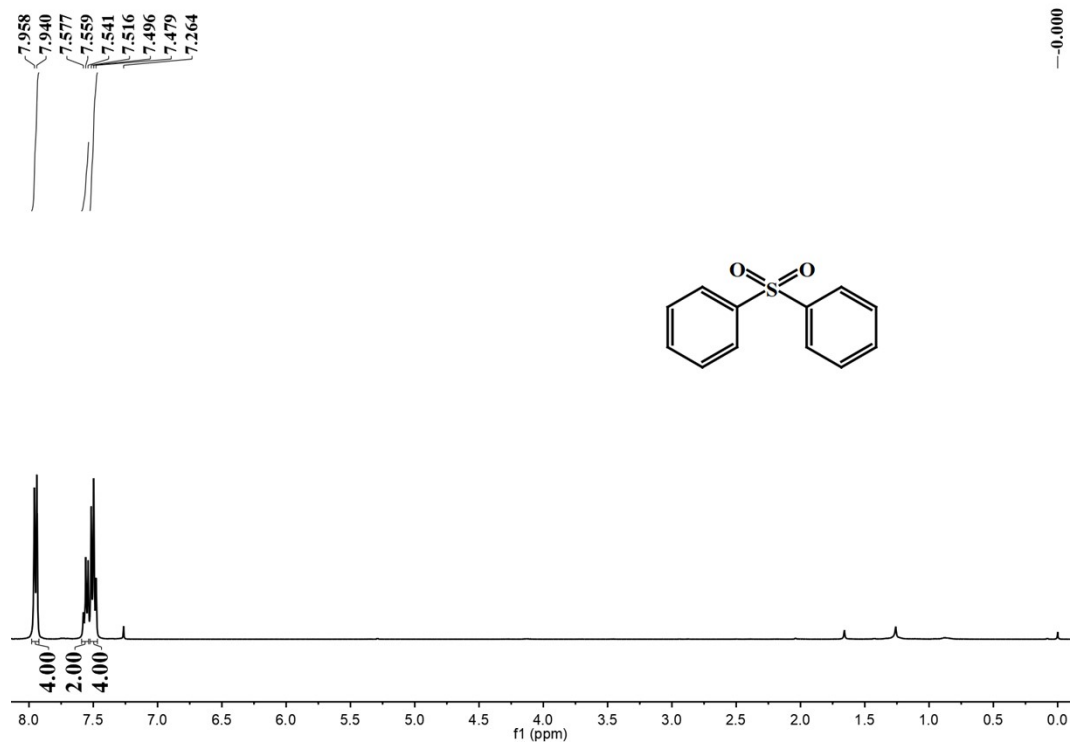
Conversion 65%, Selectivity 95%, white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.71 (d, J = 7.0 Hz, 1H), 7.60 (t, J = 7.0 Hz, 1H), 7.54 (t, J = 7.4 Hz, 1H), 7.41 (d, J = 7.6 Hz, 1H), 6.47 (s, 1H), 2.28 (s, 3H).

# 7 <sup>1</sup>H NMR

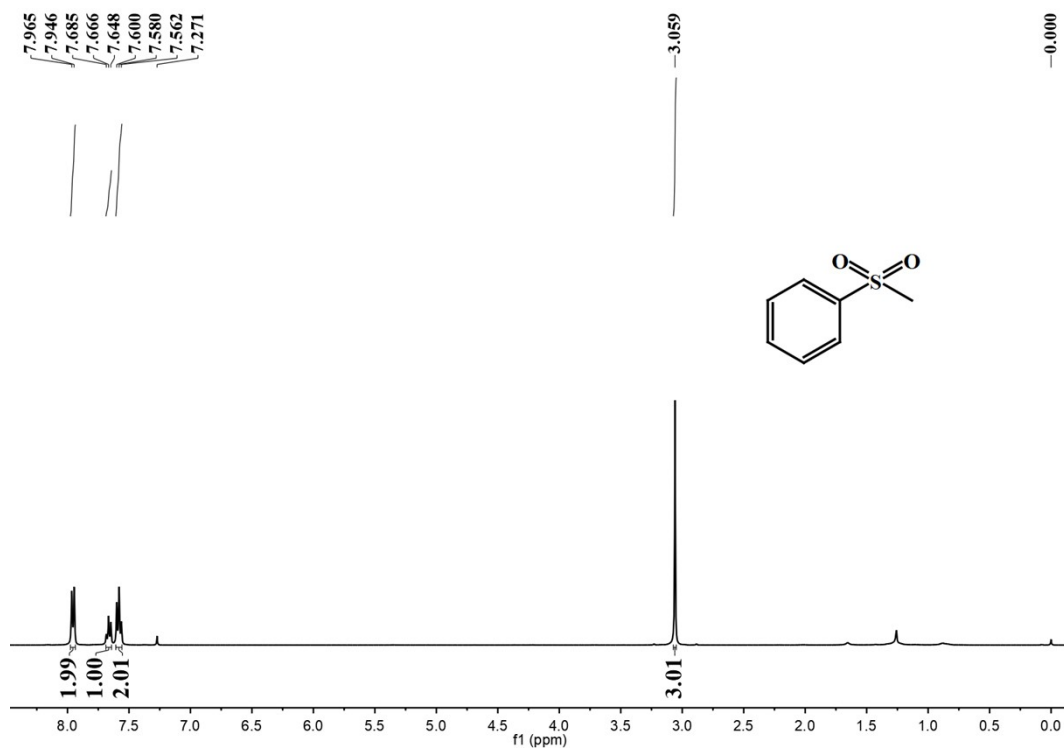
## Dibenzothiophene sulfone (2a)



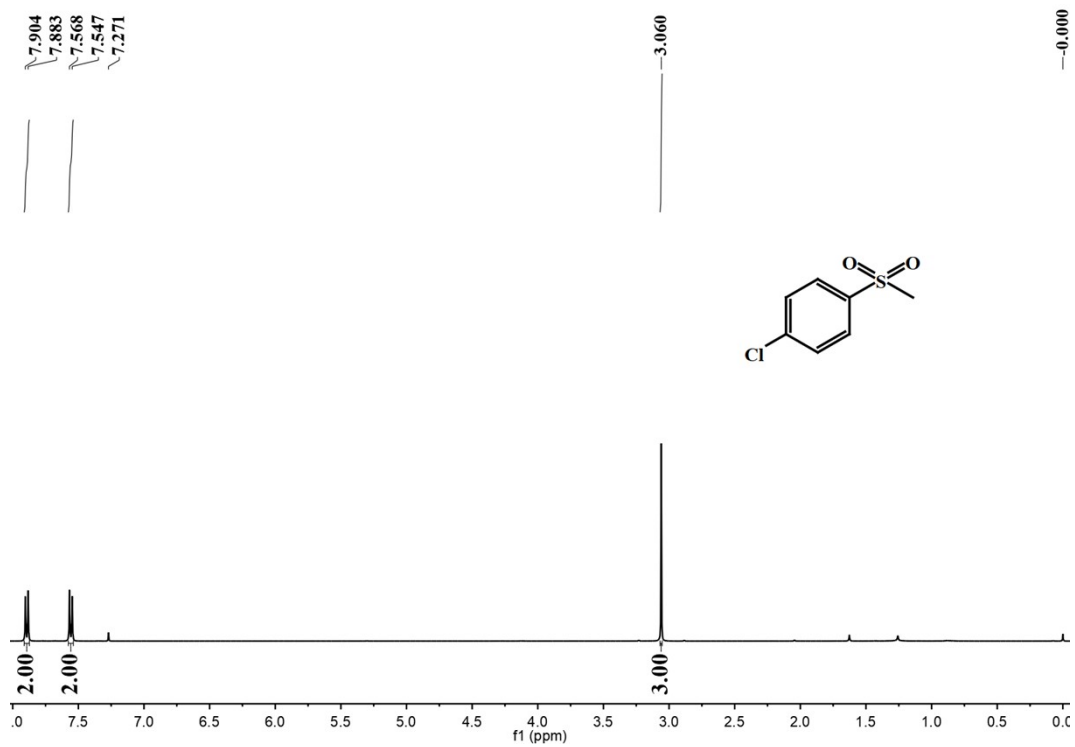
## Diphenyl sulfone (2b)



### Methyl phenyl sulfone (2c)

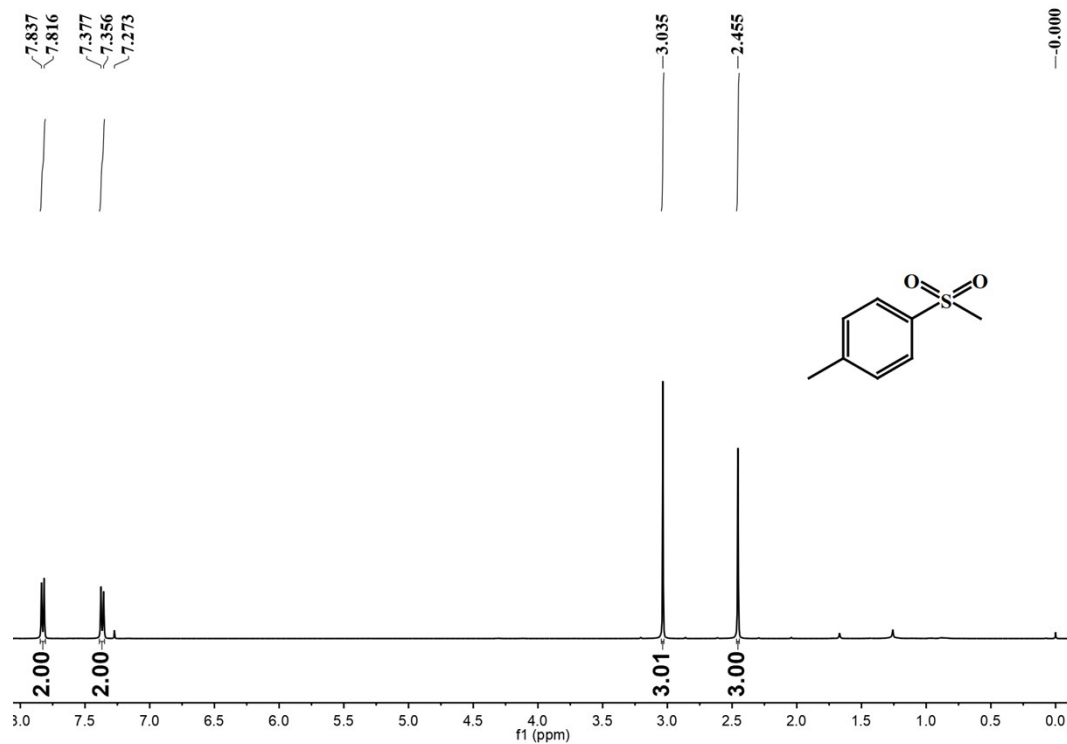


### 4-Methylsulfonyl chlorobenzene (2d)





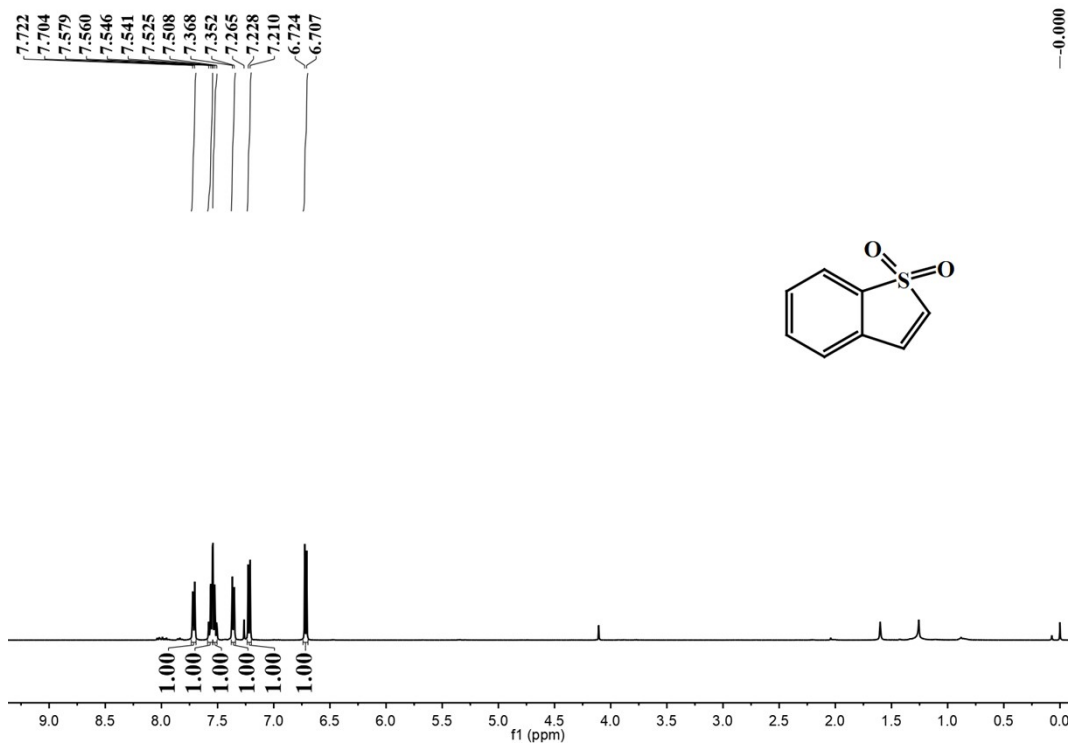
### Methyl p-tolyl sulfone (2e)



### (4-Methoxyphenyl)methylsulfone (2f)



### Thianaphene-1,1-dioxide (2g)



### 3-Methylbenzo[b]thiophene dioxide (2h)

