Ligand Exchange in the Reaction of Ferrocene and $RuCl_3$

By GEORGE J. GAUTHIER

[*Frank J. Seiler Research Laboratory (OAR), USAF Academy, Colorado* 80840]

THERE have been several reports[1] of ligand-exchange reactions in ferrocene and substituted ferrocenes. In contrast, no reactions involving exchange of the cyclopentadienyl ligands of ferrocene to a different metal atom have been reported. I report a novel ligand exchange reaction between ferrocene and $RuCl_3$ which gives ruthenocene.

The reaction was performed using a mixture of anhydrous ferrocene and $RuCl_3$ in a sealed evacuated Carius tube heated with shaking in an oil bath at 250° for 1—2 days. The metallocene mixture was separated from inorganic salts and decomposition by-products by sublimation and the metallocenes were purified by g.l.c. or preparative t.l.c. The conversion of $RuCl_3$ into ruthenocene was $50 \pm 5\%$ by ^1H n.m.r. and g.l.c. analysis of the metallocene mixture. The yield of ruthenocene was 38—45% depending on the method of separation.

The reaction is sensitive to water and oxygen. Reactions with a 3 : 1 ferrocene: $RuCl_3$ molar ratio gave only $35 \pm 3\%$ conversion but an 8 : 1 ratio gave $50 \pm 5\%$. No reaction was observed below 210°. Anhydrous $RuCl_3$ was prepared by heating a mixture of $RuCl_3$ in thionyl chloride and high-vacuum distillation of the excess of solvent.

A similar reaction with 1,1′-diethylferrocene gave a small amount of 1,1′-diethylruthenocene, ^1H n.m.r. (CCl_4) τ 5·68 (singlet, 8 protons), 7·83 (quartet, 4 protons), and 8·94 (triplet, 6 protons) as well as other polyalkylated metallocenes.

(*Received, April 14th,* 1969; *Com.* 509.)

[1] A. N. Nesmeyanov, N. A. Vol'kenau, and I. N. Bolesova, *Doklady Akad. Nauk S.S.S.R.*, 1963, **149**, 615; *Tetrahedron Letters*, 1963, 1725; D. E. Bublitz, *Canad. J. Chem.*, 1964, **42**, 2381; A. N. Nesmeyanov, N. A. Vol'kenau, and L. S. Shilovtseva, *Doklady Akad. Nauk S.S.S.R.*, 1965, **160**, 1327; A. N. Nesmeyanov, N. A. Vol'kenau, and I. N. Bolesova, *ibid.*, 1966, **166**, 607; D. E. Bublitz, *J. Organometallic Chem.*, 1969, **16**, 149.