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Reaction of Perfluoro-1-methylbenzocyclobutene with Pentafluorobenzene in SbF_5 Medium

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Abstract—A reaction of perfluoro-1-methylbenzocyclobutene with pentafluorobenzene in SbF_5 medium followed by treating the reaction mixture with water gave rise to perfluoro-1-methyl-1-phenylbenzocyclobutene, perfluoro-1-methyl-2-phenylbenzocyclobutene, 2-hydroxyperfluoro-1-methyl-2-phenylbenzocyclobutene, and also to small amounts of 1-(2-trifluoromethyltetrafluorophenyl)-1-pentafluorophenyl-2,2,2-trifluoroethane, and perfluoro-1-(2-methylphenyl)-1-phenylethylene. In a crystal of (*E*)-2-hydroxyperfluoro-1-methyl-2-phenylbenzocyclobutene for a dimer molecular pair a π-stacking interaction between pentafluorophenyl groups was found.

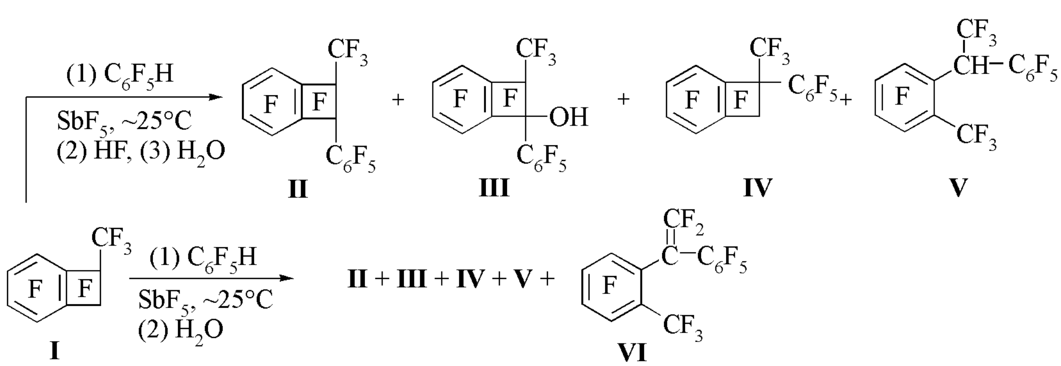
We investigated formerly the pentafluorophenylation of perfluorobenzocycloalkenes with pentafluorobenzene in SbF_5 medium providing perfluoro-1--phenylbenzocycloalkenes [1]. In reactions of 1-phenylindane and 1-arylbenzocyclobutenes with antimony pentafluoride cationoid skeleton rearrangements were observed in the series of polyfluoroarylbenzocycloalkenes [2].

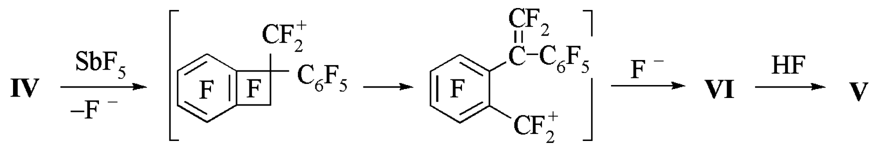
In extension of the investigation of pentafluorophenylation of polyfluorobenzoalkenes, and also aiming at preparation of polyfluorobenzocyclobutenes containing a pentafluorophenyl group alongside the trifluoromethyl one we carried out a reaction between perfluoro-1-methylbenzocyclobutene and pentafluorobenzene in SbF_5 medium followed by treating the reaction mixture first with anhydrous HF and then by water to obtain perfluoro-1-methyl-2-phenylbenzocyclobutene (**II**) ($Z:E \sim 1:3.5$), alongside with 2-hydroxyperfluoro-1-methyl-2-phenylbenzocyclobutene (**III**) ($Z:E \sim 1:1.3$), perfluoro-1-methyl-1-phenylbenzocyclobutene (**IV**), and 1-(2-trifluoromethyltetrafluorophenyl)-1-pentafluorophenyl-2,2,2-trifluoroethane (**V**). The hydrolysis of the reaction mixture without preliminary treating with anhydrous HF furnished mainly hydroxy derivative **III** ($Z:E \sim 1:1.3$) alongside compounds **II** ($Z:E \sim 1:4$) and **IV**; also formed a little of diphenylethane **V** and perfluoro-1-(2-methylphenyl)-1-phenylethylene (**VI**).

Pentafluorobenzene apparently enters into electrophilic substitution with compound **I** analogously to its reaction with perfluorobenzocycloalkenes [1].

Formation of 1-methyl-1-phenyl derivative **IV** alongside 1,2-isomer **II** is consistent with published data [3].

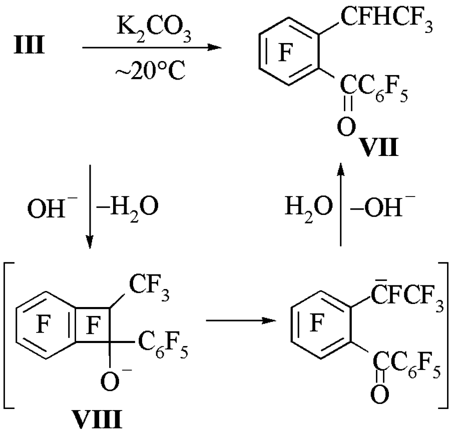
As a result of four-membered ring opening effected by SbF_5 from compound **IV** forms isomer **VI** with a double bond as it happens in reaction of perfluorin-





ated 1,1-dialkylbenzocyclobutenes with antimony pentafluoride [4]. The addition of HF to the double bond of compound **VI** furnishes diphenylethane **V**.

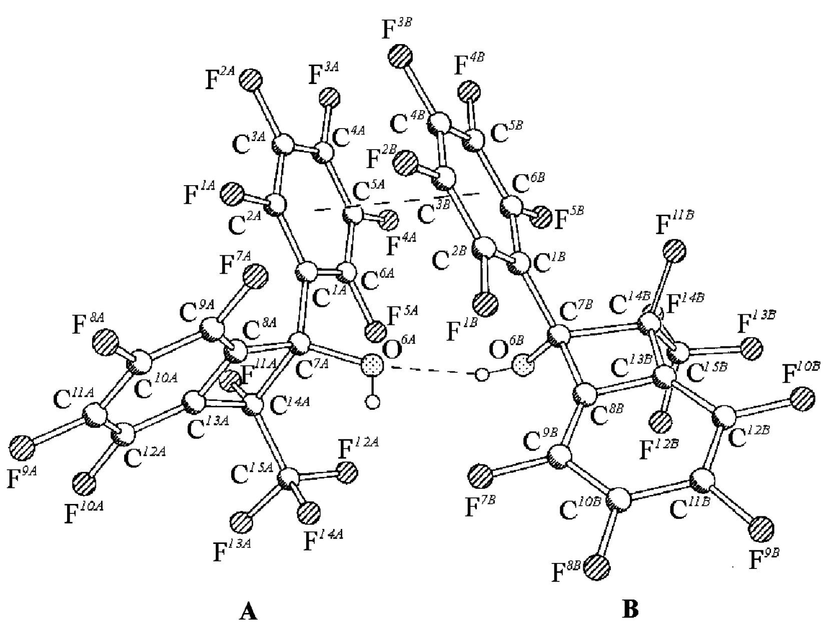
Hydroxy derivative **III** stable against acids when treated with water solution of K_2CO_3 or passed



through a column charged with silica gel (at pH ≥ 7) was converted into 1-(1,2,2,2-tetrafluoroethyl)nonafluorobenzophenone (**VII**). The reaction apparently takes the route similar to the haloform decomposition through an intermediate anion **VIII** arising by deprotonation of hydroxy compound **III**.

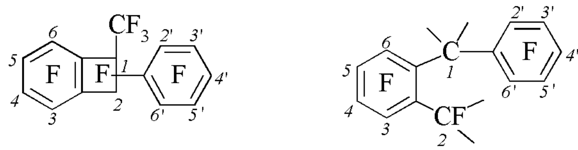
The composition and structure of compounds **II**–**VII** were derived from elemental analyses and spectral characteristics. The assignment of signals in ^{19}F NMR spectra was performed basing on the chemical shifts, multiplicity, and integral intensity thereof (Table 1).

Compound *E*-**III** was subjected to X-ray diffraction study. The independent part of the cell contains two independent molecules *E*-**IIIA** and *E*-**IIIB** whose geometric parameters coincide within the error limits (see figure), but the fragment C–OH is an exclusion with different C–O bond lengths [1.419(5) and 1.400(5) Å in *E*-**IIIA** and *E*-**IIIB** molecules respectively] and different orientation of the OH group [the torsion angle $C^8C^7O^6H^6$ is 41° and 79° in *E*-**IIIA** and *E*-B molecules respectively]. A special feature of this crystal structure is a π-stacking interaction between pentafluorophenyl groups in the dimer pair of molecules *E*-**IIIA** and *E*-**IIIB** connected by a



Structure of (*E*)-2-hydroxyperfluoro-1-methyl-2-phenylbenzocyclobutene (*E*-**III**) (**A** and **B** molecules).

Table 1. ^{19}F and ^{1}H NMR spectra of compounds **II**–**VII**



Compd. no.	δ, ppm									*J*, Hz					
	F^{1} (H)	F^{2}	F^{3}	F^{4}	F^{5}	F^{6}	$F^{2',6'}$	$F^{3',5'}$	$F^{4'}$	$J_{3,4}$	$J_{3,5}$	$J_{3,6}$	$J_{4,5}$	$J_{4,6}$	$J_{5,6}$
Z-**II**	1.2 86.4[a]	30.7	29.0	20.1	20.0	27.3	23.1[b]	2.2	14.7	18	8	23	18	7	18
E-**II**[c]	8.8 86.8[a]	31.8	29.4	20.4	19.8	27.0	22.2	2.2	14.9	18	9	23	18	8	18
Z-**III**[d]	0.3 85.1[a]	–	27.2	19.1	16.6	26.5	25.5 16.9	1.6	12.2	19	7	24	18	7	19
E-**III**[d]	9.1 87.5[a]	–	27.3	19.2	16.9	26.4	20.7	1.7	12.3	19	8	24	19	8	19
IV	92.2[a]	70.6 and 60.5[e]	26.4	17.5	20.2	31.9	28.9 19.0[f]	2.3 2.0	13.4	20	8	23	18	9	18
V[g]	96.4[a] (5.7)	108.1[a]	28.2	13.0	15.8	~32	23.9	1.4	10.8	21	9	9	21	8	21
VI	87.2[h] 86.2[h]	105.2[a]	28.4 or 26.4	14.8 or 12.3	14.8 or 12.3	28.4 or 26.4	23.4	0.9	10.5						
VII[i]	(6.1)	–37.5 84.9[a]	28.2	14.5	13.6	23.4	22.0	2.5	17.7	21	8	11	20	6	22

[a] CF_3.
[b] At 90°C.
[c] $J_{2'(6')3}$ 15 Hz.
[d] $J_{2'(6')3}$ 12 Hz.
[e] *AB*-system, J_{AB} 206 Hz.
[f] $F^{2'}$, $J_{2',6}$ 86 Hz.
[g] J_{F^6,CF_3^1} 22, J_{F^3,CF_3^2} 28 Hz.
[h] CF_2. [i] $J_{2,3}$ 15, J_{F^3,CF_3} = J_{F^2,CF_3} 13, $J_{F^2,H}$ 43, J_{H,CF_3} 6 Hz.

hydrogen bond $O^{6B}–H^{6B}\cdots O^{6A}$. The angle between the planes and the distance measured as average from the center of one ring to the plane of the other are equal respectively to 10.9° and 3.586 Å, and the distance between the centers of the benzene rings is 3.853 Å. A similar polyfluoroaryl–polyfluoroaryl π-stacking interaction between heptafluoronaphthyl fragments with interplane distance of 3.501 Å was called unprecedented in [5] for formerly only arene–arene [6] and arene–polyfluoroarene [7] interactions had been known. It should be noted that calculations for benzene [8] in the case of parallel-translated dimer gave the interplane distance of 3.5 Å, the distance between the centers of the rings of 3.9 Å, displacement of centers of 1.8 Å, and the negative energy of interaction of 2.5 kcal mol^{-1}.

The dissimilarity in the hydroxy groups orientation originating likely from the presence of π-stacking interaction in the dimer pair resulted in lack of participation in a hydrogen bond of the hydroxy group from *E*-**IIIA** molecule whereas the hydrogen of the hydroxy group from the *E*-**IIIB** molecule is involved in a bond $O^{6B}–H^{6B}\cdots O^{6A}$ (distance $H^{6B}\cdots O^{6A}$ 2.33(5) Å, angle $O^{6B}–H^{6B}\cdots O^{6A}$ 146°).

The four-membered ring in the *E*-**IIIA** molecule is somewhat more flat than in *E*-**IIIB** one [within ±0.019(3) and ±0.045(3) Å respectively]. Torsional angles $C^2C^1C^7C^8$ [15.9(6) and 16.3(6)°] and angles between the planes of the benzene ring and the bicyclic fragment [62.7(1) and 65.4(1)°] have close values in the molecules *E*-**IIIA** and *E*-**IIIB** respectively.

In the cyclobutene fragment the bond C^{14}–C^7 [1.604(6) Å in *E*-**IIIA** and 1.608(5) Å in *E*-**IIIB**] is longer than the corresponding bond in the perfluorobenzocyclobutene [9] and in benzocyclobutene [10] (1.574 and 1.576 Å respectively), but in 1-hydroxy-2,2-dimethyl-4-isopropyl-1-[2-(2-methyl-1,3-dioxolan-2-yl)propan-2-yl]benzocyclobutene it is still longer (1.659 Å) [11]. At the same time the length of this bond is close to the average value [1.598(40) Å] for 63 structures from the Cambridge Structural Database [12] containing benzocyclobutene fragment. The bond C^8–C^{13} [1.380(5) Å in *E*-**IIIA** and 1.371(6) Å in *E*-**IIIB**] is somewhat shortened as compared to the published data for benzocyclobutene (1.392 Å) [10] and perfluorobenzocyclobutene (1.395 Å) [9], and also compared to the mean value of 1.390(17) Å. The bond lengths of the fragment C^9–C^{10}–C^{11}–C^{12} [1.360(7), 1.370(8), 1.372(9) Å for *E*-**IIIA** and 1.356(6), 1.390(6) and 1.363(6) Å for *E*-**IIIB**] are a little shorter than the corresponding bonds in the perfluorobenzocyclobutene (1.386, 1.408, 1.386 Å).

According to X-ray diffraction analysis in compound *E*-**III** CF_3 and C_6F_6 groups are located *trans* with respect to each other. In the ^{19}F NMR spectrum of the compound the signal of fluorine attached to tertiary carbon is observed downfield (δ_{F1} 9.4 ppm) with respect to such atom in the *Z*-isomer (δ_{F1} 0.3 ppm). In keeping with these data *Z*-configuration was assigned to the isomer of compound **II** whose analogous fluorine atom appeared as a signal at δ_{F1} 1.2 ppm, and to the isomer with δ_{F1} 8.8 ppm was assigned *E*-configuration.

In the ^{19}F NMR spectra of compounds *E*-**II** and *E*-**III** same as in that of perfluoro-1-phenylbenzocyclobutene [1] at room temperature the atoms $F^{2',6'}$ of the pentafluorophenyl group appear as one broadened signal. Unlike that in the spectra of compounds *Z*-**III** and **IV** from analogous fluorine atoms two broad signals are observed suggesting that the rotation of the pentafluorophenyl group is hampered in these compounds. In the spectrum of isomer *Z*-**II** registered at room temperature the signals from atoms $F^{2',6'}$ of the pentafluorophenyl group are not seen, and at raising the temperature to 90°C they appear as one broad peak.

EXPERIMENTAL

^{19}F and ^1H NMR spectra of reaction mixtures and individual compounds in $CHCl_3$ ($c \leq 10$ mol%) were registered on Bruker WP-200SY at operating frequencies 188.3 MHz and 200 MHz using as internal references C_6F_6 or TMS respectively. IR spectrum of compound **VII** solution was recorded on Vector-22 instrument. Elemental composition of compounds was determined from the high resolution mass spectra taken on Finnigan MAT 8200 mass spectrometer. The GLC analyses were carried out on chromatograph LKhM-72 (50–270°C, 4000 × 4 mm, SKTFT-50 or E-301 on Chromosorb W, 15(25):100, He, 60 ml min^{-1}). GC-MS analysis was performed on Hewlett-Packard G108A instrument comprising a gas chromatograph HP 5890 of II series and mass-selective detector HP 6971 (electron impact, 70 eV); capillary column HP 5 (5% of diphenyl – 95% of dimethylsiloxane; 30m × 0.25 mm × 0.25 μm). carrier gas helium, flow rate 1 ml min^{-1}).

X-ray diffraction study of isomer *E*-**III** was performed on diffractometer Bruker P4 [Mo K_α-radiation, graphite monochromator, θ/2θ-scanning, 2θ < 45°, experiment was carried out at –70(2)°C]. Rhombic crystals: *a* 14.703(1), *b* 11.281(2), *c* 35.575(4) Å, V 5900.6(13) Å^3, space group P*bca*, $C_{15}HF_{13}O$, Z 16, *M* 444.16, d_{calc} 2.000 g/cm^3, μ 0.237 mm^{-1}. Crystal habit 0.16 × 0.70 × 0.72 mm. Intensity of 3178 independent reflections was measured, and corrections were done for absorption (transmission 0.87–0.96). The structure was solved by the direct method along the routine SHEXS-97 and refined by the least-squares procedure in anisotropic-isotropic (for H atoms) approximation using program SHELXL-97 till wR_2 0.1135, S 1.041 for all reflections (R 0.0400 for 2314 $F_0 > 4\sigma$). The position of the hydrogen in the hydroxy group was localized from the difference synthesis. The obtained coordinates of nonhydrogen atoms are available from the authors by request, the main bond lengths and bond angles are given in Table 2.

Reaction of perfluoro-1-methylbenzocyclobutene (I) with pentafluorobenzene in SbF_5 medium. (a) To a stirred mixture of compound **I** (1.95 g, 6.5 mmol), SbF_5 (4.26 g, 19.6 mmol) and C_6F_6 (3.5 ml) was added C_6F_5H (1.21 g, 7.2 mmol) within 5 min at 23–27°C. The mixture was stirred at this temperature for 4 h, then it was treated with water at 0–20°C, extracted with $CHCl_3$, the organic layer was separated, dried with $MgSO_4$; $CHCl_3$, C_6F_6 and excess C_6F_5H were distilled off to give 2.8 g of

Table 2. Selected bond lengths (Å) and bond angles (deg) of molecules A, B in compound *E*-**III**

Angle (bond)	A	B	Angle	A	B
(O^6–C^7)	1.419(5)	1.400(5)	$C^1C^7C^{14}$	113.3(4)	112.2(3)
(C^1–C^7)	1.513(6)	1.518(5)	$C^9C^8C^{13}$	122.0(4)	121.1(4)
(C^7–C^8)	1.512(6)	1.521(6)	$C^9C^8C^7$	143.2(4)	144.3(4)
(C^7–C^{14})	1.604(6)	1.608(5)	$C^{13}C^8C^7$	94.7(4)	94.5(3)
(C^8–C^9)	1.369(6)	1.366(5)	$C^{10}C^9C^8$	117.3(5)	117.5(4)
(C^8–C^{13})	1.371(6)	1.380(5)	$C^9C^{10}C^{11}$	121.4(5)	121.9(4)
(C^9–C^{10})	1.360(7)	1.356(6)	$C^{10}C^{11}C^{12}$	121.4(5)	120.5(4)
(C^{10}–C^{11})	1.370(8)	1.390(6)	$C^{11}C^{12}C^{13}$	117.3(5)	117.6(4)
(C^{11}–C^{12})	1.372(9)	1.363(6)	$C^8C^{13}C^{12}$	120.5(5)	121.3(4)
(C^{12}–C^{13})	1.375(7)	1.373(6)	$C^8C^{13}C^{14}$	94.2(3)	93.8(3)
(C^{13}–C^{14})	1.489(7)	1.499(6)	$C^{12}C^{13}C^{14}$	145.0(5)	144.1(4)
(C^{14}–C^{15})	1.518(7)	1.517(6)	$F^{11}C^{14}C^{13}$	116.7(4)	117.5(3)
$O^6C^7C^8$	114.4(4)	116.0(3)	$F^{11}C^{14}C^{15}$	106.5(4)	105.4(3)
$O^6C^7C^1$	107.8(3)	110.7(3)	$C^{13}C^{14}C^{15}$	115.4(4)	114.6(3)
$C^8C^7C^1$	119.1(3)	118.1(3)	$F^{11}C^{14}C^7$	114.2(3)	115.2(3)
$O^6C^7C^{14}$	116.7(3)	112.9(3)	$C^{13}C^{14}C^7$	86.5(3)	86.6(3)
$C^8C^7C^{14}$	84.5(3)	84.5(3)	$C^{15}C^{14}C^7$	116.9(4)	117.4(3)

mixture containing according to GLC and ^{19}F NMR data the following compounds: 6% (**II**) ($Z:E$~1:4), 74% (**III**) ($Z:E$~1:1.3), 8% (**IV**), 2% (**V**), and 2% (**VI**). The mixture was subjected to column chromatography on silica gel using as eluent $CHCl_3$ preliminary washed with HCl taken in 10-fold excess (by volume). We isolated 0.4 g of a mixture of compounds *E*-**II**, *Z*-**II, IV, V** and **VI** in ~2:0.5:4:1:1 ratio according to ^{19}F NMR data, 0.42 g of hydroxy derivative *Z*-**III**, 1.31 g of compounds *Z*-**III** and *E*-**III** mixture in ~1:1.5 ratio ^{19}F NMR data, and 0.39 g of hydroxy derivative *E*-**III**, mp 62–63°C (from hexane).

Isomer *Z*-III. Found, %: C 40.80; H 0.21; F 55.67. $C_{15}HF_{13}O$. Calculated, %: C 40.56; H 0.23; F 55.61. **Isomer *E*-III**. Found, %: C 40.38; H 0.31; F 55.12. $C_{15}HF_{13}O$. Calculated, %: C 40.56; H 0.23; F 55.61.

Besides by chromatography on silica gel (eluent hexane) from a mixture of compounds **II, IV–VI** obtained from several runs we isolated isomer *E*-**II** and fractions enriched with the other components.

From 0.79 g of mixture containing compounds in the following amounts: 60% (*E*-**II**), 25% (*Z*-**II**), and 8% (**V**), was isolated 0.14 g of *E*-**II**, 0.1 g of mixture containing *E*-**II** (46%), *Z*-**II** (35%), and **V** (16%), and also several fractions (0.39 g), containing compounds *E*, *Z*-**II, V** in different ratios. In a similar way from 0.62 g of a mixture composed of 11% of *E*-**II**, 9% of *Z*-**II**, 65% of **IV**, 5% of **V**, and 5% of **VI** was obtained 0.13 g of isomers mixture *E*-**II** (21%), **IV** (42%), **VI** (31%), and several fractions (0.33 g) containing compounds *E*, *Z*-**II, IV–VI** in different ratios. The composition of these mixtures was determined from GC-MS and ^{19}F NMR data. In the mass spectrum of compound **V** was observed the molecular ion peak *m/z* 466, and in the mass spectra of individual isomers *E*-**II,** *Z*-**II**, **VI** and in isomers mixture *E*-**II + IV** appear molecular ion peaks *m/z* 446.

Isomer *E*-II. Found: M^+ 445.9762. $C_{15}F_{14}$. Calculated: *M* 445.9776.

Mixture of compounds *E*, *Z*-II + V. Found *E*, *Z*-**II**: M^+ 445.9771. $C_{15}F_{14}$. Calculated: *M* 445.9776. Found **V**: M^+ 465.9866. $C_{15}HF_{15}$. Calculated: *M* 465.9839.

Mixture of compounds *1E*-II + IV + VI. Found: M^+ 445.9762. $C_{15}F_{14}$.Calculated: *M* 445.9776.

(b) To a stirred mixture of compound **I** (2.82 g, 9.5 mmol), SbF_5 (6.16 g, 29.4 mmol) and C_6F_6 (5 ml) was added C_6F_5H (1.75 g, 10.4 mmol) within 0.5 h at 8–12°C. The mixture was stirred at 23–27°C for 4 h, then it was treated with anhydrous HF (15 ml) and poured on ice. The reaction products were extracted into $CHCl_3$. The organic layer was separated, dried with $MgSO_4$; $CHCl_3$, C_6F_6 and excess C_6F_5H were distilled off to give 3.8 g of mixture containing according to GLC and ^{19}F NMR data 65%

of **II** ($Z:E$~1:3.5), 19% of **III** ($Z:E$~1:1.3), 5% of **IV**, and 5% of **V**. By means of column chromatography on silica gel using as eluent $CHCl_3$ treated as indicated in procedure (a) we isolated 2.7 g of mixture of compounds **II, IV**, and **V**, and 0.082 g of hydroxy derivatives **III**.

Reaction of 2-hydroxyperfluoro-1-methyl-2-phenylbenzocyclobutene (III) with water solution of K_2CO_3. A solution of 0.25 g (0.56 mmol) of compound **III** ($Z:E$ ~ 1:1.3) in $CHCl_3$ was added to 2 g of 10% water solution of K_2CO_3 (0.2 g, 1.45 mmol), and the mixture was stirred for 2.5 h at 21°C. The reaction mixture was treated with water, acidified with 5% HCl, extracted with $CHCl_3$, the organic layer was separated, dried with $MgSO_4$, $CHCl_3$ was distilled off, and as a result 0.24 g of compound **VII** was obtained (yield 96%), mp 35–37°C (from hexane). IR spectrum, ν, cm^{-1}: 1698 (C=O). Found, %: C 40.74; H 0.37; F 55.31. $C_{15}HF_{13}O$. Calculated, %: C 40.56; H 0.23; F 55.61.

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