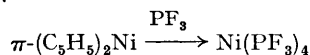


A New Synthesis of Tetrakis(trifluorophosphine)nickel(0), Ni(PF₃)₄

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CURRENT interest in phosphorus trifluoride complexes of transition metals¹⁻⁴ leads us to report a new synthesis of tetrakis(trifluorophosphine)nickel(0) by the reaction between phosphorus trifluoride and nickelocene under very mild conditions.



Thus heating nickelocene (2.4 mmoles) and PF₃ (6.1 mmoles) in a sealed Pyrex glass tube (ca. 80 c.c. volume) at 60° for 96 hours leads to ~20% conversion into Ni(PF₃)₄ [95% based on PF₃ consumed]. The product was identified by its volatility and characteristic i.r. spectrum,^{1a} while the ¹⁹F n.m.r. spectrum showed the same complex pattern described previously.² [ϕ_{F} (rel. to CCl₃F)

+16.4 p.p.m.; $J_{\text{P-F}} = 1260$ c./sec. (between the two sharpest lines)]. At slightly higher pressures some Ni(PF₃)₄ was formed after 3 days even at room temperature.

This synthetic approach contrasts with existing preparative methods using Ni(CO)₄, Ni, or NiI₂^{1a,2} which necessitate high temperatures and pressures. The ease with which both C₅H₅ rings are displaced from nickelocene parallels other recent work⁵ in which fluorophosphine complexes of the type (R_nPF_{3-n})₃Mo(CO)₃ (R = F, CCl₃, CF₃, C₃F₇) are also readily formed by displacement of cycloheptatriene from C₇H₈Mo(CO)₃ under similarly mild conditions.

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⁵ J. F. Nixon and C. G. Barlow, unpublished results.