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Hydrogenolysis of 7-Phenyltricyclo[4.1.0.0^{2,7}]heptan-1-carboxylic Acid. Synthesis of Ketones with Tricyclo[4.4.0.0^{2,7}]decane and Tricyclo[5.4.0.0^{2,8}]undecane Skeletons

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Abstract—Hydrogenation of 7-phenyltricyclo[4.1.0.0^{2,7}]heptane-1-carboxylic acid over Raney nickel occurred in the *syn*-stereoselective fashion to give *anti*-7-phenylbicyclo[3.1.1]heptane-*exo*-6-carboxylic acid. The latter was used to synthesize 4,5-benzotricyclo[4.4.0.0^{2,7}]dec-4-en-3-one and two isomeric higher homologs, 5,6-benzotricyclo[5.4.0.0^{2,8}]undec-5-en-3- and -4-ones.

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The strained bicyclobutane system undergoes cleavage by the action of catalytically excited hydrogen. The depth and direction of hydrogenolysis of bicyclobutane system depend on the substitution pattern therein [1]. Most frequently, cleavage of two C–C bonds (usually central and lateral) occurs simultaneously; bicyclobutane is thus converted into butane, and tricyclo[4.1.0.0^{2,7}]-heptane, into methylcyclohexane [2]. Examples of hydrogenolysis with cleavage of only the C–C bond were also reported, but this reaction pathway generally does not predominate [3].

It is known that α,β-unsaturated carboxylic acids, e.g., cinnamic acid, can be effectively hydrogenated over nickel–aluminum alloy in alkaline medium [4]. Taking into account partially double character of the central bicyclobutane C–C bond [1], 7-phenyltricyclo[4.1.0.0^{2,7}]-heptane-1-carboxylic acid (**I**) may be regarded as an analog of cinnamic acid. Therefore, we thought it to be reasonable to apply the above reducing agent to effect hydrogenolysis of acid **I**. Powdered Raney nickel (nickel–aluminum alloy at a weight ratio of 1:1) was added in portions under vigorous stirring to a solution of acid **I** in aqueous alkali at 80–85°C. The reaction involved exclusively the central bicyclobutane bond with strict *endo*,*syn* stereoselectivity to produce *anti*-7-phenylbicyclo[3.1.1]heptane-*exo*-6-carboxylic acid (**II**) as the only product in almost quantitative yield* (Scheme 1).

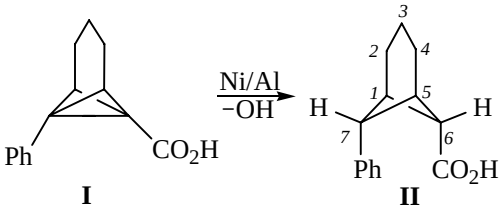
For preliminary Communication, see [5].

The structure of acid **II** and methyl ester **III** derived therefrom was confirmed by the ^{1}H and ^{13}C NMR spectra (see below).

anti-7-Phenylbicyclo[3.1.1]heptane-*exo*-1-carboxylic acid (**II**) seemed to be very promising from the viewpoint of obtaining strained structures in which the 6- and 7-positions of norpinane fragment are linked through a hydrocarbon bridge. In the present communication, the synthetic potential of acid **II** is illustrated by three examples. We have synthesized ketone **IV** having a tricyclo[4.4.0.0^{2,7}]decane skeleton and two its higher homologs, isomeric ketones **V** and **VI** having a tricyclo-[5.4.0.0^{2,8}]undecane skeleton (Scheme 2).

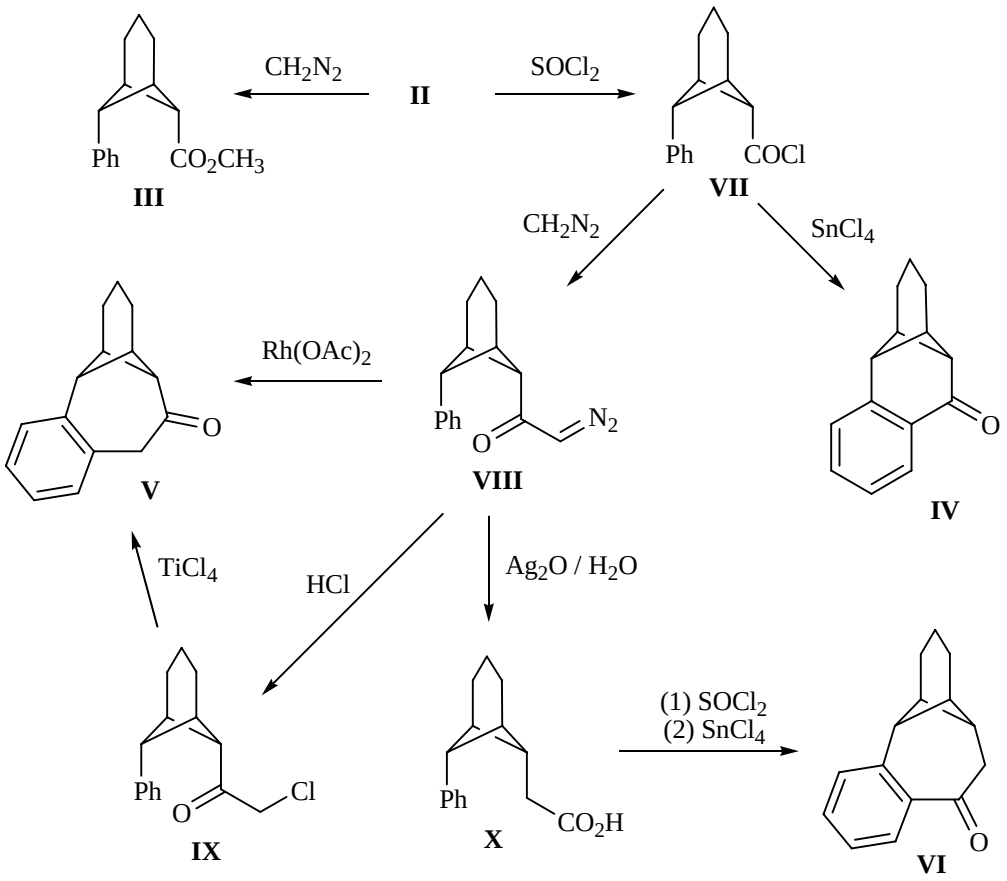
To obtain ketone **IV**, acid **II** was converted into the corresponding acyl chloride **VII** which was subjected to intramolecular acylation in the presence of $SnCl_4$ as catalyst. Treatment of acyl chloride **VII** with diazomethane gave diazo ketone **VIII** which reacted with hydrogen chloride, yielding chloro ketone **IX**. Ketone **V**

Scheme 1.



I **II**

Scheme 2.



was synthesized by both deazotization of compound **VIII** by the action of rhodium acetate and by intramolecular alkylation of **IX** in the presence of titanium(IV) chloride. Finally, ketone **VI** was prepared by intramolecular acylation of acid **X** which was obtained in turn by the Arndt–Eistert reaction from acid **II** through intermediate diazo ketone **VIII**. All compounds **II**–**X** were characterized by the ^{1}H and ^{13}C NMR spectra.

Both bicyclic compounds **II**, **III**, and **VII**–**X** and benzo-fused tricyclic ketones **IV**–**VI** formally belong to the *exo*-6-*anti*-7-disubstituted bicyclo[3.1.1]heptane series. In the ^{1}H NMR spectra of bicyclic derivatives **II**, **III**, and **VII**–**X**, respective protons of the bicyclo-[3.1.1]heptane skeleton have almost similar chemical shifts. An exception is compound **X**; as might be expected, the 1-H/5-H and 6-H signals in its ^{1}H NMR spectrum appear in a stronger field. A specific feature of the above compounds is that the 1-H/5-H signal is a broadened singlet since all vicinal coupling constant for those protons are fairly small ($J \approx 0$–1 Hz) [6]. Finally, a strong evidence in favor of the assumed configuration of molecules **II**, **III**, and **VII**–**X** is provided by the multiplicities of the 6-H and 7-H signals. In all cases, these signals appear as two doublets (in the spectrum of **X** the 6-H signal is a doublet of triplets) due to long-range spin–spin coupling between nonequivalent 6-H and 7-H protons (*W*-coupling [7]).

Useful information was also obtained from the ^{13}C NMR spectra of bicyclic compounds **II**, **III**, and **VII**–**X**. In each case, we observed five carbon signals (two of which had a double intensity) belonging to the norpinane fragment; their position was weakly sensitive to the substituent nature on C^{6}. As above, an exception was compound **X** whose C^{6} signal was displaced by about 5 ppm upfield due to remoteness of the carbonyl group.

The trimethylene bridges in benzo-fused tricyclic ketones **IV**–**VI**, as well as in bicyclic compounds **II**, **III**, **VII**–**X**, were characterized by almost similar chemical shifts and multiplicities of signals of the corresponding protons and carbon atoms in the ^{1}H and ^{13}C NMR spectra. Differences were observed in the spectral patterns for

the cyclobutane fragment in going from bicyclic compounds **II**, **III**, and **VII**–**X** to tricyclic ketones **IV**–**VI**; these differences resulted from incorporation of the norpinane fragment into tricyclic structure and change of the orientation of the aromatic ring. Some specificity is intrinsic to ketone **IV** (but not to **V** or **VI**): in the ^{1}H NMR spectrum of **IV**, two nonequivalent protons structurally analogous to 6-H and 7-H in bicyclic compounds **II**, **III**, and **VII**–**X** gave rise to one broadened singlet because of accidental almost complete coincidence of their chemical shifts. In the ^{13}C NMR spectra of **IV**–**VI**, two signals from carbon atoms of the cyclobutane fragment were displaced downfield by about 10 ppm relative to the respective signals of bicyclic compounds **II**, **III**, and **VII**–**X**.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were measured from solutions on in $CDCl_3$ on a Bruker DPX–300 spectrometer at 300.13 and 75.47 MHz, respectively. The elemental compositions were determined on Hewlett–Packard HP-185B CHN analyzer. Analytical thin-layer chromatography was performed on Silufol UV-254 plates. The products were isolated and purified by column chromatography on silica gele L (40–100 μm, Chemapol). 7-Phenyltricyclo[4.1.0.0^{2,7}]heptane-1-carboxylic acid (**I**) was synthesized according to the procedure described in [8].

***anti*-7-Phenylbicyclo[3.1.1]heptane-*exo*-6-carboxylic acid (II).** Acid **I**, 6.42 g (0.03 mol), was dissolved in a solution of 40 g (1 mol) of sodium hydroxide in 300 ml of water, the mixture was heated to 80–85°C, and 13.5 g (0.25 mol of Al) of Raney nickel was added in portions under stirring over a period of 1.5 h. The mixture was stirred for 3 h, the heating bath was removed, and the mixture was diluted with 200 ml of water and filtered. The filtrate was cooled and acidified to pH 1 by adding 15% hydrochloric acid under stirring. The precipitate was filtered off, thoroughly washed with water, and dried in a vacuum desiccator. Yield 6.23 g (96%), mp 117°C (from hexane). ^{1}H NMR spectrum, δ, ppm: 1.82–2.02 m (2H, 3-H), 2.12–2.28 m (4H, 2-H, 4-H), 2.49 d (1H, 6-H, J = 4.5 Hz), 2.94 d (1H, 7-H, J = 4.5 Hz), 3.09 br.s (2H, 1-H, 5-H), 7.12–7.40 m (5H, H_{arom}), 10.37 br.s (1H, CO_2H). ^{13}C NMR spectrum, δ_C, ppm: 15.7 (C^3); 30.9 (C^2, C^4); 41.0 (C^1, C^5); 46.8 (C^6); 49.9 (C^7); 125.6, 127.1, 127.8, 141.4 (C_{arom}); 180.8 (C=O). Found, %: C 77.89; H 7.51. $C_{14}H_{16}O_2$. Calculated, %: C 77.75; H 7.46.

Methyl *anti*-7-phenylbicyclo[3.1.1]heptane-*exo*-6-carboxylate (III) was synthesized by adding a solution of diazomethane in diethyl ether to a solution of acid **II** in diethyl ether until the mixture turned persistently yellow. The solvent was removed, and the residue was recrystallized from hexane. Yield nearly quantitative, mp 27°C, R_f 0.71 (hexane–diethyl ether, 4:1). ^{1}H NMR spectrum, δ, ppm: 1.85–2.05 m (2H, 3-H), 2.16–2.30 m (4H, 2-H, 4-H), 2.45 d (1H, 6-H, J = 4.5 Hz), 2.97 d (1H, 7-H, J = 4.5 Hz), 3.14 br.s (2H, 1-H, 5-H), 3.23 s (3H, OCH_3), 7.11–7.21 m (1H, H_{arom}), 7.25–7.35 m (4H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 15.8 (C^3); 30.8 (C^2, C^4); 41.3 (C^1, C^5); 47.0 (C^6); 49.5 (C^7); 51.0 (OCH_3); 125.5, 127.1, 127.7, 141.8 (C_{arom}); 175.0 (C=O). Found, %: C 78.29; H 7.80. $C_{15}H_{18}O_2$. Calculated, %: C 78.23; H 7.88.

4,5-Benzotricyclo[4.4.0.0^{2,7}]dec-4-en-3-one (IV). A solution of 0.52 g (2 mmol) of anhydrous tin(IV) chloride in 5 ml of benzene was added in one portions to a solution of 0.47 g (2 mmol) of acyl chloride **VII** in 10 ml of anhydrous benzene. The mixture was stirred and kept for 6 h at 25°C, 20 ml of 5% hydrochloric acid was added, and the mixture was vigorously stirred. The organic phase was separated, washed in succession with water (3×10 ml), a 5% solution of sodium hydrogen carbonate (3×10 ml), and water again (2×10 ml), and dried over sodium sulfate. The solvent was removed under reduced pressure, and the residue was recrystallized from hexane. Yield 0.32 g (81%), mp 41°C, R_f 0.73 (hexane–diethyl ether, 1:1). ^{1}H NMR spectrum, δ, ppm: 1.82–1.95 m (2H, 9-H), 1.97–2.21 m (4H, 8-H, 10-H), 2.89 br.s (2H, 2-H, 6-H), 2.98 br.s (2H, 1-H, 7-H), 7.24 d (1H, H_{arom}, J = 8.0 Hz), 7.33 t (1H, H_{arom}, J = 8.0 Hz), 7.43 t (1H, H_{arom}, J = 8.0 Hz), 7.96 d (1H, H_{arom}, J = 8.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 16.0 (C^9); 28.7 (C^8, C^{10}); 47.0 (C^6); 53.7 (C^1, C^7); 56.9 (C^2); 125.1, 126.1, 126.8, 129.3, 133.0, 151.3 (C_{arom}); 201.2 (C=O). Found, %: C 84.75; H 7.15. $C_{14}H_{14}O$. Calculated, %: C 84.81; H 7.12.

5,6-Benzotricyclo[5.4.0.0^{2,8}]undec-5-en-3-one (V). *a*. Diazo ketone **VIII**, 0.24 g (1 mmol), was dissolved in 30 ml of benzene, 10 mg of rhodium(II) acetate was added, and the mixture was heated for 1 h under reflux (until the initial diazo ketone disappeared according to the TLC data). The solvent was removed under reduced pressure, and the residue was purified by flash chromatography on silica gel. Yield 0.14 g (68%), mp 31°C (from ethanol), R_f 0.81 (hexane–diethyl ether, 1:1). ^{1}H NMR spectrum, δ, ppm: 1.84–1.98 m (2H, 10-H),

2.02–2.20 m (4H, 9-H, 11-H), 2.78 d (1H, 7-H, J = 4.4 Hz), 2.92 d (1H, 2-H, J = 4.4 Hz), 3.01 br.s (2H, 1-H, 8-H), 3.58 s (2H, 4-H), 7.11–7.24 m (2H, H_{arom}), 7.33 t (1H, H_{arom}, J = 7.6 Hz), 7.43 d (1H, H_{arom}, J = 7.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 16.4 (C^{10}); 31.6 (C^9, C^{11}); 46.3 (C^4); 48.8 (C^7); 54.0 (C^1, C^8); 61.8 (C^2); 124.1, 125.7, 126.8, 128.4, 138.7, 142.0 (C_{arom}); 211.7 (C=O). Found, %: C 84.73; H 7.67. $C_{15}H_{16}O$. Calculated, %: C 84.87; H 7.60.

b. A solution of 0.19 g (1 mmol) of anhydrous titanium(IV) chloride in 5 ml of benzene was added in one portion to a solution of 0.25 g (1 mmol) of chloro ketone **IX** in 10 ml of benzene, and the mixture was stirred for 2 h at room temperature (until the initial chloro ketone disappeared according to the TLC data). The mixture was then treated with 10 ml of 5% hydrochloric acid and vigorously stirred. The organic layer was separated, washed in succession with water (4×10 ml), a 5% solution of sodium hydrogen carbonate (3×10 ml), and water again (2×10 ml), and dried over sodium sulfate. The solvent was removed under reduced pressure, and the residue was purified by flash chromatography on silica gel. Yield 0.11 g (52%).

5,6-Benzotricyclo[5.4.0.0^{2,8}]undec-5-en-4-one (VI). A solution of 0.24 g (2 mmol) of freshly distilled thionyl chloride in 5 ml of benzene was carefully added to 0.25 g (1 mmol) of acid **X**, and the mixture was stirred for 4 h at room temperature. Volatile components were removed under reduced pressure, the residue was dissolved in 5 ml of anhydrous benzene, and a solution of 0.27 g (1 mmol) of anhydrous tin(IV) chloride in 5 ml of benzene was added. The mixture was stirred for 6 h at room temperature, 10 ml of 5% hydrochloric acid was added, and the organic layer was separated, washed in succession with water (3×5 ml), a 5% solution of sodium hydrogen carbonate (3×5 ml), and water again (2×5 ml), and dried over sodium sulfate. Yield 0.15 g (71%), mp 38°C (from ethanol), R_f 0.76 (hexane–diethyl ether, 1:1). ^1H NMR spectrum, δ, ppm: 2.02–2.13 m (2H, 10-H), 2.15–2.24 m (4H, 9-H, 11-H), 2.30 d.t (1H, 2-H, J = 4.5, 8.0 Hz), 2.38 d (2H, 3-H, J = 8.0 Hz), 2.48 d (1H, 7-H, J = 4.5 Hz), 2.59 br.s (2H, 1-H, 8-H), 7.18 d (1H, H_{arom}, J = 7.9 Hz), 7.31 t (1H, H_{arom}, J = 7.9 Hz), 7.44 t (1H, H_{arom}, J = 7.9 Hz), 7.98 d (1H, H_{arom}, J = 7.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 16.3 (C^{10}); 31.6 (C^9, C^{11}); 45.7 (C^3); 48.7 (C^2); 52.5 (C^1, C^8); 53.1 (C^7); 125.9, 127.1, 127.4, 131.4, 133.8, 151.0 (C_{arom}); 202.9 (C=O). Found, %: C 84.70; H 7.71. $C_{15}H_{16}O$. Calculated, %: C 84.87; H 7.60.

***anti*-7-Phenylbicyclo[3.1.1]heptane-*exo*-6-carbonyl chloride (VII).** A solution of 2.38 g (20 mmol) of freshly distilled thionyl chloride in 10 ml of benzene was carefully added to 2.16 g (10 mmol) of acid **II**, and the mixture was stirred for 4 h at room temperature. Volatile components were removed under reduced pressure, 50 ml of hexane was added to the residue, the mixture was filtered, and the filtrate was evaporated to a volume of ~10 ml. After prolonged keeping in a refrigerator (–5°C), colorless crystals of acyl chloride **VII** separated and were filtered off. Yield 1.86 g (79%), mp 47°C (from hexane). ^1H NMR spectrum, δ, ppm: 1.86–2.07 m (2H, 3-H), 2.18–2.34 m (4H, 2-H, 4-H), 2.47 d (1H, 6-H, J = 4.5 Hz), 3.03 d (1H, 7-H, J = 4.5 Hz), 3.18 br.s (2H, 1-H, 5-H), 7.12–7.45 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 16.0 (C^3); 31.1 (C^2, C^4); 41.6 (C^1, C^5); 47.1 (C^6); 49.6 (C^7); 125.4, 127.2, 127.9, 142.0 (C_{arom}); 167.2 (C=O). Found, %: C 71.81; H 6.52. $C_{14}H_{15}ClO$. Calculated, %: C 71.64; H 6.44.

2-Diazo-1-(*exo*-7-phenylbicyclo[3.1.1]heptan-*anti*-1-yl)ethanone (VIII). A round-bottom flask equipped with a pressure-relief valve was charged with a solution of 2.35 g (10.0 mmol) of acyl chloride **VII** in 30 ml of anhydrous diethyl ether. A solution of 1.26 g (30 mmol) of diazomethane in diethyl ether was carefully added, and the mixture was kept first for 2 h at 0°C and then for 12 h at room temperature. The solvent and traces of diazomethane were removed under reduced pressure (water-jet pump). The residue, a bright yellow oily substance (2.4 g, ~100%) was used in further syntheses without additional purification. An analytical sample was obtained by purification of the crude product by flash chromatography on silica gel L (40–100 μm). Light yellow oily liquid, R_f 0.17 (hexane–diethyl ether, 1:1). ^1H NMR spectrum, δ, ppm: 1.82–2.08 m (2H, 3-H), 2.12–2.32 m (4H, 2-H, 4-H), 2.48 d (1H, 6-H, J = 4.6 Hz), 2.96 d (1H, 7-H, J = 4.6 Hz), 3.08 br.s (2H, 1-H, 5-H), 5.05 br.s (1H, CHN_2), 7.11–7.22 m (1H, H_{arom}), 7.24–7.35 m (4H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 15.8 (C^3); 30.8 (C^2, C^4); 41.2 (C^1, C^5); 46.7 (C^6); 49.0 (C^7); 53.6 (CN_2); 126.1, 127.1, 128.2, 141.2 (C_{arom}); 203.9 (C=O). Found, %: C 75.05; H 6.65; N 11.57. $C_{15}H_{16}N_2O$. Calculated, %: C 74.94; H 6.71; N 11.70.

2-Chloro-1-(*exo*-7-phenylbicyclo[3.1.1]heptan-*anti*-1-yl)ethanone (IX). Diazo ketone **VIII**, 0.240 g (1 mmol), was dissolved in 10 ml of methylene chloride, 2 ml of a saturated solution of HCl in methylene chloride was added, and the mixture was stirred for 2 h at room temperature (until the initial diazo ketone disappeared

according to the TLC data). The mixture was washed with water (3×10 ml) and dried over sodium sulfate. Yield 0.22 g (91%), mp 49°C (from ethanol), R_f 0.66 (hexane–diethyl ether, 2 : 1). ^{1}H NMR spectrum, δ, ppm: 1.84–1.97 m (2H, 3-H), 2.10–2.31 m (4H, 2-H, 4-H), 2.53 d (1H, 6-H, J = 4.6 Hz), 2.91 d (1H, 7-H, J = 4.6 Hz), 3.19 br.s (2H, 1-H, 5-H), 4.67 s (1H, CH_2Cl), 7.05–7.32 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 16.1 (C^3); 31.3 (C^2, C^4); 41.8 (C^1, C^5); 46.9 (C^6); 50.0 (C^7); 51.2 (CH_2Cl); 126.2, 127.4, 128.4, 141.1 (C_{arom}); 204.4 (C=O). Found, %: C 72.54; H 6.81. $C_{15}H_{17}ClO$. Calculated, %: C 72.43; H 6.89.

***anti*-2-(*exo*-7-Phenylbicyclo[3.1.1]hept-6-yl)acetic acid (X).** A solution of 0.48 g (2 mmol) of diazo ketone **VIII** in 10 ml of dioxane was added dropwise under stirring to a mixture of 0.46 g (2 mmol) of freshly prepared silver oxide, 0.5 g of Na_2CO_3, and 0.3 g of $NaHSO_3$ in 20 ml of water, heated to 50–60°C. The mixture was then stirred for 2 h, raising the temperature to 90–100°C (disappearance of the initial diazo ketone was monitored by TLC). The solution was cooled, diluted with water, and acidified to pH 1 with dilute nitric acid, and the precipitate was filtered off, washed with water, and dried in a vacuum desiccator. Yield 0.30 g (66%), mp 102°C (from hexane), R_f 0.42 (hexane–diethyl ether, 1 : 1). ^{1}H NMR spectrum, δ, ppm: 1.87–1.99 m (2H, 3-H), 2.10 d.t (1H, 6-H, J = 4.4, 8.0 Hz), 2.13–2.26 m (4H, 2-H, 4-H), 2.31 d (2H, CH_2CO, J = 8.0 Hz), 2.67 br.s (2H, 1-H, 5-H), 3.00 d (1H, 7-H, J = 4.4 Hz), 7.14–7.42 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 15.6 (C^3); 31.8 (C^2, C^4); 37.1 (**C**H_2CO); 41.5 (C^1, C^5); 41.6 (C^6); 47.4 (C^7); 125.3, 126.7, 128.3, 144.5 (C_{arom}); 179.4 (C=O). Found, %: C 78.34; H 7.93. $C_{15}H_{18}O_2$. Calculated, %: C 78.23; H 7.88.

REFERENCES

1. Hoz, S., *The Chemistry of the Cyclopropyl Group*, Rappoport, Z., Ed., Chichester: Wiley, 1987, vol. 1, p. 1121.
2. Stahl, K.J., Hertzsch, W., and Musso, H., *Justus Liebigs Ann. Chem*., 1985, p. 1474.
3. Velltuzo, A.F. and Griffin, G.W., *J. Am. Chem. Soc*., 1965, vol. 87, p. 3021; Turner, R.B., Goebel, P., Mallon, B.J., Doering, W.E., Coburn, J.F., and Pomerantz, M., *J. Am. Chem. Soc*., 1968, vol. 90, p. 4315.
4. Fieser, L.F. and Fieser, M., *Reagents for Organic Synthesis*, New York: Wiley, 1968. Translated under the title *Reagenty dlya organicheskogo sinteza*, Moscow: Mir, 1970, vol. 2, p. 434.
5. Zolotarev, R.N. and Razin, V.V., *Russ. J. Org. Chem*., 1998, vol. 34, p. 1790.
6. Wiberg, K.B. and Hess, B.A., *J. Org. Chem*., 1966, vol. 31, p. 2250.
7. Günther, H., *NMR Spectroscopy: an Introduction*, Chichester: Wiley, 1980. Translated under the title *Vvedenie v kurs spektroskopii YaMR*, Moscow: Mir, 1984, p. 132.
8. Koptelov, Yu.B., Kostikov, R.R., and Molchanov, A.P., *Zh. Org. Khim*., 1989, vol. 25, p. 2024; Razin, V.V. and Makarychev, Yu.A., *Zh. Org. Khim*., 1992, vol. 28, p. 2490.