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A novel organometallic macrocycle containing a tetrahedral $Mo_2Fe(\mu_3\text{-}S)$ cluster core

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Key indicators

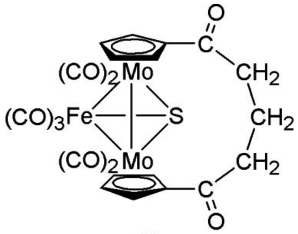
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.020
wR factor = 0.051
Data-to-parameter ratio = 12.6

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

The title compound, [μ_2-η^5,η^5-1,1′-(propane-1,3-diyldicarbonyl)dicyclopentadienyl]-μ_3-thio-heptacarbonylirondimolybdenum(2Fe—Mo, Mo—Mo), $[Mo_2Fe(C_5H_6O_2)S(CO)_7]$, consists of a tetrahedral $Mo_2Fe(\mu_3\text{-}S)$ cluster core and a 1,1′-(propane-1,3-diyldicarbonyl)dicyclopentadienyl ligand. The cyclopentadienyl rings, coordinated to two Mo atoms, make a dihedral angle of 66.57 (10)°.

Comment

Macrocyclic compounds containing organometallic cluster cores have potential applications in catalysis and the synthesis of novel structures (Roberts & Geoffroy, 1982). As part of our continuing study of the construction of such cluster macrocycles *via* self-assembly cyclization reactions (Song *et al.*, 2000, 2002), we have synthesized and characterized the title compound, (I), which contains a tetrahedral $Mo_2Fe(\mu_3\text{-}S)$ cluster core and a 1,1′-(propane-1,3-diyldicarbonyl)dicyclopentadienyl ligand.



(I)

Single-crystal analysis of (I) reveals that this compound contains a tetrahedral $Mo_2Fe(\mu_3\text{-}S)$ cluster core linked to a 1,1′-(propane-1,3-diyldicarbonyl)dicyclopentadienyl ligand, which is coordinated to two Mo atoms, one set of three CO ligands bonded to the Fe atom, and two sets of two CO ligands attached to two Mo atoms, respectively (Fig. 1). Each Cp ring and the carbonyl group attached to it are essentially coplanar. The two Cp rings form a dihedral angle of 66.57 (10)°. The π systems of the C═O double bonds belonging to the organic ligand are quite well conjugated with the Cp rings. Therefore, the C—C single-bond lengths C4—C6 [1.485 (4) Å] and C10—C11 [1.478 (4) Å] are shorter than a normal C—C single σ bond. Other dimensions in (I) are as expected.

Experimental

All reactions were carried out under an N_2 atmosphere. THF was distilled from Na/benzophenone. Other solvents were of reagent grade and used without further purification. Starting materials $[C_5H_4C(O)CH_2]_2CH_2Na_2$ (Bitterwolf, 1990) and [MoFeCo(μ_3-

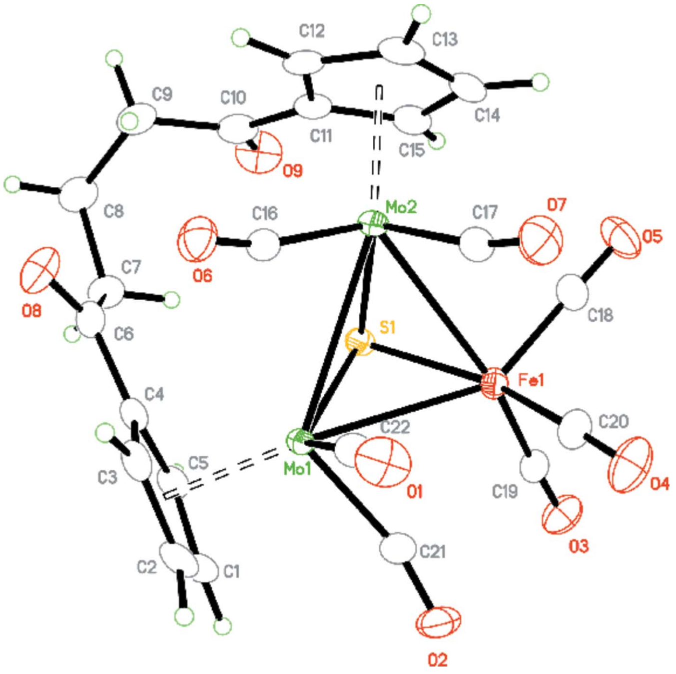


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

S)(CO)$_8$]$_2$[C$_5$H$_4$C(O)CH$_2$]$_2$CH$_2$ (Song *et al.*, 1995) were prepared according to literature procedures. [C$_5$H$_4$C(O)CH$_2$]$_2$CH$_2$Na$_2$ (0.5 mmol) was added to a solution of Mo(CO)$_6$(1.0 mmol) in THF (15 ml). The mixture was refluxed for 20 h, cooled to 298 K, and [MoCoFe(μ_3-S)(CO)$_8$]$_2$[C$_5$H$_4$C(O)CH$_2$]$_2$CH$_2$ (0.4 mmol) was added. The mixture was refluxed for another 10 h. The solvent was removed under reduced pressure and the resulting residue was purified by thin-layer chromatography (SiO$_2$, CH$_2$Cl$_2$/Et$_2$O, 20:1) to give (I) as a brown solid (8% yield). Suitable single crystals of (I) were obtained from *n*-hexane and CH$_2$Cl$_2$ (1:1) at 298 K.

Crystal data

[Mo$_2$Fe(C$_5$H$_6$O$_2$)S(CO)$_7$]
M_r = 702.12
Monoclinic, $P2_1/n$
a = 10.123 (2) Å
b = 18.835 (4) Å
c = 12.326 (2) Å
β = 105.255 (2)°
V = 2267.3 (8) Å^3

Z = 4
D_x = 2.057 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.87 mm^{-1}
T = 298 (2) K
Block, nut brown
0.46 × 0.25 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{\min}$ = 0.481, $T_{\max}$ = 0.730

9389 measured reflections
3990 independent reflections
3673 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.019
$\theta_{\max}$ = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
S = 1.03
3990 reflections
316 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0277P)^2 + 0.523P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\min}$ = −0.44 e Å^{-3}

All H atoms were included in calculated positions and refined as riding on their carrier atoms, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Constrained distances: C—H = 0.98 (aromatic CH) and 0.97 Å (methylene CH$_2$).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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