433.4, 422.2, 358.7, 337.5, 283.5, 239.1, 223.7, 198.3, 177.4, 150.4; UV-vis [CH_3COCH_3, λ (nm)]: 444, 402, 340, 315; ^{1}H NMR [CD_3COCD_3, δ (p.p.m.)]: 7.20–7.60 (*m*, 10H, SPh), 3.46 (*t*, 8^aH, $C^aH_2CH_2CH_2CH_3$), 1.83 (*m*, 8^bH, $CH_2C^bH_2CH_2CH_3$), 1.47 (*m*, 8^cH, $CH_2CH_2C^cH_2CH_3$), 1.01 (*t*, 12^dH, $CH_2CH_2CH_2C^dH_3$).

Crystal data

$(C_{16}H_{36}N)[Mo_2I-(C_6H_5S)_2(CO)_6]$
$M_r = 947.62$
Monoclinic
$P2_1/n$
$a = 9.8951\ (4)$ Å
$b = 16.2430\ (7)$ Å
$c = 25.0787\ (10)$ Å
$\beta = 90.444\ (1)°$
$V = 4030.7\ (3)$ Å^3
$Z = 4$
$D_x = 1.562$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6187 reflections
$\theta = 1.49–25.02°$
$\mu = 1.531$ mm^{-1}
$T = 293\ (2)$ K
Needle
$0.40 \times 0.15 \times 0.10$ mm
Black

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.698$, $T_{max} = 0.894$
13 287 measured reflections
6816 independent reflections

4719 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 25.02°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 29$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.089$
$S = 1.054$
6816 reflections
415 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0168P)^2 + 2.8450P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.384$ e Å^{-3}
$\Delta\rho_{min} = -0.428$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

I—Mo1	2.9460 (6)	Mo1—Mo2	2.8824 (7)
I—Mo2	2.9599 (6)	Mo2—C6	1.947 (7)
Mo1—C3	1.919 (7)	Mo2—C5	1.992 (6)
Mo1—C1	2.004 (7)	Mo2—C4	2.000 (7)
Mo1—C2	2.017 (7)	Mo2—S1	2.4783 (15)
Mo1—S1	2.4675 (15)	Mo2—S2	2.4836 (15)
Mo1—S2	2.4714 (15)		
Mo1—I—Mo2	58.43 (2)	Mo1—S1—Mo2	71.29 (4)
S1—Mo1—S2	108.84 (5)	Mo1—S2—Mo2	71.14 (4)
S1—Mo2—S2	108.10 (5)		

Data were collected over a hemisphere of reciprocal space by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 4.95 cm. Coverage of the unique set was over 99% complete to at least 25° in θ. Crystal decay was monitored by measurement of duplicate reflections. H atoms were all located theoretically and not refined.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SMART* and *SAINT* (Siemens, 1994*b*). Data reduction: *XPREP* in *SHELXTL* (Siemens, 1994*a*). 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*.

We are grateful to NNSF and SKLSC for financial support of this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1215). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 298–300

A dimolybdenum(I) carbonyl compound with thiolate and carboxylate bridges: tetrabutylammonium bis(μ-benzenethiolato-*S*:*S*)hexacarbonyl-μ-pivalate-*O*:*O*′-dimolybdenum(*Mo*—*Mo*)

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(*Received 10 June 1998; accepted 18 September 1998*)

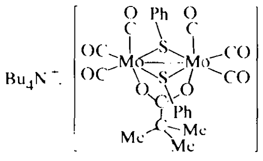
Abstract

In the anion of the title compound, $(C_{16}H_{36}N)[Mo_2(C_5H_9O_2)(C_6H_5S)_2(CO)_6]$, each Mo atom and three terminal carbonyl groups form a *fac*-$Mo(CO)_3$ fragment, and two Mo atoms and two benzenethiolate bridging

ligands form a planar Mo_2S_2 unit, with the two phenyl rings in an *anti* configuration. The pivalate acts as a conventional μ,η^2-ligand bridge to two Mo atoms, to form an approximately planar five-membered ring. The two $[MoOS_2(CO)_3]$ octahedra share a common S–S edge.

Comment

We have been investigating the syntheses and structures of dimolybdenum(I) compounds containing bridging thiolate, as well as other inorganic and organic ligands. It has been found that the phenyl rings of two benzenethiolate ligands adopt either *syn* or *anti* configurations when various bridging ligands are introduced to substitute the axial CO in the parent compound, $[Mo_2(C_6H_5S)_2(CO)_6]$, (I) (Pan *et al.*, 1998). The introduction of a halogen-ion bridging ligand leads to a *syn* configuration, while a carboxylate ligand leads to an *anti* configuration. Despite the greater steric hindrance of the pivalate in the title compound, (II), the two phenyl rings are in an *anti* configuration.



(II)

The structure of the anion of (II) is shown in Fig. 1. Each Mo atom has a distorted octahedral geometry, involving three C atoms from carbonyls, two S atoms from benzenethiolate bridging groups and one O atom from the pivalate ligand. Two *fac*-$Mo(CO)_3$ fragments are linked together by two benzenethiolate and one pivalate, forming an edge-sharing bi-octahedral structure.

The structure of the Mo_2S_2 unit in (II) is similar to that in compound (I) (Zhuang *et al.*, 1995). The introduction of a pivalate ligand causes the Mo_2S_2 unit to contract slightly, resulting in smaller Mo—S—Mo angles and a shorter Mo—Mo distance, but the Mo—S distances are essentially equal to those of (I). The *trans*-Mo—C distances are considerably shorter than those *cis* to the pivalate ligand. This indicates that a tetrabridged compound substituting the axial CO in compound (II) further, with a pivalate, would be difficult to prepare.

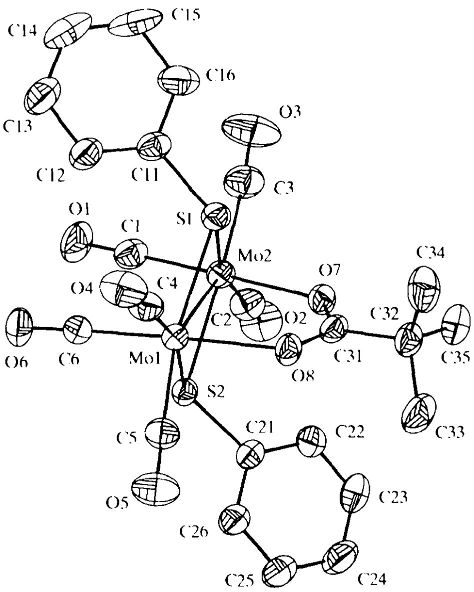


Fig. 1. View of the anion of (II), with displacement ellipsoids shown at the 30% probability level and H atoms omitted for clarity.

The pivalate ligand substitutes two axial carbonyls of (I), forming an approximately planar five-membered Mo–O–C–O–Mo ring. The Mo—Mo distance is long at 2.9071 (8) Å, leading to a slightly larger O—C—O angle [125.1 (6)°], and slightly more acute O8—Mo1—Mo2 [81. 13 (11)°] and O7—Mo2—Mo1 [81.45 (11)°] angles in the five-membered ring. It is worth noting that the Mo1—O8 distance [2.215 (4) Å] is slightly shorter than the Mo2—O7 distance [2.234 (4) Å], but the O7—C31 distance [1.257 (7) Å] is equal to the O8—C31 distance [1.260 (7) Å].

Experimental

The title compound was synthesized by the reaction of $Mo_2(CO)_8(C_6H_5S)_2$ (Zhuang *et al.*, 1984; Smith *et al.*, 1987) with Bu_4NBr and $(CH_3)_3CCOONa$ (1:1:1) in acetone, and crystallized by adding ^{i}PrOH to the filtrate and allowing it to stand at a temperature below 273 K for several days. Analysis calculated for $C_{39}H_{55}Mo_2NO_8S_2$: C 50.75, H 5.97, N 1.52%; found: C 50.76, H 6.01, N 1.54%; IR (KBr, cm^{-1}, ν_{CO}): 1997.9 (*s*), 1949.6 (*s*), 1930.4 (*s*), 1905.3 (*s*), 1889.9 (*s*), 1847.5 (*s*), 1820.5 (*s*); ν_{COO}: 1540.9 (*s*), 1417.4 (*s*).

Crystal data

$(C_{16}H_{36}N)[Mo_2(C_5H_9O_2)(C_6H_5S)_2(CO)_6]$
$M_r = 921.84$
Monoclinic
$P2_1/c$
$a = 14.4791$ (2) Å
$b = 12.9825$ (2) Å
$c = 24.4658$ (3) Å
$\beta = 104.790$ (1)°
$V = 4446.58$ (11) Å^3
$Z = 4$
$D_x = 1.377$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8192 reflections
$\theta = 1.45$–23.26°
$\mu = 0.704$ mm^{-1}
$T = 293$ (2) K
Block
0.36 × 0.32 × 0.28 mm
Green

Data collection

Siemens SMART CCD diffractometer

4577 reflections with $I > 2\sigma(I)$

ω scans
Absorption correction:
empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.562$, $T_{max} = 0.862$
16 882 measured reflections
6373 independent reflections

$R_{int} = 0.038$
$\theta_{max} = 23.26°$
$h = -16 \rightarrow 15$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 27$
Intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.135$
$S = 1.011$
6373 reflections
384 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 11.5194P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.817$ e Å^{-3}
$\Delta\rho_{min} = -0.622$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Mo1—C6	1.932 (7)	Mo2—C2	2.009 (8)
Mo1—C4	1.995 (8)	Mo2—C3	2.013 (8)
Mo1—C5	1.998 (7)	Mo2—O7	2.234 (4)
Mo1—O8	2.215 (4)	Mo2—S2	2.460 (2)
Mo1—S1	2.477 (2)	Mo2—S1	2.464 (2)
Mo1—S2	2.482 (2)	O7—C31	1.257 (7)
Mo1—Mo2	2.9071 (8)	O8—C31	1.260 (7)
Mo2—C1	1.936 (8)		
S1—Mo1—S2	107.36 (6)	Mo2—S1—Mo1	72.08 (5)
O8—Mo1—Mo2	81.13 (11)	Mo2—S2—Mo1	72.06 (5)
S2—Mo2—S1	108.45 (6)	O7—C31—O8	125.1 (6)
O7—Mo2—Mo1	81.45 (11)		

Data were collected over a hemisphere of reciprocal space by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 4.95 cm. Coverage of the unique set was over 99% complete to at least 23° in θ. Crystal decay was monitored by measurement of duplicate reflections. H atoms were all located theoretically and were not refined. The lengths of some C—C bonds in the cation are shorter than is reasonable; this is due to high thermal motion and disorder.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SMART* and *SAINT* (Siemens, 1994*a*). Data reduction: *XPREP* in *SHELXTL* (Siemens, 1994*b*). Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: Siemens *SHELXTL*.

We are grateful to NNSF and SKLSC for financial support of this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1216). Services for accessing these data are described at the back of the journal.

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trans-Diaquabis(quinoline-2-carboxylato-*N*,*O*)cobalt(II)–water–ethanol (1/2/2)

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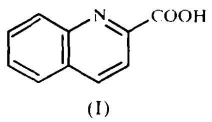
Abstract

The title compound, $[Co(C_{10}H_6NO_2)_2(H_2O)_2]\cdot 2H_2O\cdot 2C_2H_5OH$, contains a six-coordinate Co^{II} ion at a center of symmetry. The Co^{II} ion displays distorted octahedral coordination geometry defined by the two quinoline N atoms, two O atoms of the carboxylate groups and two O atoms of the water molecules. All of the corresponding pairs of ligand atoms lie in *trans* positions with respect to each other. Molecules are linked together by an intermolecular hydrogen-bonding network involving the uncoordinated water and ethanol molecules.

Comment

2-Quinolinecarboxylic acid, (I), is an intermediate tryptophan metabolite and is known to chelate transition metal ions (Martell & Smith, 1974). Crystal structures of complexes of 2-quinolinecarboxylic acid have been determined for several metal ions, including Cu^{II} (Haendler, 1986), Mn^{II} (Haendler, 1996; Okabe & Koizumi, 1997) and Fe^{II} (Okabe & Makino, 1998). We have carried out the structural analysis of the Co^{II} complex, (II), and report the results here.



(I)

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