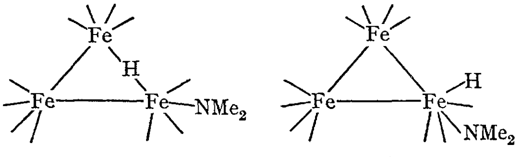
Formation of $HFe_3(CO)_{11}NMe_2$[1]

By ILSONG RHEE*, MEMBO RYANG, and SHIGERU TSUTSUMI

(*Department of Chemical Technology, Faculty of Engineering, Osaka University, Miyakojima, Osaka, Japan*)

BENZOYL CHLORIDE reacts with $Fe_3(CO)_{12}$ in benzene to give triphenylmethane. Assuming that the benzene reacted with a reaction intermediate, we repeated the experiment using dimethylformamide as solvent instead of benzene (80°, 24 hr.). The reaction mixture was hydrolysed with 6N-hydrochloric acid, and extracted with diethyl ether. The residual oil obtained after removal of the solvent was chromatographed on silica gel. Elution of the first band with light petroleum (b.p.40–60°) gave a red-black complex (1·0 g). This complex was recrystallized from n-pentane (at −70°), m.p. 162—164° (decomp.) and was shown to be relatively stable at room temperature but decomposed slowly on standing in air. The composition of the complex ($C_{13}H_7O_{11}NFe_3$) was confirmed by microanalysis. The n.m.r. spectrum of the complex had absorptions at τ 6·0 (singlet, 6H) and 27·80 (singlet, 1H), and the i.r. spectrum showed the presence of terminal carbonyls ($\nu_{Fe\text{-}Co}$ 2080—1978 cm.$^{-1}$) and an iron–hydrogen bond ($\delta_{Fe\text{-}H}$ 770 cm.$^{-1}$). Hydrogen was detected in the products of thermal decomposition of the complex by gas microanalysis. Thus, the structure of the complex is thought to be (Ia) or (Ib) on the basis of the structure of $Fe_3(CO)_{12}$.[2]



(Ia) (Ib)

[Carbonyl groups omitted]

It is interesting to note that this complex has both a metal–nitrogen σ-bond and a metal–hydrogen bond, although *X*-ray diffraction studies will be necessary to determine the exact structure.

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[1] For relevant papers, see: H. D. Kaesz, R. Bau, and M. R. Churchill, *J. Amer. Chem. Soc.*, 1967, **89**, 2775; J. A. J. Jarvis, B. E. Job, B. T. Kilbourn, R. H. B. Mais, P. G. Owston, and P. F. Todd, *Chem. Comm.*, 1967, 1149; I. Rhee, M. Ryang, and S. Tsutsumi, *J. Organometallic Chem.*, 1967, **9**, 361; I. Rhee, N. Mizuta, M. Ryang, and S. Tsutsumi. *Bull. Chem. Soc. Japan*, in the press.

[2] C. H. Wei and L. F. Dahl, *J. Amer. Chem. Soc.*, 1966, **88**, 1821.