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# 1,2-Bis(ferrocenylcarbonylthioureido)ethane chloroform disolvate

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**Key indicators**

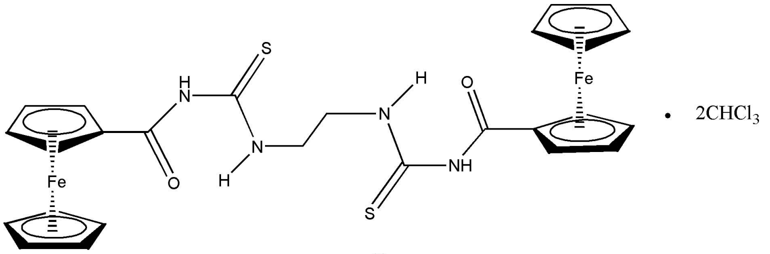
Single-crystal X-ray study
$T$ = 183 K
Mean $\sigma$(C–C) = 0.010 Å
$R$ factor = 0.068
$wR$ factor = 0.159
Data-to-parameter ratio = 14.6

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

The centrosymmetric main molecule of the title compound, $[Fe_2(C_5H_5)_2(C_{16}H_{16}N_4O_2S_2)]\cdot 2CHCl_3$, contains two linked ferrocene groups and possesses intramolecular N—H⋯O hydrogen bonds with an $S(6)$ graph-set motif. Two $CHCl_3$ solvent molecules interact with the main species by way of C—H⋯O interactions. Intermolecular N—H⋯S and C—H⋯S interactions link the molecules into one-dimensional chains with an $R_2^1(7)$ motif.

## Comment

Compounds containing ferrocene building blocks have been widely studied owing to their potential in catalysis, materials science, molecular devices, hydrometallurgy, *etc*. (Hayashi *et al.*, 1989; Andrieux *et al.*, 1980; Slone *et al.*, 1997). Variously substituted ferrocene derivatives bearing ligating groups with different donor atoms have been proposed and successfully tested for some of these applications (Benito *et al.*, 1993; Grossel *et al.*, 1991; Yuan *et al.*, 2001). In our studies, we designed and synthesized the title compound, (I), an acyclic acylthiourea compound containing two ferrocene groups. We report here its crystal structure (Fig. 1). The complete molecule is generated from the atoms of the asymmetric unit by an inversion centre located at the mid-point of the C13—C13$^i$ bond [symmetry code: (i) $-x$, $1-y$, $1-z$].



(I)

An intramolecular N—H⋯O hydrogen bond exists for the —C(O)—NH—C($S$)—N′H— group (Fig. 1), forming a six-membered ring. This ring is almost in the same plane as the

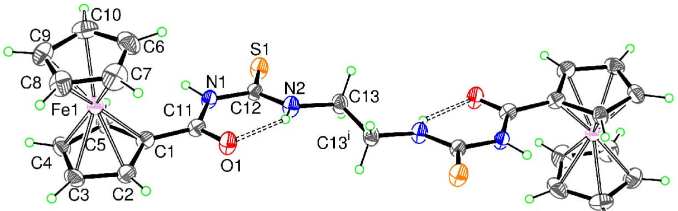


**Figure 1**
A view of the $C_{26}H_{26}N_4O_2S_2Fe_2$ species in (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). The N—H⋯O interactions are indicated by dashed lines. [Symmetry code: (i) $-x$, $1-y$, $1-z$.]

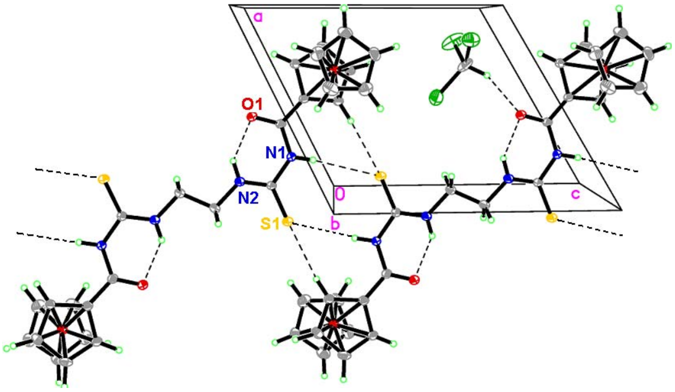


**Figure 2**
Packing of (I), viewed dowm [010], with intramolecular N—H···O and intermolecular C—H···O, N—H···S and C—H···S interactions (Table 1) indicated by dashed lines.

cyclopentadienyl (Cp) ring to which it is attached. (I) contains two co-crystallized $CHCl_3$ solvent molecules which interact with the main species by way of C—H···O bonds, with a short H···O distance of 2.11 Å (Table 1). Two non-classical intermolecular C—H···S and N—H···S interactions link the main molecules into one-dimensional chains with an $R_2^1(7)$ motif (Bernstein *et al.*, 1995) in the [001] direction. The N—H···S$^i$ and C—H···S$^i$ [symmetry code: (i) $-x, 1-y, -z$] interactions involve the same acceptor S atom (Fig. 2). The donor C—H bond is part of a ferrocene Cp ring.

## Experimental

Compound (I) was prepared by the following method (Wang *et al.*, 1993; Yuan *et al.*, 1997). KSCN (1.6 g, 15.9 mmol) was dissolved in acetone (30 ml) and added dropwise to an acetone solution (45 ml) of FcCOCl (3.6 g, 14.5 mmol) and refluxed for 30 min. The red acetone solution of FcCONCS was obtained on filtration from the precipitated KCl and used *in situ* for the following reactions without further separation and purification. Ethylenediamine dissolved in acetone (10 ml) was added dropwise to the acetone solution of ferrocenoyl isothiocyanate at room temperature. An orange–yellow solid rapidly appeared. The mixture was refluxed for 5 h and the orange–yellow powder obtained by filtration was washed with acetone. The product was purified using a silica-gel (160–220 mesh) chromatographic column (yield: 79%). The product was dissolved in chloroform and the solution was set aside at room temperature. As the solvent slowly evaporated, crystals of (I) were formed.

### Crystal data

$[Fe_2(C_5H_5)_2(C_{16}H_{16}N_4O_2S_2)]\cdot 2CHCl_3$
$M_r = 841.06$
Triclinic, $P\overline{1}$
$a = 9.705\ (4)$ Å
$b = 10.205\ (4)$ Å
$c = 10.866\ (4)$ Å
$\alpha = 96.481\ (5)^\circ$
$\beta = 111.579\ (5)^\circ$
$\gamma = 116.199\ (5)^\circ$
$V = 847.6\ (6)$ Å$^3$
$Z = 1$
$D_x = 1.648$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3513 reflections
$\theta = 2.4$–$25.4^\circ$
$\mu = 1.49$ mm$^{-1}$
$T = 183\ (2)$ K
Slab, yellow
0.30 × 0.30 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.664$, $T_{max} = 0.866$
2917 measured reflections
2921 independent reflections
2619 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.0^\circ$
$h = -11 \rightarrow 11$
$k = -8 \rightarrow 12$
$l = -12 \rightarrow 12$

### Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.159$
$S = 1.13$
2921 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 1.4465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.63$ e Å$^{-3}$
$\Delta\rho_{min} = -0.58$ e Å$^{-3}$

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H16···O1 | 0.88 | 1.92 | 2.616 (5) | 135 |
| N1—H15···S1$^i$ | 0.88 | 2.83 | 3.663 (4) | 158 |
| C5—H5···S1$^i$ | 0.95 | 2.82 | 3.660 (5) | 147 |
| C14—H17···O1$^{ii}$ | 1.00 | 2.11 | 3.044 (7) | 155 |

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $x, y, 1+z$.

All H atoms were placed in calculated positions (C—H = 0.93–0.98 Å and N—H = 0.86 Å) and refined as riding with the constraint $U_{iso}(H) = 1.2U_{eq}$(carrier) applied.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2000); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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