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Synthesis of β-amino-α-ferrocenyl alcohols

Hai-Ying Zhao, Zhan-Xi Bian* and Bao-Guo Li

College of Chemistry and Chemical Engineering, Inner Mongolia University, Huhhot 010021, China

β-Amino-α-ferrocenyl alcohols [$FcC(OH)(R)CH_2NH_2$] (R = Me, Et, ^{n}Pr, ^{i}Pr, Ph, *p*-$MeOC_6H_4$, *o*-ClC_6H_4, *m*-ClC_6H_4, *p*-ClC_6H_4, Fc; Fc = $C_5H_4FeC_5H_5$) were prepared by the reduction of cyanohydrin trimethylsilyl ethers of acylferrocenes with lithium aluminum hydride. All new compounds were characterised by elemental analysis, IR and ^{1}H NMR spectroscopies. The X-ray crystal structure of β-amino-α,α-diferrocenylethanol shows that it has a polymeric chain structure with hydrogen bonding interactions between the OH proton and the N of NH_2.

Keywords: β-amino-α-ferrocenyl alcohol, cyanohydrin trimethylsilyl ether of acylferrocene, reduction

β-Aminoalcohols are useful intermediates in organic chemistry, for example, they serve for the synthesis of various heterocycles,[1] chelate complexes,[2] the ring expansion of cycloalkanones,[3] and have some interesting pharmacological properties.[4] The classical methods for the preparation of β-aminoalcohols have been developed including reduction of ketone cyanohydrins[5] or β-nitromethyl alcohols.[3b,6] Both procedures have suffered from lack of generality and low overall yields, as does the reaction of ammonia with epoxide,[1] which is limited by the nature of the epoxide. However, the reduction of the protected cyanohydrins[7] can be carried out efficiently, affording good yields of β-aminoalcohols. In 1974, Evans synthesised successfully a series of aliphatic and aromatic β-aminoalcohols through the reduction of cyanohydrin trimethylsilyl ethers with lithium aluminum hydride.[7a] Nevertheless, there are no reports on the preparation of β-amino-α-ferrocenylalcohols until now. Here, we disclose the synthesis of β-amino-α-ferrocenylalcohols by the reduction of cyanohydrin trimethylsilyl ether of acylferrocene[8] (which was reported by us previously) with lithium aluminum hydride in ethylether (Scheme 1). The crystal structure of **2j** was further defined by X-ray diffraction. Since they contain ferrocene, those β-aminoalcohols can act as bidentate ligands and may be converted into multinuclear compounds,[5b] and as potential burning-rate catalysts for propellants can also be applied to the space industry.[9]

Experimental

General

Melting points were obtained with a XT7-4 apparatus and are uncorrected. ^{1}H NMR spectra were recorded in $CDCl_3$ solutions on a Bruker DRX-500 spectrometer with $SiMe_4$ as internal standard. IR spectra were recorded on a NEXUS-670FT-IR spectrophotometer using KBr pellets. Elemental analyses were carried out on a Perkin-Elemer-2400 apparatus. X-ray data were collected on a Bruker Smart CCD area detector. Silica gel (60H) plates and silica gel (100–200 mesh) were used for analytical TLC and column chromatography, respectively. Cyanohydrin trimethylsilyl ethers were prepared according to literature methods.[8] Dry Et_2O was distilled from sodium. All other solvents and regents were obtained from commercial sources and used without further purification.

General procedure for the synthesis of **2a–2j**

A dry 100 ml 3-neck round-bottomed flask equipped with magnetic stirring bar, reflux condenser and $CaCl_2$ drying tube was charged with a suspension of $LiAlH_4$ (3–20 mmol) in anhydrous ethylether (15–50 ml). To this suspension was added a solution of compound **1** (2 mmol) in anhydrous ethylether (20 ml) dropwise at a rate maintaining gentle reflux. Stirring was continued under reflux for 1.5 h after the addition had been completed. Destruction of the excess of $LiAlH_4$ was completed by cautious addition of ice-cold water, cooling in an ice water bath. The granular precipitate was filtered off, washed with ethylether, and the combined organic phase was dried over anhydrous potassium carbonate. Removal of the solvent *in vacuo* yielded the crude product that was purified by recrystallisation from ethanol–petroleum ether (60–90°C). [The crude product of compound **2g** was purified by silica gel column chromatography using 3:1 petroleum ether (60–90°C)–ethylether as eluent]. The ratio of $LiAlH_4$ to **1a–j** and yields are listed in Table 1.

2a: Red orange crystals, m.p. 82–84°C. IR (KBr) ν 3400–2650 (OH), 3361, 3279 (NH_2), 3094 (Fc, CH), 1606 (δ_{N-H}) cm^{-1}; ^{1}H NMR ($CDCl_3$, 500MHz) δ 1.51 (s, 3H, CH_3), 1.87 (br, 3H, OH, NH_2, disappear with D_2O), 2.74 (q, 2H, CH_2), 4.10, 4.19, 4.24 (3s, together 9H, ferrocenyl-*H*). Calc. for $C_{13}H_{17}FeNO$: C, 60.3; H, 6.6; N, 5.4. *Anal.* Found: C, 59.6; H, 7.3; N, 5.4%.

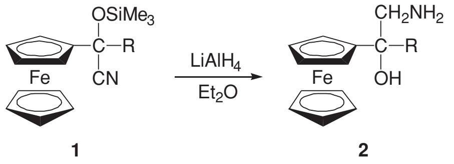
2b: Yellow crystals, m.p. 46–47°C. IR (KBr) ν 3400–3000 (OH), 3348, 3290 (NH_2), 3089 (Fc, CH), 1580 (δ_{N-H}) cm^{-1}; ^{1}H NMR ($CDCl_3$, 500MHz) δ 0.85 (t, 3H, CH_3), 1.72 (m, 1H, $CHHCH_3$), 1.87 (m, 1H, $CHHCH_3$), 2.30 (br, 3H, OH , NH_2, disappear with D_2O), 2.96 (q, 2H, CH_2NH_2), 4.14, 4.21, 4.25 (3s, together 9H, ferrocenyl-*H*). Calc. for $C_{14}H_{19}FeNO$: C, 61.6; H, 7.0; N, 5.1. *Anal.* Found: C, 61.3; H, 7.0; N, 4.8%.

2c: Orange crystals, m.p. 54–55°C. IR (KBr) ν 3500–3100 (OH), 3370, 3299 (NH_2), 3086 (Fc, CH), 1587 (δ_{N-H}) cm^{-1}; ^{1}H NMR ($CDCl_3$, 500MHz) δ 0.90 (t, 3H, CH_3), 1.20 (m, 1H, $CHHCH_3$),

Table 1 The ratio of $LiAlH_4$ to **1a–j** and yields

Substrate **1a–j**	R	Ratio ($LiAlH_4$:**1a–j**)	Yield[a] (%) **2a–j**
a	CH_3	1.5:1	73.3
b	C_2H_5	2:1	68.4
c	$CH_2CH_2CH_3$	2:1	71.3
d	$CH(CH_3)_2$	2:1	54.3
e	Ph	1.9:1	50.0
f	*p*-CH_3OPh	5.2:1	71.2
g	*o*-ClPh	7:1	38
h	*m*-ClPh	5.2:1	46.9
i	*p*-ClPh	5.2:1	46.9
j	Fc	10:1	58.3

[a]Yield of isolated product



a R = Me
b R = Et
c R = ^{n}Pr
d R = ^{i}Pr
e R = Ph
f R = *p*-$MeOC_6H_4$
g R = *o*-ClC_6H_4
h R = *m*-ClC_6H_4
i R = *p*-ClC_6H_4
j R = Fc, Fc = $C_5H_4FeC_5H_5$

Scheme 1

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* Correspondent. E-mail: bzx1957@sina.com

1.34 (m, 1H, C*H*HCH$_3$), 1.61 (m, 1H, CH*H*CH$_2$CH$_3$), 1.74 (m, 1H, C*H*HCH$_2$CH$_3$), 2.14 (br, 3H, O*H* , N*H*$_2$, disappear with D$_2$O), 2.88 (q, 2H, C*H*$_2$NH$_2$), 4.12, 4.20, 4.23 (3s, together 9H, ferrocenyl-*H*). Calc. for $C_{15}H_{21}FeNO$: C, 62.7; H, 7.3; N, 4.9. *Anal.* Found: C, 62.0; H, 7.8; N, 4.8%.

2d: Yellow crystals, m.p. 65–67°C. IR (KBr) ν 3500–3200 (OH), 3365, 3302, (NH$_2$), 3094 (Fc, CH), 1579 (δ_{N-H}) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 0.75 (dd, 6H, CH(C*H*$_3$)$_2$), 2.01 (m, 1H, C*H*(CH$_3$)$_2$), 2.20 (br, 3H, O*H* , N*H*$_2$, disappear with D$_2$O), 3.07 (q, 2H, C*H*$_2$NH$_2$), 4.0, 4.19, 4.23, 4.29 (4s, together 9H, ferrocenyl -*H*). Calc. for $C_{15}H_{21}FeNO$: C, 62.7; H, 7.4; N, 4.9. *Anal.* Found: C, 62.2; H, 7.6; N, 4.4%.

2e: Yellow crystals, m.p. 136–138°C. IR (KBr) ν 3500–3200 (OH), 3364, 3301 (NH$_2$), 3097 (Fc, CH), 3078, 3023 (Ar, CH), 1598 (δ_{N-H}, Ar, ν_{C-C}) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 2.21 (br, 3H, O*H* , N*H*$_2$, disappear with D$_2$O), 3.20 (q, 2H, C*H*$_2$NH$_2$), 4.03, 4.16, 4.20, 4.24, 4.32 (5s, together 9H, ferrocenyl-*H*), 7.25–7.48 (m, 5H, Ar). Calc. for $C_{18}H_{19}FeNO$: C, 67.3; H, 6.0; N, 4.4. *Anal.* Found: C, 67.1; H, 6.3; N, 4.2%.

2f: Orange crystals, m.p. 87–88°C. IR (KBr) ν 3400–2800 (OH), 3347, 3293 (NH$_2$), 3090 (Fc, CH), 3050, 3021 (Ar, CH), 1581 (δ_{N-H}), 1247 (OCH$_3$) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 3.18 (br, 3H, O*H*, N*H*$_2$, disappear with D$_2$O), 3.52 (q, 2H, C*H*$_2$NH$_2$), 3.82 (s, 3H, OC*H*$_3$), 4.01, 4.15, 4.21, 4.23, 4.30 (5s, together 9H, ferrocenyl-*H*), 6.88 (d, 2H, Ar), 7.37 (d, 2H, Ar). Calc. for $C_{19}H_{21}FeNO_2$: C, 65.0; H, 6.0; N, 4.0. *Anal.* Found: C, 64.7; H, 6.5; N, 3.8%.

2g: Orange crystals, m.p. 50–51°C.IR (KBr) ν 3500–3200 (OH), 3381, 3319 (NH$_2$), 3093 (Fc, CH), 3030 (Ar, CH), 1569 (δ_{N-H}) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 3.15 (br, 3H, O*H* , N*H*$_2$, disappear with D$_2$O), 3.40 (q, 2H, C*H*$_2$NH$_2$), 3.92, 4.07, 4.14, 4.16, 4.41 (5s, together 9H, ferrocenyl-*H*), 7.00–7.22 (m, 3H, Ar), 7.92 (d, 1H, Ar). Calc. for $C_{18}H_{18}ClFeNO$: C, 60.8; H, 5.1; N, 3.9. *Anal.* Found: C, 60.6; H, 5.5; N, 3.7%.

2h: Yellow crystals, m.p. 89–90°C. IR (KBr) ν 3500–3250 (OH), 3364, 3301 (NH$_2$), 3078 (Fc, CH), 3028 (Ar, CH), 1570 (δ_{N-H}) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 3.21 (br, 3H, O*H* , N*H*$_2$, disappear with D$_2$O), 3.60 (q, 2H, C*H*$_2$NH$_2$), 4.02, 4.17, 4.22, 4.25, 4.30 (5s, together 9H, ferrocenyl-*H*), 7.23–7.51 (m, 4H, Ar). Calc. for $C_{18}H_{18}ClFeNO$: C, 60.8; H, 5.1; N, 3.9. *Anal.* Found: C, 61.1; H, 5.5; N, 3.7%.

2i: Yellow crystals, m.p. 116.5–117.5°C. IR (KBr) ν 3400–2700 (OH), 3356, 3298 (NH$_2$), 3090 (Fc, CH), 3031 (Ar, CH), 1595 (δ_{N-H}, Ar, ν_{C-C}) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 3.20 (br, 3H, O*H* , N*H*$_2$, disappear with D$_2$O), 3.50 (q, 2H, C*H*$_2$NH$_2$), 4.0, 4.17, 4.21, 4.25, 4.30 (5s, together 9H, ferrocenyl-*H*), 7.31 (d, 2H, Ar), 7.40 (d, 2H, Ar). Calc. for $C_{18}H_{18}ClFeNO$: C, 60.8; H, 5.1; N, 3.9. *Anal.* Found: C, 61.3; H, 5.5; N, 3.8%.

2j: Yellow crystals, m.p. 154–155°C. IR (KBr) ν 3500–3250 (OH), 3378, 3310 (NH$_2$), 3095 (Fc, CH), 1590 (s, δ_{NH}) cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 1.51 (br, 3H, OH, NH$_2$, disappear with D$_2$O), 3.20 (s, 2H, C*H*$_2$NH$_2$), 4.13, 4.17, 4.19, 4.22 (4s, together 18H, 2 × ferrocenyl-*H*). Calc. for $C_{22}H_{23}Fe_2NO$: C, 61.6; H, 5.4; N, 3.3. *Anal.* Found: C, 61.3; H, 5.5; N, 3.1%.

Single crystal X-ray diffraction analysis of **2j**

Data **2j** were collected on a Bruker Smart CCD area detector crystal diffractometer with Mo-Kα radiation (λ = 0.71073Å) at 293K in the φ–ω scan mode, and corrected for absorption semiempirically. The structure was solved by direct method using the SHELXS-97[10] program and refined by full-matrix least squares on F^2 with the SHELXL-97.[11] The non-hydrogen atoms were refined anisotro-pically, whereas the hydrogen atoms were located from difference Fourier maps or were placed in calculated positions and refined isotropically. Crystal data and structure refinement parameters are listed in Table 2.

Results and discussion

Synthesis

10 new β-amino-α-ferrocenyl alcohols were obtained by the reduction of corresponding cyanohydrin trimethylsilyl ethers with LiAlH$_4$. According to the literature method,[7a] only under a N$_2$ atmosphere, can the reduction be carried out efficiently. However, we found that maintaining reflux of the reaction mixture can also cut off the air, so we simplified the process.

We investigated the influence of steric effect of a substrate and found that the reduction reaction was very sensitive to the steric hindrance around the α-C atoms. As is shown in Table 1, the yield was high when the substrate bears a less bulky group as in **1a** (R = Me) and **1b** (R = Et), while in the case of hindered **1d** (R = ^{i}Pr) the yield of **2d** decreased to 54.3%. With two bulky ferrocenyls, compound **1j** was treated with a large amount of LiAlH$_4$ (10 equiv.) to give **2j** in 58.3% yield. Similarly, the quantity of reductive agent was larger and the yield was lower in the case of **1g** which contains an *ortho* substituted benzene ring (*o*-Cl, LiAlH$_4$: **1g** = 7:1, yield 38%) than that of unsubstituted substrate **1e** (LiAlH$_4$: **1e** = 1.9:1, yield 50%). Therefore, the higher steric hindrance of the substrates, the more difficult is the reduction reaction.

Table 2 Crystal data and structure refinement for compound **2j**

Compound	**2j**
Empirical formula	$C_{22}H_{23}Fe_2NO$
Formula weight	429.11
Crystal system	Orthorhombic
Space group	Pna2$_1$
a(Å)	12.1450(9)
b(Å)	12.2301(9)
c(Å)	12.1686(9)
α (°)	90
β (°)	90
γ (°)	90
V(Å^3)	1807.5(2)
Z	4
Calculate density (Mg m^{-3})	1.577
Absorption coefficient(mm^{-1})	1.615
F(000)	888
θ range for data collection (°)	2.36–28.29
Limiting indices	$-16 \le h \le 11$, $-15 \le k \le 16$, $-16 \le l \le 15$
Reflection collection/unique	10635/4074
Max. and min. transmission	1.0000 and 0.8622
Data / restraints / parameters	4074 / 1 /276
Goodness-of-fit on F^2	0.670
Final R indices [$I > 2\sigma(I)$] R$_1$, wR$_2$	0.0452, 0.0544
R indices (all data) R$_1$, wR$_2$	0.1043, 0.0651
Largest diff. peak and hole (e. Å^3)	0.670, –0.312

Spectral properties

The β-amino-α-ferrocenyl alcohols **2a–j** were characterised by means of spectroscopic methods. In the IR spectra, the stretching vibrations of O–H shifted to lower frequencies and showed broad and strong bands (3500–3200 cm^{-1} or 3400–2650 cm^{-1}), compared to the free O–H (3650–3610 cm^{-1}). Those indicated the possibility of hydrogen bonding interactions, which is confirmed by X-ray crystal structure below. The double peaks at around 3360 and 3300 cm^{-1} were assigned to ν_{N-H} of primary amine. The medium intensity absorption in the 3090 cm^{-1} region revealed the existence of ferrocenyl groups.

In the ^{1}H NMR spectrum, the ferrocenyl protons resonated in the region of δ 4.41–4.00. The two methyl protons [CH(C*H*$_3$)$_2$] of **2d** were characterised by a typical double doublet at δ 0.75 (*J* = 6.6Hz) due to the influence of the chiral carbon atom attached to the isopropyl group. Analogously, the ^{1}H NMR spectrum of **2b** and **2c** showed two groups of multiplets for methylene protons in ethyl and propyl, respectively, and the methylene protons attached to the amino-group showed a quartet at δ 2.74–3.07 for **2a–2d** and δ 3.20–3.60 for **2e–2i**. The chemical shift of the latter occurred to lower field because of the deshielding effect of the phenyl ring or ferrocenyl. For the same reason, the chemical shift of OCH$_3$ protons occurred at a lower field in **2f** (δ 3.82) than those of alkylmethylether (δ 3.20).

X-ray structure analysis of **2j**

To firmly establish the structures of the new compounds **2a–j**, X-ray crystallographic analysis of compound **2j** was carried out. Atomic coordinates and equivalent isotropic displacement parameters are listed in Table 3. Selected bond distances and angles are given in Table 4. The molecular structure and the crystal structure are shown in Figs 1 and 2. The bond angles around C11, which range from 105.0°(6) to 111.2°(5), are close to the value for a regular tetrahedron. The dihedral angle between the two cyclopentadiene rings (C6, C7, C8 C9, C10 and C13, C14, C15 C16, C17) is 75.73°. The bond angle of C11–C12–N 121.9°(7) is greater the than normal tetrahedral value of 109.5°, which shows nonequivalent sp^3 hybridisation for the C12 atoms. There exists intermolecular hydrogen bonding interactions between the hydroxyl hydrogens and the nitrogens of the NH$_2$ group (O–H17…N : bond distance 3.303Å(7), bond angle 163.62°(6)). The hydrogen bonds stabilise the crystal structure by forming a polymeric chain structure.

Full details of data collection and structure refinements have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 238226 for compound **2j**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (www.ccdc.cam.ac.uk/conts/retrieving.html; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk)

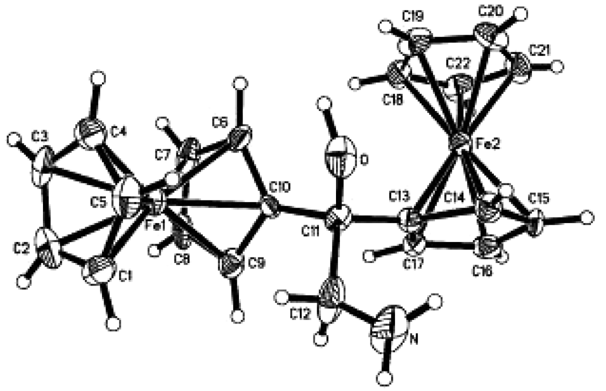

Fig. 1 X-ray structure of **2j**.

Table 3 Atomic coordinates ($\times 10^4$) an equivalent isotropic displacement parameters (Å 2$\times 10^4$) for **2j**

	X	*Y*	*Z*	*U*(eq)
Fe(1)	2374(1)	6641(1)	5534(1)	36(1)
Fe(2)	1269(1)	2066(1)	5557(1)	35(1)
N	-556(5)	4883(5)	3326(6)	107(3)
O	32(3)	4810(3)	5680(6)	59(1)
C(1)	1632(8)	7917(6)	4760(7)	57(3)
C(2)	2643(7)	8227(7)	5160(8)	62(3)
C(3)	2659(7)	8084(7)	6295(8)	56(3)
C(4)	1647(7)	7676(6)	6604(7)	48(2)
C(5)	1008(5)	7566(4)	5661(11)	53(2)
C(6)	2405(6)	5127(6)	6226(6)	42(2)
C(7)	3450(6)	5529(7)	6113(8)	51(2)
C(8)	3673(7)	5750(7)	5001(9)	47(2)
C(9)	2737(6)	5466(6)	4418(6)	44(2)
C(10)	1944(5)	5069(5)	5162(5)	29(2)
C(11)	815(5)	4601(5)	4845(5)	34(2)
C(12)	349(6)	5191(5)	3793(7)	77(3)
C(13)	901(4)	3402(5)	4609(4)	32(1)
C(14)	2(5)	2623(5)	4606(5)	46(2)
C(15)	382(6)	1616(6)	4222(5)	40(2)
C(16)	1494(7)	1777(7)	3939(6)	48(2)
C(17)	1828(6)	2843(6)	4190(5)	38(2)
C(18)	2384(6)	2219(6)	6790(5)	51(2)
C(19)	1344(8)	2479(7)	7176(6)	57(3)
C(20)	705(7)	1553(8)	7044(6)	50(2)
C(21)	1323(7)	731(6)	6555(6)	51(2)
C(22)	2368(6)	1166(6)	6425(6)	56(2)

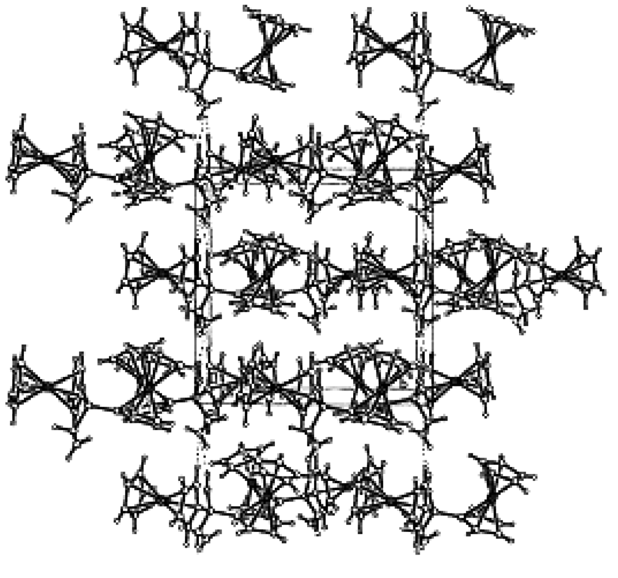

Fig. 2 Perspective drawing of the unit cell of **2j**.

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Table 4 Selected bond distances (Å) and angles (°) of **2j**

Bond distances				Bond angles			
N–C(12)	1.293(6)	Fe(1)–C(10)	2.043(6)	C(10)–C(11)–C(12)	110.8(5)	C(8)–Fe(1)–C(9)	40.0(3)
C(10)–C(11)	1.535(7)	Fe(2)–C(13)	2.049(5)	C(10)–C(11)–C(13)	110.6(5)	C(9)–Fe(1)–C(10)	40.5(2)
C(11)–C(12)	1.575(8)	Fe(2)–C(14)	2.043(6)	C(13)–C(11)–C(12)	108.6(5)	C(6)–Fe(1)–C(10)	40.5(2)
C(11)–C(13)	1.498(7)	Fe(2)–C(15)	2.026(7)	O–C(11)–C(10)	110.6(5)	C(14)–Fe(2)–C(13)	41.5(2)
O–C(11)	1.415(7)	Fe(2)–C(16)	2.019(7)	O–C(11)–C(12)	105.0(6)	C(15)–Fe(2)–C(14)	40.1(3)
Fe(1)–C(1)	2.034(8)	Fe(2)–C(17)	2.033(7)	O–C(11)–C(13)	111.2(5)	C(16)–Fe(2)–C(15)	40.7(3)
Fe(1)–C(2)	2.019(9)	Fe(2)–C(18)	2.030(6)	N–C(12)–C(11)	121.9(7)	C(16)–Fe(2)–C(17)	40.4(3)
Fe(1)–C(3)	2.023(8)	Fe(2)–C(19)	2.035(8)	C(2)–Fe(1)–C(1)	39.7(3)	C(17)–Fe(2)–C(13)	40.5(2)
Fe(1)–C(4)	2.019(8)	Fe(2)–C(20)	2.034(8)	C(2)–Fe(1)–C(3)	40.3(2)	C(18)–Fe(2)–C(19)	39.8(3)
Fe(1)–C(5)	2.014(5)	Fe(2)–C(21)	2.036(7)	C(4)–Fe(1)–C(3)	39.9(3)	C(20)–Fe(2)–C(19)	39.7(3)
Fe(1)–C(6)	2.034(7)	Fe(2)–C(22)	2.027(7)	C(5)–Fe(1)–C(4)	40.4(4)	C(20)–Fe(2)–C(21)	39.9(3)
Fe(1)–C(7)	2.012(8)			C(5)–Fe(1)–C(1)	40.5(3)	C(22)–Fe(2)–C(21)	39.8(3)
Fe(1)–C(8)	2.024(9)			C(7)–Fe(1)–C(6)	39.5(3)	C(22)–Fe(2)–C(18)	39.3(3)
Fe(1)–C(9)	2.026(7)			C(7)–Fe(1)–C(8)	40.8(2)		

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