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Neutral and Cationic Paramagnetic Amino-
amidinate Fe(II) Complexes; ^{19}F NMR Evidence
for Interactions with Weakly Coordinating Anions

Timo J.J. Sciarone, Christian A. Nijhuis, Auke Meetsma, Bart Hessen*

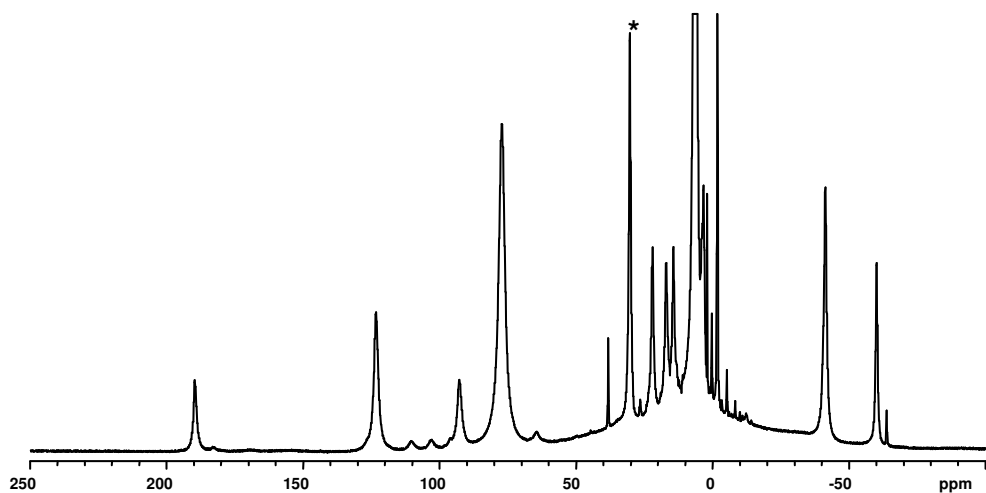


Figure S1 ^1H NMR (500 MHz, C_6D_6 , RT) spectrum of **4**. Asterisk indicates $\text{C}_6\text{D}_5\text{H}$

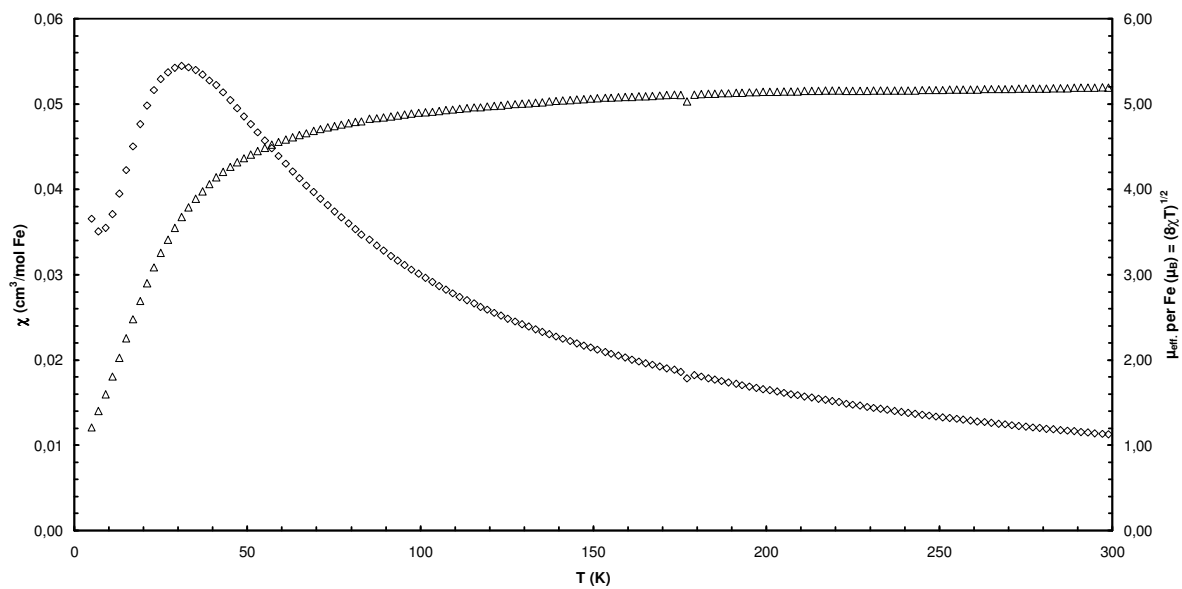


Figure S2 Temperature dependence of magnetic susceptibility and μ_{eff} for **1a**.

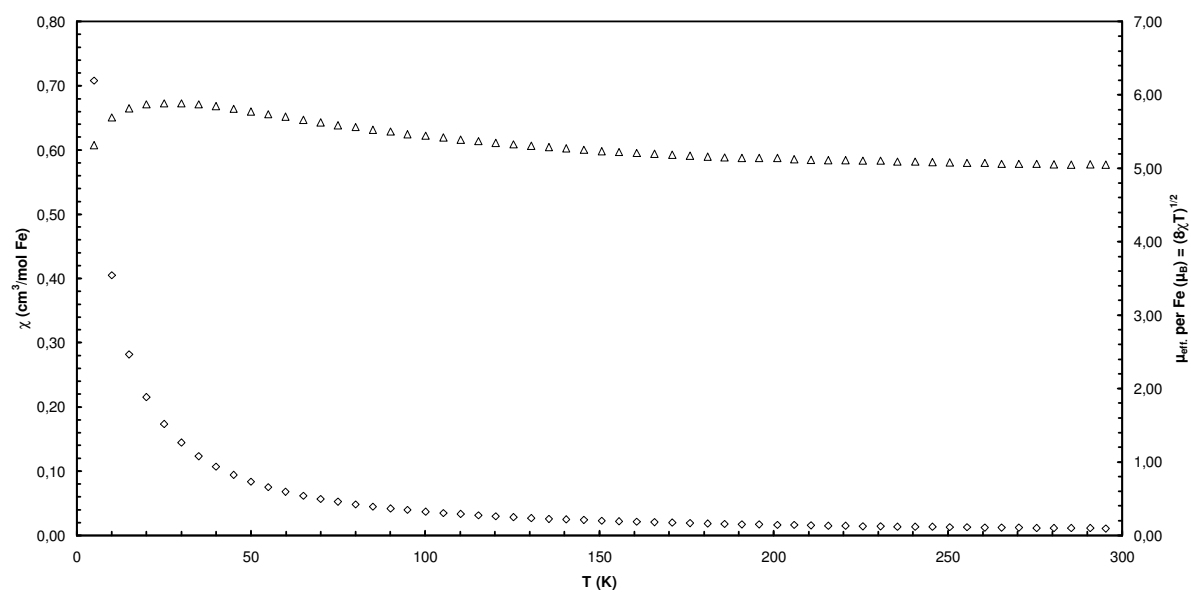


Figure S3 Temperature dependence of magnetic susceptibility and μ_{eff} for **1b**.

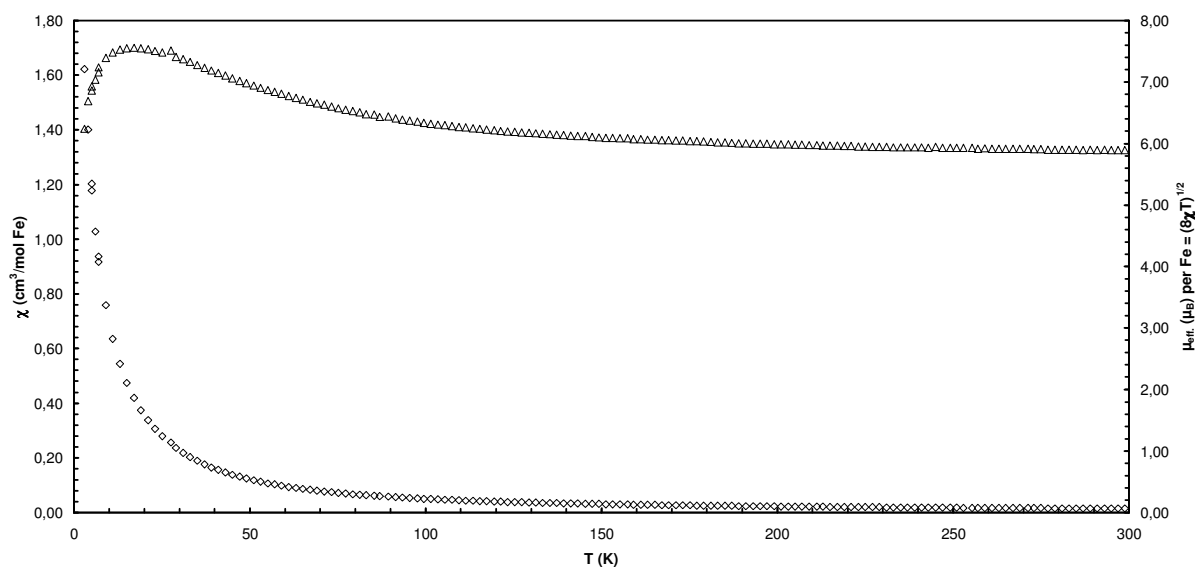


Figure S4 Temperature dependence of magnetic susceptibility and μ_{eff} for **4**.

Table S1 Crystal, collection and refinement data for complexes **1a**, **2** and **3**.

	1a	2	3
formula	C ₄₆ H ₆₄ N ₆ Fe ₂ Cl ₂	C ₄₆ H ₆₄ N ₆ Fe	C ₂₆ H ₃₂ N ₃ O ₃ FeCl· 2C ₄ H ₈ O
Fw	883.66	756.90	670.07
cryst. dim. (mm)	0.12 x 0.25 x 0.51	0.44 x 0.19 x 0.17	0.20 x 0.15 x 0.05
colour, habit	yellow, block	yellow, prism	yellow, plate
crystal system	Monoclinic	Orthorhombic	Monoclinic
space group, no. ⁴⁵	<i>P2₁/c</i> , 14	<i>Pbca</i> , 61	<i>P2₁/n</i> , 14
<i>A</i> (Å)	14.889(2)	17.6967(8)	9.4467(8)
<i>B</i> (Å)	9.889(1)	18.5086(9)	20.014(2)
<i>C</i> (Å)	16.661(1)	25.923(1)	18.161(2)
<i>A</i> (°)	90	90	90
<i>B</i> (°)	116.03(1)	90	96.305(1)
<i>Γ</i> (°)	90	90	90
<i>Z</i>	2	8	4
<i>V</i> (Å ³)	2204.3(4)	8490.9(7)	3412.9(6)
ρ_{calc} (g/cm ³)	1.331	1.184	1.304
Diffractometer	Enraf Nonius CAD-4F	Bruker Smart CCD	APEX Bruker Smart CCD
Θ range (°)	1.35 – 27.0	2.24 – 27.31	2.33 – 23.74
λ (Å)	0.71073 (Mo K α)	0.71073 (Mo K α)	0.71073 (Mo K α)
scan mode	φ - and ω -scans		
<i>T</i> (K)	180	100(1)	105(1)
data collect. time (h)	84.3	8.3	27.0
No. of meas. refl.	5266	71048	31758
No. of unique refl.	4802	9731	9004
<i>M</i> (cm ⁻¹)	8.2	3.94	5.64
No. of parameters	381	734	416
No. of restraints			
weighting scheme: a,b ^[a]	0.1087, 0.2692	0.0498, 4.7606	0.0945, 0.0
<i>R</i> (<i>F</i>) for <i>F</i> ₀ ≥ 4σ(<i>F</i> ₀) ^[b]	0.0482	0.0429	0.0628
<i>wR</i> (<i>F</i> ²) ^[c]	0.1443	0.1094	0.1737
res. el. dens. (e/Å ³)	-0.90, 0.89(11)	-0.46, 0.45(6)	-0.47, 0.8(8)
GoF ^[d]	1.040	1.036	0.978

^[a] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2, 0) + 2F_c^2] / 3$

^[b] $R(F) = \sum (||F_o| - |F_c||) / \sum |F_o|$,

^[c] $wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$,

^[d] $GoF = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$, $n = \# \text{ refl.}$, $p = \# \text{ param. refined.}$

^[e] The conventional space group is *C2/c*.

Transformation: $\begin{pmatrix} a' \\ b' \\ c' \end{pmatrix}_{C2/c} = \begin{pmatrix} -1 & 0 & -1 \\ 0 & 1 & 0 \\ 1 & 0 & 0 \end{pmatrix} \begin{pmatrix} a \\ b \\ c \end{pmatrix}_{I2/a}$ $a' = 29.221$, $b' = 8.132$, $c' = 18.514$ Å, $\beta' = 127.72^\circ$

Table S2 Crystal, collection and refinement data for complexes **1b** and **4**.

	1b	4
formula	C ₂₈ H ₄₈ N ₆ Fe ₂ Cl ₂ Si ₂	C ₄₂ H ₆₂ N ₆ Fe ₂ Si ₂
Fw	707.50	818.86
cryst. dim. (mm)	0.10 x 0.20 x 0.24	0.22 x 0.19 x 0.17
colour, habit	orange, plate	red, parallelepiped
crystal system	Monoclinic	Monoclinic
Space group, no. ⁴⁵	<i>I</i> 2/a ^[e] , 15	<i>C</i> 2/c, 15
<i>a</i> (Å)	18.514(1)	14.9022(9)
<i>b</i> (Å)	8.132(1)	13.1648(8)
<i>c</i> (Å)	23.124(6)	22.578(1)
α (°)	90	90
β (°)	91.58(1)	103.166(1)
γ (°)	90	90
<i>Z</i>	4	4
<i>V</i> (Å ³)	3460.1(10)	4313.0(4)
ρ_{calc} (g/cm ³)	1.350	1.261
Diffractometer	Enraf Nonius CAD-4F	Bruker Smart APEX CCD
θ range (°)	1.39 – 27.0	2.41 – 23.13
λ (Å)	0.71073 (Mo K α)	0.71073 (Mo K α)
scan mode	ϕ - and ω -scans	
<i>T</i> (K)	130	125(1)
data collect. time (h)	127.3	11.9
no. of meas. refl.	7598	20647
no. of unique refl.	3751	5349
μ (cm ⁻¹)	10.83	7.64
no. of parameters	265	288
no. of restraints	6	2
weighting scheme: a,b ^[a]	0.0252, 13.57	0.0486, 2.6612
<i>R</i> (<i>F</i>) for <i>F</i> ₀ ≥ 4 σ (<i>F</i> ₀) ^[b]	0.0514	0.0483
<i>wR</i> (<i>F</i> ²) ^[c]	0.1128	0.1163
res. el. dens. (e/Å ³)	-0.84, 0.81(7)	-0.45, 0.46(7)
GoF ^[d]	1.069	1.017

^[a] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2, 0) + 2F_c^2] / 3$

^[b] $R(F) = \sum (||F_o| - |F_c||) / \sum |F_o|$,

^[c] $wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$,

^[d] $GoF = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$, $n = \# \text{ refl.}$, $p = \# \text{ param. refined.}$

^[e] The conventional space group is *C*2/*c*.

Transformation: $\begin{pmatrix} a' \\ b' \end{pmatrix} = \begin{pmatrix} -1 & 0 & -1 \\ 0 & 1 & 0 \end{pmatrix} \begin{pmatrix} a \\ b \end{pmatrix}$

(*c'*) *C*2/*c* (1 0 0) (*c*) *I*2/a $a' = 29.221$, $b' = 8.132$, $c' = 18.514$ Å, $\beta' = 127.72^\circ$