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Synthesis of a trinuclear cation $[H_3Ru_3(Fc\text{-}arene)(C_6Me_6)_2(O)]^+$ containing a ferrocenyl group tethered to an arene ligand

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Abstract

Ferrocene carboxylic acid 2-cyclohexa-1,4-dienyl-ethyl ester (**1**) is prepared from ferrocene carboxylic acid and 2-cyclohexa-1,4-dienyl-ethanol. This diene reacts with $RuCl_3 \cdot nH_2O$ in refluxing ethanol to afford quantitatively $[Ru(Fc\text{-}arene)Cl_2]_2$ (**2**) (Fc-arene = ferrocene carboxylic acid phenethyl ester). The dinuclear complex **2** reacts with triphenylphosphine to give the mononuclear complex $[Ru(Fc\text{-}arene)(PPh_3)Cl_2]$ (**3**). The trinuclear arene-ruthenium cluster cation $[H_3Ru_3(Fc\text{-}arene)(C_6Me_6)_2(O)]^+$ (**4**) is synthesised from the dinuclear precursor $[H_3Ru_2(C_6Me_6)_2]^+$ and the mononuclear complex $[Ru(Fc\text{-}arene)(H_2O)_3]^{2+}$, accessible from **2** in aqueous solution. The water-soluble trinuclear cluster cation **4** catalyses the hydrogenation of benzene to give cyclohexane under biphasic conditions.

Keywords: Arene ligands; Biphasic catalysis; Cluster compounds; Ferrocene; Hydrogenation; Phosphine ligands; Ruthenium complexes

1. Introduction

Recently we have shown that the water-soluble cluster cation $[H_3Ru_3(C_6H_6)(C_6Me_6)_2(O)]^+$ to be catalytically active in the hydrogenation of benzene to cyclohexane under biphasic conditions [1]. Experimental as well as modelling studies suggest that the substrate molecule is incorporated in the hydrophobic pocket spanned by the three arene ligands, suggesting the catalytic reaction to occur within this host-guest complex [2,3]. On the other hand, it is well known that rhodium and iridium complexes with chiral Josiphos ligands (phosphine ligands containing a ferrocenyl group) are highly active and selective catalysts for the enantioselective hydrogenation of enamides, itaconic acid derivatives and acetoacetates [4]. Therefore, we decided to incorporate a ferrocenyl group tethered to the benzene ligand in $[H_3Ru_3(C_6H_6)(C_6Me_6)_2(O)]^+$, in order to study the effect of a ferrocenyl moiety on the size of the hydrophobic pocket and the catalytic activity of the water-soluble trinuclear ruthenium cluster.

2. Experimental

2.1. General

All manipulations were carried out by routine under nitrogen atmosphere. De-ionised water and organic solvents were degassed and saturated with nitrogen prior to use. NMR spectra were recorded on a Varian 200 MHz spectrometer. IR spectra were recorded on a Perkin–Elmer 1720X FT-IR spectrometer (4000–400 cm^{-1}). Microanalyses were performed by the Laboratory of Pharmaceutical Chemistry, University of Geneva (Switzerland). Electro-spray mass spectra were obtained in positive-ion mode with an LCQ Finnigan mass spectrometer. Organic products were analysed by gas chromatography (GC) on a DANI 86.10 HT gas chromatograph using a CHROMPACK Carbowax WCOT fused silica column. The starting dinuclear dichloro complexes $[Ru(C_6Me_6)Cl_2]_2$ [5] and $[H_3Ru_2(C_6Me_6)_2]^+$ [6] were prepared according to published methods.

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2.2. *Syntheses*

2.2.1. *Ferrocene carboxylic acid 2-cyclohexa-1,4-dienyl-ethyl ester (**1**)*

A solution of ferrocene carboxylic acid (2.41 g, 10.48 mmol), *N,N*-dicyclohexylcarbodiimide (3.30 g, 15.99 mmol), 4-(dimethylamino)pyridine (1 g, 8.18 mmol), 4-pyrrolidinopyridine (1.20 g, 8.10 mmol), and 2,5-dihydrophenethylalcohol (1 g, 8.06 mmol) in CH_2Cl_2 (80 ml) was stirred under nitrogen at room temperature during 3 days. The resulting solution was filtered through Celite to remove *N,N*-dicyclohexylurea, and the filtrate concentrated under reduce pressure. A chromatogram of the residue was recorded on a silica gel column, eluting with hexane/acetone (10:1). The pure product was isolated from the first fraction, giving **1** as red oil. Yield: 2.54 g (94%).

Spectroscopic data: IR (solution in $CHCl_3$, cm^{-1}): v = 1705 (C=O ester). ^1H NMR (200 MHz, $CDCl_3$): δ = 5.76 (m, 2H, ethylenic *H*), 5.61 (m, 1H, ethylenic *H*), 4.82 (m, 2H, Cp*H*), 4.41 (m, 2H, Cp*H*), 4.34 (t, 2H, ¬OCH_2CH_2¬, ^3J = 6.84 Hz), 4.21 (s, 5H, Cp*H*), 2.75 (m, 4H, ¬$CHCH_2CH$¬), 2.42 (t, 2H, ¬OCH_2CH_2¬, ^3J = 6.84 Hz). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 172.02, 131.69, 124.43, 124.33, 121.08, 71.54, 70.40, 70.21, 70.04, 62.77, 36.93, 29.40, 27.06. MS (EI mode, $CHCl_3$): m/z = 336 $[M]^+$. *Anal.* Calc. for $C_{19}H_{20}FeO_2$: C, 67.88; H, 6.00. Found: C, 67.85; H, 5.98%.

2.2.2. *[Ru(Fc-arene)Cl_2]$_2$ (**2**)*

To a solution of ruthenium trichloride hydrate (350 mg, 1.34 mmol) in ethanol (40 ml) was added **1** (1.80 g, 5.36 mmol), and the mixture was refluxed overnight. The yellow-brown precipitate was filtered, washed with ether and acetone, and dried under vacuum to give $[Ru\{C_6H_5(CH_2)_2O_2CC_5H_4FeC_5H_5\}Cl_2]_2$. Yield: 590 mg (87%).

Spectroscopic data: IR (KBr, cm^{-1}): v = 1707 (C=O ester). ^1H NMR (200 MHz, dmso-d_6): δ = 6.11¬5.79 (m, 5H, (Ru¬C_6H_5), 4.75 (t, 2H, Cp*H*, ^3J = 1.83 Hz), 4.47 (t, 2H, Cp*H*, ^3J = 1.83 Hz), 4.43 (t, 2H, 2H, ¬OCH_2CH_2¬, ^3J = 5.86 Hz), 4.12 (s, 5H, Cp*H*), 2.86 (t, 2H, ¬OCH_2CH_2¬, ^3J = 5.86 Hz). ^{13}C NMR (50 MHz, dmso-d_6): δ = 171.18, 104.00, 89.26, 87.03, 84.88, 72.21, 71.04, 70.48, 70.28, 63.53, 32.91. MS (EI mode, dmso): m/z = 976.5 [M¬Cl]$^+$. *Anal.* Calc. for $C_{38}H_{36}Cl_4Fe_2O_4Ru_2$: C, 45.08; H, 3.58. Found: C, 44.89; H, 3.56%.

2.2.3. *[Ru(Fc-arene)(PPh_3)Cl_2] (**3**)*

To a suspension of **2** (200 mg, 0.20 mmol) in 25 ml of CH_2Cl_2 was added triphenylphosphine (114 mg, 0.43 mmol). The mixture was stirred at room temperature during 24 h and filtered through Celite to eliminate insoluble degradation materials. The product was purified on a silica gel column, eluting CH_2Cl_2/acetone (10:2) to give **3** as an orange¬brown powder. Orange crystals suitable for X-ray analysis were obtained by crystallisation from $CHCl_3$/*n*-hexane. Yield: 153 mg (50%).

Spectroscopic data: IR (KBr, cm^{-1}): v = 1698 (C=O ester). ^1H NMR (200 MHz, $CDCl_3$): δ = 7.78 (m, 6H, P¬C_6H_5), 7.43 (m, 9H, P¬C_6H_5), 5.41¬5.27 (m, 5H, Ru¬C_6H_5), 4.78 (m, 2H, Cp*H*), 4.55 (m, 2H, Cp*H*), 4.43 (m, 2H, ¬OCH_2CH_2¬), 4.15 (s, 5H,Cp*H*), 3.07 (t, 2H, ¬OCH_2CH_2¬). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 171.71, 134.52, 134.33, 130.85, 130.81, 128.58, 128.38, 108.02, 89.93, 88.53, 82.57, 71.77, 70.81, 70.35, 70.04, 62.86, 32.88. ^{31}P NMR (81 MHz, $CDCl_3$): δ = 28.63 (s). MS (EI mode, $CHCl_3$): m/z = 733 [M¬Cl]$^+$. *Anal.* Calc. for $C_{37}H_{33}Cl_2Fe_1O_2P_1Ru_1$: C, 57.83; H, 4.33. Found: C, 58.07; H, 4.38%.

2.2.4. *$[H_3Ru_3(Fc\text{-}arene)(C_6Me_6)_2(O)]^+$ (**4**)*

To a solution of $[H_3Ru_2(C_6Me_6)_2][BF_4]$ (120 mg, 0.19 mmol) in acetone (40 ml) and H_2O (5 ml) was added **2** (150 mg, 0.15 mmol). The mixture was heated to 60 °C for 45 h in a closed pressure Schlenk tube. The resulting red solution was filtered on Celite and evaporated to dryness; the residue was dissolved in CH_2Cl_2 (10 ml) and purified on silica-gel plates (eluent: CH_2Cl_2/Acetone 2:1) to give pure [**4**][BF_4] as red crystalline powder. Red crystals suitable for X-ray analysis were obtained by crystallisation from acetone/*n*-hexane. Yield: 80 mg (39%).

Spectroscopic data: IR (KBr, cm^{-1}): v = 1709 (C=O ester). ^1H NMR (200 MHz, acetone-d_6): δ = 6.01 (m, 2H, Ru¬C_6H_5), 5.62 (m, 3H, Ru¬C_6H_5), 4.81 (t, 2H, Cp¬*H*, ^3J = 1.96 Hz), 4.66 (t, 2H, ¬OCH_2CH_2¬, ^3J = 6.83 Hz), 4.50 (t, 2H, Cp¬*H*, ^3J = 1.96 Hz), 4.20 (s, 5H, Cp¬*H*), 2.93 (t, 2H, ¬OCH_2CH_2¬, ^3J = 6.83 Hz), 2.35 (s, 36H, Ru¬$C_6(CH_3)_6$), −19.16 (d, 2H, Ru hydride, ^2J = 3.42 Hz), −19.85 (t, 1H, Ru hydride, ^2J = 3.42 Hz). ^{13}C NMR (50 MHz, acetone-d_6): δ = 172.84, 102.61, 94.92, 85.55, 80.26, 78.50, 71.64, 70.17, 70.02, 69.90, 63.71, 34.06, 17.47. MS (ESI, positive mode, acetone) : m/z = 982 [M+2H]$^+$. *Anal.* Calc. for $C_{43}H_{57}B_1F_4Fe_1O_3Ru_3$: C, 48.37; H, 5.38. Found: C, 48.12; H, 5.36%.

2.3. *Catalytic runs*

A solution of [**4**][BF_4] (10 mg) in 10 ml of degassed water was placed in a 100 ml stainless steel autoclave, and the substrate benzene was added with a 1/1000 ratio catalyst/substrate. After purging four times with hydrogen, the autoclave was pressurised with hydrogen (60 bar) and heated to 110 °C in an oil bath under vigorous stirring. After 4 h, the autoclave was placed in an ice-bath and the pressure released. The two-phase system was separated by decanting. The aqueous phase containing the catalyst was evaporated to dryness under vacuum, and the residue was analysed by NMR and mass spectroscopy. The organic phase containing cyclohexane and benzene was analysed by NMR spectroscopy and GC.

2.4. *X-ray crystallography*

Crystals of [**3**], and [**4**][BF_4] were mounted on a Stoe Image Plate Diffraction system equipped with a ϕ circle goniometer, using Mo Kα graphite monochromated radiation ($\lambda = 0.71073$ Å) with ϕ range 0–200°, increment of 1.3 and 1.5°, $D_{max} - D_{min} = 12.45 - 0.81$ Å. The structures were solved by direct methods using the program SHELXS-97 [7]. The refinement and all further calculations were carried out using SHELXL-97 [8]. In [**3**] and [**4**][BF_4] the hydrogen atoms have been included in calculated positions and treated as riding atoms using the SHELXL default parameters. All non-H atoms were refined anisotropically, using weighted full-matrix least-square on F^2. Crystallographic details are summarised in Table 1. Figures were drawn with ORTEP [9].

Full tables of atomic parameters, bond lengths and angles are deposited at the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, Deposition numbers: [**3**] 208397, [**4**][BF_4] 208398.

Table 1
Crystallographic and selected experimental data of [**3**], and [**4**][BF_4]

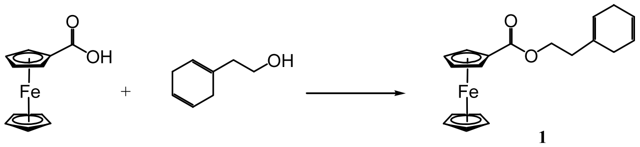
	[**3**]	[**4**][BF_4]·$(CH_3)_2CO$
Chemical formula	$C_{37}H_{33}Cl_2FeO_2PRu$	$C_{46}H_{63}BF_4FeO_4Ru_3$
Formula weight	768.42	1125.83
Crystal system	monoclinic	triclinic
Space group	$P2_1/n$	$P\bar{1}$
Crystal colour and shape	orange rod	orange plate
Crystal size	0.28 × 0.08 × 0.08	0.50 × 0.40 × 0.12
a (Å)	13.361(4)	11.771(1)
b (Å)	13.944(3)	14.671(1)
c (Å)	17.651(5)	14.696(1)
α (°)	90	117.90(1)
β (°)	105.00(3)	90.53(1)
γ (°)	90	94.47(1)
V (Å^3)	3176.6(15)	2232.9(4)
Z	4	2
T (K)	153(2)	153(2)
D_{calc} (g cm^{-3})	1.607	1.674
μ (mm^{-1})	1.185	1.372
Scan range (°)	$4.30 < 2\theta < 52.02$	$4.40 < 2\theta < 51.80$
Unique reflections	6065	8132
Reflections used [$I > 2\sigma(I)$]	3046	6483
R_{int}	0.1268	0.0383
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0584$, wR_2 0.1233	$R_1 = 0.0319$, wR_2 0.0799
R indices (all data)	$R_1 = 0.1270$, wR_2 0.1403	$R_1 = 0.0428$, wR_2 0.0833
Goodness-of-fit	0.845	0.994
Max, Min $\Delta\rho$/ *e* (Å^{-3})	1.326, −1.242	1.205, −0.666

Table 2
Selected bond lengths (Å) and angles (°) for [**3**], and [**4**][BF_4]

	[**3**]		[**4**][BF_4]
Interatomic distances			
O(1)–C(8)	1.460(9)	O(1)–C(8)	1.449(5)
O(2)–C(9)	1.192(10)	O(2)–C(9)	1.207(5)
O(1)–C(9)	1.336(9)	O(1)–C(9)	1.357(5)
Ru(1)–P(1)	2.337(2)	Ru(1)–Ru(2)	2.7422(6)
Ru(1)–Cl(1)	2.407(2)	Ru(2)–Ru(3)	2.7845(5)
Ru(1)–Cl(2)	2.409(2)	Ru(1)–Ru(3)	2.7468(5)
Angles			
Cl(1)–Ru(1)–P(1)	87.76(7)	Ru(1)–Ru(2)–Ru(3)	59.599(15)
Cl(1)–Ru(1)–Cl(2)	88.55(8)	Ru(1)–Ru(3)–Ru(2)	59.435(15)
P(1)–Ru(1)–Cl(2)	89.02(7)	Ru(2)–Ru(1)–Ru(3)	60.965(14)
		Ru(1)–O(3)–Ru(2)	86.36(9)
		Ru(1)–O(3)–Ru(3)	86.58(9)
		Ru(2)–O(3)–Ru(3)	88.32(9)

3. Results and discussion

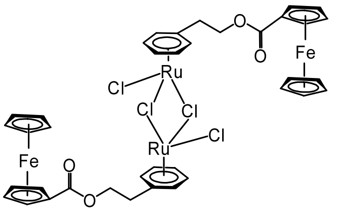
The hydroxi function of 2-cyclohexa-1,4-dienyl-ethanol, accessible by standard *Birch* reduction [10], is available for esterification by classical methods [11]. 2-Cyclohexa-1,4-dienyl-ethanol reacts with ferrocenyl carboxylic acid in dichloromethane, in the presence of condensation agents (*N,N*-dicyclohexylcarbodiimine, 4-(dimethylamino)pyridine, 4-pyrrolidinopyridine) to give the corresponding ester-ferrocenyl derivative (**1**).



1

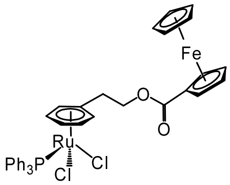
The infrared spectrum of **1** exhibits the characteristic ν_{CO} absorption at 1705 cm^{-1} of the ester function and the ν bands at 1459, 1280 and 1142 cm^{-1} due to the presence of a ferrocene moiety.

The dienyl function of **1** reacts with $RuCl_3 \cdot nH_2O$, in refluxing ethanol to afford quantitatively [Ru(Fc-arene)Cl_2]$_2$ (**2**) (Fc-arene = ferrocene carboxylic acid phenethyl ester). The dinuclear complex is only sparingly soluble in CH_2Cl_2, and decomposes slowly in DMSO to form the well known species [$RuCl_2(DMSO)_4$] [12].



2

To gain further insight in the possible structural effects on the presence of a ferrocenyl group tethered to an arene ligand, the mononuclear triphenylphosphine compound [Ru(Fc-arene)(PPh$_3$)Cl$_2$] (**3**) has been synthesised. The dinuclear complex **2** reacts with 2 equivalents of triphenylphosphine in CH$_2$Cl$_2$ to afford in moderate yield complex **3**. The formation of **3** is conveniently monitored by ^{31}P NMR spectroscopy. The ^{31}P NMR signal is shifted downfield by 32.9 ppm (as compared to uncoordinated triphenylphosphine).



3

The orange air-stable compound is crystallised from a slow diffusion of hexane into a chloroform solution containing **3**. A single-crystal X-ray analysis was performed, confirming the molecular structure of **3**, see Fig. 1. Selected bond distances and angles are given in Table 2.

The ruthenium atom possesses a pseudo-octahedral geometry, and the metrical parameters around the metallic core compare well with those of similar three-legged piano-stool [Ru(η^6-arene)(PPh$_3$)Cl$_2$] complexes [13¬15]. The ferrocene moiety is in the eclipsed conformation. The phenethyl substituent is rotated out of the ester plane by 75.5(9)°, allowing no π-interaction between the Cp ring and the η^6-arene ligand. A similar behaviour has been observed for benzoylferrocene [16] and for methylenebenzotriazolylferrocene derivatives [17]. The C(9)¬O(1) distance [1.336(9) Å] is shorter than that of ferrocenecarboxylic acid benzotriazole ester [1.427(2) Å], which is known to have a weak and reactive ester bond [18]. These observations are in agreement with the stability of the ester bond observed for **3**. The stability of the ester function allows us to synthesise the trinuclear cation [H$_3$Ru$_3$(Fc-arene)(C$_6$Me$_6$)$_2$(O)]$^+$ (**4**) in good yield.

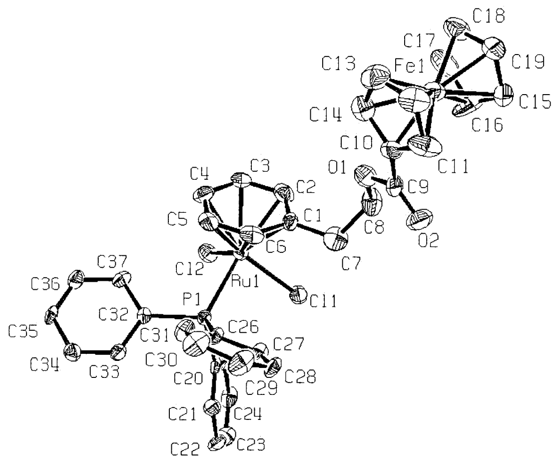
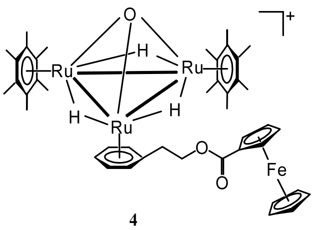


Fig. 1. Molecular structure of **3** at 50% probability level, H atoms omitted for clarity.

The trinuclear cation [H$_3$Ru$_3$(Fc-arene)(C$_6$Me$_6$)$_2$(O)]$^+$ (**4**) is synthesised in a water/acetone solution from the dinuclear precursor [H$_3$Ru$_2$(C$_6$Me$_6$)$_2$]$^+$ [19] and [Ru(Fc-arene)(H$_2$O)$_3$]$^{2+}$, the mononuclear analogue of the known cation [(C$_6$H$_6$)Ru(H$_2$O)$_3$]$^{2+}$ [20], accessible from **2** in aqueous solution.



4

The molecular structure of **4** is shown in Fig. 2. The metal core consists of three ruthenium atoms, the three Ru¬Ru distances being in accordance with a metal¬metal single bond. The three ruthenium atoms are capped by a μ_3-oxo ligand which is almost symmetrically coordinated. The three hydrido ligands bridging the three ruthenium¬ruthenium bonds could be localised and fixed. Selected bond lengths and angles are listed in Table 2. The triruthenium framework is comparable with [H$_3$Ru$_3$(C$_6$H$_2$Me$_4$)$_3$(O)]$^+$ [5], [H$_3$Ru$_3$(C$_6$H$_6$)(C$_6$Me$_6$)$_2$(O)]$^+$ [1], [H$_3$Ru$_3${C$_6$H$_5$(CH$_2$)$_2$OH}(C$_6$Me$_6$)$_2$(O)]$^+$ and [H$_3$Ru$_3${C$_6$H$_5$(CH$_2$)$_3$OH}(C$_6$Me$_6$)$_2$(O)]$^+$ [3], showing similar geometric parameters, differences appear only at the periphery. In the crystal structure, the ferrocene moiety shows no interaction with the triruthenium framework. The ferrocene moiety which is in the eclipsed conformation, is surrounded by acetone solvent molecules, and tetrafluoroborate ions. The torsion angle formed by C(1)¬C(7)¬C(8)¬O(1) [71.0(5)°] is identical to the one observed in **3** [70.3(10)°].

Compound [**4**][BF$_4$] is indeed catalytically active for the hydrogenation of benzene under biphasic conditions, it shows a catalytic activity of 157 h^{-1} (TOF) for a catalyst/substrate ratio 1:1000, at 110 °C under 60 bar

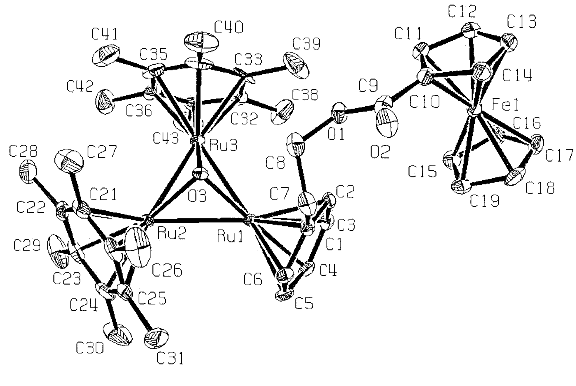


Fig. 2. Molecular structure of [**4**]BF$_4$; tetrafluoroborate ion, acetone molecule, and H atoms omitted for clarity; displacement ellipsoids are drawn at the 35% probability level.

H_2 during 4 h, as compared to 190 h^{-1} for $[H_3Ru_3(C_6H_6)(C_6Me_6)_2(O)][BF_4]$ under the same conditions. Unfortunately, it appears that, after a catalytic run, **4** is partially decomposed into mono- and dinuclear species, among which, we identified by NMR spectroscopy $[H_3Ru_2(C_6Me_6)_2]^+$. The fate of the ferrocenyl–arene–ruthenium moiety is unclear, no decomposition products containing a ferrocenyl group have been identified so far by ^1H NMR spectroscopy.

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