A New Synthesis of Tetrakistrifluorophosphinenickel(0), $Ni(PF_3)_4$

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CURRENT interest in phosphorus trifluoride complexes of transition metals[1–4] leads us to report a new synthesis of tetrakistrifluorophosphinenickel(0) by the reaction between phosphorus trifluoride and nickelocene under very mild conditions.

$$\pi\text{-}(C_5H_5)_2Ni \xrightarrow{PF_3} Ni(PF_3)_4$$

Thus heating nickelocene (2·4 mmoles) and PF_3 (6·1 mmoles) in a sealed Pyrex glass tube (*ca.* 80 c.c. volume) at 60° for 96 hours leads to ~20% conversion into $Ni(PF_3)_4$ [95% based on PF_3 consumed]. The product was identified by its volatility and characteristic i.r. spectrum,[1a] while the ^{19}F n.m.r. spectrum showed the same complex pattern described previously.[2] [ϕ_F (rel. to CCl_3F) +16·4 p.p.m.; $J_{P\text{-}F}$ = 1260 c./sec. (between the two sharpest lines)]. At slightly higher pressures some $Ni(PF_3)_4$ was formed after 3 days even at room temperature.

This synthetic approach contrasts with existing preparative methods using $Ni(CO)_4$, Ni, or NiI_2[1a,2] which necessitate high temperatures and pressures. The ease with which both C_5H_5 rings are displaced from nickelocene parallels other recent work[5] in which fluorophosphine complexes of the type $(R_nPF_{3-n})_3Mo(CO)_3$ (R = F,CCl_3,CF_3,C_3F_7) are also readily formed by displacement of cycloheptatriene from $C_7H_8Mo(CO)_3$ under similarly mild conditions.

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