

Preliminary communication

Tetrakis(pentafluorophenyl)vanadium

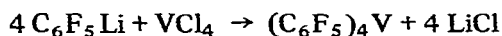
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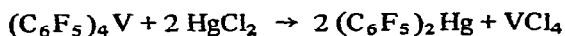
Tetrabenzylvanadium is the only example known of a thermally stable compound of vanadium(IV) with four V–C σ -bonds; its synthesis has been briefly described recently¹. First attempts to prepare R₄V compounds (R = CH₃, C₂H₅, C₄H₉², C₆H₅³) were unsuccessful: in all cases formation of organic vanadium compounds with the metal in its lower oxidation state was observed. It is well known that the thermal stabilities of organic compounds of transition metals increase on replacing aryl radicals by perfluoroaryl radicals. Here we present information on the synthesis of tetrakis(pentafluorophenyl)vanadium resulting from the reaction of vanadium tetrachloride etherate with pentafluorophenyllithium.

To an ether/hexane solution of C₆F₅Li (0.096 mol), at –70° and with vigorous stirring, was slowly added an ether solution of VCl₄ (0.024 mol). The reaction mixture was left under these conditions for 12 h and was then allowed to come gradually to room temperature. The colour of the solution became violet, and lithium chloride (0.090 mol) was precipitated.



After the lithium chloride had been removed the solution was hydrolyzed with a dilute solution of HCl. Pentafluorobenzene (0.085 mol) and a small amount of decafluorophenyl (0.003 mol) were detected in the products of the hydrolysis, in the ratio V/C₆H₅ 1/3.8, by gas-liquid chromatography.

A solution of HgCl₂ (0.020 mol) in THF was added to an ether/hexane solution of tetrakis(pentafluorophenyl)vanadium (0.010 mol). An exothermic reaction occurred, the colour of the solution became brown, and bis(pentafluorophenyl)mercury was precipitated, yield 0.019 mol of (C₆F₅)₂Hg, m.p. 140° (lit.⁴ 142°).



Tetrakis(pentafluorophenyl)vanadium crystallized slowly from the reaction solution at 0°C. The violet-coloured crystals of $(C_6F_5)_4V \cdot 2(C_2H_5)_2O$ had m.p. 220°(dec.). (Found: C, 44.43; H, 2.47; V, 7.00. $C_{32}H_{20}F_{20}O_2V$ calcd.: C, 44.29; H, 2.30; V, 5.89%.) Infrared spectrum of the product contained the following absorption bands 840 m, 950 (sh), 960 s, 1005 s, 1020 w, 1055 w, 1080 s, 1185 m, 1470 s, 1520 s, 1540 s, and 1650 m cm^{-1} . The compound is extremely sensitive to moisture.

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