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2-Pyridylmetallocenes: Part 3 [1]. Cyclomercuration of 2-pyridyl-ferrocene and 1-(2-pyridyl)-2-methyl-ferrocene. Molecular structures of $[1\text{-}(2\text{-}C_5H_4N)\text{-}2\text{-}(CH_3)C_5H_3]Fe(C_5H_5)$, $[1\text{-}(ClHg)\text{-}2\text{-}(2\text{-}C_5H_4N)C_5H_3]Fe(C_5H_5)$ and $[1\text{-}(ClHg)\text{-}2\text{-}(2\text{-}C_5H_4N)\text{-}3\text{-}(CH_3)C_5H_2]Fe(C_5H_5)$

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Mercuration of 2-pyridylferrocene $[(2\text{-}C_5H_4N)C_5H_4]Fe(C_5H_5)$ (**1a**) and its 2-methylated derivative **1b** with $Hg(OAc)_2$ followed by treatment with LiCl gave after chromatographic workup the cyclomercurated and $[1\text{-}(ClHg)\text{-}2\text{-}(2\text{-}C_5H_4N)\text{-}3\text{-}(R)C_5H_2]Fe(C_5H_5)$ (R = H: **2a**; Me: **2b**)) in 43% and 21% yield. The molecular structures of **1b**, **2a** and **2b** have been determined by X-ray crystallography.

1. Introduction

The mercuration of ferrocene to give chloromercuriferrocene was reported by Nesmeyanov et al. already in 1954 [2]. Later on several substituted ferrocenes were also successfully mercurated. The so-called "cyclomercuration" or "ortho-mercuration" of cyclopentadienyl complexes bearing nitrogen containing substituents was studied intensively by the group of Y.J. Wu and recently by Attar et al. [3]. The mechanism of the mercuration of ferrocene was studied by A.F. Cunningham [4]. He could show that primary attack of mercury occurs at the iron atom, followed by an endo attack on the coordinated cyclopentadienyl ring. Eventually, oxidation of iron to give a ferricenium species might happen as well, as we could show earlier for the mercuration with $HgCl_2$ [5]. With a nitrogen containing substituent on the cyclopentadienyl ring, alternatively coordination of the nitrogen lone pair might happen first [6].

We recently reported the cyclomercuration of tricarbonylcyclopentadienylrhenium [1] and observed a rather short Hg–N interaction, while the C–Hg–Cl bond still remained linear. Here we present our results with the mercuration of 2-pyridyl-ferrocene and its 2-methylated derivative.

2. Experimental

All reactions were performed under an atmosphere of argon using standard Schlenk-techniques. The solvents used for the lithiation reactions (THF and Et_2O) were obtained from Aldrich in the highest available quality and used without further purification. The other solvents were of standard quality. The reagents *n*-BuLi (2.5 m in hexane); 2-bromopyridine; $Hg(OAc)_2$ and MeI were obtained from Aldrich and used without further purification. 2-Pyridylferrocene (**1a**) was prepared as recently reported by us [7].

NMR: JEOL ECP-270 and ECX-400, Ref. $CHCl_3$ $\delta(^1H) = 7.240$ ppm, $\delta(^{13}C) = 77.0$ ppm; Ref. $CHDCl_2$ $\delta(^1H) = 5.300$ ppm, $\delta(^{13}C) = 53.8$ ppm. The numbering scheme for the assignment of NMR spectra is shown in Scheme 1.

MS: Finnigan MAT 90 and JEOL Mstation 700.

The elemental analysis of **1b** was performed at the microanalytical laboratory at the chemistry department of Ludwig-Maximilians University, Munich. Due to local restrictions, combustion analyses of mercury containing compounds could not be performed.

2.1. Synthesis of 2-methyl-1-(2-pyridyl)-ferrocene (**1b**)

A solution of **1a** (0.24 g, 0.91 mmol) in Et_2O (10 mL) was treated with *n*-BuLi solution (1.0 mL, 2.5 mmol) with stirring for 45 min. Then the mixture was cooled to −78 °C and MeI (0.14 mL, 2.2 mmol) was added. Stirring was continued for 30 min at this temperature and then for 2 h at room temperature. Evaporation

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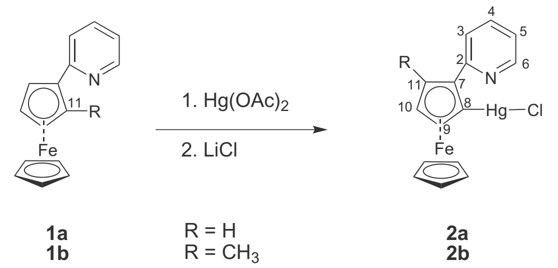


Scheme 1. Synthesis of **2a** and **2b** and numbering scheme for NMR spectra.

of the solvent in vacuo left a red oily residue, which was chromatographed on alumina using CH_2Cl_2 as eluent. After removal of the solvent a red sticky oil remained. Yield: 0.20 g (79%). EA (calcd/found): C: 69.34/68.26; H: 5.46/5.82, N: 5.05/4.65%. ^1H NMR (270 MHz, $CDCl_3$): δ = 8.52 ("ddd", 1H, H6), 7.58 ("dt", 1H, H4) 7.53 ("td", 1H, H3), 7.07 ("ddd", 1H, H5), 4.67 ("dd", 1H, H8), 4.25 ("dd", 1H, H10), 4.19 ("t", 1H, H9), 4.10 (s, 5H, C_5H_5), 2.35 (s, 3H, CH_3). ^{13}C NMR (67.9 MHz, $CDCl_3$): δ = 160.0 (C2), 148.9 (C6), 135.5 (C4), 122.2 (C5), 120.3 (C3), 83.6 (C7), 83.3 (C11), 72.0 (C10), 68.9 (C9), 67.0 (C8), 70.2 (C_5H_5), 15.4 (CH_3). MS (DEI$^+$): m/z = 277.1 (M$^+$, 100%), 211.1 (M$^+$−C_5H_6, 31%), 155.1 (M$^+$−C_5H_6–Fe, 9%).

2.2. Synthesis of chlorido-[2-(2-pyridyl)-ferrocenyl-κC]-mercury (*2a*) and chlorido-[3-methyl-2-(2-pyridyl)-ferrocenyl-κC]-mercury (*2b*)

A solution of the ferrocenylpyridine (**1a**: 0.26 g, 0.99 mmol/**1b:** 0.13 g, 0.47 mmol) in CH_2Cl_2 (20 mL) was treated with a solution of $Hg(OAc)_2$ (0.32 g/0.16 g, 1.00 mmol/0.50 mmol, respectively) in MeOH (40 mL/19 mL). After stirring for 90 min a solution of LiCl (0.070 g, 1.7 mmol/0.040 g, 0.94 mmol) in MeOH (30 mL/14 mL) was added and stirring was continued for 16 h/3 days. Then a mixture of CH_2Cl_2 (40 mL/15 mL) and water (60 mL/20 mL) was added with vigorous stirring. The aqueous phase was separated and extracted with CH_2Cl_2 (80 mL/40 mL). The combined organic phases were stirred with $MgSO_4$, filtered and evaporated to dryness. The remaining orange residue was chromatographed on alumina. Elution with hexane/CH_2Cl_2 (2:1/1:2) yielded the desired products after evaporation to dryness as orange solids. Yields: 0.21 g (43%) **2a**, 0.050 g (21%) **2b**. Recrystallization from CH_2Cl_2 by slow evaporation at room temperature gave crystals suitable for X-ray structure analysis.

2a: ^1H NMR (270 MHz, CD_2Cl_2): δ = 8.40 ("dd", 1H, H6), 7.62 ("dt", 1H, H4), 7.40 ("d" br, 1H, H3), 7.15 ("ddd", 1H, H5), 5.00 (br s, 1H, H11), 4.61 (br, s, J_{H-Hg} = 36 Hz, 1H, H10), 4.39 (br s, J_{H-Hg} = 54 Hz, 1H, H9), 4.06 (s, 5H, Cp). ^{13}C NMR (68 MHz, CD_2Cl_2): δ = 158.4 (C2), 148.6 (C6), 137.3 (C4), 121.6 (C5), 120.1 (C3), 86.8 (J_{C-Hg} = 403 Hz, C7), 83.6 (C8), 75.8 (J_{C-Hg} = 261 Hz, C10), 73.4 (J_{C-Hg} = 228 Hz, C9), 70.4 (Cp), 67.9 (C11). MS (DEI$^+$): m/z = 726.2 (L_2Hg^+), (L ≡ Fe(Cp)($C_5H_3C_5H_4N$)), 499.1 (M$^+$), 261.1 (M$^+$−HgCl).

2b: ^1H NMR (270 MHz, CD_2Cl_2): δ = 8.47 ("td", 1H, H6), 7.63 (m, 2H, H3 + 4), 7.18 ("ddd", 1H, H5), 4.47 (m, J_{H-Hg} = 42 Hz, 1H, H10), 4.20 (m, J_{H-Hg} = 31 Hz, 1H, H9), 3.99 (s, 5H, Cp), 2.39 (s, 3H, CH_3). ^{13}C NMR (68 MHz, CD_2Cl_2): δ = 159.4 (C2), 148.80 (C6), 137.2 (C4), 121.4 (C5), 121.2 (C3), 85.7 (C7), 84.0 (C8 + C11), 76.9 (C10), 73.8 (C9), 71.0 (Cp), 17.2 (CH_3). MS (DEI$^+$): m/z = 513.3 (M$^+$), 276.2 (M$^+$–HgCl, 100%).

2.3. Crystal structure determinations

Suitable single crystals of **1b** and **2a** were mounted on the tip of a glass fiber, and investigated on a Kappa CCD diffractometer, using Mo Kα radiation (λ = 0.71073 Å). Crystals of **2b** were measured on an Oxford XCalibur diffractometer. Details of data collections and

Table 1
Experimental details of the crystal structure determinations.

Compound	**1b**	**2a**	**2b**
Empirical formula	$C_{16}H_{15}FeN$	$C_{15}H_{12}ClFeHgN$	$C_{16}H_{14}ClFeHg$ N
Formula weight	277.14	498.15	512.17
Crystal system	monoclinic		
Space group	$P2_1/c$		
Unit cell dimensions			
a (Å)	16.3585(10)	11.3288(2)	8.9743(9)
b (Å)	8.7964(5)	10.3721(3)	17.4340(17)
c (Å)	17.6471(11)	12.8948(3)	9.9739(8)
ß (°)	103.170(6)	112.4589(15)	107.898(9)
Volume (Å^3)	2472.6(3)	1400.26(6)	1485.0(2)
Z	4 × 2	4	4
D_{calc} (Mg/m^3)	1.489	2.363	2.291
Absorption coefficient (mm^{-1})	1.198	12.160	11.470
F(000)	1152	928	960
Crystal size (mm^3)	0.24 × 0.17 × 0.1	0.151 × 0.087 × 0.068	0.16 × 0.14 × 0.04
Theta range for data collection	4.19° to 26.36°	3.22° to 27.47°	4.22° to 26.35°
Index ranges	$-20 \leqslant h \leqslant 15$, $-10 \leqslant k \leqslant 10$, $-22 \leqslant l \leqslant 19$	$-14 \leqslant h \leqslant 14$, $-13 \leqslant k \leqslant 13$, $-16 \leqslant l \leqslant 16$	$-11 \leqslant h \leqslant 10$, $-19 \leqslant k \leqslant 21$, $-9 \leqslant l \leqslant 12$
Reflections collected	10042	22472	5997
Independent reflections	4993 [R_{int} = 0.0501]	3207 [R_{int} = 0.0516	3012 R_{int} = 0.0628
Completeness to theta (max)°	99.0%	99.8%	99.6%
Absorption correction	Semi-empirical from equivalents		
Maximum and minimum transmission	0.8871 and 0.8652	0.3595 and 0.2355	0.6320 and 0.1357
Data/restraints/parameters	4993/0/327	3207/0/220	3012/0/183
Goodness-of-fit on F^2	0.820	1.039	0.999
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0420, wR_2 = 0.0781	R_1 = 0.0207, wR_2 = 0.0432	R_1 = 0.0378, wR_2 = 0.0789
R indices (all data)	R_1 = 0.0814, wR_2 = 0.0839	R_1 = 0.0283, wR_2 = 0.0451	R_1 = 0.0526, wR_2 = 0.0883
Extinction coefficient			0.00100(18)
Largest difference peak and hole (e Å^{-3})	0.626 and −0.392	0.591 and −1.003	1.627 and −1.723
CCDC number	883793	883794	883795

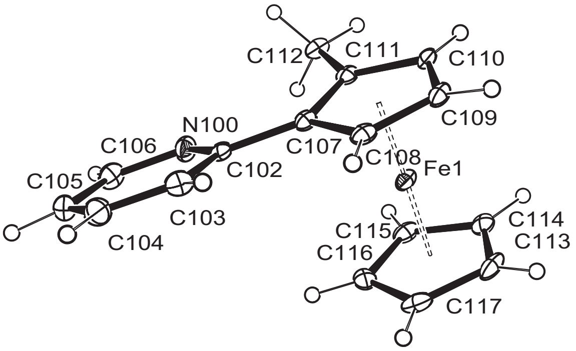


Fig. 1. Molecular structure of **1b**.

Table 2
Comparison of important bond parameters in **1b**, **2a**, and **2b**.

[1]	1b	2a	2b
C2–N	1.340(4)/1.342(4)	1.346(5)	1.346(9)
C2–C7	1.473(4)/1.481(4)	1.485(5)	1.487(10)
N–Hg	NA	2.713(3)	2.748(6)
C8–Hg	NA	2.033(4)	2.036(8)
Hg–Cl	NA	2.313(2)	2.313(3)
C8–Hg–Cl	NA	177.0(1)	177.8(2)
N–C2–C7–C11	13.4(5)/−5.4(4)	172.6(2)	165.1(7)
C2–C7–C11–C_R	0.2(6)/2.3(5)	NA	−1.6(13)
N–C2–C7–C8	−166.5(3)/174.5(3)	−5.80(5)	−15.7(10)
C2–C7–C8–Hg	NA	3.55(5)	8.59(9)
Ct(1)–Fe	1.6436(5)/1.6453(4)	1.654(2)	1.648(3)
Ct(2)–Fe	1.6518(5)/1.6557(4)	1.656(2)	1.656(3)
Ct(1)–Fe–Ct(2)	178.63/8)/178.20(8)	178.9(1)	177.91(17)
C7–Ct(1)–Ct(2)–C_{cp}	2.9/3.7	−18.9	−2.71

[1] The atom numbering corresponds to Scheme 1, not to the individual atom names in the structures. Ct(1) and Ct(2) are the centroids of the substituted and unsubstituted cyclopentadienyl rings. C_{cp} is the carbon atom of the unsubstituted cyclopentadienyl ring next to the projection of the C7–Ct(1) vector onto this ring.

refinement are collected in Table 1. For the solution and refinement of all structures the program package WINGX was used [8].

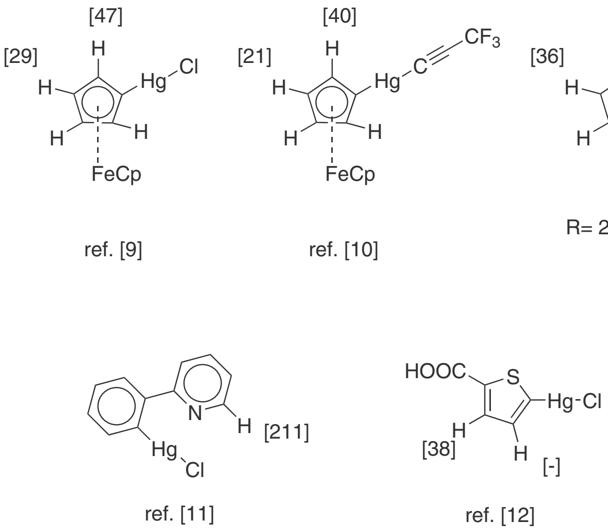
3. Results and discussion

Recently we reported a new synthetic approach towards the preparation of 2-pyridylmetallocenes η^5-[(2-C_5H_4N)(C_5H_4)]ML_n (ML_n = CpFe (**1a**), CpRu and $Mn(CO)_3$) [8]. For the ferrocene derivatives we could show that α-halogenation was possible using a reaction sequence of lithiation and electrophilic halogenation. Similarly, lithiation of pyridylferrocene **1a** followed by methylation with methyl iodide yielded 1-methyl-2-(2-pyridyl)-ferrocene (**1b**) in an isolated yield of 79%. Crystals of this compound were isolated and examined by X-ray diffraction. The molecular structure is shown in Fig. 1. Important bond parameters are collected in Table 2.

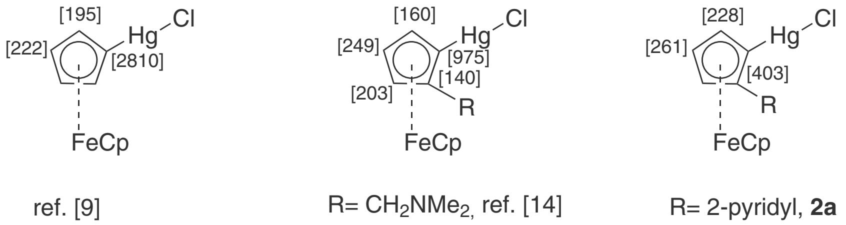
1b crystallizes in the monoclinic space group $P2_1/c$ with two independent molecules in the unit cell. The distance Ct(1)–Fe (the abbreviation "Ct" stands for "centroid") of the substituted cyclopentadienyl ring from the iron atom is slightly shorter than the distance Ct(2)–Fe of the unsubstituted ring, with both rings being nearly ideally eclipsed (2.9° and 3.7° in both molecules). The main difference between the two independent molecules is the degree of twisting of the cyclopentadienyl and the pyridine ring as indicated by the torsion angles N–C2–C7–C11. Quite interesting is the fact that contrary to the halogenated pyridylferrocenes the methyl substituent is situated next to the pyridine nitrogen atom. One might speculate on a weak hydrogen bridge CH···N between the methyl carbon and the pyridine nitrogen as a reason for this observation.

When solutions of **1a**,**b** in CH_2Cl_2 were treated at room temperature with a slight excess of a MeOH solution of mercuric acetate, followed by a MeOH solution of lithium chloride, the mercurated complexes **2** and **3** could be isolated in 43% and 21% yield, respectively, after chromatographic work-up (Scheme 1).

Both compounds were obtained as orange microcrystalline solids. They were characterized by ^{1}H, ^{13}C NMR and mass spectra, and, after recrystallization, by X-ray crystallography. The ^{1}H NMR spectra show for the two cyclopentadienyl protons in α- and ß-position to the HgCl substituent satellites due to ^{1}H–^{199}Hg coupling. Scheme 2 shows our data in comparison with some compounds from the literature [9,10]. No satellites are observed for the pyridine-H6 atom, and also its chemical shift is only slightly different from the starting pyridylferrocenes. This is in sharp contrast to the situation in mercurated phenylpyridine (see Scheme 2): There the corresponding proton is shifted to high field by nearly 1 ppm, and a substantial ^3J(H–Hg) of 211 Hz was reported [11]. This hints at a very weak interaction between the mercury and nitrogen atoms in **2a** and **2b**. However, due to the strong dependence "on spatial orientation and hybridization of bonds between coupling nuclei" ^{1}H and ^{199}Hg [12] this observation has to be carefully interpreted. Thus, in 5-chloromercurio-thiophene-2-carboxylic acid



Scheme 2. Observed *J*(H–Hg) in **2a**, **b** and some related compounds from the literature, *J* values (in Hz) in brackets.



Scheme 3. Observed $J(^{13}C–^{199}Hg)$ in **2a** and some related compounds from the literature, *J* values (in Hz) in brackets.

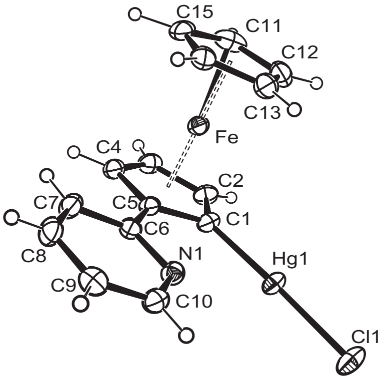


Fig. 2. Molecular structure of **2a**.

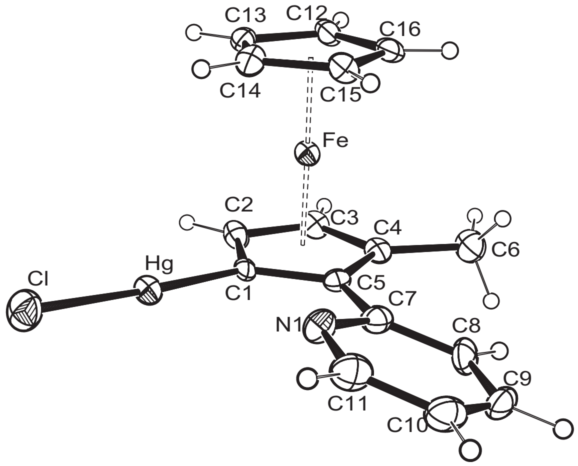


Fig. 3. Molecular structure of **2b**.

(see Scheme 2) a substantial ^4J(H–Hg) was found, while ^3J(H–Hg) was not observed at all [12].

The ^{13}C NMR spectrum of **2a** showed carbon-mercury couplings for the cyclopentadienyl carbon atoms C7, C9 and C10, ^1J(C–Hg) of C8 and ^3J(C–Hg) of C11 were not observed, as well as no carbon-mercury couplings could be detected for **2b**, most likely due to a poor signal–noise ratio. Atoms were assigned by the known [13] sequence $^1J > ^3J > ^2J > ^4J$. Scheme 3 shows our data in comparison with some literature data [9,14].

The mass spectra of both compounds showed the molecular ions; additionally the mass peak of the symmetrical bis-ferrocenyl-mercury compound could be detected for compound **2a**. However, it cannot be decided, if this known symmetrization reaction e.g., [14,6] occurred already during dissolution before the measurement or only in the mass spectrometer [13].

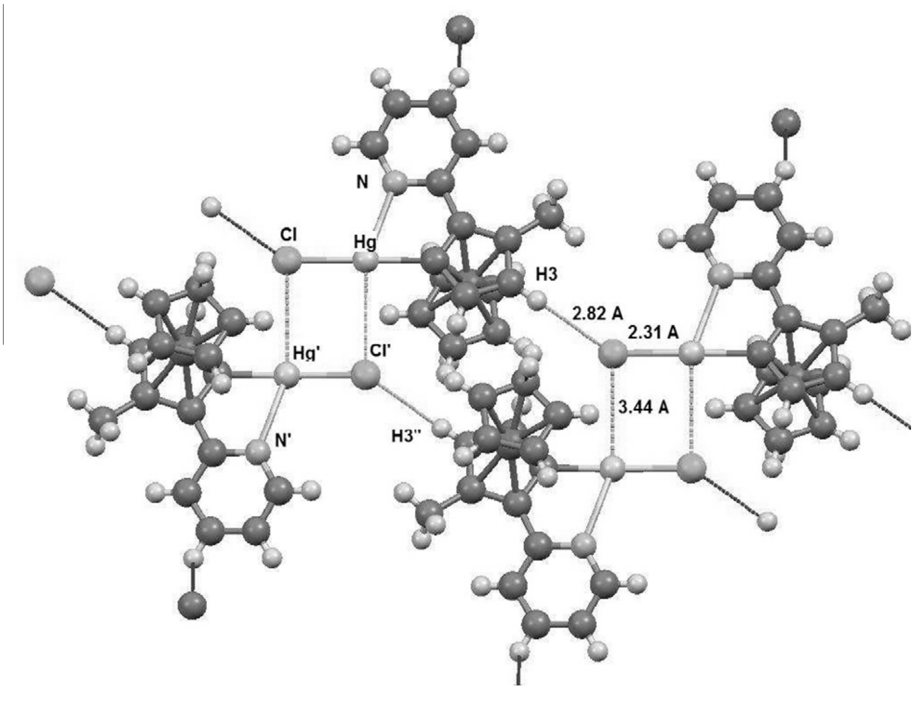


Fig. 4. View of the polymeric structure of **2b** in the crystal.

From CH_2Cl_2 solutions of both mercurated compounds crystals separated that were suitable for X-ray diffraction analysis. Views of the molecular structures of both compounds are depicted in Figs. 2 and 3, important bond parameters are collected in Table 2.

The Hg–C and Hg–Cl distances and the C–Hg–Cl angle are nearly identical in both compounds, however, the Hg–N distance is slightly larger in **3**. In comparison to the structure of (2-pyridylphenyl)mercury chloride [15] the bond lengths Hg–Cl are identical in **2** and **3**, while Hg–C are shorter and Hg–N are longer in our compounds. In the structure of the $Cr(CO)_3$ complex of the mercurated phenylpyridine the Hg–C and Hg–Cl distances are the same as in the monometallic compound, while the Hg–N distance is elongated, but still shorter than in **2** and **3** [6]. The torsion angle between cyclopentadienyl and pyridine ring (N–C2–C7–C11 in Scheme 1) is in **3** significantly larger than in **4**, suggesting sub-optimal interaction of the nitrogen lone pair and the mercury atom. While in **2** both cyclopentadienyl rings are half-way between eclipsed and staggered conformation ("ideal" torsion angles C7–Ct(1)–Ct(2)–C_{cp} would be 0 and 36°), the eclipsed conformation in **1b** remains conserved in **3**.

One particularly interesting feature in the crystal structure of the above-mentioned (2-pyridylphenyl)mercury chloride is the formation of tetranuclear $[Hg_4L_4Cl_4]$ units via stacking of two Hg_2Cl_2 rings with two triply bridging chlorine atoms [17]. Within one Hg_2Cl_2 dimer there were two short and two long Hg–Cl distances of 2.31 and ca. 3.4 Å, while the Hg–Cl distance between two dimers was 3.18 Å.

In the crystal of **2** there is also a weak contact between two neighboring Hg–Cl groups, however with a distance of more than 3.8 Å.

In the crystal structure of **3** another structural motif is found. Basic unit is again a dimer, formed between two inversion related molecules at (x,y,z) and $(1-x,-y,-z)$. A central Hg_2Cl_2 bridge shows two short and two long Hg′–Cl distances of 2.313 and 3.435 Å, respectively. Two such dimers associate via hydrogen bridges between the chloride ligand and the hydrogen atom H3 of the substituted cyclopentadienyl ring, with a length of 2.823 Å. Overall, a polymeric structure is formed along the *z* direction of the crystal (Fig. 4).

4. Conclusion

Pyridylferrocenes can be mercurated with $Hg(OAc)_2/LiCl$ to give $[1\text{-}(ClHg)\text{-}2\text{-}(2\text{-}C_5H_4N)\text{-}3\text{-}R\text{-}C_5H_2]Fe(C_5H_5)$ (R = H, CH_3) in modest yields. NMR spectra and crystal structure determinations show only a very weak interaction between the pyridine nitrogen and the mercury atom. Preliminary experiments show that the HgCl group can be substituted by other functional groups, but to make preparative use of such reactions the yields of the mercuration reactions have to be optimized first. Studies to this end are underway.

Appendix A. Supplementary data

CCDC 883793–883795 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk.

References

[1] Part 2: K. Sünkel, S. Weigand, Inorg. Chem. Commun. (2012), doi:10.1016/j.inoche.2012.04.001.
[2] A.N. Nesmeyanov, E.G. Perevalova, R.V. Golovnya, O.S. Nesmeyanov, Doklady. Akad. Nauk. SSSR 47 (1954) 459.
[3] (a) S.Q. Huo, Y.J. Wu, Y. Zhu, L. Yang, J. Organomet. Chem. 470 (1994) 17;
(b) J. Wu, S.Q. Huo, Y. Zhu, L. Yang, J. Organomet. Chem. 481 (1994) 235;
(c) S.Q. Huo, Y. Zhu, Y.J. Wu, J. Organomet. Chem. 490 (1995) 243;
(d) Y.J. Wu, X.L. Cui, Y.H. Liu, H.Z. Yuan, X.A. Mao, J. Organomet. Chem. 543 (1997) 63;
(e) X.L. Cui, Y.-J. Wu, C.X. Du, L.R. Yang, Y. Zhu, Tetrahedron Asymm. 10 (1999) 1255;
(f) X.L. Cui, Y.J. Wu, D.P. Zou, C.H. He, J.J. Chai, Polyhedron 18 (1999) 1023;
(g) Y. Wu, X. Cui, N. Zhou, M. Song, H. Yun, C. Du, Y. Zhu, Tetrahedron Asymm. 11 (2000) 4877;
(h) K. Lin, M. Song, Y. Zhu, Y. Wu, J. Organomet. Chem. 660 (2002) 139;
(i) H.-X. Wang, H.-C. Zhou, H.-F. Wu, Y.-J. Li, F.-Y. Geng, Acta Cryst. E61 (2005) m2570;
(k) X.-Q. Hao, X.-L. Hou, X-M. Zhao, L.-Y. Wu, J.-F. Gong, M.-P. Song, Y.-J. Wu, Synth. React. Inorg. Met.-org. Nano-met. Chem. 36 (2006) 517;
(l) K.E. Thiesen, K. Maitra, M.M. Olmstead, S. Attar, J. Organomet. Chem. 696 (2011) 1355.
[4] A.F. Cunningham, Organometallics 16 (1997) 1114.
[5] K. Sünkel, T. Kießling, J. Organomet. Chem. 637-639 (2001) 796.
[6] A. Berger, A. deCian. J.-P. Djukic, J. Fischer, M. Pfeffer, Organometallics 20 (2001) 3230.
[7] K. Sünkel, S. Weigand, Inorg. Chim. Acta 370 (2011) 224.
[8] (a) WINGX: L.J. Farrugia, J. Appl. Crystallogr. 32 (1999) 837;
(b) . SIR-97:A. Altomare, M.C. Burla, M. Camalli, G.L. Cascarano, C. Giacovazzo, A. Guagliardi, A.G.G. Moliterni, G. Polidori, R. Spagna, J. Appl. Cryst. 32 (1999) 115;
(c) . SHELX-97:G.M. Sheldrick, Acta Cryst. A64 (2008) 112.
[9] K. Sünkel and T. Kießling, unpublished.
[10] A.K. Brisdon, I.R. Crossley, R.G. Pritchard, Organometallics 24 (2005) 5487.
[11] R.V. Parish, J.P. Wright, R.G. Pritchard, J. Organomet. Chem. 596 (2000) 165.
[12] Z. Popovic, Z. Soldin, J. Plavec, D. Vikic-Topic, Appl. Organomet. Chem. 14 (2000) 598. and literature cited therein.
[13] K.E. Rowland, R.D. Thomas, Magn. Res. Chem. 23 (1985) 916. and literature cited therein.
[14] N. Seidel, K. Jacob, A.K. Fischer, K. Merzweiler, C. Wagner, M. Fontani, P. Zanello, J. Organomet. Chem. 630 (2001) 149.
[15] E.C. Constable, T.A. Leese, D.A. Tocher, J. Chem. Soc., Chem. Commun. (1989) 570.