

### Preliminary communication

## REACTION OF CYCLOHEXENE WITH A DIHYDRIDO—DINITROGEN COMPLEX OF IRON(II) AND A TETRAHYDRIDO COMPLEX OF IRON(IV)

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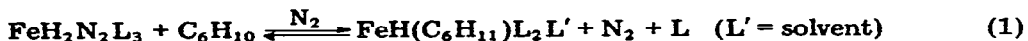
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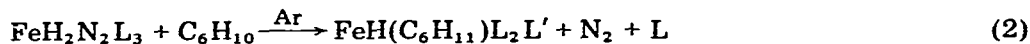
### Summary

The reaction of cyclohexene with  $\text{FeH}_2\text{N}_2\text{L}_3$  and  $\text{FeH}_4\text{L}_3$  ( $\text{L} = \text{PEtPh}_2$ ) to give the cyclohexyl derivative  $\text{FeH}(\text{C}_6\text{H}_{11})\text{L}_2\text{L}'$  ( $\text{L}' = \text{solvent}$ ) is described, and a possible mechanism suggested.

We have previously described the insertion of  $\text{CO}_2$  into the  $\text{Fe—H}$  bond of the complex  $\text{FeH}_2\text{N}_2\text{L}_3$  (I) ( $\text{L} = \text{PEtPh}_2$ ) [1], and also the reaction of that complex with ethylene [2]. We now consider the insertion of cyclohexene into the  $\text{Fe—H}$  bond of the dihydridodinitrogen complex (I) and of the tetrahydrido complex  $\text{FeH}_4\text{L}_3$  (II) ( $\text{L} = \text{PEtPh}_2$ ). The reaction was followed by means of gas-volumetric, chromatographic (GLC) and spectroscopic (IR) techniques. When the reaction was performed in the absence of solvent, the addition of cyclohexene to the solid, powdered dihydridodinitrogen iron(II) complex (I) at room temperature caused rapid evolution of nitrogen, but the product decomposed. When the complex was dissolved in tetrahydrofuran under nitrogen at room temperature, the nitrogen was eliminated slowly, as shown by the gradual disappearance of the strong band  $\nu(\text{N—N})$  of the dinitrogen complex (I) at  $2060\text{ cm}^{-1}$ . At lower temperatures (near  $0^\circ\text{C}$ ) the reaction was very slow. Free phosphine was detected by washing with *n*-pentane, but all attempts to isolate the complex in the crystalline state failed, because of its instability. Probably only one of the  $\text{Fe—H}$  bonds is involved in the reaction, since the bands  $\nu(\text{Fe—H})$  did not disappear completely at any of the examined concentrations of the hydride solution, but a short, broad band at  $1865\text{ cm}^{-1}$  (in solution) was still present in the IR spectrum:

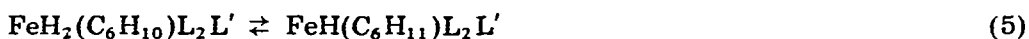
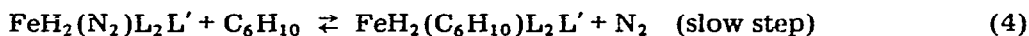
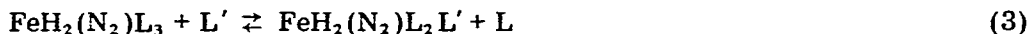


The reaction of complex I with cyclohexene, in the solid or in THF solution, was performed under nitrogen and under argon but the nitrogen of the complex was irreversibly released only when the reaction was performed under argon:

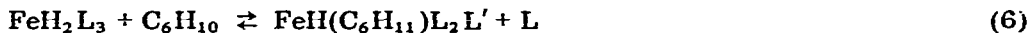


The elimination of the nitrogen was also complete when working with a molar ratio Fe:C<sub>6</sub>H<sub>10</sub> of 1:1, since the strong ν(N—N) band at 2060 cm<sup>-1</sup> of the original dinitrogen complex disappeared completely.

A possible mechanism is one in which a molecule of solvent (L') first coordinates to the complex with loss of a molecule of phosphine. Subsequently, nitrogen is released and a molecule of cyclohexene attaches to the metal to give a π-cyclohexenyl compound, which in turn is rapidly converted into a cyclohexyl derivative, in accordance with the following scheme:



The tetrahydrido complex FeH<sub>4</sub>L<sub>3</sub> in THF, at room temperature and 0°C, also reacted with cyclohexene with evolution of hydrogen and displacement of phosphine, to give the cyclohexyl derivative FeH(C<sub>6</sub>H<sub>11</sub>)L<sub>2</sub>L', identified by its IR spectrum (in solution). When the reaction was performed under nitrogen, in the first step hydrogen was replaced by nitrogen [3] and the dihydridodinitrogen complex (I) was formed. This then reacted with cyclohexene as in the scheme above. Under argon the dihydrido complex FeH<sub>2</sub>L<sub>3</sub> was first formed from the corresponding tetrahydrido complex [3], and insertion of cyclohexene then took place:



Further investigations on the insertion of olefins into the M—H bonds are in progress.

### Acknowledgment

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### References

- 1 V.D. Bianco, S. Doronzo and M. Rossi, *J. Organometal. Chem.*, **35** (1972) 337.
- 2 V.D. Bianco, S. Doronzo and M. Aresta, *J. Organometal. Chem.*, **42** (1972) C63.
- 3 M. Aresta, F. Giannoccaro, M. Rossi and A. Sacco, *Inorg. Chim. Acta*, **5** (1971) 115.