

Xiao-Yuan Wu, Jiu-Hui Liu,
Quan-Zheng Zhang,
Xiang He, Shu-Mei Chen and
Can-Zhong Lu*State Key Laboratory of Structural Chemistry,
Fujian Institute of Research on the Structure of
Matter, The Chinese Academy of Science,
Fuzhou, Fujian 350002, People's Republic of
China

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

Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.012$ Å
H-atom completeness 46%
 R factor = 0.041
 wR factor = 0.112
Data-to-parameter ratio = 12.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.A polyoxomolybdate coordinated by glycine
ligands: $\text{K}_4[\text{Mo}_8\text{O}_{26}(\text{NH}_3\text{CH}_2\text{COO})_2] \cdot 6\text{H}_2\text{O}$

The title compound, tetrapotassium bis(glycine)hexacosaoxooctamolybdenum(VI) hexahydrate, $\text{K}_4[\text{Mo}_8\text{O}_{26}(\text{NH}_3\text{CH}_2\text{COO})_2] \cdot 6\text{H}_2\text{O}$, consists of a molecular network of centrosymmetric $[\text{Mo}_8\text{O}_{26}(\text{NH}_3\text{CH}_2\text{COO})_2]^{4-}$ anions, K^+ cations and water molecules, which interact by an extensive hydrogen-bonding network. The $[\text{Mo}_8\text{O}_{26}(\text{NH}_3\text{CH}_2\text{COO})_2]^{4-}$ anion is constructed from MoO_6 and $\text{MoO}_5(\text{OOCCH}_2\text{NH}_3)$ octahedral units, where the latter has O atoms of terminal and bridging oxo groups as well as those of glycine ligands.

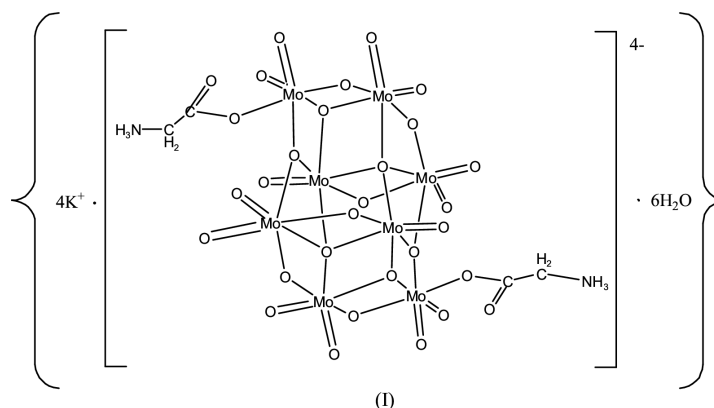
Received 12 May 2004

Accepted 1 June 2004

Online 12 June 2004

Comment

There is increasing interest in polyoxometalates, partly due to their interactions with enzymes and with different organic ligands, which enable a better understanding of the antitumor/viral activities of this class of compounds and modeling substrates involved in enzyme inhibition (Rhule *et al.*, 1998; Inoue & Yamase, 1995; Crans *et al.*, 1994). Thus, the reactivities of amino acids with polyoxometalates, as model studies for polyoxometalate–protein interactions, have attracted increasing attention. Some examples of structurally characterized polyoxoanions with covalently bound amino acids, $[\text{MMo}_6\text{O}_{21}(\text{L})_3]^{n-}$ ($M = \text{V}, \text{P}, \text{Se}, \text{Te}, \text{As}, \text{Sb}, \text{Bi}$; $L = \text{glycine}, \beta\text{-alanine}, 4\text{-aminobutyric acid}, \text{L-lysine}$), $[\text{Mo}_8\text{O}_{26}(\text{L-lysH}_2)_2]^{2-}$, $[\text{Mo}_{154}\text{O}_{462}\text{H}_{14}(\text{H}_2\text{O})_{48}(\text{HO}_2\text{C}-(\text{NH}^{3+})\text{HC}-\text{CH}_2-\text{S}-\text{S}-\text{CH}_2-\text{CH}(\text{NH}^{3+})-\text{COO}-)_{11}]^{3-}$, have been reported recently (Kortz *et al.*, 2002, 2003; Müller *et al.*, 2001). We report here the synthesis and the crystal structure of $\text{K}_4[\text{Mo}_8\text{O}_{26}(\text{NH}_3\text{CH}_2\text{COO})_2] \cdot 6\text{H}_2\text{O}$, (I) (Fig. 1), which contains octamolybdate coordinated by glycine.



In the centrosymmetric $[(\text{NH}_3\text{CH}_2\text{COO})_2\text{Mo}_8\text{O}_{26}]^{4-}$ anion of the title compound, the β -octamolybdate is coordinated by two glycines, each via one O atom of the carboxyl group. Bond-valence sum calculations (Brown & Altermatt, 1985;

Brese & O’Keeffe, 1991) indicated the valences of Mo and O to be +6 and –2, respectively. To keep the compound electrically neutral, three H atoms must be linked to each N atom.

The packing of (I) (Fig. 2) shows a network of weak N–H···O–Mo hydrogen bonds, with N···O = 2.851 (9)–3.064 (9) Å. In the crystal structure, the K1 cation is surrounded by nine O atoms, viz. eight from different polyoxoanions and one from a water molecule. The K2 cation is surrounded by eight O atoms, viz. four from different polyoxoanions and four from water molecules.

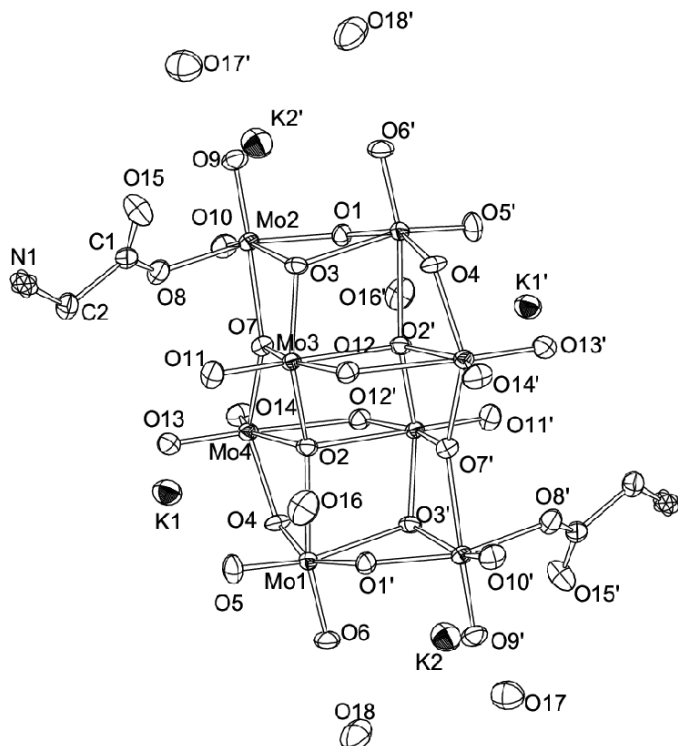


Figure 1
The molecular structure of (I) (50% displacement ellipsoids). H atoms have been omitted. The prime corresponds to symmetry code (i) in Table 1

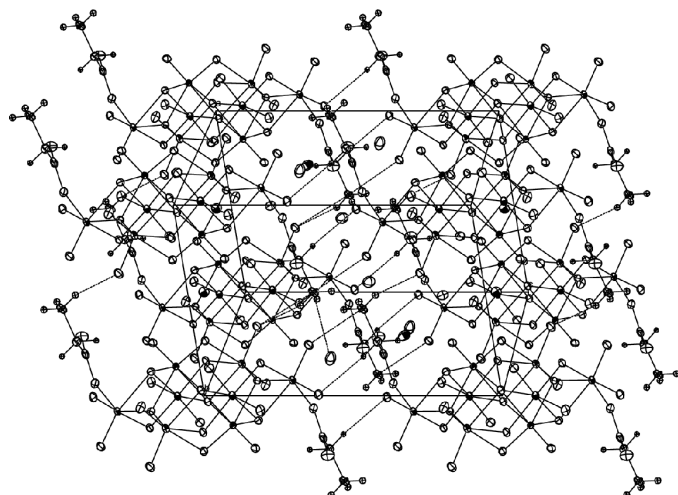


Figure 2
Packing diagram of (I), showing the hydrogen bonds as dashed lines (15% displacement ellipsoids).

Experimental

A mixture of K₂MoO₄ (5 mmol), KCl (5 mmol), H₂NCH₂COOH (15 mmol), N₂H₄·2HCl (0.5 mmol) and H₂O (10 ml) was sealed in a Teflon-lined stainless steel reactor and heated at 413 K for 4 d. After the reaction system was slowly cooled down to room temperature, small yellow crystals of (I) were obtained.

Crystal data

K₄[Mo₈O₂₆(C₂H₅NO₂)₂].6H₂O
M_r = 1598.16
 Triclinic, *P* $\bar{1}$
a = 8.1065 (3) Å
b = 9.8767 (2) Å
c = 12.4936 (5) Å
 α = 98.638 (2)°
 β = 99.295 (2)°
 γ = 113.606 (1)°
V = 878.66 (5) Å³

Z = 1
D_x = 3.020 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 4462 reflections
 θ = 1.70–25.04°
 μ = 3.35 mm⁻¹
T = 293 (2) K
 Block, yellow
 0.22 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.523, *T_{max}* = 0.715
 4462 measured reflections

3026 independent reflections
 2740 reflections with *I* > 2σ(*I*)
R_{int} = 0.026
 θ_{max} = 25.0°
h = –9 → 9
k = –11 → 11
l = –13 → 14

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.041
wR (*F*²) = 0.112
S = 1.05
 3026 reflections
 245 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0581*P*)² + 9.0351*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/*σ*)_{max} = 0.010
 Δ*ρ*_{max} = 0.80 e Å⁻³
 Δ*ρ*_{min} = –1.03 e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0011 (4)

Table 1
Selected bond distances (Å).

Mo1–O5	1.709 (5)	Mo3–O11	1.697 (5)
Mo1–O6	1.717 (5)	Mo3–O12	1.741 (5)
Mo1–O4	1.933 (5)	Mo3–O3	1.900 (5)
Mo1–O1 ⁱ	1.975 (5)	Mo3–O2	1.941 (5)
Mo1–O3 ⁱ	2.251 (5)	Mo3–O7	2.154 (5)
Mo1–O2	2.264 (5)	Mo3–O2 ⁱ	2.431 (5)
Mo2–O10	1.711 (5)	Mo4–O13	1.706 (5)
Mo2–O9	1.720 (5)	Mo4–O14	1.711 (5)
Mo2–O1	1.910 (5)	Mo4–O7	1.912 (5)
Mo2–O8	2.075 (5)	Mo4–O4	1.931 (5)
Mo2–O7	2.106 (5)	Mo4–O2	2.251 (5)
Mo2–O3	2.222 (5)	Mo4–O12 ⁱ	2.341 (5)

Symmetry code: (i) –*x*, –*y*, –*z*.

Table 2
Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1A···O6 ⁱⁱ	0.89	2.15	2.936 (9)	147
N1–H1B···O10 ⁱⁱⁱ	0.89	2.25	3.000 (9)	142
N1–H1C···O14 ^{iv}	0.89	2.26	2.851 (9)	124
N1–H1C···O10 ^{iv}	0.89	2.39	3.064 (9)	133

Symmetry codes: (ii) 1 – *x*, 1 – *y*, –*z*; (iii) –*x*, –*y*, –1 – *z*; (iv) 1 + *x*, *y*, *z*.

H atoms on C and N were positioned geometrically (N–H = 0.89 and C–H = 0.97 Å), and refined as riding, with *U*_{iso} = 1.5*U*_{eq}(N) or 1.2*U*_{eq}(C). Water H atoms were not located.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINTE* (Siemens, 1994); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97*; molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

This work was supported by the 973 Program of the MOST (001CB108906), the National Natural Science Foundation of China (90206040, 20333070 and 20303021), NSF of Fujian Province (2002F015 and 2002J006) and the Chinese Academy of Sciences.

References

- Brese, N. E. & O'Keeffe, M. (1991). *Acta Cryst.* **B47**, 192–197.
- Brown, I. D. & Altermatt, D. (1985). *Acta Cryst.* **B41**, 244–247.
- Crans, D. C., Mahroof-Tahir, M., Anderson, O. P. & Miller, M. M. (1994). *Inorg. Chem.* **33**, 5586–5590.
- Inoue, M. & Yamase, T. (1995). *Bull. Chem. Soc. Jpn.* **68**, 3055–3063.
- Kortz, U., Savelieff, M. G., Ghali, F. Y. A., Khalil, L. M., Maalouf, S. A. & Sinno, D. I. (2002). *Angew. Chem. Int. Ed.* **41**, 4070–4073.
- Kortz, U., Vaissermann, J., Thouvenot, R. & Gouzerh, P. (2003). *Inorg. Chem.* **42**, 1135–1139.
- Müller, A., Das, S. K., Kuhlmann, C., Bögge, H., Schmidtman, M., Diemann, E., Krickemeyer, E., Hormes, J., Modrow, H. & Schindler, M. (2001). *Chem. Commun.* pp. 655–656.
- Rhule, J. T., Hill, C. L. & Judd, D. A. (1998). *Chem. Rev.* **98**, 327–357.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Siemens (1994). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.