# Polymeric tri-$\mu$-oxo(pyrazine)molybdenum(VI)

## Yucang Liang, Maochun Hong,* Rong Cao, Daofeng Sun and Jiabao Weng

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: hmc@ms.fjirsm.ac.cn

**Key indicators**

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma$(C–C) = 0.012 Å

$R$ factor = 0.034

$wR$ factor = 0.086

Data-to-parameter ratio = 10.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Mo_2O_6(pz)]_n$ (pz is pyrazine, $C_4H_4N_2$), was synthisized by hydrothermal reaction of $MoO_3$, pyrazine-2-carboxylic acid and water at 443 K. The compound is a three-dimensional inorganic–organic hybrid polymer, in which pz acts as a bridging ligand. Each Mo atom is coordinated by five O atoms and one N atom in a distorted octahedral geometry, with the Mo—N distance being 2.449 (8) Å and the Mo—O distances ranging from 1.682 (7) to 2.086 (8) Å.

## Comment

Owing to the important role in the development of catalysis, electric conductivity, magnetism, non-linear optical properties and medicine, metal oxide chemistry has attracted much interest in recent years (Hill, 1998). In order to study the reaction behavior of metal oxides, organoamine ligands were often selected for the construction of inorganic–organic hybrid materials. Organoamines act in three different roles in these inorganic–organic hybrid materials (Hagrman *et al.*, 1999): (i) as charge-compensating cations, (ii) directly bonded to the metal site of the metal oxide skeleton backbone and (iii) bonded to the heterometal atom. Recently, Zubieta and co-workers chose organoamines as a ligand and reported several molybdenum oxide hybrid compounds, such as $MoO_3(2,2'$-bpy), $Mo_2O_6(2,2'$-bpy), $Mo_3O_9(2,2'$-bpy)$_2$, $Mo_4O_{13}$(Hbpa), $(H_2en)Mo_3O_{10}$ and $[H_3N(CH_2)_6NH_3][Mo_4O_{13}]$ with a one-dimensional chain structure (Kahn & Zubieta, 1993; Zapf *et al.*, 1997*a,b*, 1998; Xu *et al.*, 1996); compounds with a two-dimensional structure $[4,4'$-$H_2bpy][Mo_7O_{22}].H_2O$ (Zapf *et al.*, 1997*a,b*) and $MO_3$(py) ($M$ = Mo,W) (Johnson *et al.*, 1981) and a three-dimensional network structure $HMo_2O_6(4,4'$-bpy). Herein we report the crystal structure of a molybdenum trioxide–pyrazine complex possessing a three-dimensional framework structure prepared *via* hydrothermal reaction, *i.e.* $Mo_2O_6$(pz).
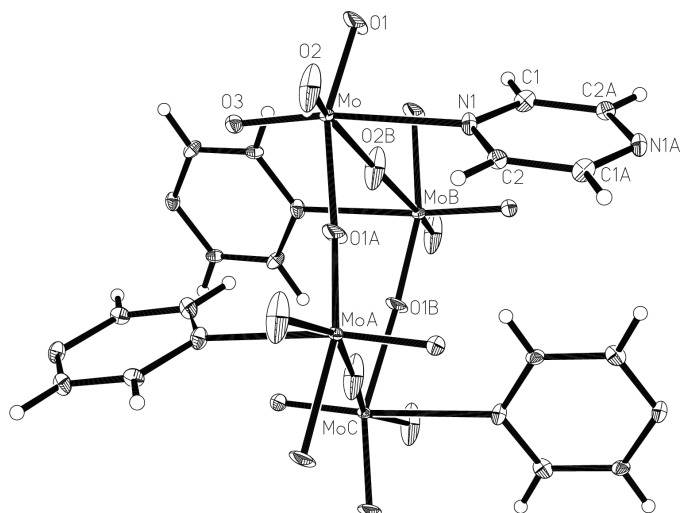


(I)
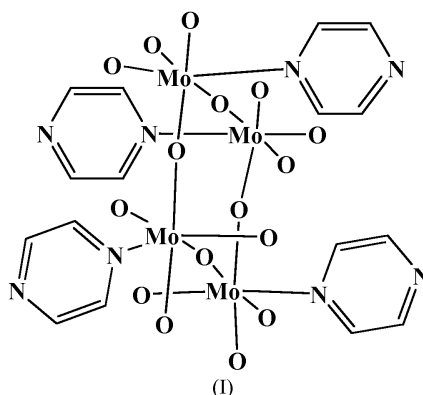
**Figure 1**
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

The title compound, (I), consists of $Mo_2O_6$ and pyrazine. $Mo_2O_6$ forms a two-dimensional layer structure by sharing O atoms. The two-dimensional layer is linked further into a three-dimensional framework through pyrazine bridging ligands. In the title compound, pyrazine came from the heat decarboxylated reaction of the pyrazine-2-carboxylic acid. Every $Mo^{VI}$ atom is coordinated by five O atoms and one pyrazine N atom to form an $[MoO_5N]$ octahedral geometry. The Mo—O bond lengths range from 1.682 (7) to 2.086 (8) Å and the Mo—N distance is 2.449 (8) Å. The O—Mo—O angles range from 78.4 (4) to 160.1 (2)° and the O—Mo—N angles range from 77.4 (2) to 171.0 (3)°.

## Experimental

A mixture of $MoO_3$ (144 mg), pyrazine-2-carboxylic acid (124 mg) and water (16 ml) was sealed in a 25 ml Teflon-lined stainless-steel reactor, heated to 443 K for 72 h, then naturally cooled to room temperature. Block-shaped black crystals suitable for X-ray analysis were obtained in 70% yield.

### Crystal data

| | |
|---|---|
| $[Mo_2O_6(C_4H_4N_2)]$ | Mo $K\alpha$ radiation |
| $M_r = 367.98$ | Cell parameters from 2590 |
| Orthorhombic, $Pbca$ | reflections |
| $a = 7.566$ (16) Å | $\theta = 2.9–25.1°$ |
| $b = 7.39$ (2) Å | $\mu = 3.20$ mm$^{-1}$ |
| $c = 14.04$ (3) Å | $T = 293$ (2) K |
| $V = 785$ (3) Å$^3$ | Block, black |
| $Z = 4$ | $0.10 \times 0.10 \times 0.06$ mm |
| $D_x = 3.113$ Mg m$^{-3}$ | |

### Data collection

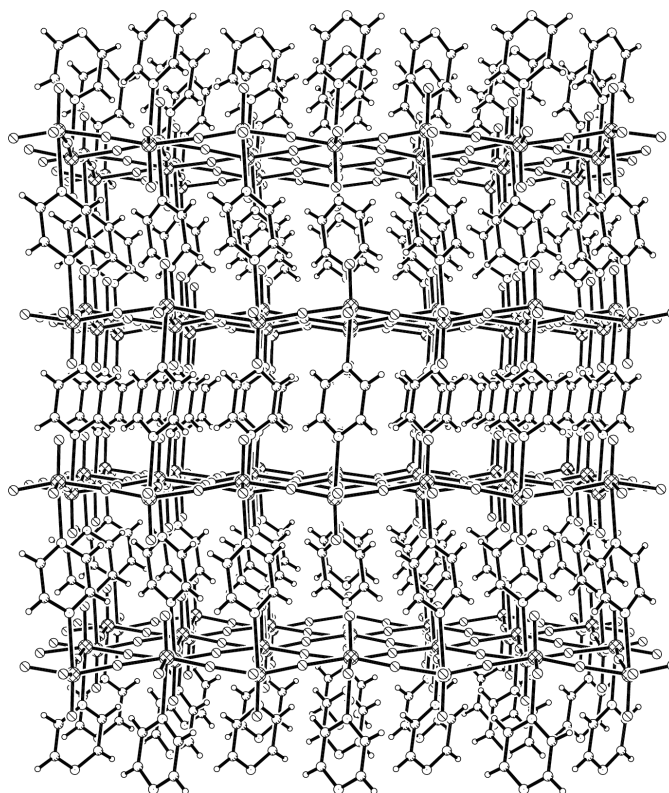| | |
|---|---|
| Siemens Smart CCD diffractometer | 630 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.027$ |
| Absorption correction: empirical | $\theta_{max} = 25.1°$ |
| (SADABS; Sheldrick, 1996) | $h = -9 \rightarrow 8$ |
| $T_{min} = 0.673$, $T_{max} = 0.825$ | $k = -3 \rightarrow 8$ |
| 3627 measured reflections | $l = -16 \rightarrow 13$ |
| 695 independent reflections | Intensity decay: none |



**Figure 2**
Packing diagram of compound (I).

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | $w = 1/[\sigma^2(F_o^2) + 18.7425P]$ |
| $wR(F^2) = 0.086$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.24$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 695 reflections | $\Delta\rho_{max} = 1.09$ e Å$^{-3}$ |
| 64 parameters | $\Delta\rho_{min} = -1.11$ e Å$^{-3}$ |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Mo—O3 | 1.682 (7) | Mo—O2 | 1.799 (8) |
| Mo—O1 | 1.755 (7) | Mo—N1 | 2.449 (8) |
| | | | |
| O3—Mo—O1 | 103.1 (3) | O2—Mo—O1$^{ii}$ | 84.0 (4) |
| O3—Mo—O2 | 101.9 (3) | O2$^i$—Mo—O1$^{ii}$ | 78.4 (4) |
| O1—Mo—O2 | 99.7 (5) | O3—Mo—N1 | 171.0 (3) |
| O3—Mo—O2$^i$ | 95.9 (3) | O1—Mo—N1 | 83.7 (3) |
| O1—Mo—O2$^i$ | 91.7 (4) | O2—Mo—N1 | 82.5 (3) |
| O2—Mo—O2$^i$ | 156.01 (9) | O2$^i$—Mo—N1 | 77.8 (3) |
| O3—Mo—O1$^{ii}$ | 95.1 (3) | O1$^{ii}$—Mo—N1 | 77.4 (2) |
| O1—Mo—O1$^{ii}$ | 160.1 (2) | | |

Symmetry codes: (i) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (ii) $x - \frac{1}{2}, y, \frac{3}{2} - z$.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SMART*; data reduction: *SMART*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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