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Key indicators

Single-crystal X-ray study
 T = 161 K
 Mean $\sigma(C-C)$ = 0.002 Å
 R factor = 0.033
 wR factor = 0.043
 Data-to-parameter ratio = 12.9

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

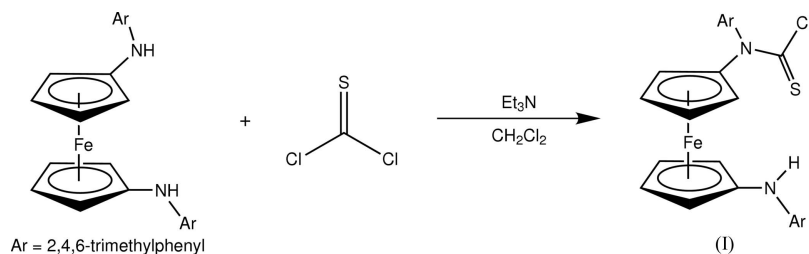
***N*-[1'-(2,4,6-Trimethylanilino)-1-ferrocenyl]-*N*-(2,4,6-trimethylphenyl)thiocarbamoyl chloride**

The title compound, $[Fe(C_{15}H_{15}ClNS)(C_{14}H_{16}N)]$, crystallizes with the thiocarbamoyl chloride unit nearly coplanar with the cyclopentadienyl ring to which it is attached. There is a slight deviation from planarity which, along with the orientation of the amino substituent on the other cyclopentadienyl ring, is suggestive of an intramolecular hydrogen bond. The long donor–acceptor $N \cdots S$ length of 3.702 (2) Å indicates that any interaction is weak.

Received 11 July 2006
 Accepted 26 July 2006

Comment

There has been widespread interest in the use of ferrocene-containing ligands in coordination chemistry (Togni & Hayashi, 1995). Previous work in our laboratory has focused on diaminoferrocene derivatives as either bidentate (Shafir *et al.*, 2003) or tetradentate (Shafir *et al.*, 2002) dianionic ligands for early transition metals. We have recently become interested in using diaminoferrocene derivatives in *N*-heterocyclic carbenes. One possible strategy for the synthesis of such materials would be to react an *N,N'*-functionalized diaminoferrocene with thiophosgene to form the cyclic urea, followed by reduction with potassium to give the desired carbene. Our initial attempts at such a cyclization, the reaction of the mesityl-functionalized diaminoferrocene, resulted in a clean reaction of only one of the two amine groups with thiophosgene. This bright-red material, (I), was found to be quite soluble in pentane at room temperature, but to crystallize as large plates upon cooling to 233 K.



The diaminoferrocene core of the structure of (I) shows eclipsed cyclopentadienyl rings with the amino substituents offset by one position on the rings. Both N1 and C20 have planar environments, with sums of angles of 360° within error. The C1–N1–C20–S1 torsion angle is 3.4 (3)°, and so the entire π system of the thiocarbamoyl chloride fragment is in conjugation with the cyclopentadienyl ring to which it is attached. A slight deviation from planarity points the S atom towards N2, and the H atom attached to this N atom points towards S1. This is suggestive of a hydrogen bond, but the $N \cdots S$ distance of 3.702 (2) Å indicates that any interaction between S1 and H31 is weak.

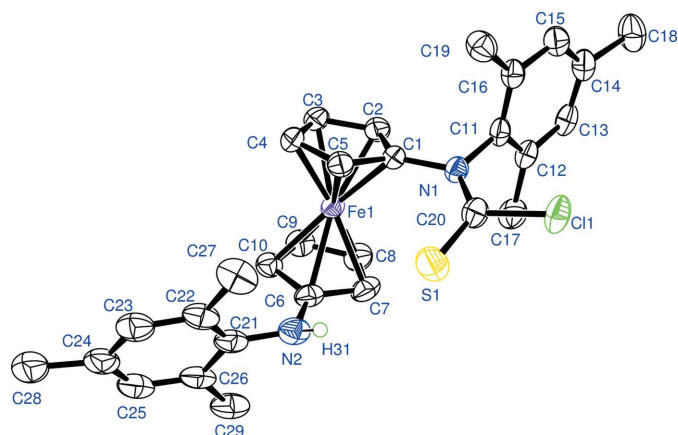


Figure 1
View of the molecular structure of (I), drawn with 50% probability displacement ellipsoids. H atoms, except H31, have been omitted.

Experimental

Under an inert atmosphere, a 5% by volume solution of CSCl_2 in CH_2Cl_2 (0.85 ml, 0.56 mmol) was added to 1,1'-bis(mesitylamino)-ferrocene (250 mg, 0.55 mmol) (Shafir *et al.*, 2003) and triethylamine (0.20 ml, 1.4 mmol) dissolved in anhydrous CH_2Cl_2 (10 ml). The solution was stirred for 2 h and anhydrous pentane (10 ml) was added. The solution was filtered and the solvent removed under vacuum. The resulting red solid (yield 273 mg, 93%) was redissolved in pentane and the solution was concentrated and cooled to 233 K for 48 h, yielding clear red crystals suitable for X-ray studies.

Crystal data

$[\text{Fe}(\text{C}_{15}\text{H}_{15}\text{CINS})(\text{C}_{14}\text{H}_{16}\text{N})]$	$Z = 4$
$M_r = 530.94$	$D_x = 1.373 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 9.6245 (5) \text{ \AA}$	$\mu = 0.79 \text{ mm}^{-1}$
$b = 20.623 (1) \text{ \AA}$	$T = 161.2 \text{ K}$
$c = 13.6569 (7) \text{ \AA}$	Plate, red
$\beta = 108.699 (1)^\circ$	$0.20 \times 0.12 \times 0.05 \text{ mm}$
$V = 2567.7 (2) \text{ \AA}^3$	

Data collection

Bruker APEX diffractometer	14858 measured reflections
ω scans	5213 independent reflections
Absorption correction: ψ scan	3992 reflections with $F^2 > 3\sigma(F^2)$
(SORTAV; Blessing, 1995)	$R_{\text{int}} = 0.019$
$T_{\text{min}} = 0.825$, $T_{\text{max}} = 0.961$	$\theta_{\text{max}} = 26.4^\circ$

Refinement

Refinement on F	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o) + 0.00022 F_o ^2]$
$wR(F^2) = 0.043$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.87$	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
3992 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
310 parameters	

Table 1

Selected geometric parameters (\AA , $^\circ$).

C11—C20	1.762 (2)	N1—C20	1.343 (3)
S1—C20	1.638 (2)		
S1...N2	3.702 (2)		
S1—C20—N1—C1	3.4 (3)		

Atom H31, attached to N2, was found in a difference Fourier map and refined isotropically. All other H atoms were placed in calculated positions ($\text{C—H} = 0.95 \text{ \AA}$), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1998); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN*.

The authors thank Drs Fredrick J. Hollander and Allen G. Oliver for assistance and advice. They also thank the DOE for funding.

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