

Generation of Chloropolyfluoromethylene-1-indanyl Cations and Their Isomerization into Chloropolyfluoromethylindenyl Cations*

V. M. Karpov and V. E. Platonov

Vorozhtsov Novosibirsk Institute of Organic Chemistry, Siberian Division, Russian Academy of Sciences,
pr. Akademika Lavrent'eva 9, Novosibirsk, 630090 Russia

Received June 2, 2000

Abstract—1-Chlorooctafluoro-3-methylene-1-indanyl, 3-(chlorofluoromethylene)heptafluoro-1-indanyl, and 2- and 3-(dichloromethylene)heptafluoro-1-indanyl cations were generated from the corresponding dihalomethyleneindans in the system $\text{SbF}_5\text{-SO}_2\text{ClF}$. Isomerization of these cations yields thermodynamically more stable polyfluoromethylindenyl cations with chlorine atoms in the five-membered ring.

We previously reported [1, 2] on the generation of perfluorinated alkylindenyl and alkylideneindanyl cations. Unlike antiaromatic indenyl cation whose relative stability is lower than that of its analog with an open π system, perfluoroalkylindenyl cations are thermodynamically more stable than isomeric perfluoroalkylideneindanyl systems [2]. With the goal of elucidating the effect of chlorine and fluorine atoms on the relative stability of haloalkylindenyl and haloalkylideneindanyl cations in the present work we studied generation of chlorine-containing polyfluoromethylene-1-indanyl cations with an open π system and their isomerization into polyfluorinated methylindenyl cations having a closed π system.

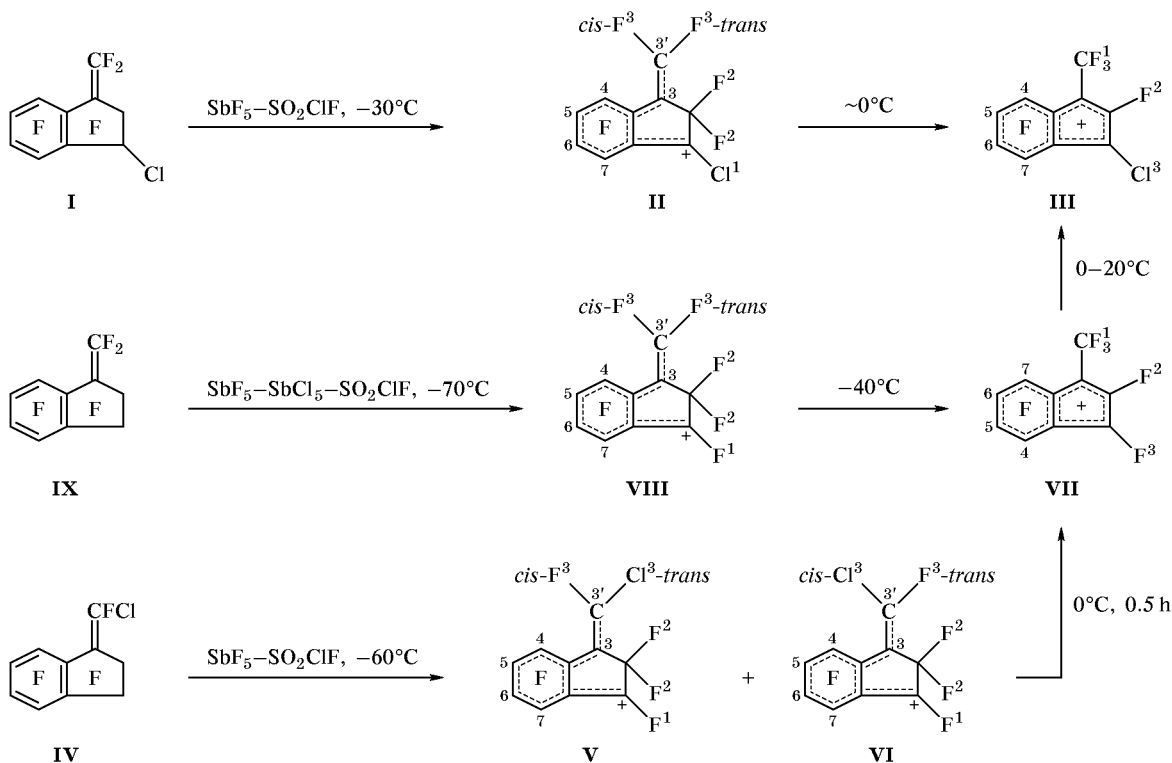
We have found that dissolution of 3-chlorononafluoro-1-methyleneindan (**I**) in a mixture of SbF_5 with SO_2ClF at low temperature gives rise to 1-chlorooctafluoro-3-methylene-1-indanyl cation (**II**). As the temperature rises, cation **II** undergoes isomerization into 3-chlorooctafluoro-1-methylindenyl cation (**III**). Likewise, from a mixture of (*Z*)- and (*E*)-1-(chlorofluoromethylene)octafluoroindans **IV** we obtained (*Z*)- and (*E*)-3-(chlorofluoromethylene)heptafluoro-1-indanyl cations **V** and **VI** whose thermal isomerization also gave ion **III** (Scheme 1). The isomerization of **V** and **VI** into **III** involves intermediate formation of perfluoro-1-methylindenyl cation (**VII**) in which the 3-fluorine atom is replaced by chlorine through the

reaction with antimony chloride fluorides generated during the process (Scheme 1). By special experiment we showed that on addition of SbCl_5 (~0.35 mol per mole of the precursor) to SbF_5 cation **VII** is converted into ion **III**. On the other hand, the isomerization of perfluoro(3-methylene-1-indanyl) cation (**VIII**) to ion **VII** is faster than its transformation into ion **II**. Perfluoro(1-methyleneindan) (**IX**) in the system $\text{SbF}_5\text{-SbCl}_5\text{-SO}_2\text{ClF}$ at -70°C gives rise to both ion **VII** and methyleneindanyl cation **VIII**. As the temperature rises to -40°C , cation **VIII** undergoes isomerization to ion **VII** without formation of ion **II**. The transformation of ion **VII** into **III** is complete when the solution is kept for 1 h at room temperature (Scheme 1).

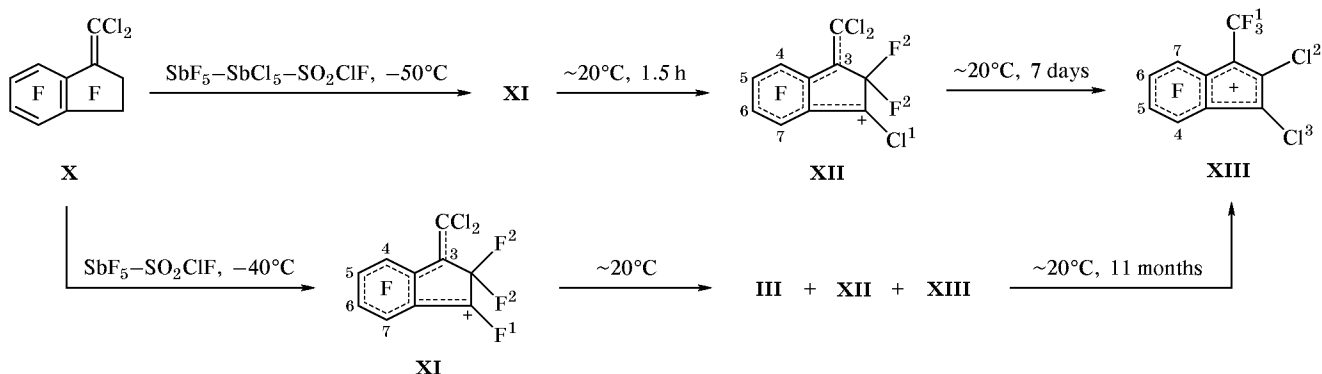
3-(Dichloromethylene)heptafluoro-1-indanyl cation (**XI**) was generated from 1-(dichloromethylene)octafluoroindane **X** in the system $\text{SbF}_5\text{-SbCl}_5\text{-SO}_2\text{ClF}$. At room temperature the 1-fluorine atom in **XI** is replaced by chlorine to afford 1-chloro-3-dichloromethylenehexafluoro-1-indanyl cation (**XII**). The latter is then converted into 2,3-dichloroheptafluoro-1-methylindenyl cation (**XIII**). In the absence of SbCl_5 the isomerization of **XI** into **XIII** proceeds at a considerably lower rate. When a solution containing ion **XI**, prepared from compound **X** and SbF_5 in SO_2ClF , was kept for 36 h at room temperature, a mixture of ions **III** and **XI-XIII** was formed. After 5.5 days, the solution contained approximately equal amounts of cations **III** and **XIII** and traces of **XII**. After 4.5 months, the ratio of ions **XIII** and **III** was about 2:1, and after 11 months, ion **III** was almost completely transformed into **XIII** (Scheme 2).

* This study was financially supported by the Russian Foundation for Basic Research (project no. 99-03-32876).

Scheme 1.



Scheme 2.

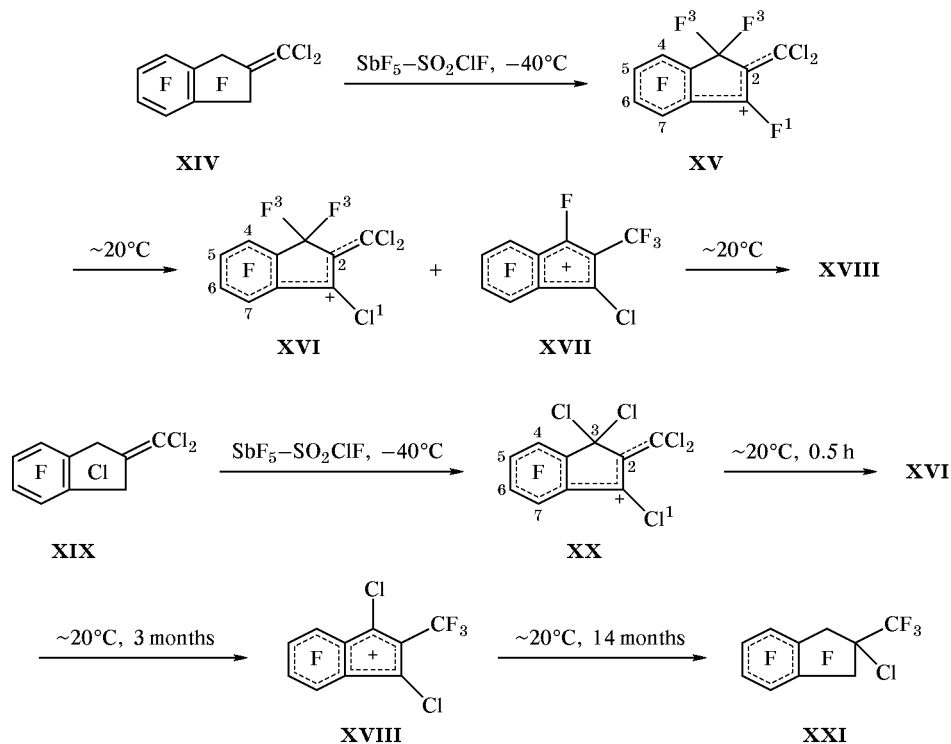


An analogous pattern was also observed with 2-(dichloromethylene)octafluoroindane (**XIV**) as precursor of cationic species. In the system $\text{SbF}_5\text{-SO}_2\text{ClF}$ at low temperature compound **XIV** gives rise to 2-(dichloromethylene)heptafluoro-1-indanyl cation (**XV**). On keeping for 11 days at room temperature, a mixture of ion **XV** (major component), 1-chloro-2-dichloromethylenehexafluoro-1-indanyl cation (**XVI**), 1-chlorooctafluoro-2-methylindenyl cation (**XVII**), and 1,3-dichloroheptafluoro-2-methylindenyl cation (**XVIII**) was obtained. After 11 months, the solution contained the same ions, but cations **XVII** and **XVIII**

were the major components (ratio $\sim 1:4$; Scheme 3). We showed previously [3] that heating of a solution of compound **XIV** in the system $\text{SbF}_5\text{-SO}_2$ for 6 h at 110°C leads to complete isomerization of ion **XV** to cation **XVIII**.

The reaction of 2-dichloromethylene-1,1,3,3-tetrachlorotetrafluoroindane (**XIX**) with SbF_5 in SO_2 generates 2-dichloromethylene-1,1,3-trichlorotetrafluoro-1-indanyl cation (**XX**) [4]; in SO_2ClF , ions **XX** and **XVI** are formed. Raising the temperature of the SO_2ClF solution to $\sim 20^\circ\text{C}$ leads to replacement of all chlorine atoms in position 3 of ion **XX** by

Scheme 3.



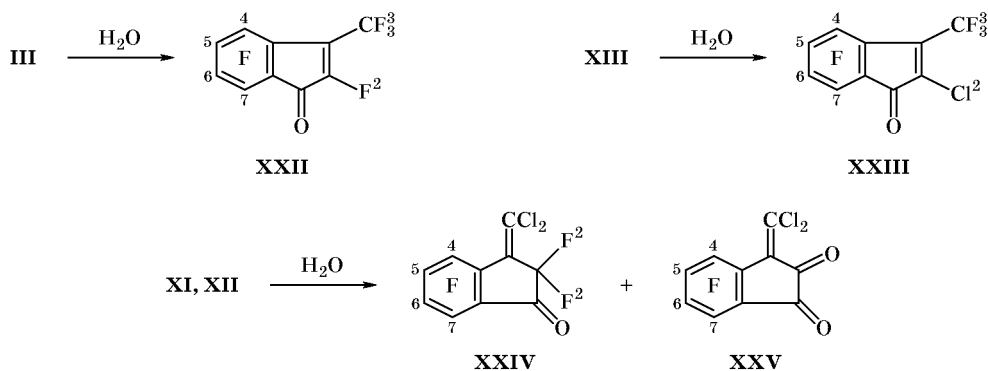
fluorine, yielding cation **XVI**. As compared to **XV**, ion **XVI** is relatively rapidly converted into methylindanyl cation **XVIII**. At room temperature the transformation is complete in 3 months. According to the ^{19}F NMR spectrum, the resulting mixture contains a small amount of 2-chloroundecafluoro-2-methylindan (**XXI**). The latter is formed as the major product when compound **XIX** was kept in the system $\text{SbF}_5\text{-SO}_2\text{ClF}$ total of 14 months.

Thus, chlorine-containing polyfluoromethylindanyl cations **III**, **XIII**, and **XVIII** which have a closed π system are thermodynamically more stable than isomeric methyleneindanyl ions with an open π

system; The stability series is as follows: **III** > **II**, **V**, **VI**; **XIII** > **XII**; **XVIII** > **XV**.

The structure of the generated cationic species was derived from their ^{19}F NMR spectra and was proved by hydrolysis of the corresponding salts (with ions **III** and **XI–XIII**). Treatment with water of solutions containing cations **III** and **XIII** gave, respectively, perfluoro(3-methylinden-1-one) (**XXII**) and 2-chloroheptafluoro-3-methylinden-1-one (**XXIII**). The hydrolysis of cation **XII** yields 3-(dichloromethylene)hexafluoro-1-indanone (**XXIV**). Ketone **XXIV** was formed in a mixture with 3-(dichloromethylene)tetrafluoro-1,2-indandione (**XXV**) by treatment with water

Scheme 4.



of a SbF_5 solution containing ion **XI** and no SO_2ClF (Scheme 4).

The ^{19}F NMR spectra of cations **VII**, **VIII** [2], **XV** [3], **XVII**, **XVIII**, and **XX** [4] were consistent with those reported previously. The spectra of methylindenyl cations **III** and **XIII** (Table 1) are analogous to that observed for ion **VII** [5], and the spectra of methyleneindanyl cations **II**, **V**, **VI**, **XI**, **XII**, and **XVI** (Table 2) resemble those of ions **VIII** [2] and **XX** [4]. In all cases, the signals from fluorine atoms attached to the sp^2 -hybridized carbon atoms are displaced downfield relative to the corresponding signals of their neutral precursors. The chemical shifts of these fluorine nuclei are likely to reflect qualitative pattern of π -charge distribution over carbon atoms attached thereto: Linear correlations were found previously between the π -charges on carbon atoms and ^{19}F chemical shifts for the other polyfluoroindenyl [2, 5] and perfluoro(3-alkylidene-1-indanyl) cations [2]. The most downfield fluorine signals are characterized by the greatest spin-spin coupling constants. In the spectra of benzyl-like methyleneindanyl cations, such signals are those from fluorine atoms located in the resonance (charge) positions, 1, 3', 5, and 7. It should be noted that J values of polyfluorinated benzyl-like [6] and arenonium ions [7] are considered to be related to direct participation of fluorine atoms in charge distribution and conjugation. The 4-F atom in ions **II**, **V**, and **VIII** occupies an off-resonance position, and the greater $J_{cis-3,4}$ (as in neutral precursors [8]) is likely to result from the short distance between the interacting nuclei rather than from positive charge distribution. It is also interesting that methylindenyl cations **III**, **VII**, and **XIII** having a cyclic π system are characterized by anomalously low coupling constants $J_{5,6}$ (<2 Hz) for fluorine atoms located *ortho* with respect to each other; by contrast, the $J_{5,6}$ values (17–21 Hz) of methyleneindanyl cations with an open π system are typical of *ortho*-fluorine atoms in a phenyl ring [9].

EXPERIMENTAL

The ^{19}F NMR spectra of salt solutions in SO_2ClF or SbF_5 and of solutions of neutral compounds in CCl_4 were recorded on Varian A-56/60A and Bruker WP-200SY spectrometers operating at 56.4 and 188.3 MHz, respectively; SO_2ClF (δ_{F} 262.8 ppm relative to C_6F_6) and C_6F_6 were used as internal references. The IR spectra were obtained on a UR-20 instrument from solutions in CCl_4 . The UV spectra were measured on a Specord UV-Vis spectrometer using heptane as solvent. The elemental compositions

were determined by high-resolution mass spectrometry on an AEI-MS 902 instrument.

Compounds **I**, **IV** [8], **X**, **XIV** [10], and **XIX** [4] as precursors of cationic species were synthesized by known procedures.

1-Chlorooctafluoro-3-methylene-1-indanyl cation (II). A solution of 0.34 g of SbF_5 in ~0.3 ml of SO_2ClF was placed in an NMR ampule and cooled to -70°C , and a solution of 0.13 g of compound **I** (molar ratio **I**– SbF_5 1:4) in ~0.3 ml of SO_2ClF was added. The mixture was stirred by shaking, and the ^{19}F NMR spectra were recorded which contained signals of ion **II**. The latter did not change in the temperature range from -60 to -30°C . The solution was then kept for 2.5 h at -3 to 5°C ; its ^{19}F NMR spectrum contained signals from cations **II** and **III** (ratio ~1:1); after 16 h, only signals of **III** were present in the spectrum.

(Z)- and (E)-3-(chlorofluoromethylene)heptafluoro-1-indanyl cations V and VI. A solution containing ions **V** and **VI** at a ratio of ~2:1 (according to the ^{19}F NMR data) was prepared by the procedure described above from 0.14 g of compound **IV** and 0.36 g of SbF_5 (molar ratio 1:4) at -70 to -60°C . The ^{19}F NMR spectrum recorded at -30°C (after raising the temperature to $\sim 0^\circ\text{C}$ for a short time) contained signals of cation **VII** (the ratio of ions **V** and **VI** was ~6:1). When the solution was kept for 0.5 h at 0 – 1°C , ions **V** and **VI** were completely converted into **VII**. After 16 h at -3 to 5°C , only signals of ion **III** were observed in the ^{19}F NMR spectrum.

Transformations of perfluoro(1-methyleneindan) (IX) in the system SbF_5 – SbCl_5 . *a.* As described above, from 0.12 g of compound **IX**, 0.33 g of SbF_5 , and 0.04 g of SbCl_5 (1:4:0.35) in SO_2ClF at -70°C a mixture of ions **VII** and **VIII** was generated. On raising the temperature to -40°C , the solution contained only ion **VIII**. The solution was then kept for 1.5 h at room temperature. According to the ^{19}F NMR data, ion **VIII** was completely converted into ion **III**.

b. To a solution of 0.11 g of SbCl_5 in 1.54 g of SbF_5 at 20°C we added 0.33 g of compound **IX** (ratio **IX**– SbF_5 – SbCl_5 1:6.76:0.35), and the mixture was stirred and kept for 5 h at 20°C . According to the ^{19}F NMR data, the resulting solution contained cation **III**. The solution was poured into ice water and treated with methylene chloride, the extract was dried over MgSO_4 , and the solvent was removed. We obtained 0.27 g of a product which was subjected to vacuum sublimation at 45 – 50°C (15–20 mm) to isolate 0.21 g of ketone **XXII**. The product was identical to that described in [11].

Table 1. ^{19}F NMR spectra of polyfluorinated 1-methylindenyl cations **III**, **VII**, and **XIII**

Cation no.	Chemical shifts δ_{F} , ppm (relative to C_6F_6)						
	CF_3	2-F	3-F	4-F	5-F	6-F	7-F
III	97.6	45.3		98.3	20.4	70.8	63.6
VII [2]	97.3	40.2	168.3	110.5	20.7	79.5	66.2
XIII	97.1			94.5	20.0	67.1	63.8

Cation no.	^{19}F - ^{19}F coupling constants J_{ij} , Hz												
	1-2	1-7	2-3	2-4	2-5	2-6	2-7	4-5	4-6	4-7	5-6	5-7	6-7
III	17	17		5	17	12	17	20	57	17	<1	11	10
VII [2]	15	15	4	4	19	13	15	21	62	15	<2	11	11
XIII		21						20	53	15	<1	11	11

3-(Dichloromethylene)heptafluoro-1-indanyl cation (XI) and 1-chloro-3-dichloromethylenehexafluoro-1-indanyl cation (XII). *a.* To a solution of 0.33 g of SbF_5 and 0.04 g of SbCl_5 in SO_2ClF we added at -75°C 0.13 g of compound **X** (ratio $\text{X}-\text{SbF}_5-\text{SbCl}_5$ 1:4:0.35), and the mixture was stirred. According to the ^{19}F NMR data, the mixture contained ion **XI** which did not change in the temperature range from -70 to -20°C . The mixture was kept for 100 min at $20-22^\circ\text{C}$; it contained cation **XII** and small amounts of ions **XI** and **XIII**. After keeping for a week at room temperature, only ion **XIII** was present in the solution. Its ^{19}F NMR spectrum did not change on keeping the mixture for an additional 7 days. The solution was poured into water and was treated as described above to isolate 0.11 g of product **XXIII** which was purified by vacuum sublimation (110°C , 15 mm). Yield 0.06 g, mp $84-86^\circ\text{C}$ (in a sealed capillary).

2-Chloroheptafluoroinden-1-one (XXIII). IR spectrum, ν , cm^{-1} : 1755 (C=O), 1640 (C=C), 1500 (fluorinated aromatic ring). UV spectrum, λ_{max} , nm ($\log \epsilon$): 325 sh (3.35), 337 (3.45), 352 (3.38), 394 (3.16). Found: $[M]^+$ 303.9562. $\text{C}_{10}\text{ClF}_7\text{O}$. Calculated: M 303.9525.

b. A solution containing ions **XI** and **XII** (~1:3) was prepared from 0.28 g of compound **X**, following the above procedure. The mixture was poured into water and extracted with methylene chloride, and the extract was dried over MgSO_4 and transferred onto a watch glass. We thus obtained 0.25 g of a mixture of compounds **X**, **XXIV**, and **XXV** at a ratio of 16:81:3 (according to the ^{19}F NMR data).

c. A solution containing cation **XI** (^{19}F NMR data) was obtained by mixing at room temperature 0.33 g of compound **X** and 1.45 g of SbF_5 (1:7). The mixture was poured into water to obtain 0.25 g of a mixture of compounds **X**, **XXIV**, and **XXV** at a ratio of 1:2:2. It was separated by column chromatography on silica gel (using carbon tetrachloride as eluent) to isolate 0.03 g of initial compound **X** and 0.08 g of ketone **XXIV**. The subsequent elution with CH_2Cl_2 gave 0.09 g of diketone **XXV** which was purified by vacuum sublimation at 100°C (15–20 mm), mp $99-100.2^\circ\text{C}$.

3-(Dichloromethylene)hexafluoroinden-1-one (XXIV). IR spectrum, ν , cm^{-1} : 1775 (C=O); 1610 (C=C); 1635, 1520, 1500 (fluorinated aromatic ring). UV spectrum, λ_{max} , nm ($\log \epsilon$): 250 (4.28), 263 sh (4.19), 273 sh (4.16), 285 sh (4.03), 332 (3.18). Found: $[M]^+$ 319.9224. $\text{C}_{10}\text{Cl}_2\text{F}_6\text{O}$. Calculated: M 319.9230.

3-(Dichloromethylene)tetrafluoroinden-1,2-dione (XXV). IR spectrum, ν , cm^{-1} : 1765, 1750 (C=O); 1575 (C=C); 1625, 1515, 1500 (fluorinated aromatic ring). UV spectrum, λ_{max} , nm ($\log \epsilon$): 273 sh (4.30), 282 (4.33), 349 (2.86). Found: $[M]^+$ 297.9186. $\text{C}_{10}\text{Cl}_2\text{F}_4\text{O}_2$. Calculated: M 297.9211.

d. Cation **XI** was generated from 0.13 g of compound **X** and 0.33 g of SbF_5 (1:4) in SO_2ClF at -40°C . The solution was then kept in a sealed ampule at room temperature, and the ^{19}F NMR spectra were recorded. After 36 h, the mixture contained ions **III**, **XI**, **XII**, and **XIII** at a ratio of 32:20:40:8; after 5.5 days, approximately equal amounts of cations **III** and **XIII** and traces of ion **XII** were detected; after

Table 2. ^{19}F NMR spectra of polyfluorinated 2- and 3-methylene-1-indanyl cations **II**, **V**, **VI**, **VIII**, **IX**, **XII**, **XV**, **XVI**, and **XX** and polyfluorinated ketones **XXII–XXV**

Cation or ketone no.	Chemical shifts δ_{F} , ppm (relative to C_6F_6)							
	1-F	2-F	<i>cis</i> -3-F	<i>trans</i> -3-F	4-F	5-F	6-F	7-F
II		45.3	119.3	115.3	37.5	95.7	24.0	63.3
V	149.5	37.8	139.5		44.1	102.6	26.1	69.5
VI	143.8	39.7		135.3	48.3	102.6	26.1	71.1
VIII [2]	147.2	38.4	122.2	116.5	40.4	103.0	26.0	70.4
XI	143.0	41.1			54.6	102.9	26.4	71.2
XII		48.0			51.9	96.2	25.1	64.9
XV	150.0			54.1	34.7	61.1	26.6	55.4
XVI				55.1	30.3	55.8	24.0	49.3
XX [4]					29.9	53.6	21.4	43.8
XXII		35.1	98.9 (CF_3)		25.6	21.4	12.0	31.0
XXIII			99.0 (CF_3)		27.3	20.6	11.9	29.9
XXIV		50.7			42.9	24.8	14.5	28.0
XXV					42.4	24.9	13.6	26.9

Cation or ketone no.	^{19}F – ^{19}F coupling constants J_{ij} , ^a Hz											
	1–2	1– <i>cis</i> -3	1–4	1–5	1–6	1–7	4–5	4–6	4–7	5–6	5–7	6–7
II							23	~9	~7	19	51	15
V	17	20	~9	35	~5	53	23	10	7	19	53	15
VI	17			~35		~50	~23	~10	~7	~19	~50	~15
VIII [2]	16	20	~9	34		~50	19	~10	~7	19	49	15
XI	19		9	36	4	58	21	10	7	21	54	17
XII							22	9	8	21	52	17
XV				15		52	22	12	13	20	31	21
XVI							20	9	12	17	30	19
XX [4]							21.5	9	13.5	20	28.5	20.5
XXII							19	5	14	16	12	20.5
XXIII							18	5	14	15	11	20
XXIV							19	7	16	18	12	20.5
XXV							19	7	16	18	12	21

^a Cation **II**: $J_{2,\text{trans-3}} = 7$, $J_{\text{cis-3},\text{trans-3}} = 64$, $J_{\text{cis-3},4} = 67$ Hz; cation **V**: $J_{\text{cis-3},4} = 89$ Hz; cation **VI**: $J_{2,\text{trans-3}} = 11$ Hz; cation **VIII**: $J_{2,\text{trans-3}} = 6$, $J_{\text{cis-3},\text{trans-3}} = 6$, $J_{\text{cis-3},4} = 64$ Hz; cation **XXII**: $J_{2,3} = 21$, $J_{2,4} = 9$, $J_{2,6} = 15.5$, $J_{\text{cis-3},4} = 20.5$ Hz; cation **XXIII**: $J_{\text{cis-3},4} = 22$ Hz.

4.5 months, the ratio of ions **XIII** and **III** was ~2:1; and after 11 months, the fraction of **XIII** was much greater than that of **III**.

1,3,3-Trichloro-2-dichloromethylenetetrafluoro-1-indanyl cation (XX) and 1-chloro-2-dichloromethylenehexafluoro-1-indanyl cation (XVI). A solution containing cation **XX** (^{19}F NMR data) was prepared at -50°C from 0.18 g of compound **XIX** and 0.38 g of SbF_5 (1:4) in SO_2ClF . The mixture was kept in a sealed ampule at room temperature. After 0.5 h, the solution contained cation **XVI**; after 4 days,

ions **XVI** and **XVIII** at a ratio of ~5:1; after 1.5 months, this ratio changed to ~1:4; and after 3 months, ions **XVI** and **XVIII** (**XVI** \ll **XVIII**) and a small amount of chloroindan **XXI** were present. After 7 months, the ratio of ions **XVIII** and **XXI** was 1.5:1; and after 14 months, only product **XXI** was detected, whereas cation **XVIII** disappeared. The mixture was poured into 5% hydrochloric acid, and the organic layer was separated and dried over MgSO_4 . We isolated 0.11 g of chloroindan **XXI** with small impurities (according to the ^{19}F NMR data).

2-Chloroundecafluoro-2-methylindan (XXI). ^{19}F NMR spectrum, δ_{F} , ppm: 94.6 (3F, CF_3), 71.9 (2F, 1- F_A and 3- F_A), 64.9 (2F, 1- F_B and 3- F_B , $J_{AB} = 260$ Hz), 24.0 (2F, 4-F and 7-F), 19.3 (2F, 5-F and 6-F). Found: $[M]^+$ 363.9467. $\text{C}_{10}\text{ClF}_{11}$. Calculated: M 363.9512.

2-(Dichloromethylene)heptafluoro-1-indanyl cation (XV). A solution containing ion **XV** was prepared from 0.13 g of compound **XIV** and 0.33 g of SbF_5 (1:4) in SO_2ClF at -50°C ; its ^{19}F NMR spectrum was recorded at -40°C , and the solution was kept in a sealed tube at room temperature. After 11 days, the ^{19}F NMR spectrum contained signals from ion **XV** (major) and **XVI–XVIII**. After 11 months, the same ions were present, but cations **XVII** and **XVIII** (molar ratio $\sim 1:4$) prevailed.

REFERENCES

1. Karpov, V.M., Platonov, V.E., and Yakobson, G.G., *Tetrahedron*, 1978, vol. 34, no. 21, pp. 3215–3218.
2. Karpov, V.M., Platonov, V.E., Chuikov, I.P., and Shchegoleva, L.N., *Russ. J. Org. Chem.*, 1999, vol. 35, no. 1, pp. 80–84.
3. Karpov, V.M., Platonov, V.E., and Yakobson, G.G., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1978, no. 11, p. 2655.
4. Karpov, V.M., Platonov, V.E., and Yakobson, G.G., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1979, no. 9, pp. 2082–2087.
5. Karpov, V.M., Platonov, V.E., and Shchegoleva, L.N., *Russ. J. Org. Chem.*, 1998, vol. 34, no. 11, pp. 1660–1665.
6. Olah, G.A. and Comisarov, M.B., *J. Am. Chem. Soc.*, 1967, vol. 91, no. 11, pp. 2955–2961; Pozdnyakov, Yu.V. and Shteingarts, V.D., *J. Fluorine Chem.*, 1974, vol. 4, no. 3, pp. 283–316.
7. Shteingarts, V.D., *Usp. Khim.*, 1981, vol. 50, no. 8, pp. 1407–1436.
8. Karpov, V.M., Platonov, V.E., Chuikov, I.P., and Yakobson, G.G., *J. Fluorine Chem.*, 1983, vol. 22, no. 5, pp. 459–473.
9. Pushkina, L.N., Stepanov, A.P., Zhukov, V.S., and Naumov, A.D., *Zh. Org. Khim.*, 1972, vol. 8, no. 3, pp. 586–597; Matthews, R.S. and Preston, W.E., *J. Org. Magn. Reson.*, 1980, vol. 14, no. 4, pp. 258–263.
10. Karpov, V.M., Platonov, V.E., Stolyarova, T.A., and Yakobson, G.G., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1981, no. 7, pp. 1586–1591.
11. Chuikov, I.P., Karpov, V.M., and Platonov, V.E., *Izv. Ross. Akad. Nauk, Ser. Khim.*, 1992, no. 6, pp. 1412–1418.