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Stereochemical factors associated with the rearrangement of (2-ethyl-1-azabuta-1,3-diene)tricarbonyliron(0) complexes

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Abstract

Reaction of (2-ethyl-1,4-diphenyl-1-azabuta-1,3-diene)tricarbonyliron(0) **4** and (2-ethyl-1-benzyl-4-phenyl-1-azabuta-1,3-diene)tricarbonyliron(0) **7** with lithium diethylamide leads to selective formation of the corresponding *endo* (1-methyl-2-amino buta-1,3-diene)tricarbonyliron(0) complexes **5** and **8**, respectively. In each case there is no evidence for the *exo* complexes **6** and **9**. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Lithium amide; Rearrangement; *Endo* enamine complexes

1. Introduction

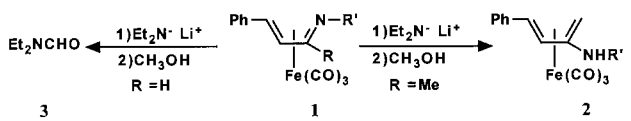
During our investigation of the reaction between (2-methyl-1-azabuta-1,3-diene)tricarbonyliron(0) **1** complexes with lithiated amines we demonstrated that when these contain a methyl group at C-2, the reaction leads to deprotonation, followed by rearrangement of the intermediate anion and re-protonation to yield secondary (2-amino homo-1,3-diene)tricarbonyliron(0) complex **2** in good yield [1,2]. Although tertiary enamine complexes in the cyclohexa-1,3-diene series have previously been reported [3], our deprotonation strategy represented the first synthesis of secondary (enamine)tricarbonyliron(0) complexes.

In cases where the substituent at C-2 of complex **1** is a hydrogen, the lithiated amine reacts by nucleophilic addition at a coordinated carbonyl group, resulting in formation of a formamide **3** after addition of a proton source [2] (Scheme 1).

In this paper we describe the effect of changing the substituent at C-2 of the coordinated 1-azabuta-1,3-diene from a methyl to an ethyl group. In principle, rearrangement of complexes **4** and **7** should lead to formation of 2-amino homo-1,3-diene complexes **5** and **6** or **8** and **9**, respectively (Scheme 2).

2. Results and discussion

Initially, the reaction between (2-ethyl-1,4-diphenyl-1-azabuta-1,3-diene)tricarbonyliron(0) **4** and lithium diethylamide was studied. Complex **4** was synthesised as follows. Condensation of aniline with 2-ethyl-4-phenyl-1-oxabuta-1,3-diene **10** in dichloromethane under titanium tetrachloride catalysis led to formation of a yellow gum after filtration and removal of the solvent under reduced pressure. Examination of the ^{1}H-NMR spectrum of the crude product mixture indicated that 1-azabuta-1,3-diene **11** was present as the major com-



3 1 2

Scheme 1.

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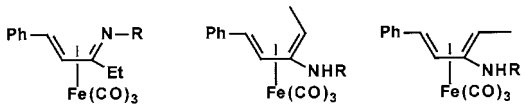
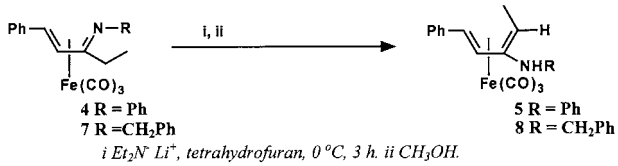


Scheme 2.



Scheme 4.

ponent (> 80% by 300 MHz ^{1}H-NMR spectroscopy). All attempts to purify **11** either by chromatography or crystallisation were unsuccessful and resulted in hydrolysis and formation of the 2-ethyl-4-phenyl-1-oxabuta-1,3-diene **10** and aniline. In view of the high yield of **11** and the difficulty encountered during its purification, the crude mixture was used directly in the complexation step. Formation of complex **4** was therefore achieved by stirring a mixture of crude 1-azabuta-1,3-diene **11** and enneacarbonyldiiron(0) in toluene at 40°C for 3 h in accordance with procedures described in the literature [4,5] to yield red crystals identified as complex **4** on the basis of their spectroscopic and analytical data (Scheme 3).

Treatment of a solution of complex **4** in tetrahydrofuran with lithium diethylamide (synthesised by stirring a solution of diethylamine in tetrahydrofuran with butyl-lithium at 0°C for 0.25 h) for 3 h at 0°C followed by a methanol quench and chromatography lead to isolation of yellow crystals identified as 2-amino homo-1,3-diene complex **5** from their spectroscopic and analytical data. There was no evidence for formation of *E,Z*-complex **6** (by 300 MHz ^{1}H-NMR spectroscopy).

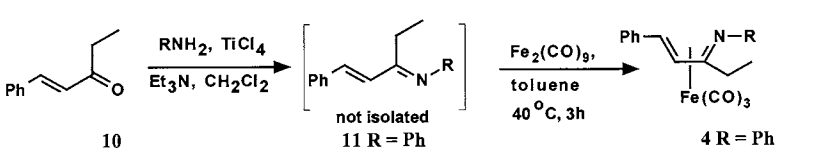
Similarly, complex **7** was synthesised by reaction of 2-ethyl-4-phenyl-1-oxabuta-1,3-diene with benzylamine followed by warming the crude reaction mixture with enneacarbonyldiiron(0). Treatment of **7** with lithium diethylamide lead to formation of the corresponding rearrangement complex **8**. There was again no evidence (by 300 MHz ^{1}H-NMR spectroscopy) for *E,Z*-complex **9** in the product mixture (Scheme 4).

The geometry of the 2-amino homo-1,3-diene ligands of complexes **5** and **8** were confirmed as *E,E* on the basis of nOe difference and ^{1}H-NMR spectroscopy. Irradiation of the signals due to the methyl groups at 1.44 and 1.32 ppm lead to an enhancement of the signals at 2.99 and 3.07 ppm due to the protons at C-4 of complexes **5** (5%) and **8** (4%), respectively. There was no evidence for any enhancement of the signal due to the *ortho* protons of the *N*-phenyl group of complex **5** or the methylene or *ortho* protons of the *N*-benzyl groups of complex **8**. These results indicate that the methyl group of complexes **5** and **8** are in the proximity of the protons at C-4. Such enhancements are only possible if the *E,E*-isomer 2-amino homo-1,3-diene ligands are complexed to the tricarbonyliron(0) moiety (Fig. 1).

Additional information regarding the geometry of the 2-amino homo-1,3-diene ligands of complexes **5** and **8** was obtained by consideration of the chemical shift of the signal due to the proton at C-1. The signal due to this proton appeared as a doublet of quartets at 3.19 ($J = 1.7$ and 7.2 Hz) and 2.84 ($J = 1.9$ and 7.2 Hz). This result confirms the nOe observations that the proton at C-1 is in the *exo* position since an *endo* proton will be situated over and highly shielded by the tricarbonyliron(0) moiety [6] and its signal in the ^{1}H-NMR spectrum is expected to appear between 0 and 1.00 ppm.

In an attempt to understand the reasons for the stereochemistry of this rearrangement reaction, the nature of the starting complexes **4** and **7** was also studied by nOe difference spectroscopy. Irradiation of the signals at 1.55 and 1.47 ppm due to the methyl group of the ethyl fragment at C-2 of complexes **4** and **7** lead to enhancements of the signals at 5.63 and 5.46 ppm due to the protons at C-3, respectively. There was no evidence for any enhancement of the signals due to the *ortho* protons of the *N*-phenyl fragment of complex **4** or the methylene or *ortho* protons of the *N*-benzyl group of complex **7**. From these results it can be assumed that the methyl group at C-1 of complexes **4** and **7** are in the proximity of the protons at C-3 (Fig. 2).

Examination of the molecular models of complexes **4** and **7** indicates that rotation of the bond between the C-2 and the ethyl fragment leads to a significant interaction between the methyl group and the substituents at nitrogen. Such a conformational constraint may be



Scheme 3.

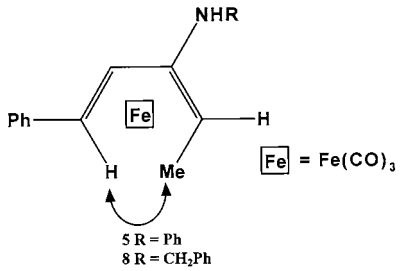


Fig. 1. Complexes **5** and **8**.

used to rationalise the stereochemistry of the rearrangement of complexes **4** and **7**.

The rearrangement of complexes **4** and **7** may therefore be described by deprotonation of the methylene of the conformationally constrained ethyl substituent at C-2. This results in formation of the carbon centred anionic complexes **12** and **13**. Complexes **12** and **13** rapidly convert to the η^3-azaallyl complexes **14** and **15**, in which the negative charges are centred on the tricarbonyliron(0) moiety. Rotation of the bond between C-2 and C-3 will lead to η^3-azaallyl complex **16** and **17**, which upon recoordination of the double bond between carbons **3** and **4** of the 1-homo-1,3-diene leads to the anionic complexes **18** and **19**, in which the negative charge is isolated on nitrogen. Protonation of **18** and **19** will lead to 2-amino-1,3-diene complexes **5** and **8** in which the methyl substituent at C-1 is exclusively in the *endo* position (Scheme 5).

In an attempt to reduce the steric interaction between the substituent at nitrogen and the ethyl substituent at C-2 of complexes **4** and **7**, the complex derived from 2-phenyl ethylamine and 2-ethyl-4-phenyl-1-oxabuta-1,3-diene was prepared. 1-Azabuta-1,3-diene **16** was synthesised by condensation of 2-phenylethylamine with 2-ethyl-4-phenyl-1-oxabuta-1,3-diene under titanium tetrachloride catalysis. Reaction of the crude reaction mixture with enneacarbonyldiiron(0) at 40°C for 3 h lead to isolation of red crystals identified as complex **17** from their spectroscopic and analytical data (Scheme 6).

Although complex **17** was stable and could be fully characterised, reaction with lithium diethylamide at 0°C lead to the formation of an unstable reaction product. It was not possible to obtain satisfactory ^{1}H-NMR

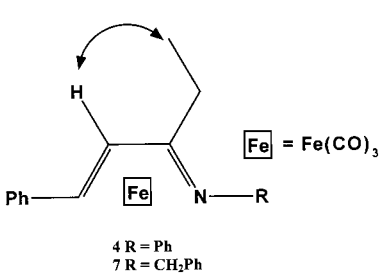
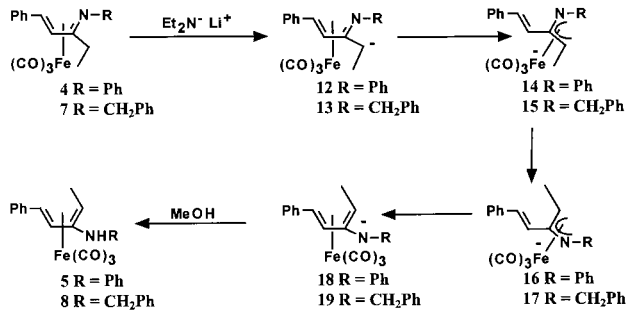


Fig. 2. Complexes **4** and **7**.



Scheme 5.

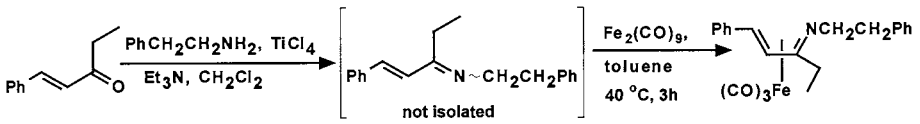
spectra of the reaction mixture even when the samples were prepared under an inert atmosphere. Consequently, the effect of changing the substituent at nitrogen to 2-phenylethyl could not be studied.

Synthesis of complexes bearing a propyl substituent at C-2 was also attempted. In accordance with previous procedures, 2-propyl-4-phenyl-1-oxabuta-1,3-diene was reacted with aniline and benzylamine. In each case the reactions yielded Michael addition products; there was no evidence for the formation of 1-azabuta-1,3-dienes **20** and **21**. Even when the crude reaction mixture was heated with enneacarbonyldiiron(0), formation of 1-azabuta-1,3-diene complexes **22** and **23** was not observed. An alternative approach to complexes **22** and **23**, where the (2-propyl-4-phenyl-1-oxabuta-1,3-diene)tetracarbo-nyliron(0) **24** was heated with aniline or benzylamine, also failed to yield complexes **22** and **23** [7]. Consequently, the effect of changing the substituent at C-2 from ethyl to propyl on this reaarangement reaction was not possible.

The work in this paper illustrates that for rearrangement of (1-azabuta-1,3-diene)tricarbonyliron(0) complexes **4** and **7** that contain an ethyl group at C-2, the relative conformation of the ethyl group with respect to the 1-azabuta-1,3-diene appears to play an important role in determining the stereochemical course of the reaction and hence the reaction products. In the cases of complexes **4** and **7**, the ethyl group is orientated away from the substituents at nitrogen and results in formation of *endo* complexes **5** and **8**. In the case of complex **17**, the conformational constraints associated with the ethyl group at C-2 appear to be absent. Although reaction of this complex with lithium diethylamide leads to unstable reaction products, these failed to give satisfactory ^{1}H- and ^{13}C-NMR spectra.

3. Experimental

All reactions under an atmosphere of nitrogen were performed using standard vacuum and Schlenk line techniques [8]. Dichloromethane was dried over calcium hydride and was distilled, toluene was dried over



10 16 17

Scheme 6.

sodium metal and was distilled and tetrahydrofuran was dried over potassium benzophenone ketyl and was distilled. Diethylamine was redistilled and butyl-lithium was used as a 1.6 M solution in hexanes. Melting points were recorded on a Kofler hot-stage micromelting point apparatus and are uncorrected. ^{1}H- and ^{13}C-NMR spectra were recorded on a Bruker AC 300 instrument at 300 and 75.4 MHz, respectively. All chemical shifts are quoted in ppm relative to a tetramethylsilane standard. Chromatography was performed on Merck (40–63 μm) silica. Filtrations through alumina were performed using deactivated Brockmann grade IV alumina.

3.1. Synthesis of (2-ethyl-1,4-diphenyl-1-azabuta-1,3-diene)tricarbonyliron(0) **4**

2-Ethyl-4-phenyl-1-oxabuta-1,3-diene **10** (0.18 g, 1.14 mmol) was dissolved in dry dichloromethane (30 ml) and stirred at 0°C under an atmosphere of nitrogen. Aniline (0.10 ml, 1.14 mmol) and triethylamine (0.49 ml, 3.50 mmol) were added and the resulting solution was stirred for 0.25 h. Titanium tetrachloride (0.091 g, 0.63 mmol) was added dropwise over a period of 10 min and the dark brown mixture produced was stirred for 0.5 h. The ice-bath was removed and stirring was continued for a further 4 h. The resulting mixture was filtered through celite to remove titanium oxide and the solvent was removed under reduced pressure to give a yellow solid. Dry toluene (30 ml) was added and the mixture was stirred for 0.25 h before being filtered to remove the precipitated amine hydrochloride and the solvent was removed under reduced pressure to yield a yellow gum. The ^{1}H-NMR spectrum of this gum showed a $>65\%$ conversion to *E*,*E* and *E*,*Z* 2-ethyl-4-phenyl-1-azabuta-1,3-diene **11**. δ_H (300 MHz; $CDCl_3$) 1.12 and 1.33 (3H, m, CH_2CH_3), 2.47 and 2.80 (2H, m, CH_2CH_3) and 6.61–7.56 (12H, m, $2\times$Ph and PhC*H*=C*H*). The crude gum containing **11** was redissolved in toluene (10 ml) and enneacarbonyldiiron(0) (0.83 g, 2.28 mmol) was added. The mixture was heated at 40°C for 3 h under an atmosphere of nitrogen. The reaction mixture was cooled to room temperature and filtered through a plug of alumina to remove the solid residues. The solvent was removed under reduced pressure to give a red/brown gum that was chromatographed on silica gel using diethyl ether:hexane (1:5) as the eluent to yield complex **4** as red crystals (0.16 g, 50% with respect to **11**). M.p. (dec.) 123–125°C; (found: C, 64.02; H, 4.43; N, 3.65% $C_{20}H_{17}NFeO_3$ requires C, 64.02; H, 4.57; N, 3.73%); $\nu_{max.}$ (C_4Cl_6) 2 053vs (C≡O), 1 991vs (C≡O) and 1 976vs cm^{-1} (C≡O); δ_H (300 MHz; $CDCl_3$) 1.55 (3H, m, CH_2CH_3), 2.46–2.80 (2H, m, CH_2CH_3), 3.21 (1H, d, $J=8.9$ Hz, PhC*H*=CH), 5.63 (1H, d, $J=8.9$ Hz, PhCH=C*H* and 6.72–7.40 (10H, m, $2\times$Ph); δ_C (75 MHz; $CDCl_3$) 13.20 (CH_2CH_3), 22.20 (CH_2CH_3), 60.21 (C4), 68.28 (C3), 121.26, 123.30, 126.62, 126.73, 128.68, 129.09, 139.46 and 149.82 ($2\times$Ph and C2).

3.2. Reaction of (2-ethyl-1,4-diphenyl-1-azabuta-1,3-diene)tricarbonyliron(0) **4** with lithium diethylamide

Butyl-lithium (1.6 M; 0.84 ml, 1.35 mmol) was added to a solution of diethylamine (0.10 g, 1.33 mmol) in tetrahydrofuran (5 ml) and the solution was cooled to 0°C and the resulting solution was stirred for 0.25 h. A solution of (2-ethyl-1,4-diphenyl-1-azabuta-1,3-diene)-tricarbonyliron(0) **4** (0.10 g, 0.27 mmol) in tetrahydrofuran (5 ml) was added and the reaction was stirred at 0°C for 3 h under an atmosphere of nitrogen before being quenched with methanol (1 ml). The resulting solution was allowed to warm to room temperature for 0.5 h and was filtered through alumina to remove the solid residues and the solvent was removed under reduced pressure to yield an orange gum. This gum was chromatographed on silica gel using diethyl ether:hexane (1:20) as the eluent to yield complex **5** as a yellow gum that crystallised on standing and was recrystallised from hexane at -20°C (0.072 g, 72%). M.p. 126–128°C; (found: C, 63.91; H, 4.39; N, 3.74%, $C_{20}H_{17}NFeO_3$ requires C, 64.02; H, 4.57; N, 3.73%); $\nu_{max.}$ (thin film) 2 039vs (C≡O) and 1 973vs, br cm^{-1} (C≡O); δ_H (300 MHz; $CDCl_3$) 1.44 (3H, d, $J=7.2$ Hz, C=CHαCH_3), 2.99 (1H, d, $J=8.6$ Hz, PhC*H*=CH), 3.19 (1H, dq, $J=1.7$ Hz and 7.2 Hz, C=C*H*αCH$_3$), 5.38 (1H, br, NH), 5.76 (1H, dd, $J=1.7$ Hz and $J=8.6$ Hz, PhCH=C*H*) and 7.00–7.42 (10H, m, $2\times$Ph); δ_C (75 MHz; $CDCl_3$) 14.33 (C=CHα*C*H$_3$), 48.48 (C=*C*HαCH$_3$), 50.49 (C4), 71.19 (C3), 119.52, 122.40, 125.97, 126.16, 128.51 and 129.53 ($2\times$Ph and C-2).

3.3. Synthesis of (1-benzyl-2-ethyl-4-phenyl-1-azabuta-1,3-diene)tricarbonyliron(0) 7

4-Phenyl-2-ethyl-1-oxabuta-1,3-diene **10** (1.0 g, 6.25 mmol) was dissolved in dry dichloromethane (30 ml) and cooled to 0°C under an atmosphere of nitrogen. To this solution was added benzylamine (0.68 ml, 6.25 mmol), dry triethylamine (2.7 ml, 19.3 mmol) and titanium tetrachloride (0.38 ml, 3.47 mmol) was added dropwise over a period of 10 min. The mixture was stirred for 30 min before the ice-bath was removed and stirring was continued for 15 h. The dark brown solution produced was filtered through celite and the solvent removed under reduced pressure to give a yellow gum that was dissolved in dry toluene and stirred for 20 min in order to precipitate the amine hydrochloride. The salt was removed by filtration, which afforded a 2:1 mixture of crude *E*,*E* and *E*,*Z* 2-ethyl-1-benzyl-4-phenyl-1-azabuta-1,3-diene **16** as a yellow gum after removal of the solvent. δ_H (300 MHz; $CDCl_3$) 1.22 (3H, m, CH_2CH_3), 2.18 (2H, m, CH_2CH_3), 4.20 (2H, m, CH_2Ph) and 7.00–7.50 (12H, m, 2 × Ph and PhC*H*=C*H*). Crude **16** was redissolved in toluene (10 ml) and enneacarbonyldiiron(0) (4.55 g, 12.5 mmol) was added and the mixture was heated at 40°C for 3 h under an atmosphere of nitrogen. The resulting dark brown mixture produced was filtered through alumina to remove the solid residues and the solvent was removed under reduced pressure to yield a brown gum. Chromatography of this gum on silica using diethyl ether:light petroleum (1:20) as the eluent gave complex **7** as red crystals, (0.88 g, 36%). M.p. (dec.) > 60°C; (found: C, 64.66; H, 4.89; N, 3.68; $C_{21}H_{19}FeNO_3$ requires C, 64.77; H, 4.92; N, 3.60); $\nu_{max.}$ (C_4Cl_6) 2 050vs (C≡O), 1 987vs (C≡O) and 1 963vs cm^{-1} (C≡O); δ_H (300 MHz; $CDCl_3$) 1.47 (3H, m, CH_2CH_3), 2.67–2.94 (2H, m, CH_2CH_3), 2.93 (1H, d, $J = 8.8$ Hz, PhC*H*=CH), 3.40 (1H, d, $J = 15.4$ Hz, PhCH*H*N), 4.07 (1H, d, $J = 15.4$ Hz, PhC*H*HN), 5.46 (1H, d, $J = 8.8$ Hz, PhCH=C*H*) and 7.12–7.37 (10H, m, 2 × Ph); δ_C (75 MHz; $CDCl_3$) 12.46 (CH_2CH_3), 21.64 (CH_2CH_3), 57.81 ($PhCH_2N$), 58.87 (C4), 67.90 (C3), 126.58, 127.07, 127.91, 128.27, 128.48, 133.51, 140.06 and 140.41(2 × Ph and C2).

3.4. Reaction of (1-benzyl-2-ethyl-4-phenyl-1-azabuta-1,3-diene)tricarbonyliron(0) 7 with lithium diethylamide

n-Butyl-lithium (1.6 M; 0.81 ml, 1.30 mmol) was added to a solution of diethylamine (0.09 ml, 1.28 mmol) in tetrahydrofuran (5 ml) at 0°C and the resulting solution was stirred at this temperature for 0.25 h under an atmosphere of nitrogen. A solution of complex **7** (0.10 g, 0.26 mmol) in tetrahydrofuran (5 ml) was added and the resulting mixture was stirred at 0°C for 1.5 h under an atmosphere of nitrogen. The reaction was quenched with methanol (1 ml) and was allowed to warm to room temperature for 0.5 h. The dark solution produced was filtered through alumina and the solvent was removed under reduced pressure to give a dark yellow gum. This gum was chromatographed on silica gel using diethyl ether:hexane (1:20) as the eluent to yield a yellow oil identified as enamine complex **8**, (0.078 g, 78%). B.p. (dec.) > 100°C; (found: C, 64.56; H, 4.70; N, 3.75; $C_{21}H_{19}FeNO_3$ requires C, 64.77; H, 4.92; N, 3.60); $\nu_{max.}$ (hexane) 1 974 (C≡O) 1 992 (C≡O), 2 053 cm^{-1} (C≡O); δ_H (300 MHz; $CDCl_3$) 1.32 (3H, d, $J = 7.2$ Hz, C=CHαCH_3), 2.84 (1H, dq, $J = 1.8$ Hz and 7.2 Hz C=C*H*αCH$_3$), 3.07 (1H, d, $J = 8.4$ Hz, PhC*H*=CH), 3.42 (1H, br, N*H*), 4.13–4.31 (2H, m, $PhCH_2N$), 5.27 (1H, dd, $J = 1.8$ Hz and 8.4 Hz, PhCH=C*H*) and 7.11–7.44 (10H, m, 2 × Ph); δ_C (75 MHz; $CDCl_3$) 14.43 (=CHαCH_3), 49.15 (NCH_2Ph), 49.66 (=*C*HαCH$_3$), 51.38 (C-4) 61.97 (C-3) 125.96, 126.10, 126.29, 127.13, 128.09, 128.32, 128.65, 129.47 and 129.15 (2 × Ph and C-2).

3.5. Synthesis of (2-ethyl-1-(2-phenylethyl)-4-phenyl-1-azabuta-1,3-diene)tricarbonyliron(O) 17

2-Ethyl-4-phenyl-1-oxabuta-1,3-diene **10** (1.00 g, 6.25 mmol) and 2-phenylethylamine (0.76 g, 6.25 mmol) were dissolved in toluene (10 ml) and the mixture was stirred for 60 h. A small aliquot was taken for ^{1}H-NMR analysis. Owing to the complexity of the spectrum, it was not possible to confirm formation of 2-ethyl-1-(2-phenylethyl)-4-phenyl-1-azabuta-1,3-diene **16**. Enneacarbonyldiiron(0) (4.55 g, 12.5 mmol) was added to crude 1-azabuta-1,3-diene **16** and the mixture was stirred at 40°C for 1 h under an atmosphere of nitrogen. The product mixture was filtered through alumina and the solvent was removed under reduced pressure to yield a brown gum. Chromatography of this gum on silica gel using diethyl ether:hexane (1:20) as the eluent, followed by removal of the solvent gave (2-ethyl-1-(2-phenylethyl)-4-phenyl-1-azabuta-1,3-diene) tricarbonyliron(0) **17** as a red gum, (0.026 g, 10%). B.p. (dec.) > 100°C; (found: C, 65.44 H, 5.22 N, 3.30; $C_{22}H_{21}NFeO_3$ requires C, 65.49; H, 5.25; N, 3.47); $\nu_{max.}$ (hexane) 2 049vs (C≡O), 1 986vs (C≡O) and 1 968vs cm^{-1} (C≡O); δ_H (300 MHz; $CDCl_3$) 1.43 (3H, m, CH_2CH_3), 2.33–3.03 (7H, m, $PhCH_2CH_2N$, PhC*H*=CH and CH_2CH_3), 5.45 (1H, d, $J = 8.8$ Hz, PhCH=C*H*) and 7.12–7.32 (10H, m, 2 × Ph); δ_C (75 MHz; $CDCI_3$) 12.43 (CH_2CH_3), 21.48 (CH_2CH_3), 38.51 and 56.04 ($PhCH_2CH_2N$), 59.14 (C-4), 67.55 (C-3), 126.16, 126.28, 126.58, 128.36, 128.53, 128.76, 133.47, 139.90 and 140.01 (2 × Ph and C-2).

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References

[1] M.J. Ackland, T.N. Danks, M.E. Howells, J. Chem. Soc. Perkin Trans. 1 (1998) 813.
[2] M.J. Ackland, T.N. Danks, M.E. Howells, Tetrahedron Letts. 37 (1996) 691.
[3] A.J. Birch, L.F. Kelly, T.P. Thompson, J. Chem. Soc. Perkin Trans. 1 (1981) 1006.
[4] S. Otsuka, T. Yoshida, A. Nakamura, Inorg. Chem. 6 (1967) 20.
[5] T.N. Danks, S.E. Thomas, J. Chem. Soc. Perkin Trans. 1 (1990) 761.
[6] (a) H.D. Preston, J.C. Davies, J. Am. Chem. Soc. 88 (1966) 1585. (b) P. Crews, J. Am. Chem. Soc. 95 (1973) 636.
[7] C.H. Cheng, M.F. Semmelhack, J. Organomet. Chem. 393 (1990) 237.
[8] D.F. Shriver, M.A. Dresdon, The Manipulation of Air-Sensitive Compounds, Wiley, Chichester, 1986.