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LETTER

Heterocycles as Coordinating Ligands
Part VII. New Pyrrolyl Iron Complexes*

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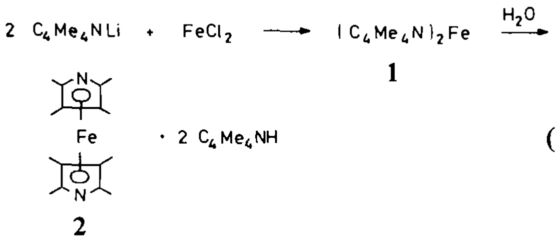
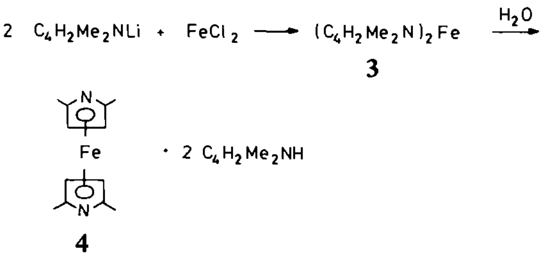
Recently [2], octamethyl-1,1'-diazaferrocene has been shown to exist as its tetramethylpyrrole adduct **2** (eqn. (1)). This result has been attributed to the stabilizing effect of both the methyl substituents and the pyrrole molecules connected to the sandwich complex via hydrogen bridges.

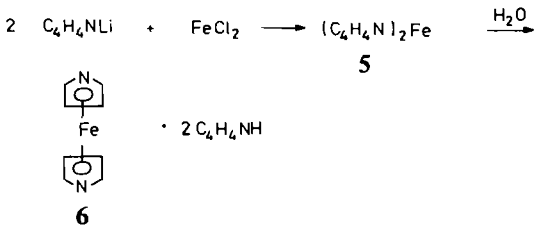
Starting with 2,5-dimethylpyrrole (eqn. (2)) or pyrrole itself (eqn. (3)), the diazaferrocene adducts **4** and **6** are obtained analogously by hydrolysis of their isolable precursors **3** and **5**† (dark brown oils). ^{13}C NMR data (see Table 1) indicate that the new compounds **3**–**6** are structural analogues



(1)



(2)



(3)

TABLE 1. NMR data of the compounds **1**–**6** (δ ppm)

Compound	^{13}C NMR	^{1}H NMR
1	3,4-Me^{a}, 9.79; 2,5-Me, 11.33; C-3,4, 113.05; C-2,5, 119.81	Me^{b}, 1.97
2	3,4-Me^{c}, 8.78; 8,9-Me, 9.21; 7,10-Me, 10.98; 2,5-Me, 12.62; C-3,4, 84.36; C-2,5, 99.29; C-8,9, 113.76; C-7,10, 120.56	3,4-Me^{b}, 1.57; 8,9-Me, 1.91; 7,10-Me, 1.98; 2,5-Me, 2.05
3	2,5-Me^{c}, 13.03; C-3,4, 106.12; C-2,5, 126.28	2,5-Me^{b}, 1.93; 3,4-CH, 5.78
4	7,10-Me^{b}, 13.09; 2,5-Me, 15.25; C-3,4, 71.67; C-2,5, 103.43; C-8,9, 106.46; C-7,10, 125.33	7,10-Me^{b}, 2.0; 2,5-Me, 2.15; 3,4-CH, 3.90; 8,9-CH, 5.82; NH, 7.67
5	C-3,4^{a}, 108.35; C-2,5, 117.46	2,3,4,5-CH^{b}, 6.08 (broad)
6	C-3,4^{d}, 72.77; C-2,5, 89.82; C-8,9, 108.06; C7,10, 118.03	3,4-CH^{d}, 4.8; 2,5-CH, 5.6; 8,9-CH, 6.2; 7,10-CH, 6.8; NH, 8.55

$^{a}C_6D_5CD_3$ at –30°. $^{b}C_6D_6$. $^{c}CD_2Cl_2$. $^{d}CD_2Cl_2$ at –30°.

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*For Part VI, see ref. 1.

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†Apparently this compound is not identical to that obtained from $FeCl_2$ and sodium pyrrolide, see ref. 3.

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of **1**[††] and **2**. As expected, the stability in this series decreases markedly on exchange of the methyl groups for hydrogen substituents. Compounds **3**, **4** and **5** may be isolated from solution and stored at low temperatures as pure compounds, as indicated by NMR data (**6** has been characterized in solution only), but no correct elemental analyses could be obtained because of their rapid decomposition at room temperature. In contrast with compound **2** [1, 5] no defined products could be obtained on reaction of **4** and **6** with Brønstedt and Lewis acids.

This result indicates methyl substitution to be helpful but not essential for the stabilization of the π-coordinated ligands in diazaferrocene structures, and therefore parallels the chemistry of cyclopentadienyliron pyrrole cations reported by us earlier [6].

Experimental

Reactions were carried out routinely in an argon atmosphere in purified solvents.

$(C_4H_2Me_2N)_2Fe$ (3) and $(C_4H_2Me_2N)_2Fe\cdot 2C_4H_2Me_2NH$ (4)

$C_4H_2Me_2NLi$ (2.87 g; 28.39 mmol), prepared from $C_4H_2Me_2NH$ and n-BuLi, was added to 1.80 g (14.2 mmol) anhydrous $FeCl_2$ in 25 ml tetrahydrofuran at room temperature. The solvent was removed *in vacuo* and replaced by 20 ml CH_2Cl_2 (from this solution **3** may be isolated). After addition of 0.25 ml H_2O the solution was filtered and cooled down to −78°. Compound **4** was isolated as an orange oil in *c.* 50% yield. MS (70 eV): *m/e* 244 ($M^+ - 2C_4H_2Me_2NH$) and further fragments.

$(C_4H_4N)_2Fe$ (5) and $(C_4H_4N)_2Fe\cdot 2C_4H_4NH$ (6)

These compounds were prepared according to the procedure reported above by use of C_4H_4NH instead of $C_4H_2Me_2NH$. Compound **6** decomposes readily on isolation.

Acknowledgements

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[††] Compound **1** forms a diamagnetic dimer in toluene at room temperature which apparently contains σ-coordinated ring ligands. In the solid state a complicated temperature-dependent equilibrium of three different structures is observed by Mössbauer spectroscopy, see ref. 4.