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Key indicators

Single-crystal X-ray study

T = 173 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.047

wR factor = 0.119

Data-to-parameter ratio = 19.9

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

The first aluminium-bridged [1.1]ferrocenophane

The structure of the title compound, bis(μ -ferrocene-1,1'-diyl)bis[chloro(*N,N,N',N'*-tetramethylethylenediamine)-aluminium], $[\text{Al}_2\text{Fe}_2\text{Cl}_2(\text{C}_5\text{H}_4)_4(\text{C}_6\text{H}_{16}\text{N}_2)_2]$, possesses a center of inversion and an *anti* conformation. Bound to each Al atom in the bridging position are one Cl atom and one molecule of *N,N,N',N'*-tetramethylethylenediamine (TMEDA). The angle between the least-squares planes of the two cyclopentadienyl (Cp) ligands is $3.6(3)^\circ$. The five C atoms in the Cp rings are staggered on average at an angle of $10.3(5)^\circ$.

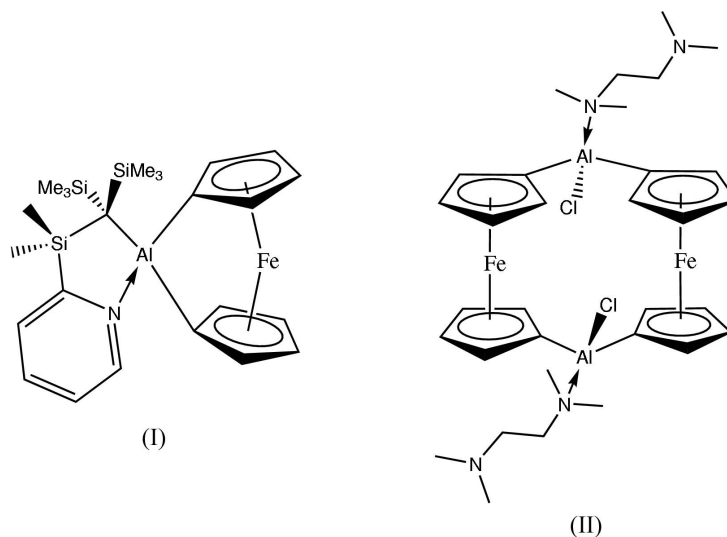
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Comment

Recently, we synthesized the first [1]aluminaferrocenophane, (I), by a metathesis reaction of 1,1'-dilithioferrocene-0.67-TMEDA and (Pytsi)AlCl₂ [Pytsi = C(SiMe₃)₂SiMe₂(2-C₅H₄N)] and TMEDA = *N,N,N',N'*-tetramethylethylenediamine] using toluene as a solvent. Compound (I) was isolated as a crystalline material with one half-molecule of ferrocene (FeCp₂) in the asymmetric unit (Schachner *et al.*, 2005). We assume that FeCp₂ results from the protolysis of 1,1'-dilithioferrocene, with toluene being the source of protons. Consequently, we substituted toluene by the less acidic solvent hexane with the intention of optimizing the synthesis of (I). However, the title compound, (II), was the only isolable product from this reaction (isolated yield 9%).



Compound (II) is a [1.1]dialuminaferrocenophane in which each Al atom still carries one Cl atom and is coordinated by one molecule of TMEDA. So far, we could neither clarify how compound (II) is formed nor optimize its synthesis by a rational approach starting from 1,1'-dilithioferrocene-0.67-

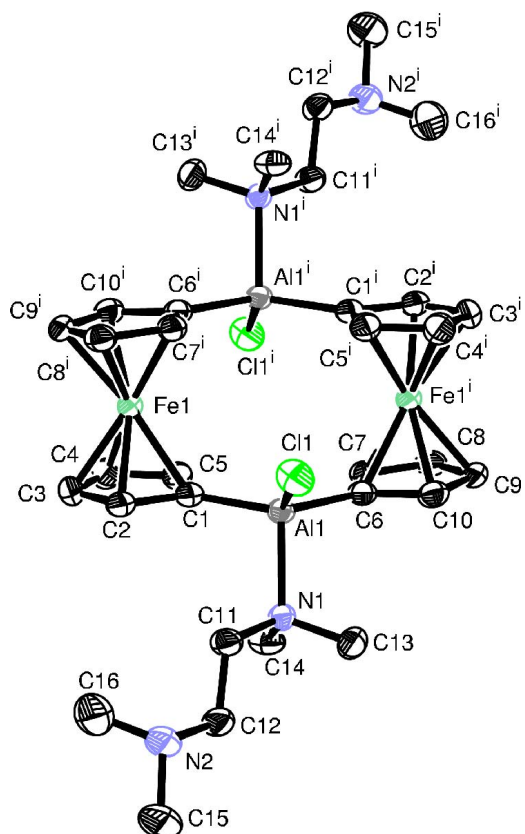


Figure 1
An ORTEP-3 view (Farrugia, 1997) of molecule (II), with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x, -y, -z$.]

TMEDA and AlCl_3 . To the best of our knowledge, there are no reports in the literature of other [1.1]-dialuminaferrocenophanes; only similar boron (Scheibitz *et al.*, 2003) and gallium [1.1]ferrocenophanes (Uhl *et al.*, 2001; Jutzi *et al.*, 2001; Althoff *et al.*, 2002, 2003) are described.

Experimental

A slurry of 1,1'-dilithioferrocene-0.67-TMEDA (582 mg, 2.11 mmol) in hexane (20 ml) was added dropwise to a cooled (263 K) slurry of (Pytsi) AlCl_2 (826 mg, 2.10 mmol) in hexane (60 ml) and stirred for 72 h at room temperature. The resulting mixture was filtered to yield a light-red solution. After removal of some hexane in vacuum, orange crystals of (II) formed at 240 K (68 mg; 9%). ^1H NMR (500 MHz, C_6D_6 , 298 K): δ 0.93–2.29 (*m*, 16H, TMEDA), 4.39 (*m*, 2H, Cp), 4.51 (*m*, 2H, Cp), 4.68 (*m*, 2H, Cp), 5.16 (*m*, 2H, Cp).

Crystal data

$[\text{Al}_2\text{Fe}_2\text{Cl}_2(\text{C}_5\text{H}_4)_4(\text{C}_6\text{H}_{16}\text{N}_2)]$
 $M_r = 725.30$
 Triclinic, $P\bar{1}$
 $a = 7.8491$ (2) Å
 $b = 10.2612$ (3) Å
 $c = 11.2111$ (5) Å
 $\alpha = 78.5212$ (12)°
 $\beta = 89.0564$ (12)°
 $\gamma = 73.722$ (2)°
 $V = 848.68$ (5) Å³

$Z = 1$
 $D_x = 1.419$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3725 reflections
 $\theta = 1.0$ – 27.5°
 $\mu = 1.09$ mm⁻¹
 $T = 173$ (2) K
 Chip, orange
 $0.20 \times 0.20 \times 0.12$ mm

Data collection

Nonius KappaCCD diffractometer
 φ scans, and ω scans with κ offsets
 Absorption correction: none
 7256 measured reflections
 3869 independent reflections
 2697 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -10 \rightarrow 10$
 $k = -13 \rightarrow 13$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.120$
 $S = 1.04$
 3869 reflections
 194 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.4054P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.76 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.51 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Al1–C1	1.954 (3)	Fe1–C2	2.034 (3)
Al1–C6	1.958 (3)	Fe1–C3	2.038 (3)
Al1–N1	2.040 (3)	Fe1–C4	2.044 (3)
Al1–Cl1	2.1594 (12)	Fe1–C5	2.053 (3)
Fe1–C1	2.081 (3)		
C1–Al1–C6	119.59 (14)	C6–Al1–Cl1	112.74 (10)
C1–Al1–N1	101.62 (12)	N1–Al1–Cl1	101.45 (8)
C6–Al1–N1	103.03 (11)		
C1–Al1–Cl1	114.94 (10)		
C6–Al1–C1–C2	179.6 (3)	Cl1–Al1–C1–C2	−41.4 (3)
N1–Al1–C1–C2	67.2 (3)		

H atoms were placed in calculated positions, with C–H distances ranging from 0.95 to 0.99 Å, and included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H})$ values constrained to be 1.2 times U_{eq} of the carrier atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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