Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

A trinuclear molybdenum cluster coordinated by *N*-phenylanthranilate

Yao Kang, Zhao-Ji Li, Ye-Yan Qin, Yu-Biao Chen, Rui-Feng Hu, Yi-Hang Wen, Jian-Kai Cheng and Yuan-Gen Yao*

The 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: yyg@ms.fjirsm.ac.cn'

Received 2 July 2002
Accepted 9 July 2002
Online 19 July 2002

Key indicators

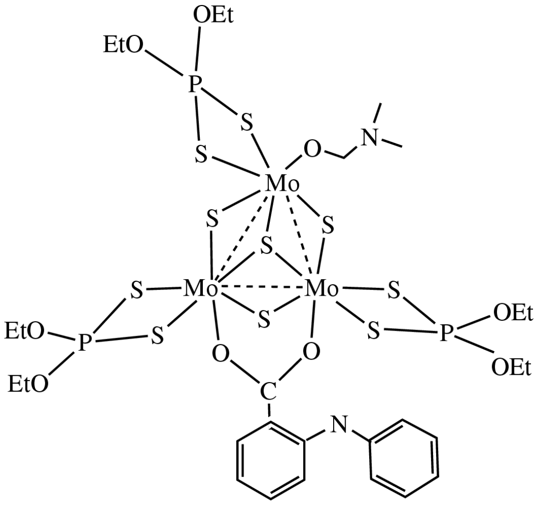
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.017 Å
R factor = 0.058
wR factor = 0.163
Data-to-parameter ratio = 17.2

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

The title compound, tris(diethyldithiophosphato)-1κ^2S,S';2κ^2S,S';3κ^2S,S'-μ-*o*-phenylanthranilato-1:3κ^2O,O'-*N*,*N*-dimethylformamide-2κO-μ_3-thio-tri-μ-thio-1:2κ^2S;1:3κ^2S; 2:3κ^2-*S*-*triangulo*-trimolybdenum(3Mo—Mo), [Mo_3S_4(*o*-C_6H_4CO-O-NH-C_6H_5)$(DTP)_3$(DMF)] or [$Mo_3S_4(C_{13}H_{10}NO_2)(C_4H_{10}O_2PS_2)_3(C_3H_7NO)$] (DTP is diethyl dithiophosphate and DMF is *N*,*N*-dimethylformamide), was synthesized from [$Mo_3S_4(DTP)_4(H_2O)$] in DMF by a ligand-substitution reaction, in which *N*-phenylanthranilate replaces the bridging DTP ligand. Its structure was determined by X-ray diffraction analysis. This ligand substitution shortens its bridged Mo—Mo bond from 2.734 (1) to 2.6847 (7) Å.

Comment

Recently, a rational synthetic route to a series of trinuclear molybdenum clusters with aromatic acid ligands has been developed in our laboratory. By this method, many derivatives with the Mo_3S_4 core have been synthesized from the cationic precursor $[Mo_3S_4(H_2O)_9]^{4+}$ or its neutral derivative [$Mo_3S_4(DTP)_4(H_2O)$] (Lin *et al.*, 1986), based on its diverse reactivity towards various metals and organic ligands. We report here the crystal structure of the title compound, [Mo_3S_4(*o*-$C_6H_4COONHC_6H_5$)$(DTP)_3$(DMF)] (DTP is diethyl dithiophosphate and DMF is *N*,*N*-dimethylformamide), (I), which was synthesized by the reaction of [$Mo_3S_4(DTP)_4(H_2O)$] with *N*-phenylanthranilic acid in a mixed solvent of DMF and EtOH.



(I)

The main structural feature of compound (I) (Fig. 1) is the typical incomplete cubane-type skeleton, which is shared by

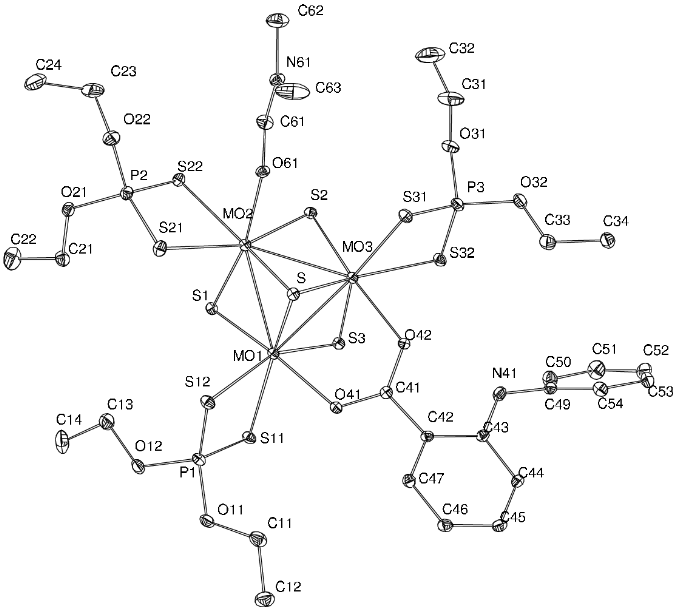


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

many trinuclear Mo—S(O) cluster compounds (Tang *et al.*, 2001). The substitution of bridging DTP by *N*-phenylanthranilate shortens the bridged Mo—Mo bond. The Mo1—Mo3 bond length of (I) is 2.6847 (7) Å. This is shorter than that of the precursor [$Mo_3S_4(DTP)_4(H_2O)$] [2.734 (1) Å; Lin *et al.*, 1986] and that of the analogous compound [$Mo_3S_4(DTP)_3(\mu\text{-}SOP(OEt)_2)(CH_3CN)$]·$CH_3CN$, (III) [2.732 (2) Å; Xia *et al.*, 1998]. This bond length is comparable to those of carboxylate-substituted trinuclear Mo—S(O) compounds, such as [$Mo_3S_4(DTP)_3(o\text{-}NO_2C_6H_4COO)(DMF)$], (II) [2.697 (1) Å; Tang *et al.*, 2001]. The Mo2—O61 bond length (to DMF) is similar to that in compound (II) [2.248 (5) Å; Tang *et al.*, 2001] and compound (III) [2.289 (1) Å; Xia *et al.*, 1998]. It is much shorter than that in [$Mo_3S_4(DTP)_4(H_2O)$].

The ^{31}P NMR spectrum of compound (I) (Fig. 2) was measured in dichloromethane. Two peaks at 108.98 p.p.m. (assigned to DTP coordinated to Mo1 and Mo3, respectively) and 109.88 p.p.m. (assigned to DTP coordinated to Mo2) were observed with the intensity ratio 2:1. Because of the steric bulk of the acid $o\text{-}C_6H_4COO\text{-}NH\text{-}C_6H_5$, no intermolecular S···S interaction is found in the crystal packing of compound (I). This contrasts with the analogous compounds [$Mo_3S_4(DTP)_3(CH_3C_6H_4SO_3)(C_2H_5OH)$] (Lu *et al.*, 1995) and [$Mo_3S_4(DTP)_3(o\text{-}HOC_6H_4COO)(CH_3CN)$] (Wu *et al.*, 1998), in which several intermolecular S···S interactions (S···S < 3.6 Å) are observed.

Experimental

[$Mo_3S_4(DTP)_4(H_2O)$] was synthesized according to the literature method of Yao *et al.* (1995); HDTP was purified before use by vacuum distillation (Bacon & Lesure, 1954). [$Mo_3S_4(DTP)_4(H_2O)$] (0.1 g, 0.085 mmol) and *N*-phenylanthranilic acid (0.1 g, 0.47 mmol) were dissolved in 10 ml DMF. After refluxing over an oil bath at 353 K for 30 min, the hot dark-brown solution was filtered into another flask, and 20 ml ethanol was then added to the solution. Rectangular black crystals precipitated over a period of 15 d (yield: 0.07 g, 65%). Analysis calculated for $C_{28}H_{47}Mo_3N_2O_9P_3S_{10}$: C,26.73, H 3.73, N 2.23%; found: C 27.10, H 3.51, N 2.20%.

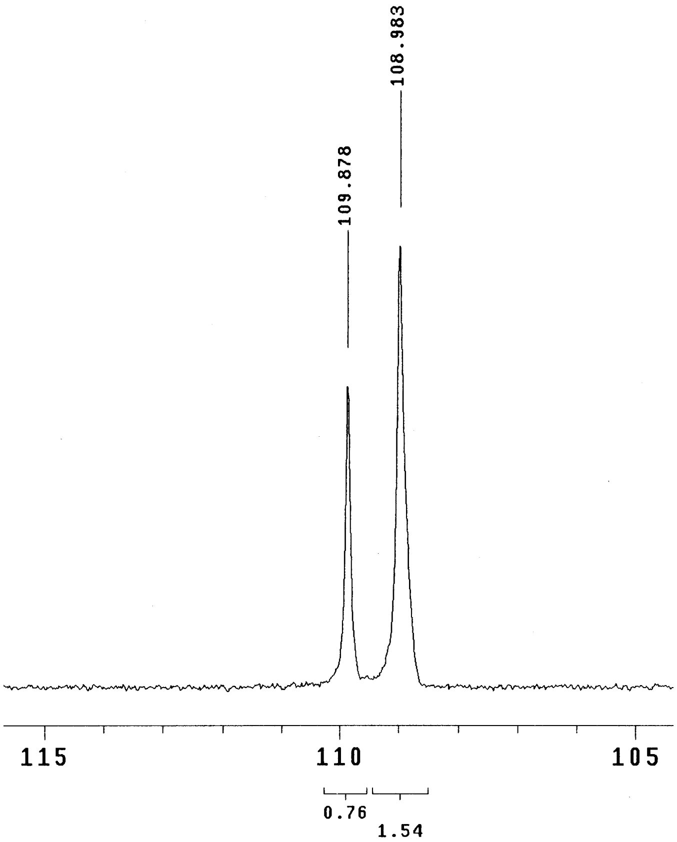


Figure 2
The ^{31}P NMR spectrum of compound (I).

Crystal data

[$Mo_3S_4(C_{13}H_{10}NO_2)$-
($C_4H_{10}O_2PS_2)_3(C_3H_7NO)$]
$M_r = 1257.01$
Triclinic, $P\overline{1}$
$a = 12.4661$ (2) Å
$b = 14.1240$ (2) Å
$c = 14.8507$ (1) Å
$\alpha = 74.787$ (1)°
$\beta = 76.232$ (1)°
$\gamma = 80.688$ (1)°
$V = 2436.84$ (5) Å^3

$Z = 2$
$D_x = 1.713$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6265 reflections
$\theta = 1.5$–25.1°
$\mu = 1.33$ mm^{-1}
$T = 293$ (2) K
Rectangular block, black
0.34 × 0.22 × 0.13 mm

Data collection

SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.630$, $T_{max} = 0.841$
12774 measured reflections

8550 independent reflections
6518 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 25.1°$
$h = -13 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -10 \rightarrow 17$

Acta Cryst. (2002). E**58**, m420–m422 Yao Kang *et al.* • [$Mo_3S_4(C_{13}H_{10}NO_2)(C_4H_{10}O_2PS_2)_3(C_3H_7NO)$] **m421**

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 6.1749P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.058$	$(\Delta/\sigma)_{\max} < 0.001$
$wR(F^2) = 0.163$	$\Delta\rho_{\max} = 0.75$ e Å^{-3}
$S = 1.18$	$\Delta\rho_{\min} = -0.73$ e Å^{-3}
8550 reflections	Extinction correction: *SHELXL*97
497 parameters	Extinction coefficient: 0.0029 (3)
H-atom parameters constrained	

The flexibility of the ethoxy group leads to the apparently short bond distances for C31—C32, C21—C22 and C23—C24.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *XPREP* in *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the NNSF of China (No. 20173063), Key Project in KIP of CAS (KJCX2-H3) and the NNSF of Fujian Province (E0020001) for financial support of this work.

References

Bacon, W. E. & Lesure, W. M. (1954). *J. Am. Chem. Soc.* **76**, 670–676.

Lin, X.-T., Lin, Y.-H., Huang, J.-L. & Huang, J.-Q. (1986). *Kexue Tongbao*, pp. 509–513.

Lu, S.-F., Huang, J.-Q., Huang, X.-Y., & Wu, Q.-J. (1995). *Jiegou Huaxue* (*Chin. J. Struct. Chem.*), **14**, 482–487.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Siemens (1994). *SAINT* and *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Tang, Y.-H., Qin, Y.-Y., Wu, L., Li, Z.-J., Kang, Y. & Yao, Y.-G., (2001). *Polyhedron*, **20**, 2911–2916.

Wu, L., Xia, J.-B., Chen, C.-N., Huang, X.-Y., Yao, Y.-G. & Lu, J.-X., (1998). *Polyhedron*, **17**, 4203–4212.

Xia, J.-B., Wu, L., Wang, Q.-M., Chen, J.-T., Wu, D.-X., Yao, Y.-G. & Lu, J.-X. (1998). *Jiegou Huaxue* (*Chin. J. Struct. Chem.*), **17**, 302–308.

Yao, Y.-G., Akashi, H., Sakane, G., Shibahara, T. & Ohtaki, H. (1995). *Inorg. Chem.* **34**, 42–48.

m422 Yao Kang *et al.* • $[Mo_3S_4(C_{13}H_{10}NO_2)(C_4H_{10}O_2PS_2)_3(C_3H_7NO)]$ *Acta Cryst.* (2002). E**58**, m420–m422