*Russian Journal of Organic Chemistry, Vol. 39, No. 12, 2003, pp. 1710–1715. Translated from Zhurnal Organicheskoi Khimii, Vol. 39, No. 12, 2003, pp. 1782–1787.*


# Electrophile-Initiated Lactonization of 7-Phenytricyclo[4.1.0.0$^{2,7}$]heptane-1-carboxylic Acid

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Received April 4, 2003

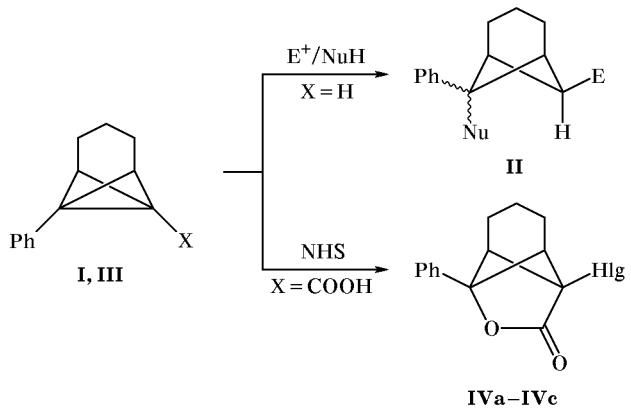
**Abstract**—Treatment of 7-phenyltricyclo[4.1.0.0$^{2,7}$]heptane-1-carboxylic acid with benzenesulfenyl chloride, mercury acetate, and hydrogen chloride leads to cleavage of the central C–C bond in the bicyclobutane fragment and heterocyclization to substituted bicyclo[3.1.1]heptane-6,7-carbolactones.

We previously found [1, 2] that 1-phenyltricyclo[4.1.0.0$^{2,7}$]heptane (**I**) takes up various nucleophiles NuH in the presence of electrophiles. Here, the addition of electrophile $E^+$ occurred predominantly (or exclusively) at the $C^1-C^7$ bond with strict *endo*-stereoselectivity and regioselectivity (at the β-position with respect to the phenyl group), yielding norpinane derivatives **II**. 7-Phenyltricyclo[4.1.0.0$^{2,7}$]heptane-1-carboxylic acid **III** is a functionally substituted derivative of **I**, which has a carboxy group at the $C^1-C^7$ bond. Taking into account the orienting effect of the phenyl group, we anticipated that acid **III** should undergo lactonization by the action of electrophiles. In fact, the first example of such transformation is the reaction of acid **III** with *N*-halosuccinimides (NHS) to afford halogen-substituted lactones **IVa**–**IVc** [3] (Scheme 1).

In the present work we studied transformations of acid **III**, initiated by other electrophilic reagents, such as benzenesulfenyl chloride, mercury acetate, hydrogen chloride, bis(benzonitrile)dichloropalladium(II), and iodotrimethylsilane (generated *in situ*). It should be noted that all these reagents were shown to be suitable for lactonization of unsaturated carboxylic acids [4]. The reaction of benzenesulfenyl chloride with acid **III** in methylene chloride in the presence of triethylamine gave the expected product, phenylsulfanyl-substituted lactone **IVd** in a high yield (Scheme 2). Treatment of acid **III** with mercury acetate in $CH_2Cl_2$ leads to heterocylization with formation of lactone **IVe** (X = OAc). The latter was converted via exchange reaction into the corresponding bromomercurio and chloromercurio derivatives **IVe** (X = Br, Cl). The reaction of **IVe** (X = Br) with bromine gave known bromo lactone **IVa**, and compound **IVe** (X = Cl) was treated with a solution of hydrogen chloride in $CH_2Cl_2$ to obtain lactone **IVf**. The same product (**IVf**) was also synthesized by reduction of **IVa** with tributylstannane.* We made an attempt to obtain lactone **IVf** directly from acid **III**. We succeeded in effecting this transformation by the action of hydrogen chloride in methylene chloride, the yield of **IVf** being nearly quantitative. Lactone **IVf** was also synthesized in a considerably lower yield by prolonged heating of acid **III** in boiling acetonitrile in the presence of $PdCl_2(PhCN)_2$. Taking into account the data of [6], lactonization of acid **III** is initiated by hydrogen chloride which is formed by reaction of palladium chloride with traces of moisture.

Obviously, the formation of lactones **IVd**, **IVe** (X = OAc), and **IVf**, as well as of halogen-substituted lactones **IVa**–**IVc** prepared previously, involves

**Scheme 1.**




**I**, X = H; **III**, X = $CO_2$; **IV**, Hlg = Br (**a**), Cl (**b**), I (**c**).

* For preliminary communication, see [5].

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**Table 1.** $^1$H NMR spectra of lactones **IVa** and **IVd–IVf** in $CDCl_3$, δ, ppm

| Compound no. | 3-H | 2-H, 4-H | 1-H, 5-H | Ph | Other protons |
|---|---|---|---|---|---|
| **IVa** | 1.16–1.37<br>1.51–1.69 | 2.01–2.19 | 3.63 br.s | 7.34–7.43 (2H)<br>7.43–7.55 (3H) | – |
| **IVd** | 1.18–1.37<br>1.66–1.83 | 1.91–2.06<br>2.06–2.24 | 3.56 br.s | 7.25–7.40 (5H)<br>7.40–7.50 (3H)<br>7.60–7.72 (2H) | – |
| **IVe** (X = OAc) | 1.43–1.60<br>1.71–1.92 | 1.95–2.20 | 3.78 br.s | 7.24–7.35 (2H)<br>7.38–7.50 (3H) | 2.08 s ($CH_3$) |
| **IVe** (X = Br) | 1.44–1.62<br>1.64–1.83 | 1.97–2.19 | 3.84 br.s | 7.24–7.41 (2H)<br>7.42–7.56 (3H) | – |
| **IVe**[a] (X = Cl) | 1.13–1.29<br>1.54–1.72 | 1.75–1.91<br>2.05–2.23 | 3.67 br.s | 7.32–7.55 (5H) | – |
| **IVf** | 1.40–1.60 | 1.85–2.00<br>2.00–2.15 | 3.60 br.s | 7.18–7.37 (2H)<br>7.37–7.53 (3H) | 3.01 s (6-H) |

[a] In DMSO-$d_6$.

**Table 2.** $^{13}$C NMR spectra of lactones **IVa** and **IVd–IVf** in $CDCl_3$, $\delta_C$, ppm

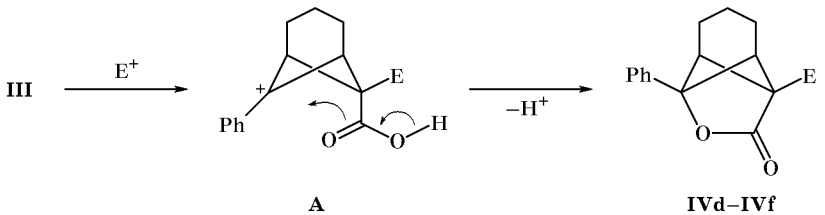
| Compound no. | $C^3$ | $C^2$, $C^4$ | $C^1$, $C^5$ | $C^6$ | $C^7$ | C=O [$CH_3$] | Ph |
|---|---|---|---|---|---|---|---|
| **IVa** | 13.8 | 21.8 | 62.2 | 61.4 | 88.9 | 170.8 | 127.2, 129.0, 130.0, 131.5 |
| **IVd** | 14.3 | 21.1 | 59.6 | 61.2 | 89.0 | 174.6 | 127.0, 128.0, 128.8, 128.85, 129.7, 130.3, 132.0, 133.2 |
| **IVe** (X = OAc) | 15.0 | 24.1 | 64.0 | 67.3 | 94.2 | 177.8, 178.9 [23.1] | 127.7, 129.1, 129.7, 131.5 |
| **IVe** | 15.0 | 23.8 | 64.1 | 74.8 | 93.8 | 178.3 | 127.3, 128.8, 129.5, 130.9 |
| **IVe**[a] (X = Cl) | 14.8 | 23.7 | 63.6 | 74.6 | 93.3 | 179.1 | 127.7, 129.0, 129.4, 132.5 |
| **IVf** | 15.6 | 22.8 | 57.7 | 46.0 | 92.0 | 177.1 | 127.9, 128.8, 129.4, 130.6 |

[a] In DMSO-$d_6$.

intermediate norpinane cation **A** which is generated by strictly *endo*-selective [7] attack by electrophile $E^+$ on the $C^1$ atom of acid **III**. The reaction is completed by intramolecular nucleophilic attack by the carboxy group on the cationic center (Scheme 2).

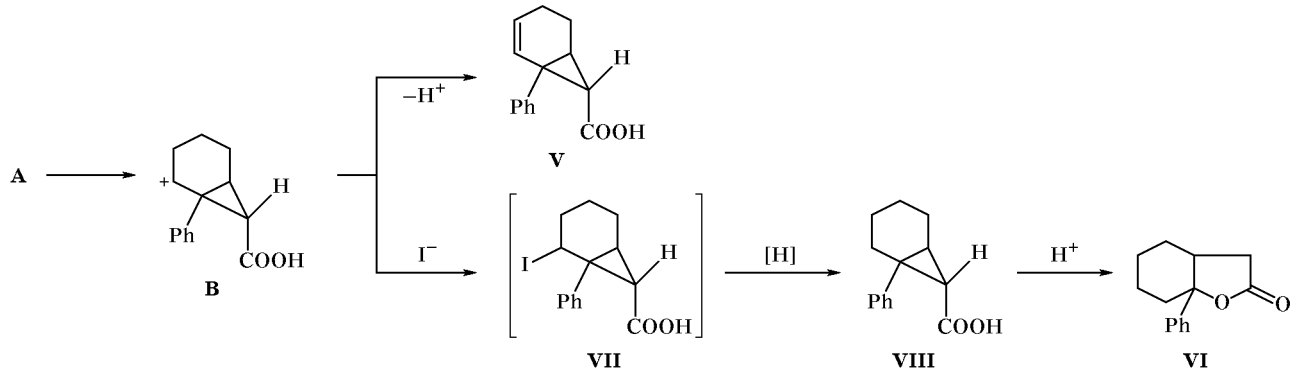
The structure of newly synthesized lactones **IVd**, **IVe**, **IVf** was convincingly proved by the $^1$H and $^{13}$C NMR spectra (Tables 1 and 2); for comparison, the data for previously described bromo lactone **IVa** are also given.

**Scheme 2.**




E = PhS (**d**), HgX (**e**), H (**f**).

**Scheme 3.**




Unlike hydrogen chloride, the other reagents giving rise to electrophilic hydrogen species induced more profound transformations of the tricycloheptane skeleton of initial acid **III**. For example, in the presence of a catalytic amount of sulfuric acid in dioxane compound **III** underwent isomerization into acid **V** having a norcarane skeleton. When gaseous hydrogen iodide was passed through a solution of acid **III** in methylene chloride, two products were formed at a ratio of 11 : 2. Among these, only the minor product was the expected lactone **IVf**. The major product, lactone **VI**, was also synthesized by reaction of **III** with chlorotrimethylsilane in the presence of NaI and water. Presumably, the system $Me_3SiCl/NaI/H_2O$ generates hydrogen iodide which initiates the above transformation of acid **III**. Obviously, the reaction path leading to acid **V** includes intermediate formation of norcaranyl cation **B**. The latter is also precursor of **VI**. As shown in Scheme 3, cation **B** reacts with HI to give iodo-substituted acid **VII** which is reduced to **VIII**, and lactonization of **VIII** yields final product **VI**. Cation **B** is formed as a result of the known cyclobutyl–cyclopropylcarbinyl isomerization of cation **A** (E = H). Taking into account the steric structure of **A** and stereospecificity of the isomerization [8], the carboxy group in cation **B** and acids **V**, **VII**, and **VIII** should be oriented *exo*. The last stage in the formation of lactone **VI** (Scheme 3) was proved by direct lactonization of acid **VIII** which was obtained by independent synthesis via carbene reaction of 1-phenylcyclohexene with ethyl diazoacetate. Acids **V** and **VIII** were treated with diazomethane to obtain the corresponding methyl ethers **IX** and **X**. The structure of **V** and **IX** was proved by the $^1H$ and $^{13}C$ NMR spectra. Specifically, the multiplicity of signals belonging to the olefinic protons is typical of 2-norcarene derivatives [9]. The structure of **VIII** and **X** unambiguously follows from the procedure of their synthesis and is confirmed by spectral data. The vicinal coupling constant of the 7-H proton, $J$ = 4.9–5.7 Hz, indicates *exo*-configuration of molecules **V**, **VIII**, **IX**, and **X** [9].

The $^1H$ and $^{13}C$ NMR spectra of lactone **VI** are consistent with the proposed structure: a characteristic chemical shift of the $C^1$ atom ($\delta_C$ 88.2 ppm) may be noted as an example. *cis*-Junction of the bicyclic skeleton of **VI** was assumed on the basis of the result of lactonization of the methyl analog of **VIII**, reported previously [4].

## EXPERIMENTAL

The $^1H$ and $^{13}C$ NMR spectra were recorded on a Bruker DPX-300 spectrometer operating at 300.13 and 75.47 MHz, respectively; $CDCl_3$ or DMSO-$d_6$ was used as solvent. Elemental analysis was performed on an HP 185B CHN-analyzer. Silufol UV-254 plates were used for analytical TLC. The products were separated and purified by column chromatography on silica gel L 40/100 μm (Chemapol). Bis(benzonitrile)dichloropalladium(II) [11] and benzenesulfenyl chloride [12] were prepared by known methods. 7-Phenyltricyclo[4.1.0.0$^{2,7}$]heptane-1-carboxylic acid (**III**), mp 168°C, was synthesized by the procedure reported in [3].

**6-Bromo-7-phenylbicyclo[3.1.1]heptane-6,7-carbolactone (IVa).** A solution of 0.49 g (1 mmol) of bromomercurio lactone **IVe** (X = Br) in 15 ml of methylene chloride was added to a solution of 0.32 g (2 mmol) of bromine in 20 ml of methylene chloride. The mixture was stirred for 10 h and treated with water. The organic phase was separated, washed with a solution of sodium thiosulfate and with water, and dried over anhydrous sodium sulfate. The solvent was removed to leave 0.28 g (96%) of lactone **IVa**,

mp 116°C; published data [3]: mp 116°C; $R_f$ 0.55 (hexane–ether, 1 : 1).

**7-Phenyl-6-phenylsulfanylbicyclo[3.1.1]heptane-6,7-carbolactone (IVd).** A flask was charged under argon with 1.07 g (5 mmol) of acid **III**, 0.57 g (5.6 mmol) of triethylamine, and 10 ml of methylene chloride. The flask was capped with a septum, the mixture was cooled to –25°C, and a solution of 1.01 g (7 mmol) of benzenesulfenyl chloride in 10 ml of methylene chloride was added using a syringe over a period of 10 min under stirring. The mixture was stirred for 1 h at –25°C and for 3 h at room temperature. The solvent was removed under reduced pressure, 20 ml of benzene was added to the residue, and the solution was filtered. Purification by column chromatography gave 1.41 g (86%) of lactone **IVd**. mp 97°C (hexane). $R_f$ 0.52 (hexane–ether, 2 : 1). Found, %: C 74.42, 74.54; H 5.60, 5.67. $C_{20}H_{18}O_2S$. Calculated, %: C 74.50; H 5.63.

**Reaction of acid III with mercury acetate.** Mercury acetate, 5.58 g (18 mmol), was added to a solution of 3.21 g (15 mmol) of acid **III** in 75 ml of methylene chloride, and the mixture was stirred for 24 h. Water, 80 ml, was added, and the organic phase was separated, washed with water (3 × 30 ml), and dried over anhydrous sodium sulfate. Yield of 6-acetoxymercurio-7-phenylbicyclo[3.1.1]heptane-6,7-carbolactone (**IVe**, X = OAc) 5.98 g (85%). mp 117°C (from methanol–ether). Found, %: C 40.86, 40.88; H 3.51, 3.54. $C_{16}H_{16}HgO_4$. Calculated, %: C 40.64; H 3.41. A solution of 0.24 g (2 mmol) of potassium bromide in 7 ml of water was added to a solution of 0.95 g (2 mmol) of compound **IVe** (X = OAc) in 5 ml of methanol. The resulting suspension was stirred for 4 h at room temperature and diluted with 5 ml of water, and 30 ml of chloroform was added. The organic phase was separated, and the aqueous phase was extracted with chloroform (3 × 10 ml). The extracts were combined with the organic phase, washed with water, and dried over anhydrous sodium sulfate. Removal of the solvent left 0.93 g (94%) of 6-bromomercurio-7-phenylbicyclo-[3.1.1]heptane-6,7-carbolactone (**IVe**, X = Br). mp 129°C (from chloroform). Found, %: C 34.16, 34.18; H 2.69, 2.71. $C_{14}H_{13}BrHgO_2$. Calculated, %: C 34.06; H 2.65.

Likewise, from 0.95 g (2 mmol) of lactone **IVe** (X = OAc) in 5 ml of methanol and 7 ml of a solution of sodium chloride (0.12 g, 2 mmol) in water we obtained 0.83 g (83%) of 6-chloromercurio-7-phenylbicyclo[3.1.1]heptane-6,7-carbolactone (**IVe**, X = Cl). mp 125°C (from chloroform). Found, %: C 42.07, 42.12; H 3.58, 3.63. $C_{14}H_{13}ClHgO_2$. Calculated, %: C 42.06; H 3.53.

**7-Phenylbicyclo[3.1.1]heptane-6,7-carbolactone (IVf).** *a*. A solution of HCl in methylene chloride, 0.5 ml (obtained by shaking a mixture of methylene chloride with hydrochloric acid and subsequent drying over $CaCl_2$), was added to a solution of 2.14 g (10 mmol) of acid **III** in 20 ml of methylene chloride. The mixture was stirred for 3 h at room temperature, the solvent was removed under reduced pressure, the residue was diluted with 30 ml of diethyl ether, and the ether solution was washed with a 5% aqueous solution of sodium hydrogen carbonate and with water, dried over anhydrous sodium sulfate, and evaporated to obtain 2.03 g (95%) of lactone **IVf**.

*b*. Dry hydrogen chloride was passed over a period of 2 h through a suspension of 0.45 g (1 mmol) of lactone **IVe** (X = Cl) in 30 ml of carbon tetrachloride. An aqueous solution of sodium acetate was then added, and the organic phase was separated, washed with water, and dried over anhydrous sodium sulfate. Removal of the solvent left 0.12 g (55%) of **IVf**.

*c*. Bis(benzonitrile)dichloropalladium(II), 19.2 mg, (1 mmol), was added to a solution of 1.07 g (5 mmol) of acid **III** in 20 ml of acetonitrile, and the mixture was stirred for 50 h under reflux in an argon atmosphere. The mixture was then filtered through a 2-cm layer of silica gel, the solvent was removed from the filtrate under reduced pressure, and the residue was purified by flash chromatography on silica gel using hexane–methylene chloride (2 : 1) as eluent. Yield of **IVf** 0.37 g (35%).

*d*. Azobis(isobutyronitrile), 25 mg, was added to 440 mg (1.5 mmol) of lactone **IVa** and 495 mg (1.7 mmol) of tributylstannane in 30 ml of anhydrous benzene, and the mixture was heated for 7 h under reflux, a 10–15-mg portion of AIBN being added every ~1.5 h. The product was purified by column chromatography. Yield 151 mg (47%). mp 76°C (from hexane). $R_f$ 0.48 (hexane–ether, 1 : 1). Found, %: C 78.46, 78.45; H 6.58, 6.56. $C_{14}H_{14}O_2$. Calculated, %: C 78.48; H 6.59.

**1-Phenylbicyclo[4.1.0]hept-2-ene-*exo*-7-carboxylic acid (V).** Concentrated sulfuric acid, 50 mg, was added to a solution of 0.214 g (1 mmol) of acid **III** in 7 ml of dioxane, and the mixture was stirred for 50 h at room temperature. The solution was neutralized with 2 g of powdered barium carbonate, the solvent was evaporated, and the residue was recrystallized to obtain 0.175 g (85%) of acid **V**. mp 105°C (from hexane). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 1.79–1.96 (2H, 5-H), 2.05–2.20

(2H, 4-H), 2.23 d (1H, 7-H, $J$ = 5.7 Hz), 2.52 br.s (1H, 6-H), 5.60 d.d (1H, 3-H, $J$ = 9.9, 7.1 Hz), 5.99 d (1H, 2-H, $J$ = 9.9 Hz), 7.19–7.38 (5H, Ph), 10.78 (1H, $CO_2H$). $^{13}C$ NMR spectrum (DMSO-$d_6$), $\delta_C$, ppm: 17.2, 19.8, 27.3, 32.0, 36.0, 124.4, 126.7, 128.2 (2C), 128.7 (2C), 131.7, 140.7, 176.2 (C=O). Found, %: C 78.42, 78.44; H 6.60, 6.61. $C_{14}H_{14}O_2$. Calculated, %: C 78.48; H 6.59.

**Methyl 1-phenylbicyclo[4.1.0]hept-2-ene-*exo*-7-carboxylate (IX)** was synthesized by treatment of acid **V** with a solution of diazomethane in diethyl ether. $^1H$ NMR spectrum ($CDCl_3$), δ, ppm: 1.81–2.0 (2H), 2.08–2.25 (2H), 2.32 d (1H, 7-H, $J$ = 5.6 Hz), 2.59–2.64 (1H), 3.44 s (3H, $OCH_3$), 5.61 d.d.d (1H, 3-H, $J$ = 9.7, 7.0, 1.4 Hz), 6.03 d (1H, 2-H, $J$ = 9.7 Hz), 7.22–7.38 (5H, Ph). $^{13}C$ NMR spectrum ($CDCl_3$), $\delta_C$, ppm: 17.2, 19.9, 26.5, 32.2, 34.8, 51.2 ($OCH_3$), 124.1, 126.2, 128.2 (2C), 128.7 (2C), 131.9, 141.1, 170.6 (C=O).

**6-Phenyl-7-oxabicyclo[4.3.0]nonan-8-one (VI).** *a*. Freshly distilled chlorotrimethylsilane (bp 57°C), 0.12 g (1.1 mmol), sodium iodide, 0.15 g (1 mmol), and water, 18 mg (1 mmol), were added to a solution of 0.21 g (1 mmol) of acid **III** in 25 ml of benzene. The mixture was stirred for 10 h, washed with a solution of sodium thiosulfate and with water, and dried over anhydrous magnesium sulfate. After removal of the solvent, the residue was purified by flash chromatography on silica gel using hexane–methylene chloride (6:1) as eluent. Yield 0.17 g (78%). Oily substance, $R_f$ 0.64 (hexane–methylene chloride, 6:1). $^1H$ NMR spectrum ($CDCl_3$), δ, ppm: 1.26–1.54 (2H), 1.55–1.71 (2H), 1.72–1.85 (2H), 1.92–2.25 (1H), 2.13 d (1H, $J$ = 16.4 Hz) and 2.42 d.d (1H, $J$ = 16.4, 6.7 Hz) (7-$CH_2$), 2.17–2.27 (1H), 2.63–2.74 (1H, 6-H), 7.22–7.48 (5H, Ph). $^{13}C$ NMR spectrum ($CDCl_3$), $\delta_C$, ppm: 21.6, 23.6, 29.2, 37.4, 37.6, 40.4, 88.2 ($C^1$), 125.1 (2C), 128.0, 128.9 (2C), 144.7, 177.6 (C=O). Found, %: C 77.66, 77.69; H 7.49, 7.56. $C_{14}H_{16}O_2$. Calculated, %: C 77.75; H 7.46.

*b*. Chlorotrimethylsilane, 0.12 g (1.1 mmol), sodium iodide, 0.15 g (1 mmol), and water, 18 mg (1 mmol), were added to a solution of 0.22 g (1 mmol) of acid **VIII** in 25 ml of benzene. The subsequent procedure was the same as described above in *a*. Yield of **VI** 0.18 g (81%).

**Reaction of acid III with hydrogen iodide.** Dry hydrogen iodide was passed over a period of 1 h through a solution of 0.21 g (1 mmol) of acid **III** in 30 ml of methylene chloride. The solution was washed in succession with a solution of sodium thiosulfate, a solution of sodium hydrogen carbonate, and water and dried over anhydrous magnesium sulfate. After removal of the solvent, the residue (0.25 g) was a mixture of lactones **IVf** and **VI** at a ratio of 2:11 (according to the $^1H$ NMR data).

**1-Phenylbicyclo[4.1.0]heptane-*exo*-7-carboxylic acid (VIII).** A solution of 8 g (0.07 mol) of ethyl diazoacetate in 5 ml of octane was added over a period of 1 h with stirring under nitrogen to a mixture of 15.8 g (0.1 mol) of 1-phenylcyclohexene and 50 mg of anhydrous copper sulfate in 10 ml of octane, heated to 125°C. When the addition was complete, the mixture was heated for an additional 10 min (until nitrogen no longer evolved). The mixture was cooled, 8 g of potassium hydroxide in 150 ml of ethanol was added, and the mixture was heated for 3 h under reflux with stirring, cooled, and diluted with 300 ml of water. Volatile organic substances were removed by steam distillation, and the still residue was concentrated to a volume of ~100 ml and acidified to pH 1 by adding dilute sulfuric acid on cooling. The precipitate was filtered off and repeatedly recrystallized from aqueous methanol. Yield of acid **VIII** 2.1 g (14%). mp 181°C. $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 1.15–1.31 (2H), 1.32–1.48 (2H), 1.60–1.76 (2H), 1.83 d (1H, 7-H, $J$ = 5.1 Hz), 1.95–2.03 (3H), 2.05–2.22 (2H), 7.10–7.32 (5H, Ph), 11.71 br.s (1H, $CO_2H$). $^{13}C$ NMR spectrum (DMSO-$d_6$), $\delta_C$, ppm: 21.55, 21.65, 23.0, 24.6, 32.1, 34.0, 36.4, 126.8, 128.9 (2C), 129.1 (2C), 145.4, 173. Found, %: C 77.57, 77.82; H 7.54, 7.37. $C_{14}H_{16}O_2$. Calculated, %: C 77.75; H 7.46.

**Methyl 1-phenylbicyclo[4.1.0]heptane-*exo*-7-carboxylate (X)** was synthesized by treatment of acid **VIII** with a solution of diazomethane in ether. $^1H$ NMR spectrum ($CDCl_3$), δ, ppm: 1.25–1.55 (4H), 1.74–1.91 (2H), 1.93 d (1H, 7-H, $J$ = 4.9 Hz), 2.12–2.30 (3H), 3.43 s (3H, $OCH_3$), 7.17–7.34 (5H, Ph). $^{13}C$ NMR spectrum ($CDCl_3$), $\delta_C$, ppm: 21.0, 21.05, 22.5, 24.7, 31.7, 33.0, 36.6, 51.1 ($OCH_3$), 126.1, 128.0 (2C), 128.1 (2C), 144.2, 172.2 (C=O).

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