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Reactions of [Fluoro(methylsulfonyloxy)iodo]benzene: III. Reactions of [Fluoro(methylsulfonyloxy)iodo]benzene with Norbornene and Some Its Derivatives

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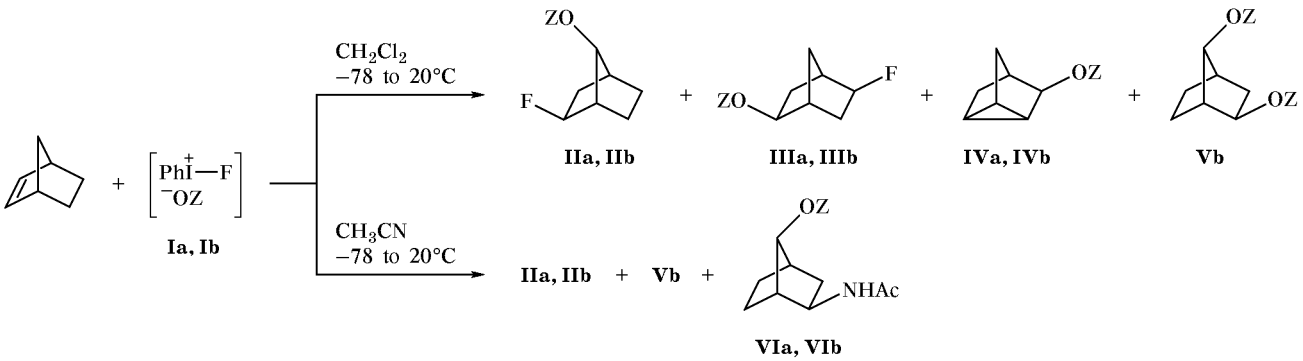
Abstract—Reactions of [fluoro(methylsulfonyloxy)iodo]benzene with norbornene, *cis*-5-norbornene-*endo*-2,3-dicarboxylic acid, and *cis*-5-norbornene-*endo*-2,3-dicarboxylic anhydride were studied. A new mild procedure was developed for introduction of fluoro and sulfonyloxy or two sulfonyloxy groups into molecules of norbornene and its derivatives.

Among tervalent iodine compounds, the most interesting are its sulfonyloxy derivatives primarily due to their high reactivity and wide application in organic synthesis [1]. For example, [fluoro(sulfonyloxy)iodo]benzene having a readily departing sulfonyloxy group exhibits pronounced electrophilic properties [2]; it was used by us for transformation of terminal [3–5] and internal acetylenes [6], ketones [7], and also monocyclic [8] and acyclic olefins [9]. With the goal of extending the series of appropriate substrates, in the present work we studied the reactions of [fluoro(sulfonyloxy)iodo]benzenes **Ia** and **Ib** with norbornene and some its derivatives.

In keeping with published data [10, 11] on reactions of electrophilic reagents with alkenes and on reactions of tervalent iodine compounds with norbornene [12, 13], we expected that compounds **Ia** and **Ib** would react with norbornene in nonpolar organic solvents to give the corresponding *cis*-1-fluoro-2-sulfonyloxy or *cis*-1,2-bis(sulfonyloxy) derivatives. However, even in such weakly polar and weakly ionizing solvent as methylene chloride, compounds **Ia** and **IIb** vigorously reacted with norbornene to afford products of profound skeletal rearrangements. The reactions were carried out following a general procedure: reagents **Ia** and **Ib** were generated *in situ* in methylene chloride at –78°C, a solution of norbornene in the same solvent was added, and the temperature was gradually raised to ambient. The progress of reactions was monitored by thin-layer chromatog-

Scheme 1.



Z = CF_3SO_2 (**a**), CH_3SO_2 (**b**).

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Table 1. Reactions of norbornene with compounds **Ia** and **Ib**

Reagent	Reaction time	Products (yield, %)
[PhI$^+$F$^-$OTf] (**Ia**)	7 h	**IIa** (25), **IIIa** (12), **IVa** (8)
[PhI$^+$F$^-$OMs] (**Ib**)	8 h	**IIb** (26), **IIIb** (18), **IVb** (11), **Vb** (13)

raphy on silica gel. The products were separated by column chromatography on silica gel. Products **II**–**VI** were isolated in the pure state. Unlike unstable compounds **IIa**, **IIb**, **IIIa**, **IIIb**, **VIa**, and **VIb**, product **Vb** can be stored in the cold for several days without appreciable decomposition (Scheme 1, Table 1).

The reactions of compounds **Ia** and **Ib** with norbornene under similar conditions but in a more polar solvent, namely acetonitrile, involves the solvent as external nucleophile. As a result, products **VIa** and **VIb** were formed together with **II** and **Vb**.

The structure of compounds **II**–**VI** was proved by the ^{1}H, ^{13}C, and ^{19}F NMR spectral data (Tables 2, 3) and elemental analyses. The NMR spectra were analyzed using ^{1}H–^{1}H, ^{1}H–^{19}F, and ^{13}C–^{19}F coupling constants. The *exo* orientation of the fluorine atom in **II** and **III** follows from the coupling constant $^3J_{C,F}$ whose value depends on the dihedral angle FCCC (φ); as in *exo*-fluoronorbornane [14], the bridging carbon atom (C^7) is characterized by $^3J_{C,F} \leq 1$ Hz (φ ≈ 80°), and methylene carbon atoms in the six-membered ring show $^3J_{C,F}$ values of 9–11 Hz (φ ≈ 170°). Analogous stereochemical relations are observed for $^3J_{H,F}$ (cf. [15]) and $^3J_{H,H}$. The coupling constant $^3J_{H,trans\text{-}H}$ for the methylene bridge containing fluorine is small (1.5–1.9 Hz). Important parameters for structural assignments in norbornane derivatives are vicinal coupling constants between the methylene protons and protons in the bridgehead

Scheme 2.

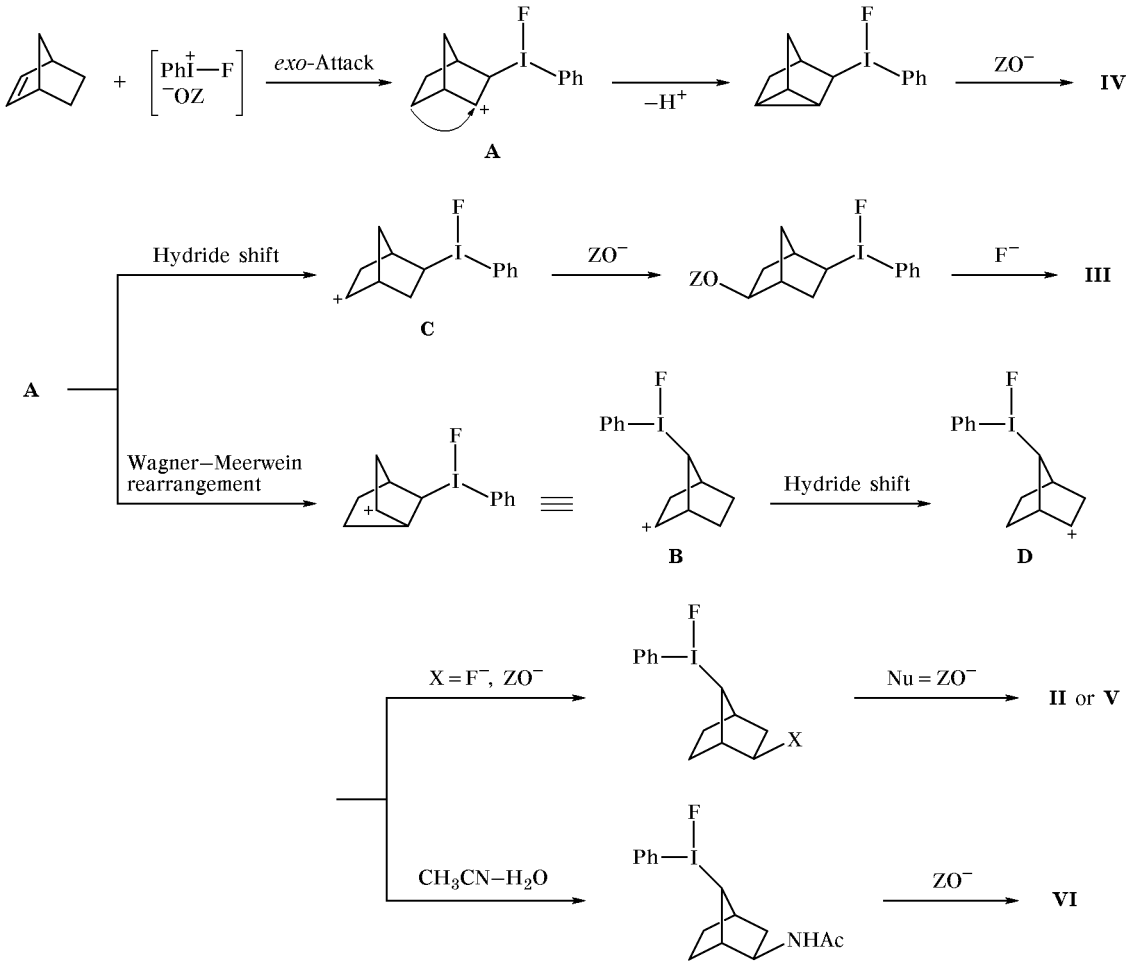


Table 2. ^{1}H NMR spectra (400 MHz) of compounds **II**–**V**, δ, ppm, $J_{H,H}$ and $J_{H,F}$,[a] Hz

Proton	**IIa**	**IIIa**	**IVa**	**IIb**	**IIIb**	**IVb**	**Vb**
1-H	2.358 (8.3), $J_{1,2}$ = 1.5, $J_{1,6}$ = 4.4	2.416, $J_{1,exo\text{-}6}$ = 4.8	1.38	2.423 (8.3), $J_{1,2}$ = 1.5, $J_{1,6}$ = 4.4	2.485, $J_{1,exo\text{-}6}$ = 4.9	1.29	2.803, $J_{1,2}$ = 1.5, $J_{1,6}$ = 4.2
2-H	4.278 (55.3), $J_{2,endo\text{-}3}$ = 6.8, $J_{2,exo\text{-}3}$ = 1.5	3.126, $J_{2,endo\text{-}3}$ = 8.3, $J_{2,exo\text{-}3}$ = 4.3	1.39	4.423 (55.9), $J_{2,endo\text{-}3}$ = 6.2, $J_{2,exo\text{-}3}$ = 1.4	3.210, $J_{2,endo\text{-}3}$ = 8.4, $J_{2,exo\text{-}3}$ = 4.2	1.36	4.786, $J_{2,endo\text{-}3}$ = 7.5, $J_{2,exo\text{-}3}$ = 3.5
3-H	1.536 (*exo*), 1.335 (*endo*), $J_{exo\text{-}3,4}$ = 4.2	1.740 (*endo*), 1.540 (*exo*), $J_{exo\text{-}3,endo\text{-}3}$ = 14.2, $J_{exo\text{-}3,4}$ = 5.1	3.378	1.528 (*exo*), 1.318 (*endo*), $J_{exo\text{-}3,4}$ = 4.1	1.813 (*endo*), 1.605 (*exo*), $J_{exo\text{-}3,endo\text{-}3}$ = 14.2, $J_{exo\text{-}3,4}$ = 5.0	3.45	1.777 (*exo*), 1.543 (*endo*), $J_{exo\text{-}3,4}$ = 4.9
4-H	2.016	2.541 (8.0), $J_{4,5}$ = 1.3	2.239	2.111	2.593 (6.4), $J_{4,5}$ = 1.3	2.256	2.52, $J_{3,4}$ = 5.0
5-H	1.628 (*exo*), 0.640 (*endo*), $J_{endo\text{-}5,endo\text{-}6}$ = 8.4	4.671, (55.5), $J_{endo\text{-}5,endo\text{-}6}$ = 6.3	1.526 (*endo*), 1.587 (*exo*), $J_{endo\text{-}5,exo\text{-}5}$ = 10.9	1.646 (*exo*), 0.621 (*endo*), $J_{endo\text{-}5,endo\text{-}6}$ = 8.4	4.702 (55.6), $J_{endo\text{-}5,endo\text{-}6}$ = 6.4	1.532 (*endo*), 1.599 (*exo*), $J_{endo\text{-}5,exo\text{-}5}$ = 10.9	1.783 (*exo*), 1.135 (*endo*), $J_{endo\text{-}5,endo\text{-}6}$ = 8.8
6-H	1.630 (*exo*), 0.585 (*endo*)	1.758 (*exo*) (39.2), 1.888 (*endo*) (24.8)	1.34	1.701 (*exo*), 0.612 (*endo*)	1.821 (*exo*) (40.0), 1.917 (*endo*) (17.8)	1.376	1.756 (*exo*), 1.121 (*endo*)
7-H	3.427	1.648, 1.715, $J_{7,7}$ = 10.7	1.360, 1.854, $J_{7,7}$ = 10.2	3.502	1.687, 1.689, $J_{7,7}$ = 10.7	1.320, 1.798, $J_{7,7}$ = 10.3	4.767
Me				3.218	3.345	3.322	3.232, 3.254

[a] In parentheses.

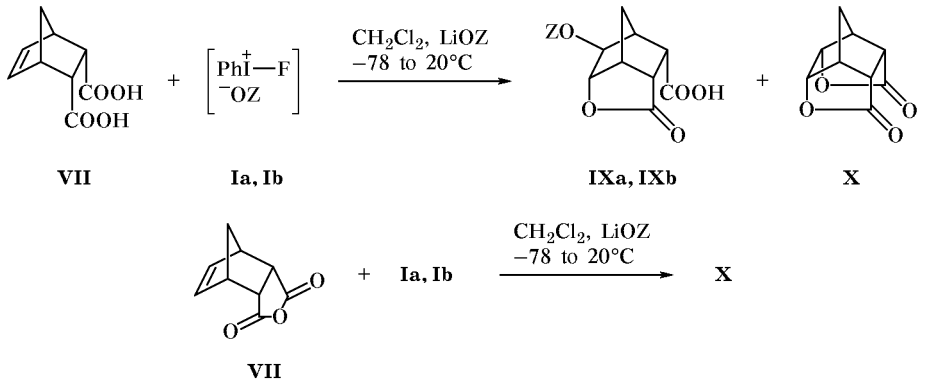
positions [16]: for *exo*-protons, the $^3J_{H,H}$ value is 4–5 Hz, and for *endo*-protons, it is close to zero.

The configuration of substituents in positions *2* and *7* of compound **Vb** was determined on the basis of high-resolution ^{1}H NMR spectra. The *exo* orientation of the substituent on C^2 follows from the mode of splitting of the bridgehead proton signal. The 4-H signal is a triplet with J = 5 Hz due to coupling with the neighboring *exo*-5-H and *exo*-3-H ptorons. By contrast, the 1-H signal appears in the spectrum as a doublet due to coupling with only one *exo*-6-H proton. The *syn* configuration of the 7-substituent is indicated by almost similar chemical shifts of 7-H and 2-H (δ 4.767 and 4.786 ppm, respectively); this suggests the absence of deshielding effect of the *exo*-methylsulfonyloxy group on 7-H.

The proposed structure of **VI** is fully consistent with the data of ^{1}H and ^{13}C NMR and IR spectroscopy and elemental analysis. The configuration of substituents in molecule **VI** was derived from the ^{1}H NMR spectra. The C**H**OZ signal is a doublet at δ 3.59–3.77 ppm; its position indicates that the sulfonyloxy group is attached to C^7. The set of coupling constants of the C**H**N proton (δ 4.45–4.47 ppm) corresponds to its *endo* orientation. The large constant (8.4–8.6 Hz) characterizes *endo*–*cis*-coupling of skeletal protons, and the medium constant (5.4–5.7 Hz) belongs to *trans*-coupling. The ^4J(HCF–HCN) constant equal to 1.5–1.7 Hz indicates *anti*-orientation of 7-H.

The structure of products **II**–**VI** suggests the following mechanism of the reaction of norbornene with [fluoro(sulfonyloxy)iodo]benzenes **Ia** and **Ib** (Scheme 2). *exo*-2,5-Disubstituted products **III** are likely to be formed as a result of *exo*-attack by the reagent at the double bond of the substrate with subsequent 3,5-hydride shift and stabilization of

Scheme 3.



Z = CF_3SO_2 (**a**), CH_3SO_2 (**b**).

intermediate carbocation **C** through addition of fluoride ion (from fluorophenyliodonium cation). 2,7-Disubstituted norbornanes **II** and **V** can be formed via *exo*-attack by iodonium ion on the double bond of the substrate. The primary intermediate, carbocation **A**, undergoes Wagner–Meerwein rearrangement to cation **B** with the iodonium moiety in position *7* of the norbornane skeleton. Presumably, cation **B** is a direct precursor of product **II**.

Next we turned to reactions of compounds **Ia** and **Ib** with norbornene derivatives, in particular with *cis*-5-norbornene-*endo*-2,3-dicarboxylic acid (**VII**) and its anhydride **VIII**. These substrates were selected on the basis of published data [17, 18], according to which introduction of one or more substituents into the norbornene molecule can change the direction of electrophilic reactions. We have found that compounds **Ia** and **Ib** react with acid **VII** in the presence of the corresponding lithium sulfonate to give lactones **IXa** and **IXb** or bis-lactone **X**. The latter is also formed by reaction of **Ia** and **Ib** with anhydride **VIII** (Scheme 3). When the reaction of acid **VII** with compounds **Ia** and **Ib** was performed in acetonitrile or in the absence of lithium sulfonate, bis-lactone **X** was the only product.

The structure of compounds **IXa**, **IXb**, and **X** was proved by spectral methods. The IR spectra of **IX** and **X** contained an absorption band in the region 1780–1790 cm^{-1}, which is typical of carbonyl group in five-membered lactone ring [19]. Insofar as the molecule of bis-lactone **X** is symmetrical, only five carbon signals are observed in the ^{13}C NMR spectrum. The other spectral parameters and physical constants of compound **X** coincide with those reported in [20]. The IR spectra of **IXa** and **IXb** contain absorption bands in the regions 3379–3400 and 1735–1740 cm^{-1},

Table 3. ^{13}C (100 MHz) and ^{19}F (187 MHz) NMR spectra of compounds **II**–**V**, δ_C and δ_F, ppm[a]

Atom	IIa	IIIa	IVa	IIb	IIIb	IVb	Vb
C^1	46.97 (19.0)	41.07	12.74	46.65 (19.1)	41.12	12.56	45.77
C^2	95.26 (18.39)	43.49 (2.3)	14.97	95.34 (18.5)	45.97 (2.3)	14.67	95.34
C^3	40.09 (20.6)	31.55 (10.8)	51.33	40.18 (21.1)	31.60 (11.3)	51.29	41.39
C^4	39.67 (0.3)	42.58 (19.7)	34.73	39.45 (0.3)	42.61 (20.8)	34.11	39.95
C^5	26.24 (1.1)	95.16 (183.16)	33.64	26.33 (1.1)	94.85 (182.4)	33.56	26.28
C^6	20.91 (9.2)	39.67 (22.0)	11.45	20.98 (9.7)	39.62 (21.6)	11.39	20.19
C^7	51.08 (1.3)	32.76 (<1.0)	30.80	50.1 (1.1)	33.03 (<1.0)	30.63	51.99
C(OZ)	118.1 (318)	119.1 (319)	119.42 (319)	38.56	38.13	39.01	38.67
^{19}F	–160.53	–162.71	–161.83	–159.49	–162.73		

[a] In parentheses are given the J_{CF} values, Hz.

which are typical of carboxy group [21]. Lactones **IX** show in the ^{1}H NMR spectra signals from the 2-H and 3-H protons as broadened singlets at δ 4.56–4.64 and 4.27–4.38 ppm, respectively. The vicinal coupling constant ^3J(2-H–3-H) is negligible. This indicates that the ZO group occupies the *exo* position and that molecule **IX** has *trans* configuration. The 7-H signals are doublets, δ 1.25 and 1.3 ppm, with a geminal coupling constant of 10.5 Hz.

Thus our study showed that [fluoro(sulfonyloxy)-iodo]benzene reacts with norbornene and its derivatives to give products of skeletal rearrangements or those formed with participation of both internal nucleophilic centers and external nucleophiles, e.g., solvent molecules. The addition of compounds **Ia** and **Ib** to norbornene was found to involve fluoride and sulfonate ions as nucleophilic species originating from the initial reagent. A new method was developed for introduction of fluoro and sulfonyloxy or two sulfonyloxy groups into the norbornene molecule under mild conditions. The procedure was used to synthesize new representatives of the rare fluoronorbornyl sulfonate and norbornadiyl disulfonate series.

EXPERIMENTAL

The IR spectra were recorded on a UR-10 spectrometer from solutions in carbon tetrachloride. The ^{1}H, ^{13}C, and ^{19}F NMR spectra were obtained on a Varian-400 instrument at 400, 100, and 187 MHz, respectively. The ^{1}H and ^{13}C chemical shifts were measured relative to the solvent signals or tetramethylsilane as internal reference. The ^{19}F chemical shifts were measured relative to trifluoroacetic acid or chlorotrifluoromethane. The purity of compounds was checked by thin-layer chromatography on Silufol plates; TLC was also used to monitor the progress of reactions. Methylene chloride and acetonitrile were purified by the procedure described in [22].

Reactions of norbornene with compounds Ia and Ib in methylene chloride. A solution of 6.5 mmol of norbornene in 10 ml of methylene chloride was added at –78°C under argon to a suspension of 5.6 mmol of compound **Ia** or **Ib** in 20 ml of methylene chloride. The mixture was stirred for 5 h at –40°C and for 2 h at room temperature and was poured into cold water. The mixture was then filtered through a thin layer of silica gel, washed with a 10% solution of sodium thiosulfate, and the aqueous phase was extracted with chloroform. The extract was dried over magnesium sulfate and evaporated under reduced pressure. The oily residue was subjected to column chromatography on silica gel (Silpearl) using 4:1 hexane–ethyl acetate as eluent. Compounds **IIa**–**IVa** (from **Ia**) and **IIb**–**Vb** (from **Ib**) were isolated as unstable oily liquids. Their spectral parameters are given in Tables 2 and 3.

***exo*-2-Fluoro-*syn*-7-trifluoromethylsulfonyloxynorbornane (IIa).** R_f 0.32. Found, %: C 36.67; H 3.94. $C_8H_{10}F_4O_3S$. Calculated, %: C 36.64; H 3.82.

***exo*-5-Fluoro-*exo*-2-trifluoromethylsulfonyloxynorbornane (IIIa).** R_f 0.41. Found, %: C 36.89; H 4.02. $C_8H_{10}F_4O_3S$. Calculated, %: C 36.64; H 3.82.

3-Trifluoromethylsulfonyloxynortricyclene (IVa). R_f 0.53. Found, %: C 39.87; H 3.88. $C_8H_9F_3O_3S$. Calculated, %: C 39.67; H 3.72.

***exo*-2-Fluoro-*syn*-7-methylsulfonyloxynorbornane (IIb).** R_f 0.24. Found, %: C 46.32; H 6.33. $C_8H_{13}FO_3S$. Calculated, %: C 46.15; H 6.25.

***exo*-5-Fluoro-*exo*-2-methylsulfonyloxynorbornane (IIIb).** R_f 0.44. Found, %: C 46.27; H 6.12. $C_8H_{13}FO_3S$. Calculated, %: C 46.15; H 6.25.

3-Methylsulfonyloxynortricyclene (IVb). R_f 0.59. Found, %: C 51.14; H 6.48. $C_8H_{12}O_3S$. Calculated, %: C 51.06; H 6.38.

***exo*-2,*syn*-7-Bis(methylsulfonyloxy)norbornane (Vb).** R_f 0.31. mp 108°C. Found, %: C 38.15; H 5.79. $C_9H_{16}O_6S_2$. Calculated, %: C 38.03; H 5.63.

Reactions of norbornene with compounds Ia and Ib in acetonitrile. Following the above procedure but using acetonitrile instead of methylene chloride we isolated products **IIa** and **VIa** (from **Ia**) and **IIb**, **Vb**, and **VIb** (from **Ib**). The products were separated by column chromatography using 4:1 hexane–ethyl acetate as eluent.

***exo*-2-Acetamido-*syn*-7-trifluoromethylsulfonyloxynorbornane (VIa).** R_f 0.58; yield 18% (yield of **IIa** 25%). IR spectrum (CCl_4), ν, cm^{-1}: 3320 (NH); 1640 (C=O); 1444, 1268, 1222, 1152, 941 (OSO_2CF_3). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.38–1.96 m (6H), 1.98 s (3H, CH_3), 2.42 s (2H, 1-H, 4-H), 3.77 d (1H, 7-H, J = 1.53 Hz), 4.45 d.d.t (1H, J_t = 8.54, J_d = 5.49, J_d = 1.53 Hz), 6.18 d (1H, NH, J = 5.5 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 23.51, 24.32, 27.22, 31.97, 33.21, 43.62, 48.87 (C^7), 52.32 (C^2), 118.45 q (CF_3, $J_{C,F}$ = 318 Hz), 170.42 (C=O). Found, %: C 39.99; H 4.56. $C_{10}H_{14}F_3NO_4S$. Calculated, %: C 39.87; H 4.65.

***exo*-2-Acetamido-*syn*-7-methylsulfonyloxynorbornane (VIb).** R_f 0.47; yield 11% (yields of **IIb** and **Vb** 22% and 15%, respectively). IR spectrum (CCl_4), ν, cm^{-1}: 3320 (NH); 1640 (C=O); 1355, 1186, 947

(S=O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.41–1.89 m (6H), 1.948 s (3H, CH_3), 2.38 s (2H, 1-H, 4-H), 3.23 s (3H, CH_3SO_2O), 3.59 d (1H, 7-H, J = 1.7 Hz), 4.47 d.d.t (1H, J_t = 8.6, J_d = 5.7, J_d = 1.7 Hz), 6.01 d (1H, NH, J = 5.7 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 23.51, 24.32, 27.22, 31.97, 33.21, 38.94, 43.62, 48.87 (C^7), 52.32 (C^2), 170.42 (C=O). Found, %: C 48.46; H 6.75. $C_{10}H_{17}NO_4S$. Calculated, %: C 48.58; H 6.88.

Reactions of *cis*-5-norbornen-*endo*-2,3-dicarboxylic acid with compounds Ia and Ib in methylene chloride. The reactions were carried out according to the preceding procedure with 4.72 mmol of dicarboxylic acid **VII** and 4.72 mmol of compound **Ia** or **Ib** in the presence of 12 mmol of the corresponding lithium sulfonate. Products **IX** and **X** were isolated by chromatography.

Bis-lactone X. R_f 0.43 (ethyl acetate–hexane, 1:1); yield 54%. mp 264–265°C; published data [20]: mp 263°C. IR spectrum (CCl_4), ν, cm^{-1}: 1780 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.77 s (2H, CH_2), 3.02 m (2H, CH), 3.33 m (2H, HCCO), 4.72 m (2H, CHO). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 28.13 s (1C, CH_2), 45.98 s (2C, CH), 46.99 s (2C, H**C**CO), 76.27 s (2C, CHO), 176.79 s (2C, CO).

5-Oxo-*exo*-2-trifluoromethylsulfonyloxy-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxylic acid (IXa). R_f 0.27 (ethyl acetate–hexane, 1:1); yield 13%. mp 222–229°C. IR spectrum (CCl_4), ν, cm^{-1}: 3400, 1735 (COOH); 1780 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.31 d (1H, 8-H, $J_{8,8}$ = 10.5 Hz), 1.56 d (1H, 8-H, $J_{8,8}$ = 10.5 Hz), 2.18 br.s (2H, 1-H, 7-H), 2.35 br.s (1H, 9-H), 2.93 br.s (1H, 6-H), 4.28 br.s (1H, 3-H), 4.64 br.s (1H, 2-H). Found, %: C 36.28; H 2.69. $C_{10}H_9F_3O_7S$. Calculated, %: C 36.36; H 2.73.

***exo*-2-Methylsulfonyloxy-5-oxo-4-oxatricyclo-[4.2.1.0^{3,7}]nonane-*endo*-9-carboxylic acid (IXb).** R_f 0.31 (ethyl acetate–hexane, 1:1); yield 19% (yield of bis-lactone **X** 62%). mp 196–198°C. IR spectrum (CCl_4), ν, cm^{-1}: 3379, 1740 (COOH); 1785 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.25 d (1H, 8-H, $J_{8,8}$ = 10.5 Hz), 1.49 d (1H, 8-H, $J_{8,8}$ = 10.5 Hz), 2.27 br.s (2H, 1-H, 7-H), 2.77 br.s (1H, 9-H), 3.03 br.s (1H, 6-H), 3.43 s (3H, CH_3SO_2O), 4.25 br.s (1H, 3-H), 4.56 br.s (1H, 2-H). Found, %: C 43.57; H 4.51. $C_{10}H_{12}O_7S$. Calculated, %: C 43.48; H 4.35.

Reactions of *cis*-5-norbornen-*endo*-2,3-dicarboxylic acid anhydride with compounds Ia and Ib in methylene chloride. Following the above procedure, from 4.72 mmol of anhydride **VIII** and 4.72 mmol of compound **Ia** or **Ib** in the presence of 12 mmol of the corresponding lithium sulfonate we obtained bis-lactone **X** in 58 and 54% yield, respectively.

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