Electronic Supplementary Material (ESI) for New Journal of Chemistry

Synthesis, structural characterisation and electrochemistry of a ferrocenyl-substituted 4-quinazolinone and some related heterocycles

Jiří Tauchman, Kateřina Hladíková, Filip Uhlík,
Ivana Císařová and Petr Štěpnička*

Supporting Information

Contents

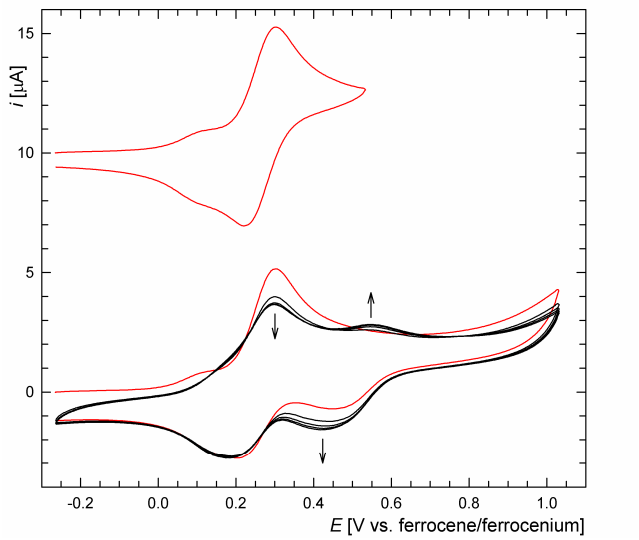

Figure S1. Cyclic voltammograms of compound **3** recorded with the switching potential set close after the first wave (top) and to a more positive potential (bottom). The first scans are shown in red and the arrows indicate trends observed upon repeated cycling. Conditions: Pt disc electrode (2 mm diameter), 0.5 mM solution in 1,2-dichloroethane with 0.1 M $Bu_4N[PF_6]$ supporting electrolyte, scan rate: 0.2 V s^{-1} in both cases.

Table S1. Comparison of bond distances and angles determined from single-crystal X-ray diffraction data and by DFT calculations for **2**.

Parameter		X-ray data	DFT gas phase	DFT crystal
Fe	-C1	2.0301(13)	2.0509	2.0264
Fe	-C2	2.0390(16)	2.0472	2.0254
Fe	-C3	2.0462(17)	2.0588	2.0286
Fe	-C4	2.0455(14)	2.0556	2.0199
Fe	-C5	2.0368(15)	2.0437	2.0133
Fe	-C6	2.0357(17)	2.0525	2.0350
Fe	-C7	2.0417(17)	2.0524	2.0314
Fe	-C8	2.0447(17)	2.0548	2.0328
Fe	-C9	2.047(2)	2.0575	2.0425
Fe	-C10	2.034(2)	2.0550	2.0419
O	-C14	1.238(2)	1.2238	1.2540
N11	-C12	1.2983(18)	1.3010	1.3203
N11	-C16	1.3875(17)	1.3852	1.3852
N13	-C12	1.3819(19)	1.3808	1.3882
N13	-C14	1.3714(18)	1.4017	1.3911
C1	-C5	1.436(2)	1.4391	1.4516
C1	-C12	1.4638(18)	1.4676	1.4672
C1	-C2	1.430(2)	1.4348	1.4474
C2	-C3	1.423(2)	1.4217	1.4330
C3	-C4	1.420(3)	1.4275	1.4387
C4	-C5	1.425(2)	1.4246	1.4374
C6	-C7	1.411(2)	1.4275	1.4350
C6	-C10	1.423(4)	1.4273	1.4366
C7	-C8	1.404(3)	1.4276	1.4366
C8	-C9	1.405(3)	1.4276	1.4363
C9	-C10	1.410(3)	1.4269	1.4349
C14	-C15	1.452(2)	1.4653	1.4613
C15	-C17	1.4029(19)	1.4031	1.4107
C15	-C16	1.406(2)	1.4176	1.4287
C16	-C20	1.405(2)	1.4102	1.4198
C17	-C18	1.375(2)	1.3863	1.3936
C18	-C19	1.403(2)	1.4070	1.4143
C19	-C20	1.378(2)	1.3861	1.3948

(Table S1 continued)

C1	-Fe	-C2	41.15(6)	40.99	41.86
C1	-Fe	-C3	68.87(6)	68.52	70.26
C1	-Fe	-C4	68.90(6)	68.70	70.51
C1	-Fe	-C5	41.36(6)	41.15	42.12
C1	-Fe	-C6	118.67(6)	118.94	117.44
C1	-Fe	-C7	151.86(7)	152.88	150.87
C1	-Fe	-C8	166.86(7)	165.24	166.94
C1	-Fe	-C9	129.19(7)	127.56	128.58
C1	-Fe	-C10	109.09(6)	108.17	108.00
C2	-Fe	-C3	40.77(6)	40.51	41.40
C2	-Fe	-C4	68.68(6)	68.50	70.22
C2	-Fe	-C5	69.36(6)	68.97	70.71
C2	-Fe	-C6	108.49(8)	107.35	106.46
C2	-Fe	-C7	117.74(7)	118.26	115.66
C2	-Fe	-C8	150.65(7)	152.46	149.63
C2	-Fe	-C9	168.05(7)	165.31	167.60
C2	-Fe	-C10	129.86(7)	127.20	128.38
C3	-Fe	-C4	40.61(7)	40.60	41.63
C3	-Fe	-C5	68.89(7)	68.51	70.41
C3	-Fe	-C6	128.45(9)	126.49	126.46
C3	-Fe	-C7	107.54(7)	107.23	104.98
C3	-Fe	-C8	117.19(7)	118.72	115.42
C3	-Fe	-C9	150.13(8)	153.22	150.33
C3	-Fe	-C10	167.67(9)	164.50	166.14
C4	-Fe	-C5	40.87(6)	40.67	41.76
C4	-Fe	-C6	165.96(8)	164.12	164.91
C4	-Fe	-C7	127.53(7)	126.51	125.79
C4	-Fe	-C8	107.47(7)	107.69	105.40
Fe	-C2	-C1	69.10(9)	69.64	69.11
C1	-C2	-C3	107.81(14)	108.18	108.21
Fe	-C3	-C4	69.67(10)	69.58	68.86
Fe	-C3	-C2	69.34(9)	69.30	69.18
C2	-C3	-C4	108.28(14)	108.28	108.24
Fe	-C4	-C5	69.24(8)	69.22	68.87
C3	-C4	-C5	108.52(13)	108.11	108.23
Fe	-C4	-C3	69.72(9)	69.82	69.51
C1	-C5	-C4	107.36(14)	108.02	107.89
Fe	-C5	-C1	69.07(8)	69.69	69.43

(Table S1 continued)

Fe	-C5	-C4	69.89(9)	70.11	69.37
C7	-C6	-C10	107.38(18)	107.91	107.93
Fe	-C6	-C10	69.48(12)	69.76	69.63
Fe	-C6	-C7	69.98(10)	69.64	69.20
Fe	-C7	-C8	70.02(10)	69.75	69.35
C6	-C7	-C8	107.93(18)	108.05	107.96
Fe	-C7	-C6	69.52(10)	69.65	69.47
C7	-C8	-C9	108.99(16)	107.97	108.11
Fe	-C8	-C7	69.78(10)	69.57	69.25
Fe	-C8	-C9	70.01(11)	69.79	69.73
C8	-C9	-C10	107.4(2)	107.97	107.83
Fe	-C9	-C8	69.84(13)	69.58	69.00
Fe	-C9	-C10	69.30(14)	69.60	69.41
Fe	-C10	-C6	69.60(13)	69.57	69.11
Fe	-C10	-C9	70.29(13)	69.79	69.45
C6	-C10	-C9	108.28(19)	108.10	108.17
N11	-C12	-N13	123.34(12)	122.65	122.81
N13	-C12	-C1	117.51(12)	117.27	117.94
C4	-Fe	-C9	117.19(8)	119.43	116.72
C4	-Fe	-C10	151.12(9)	153.82	151.79
C5	-Fe	-C6	152.37(7)	153.78	152.34
C5	-Fe	-C7	165.54(6)	164.37	165.23
C5	-Fe	-C8	127.82(7)	126.95	126.86
C5	-Fe	-C9	107.68(8)	108.22	107.07
C5	-Fe	-C10	118.19(8)	119.69	118.08
C6	-Fe	-C7	40.50(7)	40.70	41.33
C6	-Fe	-C8	67.83(7)	68.47	69.64
C6	-Fe	-C9	68.43(10)	68.41	69.55
C6	-Fe	-C10	40.93(10)	40.67	41.26
C7	-Fe	-C8	40.20(8)	40.68	41.40
C7	-Fe	-C9	68.00(8)	68.38	69.63
C7	-Fe	-C10	68.16(8)	68.38	69.51
C8	-Fe	-C9	40.15(8)	40.63	41.27
C8	-Fe	-C10	67.59(7)	68.36	69.42
C9	-Fe	-C10	40.42(9)	40.60	41.13
C12	-N11	-C16	117.09(13)	117.77	117.70
C12	-N13	-C14	123.48(12)	124.95	123.78
Fe	-C1	-C12	124.14(9)	126.37	126.09

(Table S1 continued)

Fe	-C1	-C2	69.76(8)	69.37	69.03
Fe	-C1	-C5	69.57(8)	69.16	68.46
C5	-C1	-C12	127.72(14)	128.37	128.73
C2	-C1	-C12	124.20(13)	124.22	123.80
C2	-C1	-C5	108.02(12)	107.40	107.43
Fe	-C2	-C3	69.89(9)	70.18	69.42
N11	-C12	-C1	119.15(13)	120.07	119.24
O	-C14	-C15	124.41(13)	126.88	124.91
N13	-C14	-C15	114.67(13)	112.57	114.44
O	-C14	-N13	120.87(13)	120.55	120.63
C16	-C15	-C17	120.54(13)	120.67	120.55
C14	-C15	-C16	118.31(12)	119.05	118.43
C14	-C15	-C17	121.13(14)	120.28	121.00
C15	-C16	-C20	118.69(12)	118.43	118.18
N11	-C16	-C15	122.98(13)	123.01	122.78
N11	-C16	-C20	118.33(14)	118.56	119.04
C15	-C17	-C18	119.95(15)	119.99	120.37
C17	-C18	-C19	119.77(14)	119.75	119.50
C18	-C19	-C20	120.91(15)	120.82	120.88
C16	-C20	-C19	120.10(15)	120.34	120.52

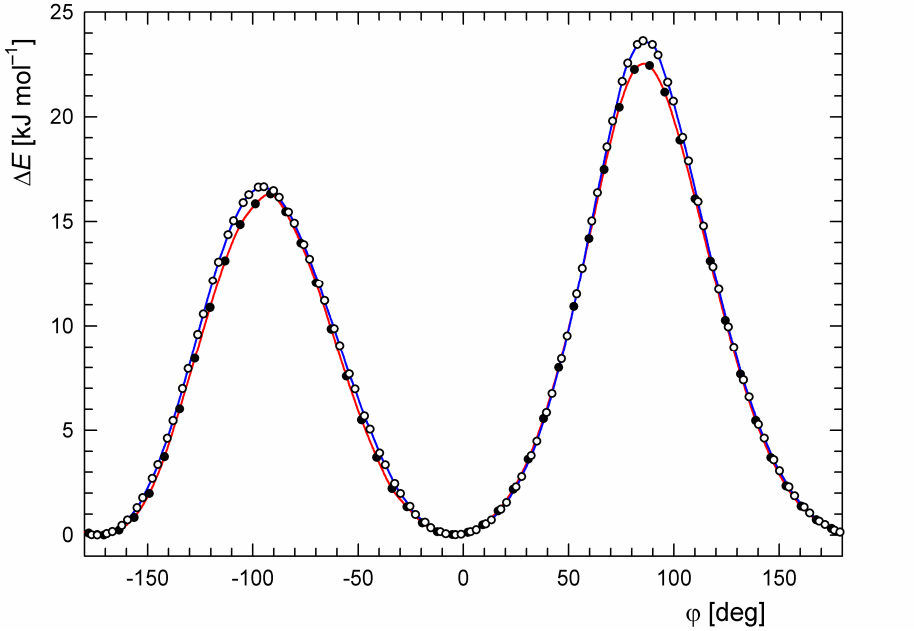

Figure S2. The dependence of the relative potential energy on the torsion angle N11-C12-C1-C2 (φ) as calculated by two different DFT methods: B3LYP/6-31G* – filled circles and red line, PBE/6-31G* – empty symbols and blue line.

Table S2. Selected crystallographic data and structure refinement parameters for **1-7** and **9**.[a]

Compound	**1**	**2**	**3**	**4**
Formula	$C_{18}H_{16}FeN_2O_2{\cdot}½H_2O$	$C_{18}H_{14}FeN_2O$	$C_{18}H_{14}FeN_2S$	$C_{18}H_{13}FeNO_2$
M	357.19	330.16	346.22	331.14
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic
Space group	*C*2/*c* (no. 15)	$P2_1/c$ (no. 14)	$P2_1/c$ (no. 14)	$P2_1/c$ (no. 14)
a/Å	12.0028(4)	13.2738(4)	14.1216(4)	10.3361(3)
b/Å	13.0095(5)	8.4806(2)	8.5142(2)	7.9197(2)
c/Å	20.3429(7)	14.0205(4)	13.7245(4)	17.1157(5)
α/°				
β/°	99.216(1)	114.859(1)	115.626(1)	98.984(1)
γ/°				
V/Å^3	3135.6(2)	1432.05(7)	1487.84(7)	1383.88(7)
Z	8	4	4	4
D_c/g mL^{-1}	1.513	1.531	1.550	1.594
μ(Mo Kα)/mm^{-1}	0.977	1.055	1.150	1.096
Diffractions collected	26062	23072	24353	23033
Independent/obsd[b] diffrns	3619/3045	3275/2992	3412/3171	3187/2855
R_{int}[c]/%	2.64	1.77	1.56	2.23
R[c] observed diffrns/%	2.85	2.50	2.13	2.40
R, wR[c] all data/%	3.74, 8.33	2.84, 6.79	2.38, 5.77	2.87, 6.24
Δρ/e Å^{-3}	0.34, –0.71	0.39, –0.36	0.36, –0.31	0.32,–0.29
CCDC reference no.	922133	922134	922135	922136

Table S3 (continued)

Compound	**5**	**6**[d]	**7**	**9**
Formula	$C_{18}H_{13}FeNS_2$	$C_{18}H_{13}FeNOS$	$C_{18}H_{17}FeN_3O_2$	$C_{23}H_{25}FeN_5O$
M	363.23	347.20	363.20	459.33
Crystal system	triclinic	monoclinic	monoclinic	monoclinic
Space group	*P*–1 (no. 2)	$P2_1/n$ (no. 14)	*C*2/*c* (no. 15)	$P2_1/c$ (no. 14)
a/Å	7.3774(3)	13.1577(14)	16.2896(4)	11.9743(3)
b/Å	9.9499(4)	8.6907(9)	12.4546(3)	12.3974(3)
c/Å	10.5660(4)	13.8343(16)	16.4543(4)	14.2253(3)
α/°	87.451(1)			
β/°	79.770(1)	114.783(3)	111.719(1)	92.713(1)
γ/°	74.670(1)			
V/Å^3	736.10(5)	1436.3(3)	3101.3(1)	2109.38(9)
Z	2	4	8	4
D_c/g mL^{-1}	1.639	1.606	1.556	1.446
μ(Mo Kα)/mm^{-1}	1.301	1.195	0.997	0.746
Diffractions collected	9376	12897	25230	16290
Independent/obsd[b] diffrns	3375/2944	3127/2630	3570/3316	4844/4113
R_{int}[c]/%	2.64	6.32	1.73	1.86
R[c] observed diffrns/%	2.94	4.61	2.24	2.87
R, wR[c] all data/%	3.63, 7.97	5.60, 12.98	2.48, 6.12	3.74, 7.74
Δρ/e Å^{-3}	0.53, –0.34	0.92, –1.18	0.34, –0.29	0.29,–0.30
CCDC reference no.	922137	922138	922139	922140

[a] Common details: T = 150(2) K. [b] $I > 2\sigma(I)$. [c] Definitions: $R_{int} = \Sigma \left| F_o^2 - F_o^2(\text{mean}) \right| / \Sigma F_o^2$, where F_o^2(mean) is the average intensity of symmetry-equivalent diffractions. $R = \Sigma \left| \left| F_o \right| - \left| F_c \right| \right| / \Sigma \left| F_o \right|$, $wR = [\Sigma\{w(F_o^2 - F_c^2)^2\}/\Sigma\, w(F_o^2)^2]^{1/2}$. [d] This compound tends to form crystalline aggregates. A lower quality of the available crystals (defects and possible contributions from minor domains) was manifested by a relatively lower quality of the diffraction data.