According to the published data,[2] the isosteric heat of the adsorption of xenon on the NaX zeolite at 165 K varies from 21.1 to 24.8 kJ mol^{-1} as a function of the amount adsorbed. These values are close to the isochoric heats of vaporization and sublimation mentioned above. It can be suggested that the cavities of the NaX zeolite act as a reservoir for the adsorbed xenon ensuring that its volume remains constant when the temperature changes. Evidently, the conditions in this case are not strictly isochoric, since the volume of zeolite crystals changes slightly both in adsorption and following an increase in the temperature. These corrections can be found from experimental data on the deformation of zeolites.

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Cyclopropanation of buckminsterfullerene *via* olefin metathesis reaction

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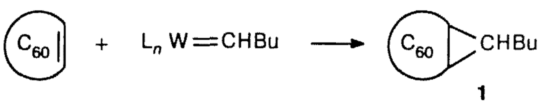
Buckminsterfullerene C_{60} is known to enter into cycloaddition reactions with carbenes during the interactions with diazocompounds, azides, chlorocarbenes,[1] and Fischer carbenes.[2] In this case methanofullerene and methanoannulene (fulleroid) derivatives are formed depending on the type of substituents in carbene.[3]

A catalytic reaction of olefin metathesis, which is the redistribution of alkylidene moieties resulting in new unsaturated molecules, occurs involving metalcarbene intermediates.[4]

$$L_nM{=}CHR^1 + R^2CH{=}CHR^3 \longrightarrow R^2CH{=}CHR^1 + L_nM{=}CHR^3$$

However, cyclopropanation of unsaturated compounds containing electron-withdrawing double bonds can take place under metathesis conditions.[5] For this reason, we should expect that tungsten carbene complexes formed in the metathesis reaction of 1-hexene in the presence of a homogeneous system WCl_6—1,1,3,3-tetramethyl-1,3-disilacyclobutane (SCB)[6], can add to electron-withdrawing bonds of fullerene to yield methanofullerene.



1

The compound **1** obtained was characterized by spectral methods (IR, ^1H and ^{13}C NMR spectroscopy); its composition is consistent with the elemental analysis data.

The methanofullerene structure of adduct **1**, formed as a result of carbene addition to the (6,6) junction of fullerene is supported by the presence of signals of the bridgehead and foot carbons of the cyclopropane moiety (at δ 40.30 and 72.30) in its ^{13}C NMR spectrum, and the signal of the methine proton at δ 5.29 in its ^1H NMR spectrum. These data are consistent with the NMR spectral parameters of methanofullerenes.[3]

The result presented is the first example of the use of metathesis reaction for the synthesis of fullerene derivatives.

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The reactions were carried out under conditions excluding the presence of moisture and air oxygen. A toluene solution of a WCl_6—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. $C_{65}H_{10}$. 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}). ^{1}H 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} Cr(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

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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 polyhydrazones[1]) 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 EtOH—$N_2H_4 \cdot H_2O$ (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. $C_6H_{10}N_{12}$. Calculated (%): C, 28.80; H, 4.00; N, 67.20. MS, *m/z* (I_{rel} (%)): 250 [M]$^+$ (5.77); 234 [M$-$NH$_2$]$^+$ (1.92); 222

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