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Synthesis and skeletal rearrangements of perfluorinated 4-alkyl- and 4-phenyl-tetralin-1-ones under the action of antimony pentafluoride

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ABSTRACT

Heating of perfluorinated 1-methyl- and 1-ethyltetralins with SiO_2 in an SbF_5 medium at 100 °C results in perfluoro-4-alkyltetralin-1-ones formation. Perfluoro-4-methyltetralin-1-one, under the action of SbF_5 at 180 °C with subsequent treatment of the reaction mixture with water, is converted to perfluoro-3,3-dimethylindan-1-one and perfluoro-3,4-dimethylisochromen-1-one. Perfluoro-4-ethyltetralin-1-one, under similar conditions, forms perfluoro-3-ethyl-3-methylindan-1-one, perfluoro-4-ethyl-3-methylisochromen-1-one and perfluoro-2-methyltetralin. Reaction of perfluorotetralin-1-one with pentafluorobenzene in the presence of SbF_5 at 50–55 °C leads to the formation of perfluoro-4-phenyltetralin-1-one, which under the action of SbF_5 at 75 °C isomerizes into perfluoro-3-methyl-3-phenylindan-1-one. Heating of the latter with SbF_5 at 75–95 °C gives, after treatment of the reaction mixture with water, perfluoro-2-(2-methylphenyl)-3-phenylpropenoic acid and perfluoro-4-methyl-3-phenylisochromen-1-one.

1. Introduction

Previously, skeletal transformations of carbonyl derivatives of perfluorinated benzocyclobutene, indan, tetralin and a number of their perfluoroalkyl derivatives under the action of SbF_5 or SiO_2–SbF_5 have been found [1–7]. Carbocationic skeletal rearrangements of polyfluorinated ketones had not been known before. In this connection investigation of skeletal transformations of polyfluorobenzocycloalkenones is important to fundamental organic chemistry and can be useful for expansion of the range of available reactions of polyfluorinated compounds which find broad application in various fields, especially in materials science [8,9].

In our previous studies it was found that skeletal transformations of perfluorobenzocycloalken-1-ones under the action of SbF_5 occur along with disproportionation to benzocycloalkenes and benzocycloalkenediones, which also undergo skeletal transformations under these reaction conditions [3]. As a result, the reactions proceed unselectively. Thus, the reaction of perfluorotetralin-1-one (**1**) with SbF_5 gives a mixture of perfluorinated tetralin, benzocondensed five- and six-membered oxygen-containing heterocyclic compounds and alkylbenzoic acids (Scheme 1).

Rearrangements of perfluorinated alkyl substituted benzocyclobutenones [4,7] and indanones [5,6] proceed more selectively because the replacement of the fluorine atom in benzylic position by an alkyl group makes impossible the disproportionation of the initial ketone. Skeletal transformations of substituted polyfluorotetralones have not been investigated before. In this connection this work describes synthesis and behaviour of perfluorinated 4-methyl- (**2**), 4-ethyl- (**3**) and 4-phenyl-tetralin-1-one (**4**) in the presence of SbF_5 with the view of studying the possibility of their cationic skeletal transformations and influence of a substituent in the aliphatic ring on the reaction route.

2. Results and discussion

2.1. Skeletal rearrangements of tetralones 2–4 under the action of SbF_5

We have found that perfluoro-4-alkyltetralin-1-ones undergo skeletal transformations under the action of SbF_5 at high temperature. Thus, heating of methyltetralone **2** with antimony pentafluoride at 180 °C with further treatment of the reaction mixture with water leads to the formation of perfluoro-3,3-dimethylindan-1-one (**5**) and perfluoro-3,4-dimethylisochromen-1-one (**6**) as major products (Scheme 2).

The formation of similar products is also observed in the reaction of ethyltetralone **3** with SbF_5 at 180 °C, but in this case the reaction mixture contains perfluoro-3-ethyl-3-methylindan-1-one (**7**) and perfluoro-4-ethyl-3-methylisochromen-1-one (**8**) together with significant amount of perfluoro-2-metyltetralin (**9**) (Scheme 2). The latter product has a modified carbon framework containing one carbon atom less than the parent ketone **3**. The reaction

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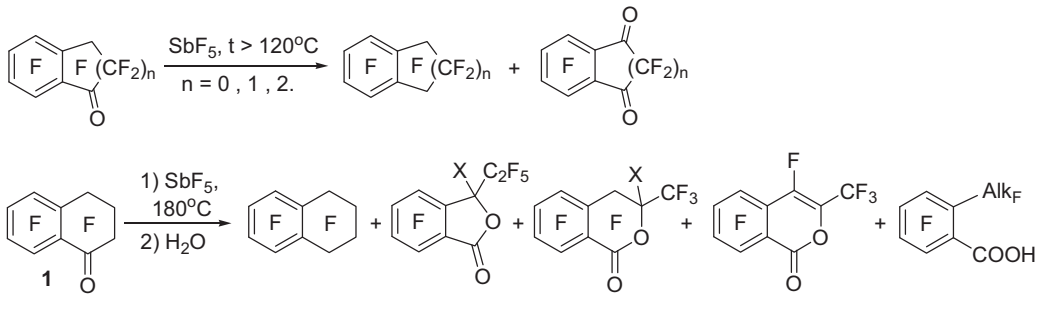
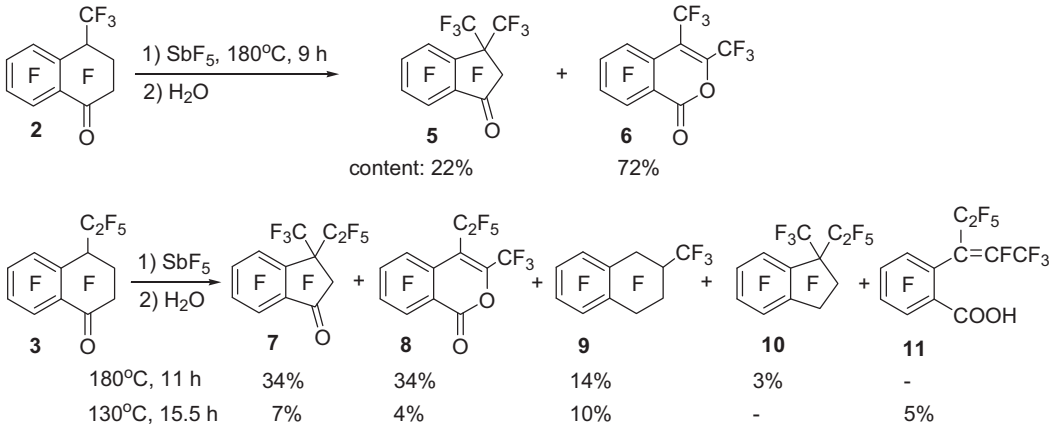


Scheme 1.



Scheme 2.

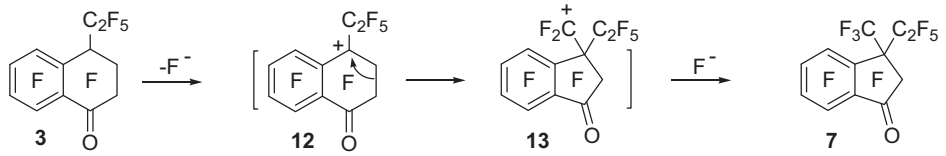
mixture also contains small amounts of perfluoro-1-ethyl-1-methylindan (**10**). Interaction of ethyltetralone **3** with SbF_5 at 130 °C leads to the formation of a mixture of compounds **7**–**9** together with perfluoro-2-(pent-2-en-3-yl)benzoic acid (**11**) and a large amount (74%) of unchanged ketone **3**.

The dialkylindanones **5** and **7** formed in the reaction of tetralones **2** and **3** with antimony pentafluoride are products of a six-membered aliphatic ring contraction. Scheme 3 illustrates this transformation using tetralone **3** as an example. Apparently, ketone **3** under the action of SbF_5 generates the benzyl type cation **12**, which undergoes isomerization into cation **13** by migration of an alkyl moiety. Subsequent addition of fluoride anion gives the dialkylindanone **7**. This scheme is analogous to that for the six-membered aliphatic ring contraction in the reaction of perfluorinated 5-ethyl- and 5,8-diethyl-tetralins under the action of antimony pentafluoride [10].

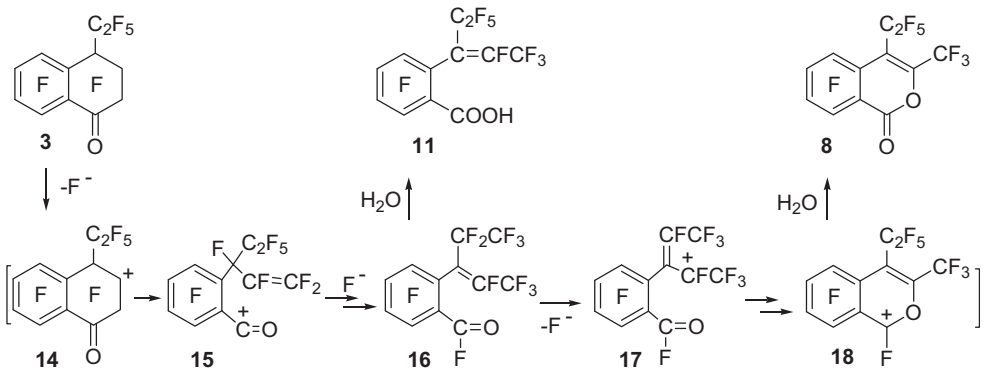
Cleavage of the six-membered aliphatic ring is another route of transformation of tetralones **2** and **3** under the action of SbF_5. This route leads to the formation of isochromenone **6** from tetralone **2** and products **8**, **11** from tetralone **3**. The latter transformation can be rationalized by Scheme 4. Initially, ketone **3** under the action of SbF_5 seems to generate cation **14**. The alicyclic six-membered ring of the latter may undergo ring opening to yield the benzoyl type ion **15**. Fluoride anion addition to cation **15** and isomerization of the double bond inside the chain form the acyl fluoride **16**. This scheme is analogous to that for the six-membered ring opening of perfluorinated tetralin-1-one (**1**) and tetralin-1,4-dione under the action of antimony pentafluoride [3]. Compound **16** under the action of antimony pentafluoride generates the allyl type cation **17**. Intramolecular attack of the positively charged carbon atom of the allylic system of cation **17** at the fluorocarbonyl oxygen atom and subsequent isomerization of the double bond into the endocyclic position gives the isochromenyl cation **18**. The formation of perfluoroalkylisochromenyl cations in the reaction of 2-alkenylbenzoyl fluorides with SbF_5 was observed earlier [3,4]. Hydrolysis of a salt of cation **18** and acyl fluoride **16** gives compounds **8** and **11**, respectively (Scheme 4). Transformation of tetralone **2** to isochromenone **6** proceeds in a similar way.

The mechanism of methyltetralin **9** formation in the reaction of tetralone **3** with antimony pentafluoride is unclear. However, it may be conjectured that the route of this transformation includes a series of cationic rearrangements and decarbonylation [11] of an intermediate acyl fluoride.

Phenyltetralone **4** also undergoes ring contraction under the action of SbF_5. Thus, heating of a solution of phenyltetralone **4** in an SbF_5 medium at 75 °C with further treatment of the reaction mixture with water leads to the formation of perfluoro-3-methyl-3-phenylindan-1-one (**19**). The reaction mixture also contains



Scheme 3.



Scheme 4.

perfluoro-2-(2-methylphenyl)-3-phenylpropenoic acid (**20**) and small amounts of perfluoro-4-methyl-3-phenylisochromen-1-one (**21**) (Scheme 5). The reaction of phenyltetralone **4** with SbF_5 in the presence of HF (1.5 equiv.) gives indanone **19** without producing compounds **20** and **21**.

Milder conditions of ring contraction reaction of tetralone **4** as compared with the corresponding reactions of tetralones **2** and **3** can be attributed to a higher concentration of reacting perfluoro-4-oxo-1-phenyltetralin-1-yl cation (**22**) as compared to the corresponding perfluoro-4-oxo-1-alkyltetralin-1-yl cations (for the mechanism of ring contraction, see Scheme 3). Indeed, according to ^{19}F NMR data phenyltetralone **4** dissolved in SbF_5 is transformed to a salt of cation **22**, whereas the tetralones **2** and **3** give complexes **2c** and **3c** in which antimony pentafluoride is attached to the oxygen atom of the carbonyl group (Scheme 5). Therefore, perfluoro-4-oxo-1-alkyltetralin-1-yl cations are not formed in sufficient concentration for ^{19}F NMR detection.

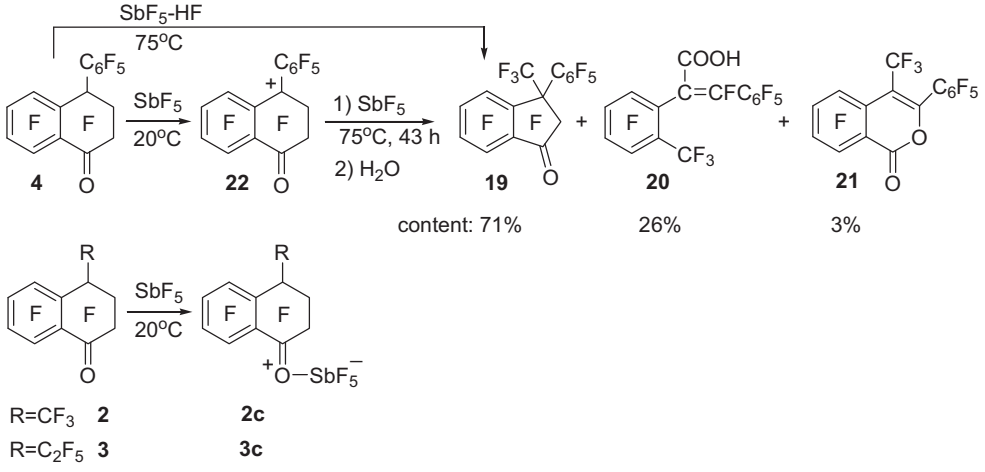
Compounds **20** and **21** formed in the reaction of phenyltetralone **4** with SbF_5 at 75 °C are products of further transformations of indanone **19** under the reaction conditions. Thus, heating of indanone **19** with antimony pentafluoride at 75 °C gives a mixture of compounds **20** and **21** in similar ratio. Increasing of the reaction temperature to 95 °C leads to the formation of compound **21** as the major product (Scheme 6). It should be noted that, in contrast to indanone **19**, participation of indanones **5** and **7** in the formation of products **6**, **8**, **9**, **11** seems unlikely, since it has been shown in a separate experiment that compounds **5** and **7** practically do not change under the reaction conditions of tetralones **2**, **3** with SbF_5.

Formation of compounds **20** and **21** in the reaction of indanone **19** with SbF_5 apparently proceeds in the following way (Scheme 6). At first, compound **19** with SbF_5 seems to generate cation **23**. Then the pentaflurophenyl group migration to the cationic center leads to the formation of cation **24**. The five-membered ring of the latter may undergo ring opening to yield the benzoyl type ion **25**, which isomerizes into the allyl type cation **26**. Another way to ion **26** is pentaflurophenyl group migration in cation **27** with subsequent ring opening and addition-elimination of a fluoride ion. An intramolecular attack of one or another positively charged carbon atom of the allyl system of cation **26** at the fluorocarbonyl oxygen atom can give cation **28** or cation **29**. Isomerization of cation **28** into cation **30** followed by ring opening and addition-elimination of a fluoride ion results in the formation of the acyl type cation **31**. Its hydrolysis gives acid **20**. Isomerization of the double bond of cation **29** into the endocyclic position forms cation **32**. Its hydrolysis gives compound **21**.

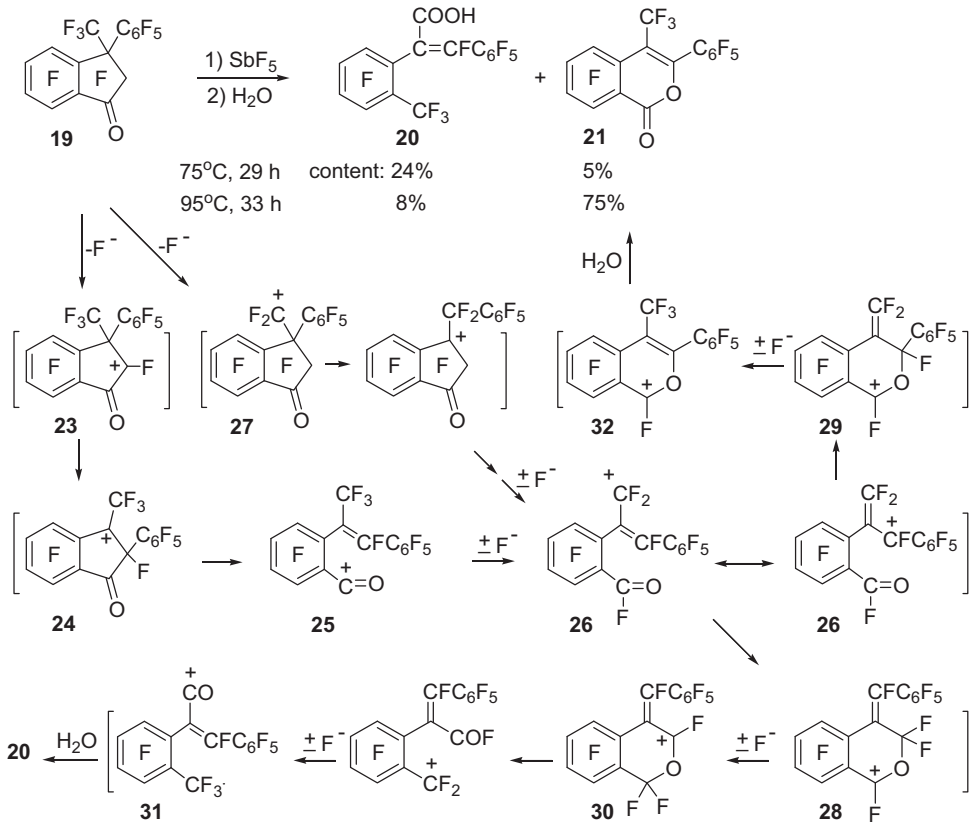
As already mentioned, the reaction of phenyltetralone **4** with SbF_5 in the presence of HF gives the indanone **19** without compounds **20** and **21**. Taking into account the proposed reaction routes this effect of HF can be explained by suppression of generation of unstable cations **23** and/or **27**, which are supposed to be the intermediates in products **20** and **21** formation (Scheme 6). However, generation of the more stable cation **22** in a concentration sufficient for the reaction in this medium is still possible (Scheme 5).

2.2. Synthesis of tetralones 2–4

The required alkyltetralones **2** and **3** were synthesized in high yield by the reaction of tetralins **33** and **34** with SiO_2 in the presence of SbF_5 (Scheme 7). This method has been used earlier for



Scheme 5.

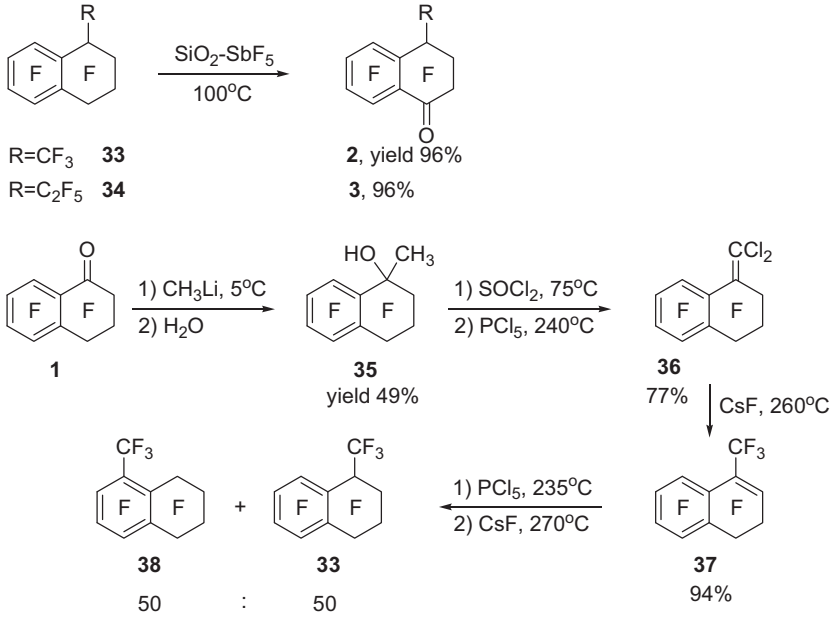


Scheme 6.

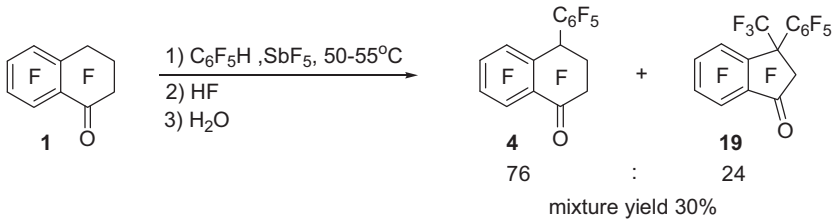
the synthesis of mono- and di-carbonyl derivatives of perfluorobenzocycloalkenes [3].

Methyltetralin **33** was obtained by the following method (Scheme 7). Initially 2,2,3,3,4,4,5,6,7,8-decafluoro-1-methyltetralin-1-ol (**35**) was prepared by reaction of tetralone **1** with CH_3Li. Treatment of compound **35** with $SOCl_2$ at 75 °C and subsequent chlorination with PCl_5 at 240 °C gave 1-(dichloromethylene)-2,2,3,3,4,4,5,6,7,8-decafluorotetralin (**36**). Heating of the latter with CsF at 260 °C led to chlorine replacement by fluorine with isomerization of the double bond to an endocyclic position. As a result perfluoro-4-methyl-1,2-dihydronaphthalene (**37**) was formed. Chlorination of double bond in compound **37** with PCl_5 at 235 °C and subsequent heating of the product obtained with CsF at 270 °C gave methyltetralin **33** together with perfluoro-5-methyltetralin (**38**).

It should be noted that ^{19}F NMR data for compounds named in article [12] as "perfluorinated 1- and 2-methyltetralins" correspond with the structures of perfluorinated 5- and 6-methyltetralins,



Scheme 7.



Scheme 8.

containing CF_3 group in the aromatic ring, and disagree with the ^{19}F NMR spectra of 1- and 2-methyltetralins **33** and **9** obtained in this work.

Perfluoro-1-phenyltetralin does not react with SiO_2 in the presence of SbF_5 at 70 °C, therefore phenyltetralone **4** was synthesized by reaction of tetralone **1** with pentafluorobenzene in the presence of SbF_5 at 50–55 °C with further treatment of the reaction mixture with anhydrous HF and then with water (Scheme 8). The reaction proceeds more slowly as compared with the corresponding reaction of perfluorotetralin [13], the reaction mixture after 50 h contains phenyltetralone **4** along with considerable amount of unchanged ketone **1**. Despite the low reaction temperature the reaction mixture also contains small amounts of indanone **19**.

2.3. ^{19}F NMR characterization

The structures of the compounds were established by HRMS and spectral characteristics. Assignment of signals in the ^{19}F NMR spectra of compounds was made on the basis of chemical shifts of the signals, their fine structure and integral intensities. Patterns observed in the spectra of indanones **7**, **19** are in agreement with those for compound **5** [14] and perfluoroindan-1-one [2], in the spectra of tetralones **2**, **3**, **4** – with those for tetralone **1** [3], in the spectrum of methyltetralin **33** – with those for ethyltetralin **34** [15], in the spectrum of compound **21** – with those for dimethylisochromenone **6** [5]. Compound **20** is formed as a mixture of *E*- and *Z*-isomers. Their configuration was not assigned because resolution of signals in the ^{19}F NMR spectrum was insufficient for fine structure analysis. Compounds **5** [14], **6** [5], **8**, **11** [4] were identified by comparison of the ^{19}F NMR data with data for authentic samples. Assignment of signals in the ^{19}F NMR spectrum of cation **22** was made by analogy with that for perfluoro-1-phenyltetralin-1-yl cation [13]. Assignment of signals in the ^{19}F NMR spectra of complexes **2c** and **3c** was made by analogy with that for complex of tetralone **1** with SbF_5 [3] and perfluoro-1-hydroxytetralin-1-yl cation [2].

3. Conclusion

Novel perfluoro-4-R-tetralin-1-ones (R = CF_3 (**2**), C_2F_5 (**3**), C_6F_5 (**4**)) were synthesized. The behaviour of tetralones **2**–**4** under the action of SbF_5 was investigated. As a result new carbocationic skeletal rearrangements were found. These compounds undergo six-membered aliphatic ring contraction to give the corresponding perfluoro-3-R-3-methylindan-1-ones **5**, **7** and **19**. In the case of alkyltetralones **2** and **3** ring contraction occurs along with ring cleavage leading finally to the formation of isochromenones **6** and **8**; in the reaction of ethyltetralone **3** is also formed considerable amount of 2-methyltetralin. The pentafluorophenyl group in tetralone **4** facilitates the process of ring contraction in comparison with the perfluoroalkyl groups. The indanone **19** formed in the reaction of tetralone **4** with SbF_5 undergoes skeletal rearrangements under the reaction conditions to give diarylpropenoic acid **20** and isochromenone **21**, whereas the formation of indanones **5** and **7** is not accompanied by further transformations. The presence of HF in the reaction media allow to prevent indanone **19** rearrangements.

4. Experimental

IR spectra were taken on a Bruker Vector 22 IR spectrophotometer. ^{19}F and ^{1}H NMR spectra were recorded on a Bruker AV 300 instrument (282.4 MHz and 300 MHz, respectively). Chemical shifts are given in δ ppm from CCl_3F (^{19}F) and TMS (^{1}H), *J* values in Hz; C_6F_6 and SO_2ClF (−162.9 and 99.9 ppm from CCl_3F) and $CHCl_3$ (7.24 ppm from TMS) were used as internal standards. The molecular masses of the compounds were determined by high-resolution spectrometry on a Thermo Electron Corporation DFS instrument (EI 70 eV). Contents of products in the reaction mixtures were established by ^{19}F NMR spectroscopic data.

New compounds **2**, **3**, **7**, **19**–**21** and **35**–**37** were isolated and characterized in the individual state; compound **4** – in the mixture with isomer **19**. Isomeric compounds **9** and **10**, **33** and **38** were isolated and characterized in their mixtures.

Antimony pentafluoride was distilled at atmospheric pressure (bp 142–143 °C); SiO_2 was prepared by heating of silica gel at 400–450 °C; ketone **1** and compound **34** were synthesized according to Refs. [14,15], respectively. The reactions were carried out in glassware or in a nickel bomb (*V* = 10 ml).

4.1. Reaction of perfluoro-4-methyltetralin-1-one (2) with SbF_5

A solution of compound **2** (0.97 g) in SbF_5 (5.96 g) (molar ratio, 1:11) was prepared and ^{19}F NMR spectrum of the solution was recorded at 20 °C. The spectrum contained signals of complex **2c**. Then the solution was heated at 180 °C for 9 h in the nickel bomb. The mixture was treated with 5% hydrochloric acid and extracted with CH_2Cl_2. The extract was washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. The solvent was distilled off to give 0.65 g of a mixture containing (GC–MS) 22% of **5**, 56% of **6** and 6% of perfluoro-4-fluorocarbonyl-3-methylisochromen-1-one [5] together with unidentified impurities.

4.1.1. Complex (2c)

^{19}F NMR (SbF_5): δ −72.6 (3F, CF_3), −100.3 (1F, F-8), −104.5 (1F, F-6), −110.5 ($1F_A$) and −121.4 ($1F_B$, $J_{A,B}$ = 324, CF_2-2), −115.6 ($1F_A$) and −132.2 ($1F_B$, $J_{A,B}$ = 278, CF_2-3), −121.4 (1F, F-5), −138.2 (1F, F-7), −183.2 (1F, F-4).

4.2. Reaction of perfluoro-4-ethyltetralin-1-one (3) with SbF_5

1. A solution of compound **3** (1.03 g) in SbF_5 (6.29 g) (molar ratio, 1:12) was prepared and ^{19}F NMR spectrum of the solution was recorded at 20 °C. The spectrum contained signals of complex **3c**. Then the solution was heated at 130 °C for 15.5 h in the nickel bomb. The mixture was treated with 5% hydrochloric acid and extracted with CH_2Cl_2. The extract was dried over $MgSO_4$. The solvent was distilled off to give 0.96 g of a mixture of compounds **3**, **7**, **8**, **9** and **11** (*E*:*Z* ~ 15:85) in the ratio 74:7:4:10:5.
2. A mixture of compound **3** (1.12 g) and SbF_5 (6.80 g) (molar ratio, 1:12) was heated at 180 °C for 3 h in a nickel bomb. The mixture

was treated with 5% hydrochloric acid and extracted with CH_2Cl_2. The extract was washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. The solvent was distilled off to give 0.85 g of a mixture containing ~90% compounds **7**, **8**, **9** and **10** in the ratio 40:32:27:1. The solvent was distilled off and the residue was dissolved in 5 ml of diethyl ether. To this solution 10 ml of water and 1 g of $NaHCO_3$ were added. The mixture was stirred at room temperature for 5 days, and then organic layer was separated and dried over $MgSO_4$. The solvent was distilled off to give 0.47 g of a mixture of compounds **7**, **9** and **10** in the ratio 60:39:1.

3. Analogously to the previous experiment, the reaction of compound **3** (0.85 g) and SbF_5 (5.43 g) (molar ratio, 1:12.5) gave (180 °C, 11 h) 0.64 g of a mixture containing ~85% of compounds **7**, **8**, **9** and **10** in the ratio 40:40:16:4 and subsequent hydrolysis gave 0.33 g of a mixture of compounds **7**, **9** and **10** in the ratio 68:26:6. Column chromatography (hexane as eluent) of mixture (1.30 g) with the same composition (obtained in several analogous experiments) gave 0.39 g of individual compound **7** and 0.25 g of a mixture containing compounds **9** and **10** in the ratio 80:20. Column chromatography of this mixture gave a fraction (0.02 g) containing (GC–MS) 7% of tetralin **9** and 86% of indan **10**, and a fraction (0.06 g) containing (GC–MS) 84% of tetralin **9** and 12% of indan **10**.

*4.2.1. Complex (**3c**)*

^{19}F NMR (SbF_5): δ −78.5 (3F, CF_3), −109.9 ($1F_A$) and −120.1 ($1F_B$, $J_{A,B}$ = 328, CF_2-2), −100.6 (1F, F-8), −104.7 (1F, F-6), −112.2 ($1F_A$) and −130.0 ($1F_B$, $J_{A,B}$ = 274, CF_2-3), −113.7 ($1F_A$) and −115.0 ($1F_B$, $J_{A,B}$ = 302, **CF_2**CF_3), −121.5 (1F, F-5), −138.1 (1F, F-7), −185.2 (1F, F-4).

*4.2.2. Perfluoro-3-ethyl-3-methylindan-1-one (**7**)*

Liquid. IR (CCl_4) ν, cm^{-1}: 1784 (C=O); 1515, 1507 [fluorinated aromatic ring (FAR)]. ^{19}F NMR ($CDCl_3$): δ −63.7 (3F, CF_3-3), −78.9 (3F, CF_2**CF_3**), −108.4 ($1F_A$) and −110.2 ($1F_B$, $J_{A,B}$ = 297, **CF_2**CF_3), −114.9 ($1F_A$) and −118.8 ($1F_B$, $J_{A,B}$ = 284, CF_2-2), −130.5 (1F, F-4), −132.8 (1F, F-7), −136.6 (1F, F-5), −144.7 (1F, F-6); $J_{4,5}$ = 19.5, $J_{4,6}$ = 9, $J_{4,7}$ = 16.5, $J_{5,6}$ = 19, $J_{5,7}$ = 13, $J_{6,7}$ = 21. HRMS *m*/*z*, 425.9710 (M^+). Calcd. for $C_{12}F_{14}O$ = 425.9725.

*4.2.3. Perfluoro-2-methyltetralin (**9**)*

Liquid. ^{19}F NMR (hexane): δ −70.6 (3F, CF_3), −95.9 ($1F_A$) and −102.1 ($1F_B$, $J_{A,B}$ = 305, CF_2-1), −96.6 ($1F_A$) and −116.9 ($1F_B$, $J_{A,B}$ = 293, CF_2-4), −119.5 ($1F_A$) and −136.7 ($1F_B$, $J_{A,B}$ = 286, CF_2-3), −135.0 (1F) and −135.6 (1F, F-5, F-8), −145.1 (2F, F-6, F-7), −185.7 (1F, F-2). HRMS *m*/*z*, 397.9770 (M^+). Calcd. for $C_{11}F_{14}$ = 397.9776.

*4.2.4. Perfluoro-1-ethyl-1-methylindan (**10**)*

Liquid. ^{19}F NMR (hexane): δ −63.5 (3F, CF_3-1), −78.1 (3F, CF_2**CF_3**), −104.0 ($1F_A$) and −107.4 ($1F_B$, $J_{A,B}$ = 260, CF_2-3), −106.4 ($1F_A$) and −107.7 ($1F_B$, $J_{A,B}$ = 298, **CF_2**CF_3), −115.6 (2F, CF_2-2), −129.8 (1F, F-7), −138.7 (1F, F-4), −143.7 (1F, F-6), −145.2 (1F, F-5); $J_{4,5}$ = 20.5, $J_{4,6}$ = 8, $J_{4,7}$ = 16, $J_{5,6}$ = 18.5, $J_{5,7}$ = 9.5, $J_{6,7}$ = 19. HRMS *m*/*z*, 447.9739 (M^+). Calcd. for $C_{12}F_{16}$ = 447.9744.

*4.3. Reaction of perfluoro-4-phenyltetralin-1-one (**4**) with SbF_5*

To a mixture of isomers **4** and **19** (0.19 g) in the ratio 76:24 and SbF_5 (1.19 g) (molar ratio (**4** + **19**):SbF_5 = 1:13.5) SO_2ClF (0.14 g) was added at 0 °C and ^{19}F NMR spectrum of the solution was measured at 20 °C. The spectrum contained signals of cation **22** and indanone **19** in the ratio 75:25. The solution was heated at 75 °C for 43 h and then poured into 5% hydrochloric acid and extracted with CH_2Cl_2. The extract was dried over $MgSO_4$. The solution contained compounds **19**, **20** and **21** in the ratio 71:26:3. The extract was washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. The solvent was distilled off to give 0.13 g of a mixture, which contained compounds **19** and **21** in the ratio 96:4. An analytical sample of indanone **19** was prepared by crystallization. The aqueous solution of $NaHCO_3$ was acidified with HCl, extracted with CH_2Cl_2 and dried over $MgSO_4$. The solvent was distilled off and the residue was sublimed (150 °C, 3 Torr) to give 0.04 g of acid **20** (mixture of isomers in the ratio 80:20).

*4.3.1. Perfluoro-4-oxo-1-phenyltetralin-1-yl cation (**22**)*

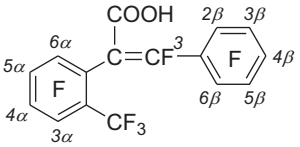
^{19}F NMR (SbF_5–SO_2ClF): δ −77.1 (1F, F-6), −88.8 (1F, F-8), −96.2 (1F, F-*para*), −110.2 (4F, F-*ortho*, CF_2-2), −112.7 (1F, F-5), −122.3 (2F, CF_2-3), −130.0 (1F, F-7), −148.4 (2F, F-*meta*).

*4.3.2. Perfluoro-3-methyl-3-phenylindan-1-one (**19**)*

mp 73.5–74 °C (hexane). IR (CCl_4) ν, cm^{-1}: 1772 (C=O); 1531, 1504 (FAR). ^{19}F NMR [$CO(CD_3)_2$]: δ −61.3 (3F, CF_3), −104.7 ($1F_A$) and −113.9 ($1F_B$, $J_{A,B}$ = 283, CF_2), −132.1 (2F, F-*ortho*), −133.5 (1F, F-7), −135.2 (1F, F-4), −135.9 (1F, F-5), −147.2 (1F, F-6), −148.7 (1F, F-*para*), −159.8 (2F, F-*meta*); $J_{CF3-F(2A)}$ = 2.5, $J_{CF3-F(2B)}$ = 25.5, $J_{CF3-F(ortho)}$ = 26.5, $J_{CF3-F(4)}$ = 19.5, $J_{ortho,para}$ = 6, $J_{ortho,2A}$ = 16.5, $J_{ortho,2B}$ = 2.5, $J_{ortho,4}$ = 2, $J_{meta,para}$ = 21, $J_{4,5}$ = 19.5, $J_{4,6}$ = 7.5, $J_{4,7}$ = 15.5, $J_{5,6}$ = 18.5, $J_{5,7}$ = 13, $J_{6,7}$ = 20.5. HRMS *m*/*z*, 473.9721 (M^+). Calcd. for $C_{16}F_{14}O$ = 473.9720.

*4.3.3. Perfluoro-2-(2-methylphenyl)-3-phenylpropenoic acid (**20**)*

Mixture of two isomers, ratio **20a**:**20b** = 80:20; mp 132–143 °C (hexane). IR (CCl_4) ν, cm^{-1}: ~3000 b (OH); 1754, 1719, 1670, 1647 (O=C–C=C); 1511, 1485 (FAR). ^1H NMR ($CDCl_3$): δ 9.10 (bs, OH).



Isomer **20a**: ^{19}F NMR ($CDCl_3$): δ −57.6 (3F, CF_3), −65.7 (1F, F-3), −135.5 (1F, F-6α), −137.5 (1F, F-3α), −137.9 (2F, F-2β, F-6β), −147.9 (1F, F-4β), −148.3 (1F, F-5α), −150.9 (1F, F-4α), −161.2 (2F, F-3β, F-5β); $J_{CF3-F(3\alpha)}$ = 22, $J_{3,6\alpha}$ = 4, $J_{3,4\beta}$ = 4, $J_{3\alpha,4\alpha}$ = 21, $J_{3\alpha,5\alpha}$ = 8, $J_{3\alpha,6\alpha}$ = 10.5, $J_{4\alpha,5\alpha}$ = 20.5, $J_{4\alpha,6\alpha}$ = 5.5, $J_{5\alpha,6\alpha}$ = 22.5, $J_{2\beta,4\beta}$ = $J_{6\beta,4\beta}$ = 4, $J_{3\beta,4\beta}$ = $J_{5\beta,4\beta}$ = 21.

Isomer **20b**: −57.6 (3F, CF_3), −69.0 (1F, F-3), −135.4 (1F, F-6α), −136.2 (1F, F-3α), −136.8 (2F, F-2β, F-6β), −146.0 (1F, F-4β), −147.4 (1F, F-5α), −149.6 (1F, F-4α), −159.3 (2F, F-3β, F-5β). HRMS (mixture of two isomers) *m*/*z*, 471.9768 (M^+). Calcd. for $C_{16}H_1F_{13}O_2$ = 471.9764.

*4.4. Reaction of perfluoro-4-phenyltetralin-1-one (**4**) with SbF_5 in the presence of HF*

A mixture of SbF_5, HF and perfluoro-1-phenylindan was prepared by the reaction of C_6F_5H (0.11 g, 0.65 mmol) with perfluoroindan (0.19 g, 0.64 mmol) in SbF_5 (1.11 g, 5.10 mmol) [13]. Then the mixture of isomers **4**:**19** = 76:24 (0.20 g, 0.43 mmol) was added. The resulting solution was heated at 75 °C for 48 h, treated with 5% hydrochloric acid, extracted with CH_2Cl_2 and dried over $MgSO_4$. The solvent was distilled off to give 0.44 g of a mixture, which contained compound **19**, perfluoro-1-phenylindan and perfluoro-1-phenylindan-1-ol in the ratio 40:15:45.

*4.5. Reaction of perfluoro-3-methyl-3-phenylindan-1-one (**19**) with SbF_5*

1. A mixture of compound **19** (0.20 g) and SbF_5 (1.27 g) (molar ratio, 1:13.8) was heated at 75 °C for 29 h. The mixture was

treated with 5% hydrochloric acid and extracted with CH_2Cl_2. The solution contained compounds **19**, **20** and **21** in the ratio 71:24:5. The extract was washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. The solvent was distilled off to give 0.15 g of mixture containing compounds **19** and **21** in the ratio 93:7. The aqueous solution of $NaHCO_3$ was acidified with HCl, extracted with CH_2Cl_2 and dried over $MgSO_4$. The solvent was distilled off and the residue was sublimed (140 °C, 3 Torr) to give 0.04 g of acid **20** (mixture of isomers in the ratio 80:20).

2. Analogously to the previous experiment, the reaction of compound **19** (0.11 g) and SbF_5 (1.17 g) (molar ratio, 1:23) gave (75 °C, 12 h, then 95 °C 33 h) an extract containing compounds **19**, **20** and **21** in the ratio 17:8:75. The extract was washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. The solvent was distilled off to give 0.09 g of a mixture containing compounds **19** and **21** in the ratio 19:81. Crystallization from hexane gave 0.042 g of compound **21**.

4.5.1. Perfluoro-4-methyl-3-phenylisochromen-1-one (21)

Mp 137.5–138.5 °C (hexane). IR (CCl_4) ν, cm^{-1}: 1790 (C=O); 1512, 1483 (FAR). ^{19}F NMR ($CDCl_3$): δ −58.6 (3F, CF_3), −130.8 (1F, F-8), −132.2 (1F, F-5), −139.7 (2F, F-*ortho*), −140.3 (1F, F-6), −148.1 (1F, F-*para*), −148.9 (1F, F-7), −160.5 (2F, F-*meta*); $J_{CF3-F(5)}$ = 46, $J_{CF3-F(ortho)}$ = 2, $J_{ortho,para}$ = 4, $J_{meta,para}$ = 21, $J_{5,6}$ = 19, $J_{5,7}$ = 6.5, $J_{5,8}$ = 13, $J_{6,7}$ = 20.5, $J_{6,8}$ = 12, $J_{7,8}$ = 20.5. HRMS *m*/*z*, 451.9700 (M^+). Calcd. for $C_{16}F_{12}O_2$ = 451.9701.

4.6. Synthesis of perfluoro-4-methyltetralin-1-one (2)

1. To a stirred solution of tetralone **1** (7.69 g) in 20 ml of dry diethyl ether 46 ml of ether solution of CH_3Li (molar ratio, 1:1) was slowly added at 5 °C under argon atmosphere. The mixture was treated with 60 ml 5% hydrochloric acid. Organic layer was separated and the ether was distilled off the residue was dissolved in CH_2Cl_2 and dried over $MgSO_4$. Solution contained compounds **1** and **35** in the ratio 23:77. Vacuum distillation gave 5.32 g of the product (bp 60–66 °C, 3 Torr) containing compounds **1** and **35** in the ratio 5:95. Individual compound **35** (3.92 g, yield 49%) was obtained by crystallization from hexane.

4.6.1. 2,2,3,3,4,4,5,6,7,8-Decafluoro-1-methyltetralin-1-ol (35)

Mp 51–52 °C (hexane). IR (CCl_4) ν, cm^{-1}: 3589 (OH); 1527, 1491 (FAR). ^1H NMR ($CDCl_3$): δ 2.87 (1H, OH), 1.86 (3H, CH_3). ^{19}F NMR ($CDCl_3$): δ −100.7 (1F_A) and −111.9 (1F_B, $J_{A,B}$ = 290, CF_2-4), −128.5 (1F_A) and −135.4 (1F_B, $J_{A,B}$ = 271, CF_2-2 or CF_2-3), −128.8 (1F_A) and −139.1 (1F_B, $J_{A,B}$ = 273, CF_2-2 or CF_2-3), −136.3 (1F, F-8), −137.7 (1F, F-5), −146.7 (1F, F-7), −151.3 (1F, F-6); $J_{4A,5}$ = 13, $J_{4B,5}$ = 29, $J_{5,6}$ = 21, $J_{5,7}$ = 8, $J_{5,8}$ = 12, $J_{6,7}$ = 20.5, $J_{6,8}$ = 7, $J_{7,8}$ = 20.5. HRMS *m*/*z*, 342.0065 (M^+). Calcd. for $C_{11}H_4F_{10}O$ = 342.0097.

2. A mixture of compound **35** (3.82 g), $SOCl_2$ (2.5 ml) (molar ratio, 1:3), CCl_4 (2 ml) and DMF (3 drops) was heated at 75 °C (19 h). The mixture was treated with water and extracted with CCl_4. The extract was dried over $MgSO_4$. The solvent was distilled off and the residue was heated with PCl_5 (12.59 g) at 235–240 °C (24 h) in a sealed ampoule. Reaction mixture was treated with water, extracted with CH_2Cl_2 and dried over $MgSO_4$. Vacuum distillation gave 3.38 g (yield 77%) of compound **36**.

4.6.2. 1-(Dichloromethylene)-2,2,3,3,4,4,5,6,7,8-decafluorotetralin (36)

Liquid, bp 68–69 °C (2 Torr). ^{19}F NMR ($CDCl_3$): δ −106.2 (1F_A) and −111.8 (1F_B, $J_{A,B}$ = 285, CF_2-2 or CF_2-4), −108.8 (1F_A) and −120.2 (1F_B, $J_{A,B}$ = 260, CF_2-2 or CF_2-4), −126.4 (1F, F-8), −131.7 (1F_A) and −134.6 (1F_B, $J_{A,B}$ = 253, CF_2-3), −137.7 (1F, F-5), −146.7 (1F, F-7), −148.9 (1F, F-6); $J_{5,6}$ = 21, $J_{5,7}$ = 8.5, $J_{5,8}$ = 12, $J_{6,7}$ = 20, $J_{6,8}$ = 8.5, $J_{7,8}$ = 21. HRMS *m*/*z*, 391.9211 (M^+). Calcd. for $C_{11}Cl_2F_{10}$ = 391.9212. Anal. Calcd. for $C_{11}Cl_2F_{10}$: C, 33.6; Cl, 18.0%. Found: C, 33.5; Cl, 18.0%.

3. A mixture of compound **36** (3.17 g) and CsF (6.7 g) (molar ratio, 1:5.5) was heated at 250–260 °C (6 h) in a sealed ampoule, then volatile products were separated by distillation. Resulting mixture, which contained compounds **36** and **37** in the ratio 12:88, was heated with CsF (3.48 g) at 250–260 °C (10 h) again. Distillation gave 2.74 g (yield 94%) of product **37**.

4.6.3. Perfluoro-4-methyl-1,2-dihydronaphthalene (37)

Liquid. ^{19}F NMR ($CDCl_3$): δ −60.5 (3F, CF_3), −113.3 (1F, F-3), −122.2 (2F, CF_2-1), −130.3 (1F, F-5), −132.0 (2F, CF_2-2), −136.2 (1F, F-8), −146.1 (1F, F-6), −148.6 (1F, F-7); $J_{1,3}$ = 2, $J_{1,6}$ = 2.5, $J_{1,8}$ = 36, $J_{2,3}$ = 16, $J_{2,4}$ = 2, $J_{3,4}$ = 29.5, $J_{3,5}$ = 5.5, $J_{3,6}$ = 1.5, $J_{3,7}$ = 6, $J_{3,8}$ = 2, $J_{4,5}$ = 35.5, $J_{5,6}$ = 20, $J_{5,7}$ = 8, $J_{5,8}$ = 11, $J_{6,7}$ = 19.5, $J_{6,8}$ = 9.5, $J_{7,8}$ = 21. HRMS *m*/*z*, 359.9804 (M^+). Calcd. for $C_{11}F_{12}$ = 359.9803.

4. A mixture of compound **37** (2.61 g) and PCl_5 (3.15 g) (molar ratio, 1:2) was heated at 225–235 °C (6 h) in a sealed ampoule. Reaction mixture was treated with water, extracted with CH_2Cl_2 and dried over $MgSO_4$. The solvent was distilled off and the residue was heated with CsF (5.41 g) at 250–260 °C (9 h) and then at 260–270 °C (8.5 h) in a sealed ampoule. Distillation of reaction mixture gave 2.71 g (yield 94%) of tetralins **33** and **38** in the ratio 50:50.

4.6.4. Perfluoro-1-methyltetralin (33) and perfluoro-5-methyltetralin (38)

Mixture of two isomers, ratio **33**:**38** = 50:50; liquid. *Compound* **33**: ^{19}F NMR ($CDCl_3$): δ −72.6 (3F, CF_3), −100.1 (1F_A) and −107.6 (1F_B, $J_{A,B}$ = 292, CF_2-4), −123.6 (1F_A) and −133.5 (1F_B, $J_{A,B}$ = 281, CF_2-2 or CF_2-3), −130.6 (1F_A) and −136.5 (1F_B, $J_{A,B}$ = 286, CF_2-2 or CF_2-3), −131.5 (1F, F-8), −134.3 (1F, F-5), −143.9 (1F, F-7), −144.4 (1F, F-6), −177.6 (1F, F-1); $J_{CF3-F(1)}$ = 8.5, $J_{CF3-F(8)}$ = 26.5, $J_{1,8}$ = 32.5, $J_{4A,5}$ = 21, $J_{4B,5}$ = 22, $J_{5,6}$ = 21.5, $J_{5,7}$ = 9.5, $J_{5,8}$ = 11.5, $J_{6,7}$ = 20.5, $J_{6,8}$ = 10, $J_{7,8}$ = 20.

Compound **38**: ^{19}F NMR ($CDCl_3$): δ −55.6 (3F, CF_3), −103.3 (2F, CF_2-4), −105.5 (2F, CF_2-1), −117.4 (1F, F-6), −123.0 (1F, F-8), −135.5 (2F) and −136.1 (2F, CF_2-2, CF_2-3), −145.4 (1F, F-7); $J_{1,8}$ = 21, $J_{4,5}$ = 24.5, $J_{5,6}$ = 29.5, $J_{6,7}$ = 20.5, $J_{6,8}$ = 20.5, $J_{7,8}$ = 20.5. HRMS (mixture of **33** and **38**) *m*/*z*, 397.9768 (M^+). Calcd. for $C_{11}F_{14}$ = 397.9771

5. A mixture of tetralins **33** and **38** (2.59 g), SiO_2 (0.58 g) and SbF_5 (5.36 g) (molar ratio, 0.5:0.5:1.5:4) was stirred at 75 °C (6.5 h) and then at 100 °C (1 h). The mixture was treated with 5% hydrochloric acid and extracted with CH_2Cl_2. The extract was washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. The extract was dried over $MgSO_4$. The solvent was distilled off to give 1.17 g (yield 96%) of ketone **2**.

4.6.5. Perfluoro-4-methyltetralin-1-one (2)

Mp 29–31 °C. IR (CCl_4) ν, cm^{-1}: 1738 (C=O); 1518, 1487 (FAR). ^{19}F NMR (Et_2O): δ −73.9 (3F, CF_3), −117.8 (1F_A) and −127.2 (1F_B, CF_2-2), −118.6 (1F_A) and −132.9 (1F_B, CF_2-3), −131.5 (1F, F-8), −132.6 (1F, F-5), −137.6 (1F, F-6), −144.5 (1F, F-7), −185.3 (1F, F-4); $J_{CF3-F(2A)}$ = 21, $J_{CF3-F(3A)}$ = 5, $J_{CF3-F(3B)}$ = 16, $J_{CF3-F(4)}$ = 10, $J_{CF3-F(5)}$ = 16, $J_{2A,2B}$ = 303, $J_{2A,3A}$ = 3, $J_{2A,3B}$ = 12.5, $J_{2B,3A}$ = 13, $J_{2B,3B}$ = 10, $J_{2B,4}$ = 10, $J_{3A,3B}$ = 273, $J_{3A,4}$ = 16, $J_{3B,4}$ = 12.5, $J_{4,5}$ = 49.5, $J_{4,7}$ = 1.5, $J_{4,8}$ = 2, $J_{5,6}$ = 20, $J_{5,7}$ = 10, $J_{5,8}$ = 12.5, $J_{6,7}$ = 19.5, $J_{6,8}$ = 15.5, $J_{7,8}$ = 20. HRMS *m*/*z*, 375.9753 (M^+). Calcd. for $C_{11}F_{12}O$ = 375.9752.

4.7. Synthesis of perfluoro-4-ethyltetralin-1-one (3)

A mixture of compound **34** (3.74 g), SiO_2 (0.51 g) and SbF_5 (5.44 g) (molar ratio, 1:1:3) was stirred at 70 °C (1.5 h) and then at

100 °C (3 h). The mixture was treated with 5% hydrochloric acid and extracted with CH_2Cl_2. The extract was dried over $MgSO_4$. The solvent was distilled off to give 3.40 g (yield 96%) of ketone **3**.

4.7.1. Perfluoro-4-ethyltetralin-1-one (3)

Liquid. IR (CCl_4) ν, cm^{-1}: 1740 (C=O); 1517, 1489 (FAR). ^{19}F NMR (Et_2O): δ −79.4 (3F, CF_3), −114.7 (1F_A) and −131.0 (1F_B, $J_{A,B}$ = 269, CF_2-3), −115.8 (1F_A) and −125.4 (1F_B, $J_{A,B}$ = 305, CF_2-2), −116.2 (1F_A) and −117.8 (1F_B, $J_{A,B}$ = 300, **CF_2**CF_3), −131.8 (1F, F-8), −132.8 (1F, F-5), −138.0 (1F, F-6), −144.3 (1F, F-7), −188.3 (1F, F-4); $J_{4,5}$ = 59, $J_{4,8}$ = 2.5, $J_{5,6}$ = 20, $J_{5,7}$ = 10, $J_{5,8}$ = 12.5, $J_{6,7}$ = 20, $J_{6,8}$ = 15, $J_{7,8}$ = 20. HRMS *m*/*z*, 425.9733 (M^+). Calcd. for $C_{12}F_{14}O$ = 425.9725.

4.8. Synthesis of perfluoro-4-phenyltetralin-1-one (4)

A mixture of compound **1** (0.76 g), C_6F_5H (0.78 g) and SbF_5 (4.01 g) (molar ratio, 1:2:8) was heated at 50–55 °C (50 h) in a sealed ampoule. Reaction mixture was placed in a Teflon container, dissolved in 10 ml of anhydrous HF, kept at room temperature for 2 h, poured to ice and extracted with CH_2Cl_2. The extract was dried over $MgSO_4$. Solution obtaining after incomplete solvent distillation contained compounds **1**, **4**, **19**, C_6F_5H and $C_6F_5C_6F_5$ in the ratio 46:21:7:18:8. The mixture was spontaneously evaporated in the air to dryness and the residue was sublimed (100 °C, 3 Torr) to give 0.33 g (yield 30%) of mixture containing tetralone **4** and indanone **19** in the ratio 76:24.

4.8.1. Perfluoro-4-phenyltetralin-1-one (4)

Mixture of isomers **4** and **19** in the ratio 76:24. ^{19}F NMR ($CDCl_3$): δ −120.5 (1F_A) and −131.5 (1F_B, $J_{A,B}$ = 277, CF_2-2 or CF_2-3), −122.1 (1F_A) and −134.3 (1F_B, $J_{A,B}$ = 281, CF_2-2 or CF_2-3), −132.9 (1F, F-5), −133.5 (1F, F-8), −137.5 (2F, F-*ortho*), −139.2 (1F, F-6), −146.0 (1F, F-7), −148.3 (1F, F-*para*), −152.2 (1F, F-4), −160.1 (2F, F-*meta*); $J_{ortho,para}$ = 5, $J_{meta,para}$ = 21, $J_{4,5}$ = 8, $J_{4,6}$ = 2, $J_{4,7}$ = 6, $J_{5,6}$ = 20.5, $J_{5,7}$ = 8, $J_{5,8}$ = 12, $J_{6,7}$ = 20, $J_{6,8}$ = 13.5, $J_{7,8}$ = 20. HRMS (mixture of **4** and **19**) *m*/*z*, 473.9718 (M^+). Calcd. for $C_{16}F_{14}O$ = 473.9720.

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