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Acid-Catalyzed Transformations of 2-(Phenylethynyl)isoborneol

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Abstract—2-(Phenylethynyl)isoborneol was synthesized by treatment of camphor with lithium phenylacetylide. Skeletal rearrangements of the title compound under the Ritter reaction conditions afforded a mixture of *N*-(4-phenylethynyl- and 4-benzoylmethyl-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)acetamides at a ratio of 8:3. The reaction of 2-(phenylethynyl)isoborneol with formic acid involved mainly Meyer–Schuster rearrangement instead of the expected Rupe rearrangement, and the major product was 2-(1,7,7-trimethylbicyclo[2.2.1]-hept-2-ylidene)-1-phenylethanone. The minor product (~6%) was 1-(2-hydroxy-1,7,7-trimethylbicyclo[2.2.1]-hept-2-yl)-2-phenylethanone. The Ritter reaction of 2-(1,7,7-trimethylbicyclo[2.2.1]hept-2-ylidene)-1-phenylethanone selectively yielded *N*-(4-benzoylmethyl-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)acetamide.

We previously showed that acetylenic alcohols of the bicyclo[2.2.1]heptane series undergo various transformations under the Ritter reaction conditions. These transformations include both skeletal rearrangements and hydration of the triple bond, leading to ethynyl- or acetyl-substituted amides. The relative contributions of these processes are determined by the steric structure of the initial alcohol irrespective of the conditions [1].

In the present work we studied acid-catalyzed transformations of 2-(phenylethynyl)isoborneol (1,7,7-trimethyl-*endo*-2-phenylethynylbicyclo[2.2.1]heptan-2-ol, **I**) which was prepared by treatment of camphor with lithium phenylacetylide. Compound **I** reacted with acetonitrile in the presence of sulfuric acid (Ritter reaction) to give 4-substituted bornane derivatives, *N*-(1,7,7-trimethyl-4-phenylethynylbicyclo[2.2.1]hept-2-yl)acetamide (**II**) and *N*-(4-benzoylmethyl-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)acetamide (**III**) at a ratio of 8:3. Thus the Ritter reaction of acetylenic alcohol **I**, unlike its closest structural analog, 2-ethynylisoborneol (**IV**) [1], involves an appreciable contribution of hydration of the triple bond. Here, water addition at the triple bond occurs exclusively at the carbon atom in the α-position with respect to the benzene ring. The observed skeletal rearrangement is analogous to that reported for 2-ethynylisoborneol.

Amides **II** and **III** were separated by crystallization from hexane. Their structure was determined on the basis of the ^{1}H NMR, IR, and mass spectra. The IR spectrum of **II** contains an absorption band at 1645 cm^{-1}, which is typical of carbonyl stretching vibrations in amides; bands at 3290 and 1550 cm^{-1} belong to stretching vibrations of the N–H group; a weak absorption band at 2220 cm^{-1} originates from vibrations of the triple carbon–carbon bond; also, absorption bands at 3080, 3030, 1600, 755, and 690 cm^{-1} were present due to vibrations of the benzene ring. Compound **II** showed in the mass spectrum the molecular ion peak $[M]^+$ with *m*/*z* 295 and a relative intensity of 47%. In the ^{1}H NMR spectrum of **II** we observed a signal at δ 3.98 ppm due to the proton neighboring to the amide group. This signal appeared as a doublet of triplets, indicating the presence of a methylene group in the α-position with respect to the amide group. The coupling constant equal to 8.8 Hz corresponds to interaction of *endo*-2-H with *endo*-3-H (δ 2.27 ppm, d.d, ^2J = 13.2, ^3J = 8.8 Hz) and NH (δ 5.40 ppm, br.d), and the constant J = 4.8 Hz characterizes coupling with *exo*-3-H (δ 1.86 ppm, d.d.d, ^2J = 13.2, ^3J = 4.8, $^WJ_{3,exo\text{-}5}$ = 2.4 Hz). The multiplicity of the latter signal (specifically, the lack of vicinal coupling with 4-H) indicates that the 3-H proton is located in a position neighboring to substituted bridgehead carbon atom, in keeping with the assumed structure.

The IR spectrum of amino ketone **III** contained absorption bands typical of vibrations of the amide group (3275, 1640, and 1545 cm^{-1}) and benzene ring (3065, 3025, 1595, 750, and 690 cm^{-1}), as well as a band at 1680 cm^{-1}, belonging to carbonyl group

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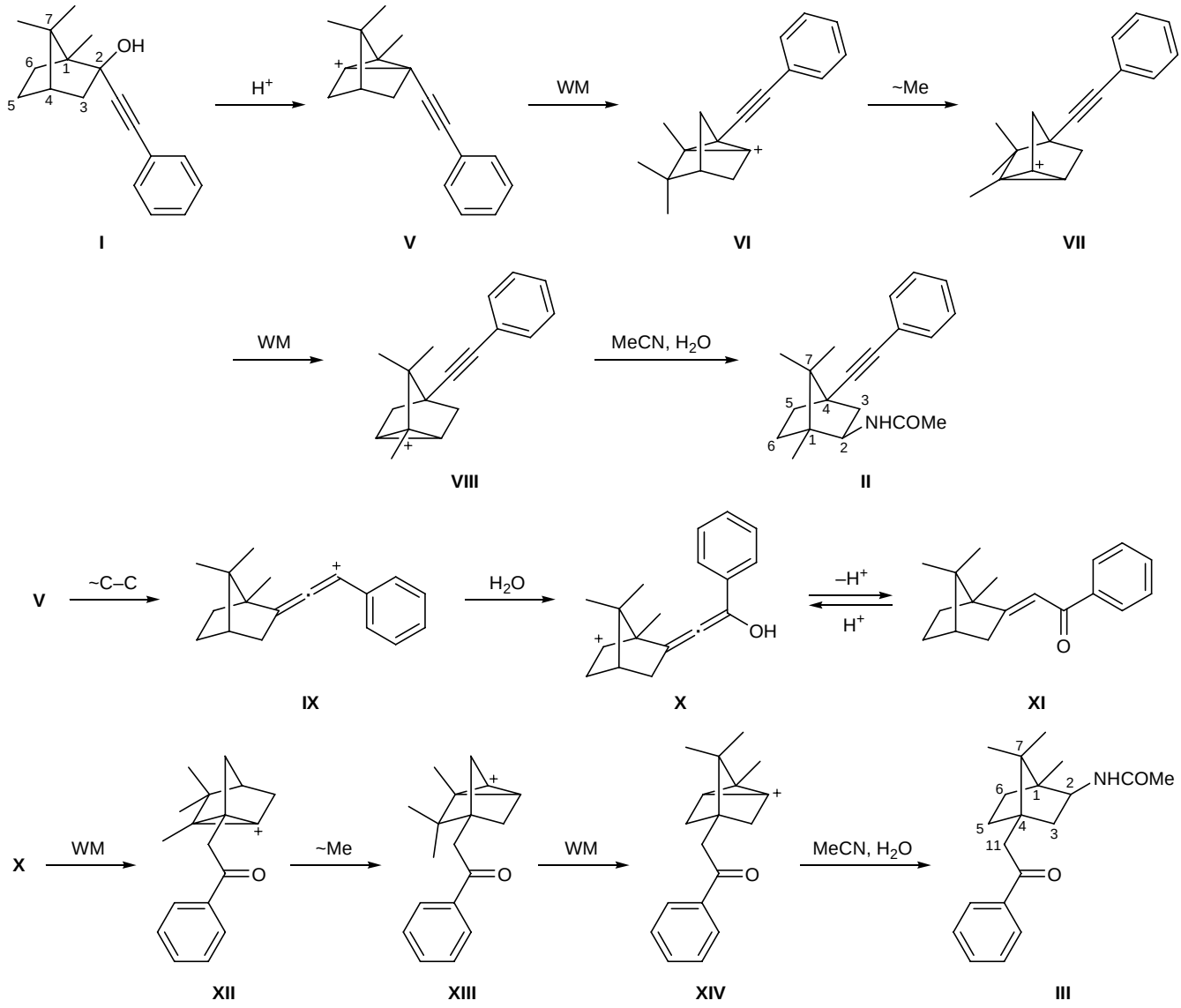
conjugated with aromatic ring. The molecular ion peak $[M]^+$ (*m*/*z* 313) in the mass spectrum of **III** had a relative intensity of 2%.

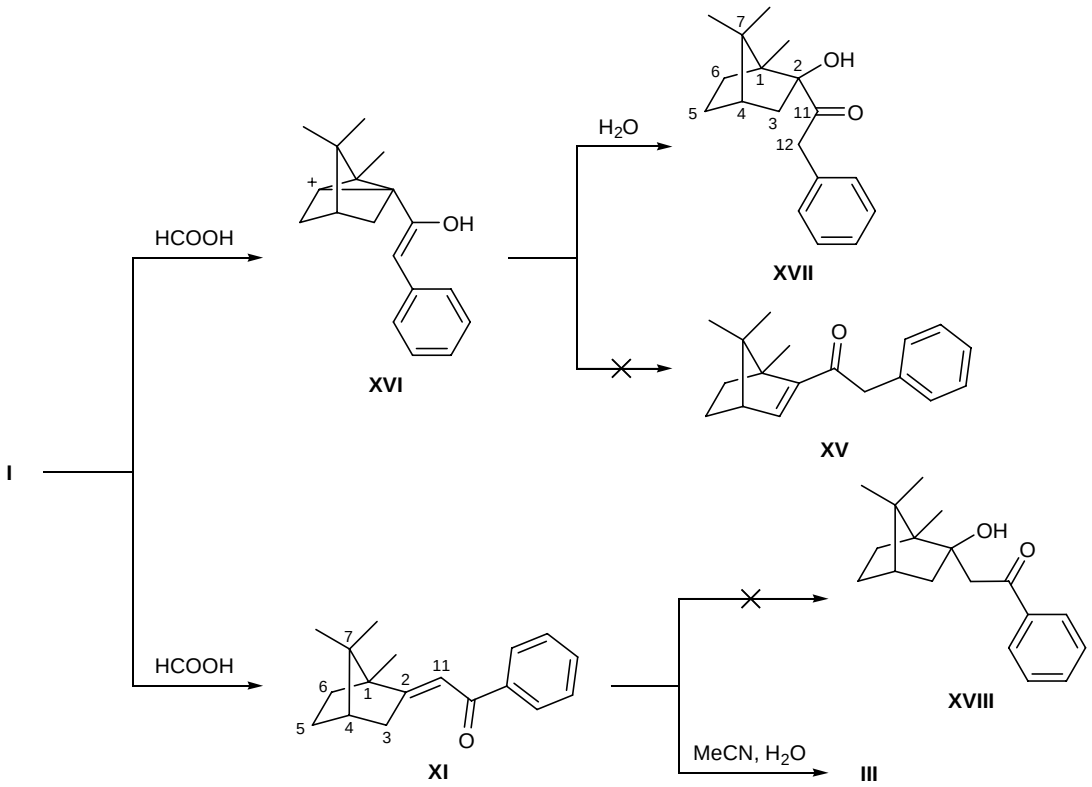
Compound **III** displayed in the ^{1}H NMR spectrum signals from the acetylamino group at δ 1.94 (s, 3H, CH_3) and 5.30 ppm (br.d, 1H, ^3J = 9.2 Hz) and a signal at δ 3.92 ppm from the proton neighboring to the amino group. The latter signal was a doublet of triplets (J = 9.2, 9.2, 4.6 Hz), and its multiplicity was similar to that of the *endo*-2-H signal in the spectrum of **II**. As with compound **II** (see above), this suggests the presence of a CH_2 group in the α-position with respect to the amide group. The *endo*-3-H signal (δ 2.17 ppm) is a doublet of doublets, ^2J = 13.2, $^3J_{endo,endo}$ = 9.2 Hz), and the *exo*-3-H signal (δ 1.89 ppm) is a double doublet of doublets (^2J = 13.2, $^3J_{endo,exo}$ = 4.6, ^WJ = 2.4 Hz), i.e., a substituent is present on C^4 of the bornane skeleton. Protons of the methylene group neighboring to the benzoyl fragment give a singlet at δ 2.94 ppm. Aromatic protons resonated at δ 7.51 (3H) and 7.92 ppm (2H) ppm as two multiplets. The downfield position of the latter signal indicates that both protons in the *ortho* positions of the benzene ring suffer from deshielding effect produced by the carbonyl group.

Scheme 1 illustrates transformations of 2-phenylethynylisoborneol (**I**) in the Ritter reaction. Obviously, the major product, *N*-(4-phenylethynyl-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)acetamide (**II**), is formed as a result of the following reaction sequence. Elimina-

Scheme 1.



Scheme 2.



tion of the hydroxy group from the initial compound gives cation **V** which is transformed into isocamphane cation **VI**. Methyl group migration to the cationic center in **VI** and the subsequent Wagner–Meerwein rearrangement give 4-phenylethynylbornane cation **VIII** which takes up nucleophile molecule (acetonitrile), and hydration of iminium cation thus formed yields amide **II**. Analogous reorganization of the carbon skeleton was described by us previously for other 2-substituted isoborneols uner the Ritter reaction conditions [1–3].

Another path of transformations of initially formed cation **V** is Meyer–Schuster rearrangement [4]: C–C bond migration to the cationic center gives phenylallene cation **IX** whose hydration leads to cation **X**. The latter undergoes a series of skeletal rearrangements (like **V** → **VI** → **VII** → **VIII** → **II**) through intermediates **XII**–**XIV**, resulting in formation of *N*-(4-benzoylmethyl-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)acetamide (**III**). The assumption that compound **III** is formed as a result of Meyer–Schuster rearrangement rather than acid-catalyzed hydration of the triple bond is supported by the following. First, the fraction of product **III** does not increase with time. Second, the reaction mixture contains a small amount (2–3%) of ketone **XI** (see below) formed by deprotonation of cation **X**. Third, an analogous mixture of compounds **II** and **III** was obtained when the reaction was carried out in anhydrous acetonitrile in the presence of boron trifluoride–ether complex, i.e., under the conditions excluding hydration of the triple bond. Obviously, the ratio of amides **II** and **III** in the reaction mixture is determined by the rates of Wagner–Meerwein and Meyer–Schuster rearrangements of initially formed cation **V**; therefore, this ratio almost does not change upon variation of the reaction conditions.

2-Phenylethynylisoborneol (**I**) underwent rearrangement into α,β-unsaturated ketone **XI** on heating with formic acid. These conditions usually favor Rupe rearrangement [5] which would lead to enone **XV** with endocyclic double bond via migration of the hydroxy group to the α-carbon atom at the triple bond. However, the predominant transformation pathway under catalysis by a weak acid was Meyer–Schuster rearrangement (see above), resulting in formation of ketone **XI**. Obviously, this pathway is more favorable from the viewpoint of thermodynamics: the formation of cross-conjugated 10π-electron system in molecule **XI** is more advantageous than the formation of molecule **XV** in which the enone fragment is not conjugated with the benzene ring.

The structure of compound **XI** was confirmed by the ^{1}H NMR, IR, and mass spectra. Its IR spectrum contained absorption bands typical of α,β-unsaturated ketones: 1665 (C=O) and 1610 cm^{-1} (C=C). Similar intensities of these bands indicate *s-cis* configuration of the enone fragment, i.e., compound **XI** is formed as a single *trans-s-cis* isomer. The assumed structure of **XI** is also supported by the data of ^{1}H NMR spectroscopy. The signal from proton at the double bond was a triplet at δ 6.78 ppm with a coupling constant of 2.4 Hz corresponding to *trans*-allyl interaction with the methylene protons on C^3. Therefore, the senior substituents (C^1 and benzoyl group) at the double C=C bond are arranged *trans*. Alternative configuration of the double bond is hardly probable for steric reasons: it would involve overlap of the effective volumes of the methyl group on C^1 and the carbonyl group (*cis-s-cis*) or benzene ring (*cis-s-trans*) in the enone fragment. The ^{1}H NMR spectrum of ketone **XI** also contained signals from methylene protons on C^3 at δ 3.00 (d.d.t, *exo*-3-H, ^2J = 19.8, $^3J_{3,4}$ = 4.0, $^WJ_{3,exo\text{-}5}$ = ^4J = 2.4 Hz) and 2.55 ppm (d.d, *endo*-3-H, ^2J = 19.8, ^4J = 2.4 Hz). An increased value of the geminal coupling constant for these protons is typical of a methylene group in the α-position with respect to a conjugated double bond system [6]. In the mass spectrum of compound **XI**, the molecular ion peak $[M]^+$ (*m*/*z* 254) had a relative intensity of 40%.

The minor component (~6%) in the reaction mixture obtained by rearrangement 2-phenylethynylisoborneol (**I**) was 1-(2-hydroxy-1,7,7-trimethylbicyclo-[2.2.1]hept-2-yl)-2-phenylethanone (**XVII**). Presumably, a small part of the initial acetylenic alcohol undergoes rearrangement according to the first step of the Rupe reaction to give nonclassical cation **XVI** (Scheme 2). However, instead of normal stabilization via elimination of proton, cation **XVI** takes up water molecule. A probable reason is considerable increase in skeletal strains accompanying formation of compounds with an endocyclic double bond. Water molecule adds to cation **XVI** at the geminal position with respect to the bulky phenylacetyl group rather than at the spatially more accessible C^6 atom. This may be due to formation of kinetically bound ion pairs rather than free carbocations under catalysis by a weak acid. An evidence in support of the above assumption is that neither initial compound **I** nor cationic intermediates derived therefrom undergo Wagner–Meerwein rearrangement which does occur under the Ritter reaction conditions (catalysis by sulfuric acid).

The structure of hydroxy ketone **XVII** was confirmed by the ^{1}H NMR, IR, and mass spectra. Compound **XVII** showed in the mass spectrum the molecular ion peak $[M]^+$ with *m*/*z* 272 and a relative intensity of 3%. Its molecular weight corresponds to addition of one water molecule to initial acetylenic alcohol **I**. The carbonyl absorption band appears in the IR spectrum of **XVII** at a frequency of 1720 cm^{-1}, which indicates the absence of conjugation between the carbonyl group and the benzene ring. Therefore, compound **XVII** has the structure of α-hydroxy ketone rather than ketone **XVIII** which could be formed by hydration of enone **XI**. Structure **XVII** is also supported by the ^{1}H NMR data. The chemical shift of the methylene protons on C^{12} is 3.88 ppm, in keeping with the position of that group in the α-position with respect to the carbonyl group and benzene ring. The corresponding signal in the spectrum of **XVIII** should appear in a stronger field by at least 0.5 ppm [7].

We also examined transformations of enone **XI** under the Ritter reaction conditions. The only product was *N*-(4-benzoylmethyl-1,7,7-trimethylbicyclo-[2.2.1]-hept-2-yl)acetamide (**III**). Obviously, the reaction involves processes analogous to those described above: protonation of the carbonyl group in enone **XI** gives carbocation **X** which rearranges according to Scheme 1 to afford cation **XIV**. Nucleophilic stabilization of the latter yields amino ketone **III**.

EXPERIMENTAL

The IR spectra were recorded on a Nicolet Protégé-460 Fourier spectrometer. The ^{1}H NMR spectra were measured on a Tesla BS-567 instrument at 100 MHz using $CDCl_3$ as solvent and HMDS as internal reference. The mass spectra were run on a Hewlett Packard HP 5972 mass-selective detector (electron impact, 70 eV) coupled with an HP 5890 gas chromatograph (HP-5MS column). The progress of reactions and the purity of products were monitored by GLC on a Chrom-5 chromatograph equipped with a 2000× 2-mm glass column packed with Apiezon L on Chromaton-N-AW-DMCS (0.16-0.20 mm).

1,7,7-Trimethyl-2-phenylethynylbicyclo[2.2.1]-heptan-2-ol (I) was synthesized by reaction of camphor with lithium phenylacetylide according to the procedure described in [8]. mp 57–59°C. ^{1}H NMR spectrum, δ, ppm: 0.90 s (3H, 1-CH_3), 1.01 s (3H, *anti*-7-CH_3), 1.10 s (3H, *syn*-7-CH_3), 1.11–1.88 m (5H, 5-H, 6-H, *endo*-3-H), 1.99 s (1H, OH), 2.04 t (1H, 4-H, $^3J_{4,exo\text{-}3}$ = $^3J_{4,exo\text{-}5}$ = 3.6 Hz), 2.30 d.d.d (1H, *exo*-3-H,

^2J = 14.0, $^3J_{3,4}$ = 3.6, $^WJ_{3,exo\text{-}5}$ = 2.4 Hz), 7.32 m (5H, H_{arom}).

Ritter reaction of 1,7,7-trimethyl-2-phenylethynylbicyclo[2.2.1]heptan-2-ol (I). The reaction was carried out according to the procedure described in [1]: concentrated sulfuric acid, 3.5 ml, was added to a solution of 5.0 g of alcohol **I** in 7 ml of acetonitrile. Evaporation of the ether extract left a mixture of acetamides **II** and **III** at a ratio of 8:3 (GLC data). Overall yield 4.5 g (76%). The product mixture was separated by crystallization from hexane. The precipitate contained mainly amide **II**, and the mother liquor contained a mixture enriched in amide **III**.

***N*-(1,7,7-Trimethyl-4-phenylethynylbicyclo-[2.2.1]hept-2-yl)acetamide (II)** was isolated by repeated crystallization from hexane. mp 164–166°C. IR spectrum, ν, cm^{-1}: 3300 (NH); 3080, 3030, 3020 w (C–H_{arom}); 2960, 2930, 2880 (C–H_{aliph}); 2220 w (C≡C); 1645 s (C=O); 1600 w (C=C_{arom}); 1550 s (NH); 755, 690 m (δC–H_{arom}). ^{1}H NMR spectrum, δ, ppm: 0.91 s (6H, 1-CH_3, *anti*-7-CH_3), 0.98 s (3H, *syn*-7-CH_3), 1.05–1.90 m (5H, 5-H, 6-H, *exo*-3-H), 1.98 s (3H, $COCH_3$), 2.27 d.d (1H, *endo*-3-H, ^2J 13.2, $^3J_{endo,endo}$ = 8.8 Hz), 3.98 d.t (1H, *endo*-2-H, $^3J_{2,endo\text{-}3}$ = $^3J_{2,NH}$ = 8.8, $^3J_{2,exo\text{-}3}$ = 4.8 Hz), 5.40 br.d (NH, $^3J_{NH,2}$ = 8.8 Hz), 7.33 m (5H, H_{arom}). Mass spectrum, *m*/*z* (I_{rel}, %): 295 $[M]^+$ (47), 280 $[M - CH_3]^+$ (5), 252 $[M - CH_3CO]^+$ (7), 236 $[M - CH_3CONH_2]^+$ (24), 221 $[M - CH_3CONH_2 - CH_3]^+$ (39), 195 $[M - C{\equiv}CC_6H_4]^+$ (76), 168 (39), 165 (29), 128 (22), 115 (40), 91 (22), 77 $[C_6H_5]$ (18), 55 (19), 43 (100).

***N*-(4-Benzoylmethyl-1,7,7-trimethylbicyclo-[2.2.1]hept-2-yl)acetamide (III)** was isolated from the mother liquor and was purified by double recrystallization from methanol, mp 108–109°C. IR spectrum, ν, cm^{-1}: 3280 (NH); 3065, 3025 w (C–H_{arom}); 2955, 2930, 2880 (C–H_{aliph}); 1680 s (C=O, ketone); 1640 s (C=O, amide); 1600, 1585 w (C=C_{arom}); 1545 s (NH); 750, 690 m (δC–H_{arom}). ^{1}H NMR spectrum, δ, ppm: 0.80 s (6H, 1-CH_3, *anti*-7-CH_3), 0.88 s (3H, *syn*-7-CH_3), 1.55–1.80 m (5H, 5-H, 6-H, *exo*-3-H), 1.94 s (3H, $COCH_3$), 2.06 d.d (1H, *endo*-3-H, ^2J = 12.8, $^3J_{endo,endo}$ = 9.2 Hz), 3.92 d.t (1H, *endo*-2-H, $^3J_{2,endo\text{-}3}$ = $^3J_{2,NH}$ = 9.2, $^3J_{2,exo\text{-}3}$ = 4.6 Hz), 5.30 br.d (1H, NH, $^3J_{NH,2}$ = 8.8 Hz), 7.50 m (3H, H_{arom}), 7.90 m (2H, *o*-H). Mass spectrum, *m*/*z* (I_{rel}, %): 313 $[M]^+$ (2), 295 $[M - H_2O]^+$ (<1), 270 $[M - CH_3CO]^+$ (<1), 254 $[M - CH_3CONH_2]^+$ (4), 229 (1), 197 (1), 194 $[M - C_6H_5COCH_2]^+$ (11), 170 (11), 134 (46), 119 (7), 105 (100), 77 $[C_6H_5]$ (40), 55 (7), 43 (33).

The Ritter reaction in the presence of BF_3–Et_2O was performed in a similar way. The reaction mixture was decomposed with excess aqueous NaOH. As a result, 4.25 g (72%) of a mixture of amides **II** and **III** was isolated, their ratio being approximately the same as in the reaction catalyzed by sulfuric acid.

Reaction of 1,7,7-trimethyl-2-phenylethynylbicyclo[2.2.1]heptan-2-ol with formic acid. Compound **I**, 5.0 g, was dissolved in 10 ml of 85% formic acid, and the mixture was heated under reflux until the reaction was complete (GLC). The mixture was cooled, neutralized with excess aqueous ammonia, and extracted with hexane. The extract was dried over magnesium sulfate, the aqueous phase was additionally extracted with diethyl ether, and the ether extract was dried separately. Evaporation of the hexane extract gave almost pure ketone **XI**.

2-(1,7,7-Trimethylbicyclo[2.2.1]hept-2-ylidene)-1-phenylethanone (XI). Yield 78%, thick transparent oily substance, n_D^{16} = 1.5650. IR spectrum, ν, cm^{-1}: 3060, 3025 (C–H_{arom}); 2955, 2870 (C–H_{aliph}); 1665 s (C=O); 1610 s (C=C); 1580 w (C=C_{arom}); 700 (δC–H_{arom}). ^{1}H NMR spectrum, δ, ppm: 0.77 s (3H, 1-CH_3), 0.97 s (3H, *anti*-7-CH_3), 1.09 s (3H, *syn*-7-CH_3), 1.50–1.95 m (5H, 4-H, 5-H, 6-H), 2.55 d.d (1H, *endo*-3-H, ^2J = 19.8, $^4J_{3,11}$ = 2.4 Hz), 3.00 d.d.t (1H, *exo*-3-H, ^2J = 19.8 , $^3J_{3,4}$ = 4.0 , $^WJ_{3,exo\text{-}5}$ = $^4J_{3,11}$ = 2.4 Hz), 6.78 t (1H, 11-H, $^4J_{11,exo\text{-}3}$ = $^4J_{11,endo\text{-}3}$ = 2.4 Hz), 7.49 m (3H, H_{arom}), 7.90 m (2H, H_{arom}). Mass spectrum, *m*/*z* (I_{rel}, %): 254 $[M]^+$ (40), 239 $[M - CH_3]^+$ (21), 226 (20), 211 (70), 193 (13), 172 (11), 149 $[M - C_6H_5CO]^+$ (12), 134 $[M - C_6H_5CO - CH_3]^+$ (7), 115 (17), 105 $[C_6H_5CO]^+$ (100), 91 (32), 77 $[C_6H_5]$ (82), 51 (18), 41 (25).

Evaporation of the ether extract afforded a mixture consisting of hydroxy ketone **XVII** and enone **XI** (~20%). This mixture was dissolved in methanol, water was added to the solution until it became turbid, and the mixture was extracted with hexane. Enone **XI** was thus transferred into the hexane extract, while the aqueous–methanolic phase was enriched with compound **XVII**. Repetition of this procedure gave a small amount of almost pure compound **XVII**.

1-(2-Hydroxy-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)-2-phenylethanone (XVII). n_D^{16} = 1.5715. IR spectrum, ν, cm^{-1}: 3470 (OH); 3060, 3030 (C–H_{arom}); 2955, 2870 (C–H_{aliph}); 1720 s (C=O); 1580 w (C=C_{arom}); 755, 690 (δC–H_{arom}). ^{1}H NMR spectrum, δ, ppm: 0.95 s (3H, 1-CH_3), 1.08 s (*anti*-7-CH_3), 1.21 s

(3H, *syn*-7-CH_3), 1.55–2.05 m (6H, 4-H, 5-H, 6-H, OH), 2.32 d (1H, *endo*-3-H, ^2J = 13.2 Hz), 2.98 d.d.d (1H, *exo*-3-H, ^2J = 13.2, $^3J_{3,4}$ = 4.0, $^WJ_{3,exo\text{-}5}$ = 2.4 Hz), 3.88 s (2H, 11-H), 7.52 m (5H, H_{arom}). Mass spectrum, *m*/*z* (I_{rel}, %): 272 $[M]^+$ (3), 254 $[M - H_2O]^+$ (37), 239 $[M - H_2O - CH_3]^+$ (17), 225 (11), 210 (34), 194 (9), 181 $[M - C_6H_5CH_2]^+$ (31), 165 $[M - C_6H_5CH_2 - H_2O]^+$ (70), 153 $[M - C_6H_5CH_2CO]^+$ (17), 135 $[M - C_6H_5CH_2CO - H_2O]^+$ (100), 119 $[C_6H_5CH_2CO]^+$ (34), 115 (11), 105 (88), 91 $[C_6H_5CH_2]^+$ (38), 77 $[C_6H_5]^+$ (17), 51 (17), 41 (22).

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