

Bis(ferrocenyl methyl ketone thiosemi-
carbazone- κ^2N,S)zinc tetrahydrofuran
hemisolvateXun Li,^{a*} Xue-Gui Cui,^a
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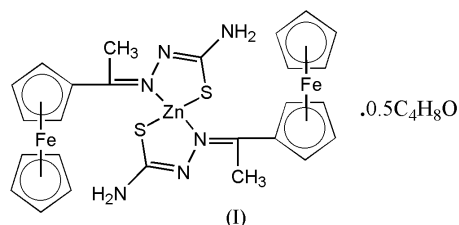
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.015\text{ \AA}$
 R factor = 0.057
 wR factor = 0.194
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title complex, $[\text{ZnFe}_2(\text{C}_5\text{H}_5)_2(\text{C}_8\text{H}_9\text{N}_3\text{S})_2] \cdot 0.5\text{C}_4\text{H}_8\text{O}$, is a Schiff base chelate. It consists of one zinc cation and two ligand anions, the cation being tetrahedrally coordinated by one N atom and one S atom from each ligands, thus forming two orthogonal five-membered chelate rings related to each other by the twofold rotation axis passing through Zn.

Comment

Complexes of transition metals with some Schiff bases containing S and N atoms have been used widely (Basavantappa & Patil, 1986) as anticancer drugs, preservatives and antibacterial agents. In particular, the biochemical behaviour and function of zinc have a very close bearing on health (Wang & Xu, 1992). The ferrocene group can also serve as an ultra-violet protecting agent (Basavantappa & Patil, 1986). In the title complex, (I), the molecular structure contains two symmetric five-membered chelate, related to each other by the twofold rotation axis passing through Zn. Zn is tetrahedrally coordinated by one N atom and one S atom from each ligand. As shown by thermogravimetric and differential thermal analysis, the thermostability of the complex is much greater than that of the ligand itself.



Experimental

Acetylferrocene was prepared by a modification of the method of Van Ryswyk & Van Hecke (1991) (m.p. 354 K; 91% yield). For the preparation of monoacetylferrocene thiosemicarbazone (ligand), (II), to a stirred solution of acetylferrocene (2.3 g, 10 mmol) in anhydrous ethanol (50 ml), thiosemicarbazide (0.91 g, 10 mmol) in distilled water (40 ml) was added dropwise; to this mixture was added acetic acid (2 ml), and the resulting solution was refluxed at 323 K for 2 h, cooled to room temperature and filtered. The precipitate was washed with anhydrous ethanol and dried *in vacuo* (m.p. 425–429 K). For the preparation of (I), compound (II) (0.304 g, 1 mmol) in 50% ethanol solution (25 ml) was refluxed for about 30 min. A solution of $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.11 g, 0.5 mmol) in 50% ethanol solution (25 ml) was added to the refluxing solution over a period of about 30 min, and the mixture was refluxed for another 2 h before being cooled. The precipitate was filtered off and dried *in vacuo*, yielding red crystals. Single crystals suitable for X-ray analysis were obtained by evaporation of a tetrahydrofuran solution slowly over a period of 5 d.

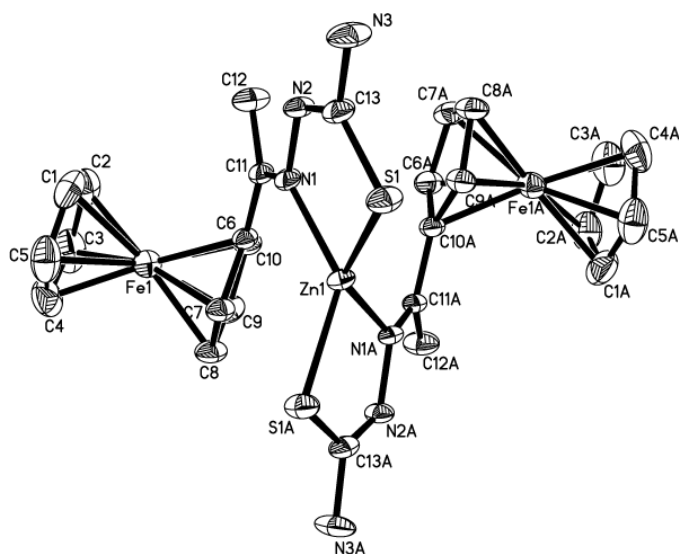


Figure 1
The molecular structure of (I), with displacement ellipsoids at the 30% probability level. Hydrogen atoms have been omitted, as has the solvent molecule. [Symmetry code: (A) $x - y + \frac{1}{3}, \frac{2}{3} - y, \frac{1}{6} - z$.]

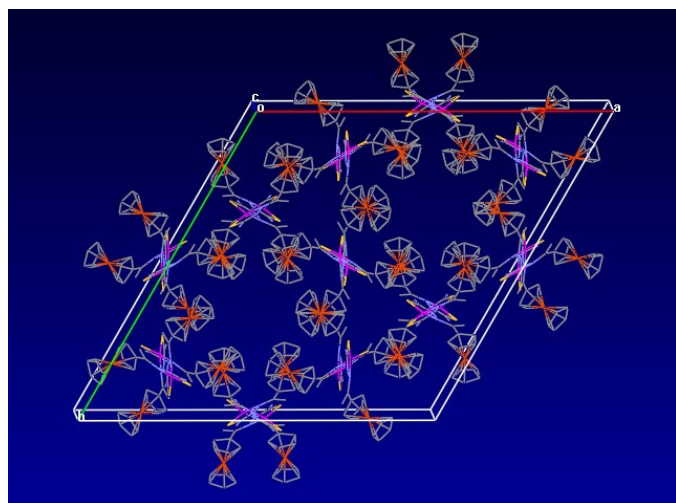


Figure 2
The crystal structure of (I), viewed along the c axis. The THF molecule has been omitted for clarity.

Crystal data

$[\text{ZnFe}_2(\text{C}_5\text{H}_5)_2(\text{C}_8\text{H}_9\text{N}_3\text{S})_2] \cdot 0.5\text{C}_4\text{H}_8\text{O}$
 $M_r = 701.79$
 Trigonal, $R\bar{3}c$
 $a = 28.912(17) \text{ \AA}$
 $c = 19.784(17) \text{ \AA}$
 $V = 14322(17) \text{ \AA}^3$
 $Z = 18$

$D_x = 1.465 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 1.0\text{--}25.0^\circ$
 $\mu = 1.81 \text{ mm}^{-1}$
 $T = 293(2) \text{ orange}$
 $0.30 \times 0.25 \times 0.20 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.613, T_{\max} = 0.714$
 19 336 measured reflections

2823 independent reflections
 1496 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.133$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -29 \rightarrow 34$
 $k = -34 \rightarrow 33$
 $l = -17 \rightarrow 23$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.194$
 $S = 0.96$
 2823 reflections
 192 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$

H atoms were positioned geometrically, with $\text{C}-\text{H} = 0.93\text{--}0.98 \text{ \AA}$ and $\text{N}-\text{H} = 0.90 \text{ \AA}$, and treated using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$. The value of R_{int} is high due to the poor quality of the crystal. The maximum residual density peak is 0.86 \AA^{-3} from atom C14.

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

References

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