

Journal of Organometallic Chemistry, 429 (1992) 13–26
 Elsevier Sequoia S.A., Lausanne
 JOM 22368

Reactions of pentafluorophenyl germanium hydrides with nickelocene

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(Received September 23, 1991)

Abstract

Both $(C_6F_5)_2Ge(H)Ge(H)(C_6F_5)_2$ and $(C_6F_5)_2GeH_2$ react with bis-(cyclopentadienyl)nickel to form a novel germylnickel cluster $[(C_6F_5)_2GeNiCp]_2$. The molecule of the complex contains a butterfly-like Ge_2Ni_2 core with strong Ge–Ni (2.257 Å) and weak Ni–Ni (2.492 Å) bonds. The tetrahedral angle between its Ni_2Ge wings is 117.6°. Interaction of nickelocene with tris(pentafluorophenyl)germane yields 30% of the ionic complex $\{[(C_6F_5)_3Ge]_2NiCp\}^- [Cp_3Ni_2]^+$. The X-ray diffraction analysis has shown that the structure of the $[(C_6F_5)_3Ge]_2NiCp^-$ anion contains the 3-membered Ge–Ni–Ge chain. The Ni–Ge bond lengths are 2.265 and 2.263 Å, the Ge–Ni–Ge angle is 127.3°. The Cp_3Ni_2 cation is a triple-decker sandwich with eclipsed conformation of the Cp-rings.

Introduction

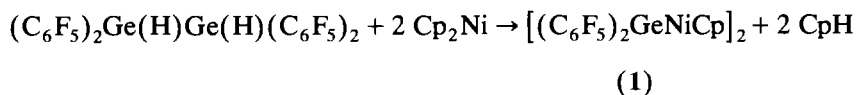
High reactivity of metallocenes toward different organoelement compounds allows Cp_2M to be used for the synthesis of polynuclear compounds [1]. Thus, a nine-nuclear branched chain containing germanium, cadmium (mercury) and nickel atoms has been obtained by reaction of Cp_2Ni with $(Ph_3Ge)_2M$ ($M = Cd, Hg$) [2]. The perfluorinated analogue $[(C_6F_5)_3Ge]_2Hg$ in the reactions with cobaltocene, chromocene and Cp_2Yb gives five-nuclear mercurates of the corresponding Cp_2M^+

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cations [3]. Vanadocene in a similar reaction forms an insertion product with Ge–V–Hg–Ge catena [4]. It is known that Cp₂V easily displaces hydrogen in some organoelement hydrides to give *d*¹ and *d*²-complexes with a vanadium–element bond [5–7]. Earlier we have shown that the interaction of Cp₂V with (C₆F₅)₂Ge(H)Ge(H)(C₆F₅)₂ leads to Cp₂VGe(C₆F₅)₂Ge(C₆F₅)₂H with the *trans* configuration of V–Ge–Ge–H [9]. Here we report on the reactions of (C₆F₅)₂GeH₂, (C₆F₅)₃GeH and (C₆F₅)₂Ge(H)Ge(H)(C₆F₅)₂ with nickelocene and the crystal structure of the product obtained.

Results and discussion

Like vanadocene, nickelocene reacts with (C₆F₅)₂Ge(H)Ge(H)(C₆F₅)₂ under mild conditions but does not give similar products, *i.e.* hydrogen and Cp₂NiGe(C₆F₅)₂Ge(C₆F₅)₂H. Instead, the four-nuclear cluster [(C₆F₅)₂GeNiCp]₂ (**1**) has been isolated and cyclopentadiene was identified among the volatile products.



The dark-red crystals of **1** have m.p. 270–272°C (decomp.) and are stable in air for a few hours. The compound is well soluble in THF, DME and in toluene and alkanes on heating. It was characterized by IR and ESR spectroscopy, element analysis and X-ray single crystal diffraction studies. The latter reveal that the molecule contains a Ge₂Ni₂ core in a “butterfly” configuration, with Ni–Ni distance of 2.492(1) Å, the Ge ⋯ Ge non-binding distance 3.219(1) Å and a dihedral angle of 117.6° between the Ni(1)–Ni(2)–Ge(1) and Ni(1)–Ni(2)–Ge(2) butterfly “wings”. The Ni–Ge bonds are significantly shorter (average 2.257(4) Å) than in metal-chain complexes [Ph₃Ge–Ni(Cp)(–MGePh₃)]₂M (2.308 and 2.329 Å for M = Cd and Hg, respectively) [2]. These distances merit a comparison with Pauling’s metallic radii (calculated for the case of valence equal to coordination number, *i.e.* for a strong single bond), being 1.223 Å for Ge and 1.154 Å for Ni [10]. The Ni–Ni bond distances in polynuclear complexes vary widely, however according to [11] even in the presence of strongly tightening bridging ligands, no distances shorter than 2.314 Å (in the complex [(Et₃P)₂Ni₂(μ-C₆H₄)] [12]) are observed; this limit is in good agreement with the double Pauling’s radius (2.308 Å). In **1** the Ni–Ni distance is much longer. On the other hand, the Ge–Ni bond is shorter than the sum of the covalent radii (2.377 Å) by 0.12 Å, probably owing to the donation of a *d*_π electron of the nickel atom onto the vacant *d*_π orbitals of the germanium atoms. A similar shortening of the Ge–Ni bond (by 0.05–0.07 Å) was found in [Ph₃Ge–Ni(Cp)(–MGePh₃)]₂M [2]. Stronger shortening of these bonds in **1** may be explained by an increased contribution of the Ge atom 4*s*-orbital to the Ge–Ni bonding, resembling that observed for transition metal–carbon bonds in the complexes with μ-carbene (:CR₂) ligands. The same order of Ge–Ni bond shortening was also found in Cp(PPh₃)NiGeCl₃ (2.248 Å) [13] and [(OC)₃NiGe(OCMe₃)₂(μ-OCMe₃)₃] (2.283 Å) [14], where the Ge 4*s*-orbital contribution is increased owing to electronegative substituents attached to this atom.

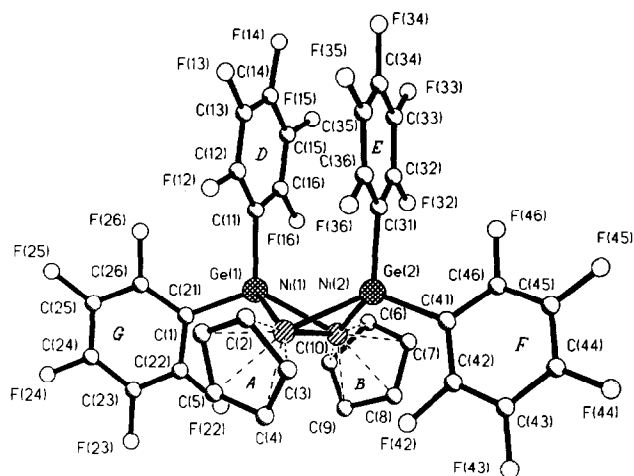


Fig. 1. The molecular structure and atom numbering scheme for $[(C_6F_5)_2GeNiCp]_2$.

Both cyclopentadienyl ligands are coordinated to their nickel atoms non-symmetrically, i.e. in a pseudo- π -allylic fashion, two atoms in each ring (C(4) and C(5) in ring *A*, C(8) and C(9) in ring *B*) being more distant from Ni than the other three by 0.07 Å on average. Such distortions are usually induced by non-equivalence of the monodentate *trans*-ligand in half-sandwich complexes with the M–C(Cp) distances lengthening in *trans* position to multiply bonded or strongly π -acceptor ligand (=O, CO, etc.) [15,16]. In the present case the longer Ni–C(Cp) distances are pseudo-*trans* to the Ge–Ni bonds and the shorter ones to the Ni–Ni bond, in agreement with the suggestion that the order of the Ge–Ni bonds is increased on account of the weakening of the Ni–Ni bond.

Both Ni atoms are situated at equal distances of 1.71 Å from the mean planes of their Cp-ligands, the latter forming a dihedral angle of 47.5°. Both 5-membered cycles are slightly non-planar. One atom of each cycle (C(4) in *A*, C(9) in *B*) is tilted out of the plane of the other four towards the coordinated nickel atom.

The Ge–C bond lengths are almost equivalent (average 1.980(7) Å) unlike the complex $Cp_2VGe(C_6F_5)_2Ge(C_6F_5)_2H$ where they vary over a wide range (1.98–2.05 Å) [9], probably owing to larger steric hindrances in the latter molecule. The perfluorophenyl cycles *D* and *E* are almost parallel (dihedral angle of 6.6°) and the distance between their planes (approx. 3.4 Å) corresponds to a close Van der Waals contact. Two other C_6F_5 -cycles are nearly normal to the former, with a dihedral angle of 78° between the *D* and *G* ring planes and 82° between the *E* and *F* planes. The molecular structure of **1** is shown in Fig. 1 (The X-ray investigation $[(C_6F_5)_2GeNiCp]_2$ was carried out by A.S. Batsanov). The bond distances and angles are given in Tables 1 and 2 and the atomic coordinates in Table 3.

The ESR monitoring of the reaction of $(C_6F_5)_2Ge(H)Ge(H)(C_6F_5)_2$ with nickelocene has shown that within a few minutes of heating of the reaction mixture at 60°C, the ESR signal appears as a singlet with $\Delta H \approx 100$ mT and g_i value of 2.30. These parameters are characteristic for Ni(d^8) complexes [17]. Most probably the

Table 1

Bond distances d (Å) in the molecule of **1**

Ni(1)–Ni(2)	2.492(1)	Ge(2)–C(41)	1.978(5)
Ni(1)–Ge(1)	2.555(1)	C(1)–C(2)	1.405(1)
Ni(1)–Ge(2)	2.253(1)	C(2)–C(3)	1.41(1)
Ni(2)–Ge(1)	2.263(1)	C(3)–C(4)	1.41(1)
Ni(2)–Ge(2)	2.256(1)	C(4)–C(5)	1.40(1)
Ni(1)–C(1)	2.066(6)	C(5)–C(1)	1.43(1)
Ni(1)–C(2)	2.056(6)	C(6)–C(7)	1.39(1)
Ni(1)–C(3)	2.073(6)	C(7)–C(8)	1.44(1)
Ni(1)–C(4)	2.140(6)	C(8)–C(9)	1.39(1)
Ni(1)–C(5)	2.115(6)	C(9)–C(10)	1.42(1)
Ni(2)–C(6)	2.045(6)	C(10)–C(6)	1.40(1)
Ni(2)–C(7)	2.063(6)	C–F	1.324(7)
		Min	
Ni(2)–C(8)	2.130(6)	Max	1.356(6)
Ni(2)–C(9)	2.142(6)	Median	1.343(7)
Ni(2)–C(10)	2.064(6)	C–C (Ph)	1.36(1)
		Min	
Ge(1)–C(11)	1.973(5)	Max	1.40(1)
Ge(1)–C(21)	1.989(5)	Median	1.38(1)
Ge(2)–C(31)	1.979(5)		

signal belongs to the $\text{CpNiGe}(\text{C}_6\text{F}_5)_2\text{Ge}(\text{C}_6\text{F}_5)_2\text{H}$ intermediate formed in the first stages of the reaction. Mixing of the reaction solution leads to a decrease of this signal. At the same time, a new signal appears. Its intensity achieves maximum in the middle reaction and then the signal decreases and completely disappears after 10 h. The parameters of the isotropic ESR spectrum of the new signal ($\Delta H \approx 1$ mT; $g_i = 2.048$) do not allow it to be connected with the definite oxidative state of Ni since the signal can be attributed both to $\text{Ni}^{3+}(d^7)$ and $\text{Ni}^+(d^9)$ complexes [18,19]. Taking into account the structure of the resulting product we suggest that this signal belongs to the complexes of CpNi with bis(pentafluorophenyl)germilene.

Table 2

Bond angles ω (°) in the molecules of **1**

Ni(2)–Ni(1)–Ge(1)	56.7(1)	Ni(1)–Ge(1)–C(21)	117.0(2)
Ni(2)–Ni(1)–Ge(2)	56.5(1)	Ni(2)–Ge(1)–C(11)	116.9(2)
Ni(2)–Ni(1)–Cp(a)	152.8(2)	Ni(2)–Ge(1)–C(21)	125.3(2)
Ge(1)–Ni(1)–Ge(2)	91.1(1)	C(11)–Ge(1)–C(21)	106.3(2)
Ge(1)–Ni(1)–Cp(a)	129.7(2)	Ni(1)–Ge(2)–Ni(2)	67.1(1)
Ge(2)–Ni(1)–Cp(a)	137.0(2)	Ni(1)–Ge(2)–C(31)	116.5(2)
Ni(1)–Ni(2)–Ge(1)	56.4(1)	Ni(2)–Ge(2)–C(41)	124.1(2)
Ni(1)–Ni(2)–Ge(2)	56.4(1)	Ni(2)–Ge(2)–C(31)	120.8(2)
Ni(1)–Ni(2)–Cp(6)	153.7(2)	Ni(2)–Ge(2)–C(41)	121.8(2)
Ge(1)–Ni(2)–Ge(2)	90.8(1)	C(31)–Ge(2)–C(41)	104.1(2)
Ge(1)–Ni(2)–Cp(6)	136.8(2)	C(Cp)–C(Cp)–C(Cp)	
Ge(2)–Ni(2)–Cp(b)	130.0(2)	Min	106.7(6)
Ni(1)–Ge(1)–Ni(2)	66.9(1)	Max	108.5(5)
Ni(1)–Ge(1)–C(11)	120.8(2)		

Table 3

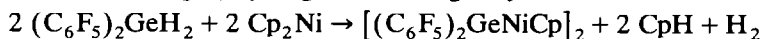
Atomic coordinates ($\times 10^4$, for H $\times 10^3$) in the structure of **1**

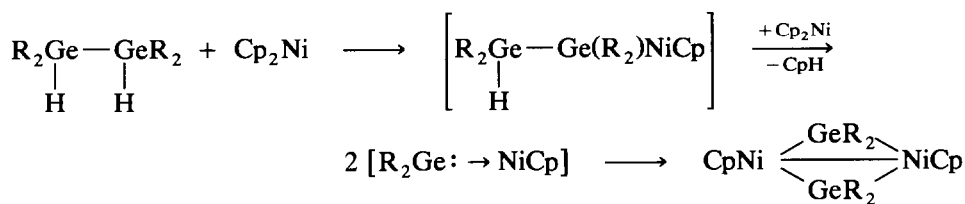
Atom	x	y	z	Atom	x	y	z
Ge(1)	1674(1)	1877(1)	623(1)	C(6)	3229(7)	3865(4)	882(3)
Ge(2)	-841(1)	3237(1)	745(1)	C(7)	2095(7)	4298(3)	1132(3)
Ni(1)	-355(1)	2019(1)	1165(1)	C(8)	1948(8)	4024(4)	1748(3)
Ni(2)	1569(1)	3089(1)	1090(1)	C(9)	3025(8)	3429(4)	1864(3)
F(12)	-550(4)	962(2)	-339(1)	C(10)	3796(7)	3317(4)	1324(3)
F(13)	-1002(4)	1139(2)	-1556(2)	C(11)	1485(6)	1873(3)	-281(2)
F(14)	760(4)	2158(2)	-2164(1)	C(12)	363(6)	1460(3)	-622(3)
F(15)	2977(4)	2984(2)	-1542(2)	C(13)	131(6)	1537(3)	-1251(3)
F(16)	3475(4)	2787(2)	-322(1)	C(14)	1016(6)	2044(4)	-1558(2)
F(22)	3651(4)	1586(2)	1843(1)	C(15)	2147(6)	2465(4)	-1243(3)
F(23)	5432(4)	377(2)	2219(2)	C(16)	2361(6)	2369(3)	-618(2)
F(24)	5888(4)	-883(2)	1463(2)	C(21)	3082(6)	992(3)	873(2)
F(25)	4435(5)	-932(2)	330(2)	C(22)	3816(6)	976(3)	1451(2)
F(26)	2630(4)	262(2)	-55(1)	C(23)	4744(7)	354(4)	1657(2)
F(32)	612(4)	4071(2)	-385(2)	C(24)	4967(6)	-285(4)	1276(3)
F(33)	-166(5)	3984(2)	-1591(2)	C(25)	4241(7)	-304(3)	703(3)
F(34)	-2602(5)	3114(3)	-2000(2)	C(26)	3315(6)	328(3)	511(2)
F(35)	-4248(4)	2344(3)	-1197(2)	C(31)	-1421(6)	3256(3)	-145(2)
F(36)	-3532(4)	2475(2)	22(2)	C(32)	-607(6)	3639(3)	-567(3)
F(42)	-1621(4)	3550(2)	2102(1)	C(33)	-983(7)	3609(4)	-1198(3)
F(43)	-3571(4)	4548(2)	2571(1)	C(34)	-2229(7)	3166(4)	-1397(2)
F(44)	-5168(4)	5606(2)	1832(2)	C(35)	-3065(7)	2781(4)	-993(3)
F(45)	-4700(4)	5685(2)	631(2)	C(36)	-2658(6)	2841(4)	-373(2)
F(46)	-2767(4)	4697(2)	146(1)	C(41)	-2148(6)	4052(3)	1092(2)
C(1)	-852(8)	808(3)	1227(3)	C(42)	-2381(6)	4069(3)	1717(2)
C(2)	-2167(7)	1259(4)	1120(3)	C(43)	-3367(6)	4576(3)	1972(2)
C(3)	-2271(6)	1796(4)	1614(3)	C(44)	-4157(6)	5122(3)	1602(3)
C(4)	-1046(7)	1652(4)	2039(2)	C(45)	-3923(6)	5150(3)	993(3)
C(5)	-144(7)	1058(3)	1800(3)	C(46)	-2934(6)	4632(3)	748(2)

Unfortunately the simultaneous presence of two paramagnetic compounds in the reaction solution prevents the study of the anisotropic ESR spectra.

The data obtained suggest a scheme for the process. We believe that in the first stage $(C_6F_5)_2Ge(H)Ge(H)(C_6F_5)_2$ reacts with Cp_2Ni to give cyclopentadiene and the unstable three-nuclear intermediate, $CpNiGe(C_6F_5)_2Ge(C_6F_5)_2H$. It should be noted that a similar vanadium complex, $Cp_2VGe(C_6F_5)_2Ge(C_6F_5)_2H$, obtained previously [9] is quite stable and may be isolated from the reaction mixture. In the case of the nickel complex, the intermediate obviously reacts with the second Cp_2Ni molecule yielding CpH and a paramagnetic complex of germilene with the $CpNi$ unit, $(C_6F_5)_2Ge:NiCp$. Formation of the latter may occur alternatively through the dinickel derivative $[CpNiGe(C_6F_5)_2Ge(C_6F_5)_2NiCp]$, decomposition of which must lead to the same germilene-nickel complex. In the last stage of the reaction a dimerization of this complex results in the final four-nuclear cluster **1** (Scheme 1).

Compound **1** can be synthesized also by the reaction of nickelocene with $(C_6F_5)_2GeH_2$. This reaction is completed in 3 h at 70°C in toluene with the formation of CpH , hydrogen and the germynickel cluster **1** in 85% yield:



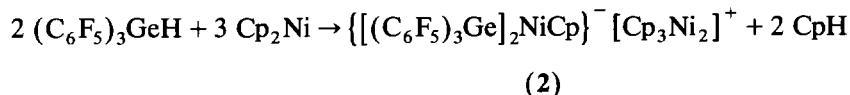


(R = C₆F₅)

Scheme 1.

It should be noted that ESR spectroscopy displayed the presence in the reaction mixture of the same unstable intermediates ($g_i = 2.048$ and 2.30), that were observed in the previous reaction.

Different types of complex have been obtained by interaction of nickelocene with (C₆F₅)₃GeH. Heating a mixture of these compounds at 70°C leads to CpH splitting, and formation of germylnickel ate-complexes $\{[(\text{C}_6\text{F}_5)_3\text{Ge}]_2\text{NiCp}\}^- [\text{Cp}_3\text{Ni}_2]^+$ (**2**) which are isolated in 30% yield as yellow-green dichroic crystals with m.p. 110°C. The product is stable in air and well soluble in THF, DME and toluene but insoluble in alkanes.



In the course of the reaction, an ESR signal was observed. Its intensity reaches a maximum in the middle of the reaction. The character of the ESR spectra ($\Delta H = 100$ mT, $g_i = 2.47$) corresponds to Ni²⁺ (d^8) configuration [17], the appearance of which may be explained by formation of the unstable complex $[(\text{C}_6\text{F}_5)_3\text{GeNiCp}]$. We believe that the formation of **2** is a result of disproportionating of this intermediate in the presence of Cp₂Ni.

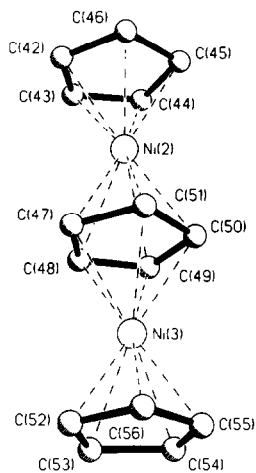


Fig. 2. The structure of the cation Cp₃Ni₂⁺.

Single-crystal X-ray analysis showed that the crystal of **2** has an ionic structure $[(C_6F_5)_3Ge]_2NiCp)^-[Cp_3Ni_2]^+$ and contains the solvating molecule of toluene.

The Cp_3Ni_2 cation is a triple-decker sandwich with eclipsed conformation of the three C-rings (Fig. 2) (The X-ray investigation of $[(C_6F_5)_3Ge]_2NiCp)^-[Cp_3Ni_2]^+$ was carried out by L.N. Zakharov). In contrast to **2**, in the structure of $[Cp_3Ni_2]^+[BF_4]^-$, the two Cp-ligands are approximately eclipsed, while the third Cp ligand is staggered with respect to the other two [20]. The relative orientation

Table 4

Selected bond lengths d (Å) in the structure of **2**

Ni(1)–Ge(1)	2.265(2)	C(17)–C(18)	1.40(2)	Ni(2)–C(44)	2.10(3)
Ni(1)–Ge(2)	2.263(3)	C(17)–F(17)	1.34(2)	Ni(2)–C(45)	2.09(4)
Ni(1)–C(37)	2.08(2)	C(18)–F(18)	1.33(2)	Ni(2)–C(46)	2.11(3)
Ni(1)–C(38)	2.11(2)	C(19)–C(20)	1.33(2)	Ni(2)–C(47)	2.16(3)
Ni(1)–C(39)	2.08(2)	C(19)–C(24)	1.39(3)	Ni(2)–C(48)	2.15(4)
Ni(1)–C(40)	2.15(2)	C(20)–C(21)	1.41(2)	Ni(2)–C(49)	2.15(3)
Ni(1)–C(41)	2.12(2)	C(20)–F(20)	1.35(2)	Ni(2)–C(50)	2.13(3)
Ge(1)–C(1)	1.98(1)	C(21)–C(22)	1.32(3)	Ni(2)–C(51)	2.11(5)
Ge(1)–C(7)	2.02(1)	C(21)–F(21)	1.34(2)	Ni(3)–C(47)	2.13(4)
Ge(1)–C(13)	2.03(1)	C(22)–C(23)	1.36(3)	Ni(3)–C(48)	2.11(4)
Ge(2)–C(19)	2.04(1)	C(22)–F(22)	1.34(2)	Ni(3)–C(49)	2.13(3)
Ge(2)–C(25)	2.03(1)	C(23)–C(24)	1.40(2)	Ni(3)–C(50)	2.18(3)
Ge(2)–C(31)	2.02(2)	C(23)–F(23)	1.34(3)	Ni(3)–C(51)	2.17(3)
C(1)–C(2)	1.39(3)	C(24)–F(24)	1.32(2)	Ni(3)–C(52)	2.10(3)
C(1)–C(6)	1.38(2)	C(25)–C(26)	1.38(2)	Ni(3)–C(53)	2.05(4)
C(2)–C(3)	1.40(3)	C(25)–C(30)	1.32(3)	Ni(3)–C(54)	2.08(3)
C(2)–F(2)	1.37(2)	C(26)–C(27)	1.37(2)	Ni(3)–C(55)	2.09(5)
C(3)–C(4)	1.38(3)	C(26)–F(26)	1.36(2)	Ni(3)–C(56)	2.09(5)
C(3)–F(3)	1.32(3)	C(27)–C(28)	1.33(3)	C(42)–C(43)	1.30(4)
C(4)–C(5)	1.32(3)	C(27)–F(27)	1.34(2)	C(42)–C(46)	1.35(5)
C(4)–F(4)	1.35(2)	C(28)–C(29)	1.37(3)	C(43)–C(44)	1.33(4)
C(5)–C(6)	1.38(2)	C(28)–F(28)	1.34(2)	C(44)–C(45)	1.37(5)
C(5)–F(5)	1.35(2)	C(29)–C(30)	1.42(3)	C(45)–C(46)	1.45(5)
C(6)–F(6)	1.32(2)	C(29)–F(29)	1.33(3)	C(47)–C(48)	1.33(4)
C(7)–C(8)	1.36(2)	C(30)–F(30)	1.34(2)	C(47)–C(51)	1.33(5)
C(7)–C(12)	1.39(2)	C(31)–C(32)	1.40(2)	C(48)–C(49)	1.33(4)
C(8)–C(9)	1.38(2)	C(31)–C(36)	1.37(2)	C(49)–C(50)	1.33(4)
C(8)–F(8)	1.34(2)	C(32)–C(33)	1.37(2)	C(50)–C(51)	1.48(4)
C(9)–C(10)	1.37(3)	C(32)–F(32)	1.33(2)	C(52)–C(53)	1.40(5)
C(9)–F(9)	1.33(2)	C(33)–C(34)	1.36(3)	C(52)–C(56)	1.31(6)
C(10)–C(11)	1.37(2)	C(33)–F(33)	1.34(2)	C(53)–C(54)	1.42(5)
C(10)–F(10)	1.34(2)	C(34)–C(35)	1.36(3)	C(54)–C(55)	1.30(5)
C(11)–C(12)	1.33(2)	C(34)–F(34)	1.36(2)	C(55)–C(56)	1.28(5)
C(11)–F(11)	1.36(2)	C(35)–C(36)	1.39(2)		
C(12)–F(12)	1.36(2)	C(35)–F(35)	1.34(2)		
C(13)–C(14)	1.37(2)	C(36)–F(36)	1.36(2)		
C(13)–C(18)	1.36(2)	C(37)–C(38)	1.36(5)		
C(14)–C(15)	1.39(2)	C(37)–C(41)	1.42(3)		
C(14)–F(14)	1.35(2)	C(38)–C(39)	1.37(3)		
C(15)–C(16)	1.38(2)	C(39)–C(40)	1.39(3)		
C(15)–F(15)	1.36(2)	C(40)–C(41)	1.31(4)		
C(16)–C(17)	1.35(3)	Ni(2)–C(42)	2.08(4)		
C(16)–F(16)	1.34(2)	Ni(2)–C(43)	2.11(4)		

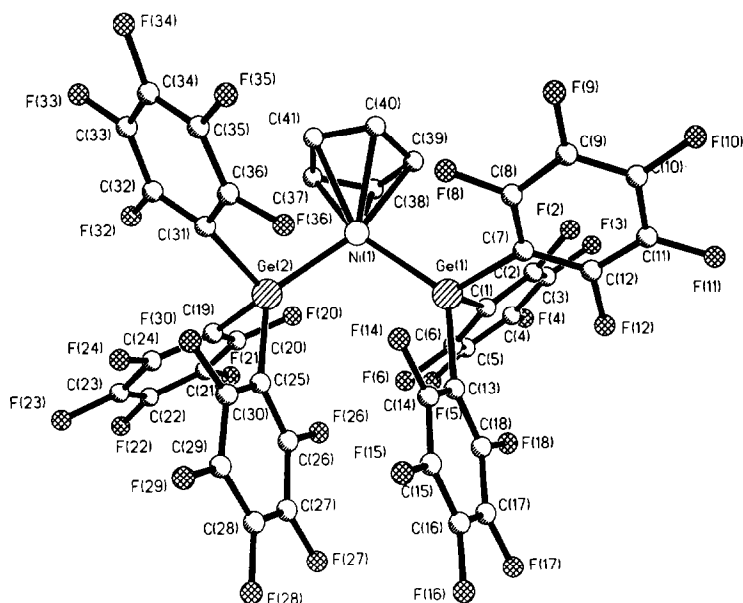


Fig. 3. The structure of the anion $\{[(C_6F_5)_3Ge]_2NiCp\}^-$.

of the three Cp-rings may be attributed to crystal packing forces. The maximum dihedral angle between the average planes of the Cp-rings is 5° , *i.e.* the three Cp-rings in the Cp_3Ni_2 cation are factually planar. The Ni–C bond lengths in the inner Cp-ring (2.11–2.16 Å (average 2.09 Å) for the Ni(2)–C bonds and 2.11–2.18 Å (average 2.14 Å) for the Ni(3)–C bonds) are somewhat longer than the Ni–C bond lengths in the other two Cp-rings (2.08–2.11 Å (average 2.09 Å) for the Ni(2)–C bonds and 2.05–2.11 Å (average 2.08 Å) for the Ni(3)–C bonds) (Table 4). These values are close to similar Ni–C distances in the Cp_3Ni_2 cation (2.17, 2.16 Å, 2.09, 2.11 Å, respectively) observed in the structure of $[Cp_3Ni_2]^+[BF_4]^-$ [20].

The $\{[(C_6F_5)_3Ge]_2NiCp\}$ anion contains a 3-membered Ge–Ni–Ge chain (Fig. 3). The Ni atom is also bonded to the Cp-ring in η^5 -fashion with an average Ni(1)–C(Cp) bond length of 2.11 Å. The Ni(1) atom is surrounded in a planar trigonal array by the centers of the cyclopentadienyl ligand, the Ge(1) and Ge(2) atoms. The Cp(center)Ni(1)Ge(1, 2) angles are 128.0° and 126.5° (Table 5). The Ge(1)–Ni(1)–Ge(2) angle is 127.5° . The Ni(1) atom deviates from the Ni(1)–Ge(1)–Cp(center) plane only by 0.03 Å. The Ni(1)–Ge(1) (2.265(2) Å), Ni(1)–Ge(2) (2.263(3) Å) bond lengths are shorter than the sum of the covalent radii of the Ni and Ge atoms (2.377 Å) by ~ 0.11 Å. As in **1**, this shortening is probably a consequence of a donation of a d_π electron of the Ni atom onto the vacant d_π orbitals of the Ge atoms.

It is interesting to note that the changing of one of the H atoms in $(C_6F_5)_2GeH_2$ or $(C_6F_5)_2Ge(H)Ge(H)(C_6F_5)_2$ with a Cp_2V unit dramatically decreases the reactivity of the remaining Ge–H group. Thus, $Cp_2VGe(C_6F_5)_2H$ and $Cp_2VGe(C_6F_5)_2Ge(C_6F_5)_2H$ do not react with Cp_2Ni even with prolonged heating of the mixture of these reagents at $80^\circ C$. Earlier we observed a similar decrease in the reactivity of vanadium derivatives in comparison with $(C_6F_5)_2GeH_2$ and

Table 5

Selected bond angles ω ($^\circ$) in the structure of **2**

Ge(1)–Ni(1)–Ge(2)	105.3(1)	C(19)–C(20)–F(20)	120.5(14)
Ni(1)–Ge(1)–C(1)	102.7(4)	C(21)–C(20)–F(20)	116.2(14)
Ni(1)–Ge(1)–C(7)	117.9(4)	C(20)–C(21)–C(22)	119.5(16)
Ni(1)–Ge(1)–C(13)	129.7(5)	C(20)–C(21)–F(21)	119.4(18)
C(1)–Ge(1)–C(7)	105.2(6)	C(22)–C(21)–F(21)	120.9(14)
C(1)–Ge(1)–C(13)	104.3(5)	C(21)–C(22)–C(23)	119.9(16)
C(7)–Ge(1)–C(13)	94.6(5)	C(21)–C(22)–F(22)	120.5(18)
Ni(1)–Ge(2)–C(19)	118.4(5)	C(23)–C(22)–F(22)	119.6(21)
Ni(1)–Ge(2)–C(25)	129.3(5)	C(22)–C(23)–C(24)	119.6(20)
Ni(1)–Ge(2)–C(31)	102.9(5)	C(22)–C(23)–F(23)	120.3(15)
C(19)–Ge(2)–C(25)	93.3(6)	C(24)–C(23)–F(23)	120.0(17)
C(19)–Ge(2)–C(31)	106.6(6)	C(19)–C(24)–C(23)	121.1(16)
C(25)–Ge(2)–C(31)	104.2(6)	C(19)–C(24)–F(24)	122.6(13)
Ge(1)–C(1)–C(2)	127.8(9)	C(23)–C(24)–F(24)	116.3(18)
Ge(1)–C(1)–C(6)	118.3(13)	Ge(2)–C(25)–C(26)	116.9(12)
C(2)–C(1)–C(6)	112.2(13)	Ge(2)–C(25)–C(30)	128.1(11)
C(1)–C(2)–C(3)	124.8(14)	C(26)–C(25)–C(30)	114.9(13)
C(1)–C(2)–F(2)	119.3(13)	C(25)–C(26)–C(27)	125.2(16)
C(3)–C(2)–F(2)	115.8(17)	C(25)–C(26)–F(26)	118.4(13)
C(2)–C(3)–C(4)	117.0(20)	C(27)–C(26)–F(26)	116.3(13)
C(2)–C(3)–F(3)	119.9(16)	C(26)–C(27)–C(28)	117.2(15)
C(4)–C(3)–F(3)	123.0(18)	C(26)–C(27)–F(27)	120.3(16)
C(3)–C(4)–C(5)	121.4(17)	C(28)–C(27)–F(27)	122.5(13)
C(3)–C(4)–F(4)	116.4(21)	C(27)–C(28)–C(29)	122.1(16)
C(5)–C(4)–F(4)	122.2(17)	C(27)–C(28)–F(28)	120.1(16)
C(4)–C(5)–C(6)	119.5(15)	C(29)–C(28)–F(28)	117.8(19)
C(4)–C(5)–F(5)	119.6(15)	C(28)–C(29)–C(30)	117.5(19)
C(6)–C(5)–F(5)	121.0(17)	C(28)–C(29)–F(29)	122.7(17)
C(1)–C(6)–C(5)	125.0(17)	C(30)–C(29)–F(29)	119.8(15)
C(1)–C(6)–F(6)	118.2(13)	C(25)–C(30)–C(29)	123.1(15)
C(5)–C(6)–F(6)	116.8(13)	C(25)–C(30)–F(30)	122.8(14)
Ge(1)–C(7)–C(8)	121.9(12)	C(29)–C(30)–F(30)	114.0(17)
Ge(1)–C(7)–C(12)	123.1(9)	Ge(2)–C(31)–C(32)	124.0(10)
C(8)–C(7)–C(12)	115.0(13)	Ge(2)–C(31)–C(36)	118.1(13)
C(7)–C(8)–C(9)	123.5(17)	C(32)–C(31)–C(36)	114.8(12)
C(7)–C(8)–F(8)	121.4(14)	C(31)–C(32)–C(33)	121.8(14)
C(9)–C(8)–F(8)	115.0(13)	C(31)–C(32)–F(32)	120.3(12)
C(8)–C(9)–C(10)	118.4(14)	C(33)–C(32)–F(32)	117.8(15)
C(8)–C(9)–F(9)	122.1(18)	C(32)–C(33)–C(34)	120.7(17)
C(10)–C(9)–F(9)	119.6(16)	C(32)–C(33)–F(33)	120.8(14)
C(9)–C(10)–C(11)	119.4(15)	C(34)–C(33)–F(33)	118.5(15)
C(9)–C(10)–F(10)	120.7(15)	C(33)–C(34)–C(35)	120.5(15)
C(11)–C(10)–F(10)	119.9(18)	C(33)–C(34)–F(34)	121.5(18)
C(10)–C(11)–C(12)	120.4(18)	C(35)–C(35)–F(34)	118.0(16)
C(10)–C(11)–F(11)	118.5(14)	C(34)–C(35)–C(36)	118.0(15)
C(12)–C(11)–F(11)	121.1(14)	C(34)–C(35)–F(35)	123.0(14)
C(7)–C(12)–C(11)	122.9(13)	C(36)–C(35)–F(35)	118.9(16)
C(7)–C(12)–F(12)	118.4(12)	C(31)–C(36)–C(35)	124.0(16)
C(11)–C(12)–F(12)	118.6(15)	C(31)–C(36)–F(36)	118.4(13)
Ge(1)–C(13)–C(14)	116.3(9)	C(35)–C(36)–F(36)	117.6(13)
Ge(1)–C(13)–C(18)	126.9(11)	C(38)–C(37)–C(41)	108.2(21)
C(14)–C(13)–C(18)	116.8(12)	C(37)–C(38)–C(39)	107.1(21)
C(13)–C(14)–C(15)	122.6(13)	C(38)–C(39)–C(40)	107.9(23)
C(13)–C(14)–F(14)	121.8(12)	C(39)–C(40)–C(41)	109.3(19)

Table 5 (continued)

C(15)–C(14)–F(14)	115.5(14)	C(37)–C(41)–C(40)	107.2(26)
C(14)–C(15)–C(16)	118.3(16)	C(43)–C(42)–C(46)	110.8(26)
C(14)–C(15)–F(15)	121.3(13)	C(42)–C(43)–C(44)	111.7(29)
C(16)–C(15)–F(15)	120.4(15)	C(43)–C(44)–C(45)	106.3(24)
C(15)–C(16)–C(17)	121.0(15)	C(44)–C(45)–C(46)	107.5(27)
C(15)–C(16)–F(16)	119.4(18)	C(42)–C(46)–C(45)	103.5(29)
C(17)–C(16)–F(16)	119.4(16)	C(48)–C(47)–C(51)	108.3(23)
C(16)–C(17)–C(18)	118.6(14)	C(47)–C(48)–C(49)	110.6(26)
C(16)–C(17)–F(17)	121.4(13)	C(48)–C(49)–C(50)	110.4(23)
C(18)–C(17)–F(17)	119.9(15)	C(49)–C(50)–C(51)	103.9(22)
C(13)–C(18)–C(17)	122.5(14)	C(47)–C(51)–C(50)	106.8(25)
C(13)–C(18)–F(18)	122.3(13)	C(53)–C(52)–C(56)	105.7(27)
C(17)–C(18)–F(18)	115.0(12)	C(52)–C(53)–C(54)	106.6(30)
Ge(2)–C(19)–C(20)	123.8(15)	C(53)–C(54)–C(55)	103.2(24)
Ge(2)–C(19)–C(24)	119.3(11)	C(54)–C(55)–C(56)	114.2(33)
C(20)–C(19)–C(24)	116.4(14)	C(52)–C(56)–C(55)	110.2(33)
C(19)–C(20)–C(21)	123.3(19)	Cp(center)–Ni(1)–Ge(1)	126.5
		Cp(center)–Ni(1)–Ge(2)	128.0

$(C_6F_5)_2Ge(H)Ge(H)(C_6F_5)_2$ in the reactions with R_2Hg ($R = Me, Et, Ph, CH_2Ph, N(SiMe_3)_2, GeEt_3$).

Experimental

All reactions were carried out in evacuated sealed ampoules. The pentafluorophenyl germanies were prepared by previously reported methods [21,22]. Melting points (given without corrections) were determined using evacuated capillaries. IR spectra were recorded on a Perkin–Elmer-577 spectrophotometer. ESR data were obtained with a Bruker ER-200 D SRC spectrometer. GLC analyses of volatile products were carried out on a Tsvet-104 chromatograph with a catarometer detector.

X-Ray diffraction study

The diffraction experiment was carried out on an unstable crystal of **1** at 173 K and on a crystal of **2** at 293 K with a fourcircle computer-controlled Siemens R3/PC diffractometer, using graphite-monochromated $Mo-K_\alpha$ radiation. SHELXTL PLUS programs were used for the calculations.

Crystal data of 1. $C_{34}H_{10}F_{20}Ge_2Ni_2$, monoclinic, space group $P2_1/n$, $a = 8.979(2)$, $b = 16.606(4)$, $c = 21.889 \text{ \AA}$, $\beta = 94.00(2)^\circ$, $V = 3256 \text{ \AA}^3$, $Z = 4$, $D_{calc.} = 2.165 \text{ g/cm}^3$, $\mu(Mo-K_\alpha) = 30.9 \text{ cm}^{-1}$.

Crystal data of 2. $C_{63}H_{28}F_{30}Ge_2Ni_3$, triclinic, space group $P\bar{1}$, $a = 12.148(2)$, $b = 14.680(3)$, $c = 19.001(4) \text{ \AA}$, $\alpha = 69.89(2)$, $\beta = 76.82(2)$, $\gamma = 73.10(2)^\circ$, $V = 3013 \text{ \AA}^3$, $Z = 2$, $D_{calc.} = 1.727 \text{ g/cm}^3$, $\mu(Mo-K_\alpha) = 20.2 \text{ cm}^{-1}$.

The intensities of the unique reflections with $I > 2\sigma(I)$ (4252 for **1** and 6555 for **2**) were measured by the θ – 2θ scan technique ($2\theta < 50^\circ$ (**1**) and 52° (**2**)). The structures were solved by direct methods. Least-squares refinement of all non-hydrogen atoms in an anisotropic approximation (including all hydrogen atoms as fixed isotropic contributions in calculated position) converged to $R = 0.037$, $R_w =$

Table 6

Atom coordinates ($\times 10^4$) and temperature factors ($\text{\AA}^2 \times 10^3$) in the structure of **2**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ^a
Ni(1)	-704(2)	2377(1)	3315(1)	39(1)
Ge(1)	839(1)	3071(1)	2765(1)	35(1)
Ge(2)	-1223(1)	2034(1)	2379(1)	37(1)
C(1)	275(11)	4416(9)	2906(7)	36(5)
C(2)	447(13)	4721(11)	3482(9)	55(7)
C(3)	-150(16)	5620(14)	3632(10)	67(9)
C(4)	-991(18)	6227(12)	3190(11)	76(9)
C(5)	-1236(14)	5966(11)	2655(9)	54(7)
C(6)	-602(13)	5086(10)	2512(8)	45(6)
C(7)	2314(11)	2461(9)	3241(8)	38(6)
C(8)	2572(12)	1490(11)	3669(9)	52(7)
C(9)	3510(16)	1073(12)	4062(9)	66(8)
C(10)	4245(12)	1656(14)	3999(9)	60(8)
C(11)	4054(13)	2616(13)	3530(8)	56(8)
C(12)	3139(11)	2991(11)	3158(8)	43(6)
C(13)	1668(10)	3318(10)	1686(7)	34(5)
C(14)	2149(13)	2493(11)	1440(8)	47(7)
C(15)	2775(13)	2545(12)	723(9)	56(7)
C(16)	2864(14)	3469(16)	231(9)	66(9)
C(17)	2422(12)	4300(12)	454(8)	50(7)
C(18)	1801(11)	4212(10)	1184(8)	40(6)
C(19)	-2749(12)	2846(11)	1991(9)	48(7)
C(20)	-3297(13)	3731(11)	2082(9)	49(7)
C(21)	-4285(14)	4332(11)	1745(9)	58(7)
C(22)	-4675(15)	4036(15)	1283(10)	69(9)
C(23)	-4162(14)	3126(14)	1175(9)	62(8)
C(24)	-3189(13)	2532(12)	1525(9)	54(7)
C(25)	-360(12)	2057(11)	1325(8)	42(6)
C(26)	-281(13)	2978(1)	820(9)	50(7)
C(27)	228(13)	3144(13)	78(8)	55(7)
C(28)	666(15)	2354(14)	-173(9)	66(8)
C(29)	607(15)	1413(15)	278(11)	72(10)
C(30)	89(14)	1298(12)	1042(10)	60(8)
C(31)	-1393(13)	616(11)	2863(8)	46(6)
C(32)	-2455(14)	352(11)	3196(8)	51(7)
C(33)	-2511(15)	-542(13)	3730(9)	59(8)
C(34)	-1524(19)	-1235(13)	3930(9)	70(9)
C(35)	-466(17)	-1046(12)	3606(9)	67(8)
C(36)	-426(13)	-111(12)	3091(8)	51(7)
C(37)	-2285(17)	2436(23)	4031(9)	90(13)
C(38)	-1770(20)	3106(17)	4100(11)	80(11)
C(39)	-729(18)	2581(16)	4348(8)	74(10)
C(40)	-662(17)	1572(15)	4493(9)	72(9)
C(41)	-1566(22)	1469(20)	4282(11)	87(13)
F(2)	1251(8)	4115(7)	3948(5)	71(5)
F(3)	117(12)	5857(9)	4176(7)	110(7)
F(4)	-1562(12)	7091(8)	3343(8)	116(7)
F(5)	-2116(9)	6555(8)	2265(6)	88(5)
F(6)	-881(7)	4867(7)	1967(5)	58(4)
F(8)	1898(8)	871(7)	3750(6)	78(5)
F(9)	3711(9)	135(8)	4501(6)	92(6)
F(10)	5158(8)	1293(8)	4385(5)	85(5)

Table 6 (continued)

Atom	x	y	z	U^a
F(11)	4802(8)	3183(9)	3470(6)	88(6)
F(12)	2968(8)	3953(7)	2708(5)	66(4)
F(14)	2090(7)	1563(6)	1897(5)	56(4)
F(15)	3245(9)	1715(8)	497(6)	81(5)
F(16)	3474(10)	3537(9)	-464(5)	93(6)
F(17)	2534(9)	5198(7)	-14(5)	76(5)
F(18)	1415(7)	5058(6)	1366(5)	54(4)
F(20)	-2934(7)	4092(7)	2529(6)	68(5)
F(21)	-4766(8)	5229(8)	1851(7)	88(6)
F(22)	-5591(9)	4610(8)	937(7)	103(6)
F(23)	-4552(9)	2820(9)	711(6)	93(6)
F(24)	-2705(8)	1678(7)	1368(5)	68(5)
F(26)	-738(7)	3799(6)	1065(5)	57(4)
F(27)	296(9)	4069(8)	-358(5)	86(5)
F(28)	1200(11)	2463(11)	-889(6)	112(7)
F(29)	1056(11)	612(9)	36(6)	105(6)
F(30)	32(10)	356(7)	1443(6)	88(5)
F(32)	-3453(7)	1003(7)	3034(5)	67(4)
F(33)	-3541(10)	-770(8)	4054(6)	88(6)
F(34)	-1564(12)	-2113(7)	4484(6)	98(6)
F(35)	534(11)	-1699(8)	3786(6)	98(6)
F(36)	636(7)	98(7)	2811(5)	67(4)
Ni(2)	4821(2)	9123(2)	1813(1)	66(1)
Ni(3)	2828(2)	7938(2)	1506(2)	70(1)
C(42)	6113(27)	8942(19)	2433(18)	117(16)
C(43)	5262(21)	9651(23)	2589(18)	119(18)
C(44)	5024(26)	10408(17)	1973(20)	117(16)
C(45)	5830(31)	10179(26)	1387(17)	133(20)
C(46)	6562(20)	9206(31)	1692(27)	145(27)
C(47)	4265(23)	7765(15)	2043(22)	116(17)
C(48)	3369(30)	8429(28)	2265(14)	124(19)
C(49)	3074(18)	9221(19)	1679(19)	88(14)
C(50)	3775(27)	9119(20)	1050(13)	106(14)
C(51)	4586(21)	8135(26)	1304(21)	133(20)
C(52)	2253(30)	6622(19)	1812(22)	128(18)
C(53)	1341(27)	7418(30)	1931(21)	140(22)
C(54)	1214(22)	8137(17)	1209(23)	115(16)
C(55)	1999(32)	7716(29)	750(18)	123(20)
C(56)	2615(28)	6872(26)	1079(26)	142(23)
C(57)	3808(10)	6066(10)	4014(8)	136(6) ^b
C(58)	4967(10)	5564(10)	3890(8)	130(6) ^b
C(59)	5847(10)	5917(10)	3998(8)	134(6) ^b
C(60)	5567(10)	6771(10)	4230(8)	150(6) ^b
C(61)	4408(10)	7272(10)	4354(8)	143(6) ^b
C(62)	3528(10)	6920(10)	4246(8)	122(6) ^b
C(63)	2898(17)	5734(15)	3909(11)	169(6) ^b

^a Equivalent isotropic U defined as one-third of the trace of the orthogonalised U_{ij} tensor. ^b Atomic coordinates of a solvating toluene molecule refined as a rigid group (C(63), first atom in the group) with fixed geometry of the Ph-ring and constraints of C(57)–C(63) bond length.

0.036 for **1** and $R = 0.090$, $R_w = 0.090$ for **2** ($w = \sigma^{-2}$ weighting scheme). The solvating toluene molecule in **2** was refined as a rigid group (C(63), first atom in group) with fixed geometry of the Ph-ring and constraints on the C(Ph)–C(Me) (C(57)–C(63)) bond length. Atomic coordinates of **1** and **2** are listed in Tables 3 and 6, respectively.

Reaction $(C_6F_5)_2Ge(H)Ge(H)(C_6F_5)_2$ with Cp_2Ni

To a solution of Cp_2Ni (0.255 g, 0.135 mmol) in toluene (10 ml) was added a solution of dihydrodigermane (0.55 g, 0.067 mmol) in toluene (30 ml). After 1 h at 80°C, the reaction mixture became red and an ESR signal ($\Delta H = 100$ mT, $g_i = 2.30$) was detected. After 2 h, a new signal ($\Delta H = 1$ mT, $g_i = 2.048$) was recorded which disappeared in 10 h. The volatile products and solvent evaporated *in vacuo*. In the volatile products, cyclopentadiene (0.08 g, 90%) was found by GLC. Recrystallization of the solid from toluene gave 0.563 g (85%) of dark-red crystals of **1**, m.p. 270–272°C (decomp.). Anal. Found: C, 38.45; H, 1.33. $C_{34}H_{10}F_{20}Ge_2Ni_2$ calc.: C, 38.49; H, 0.95% IR (Nujol): 3080vw, 1640m, 1500m, 1280m, 1080s, 960s, 830s, 810m, 800w, 610m, 370m cm^{-1} .

The reaction of $(C_6F_5)_2GeH_2$ with Cp_2Ni was carried out in a similar way. From 1.25 g (3.09 mmol) of bis(pentafluorophenyl)germane and 0.582 g (3.09 mmol) of nickelocene, 65 ml (95%) of hydrogen, 0.19 g of CpH and 1.38 g (84%) of **1** were obtained.

Reaction $(C_6F_5)_3GeH$ with Cp_2Ni

To a solution of Cp_2Ni (0.345 g, 1.83 mmol) in THF (10 ml) was added a solution of germane (0.7 g, 1.22 mmol) in THF (15 ml). After 2 h at 70°C, the reaction mixture became brown and an ESR signal ($\Delta H = 100$ mT, $g_i = 2.47$) was detected. After 4 h, the signal disappeared. In the volatile products, cyclopentadiene (0.07 g, 92%) was found by GLC. CpH and THF evaporated *in vacuo*. The residue was recrystallized from toluene and 0.3 g (30%) of yellow-green crystals of **2**, m.p. 110°C, were obtained. Anal. Found: C, 44.87; H, 1.01. $C_{63}H_{28}F_{30}Ge_2Ni_3$ calc.: C, 45.14; H, 1.68%. IR (Nujol): 3080vw, 1640m, 1500s, 1280m, 1080s, 960s, 830s, 820m, 810m, 800m, 610w, 375w cm^{-1} .

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