

The reactions were carried out under conditions excluding the presence of moisture and air oxygen. A toluene solution of a $\text{WCl}_6\text{--SCB--1-hexene--C}_{60}$ (2 : 4 : 25 : 1) mixture was heated in a sealed tube (5 h, 100 °C). After removing the liquid products by condensation, the solid residue was treated with methanol to decompose tungsten chlorides. Methanol was removed, the dark brown residue formed was twice washed with hexane and evaporated *in vacuo* (to $1 \cdot 10^{-1}$ Torr). Toluene was added, and a reddish-brown solution and an insoluble residue were obtained. Adduct **1** was isolated using TLC (Al_2O_3 , benzene--hexane (2 : 1) as the eluent, R_f 0.77) followed by extraction with toluene.

Found (%): C, 98.62; H, 1.38. $\text{C}_{65}\text{H}_{10}$. Calculated (%): C, 98.73; H, 1.27. IR (KBr), ν/cm^{-1} : 2960, 2930, 2890, 2860, 1660, 1465, 1455, 1435, 1375, 1187, 710, 581, 535 (strong bands of stretching and bending vibrations of the aliphatic C--H bonds and vibrations of the carbon skeleton of C_{60}). ^1H NMR (300 MHz, CDCl_3), δ : 0.86 (m, 3 H, CH_3); 1.22 (m, 4 H, CH_2); 2.04 (m, 2 H, CH_2); 5.29 (m, 1 H, CH) (H aliph.). ^{13}C NMR (75.47 MHz, C_6D_6 , 0.03 mol mL^{-1} $\text{Cr}(\text{acac})_3$), δ : 138--160 (C fullerene); 14.23, 22.72, 29.33, 32.18, 40.30, 72.30 (C aliph.).

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Interaction of perfluorocyclohexene with hydrazine hydrate

M. D. Bargamova* and G. G. Bargamov

A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences,
28 ul. Vavilova, 117813 Moscow, Russian Federation.
Fax: +7 (095) 135 5085

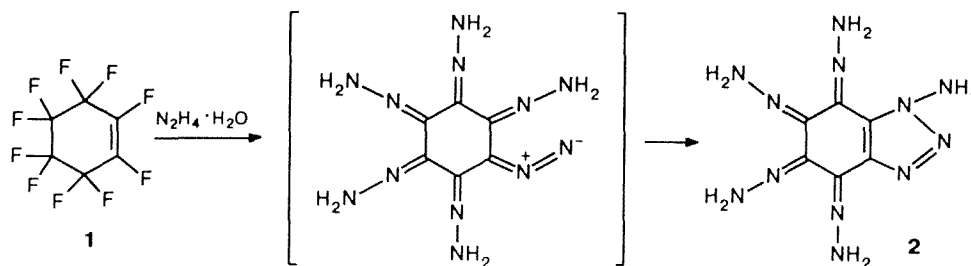
It is known that perhalocycloolefins, for example perfluorocyclobutene and 1,2-dichlorohexafluorocyclopentene, are transformed to polyhydrazones of cyclic polyketones under the action of an excess of hydrazine hydrate under mild conditions.¹ Unexpectedly it appeared that perfluorocyclohexene (**1**) (for the preparation see Ref. 2) under similar conditions is completely transformed to tetrahydrazone of 1-amino-1,2,3-triazabicyclo[4,3,0]nona-2,4-dien-5,6,7,8-tetraone (**2**) (Scheme 1).

On the basis of IR spectroscopy and mass spectrometry data and comparison of these data with parameters of the known structural analogs,^{1,3} structure **2** was assigned to the product obtained. The IR spectrum of compound **2** (unlike that from cyclic polyhydrazones¹) contains the absorption bands at 1480--1520 and

1620 cm^{-1} corresponding to the vibrations of 1,2,3-triazolic aromatic cycle (cf. Ref. 3) and fragmentation of ions in a mass spectrum of **2** is typical for compounds of similar structure (cf. Refs. 1 and 3). The structure of compound **2** will be refined by chemical methods.

Perfluorocyclohexene **1** (3 g, 0.011 mol) was added dropwise to a hydrazine hydrate solution (12 g, 0.23 mol) in 200 mL of ethanol with stirring. The homogeneous mixture was kept at 20 °C until formation of the precipitate ceased (48 h). The crystals precipitated were filtered, washed with a boiling water, acetonitrile and ether, and crystallized from a mixture of $\text{EtOH--N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (1 : 1) to give 2.3 g (80 %) of compound **2**, decomp. at 205 °C. IR, ν/cm^{-1} : 1480, 1515 s (N=N); 1550, 1570 m (C=N); 1620 m (C=C); 3200, 3380, 3400 s (NH_2). Found (%): C, 28.15; H, 4.22; N, 67.25. $\text{C}_6\text{H}_{10}\text{N}_{12}$. Calculated (%): C, 28.80; H, 4.00; N, 67.20. MS, m/z (I_{rel} (%)): 250 $[\text{M}]^+$ (5.77); 234 $[\text{M--NH}_2]^+$ (1.92); 222

Scheme 1



$[\text{M}-\text{N}_2]^+$ (3.80); 207 $[\text{M}-\text{N}_3\text{H}]^+$ (1.92); 194 $[\text{M}-2\text{N}_2]^+$ (11.54); 179 $[\text{M}-2\text{N}_2, \text{NH}]^+$ (11.50); 164 $[\text{M}-2\text{N}_2, \text{N}_2\text{H}_2]^+$ (40.38); 149 $[\text{M}-2\text{N}_2, \text{N}_3\text{H}_3]^+$ (11.58); 134 $[\text{M}-2\text{N}_2, \text{N}_4\text{H}_4]^+$ (9.62); 120 $[\text{M}-2\text{N}_2, \text{N}_5\text{H}_5]^+$ (15.38); 105 $[\text{M}-2\text{N}_2, \text{N}_6\text{H}_6]^+$ (38.46); 92 $[\text{C}_6\text{H}_5\text{NH}]$ (57.69); 78 $[\text{C}_6\text{H}_6]$ (53.82); 77 $[\text{C}_6\text{H}_5]$ (57.69); 76 $[\text{C}_6\text{H}_4]$ (47.68); 68 $[\text{C}_2\text{N}_3\text{H}_2]$ (65.38); 52 $[\text{C}_4\text{H}_4]$ (100); 42 $[\text{C}=\text{N}-\text{NH}_2]$ (7.82); 32 $[\text{N}_2\text{H}_4]$ (89.20); 28 $[\text{N}_2]$ (100); 17 $[\text{NH}_3]$ (100).

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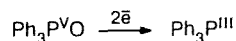
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Electrochemical deoxygenation of triphenylphosphine oxide

V. V. Yanilkin,* V. S. Gromakov, and F. F. Nigmatzyanov

A. E. Arbuzov Institute of Organic and Physical Chemistry, Kazan' Scientific Center of the Russian Academy of Sciences,
8 ul. Akad. Arbuzova, 420083 Kazan', Russian Federation.
Fax: +7 (843 2) 75 2253

Electrochemical reduction of phosphine oxides directly at the electrode involves cleavage of the P—C bond¹ or hydrogenation of the aromatic fragment.² We found that electrolysis carried out with a soluble aluminum anode in a 0.1 M solution of anhydrous aluminum trichloride as the supporting electrolyte leads to the reduction of the phosphoryl group of the triphenylphosphine oxide and yields triphenylphosphine.



The electrolysis of 3.8 g of $\text{Ph}_3\text{P}=\text{O}$ was carried out in a diaphragmless glass cell ($V = 50$ mL) with a graphite cathode in an acetonitrile—benzene (2 : 1) mixture at a constant current of 0.4 A for 2.5 h. When the electrolysis was completed, the solution was treated with 50 mL of