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# Bis($\mu$-methyl mercaptoacetato-$\kappa^2S$:$S$)bis[tricarbonyliron(**I**)]

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In the title compound, $[Fe_2(C_3H_5O_2S)_2(CO)_6]$, the asymmetric unit contains only one-half of the molecule and the central $[Fe_2S_2]$ fragment possesses a butterfly form on a twofold rotation axis. Each Fe atom is in a distorted tetragonal–pyramidal geometry and both C atoms bonded to sulfur are in equatorial positions.

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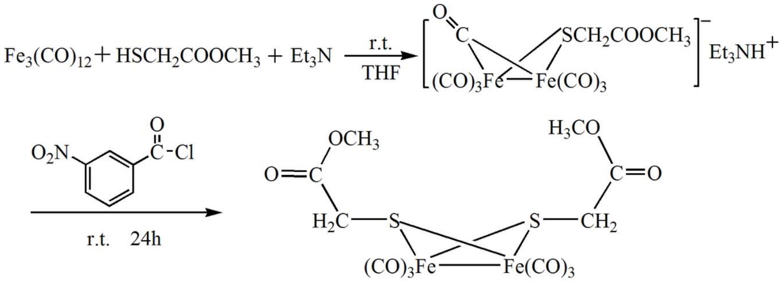
**Key indicators**

Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.027
$wR$ factor = 0.074
Data-to-parameter ratio = 14.3

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

## Comment

Recently, the structure of the redox forms of Fe-only hydrogenase have been widely studied (Liaw *et al.*, 2003). Here, we synthesized a reductive dinuclear iron-thiolate carbonyl complex, (I) (Fig. 1).



(I)

The asymmetric unit contains only one-half of the molecule. The central $[Fe_2S_2]$ fragment possesses a butterfly form and lies on a twofold rotation axis, and each Fe atom is in a distorted tetragonal pyramidal geometry (Table 1). In addition, each S-bonded C atom is in an equatorial position, showing the symmetric isomer is in the *e*,*e* (equatorial, equatorial) form (Dahl & Wei, 1963).

## Experimental

A solution of $Fe_3(CO)_{12}$ (1.5 g, 3.0 mmol), methyl mercaptoacetate (0.27 ml, 3.0 mmol) and triethylamine (0.5 ml, 3.6 mmol) in THF (40 ml) was maintained under an atmosphere of purified nitrogen and at room temperature for 45 min to give a brown-red solution. *m*-Nitrobenzoyl chloride (0.65 g, 3.5 mmol) was then added and stirred for 24 h. The solvent was removed under reduced pressure. The residue was extracted with petroleum ether/ethyl acetate (20/1 *v*/*v*) and then concentrated. The condensate was separated by column chromatography on silica gel with ethyl acetate:petroleum ether (1:20) as eluent. From the first band, red crystals for X-ray diffraction analyses were grown from hexane:$CH_2Cl_2$ (20:1) solution at 277 K (yield 0.341 g, 23.2%, m.p. 357–358 K). Spectroscopic analysis: IR (KBr, $cm^{-1}$): $\nu$ 2070, 2041, 1991, 1970; $^1H$ NMR ($CDCl_3$, p.p.m.): $\delta$ 3.71 (*s*, 6H), 3.11 (*s*, 4H). Analysis, calculated for $C_{12}H_{10}FeO_{10}S_2$: C 29.41, H 2.06%; found: C 29.19, H 1.96%.

### *Crystal data*

$[Fe_2(C_3H_5O_2S)_2(CO)_6]$
$M_r = 490.02$
Monoclinic, $C2/c$
$a = 24.339$ (4) Å
$b = 5.9939$ (9) Å
$c = 17.033$ (3) Å
$\beta = 132.189$ (1)°
$V = 1841.1$ (5) Å$^3$
$Z = 4$

$D_x = 1.768$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1715 reflections
$\theta = 2.3$–25.5°
$\mu = 1.85$ mm$^{-1}$
$T = 298$ (2) K
Block, red
0.58 × 0.45 × 0.34 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.385$, $T_{max} = 0.533$
4695 measured reflections

1715 independent reflections
1602 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 25.5°$
$h = -14 \rightarrow 29$
$k = -6 \rightarrow 7$
$l = -20 \rightarrow 17$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.074$
$S = 1.00$
1715 reflections
120 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 1.3935P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0246 (10)

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

| | | | |
|---|---|---|---|
| Fe1—C6 | 1.785 (2) | Fe1—S1$^i$ | 2.2515 (6) |
| Fe1—C5 | 1.787 (2) | Fe1—S1 | 2.2600 (7) |
| Fe1—C4 | 1.814 (3) | Fe1—Fe1$^i$ | 2.5359 (6) |
| | | | |
| C6—Fe1—C5 | 90.94 (11) | C6—Fe1—S1 | 94.25 (8) |
| C6—Fe1—C4 | 99.09 (12) | C5—Fe1—S1 | 159.51 (8) |
| C5—Fe1—C4 | 99.77 (12) | C4—Fe1—S1 | 98.96 (9) |
| C6—Fe1—S1$^i$ | 158.81 (8) | S1$^i$—Fe1—S1 | 75.13 (3) |
| C5—Fe1—S1$^i$ | 93.26 (8) | C1—S1—Fe1 | 114.71 (9) |
| C4—Fe1—S1$^i$ | 100.64 (9) | Fe1$^i$—S1—Fe1 | 68.402 (19) |

Symmetry code: (i) $-x+2, y, -z+\frac{1}{2}$.

H atoms were positioned geometrically [0.97 ($CH_2$) and 0.96 ($CH_3$) Å] and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2(1.5$ for methyl$)U_{eq}(C)$.

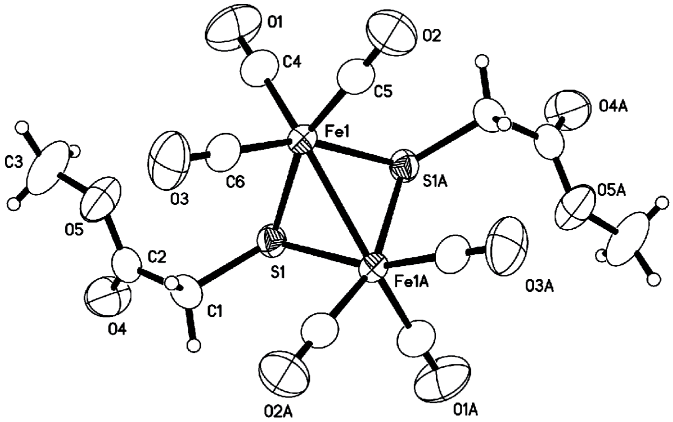


**Figure 1**
The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probalitity level. The suffix A corresponds to symmetry code (i) in Table 1.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXTL*.

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**m238** Liu *et al.* • $[Fe_2(C_3H_5O_2S)_2(CO)_6]$ *Acta Cryst.* (2006). E**62**, m237–m238