

Preliminary communication

The photoreaction of the $(\eta\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{I}$ –diisopropylamine system with pyrrole and indole

Janusz Zakrzewski

Institute of Chemistry, University of Łódź, 90-136 Łódź, Narutowicza 68 (Poland)

(Received March 23rd, 1987)

Abstract

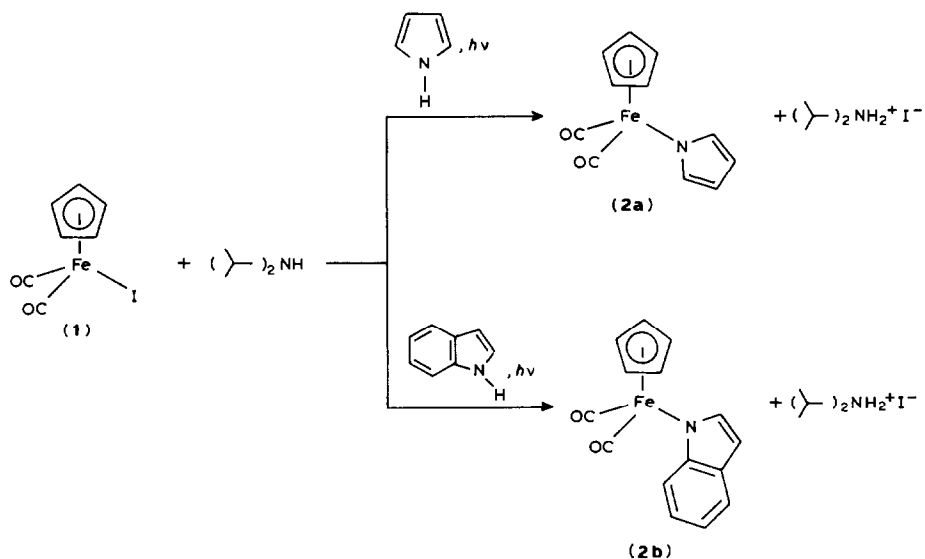
$(\eta\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{I}$ reacts with pyrrole and indole in the presence of diisopropylamine, in sunlight, to give the corresponding $(\eta\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{-}\eta^1\text{-N}$ -heterocyclic complexes in 72–88% yield.

Here I describe a simple, convenient conversion of $(\eta\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{I}$ (**1**) into the $\eta^1\text{-N}$ -pyrrolyl and $\eta^1\text{-N}$ -indolyl complexes **2a**, **2b**. The method involves the irradiation with sunlight of a mixture of **1**, pyrrole or indole and diisopropylamine in heptane at room temperature (Scheme 1). Complexes **2a**, **2b** have been previously prepared in poor yields (19 and 25%, respectively) by heating **1** with potassium salts of pyrrole and indole in benzene at 60 °C [1].

The photochemical preparation of **2a** was performed in the following manner. A suspension of **1** (1.52 g, 5 mmol) in heptane (20 ml) containing freshly distilled pyrrole (0.60 ml, 8.7 mmol) and diisopropylamine (1.50 ml, 11.5 mmol) in a glass tube, was exposed to sunlight. The precipitation of diisopropylamine hydroiodide, as well as a slight evolution of CO, only at the beginning of the reaction, was observed. The mixture was shaken occasionally and the black crystals of **1** disappeared gradually to form an orange solution with a copious beige precipitate. After 1–2 sunny days (even in January in Poland) all of **1** had been consumed (TLC control), the ammonium salt was then filtered off and washed with diethyl ether. The solvent was removed under vacuum, and the residue was crystallized from diethyl ether/pentane to afford **2a** as beautiful orange crystals (0.88 g, 72%).

Under similar conditions a mixture of **1** (1.52 g, 5 mmol), indole (0.90 g, 7.7 mmol) and diisopropylamine (1.50 ml, 11.5 mmol) gave **2b** in 88% yield.

The complexes **2a** and **2b** obtained as described above, were found, by use of IR ^1H NMR and mass spectroscopy, to be identical with authentic samples prepared by a published procedure [1]. However, I found the melting point for **2a** to be 102–103 °C (dec.), whereas Pauson reported one of 91 °C. The melting point of 141–142 °C for **2b**, however, was consistent with the reported value of 141.5–142 °C.



Scheme 1

The η^1 -pyrrolyl complex **2a** was decarbonylated in refluxing benzene (1.5 h) and gave azaferrocene [1,2], $(\eta\text{-C}_4\text{H}_4\text{N})(\eta\text{-C}_5\text{H}_5)\text{Fe}$, in 65% yield.

The above procedure is mild, clean and convenient. Its great advantage is that pyrrole (or indole) and diisopropylamine are used instead of the potassium salts of these heterocycles. The yields are significantly improved, viz. 72 and 88% compared with 19 and 25% [1].

Complexes **2a**, **2b** and azaferrocene are interesting in that they enable the study of the reactions of pyrrolyl and indolyl ligands bonded in different ways to a transition metal. This study is now in progress.

This work is being supported by the Polish Academy of Sciences grant CPBP 01.13.

References

- 1 P.L. Pauson and A.R. Quazi, *J. Organomet. Chem.*, 7 (1967) 321.
- 2 K.K. Joshi, P.L. Pauson, A.R. Quazi and W.H. Stubbs, *J. Organomet. Chem.*, 1 (1964) 471.