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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C}) = 0.011 \text{ \AA}$
Disorder in main residue
R factor = 0.035
wR factor = 0.094
Data-to-parameter ratio = 13.0

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

1-Chloromercurio-2-[(2-chlorophenyl)(3-chlorophenyl)imino]methyl]ferrocene

The title compound, $[\text{FeHg}(\text{C}_5\text{H}_5)(\text{C}_{18}\text{H}_{11}\text{Cl}_2\text{N})\text{Cl}]$, was prepared by the reaction of [(2-chlorophenyl)(3-chlorophenylimino)methyl]ferrocene, mercury acetate and lithium chloride. The $\text{N} \cdots \text{Hg}$ separation of 2.884 (5) \AA suggests a weak coordination interaction.

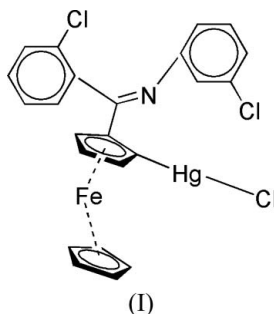
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Comment

Cyclomercurated ferrocenyl derivatives, especially those bearing *N*-donor ligands, are widely used in organic syntheses such as halogenation (Larock & Chow, 1986), transmetalation (Wu *et al.*, 1995), as well as C–C bond formation (Larock & Leach, 1982). We have recently prepared the title compound, (I), which may be an excellent cyclometallation candidate.



The molecular structure of (I) is shown in Fig. 1. The $\text{C1}=\text{N1}$ bond length (Table 1) suggests it to be a typical double bond. Although the bond distance shows that $\text{C1}-\text{C14}$ is a typical single bond, atoms C1, C14, C15, N1 and Hg1 are coplanar, the maximum atomic deviation from the mean plane being 0.089 (4) \AA (for Hg1). The separation of 2.884 (5) \AA between atoms N1 and Hg1 is much shorter than the sum of van der Waals radii. These findings may suggest a weak coordination interaction between atoms N1 and Hg1.

Experimental

A methanol solution (30 ml) of mercury(II) acetate (2 mmol) was added dropwise to a methanol/dichloromethane solution (4:1 *v/v*, 25 ml) of [(2-chlorophenyl)(3-chlorophenylimino)methyl]ferrocene (2 mmol) at room temperature. Thin-layer chromatography was used to monitor the reaction until complete. A methanol solution (20 ml) of lithium chloride (3 mmol) was then added and the mixture was stirred for a further 30 min. The solvent was removed *in vacuo* and the residue was extracted with dichloromethane three times (total 90 ml). The organic layer was dried over anhydrous sodium sulfate and concentrated to dryness under reduced pressure. The crude product was purified by chromatography (silica gel, ethyl acetate/hexane = 1:1 *v/v*); the second band of eluant was collected. The solvent was removed under reduced pressure to obtain crystals of (I).

Analysis calculated for $C_{23}H_{16}Cl_3FeHgN$: C 41.28, H 2.41, N 2.09%; found: C 41.15, H 2.45, N 2.17%.

Crystal data

$[FeHg(C_5H_5)(C_{18}H_{11}Cl_2N)Cl]$
 $M_r = 669.16$
 Triclinic, $P\bar{1}$
 $a = 8.5489$ (16) Å
 $b = 9.1426$ (18) Å
 $c = 15.213$ (3) Å
 $\alpha = 90.700$ (3)°
 $\beta = 102.656$ (3)°
 $\gamma = 107.516$ (3)°
 $V = 1102.5$ (4) Å³

$Z = 2$
 $D_x = 2.016$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3094 reflections
 $\theta = 2.6$ – 26.3 °
 $\mu = 7.99$ mm⁻¹
 $T = 294$ (2) K
 Prism, purple
 $0.24 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.175$, $T_{max} = 0.238$
 5596 measured reflections

3842 independent reflections
 3057 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.025$
 $\theta_{max} = 25.0$ °
 $h = -9 \rightarrow 10$
 $k = -10 \rightarrow 10$
 $l = -17 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.094$
 $S = 1.05$
 3842 reflections
 296 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.0748P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 1.16$ e Å⁻³
 $\Delta\rho_{min} = -1.62$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-------------|-------------|-------------|-----------|
| Hg1—C15 | 2.012 (8) | N1—C1 | 1.262 (8) |
| Hg1—C11 | 2.294 (3) | N1—C8 | 1.413 (8) |
| C15—Hg1—C11 | 177.75 (17) | C16—C15—Hg1 | 133.1 (6) |
| C1—N1—C8 | 121.7 (5) | C14—C15—Hg1 | 121.1 (5) |
| N1—C1—C14 | 118.7 (6) | Hg1—C15—Fe1 | 122.0 (3) |

The C2-containing chlorophenyl group is disordered over two positions; site-occupancy factors were refined and converged to 0.886 (6) and 0.114 (6), respectively. The benzene rings of the disordered chlorophenyl were refined as regular hexagons. All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. In the final difference Fourier map, the highest peak was 0.93 Å from atom Hg1 and the deepest hole 1.08 Å from atom Hg1.

Data collection: SMART (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve

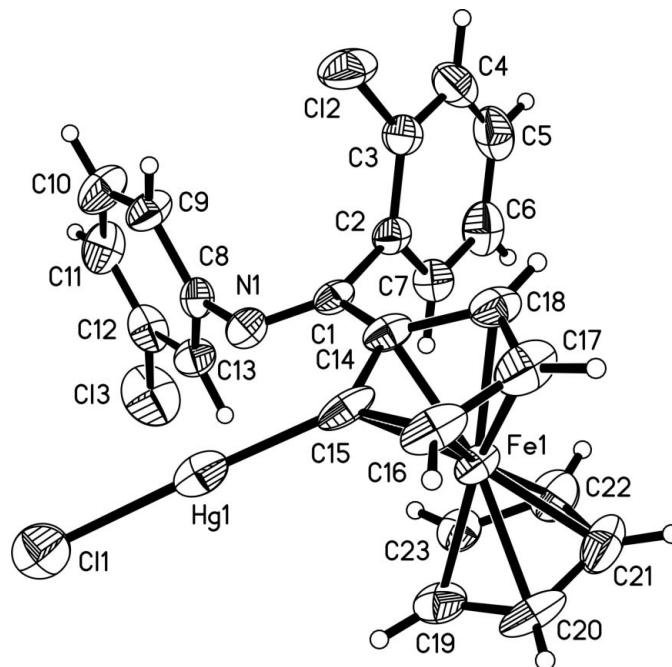


Figure 1

The molecular structure of (I), shown with 35% probability displacement ellipsoids (arbitrary spheres for H atoms). The minor component of the disordered chlorophenyl has been omitted for clarity.

structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

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