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Structural studies of the $C_4H_8N_2Fe_2(CO)_6$ complex obtained by cyclization of acetaldazine in the presence of dodecacarbonyl-triiron: investigations by X-ray diffraction and NMR of μ-1,2-(1,2-diaza-3-methylcyclopentane)diyl-bis(tricarbonyliron)

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Abstract

The molecular structure of a complex obtained from 3,4-diazahexa-2,4-diene in reaction with dodecacarbonyl-triiron proceeding via cyclization of the azine was determined by X-ray diffraction. On the basis of X-ray conformational data, the complete assignment of ^{1}H-NMR signals and a determination of geminal and vicinal coupling constants have been performed. The agreement of X-ray and NMR conformational parameters indicated that the differences in the structure of the title complex in the solid state and in solution are negligible. © 2002 Published by Elsevier Science B.V.

Keywords: Iron complexes; Azines; Acetaldazine; Bis(tricarbonyliron); Assignment of ^{1}H-NMR signals; Complementary X-ray-NMR study; Conformational analysis; μ-1,2-(1,2-Diaza-3-methylcyclopentane)diyl-bis(tricarbonyliron)

1. Introduction

In the reaction of iron carbonyls with organic substrates containing a bond between two nitrogen atoms, this bond is cleaved in most cases; however, if it is retained, usually only one nitrogen atom is involved in the coordination of the metal [1–5]. Until now, only a few complexes have been described in which the σ-bond between nitrogen atoms remains preserved and both of these atoms participate in the Fe–N–Fe bridges [6–12]. In such complexes the N_2Fe_2 core forms a nearly regular tetrahedron. Both types of complexes, denoted as **I** and **II**, are schematically shown in Fig. 1.

The geometries of the central core in complexes of type **I** and **II** are distinct. For example, the length of the Fe–Fe bond is increased by 0.1 Å in **I** as compared with **II**, and there is a dramatic change of the N–Fe–N angle from 43° in **I** to 70° in **II** [7]. Moreover, in **I** the intramolecular stress results in a decrease of stability [12,13].

As reported in the literature cited above, binuclear complexes of the type **I** were typically obtained by reacting azo-compounds with iron carbonyls [6]. In contrast, the complex characterized in the course of the present work was formed from acetaldazine $(CH_3CH{=}N–)_2$ and $Fe_3(CO)_{12}$ as a result of a cyclization of the organic substrate with rearrangement of the hydrogen atom (scheme of the reaction is shown in Fig. 2). The presence of the $NCH_2CH_2CH(CH_3)N$ moiety in the complex was documented by ^{13}C-NMR [13]. This result was surprising, because as observed earlier [4], when symmetric ketazines carrying aryl and methyl substituents or when propionaldehyde azine have been used for complexation, the N–N bond was cleaved and binuclear complexes were obtained as two geometric isomers (*anti* and *syn*), as shown in Fig. 1 (**II**). The bis(μ-iminato) system $(CH_3CH{=}N)_2Fe_2(CO)_6$ of type **II**, initially expected in the present work, was already reported [14] as occurring exclusively in the *syn* form;

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however, in the synthesis a different route than complexation of the azine has been applied.

The aim of the present work was the characterization of the complex obtained from acetaldazine and $Fe_3(CO)_{12}$, including the determination of its structure by X-ray diffraction, and the analysis of the ABMN proton spin system by NMR in solution.

2. Results and discussion

As previously reported [13], the reaction of acetaldazine $(CH_3CH{=}N–)_2$ with dodecacarbonyl-triiron resulted in a deep-orange complex **1** of the composition $C_4H_8N_2Fe_2(CO)_6$. Compared with complexes prepared from other azines [4,5], the compound was as fairly unstable and could not be separated into *anti* and *syn* isomers [13] typical for the structure shown in Fig. 1 (**II**).

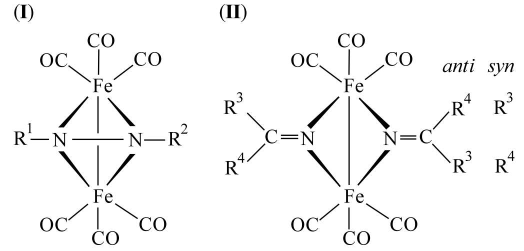

Fig. 1. Schematic structure of: (**I**), complexes $R^1R^2N_2Fe_2(CO)_6$ with N–N σ-bond, where, in known compounds $R^1{=}R^2$ or the moiety $N_2R^1R^2$ forms a cyclic ligand; and (**II**), complexes $(R^3R^4CN)_2Fe_2(CO)_6$ in which between N atoms no chemical bond occurs.

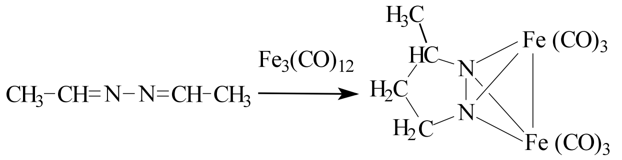

Fig. 2. Complexation reaction of acetaldazine with $Fe_3(CO)_{12}$.

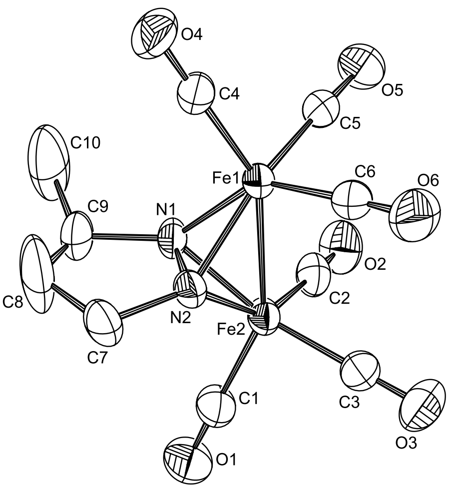

Fig. 3. ORTEP plot of **1** with the atom labeling scheme. Thermal ellipsoids are drawn at the 50% probability level. Hydrogen atoms are omitted for clarity.

2.1. Molecular structure

As determined by X-ray diffraction, the central core of complex **1** (schematically shown in Fig. 2, molecular structure is presented in Fig. 3) consists of two iron and two nitrogen atoms linked by σ-bonds and forming a nearly symmetric tetrahedron. The results obtained for **1** are in good agreement with the literature data for $(\mu\text{-}\eta^2\text{-}R_2N_2)\{Fe(CO)_3\}_2$ complexes of the general type **I**, where for known compounds $R^1 = R^2$ denotes methyl, ethyl or phenyl groups, or $N_2R^1R^2$ stands for a cyclic structure. The N–N distance is 1.402(5) Å in **1**, proving the preservation of a covalent σ-bond (literature data, single $N(sp^3)–N(sp^3)$ σ-bond 1.45(2), double N=N bond 1.24(1) Å [15]). For comparison of selected structural parameters of **1** with those of type **I** complexes reported in the literature [6–12], the data were retrieved from Cambridge Structural Database [16]. The values for **1** are as follows (in parentheses, the corresponding average values for type **I** are given): N–N distance 1.402(5) Å, (1.397); Fe–Fe 2.511(1) Å, (2.498); Fe–N average 1.903(3) Å, (1.905), angle N–Fe–N 43.2(2)°, (43.0). Furthermore, in close agreement with other compounds containing an $E_2M_2(CO)_6$ core, the carbonyl ligands in the $(Fe(CO)_3)_2$ moiety adopt in **1** a nearly synperiplanar conformation, with an average torsion angle C–Fe–Fe–C of 7°. Selected structural parameters of **1** are collected in Table 1.

The organic ligand in **1** forms a five-membered ring N(1)N(2)C(7)C(8)C(9) shaped as an open envelope with the carbon atom C(8) located 0.23(1) Å out of the least-square plane defined by the atoms N(1)N(2)C(7)C(9) (with rms deviation of 0.003 Å). The distance of carbon C(10) of the methyl group from this plane is 0.67(1) Å, and C(10) is situated on the same side of the plane as the carbon C(8). The anisotropic thermal parameters observed for both C(8) and C(10) are far larger as noted for other carbon atoms, as visualized on the ORTEP [17] plot (Fig. 3). The values of these parameters are especially high in the direction perpendicular to the plane defined by the atoms N(1)N(2)C(7)C(9), either as a result of molecular motion due to dynamic conformational transitions in the ring, or caused by structural disorder in the lattice. A distinction between these phenomena is beyond the scope of the present work.

In complexes of type **I**, the geometry around the nitrogen atoms in the tetrahedral central core N_2Fe_2 can be interpreted as a result of an $N(sp^2)$ hybridization, where the N–N bond is formed by p-orbitals and

Table 1
Selected bond lengths (Å), valence and torsion angles (°) for **1**

Bond lengths			
Fe(1)–Fe(2)	2.5111(10)	C(7)–C(8)	1.414(8)
N(1)–N(2)	1.402(5)	C(8)–C(9)	1.470(9)
Fe(1)–N(1)	1.911(3)	Fe(1)–C(4)	1.798(5)
Fe(1)–N(2)	1.894(3)	Fe(1)–C(5)	1.791(5)
Fe(2)–N(1)	1.897(3)	Fe(1)–C(6)	1.792(5)
Fe(2)–N(2)	1.909(3)	C(4)–O(4)	1.138(5)
N(1)–C(9)	1.492(5)	C(5)–O(5)	1.147(5)
N(2)–C(7)	1.478(5)	C(6)–O(6)	1.129(6)
Bond angles			
N(1)–Fe(1)–N(2)	43.24(15)	Fe(1)–N(1)–Fe(2)	82.52(13)
N(1)–Fe(2)–N(2)	43.23(14)	Fe(1)–N(2)–Fe(2)	82.64(12)
C(7)–N(2)–N(1)–C(9)	−0.6(5)	Fe(1)–N(1)–N(2)–Fe(2)	−90.43(10)
C(8)–C(9)–N(1)–N(2)	9.6(7)	C(1)–Fe(2)–Fe(1)–C(4)	−9.1(5)
N(1)–N(2)–C(7)–C(8)	−9.0(7)	C(2)–Fe(2)–Fe(1)–C(5)	−6.2(2)
N(2)–N(1)–C(9)–C(10)	147.6(7)	C(3)–Fe(2)–Fe(1)–C(6)	−5.8(2)

is localized above the line connecting both nitrogen atoms (the arrangement of bonds in the core is shown in Fig. 4). This assumption is consistent with the rather short N–N distance (1.402 Å) in **1** and in compounds of type **I** (average 1.397 Å) as compared with the length of a typical N(sp^3)–N(sp^3) σ-bond (1.45 Å). However, the nitrogen atoms in **1** deviate from the planes C(7)Fe(1)Fe(2) and C(9)Fe(1)Fe(2) outside the molecule (denoted here as positive distortion) by 0.112(5) and 0.141(5) Å, respectively. Positive or negative deviations of this kind were observed for all known compounds of type **I**, and in general the negative distortion (towards the other N atom) was noted in case of discrete substituents at nitrogen atoms, such as methyl, ethyl or phenyl groups, and also for bridging chains forming 7- or 6-membered rings. As can be seen from Table 2 in which the above deviations are listed (based on structural data from Cambridge Structural Database [16]), the highest negative distortion (−0.11 Å) occurs for fairly voluminous separate phenyl substituents, and the value close to zero (−0.02) was noticed for a six-membered ring. In contrast, the stressed five-membered rings give rise to positive deviations with an average value of 0.11 Å, and in case of bicyclo-bridges closing both five- and six-membered rings the distortion reaches 0.17 Å.

The coordination around the iron atoms in **1** can be described as substantially distorted octahedral, what is consistent with former observations for the complex $(CH_3)_2N_2Fe_2(CO)_6$ [6].

2.2. ^{1}H-NMR results

The molecular sequence N–CH$_2$–CH$_2$–CH(CH$_3$)–N in the organic ligand of **1** has been already proposed [13] on the basis of ^{13}C-NMR chemical shifts (ppm) and C–H off-resonance splittings: 56.5(t), 43.8(t), 61.5(d) and 19.7(q), respectively, for the above order of atoms. However, in contrast to the explicit carbon spectrum, the resonances in ^{1}H-NMR resulted from six nonequivalent groups of protons with nine different coupling constants, and had multiplex character due to the ABMN spin system in the rigid ring structure. Hence, the assignment of multiplets, especially to protons in positions *anti* and *syn* with respect to the methyl group, and the determination of all coupling constants required a thorough conformational NMR analysis. The experimental multiplets of H(7) *anti*, H(7) *syn*, H(8) *anti*, H(8) *syn* and H(9) are shown in Fig. 5, the spatial arrangement of protons is schematically depicted in Fig. 6, and selected spectral and structural data are collected in Table 3.

In the ^{1}H-NMR spectrum of **1**, distinct and well-separated multiplets of all proton groups have been observed, i.e. a typical doublet at 1.30 ppm (δ), $J = 6.44$ Hz, and five multiplets evincing expected symmetry in the arrangement of constituent signals. Considering the differences in chemical shifts it can be assumed that three multiplets appearing at lower fields, at 3.36, 3.51 and 3.63 ppm, originate from the protons in positions α to the nitrogen atoms, whereas the remaining multiplets at 2.38 and 2.73 ppm arise from protons at C(8). This was confirmed, and also the multiplet of H(9) was identified at 3.51 ppm by a 2-D heteronuclear ^{1}H–^{13}C COSY experiment (the ^{1}H–^{13}C spectrum is shown in

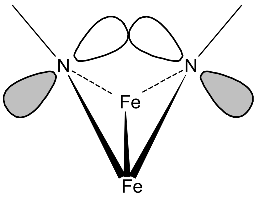

Fig. 4. Geometry of the central core N$_2$Fe$_2$ with p-orbitals forming the N–N bond.

Table 2
Deviations of nitrogen atoms from the planes C′Fe(1)Fe(2) and C″Fe(1)Fe(2), where C′ and C″ mean carbon atoms bonded with the regarded nitrogens, in complexes of the type **I**

Cambridge Structural Database refcode [16]	Δpl_N(1) (Å)	Δpl_N(2) (Å)	Ring size or substituents	Reference
MAZFCO	−0.06	−0.04	Methyl	[6]
KAVYUB	−0.05	−0.05	Ethyl	[10]
LAYZAM	−0.07	−0.11	Phenyl	[12]
BAGRUW	−0.01	−0.07	7	[9]
BCINFE	−0.02	−0.02	6	[7]
WALJUO	0.10	0.12	5	[11]
WALJUO	0.13	0.10	5	[11]
WALKAV	0.10	0.10	5	[11]
AZHPFE10	0.17	0.16	(5, 6) [a]	[8]
AZHPFE10	0.17	0.16	(5, 6) [a]	[8]
1 [b]	0.14	0.11	5	This work

Negative value means the deviation towards the next N atom.

[a] Bicyclo.

[b] Denotation used in this work.

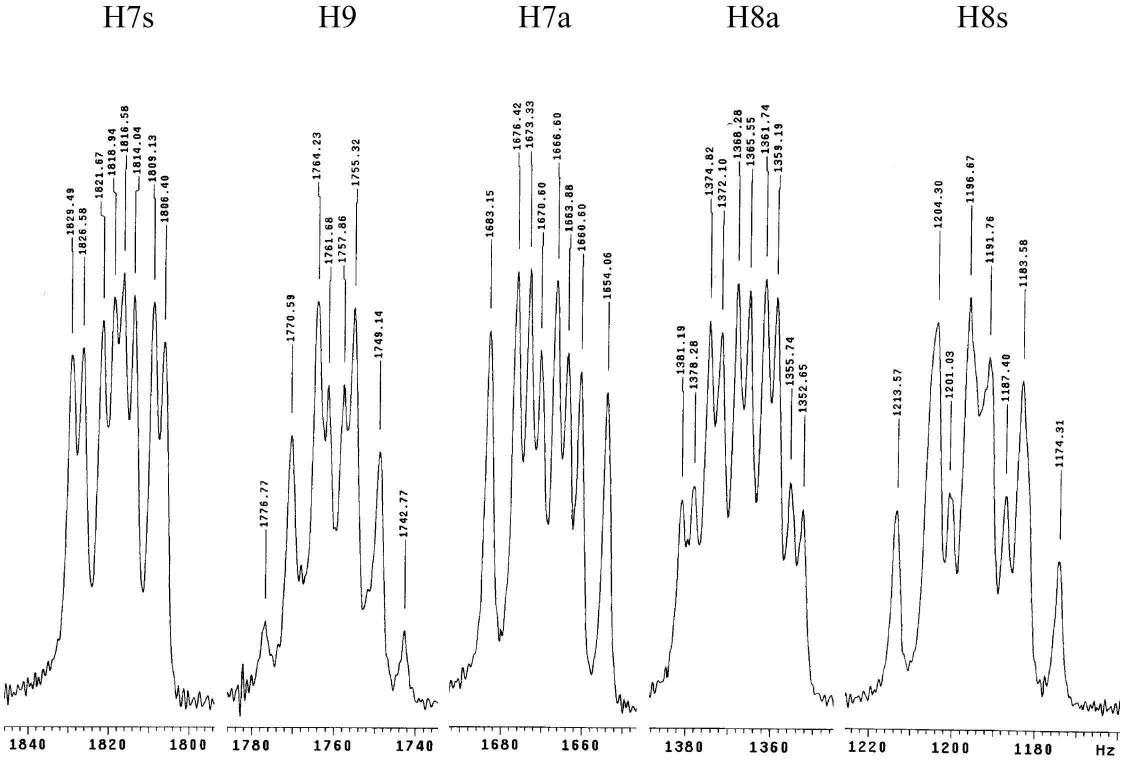

Fig. 5. The experimentally observed multiplets in the ^{1}H-NMR spectrum of **1** assigned to protons at C(7), C(8) and C(9).

Fig. 7). Nevertheless, the assignment of signals to protons *anti* and *syn* at C(8) (2.38, 2.73 ppm) and C(7) (3.36, 3.63 ppm) could only be accomplished by analysis of the coupling constants after taking into account the conformation of the ligand.

The general assumption was made that in **1** the conformational changes in solution as compared with the crystalline state are negligible in the comparative analysis of valence and particularly dihedral angles. This allowed the prediction of all coupling constants in the CH_2CH_2CH rigid system according to the correlation of Karplus and co-workers [18,19]. Karplus emphasized that deviations from his approximation can occur, especially in the case of electronegative substituents and non-typical bond lengths, thus, in this work an initial verification for **1** was necessary. Consequently, all predicted vicinal and geminal coupling values have been summarized for each proton, and the results compared with the total width of experimental multiplets. A satisfactory agreement between the pre-

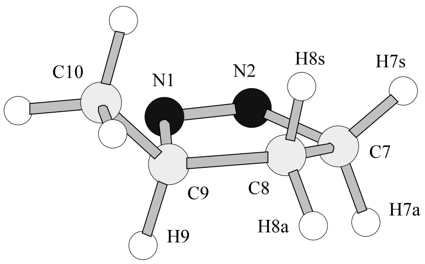

Fig. 6. Spatial arrangement of protons in **1**.

dicted and observed values was observed for all protons in the ring (as shown in Table 3), and these results validated the application of the Karplus correlation for **1**. Hence, it can be concluded that the X-ray structure of the complex remains generally intact in solution.

It should be stressed that in the X-ray analysis of **1** the positions of hydrogen atoms were not refined, thus, protons were included in locations calculated on the basis of the conformation of the carbon skeleton. Nevertheless, this accuracy turned out to be entirely adequate for the prediction of H–H coupling constants.

Based on the widths of the multiplets, the complete assignment of signals to protons in particular locations can be carried out, provided that the differences between the considered values are sufficient. As can be seen from Table 3, this is the case for protons whose resonances needed to be assigned in **1**, i.e. protons located at C(7) (the experimental widths of multiplets to be distinguished are 29.09 and 23.09 Hz) and, accordingly, at C(8) (28.54 and 39.26 Hz). Moreover, the measured values are in good agreement with predicted ones. Hence, the results of assignment are as follows: H(7) *anti* appears in the spectrum at 3.36 ppm, H(7) *syn* at 3.63, H(8) *anti* at 2.73 and H(8) *syn* at 2.38 ppm.

Consequently, on the basis of the above results the exact values of all coupling constants ^{2}J (geminal) and ^{3}J (vicinal) were determined in **1** directly from the experimental ^{1}H-NMR spectra. When needed, the multiplets were simulated [20] and then their shapes were matched with recorded signals by fine corrections of previously estimated values for coupling constants. After a satisfactory agreement of the shape of the signals has been achieved, the overlapping constituents were localized in the experimental spectra and the recognizable splittings were repeatedly measured. The final values of coupling constants are given in Table 3.

In particular, the analysis of experimental multiplets arising from protons at C(7) allowed the direct determination of a geminal coupling constant $^{2}J_{H(7a)-H(7s)}$ and all four vicinal coupling constants with protons at the neighboring carbon C(8). From the doublet given by C(10)H$_3$ the value of $^{3}J_{CH3-H(9)}$ was obtained, and the analysis of the multiplet assigned to H(9) provided the information about $^{3}J_{H(9)-H(8s)}$. Since the values of $^{3}J_{CH3-H(9)}$ and $^{3}J_{H(9)-H(8a)}$ are very close, the latter one was determined by subtraction of all remaining coupling constants from the total width of the multiplet H(9). The geminal coupling between protons at C(8) was determined by the same method which, however, resulted in increased errors.

Table 3
Selected structural and ^{1}H-NMR spectral data for **1**

Hydrogen atom	Chemical shift (ppm, δ)	Width of multiplet (observed/expected [a]) (Hz)	Number of constituents in multiplet (observed/expected) [b]
H(7a)	3.36	29.09/27.05	8/8
H(7s)	3.63	23.09/23.40	8/8
H(8a)	2.73	28.54/29.90	10/16
H(8s)	2.38	39.26/38.20	8/16
H(9)	3.51	34.00/33.52	10/16
CH$_3$(10)	1.30	–	2/2
Sequence of atoms	Angle (°) from X-ray	J_{H-H} expected [a] (Hz)	J_{H-H} found (Hz)
H(7a)–C(7)–H(7s)	109	13.5	12.63(58)
H(8a)–C(8)–H(8s)	108	15.0	13.10(19)
H(7a)–C(7)–C(8)–H(8a)	17	7.3	6.68(10)
H(7a)–C(7)–C(8)–H(8s)	135	6.7	9.86(9)
H(7s)–C(7)–C(8)–H(8a)	−104	1.6	2.73(16)
H(7s)–C(7)–C(8)–H(8s)	14	8.3	7.64(16)
H(8a)–C(8)–C(9)–H(9)	−28	6.0	5.83(20)
H(8s)–C(8)–C(9)–H(9)	−146	8.2	8.85(8)
H(9)–CH$_3$(10)	–	–	6.44(10)

Hydrogen atoms in positions *anti* and *syn* towards the CH$_3$ group are denoted as (a) and (s), respectively. Coupling constants ^{2}J and ^{3}J have been considered.

[a] According to correlation of Karplus [18].

[b] Discrepancies arise from the overlapping of signals.

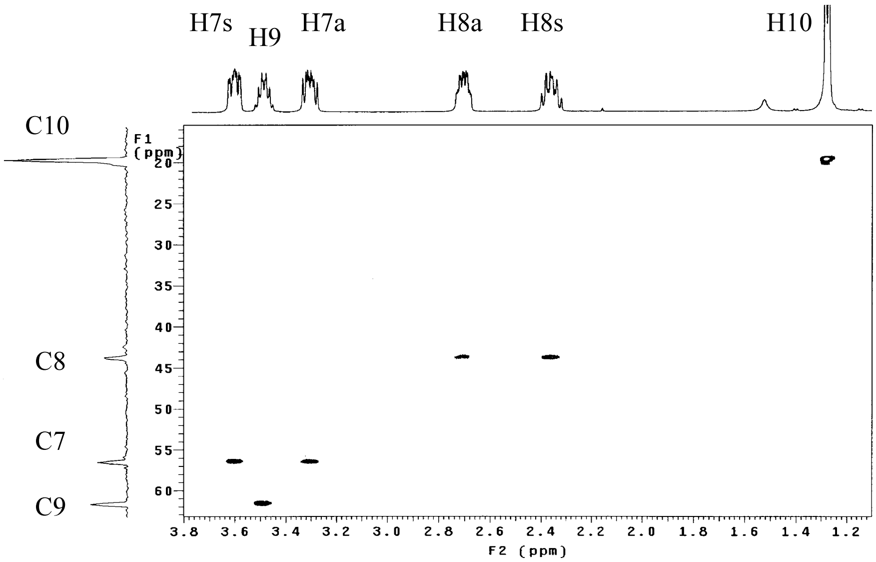

Fig. 7. 2-D heteronuclear ^{1}H–^{13}C spectrum of **1**.

3. Conclusions

The cyclization of acetaldazine to pyrazolidin-diyl ligand during complexation with dodecacarbonyl-triiron was proven by the X-ray structural analysis of the title $C_4H_8N_2Fe_2(CO)_6$ complex. The geometry around the nitrogen atoms can be interpreted in terms of an $N(sp^2)$ hybridization; however, deviations due to steric factors were observed caused by voluminous substituents or by distortions in stressed five-membered rings. A correlation between these deviations and the type of organic ligand was noted in a series of known N_2Fe_2 complexes containing a N–N bond.

A complete assignment of signals has been accomplished in a ^{1}H-NMR study of the title complex, in particular to protons in *anti* and *syn* positions relative to the methyl group, by a conformational analysis and determination of geminal and vicinal coupling constants. The values of valence and dihedral angles were known from X-ray diffraction analysis, and the Karplus relation was shown to be applicable. Hence, after assignments, the shapes of multiplets were simulated and the exact values of geminal and vicinal coupling constants have been determined from the experimental spectra. Since the Karplus correlation is effective for the crystal structure of the compound under study, differences in conformation between the crystalline and dissolved states of the molecule can be regarded as negligible.

4. Experimental

Abbreviations used: CSD, Cambridge Structural Database; X-ray, crystallographic structural analysis; r.t., room temperature; NMR, nuclear magnetic resonance; d-chloroform, deuterated chloroform; ppm, part per million; TMS, tetramethylsilane; J, coupling constant (^{2}J geminal; ^{3}J vicinal); d, doublet; t, triplet; q, quartet; av., average; S.D., standard deviation; rms, root mean square.

The complex under study, μ-1,2-(1,2-diaza-3-methylcyclopentane)diyl-bis(tricarbonyliron), denoted as **1**, was obtained according to Ref. [13]. 3,4-Diazahexa-2,4-diene (1 mmol) and triiron-dodecacarbonyl (3 mmol) were refluxed in isooctane under nitrogen, chromatographed on Kieselgel 60 and recrystallized from *n*-hexane. The deep-orange needles were fairly unstable at r.t. but could be stored at − 20 °C for several months, preferably under inert gas. The synthesis of 3,4-diazahexa-2,4-diene (acetaldazine) was accomplished according to Ref. [21].

The ^{1}H-NMR spectrum was recorded with a Varian Unity + 500 MHz instrument in d-chloroform. Chemical shifts are given in ppm, δ, measured from TMS; coupling constants in Hz.

4.1. X-ray structure determination

Single crystals of **1**, suitable for X-ray diffraction studies, were grown from *n*-hexane. X-ray data were collected on a four-circle P3 (Siemens AG) diffractometer at r.t. The selected crystallographic data, the parameters of data collections and refinement procedures are presented in Table 4.

The crystal class and the orientation matrix were obtained from the least-square refinement of the angular settings of 25 randomly found reflections in the 2θ range from 15 to 30°. Intensities were collected in the $\omega - 2\theta$ mode. Two check reflections measured every 70

Table 4
Crystal data and structure refinement for **1**

Empirical formula	$C_{10}H_8Fe_2N_2O_6$
Formula weight	363.87
Temperature (K)	293(2)
Wavelength (Å)	0.71073 (Mo–K_α)
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	
a (Å)	9.039(3)
b (Å)	12.582(4)
c (Å)	12.993(4)
β (°)	105.60(2)
V (Å^3)	1423.1(7)
Z	4
D_{calc} (Mg m^{-3})	1.698
Absorption coefficient (mm^{-1})	2.061
F(000)	728
Crystal size (mm)	0.48 × 0.36 × 0.32
Theta range for data collection (°)	2.1–25.0
Index ranges	$-10 \leq h \leq 10$, $0 \leq k \leq 14$, $0 \leq l \leq 15$
Reflections collected	2633
Independent reflections	2515 [$R_{int} = 0.0638$]
Completeness to $\theta = 25.0°$	99.7%
Absorption correction	Gaussian, from crystal shape
Max/min transmission	0.5359, 0.3686
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2515/0/181
Goodness-of-fit on F^2	1.024
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0415$, $wR_2 = 0.1107$
R indices (all data)	$R_1 = 0.0549$, $wR_2 = 0.1185$
Largest difference peak and hole (e Å^{-3})	+0.72 and −0.45

reflections, indicated appreciable (13.6%) decomposition of the crystal during data collection. A total of 2633 measured intensities were corrected for Lorentz-polarization factors and the crystal decay. An analytical absorption correction (Gaussian integration) was applied on the basis of the well defined crystals shape.

The structure was solved in $P2_1/c$ space group by direct methods using the SHELXS-86 program [22]. Full-matrix least-squares refinement method against F^2 values was carried out by using the SHELXL-97 program [23]. All non-hydrogen atoms were refined with anisotropic displacement parameters. The H atoms were included in calculated positions with isotropic U equal to 1.5 times the equivalent isotropic displacement parameter of the parent carbon atom. The C(8) and C(10) atoms showed large anisotropic displacement parameters and electron density features close to them which imply some kind of disorder but no satisfactory multisite model was derived. The final weighting scheme was, $w = [\sigma^2(F_o^2) + (0.070P)^2 + 0.82P]^{-1}$, where $P = 1/3(F_o^2 + 2F_c^2)$.

5. Supplementary material

Crystallographic data for the structural analysis of **1** have been deposited with the Cambridge Crystallographic Data Center, CCDC no. 169446. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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