0.25 was too high, and there were many peaks in the difference electron-density map which could not be incorporated into the model. A twin model was then proposed such that the crystal is composed of two twin domains, with the reciprocal lattices of these two components being coincident: $I(hkl) = (1-\alpha)I(hkl) + \alpha(\bar{k}\bar{h}\bar{l})$. In *SHELXL*93 (Sheldrick, 1993), this is incorporated into the least-squares refinement by a twin matrix of $(0\bar{1}0/\bar{1}00/00\bar{1})$ and refinement of the twin fraction α. Application of this twin law resulted in a dramatic drop in the R factor to 0.076 [isotropic refinement on $I > 3\sigma(I)$] and a clean difference electron-density map. This is an example of a twin by merohedry with the crystal twinned on the (110) plane. A linear decay correction was applied to the data (*TEXSAN*; Molecular Structure Corporation, 1989). Averaging the symmetry equivalent reflections within the two measured octants, $(+h,+k,+l)$ and $(-h,+k,+l)$, resulted in an $R_{\rm int}$ value of 0.022. The twin fraction refined to a value of 0.334 (1). Three reflections were omitted from the refinement because of large negative F_o values, *i.e.* $(\bar{2},18,1)$, $(\bar{1},14,2)$ and $(\overline{11},17,1)$. H atoms for both structures were included in the model at calculated positions using a riding model, with aromatic C—H = 0.93, secondary C—H = 0.97, tertiary C—H = 0.98 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(bonded C atom). *PLATON* (Spek, 1990) was used to calculate some metric parameters.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995) for (3); *TEXSAN* (Molecular Structure Corporation, 1989) for (4). For both compounds, program(s) used to solve structures: *SHELXS*86; program(s) used to refine structures: *SHELXL*93; molecular graphics: *ORTEP*II (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1123). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1612–1615

A Trinuclear Molybdenum Cluster Coordinated by *o*-Nitrobenzoate

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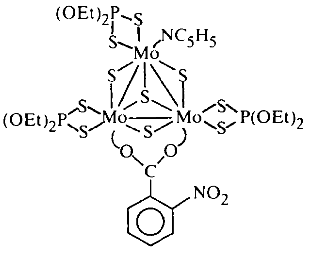
Abstract

The title compound, tris(diethyl dithiophosphato)-$1\kappa^2S,S';2\kappa^2S,S';3\kappa^2S,S'$-$\mu$-*o*-nitrobenzoato-$2\kappa O:3\kappa O'$-pyridine-$1\kappa N$-$\mu_3$-thio-tri-$\mu$-thio-$1{:}2\kappa^2S;1{:}3\kappa^2S;2{:}3\kappa^2S$-*triangulo*-trimolybdenum(3 *Mo*—*Mo*), $[Mo_3S_4(C_7H_4NO_4)(C_4H_{10}PS_2)_3(C_5H_5N)]$, was synthesized in a mixed solvent of acetonitrile and ethanol by a ligand-substitution reaction in which *o*-nitrobenzoic acid replaces the bridging diethyl dithiophosphate (DTP) ligand. The average Mo—Mo bond distance is 2.731 (2) Å. The substitution of *o*-nitrobenzoate for DTP shortens the Mo—Mo bond that it bridges to 2.693 (1) Å.

Comment

In the course of our study on the incomplete cubane-type Mo-S(O) cluster compounds, one of the authors proposed that there is quasi-aromaticity in the puckered ring of the Mo_3S_3 moiety (Chen *et al.*, 1990). We were interested in studying the electron delocalization between the Mo cluster core (Mo_3S_4) and the aromatic ligands; thus, the synthesis of the title compound, (I), was designed. The reaction of $[Mo_3S_4(DTP)_4(H_2O)]$, (II) (Lin *et al.*, 1986), with benzoic acid yielded a brown powder which proved difficult to dissolve in any solvent tried. Nevertheless, the reaction with *o*-nitrobenzoic acid and pyridine (py) gave good crystals of the title compound, $[Mo_3S_4(DTP)_3(2\text{-}NO_2C_6H_4CO_2)(py)]$ (DTP is diethyl dithiophosphate), (I).



(I)

The main structural feature of compound (I) (Fig. 1) is similar to that of the many incomplete cubane-type cluster compounds with organic ligands (Shibahara, 1991). The dihedral angle calculated between the plane through atoms Mo2, Mo3, O1, O2, C1 and C2, and that through atoms C1–C7 and N1 is 46.3 (6)°. There is a dihedral angle of 31.7 (3)° between the plane of Mo1, Mo2 and Mo3, and the plane of Mo2, Mo3, O1, O2, C1 and C2, which reveals heavily distorted octahedral Mo coordination. The substitution of bridging DTP by organic acids shortens the bridged Mo—Mo bond distance. The Mo2—Mo3 bond of the title compound is 2.693 (1) Å, which is shorter than the DTP-bridged Mo2—Mo3 bond of compound (II) [2.734 (1) Å; Lin *et al.*, 1986]. Furthermore, substitution by aromatic acids shortens the bridged Mo—Mo bond more than substitution by aliphatic acids (Huang *et al.*, 1988).

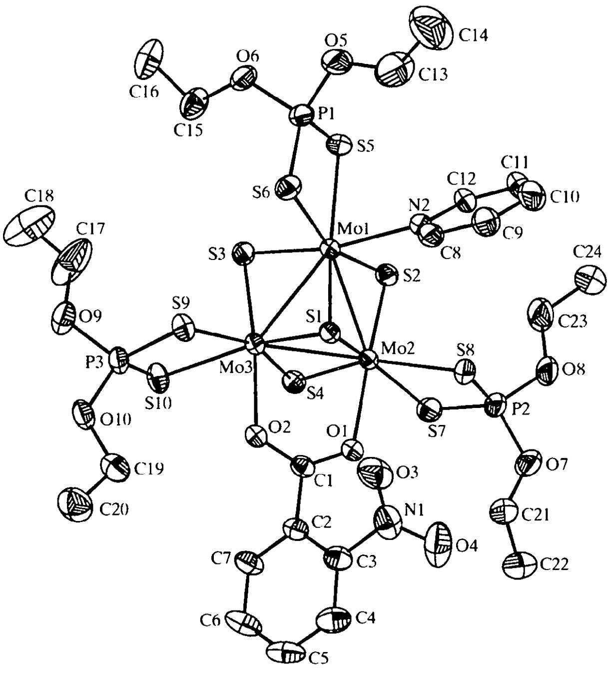


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

The ^{31}P NMR spectra of compound (I) were measured in deuterated acetone (Yao *et al.*, 1995). Two peaks at 111.16 (assigned to the two DTPs coordinated to Mo2 and Mo3) and 110.20 p.p.m. (assigned to the DTP coordinated to Mo1) were observed with the intensity ratio 2:1.

Among the calculated intermolecular atomic distances, no S···S interaction between two molecules in the cell was found. This contrasts with the analogous compounds $[Mo_3S_4(DTP)_3(CH_3C_6H_4SO_3)(C_2H_5OH)]$ (Lu *et al.*, 1995) and $[Mo_3S_4(DTP)_3(2\text{-}HOC_6H_4CO_2)(CH_3CN)]$ (Xia *et al.*, 1998), in which several intermolecular S···S interactions (S···S < 3.60 Å) are observed. The loosely coordinated pyridine ligand of each molecule occupies the space between two molecules and hinders S···S interactions.

Experimental

$[Mo_3S_4(DTP)_4(H_2O)]$ was synthesized according to the literature method of Yao *et al.* (1995); however, HDTP was purified before use by vacuum distillation (Bacon & LeSure, 1954; Olah & McFarland, 1975). In a flask, $[Mo_3S_4(DTP)_4(H_2O)]$ (0.2 g) was dissolved in ethanol–acetonitrile (40 ml, *v*/*v* = 1/1); *o*-nitrobenzoic acid (0.03 g) and a few drops of pyridine were then added. After refluxing over an oil bath at 363 K for 1 h, the hot dark-brown solution was filtered into another flask. Rectangular black crystals precipitated over a period of 10 d (yield: 0.12 g, 60%).

Crystal data

$[Mo_3S_4(C_7H_4NO_4)(C_4H_{10}PS_2)_3(C_5H_5N)]$
$M_r = 1216.97$
Triclinic
$P\bar{1}$
$a = 10.526 (6)$ Å
$b = 14.030 (4)$ Å
$c = 16.884 (4)$ Å
$\alpha = 93.16 (2)°$
$\beta = 103.19 (3)°$
$\gamma = 110.99 (3)°$
$V = 2241 (3)$ Å^3
$Z = 2$
$D_x = 1.80$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 20 reflections
$\theta = 10.50–14.95°$
$\mu = 1.41$ mm^{-1}
$T = 293$ K
Cuboid
0.80 × 0.60 × 0.40 mm
Black

Data collection

Enraf–Nonius CAD-4 diffractometer

6835 reflections with $I > 3\sigma(I)$

$\omega/2\theta$ scans
Absorption correction:
ψ scan (North *et al.*, 1968)
$T_{min} = 0.440$, $T_{max} = 0.569$
7864 measured reflections
7864 independent reflections

$\theta_{max} = 25°$
$h = 0 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 20$
3 standard reflections
every 60 reflections
intensity decay: 0.8%

Refinement

Refinement on F
$R = 0.044$
$wR = 0.055$
$S = 1.80$
6835 reflections
470 parameters
H atoms not refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 1.08$ e Å^{-3}
$\Delta\rho_{min} = -1.09$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Mo1—Mo2	2.745 (1)	Mo3—S9	2.512 (2)
Mo1—Mo3	2.756 (2)	Mo3—S10	2.558 (2)
Mo1—S1	2.335 (1)	Mo3—O2	2.201 (3)
Mo1—S2	2.291 (2)	O1—C1	1.266 (5)
Mo1—S3	2.283 (1)	O2—C1	1.263 (6)
Mo1—S5	2.568 (2)	O3—N1	1.213 (8)
Mo1—S6	2.539 (2)	O4—N1	1.202 (8)
Mo1—N2	2.368 (4)	N1—C3	1.474 (7)
Mo2—Mo3	2.693 (1)	N2—C8	1.334 (7)
Mo2—S1	2.329 (2)	N2—C12	1.345 (6)
Mo2—S2	2.288 (1)	C1—C2	1.487 (5)
Mo2—S4	2.297 (2)	C2—C3	1.384 (7)
Mo2—S7	2.555 (2)	C2—C7	1.383 (6)
Mo2—S8	2.514 (2)	C3—C4	1.374 (6)
Mo2—O1	2.239 (3)	C4—C5	1.359 (8)
Mo3—S1	2.323 (1)	C5—C6	1.37 (1)
Mo3—S3	2.282 (1)	C6—C7	1.373 (7)
Mo3—S4	2.290 (2)		
Mo2—Mo1—Mo3	58.63 (4)	S4—Mo2—O1	83.02 (9)
Mo2—Mo1—S1	53.85 (4)	S7—Mo2—S8	77.28 (6)
Mo2—Mo1—S2	53.12 (4)	S7—Mo2—O1	84.4 (1)
Mo2—Mo1—S3	98.37 (5)	S8—Mo2—O1	82.19 (9)
Mo2—Mo1—S5	139.77 (5)	Mo1—Mo3—Mo2	60.47 (3)
Mo2—Mo1—S6	140.91 (4)	Mo1—Mo3—S1	53.93 (4)
Mo2—Mo1—N2	91.9 (1)	Mo1—Mo3—S3	52.89 (4)
Mo3—Mo1—S1	53.52 (4)	Mo1—Mo3—S4	96.71 (5)
Mo3—Mo1—S2	97.74 (5)	Mo1—Mo3—S9	102.39 (5)
Mo3—Mo1—S3	52.83 (4)	Mo1—Mo3—S10	144.61 (4)
Mo3—Mo1—S5	140.04 (4)	Mo1—Mo3—O2	133.93 (8)
Mo3—Mo1—S6	102.10 (5)	Mo2—Mo3—S1	54.74 (4)
Mo3—Mo1—N2	136.71 (8)	Mo2—Mo3—S3	99.89 (4)
S1—Mo1—S2	105.44 (5)	Mo2—Mo3—S4	54.17 (4)
S1—Mo1—S3	104.39 (5)	Mo2—Mo3—S9	139.15 (4)
S1—Mo1—S5	161.46 (5)	Mo2—Mo3—S10	138.77 (5)
S1—Mo1—S6	87.10 (5)	Mo2—Mo3—O2	85.17 (9)
S1—Mo1—N2	83.89 (9)	S1—Mo3—S3	104.85 (5)
S2—Mo1—S3	98.58 (6)	S1—Mo3—S4	108.33 (6)
S2—Mo1—S5	86.65 (6)	S1—Mo3—S9	84.81 (5)
S2—Mo1—S6	160.16 (4)	S1—Mo3—S10	156.93 (5)
S2—Mo1—N2	85.4 (1)	S1—Mo3—O2	81.79 (9)
S3—Mo1—S5	87.21 (5)	S3—Mo3—S4	94.54 (6)
S3—Mo1—S6	92.92 (6)	S3—Mo3—S9	95.48 (6)
S3—Mo1—N2	169.36 (9)	S3—Mo3—S10	91.73 (6)
S5—Mo1—S6	77.77 (6)	S3—Mo3—O2	173.18 (8)
S5—Mo1—N2	83.14 (9)	S4—Mo3—S9	160.81 (4)
S6—Mo1—N2	80.7 (1)	S4—Mo3—S10	85.76 (6)
Mo1—Mo2—Mo3	60.90 (3)	S4—Mo3—O2	84.7 (1)
Mo1—Mo2—S1	54.06 (4)	S9—Mo3—S10	77.62 (6)
Mo1—Mo2—S2	53.23 (4)	S9—Mo3—O2	83.4 (1)
Mo1—Mo2—S4	96.86 (5)	S10—Mo3—O2	81.46 (9)
Mo1—Mo2—S7	141.96 (4)	Mo1—S1—Mo2	72.09 (5)
Mo1—Mo2—S8	104.58 (5)	Mo1—S1—Mo3	72.55 (5)
Mo1—Mo2—O1	133.7 (1)	Mo2—S1—Mo3	70.74 (5)
Mo3—Mo2—S1	54.52 (4)	Mo1—S2—Mo2	73.64 (5)
Mo3—Mo2—S2	99.61 (4)	Mo1—S3—Mo3	74.28 (5)
Mo3—Mo2—S4	53.94 (5)	Mo2—S4—Mo3	71.90 (5)
Mo3—Mo2—S7	139.15 (4)	Mo2—O1—C1	122.0 (3)
Mo3—Mo2—S8	138.85 (4)	Mo3—O2—C1	122.2 (2)
Mo3—Mo2—O1	83.68 (9)	O3—N1—O4	125.7 (6)
S1—Mo2—S2	105.75 (5)	O3—N1—C3	117.7 (5)
S1—Mo2—S4	107.88 (6)	O4—N1—C3	116.6 (6)
S1—Mo2—S7	158.95 (5)	Mo1—N2—C8	121.6 (3)
S1—Mo2—S8	85.23 (5)	Mo1—N2—C12	121.2 (4)
S1—Mo2—O1	81.7 (1)	C8—N2—C12	117.1 (4)
S2—Mo2—S4	93.38 (5)	O1—C1—O2	126.9 (4)
S2—Mo2—S7	88.76 (6)	O1—C1—C2	117.8 (4)
S2—Mo2—S8	99.38 (5)	O2—C1—C2	115.2 (4)
S2—Mo2—O1	172.4 (1)	C1—C2—C3	122.4 (4)
S4—Mo2—S7	85.89 (6)	N2—C8—C9	123.5 (5)
S4—Mo2—S8	158.56 (5)	N2—C12—C11	122.5 (6)

The positions of the Mo atoms were located by direct methods and those of the other atoms from successive difference Fourier syntheses. The highest peak on the final difference Fourier map had a height of 1.076 e Å^{-3} and was located at a position 1.04 Å from Mo1. The next highest peak was 1.067 e Å^{-3}, 1.13 Å from Mo3.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1987). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983) in *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *GCIF* (local program).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1109). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1615–1616

Hexapotassium μ-Oxo-bis[(citrato)-dioxomolybdenum] Dihydrate

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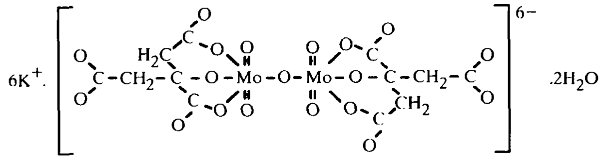
Abstract

In the title compound, hexapotassium μ-oxo-bis{dioxo[2-hydroxy-κO-1,2,3-propanetricarboxylato(4−)-$\kappa^2 O^1,O^2$]molybdenum(VI)} dihydrate, $K_6[\{MoO_2(C_6H_4O_7)\}_2O].2H_2O$, the citrate, as a tridentate ligand, is coordinated to molybdenum through the deprotonated hydroxy group, the α-carboxylate group and also through one β-carboxylate group. The coordination polyhedron of the Mo atom is distorted octahedral, containing one $[Mo_2O_5]^{2+}$ core. Principal dimensions are: Mo—O$_t$ = 1.715 (av), Mo—O$_b$ = 1.915 (2), Mo—O$_{\rm hydroxy}$ = 1.952 (3), Mo—O$_{\alpha-\rm carboxy}$ = 2.216 (4), Mo—O$_{\beta-\rm carboxy}$ = 2.261 (4) Å and Mo—O$_b$—Mo = 137.1 (4)°.

Comment

There is at present a growing interest in the chemistry of the coordination compounds of molybdenum, particularly because of their possible relationship to redox-active molybdoenzymes (Chan *et al.*, 1993; Russ, 1996). However, structural studies on oxomolybdenum complexes containing a citrate ligand are rare. One complex, $K_2Na_4[(MoO_2)_2O(cit)_2].5H_2O$ (cit = citrate), has been structurally characterized by X-ray diffraction (Zhou *et al.*, 1997), and the title complex, (I), was considered to be isomorphous with this and to have the same coordination mode. Structure analysis shows that the dimeric anion of (I) consists of the common oxo-bridged $[Mo_2O_5]^{2+}$ entity, which is non-centrosymmetric. Each Mo atom is six-coordinate, with approximately octahedral geometry. The terminal and bridging oxo groups adopt a *fac* stereochemistry. Other coordination positions are occupied by a fully deprotonated tridentate citrate ligand, which forms both five-membered and six-membered chelate rings. The O1—Mo1—O5 and O1—Mo1—O6 bond angles are 73.86 (13) and 79.68 (14)°, respectively. A comparison of the title compound with two related compounds, namely, $K_2Na_4[(MoO_2)_2O(cit)_2].5H_2O$ (Zhou *et al.*, 1997) and $Na_6[W_2O_5(cit)_2].10H_2O$ (Llopis *et al.*, 1993), indicates that both reported structures, as well as the present one, have almost the same structural features. The corresponding bond distances and angles to *M* (*M* = Mo or W) are similar, except for the *M*—O$_{\beta-\rm carboxy}$ distance and the *M*—O$_b$—*M* angle of the title compound being different to those of $Na_6[W_2O_5(cit)_2].10H_2O$ (Llopis *et al.*, 1993) {*M*—O$_{\beta-\rm carboxy}$ = 2.261 (4) Å and *M*—O$_b$—*M* = 137.1 (4)° for (I), and 2.289 (2) Å and 180° for $Na_6[W_2O_5(cit)_2].10H_2O$}.



(I)

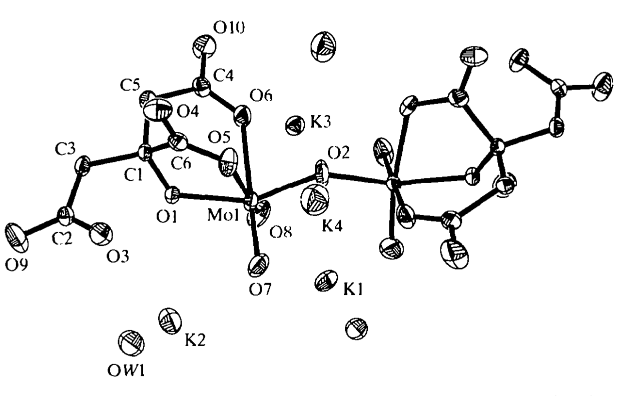


Fig. 1. A view of the title complex showing displacement ellipsoids drawn at the 50% probability level and the atom-labelling scheme.