

2-(4-Nitrophenyl)oxirane Amino Derivatives in Heterocyclization Reactions

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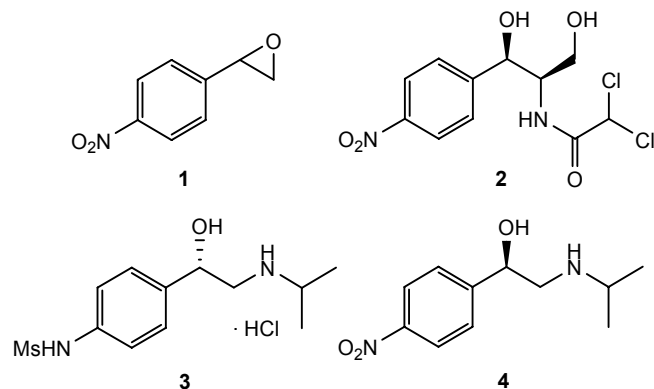
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Abstract—Reactions of 2-amino-1-(4-nitrophenyl)ethanol with various electrophilic reagents led to the formation of oxazaheterocycles (1,3-oxazolidin-2-one, 1,3-oxazolidine, morpholin-2-, -3-one, and -2,3-dione). In reactions of 2-{(bicyclo[2.2.1]hept-5-en-2-ylmethyl)amino}-1-(4-nitrophenyl)ethan-1-ol with carbonyldiimidazole, oxalyl chloride, benzaldehyde, and formaldehyde the corresponding derivatives of 1,3-oxazolidin-2-one, morpholin-2,3-dione, and 1,3-oxazolidine were obtained.

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Among the numerous epoxy compounds a special place belongs to 4-nitrostyrene oxide **1** [2-(4-nitrophenyl)oxirane]. The structural fragment of 4-nitrophenyloxirane is a very prospective scaffold existing in many biologically active substances, antibiotics, vasodilators, herbicides, plant growth regulators [1]. The most well-known pharmaceuticals are antibiotic chloramphenicol **2** [levomycetin, *D*-(–)-*threo*-1-(4-nitrophenyl)-2-(dichloroacetyl)amino]propane-1,3-diol], and its racemate synthomycine [2–4], and also β -adrenoblockers *d*-(+)-sotalol **3** and (*R*)-nifenalol **4** possessing antianginal, antiarrhythmic, and hypotensive action [5–9].

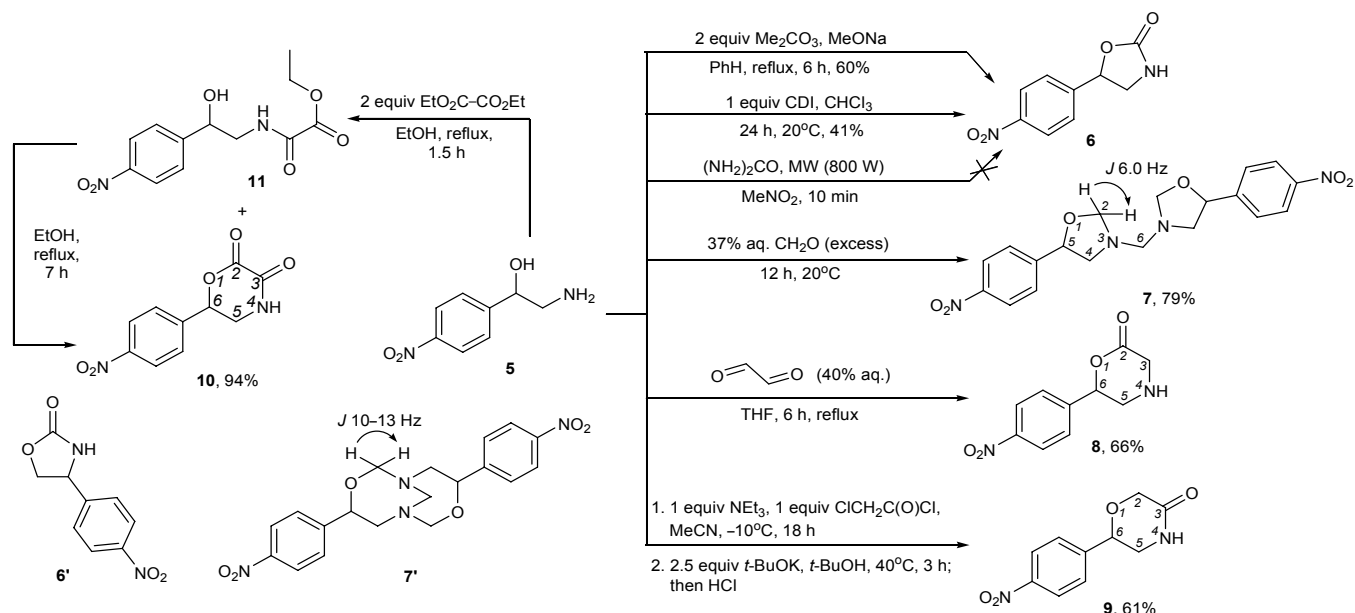


The recent studies of our scientific team revealed the analgesic, anticonvulsant, tranquillizing activity of cage sulfonamides and aminoalcohols including a 4-nitrophenyl fragment [10–12].

Here we report on an extension of the investigation in the field of aminoalcohols synthesis and their functionalization at the alternative nucleophilic centers (the preparation of *N*-acyl, *N*-sulfonyl, *O*-silyl, *O*-acyl derivatives) [13, 14]. We studied the possibility of aminoalcohols heterocyclization into five- and six-membered nonaromatic 1,3- and 1,4-oxazaheterocycles, whose practical importance had been widely described in [15–18]. Some of the obtained compounds include a secondary amine or amide moiety, which increases their synthetic potential and enables them to participate in the development of combinatorial libraries of new compounds via subsequent acylation and alkylation at the nitrogen atom.

We selected as the starting material 2-amino-1-(4-nitrophenyl)ethanol **5** [19, 20]. Its reactions with various cyclization agents led to the obtaining of a number of simplest oxazaheterocyclic derivatives **6–10**. 1,3-Oxazolidin-2-one **6** is formed by the action of carbonyldiimidazole (CDI) on the starting aminoalcohol, or in interaction with dimethyl carbonate. In the latter case the yield reached 60%. The methods known before [heating of 2-(4-nitrophenyl)oxirane **1** with urea above the melting point in solvent free conditions, or refluxing starting materials in DMF] afforded compound **6** in 7–30% yields [21], moreover regioisomeric oxazolidinone **6'** is formed as a by-product. The microwave irradiation of the mixture of aminoalcohol **5** with urea in nitromethane as described in [22] does not lead to the desired product **6** and the

Scheme 1.

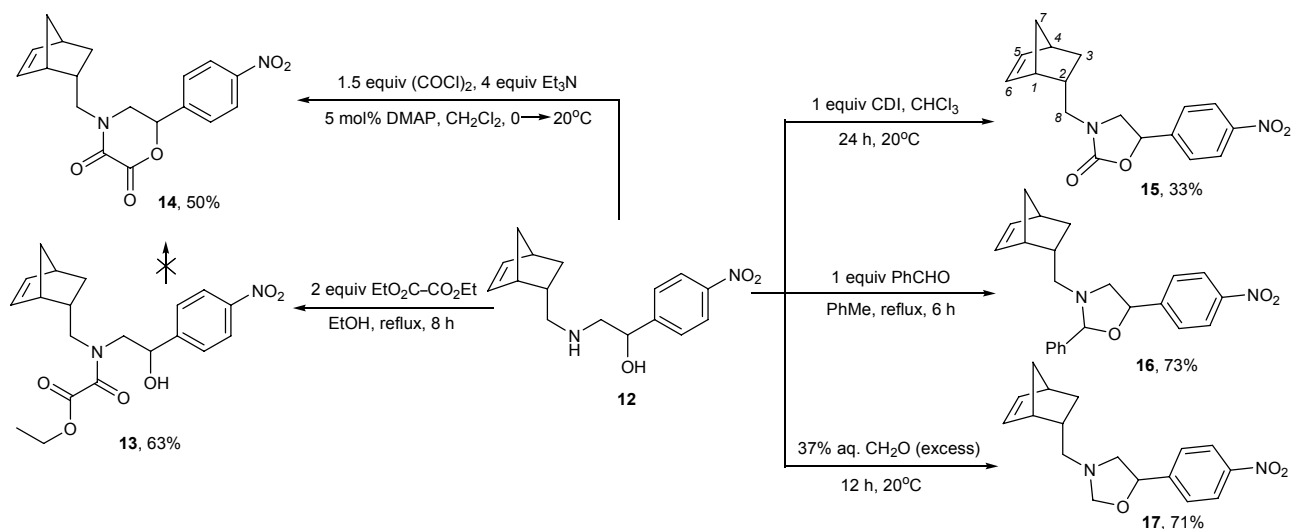


starting aminoalcohol was recovered nearly quantitatively. The reaction of compound 5 with formaldehyde yielded bisoxazolidine 7. The formation of compound 7 is confirmed by the value of the heminal spin-spin coupling constant of the protons of the OCH_2N group ($^2J_{2A,2B}$ 6 Hz). According to published data in the alternative isomer of the structure of 1,6-diaza-3,8-dioxabicyclo[4.4.1]undecane 7' this coupling constant should be of 10–13 Hz [23, 24]. Besides the formation of similar bis-products in analogous conditions was mentioned in [25, 26]. Morpholin-2- and -3-ones 8 and 9 were obtained by procedures [27, 28]. The reaction

of excess diethyl oxalate with compound 5 first gave the oxalic acid amidoester 11 (data of ^1H NMR spectrum). After 1.5 h of boiling reagents the ratio of compounds 10 and 11 was 1 : 2, at longer reaction we isolated only the derivative of morpholin-2,3-dione 10 (Scheme 1).

Since the oxazaheterocyclic derivatives of cage aminoalcohols almost have not been studied [15–18], some of the above described synthetic methods were tested on aminoalcohol 12 including a norbornene fragment. Aminoalcohol 12 was less active than its analog 5. The reaction with diethyl oxalate resulted in

Scheme 2.



amide **13**, but further cyclization into morpholin-2,3-dione **14** did not occur even after 36 h of boiling. Compound **14** formed only under the action of oxalyl chloride in the presence of a catalytic quantity of 4-dimethylaminopyridine. By reactions of aminoalcohol **12** with CDI, benzaldehyde, and formaldehyde we obtained the corresponding derivatives of 1,3-oxazolidin-2-one and 1,3-oxazolidine **15–17** (Scheme 2).

The structure of obtained compounds is confirmed by ^1H NMR and IR spectra.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Spectrum One (Perkin Elmer) using KBr pellets. ^1H NMR spectra were registered on Varian and Bruker spectrometers at operating frequencies 400 and 500 MHz with TMS as internal reference. The reaction progress was monitored and the purity of obtained compounds was checked by TLC on Silufol 60F₂₅₄ plates, eluent ethyl acetate–hexane; the plates were visualized with iodine vapor. Elemental analysis was performed using Carlo Erba analyzer. Spectral characteristics of compound **6** were in correspondence with the literature [21].

5-(4-Nitrophenyl)oxazolidin-2-one (6). *a.* To a solution of 0.50 g (2.74 mmol) of 2-amino-1-(4-nitrophenyl)ethanol **5** in 20 mL of anhydrous benzene was added 0.49 g (0.46 mL, 5.49 mmol) of dimethyl carbonate and 0.015 g (0.27 mmol, 10 mol %) of sodium methylate. The reaction mixture was boiled for 6 h, the solvent was removed in a vacuum, the residue was treated with water, neutralized with hydrochloric acid solution, the precipitate was filtered off and recrystallized from a mixture ethyl acetate–hexane. Yield 0.34 g (60%), mp 105–107°C (decomp.) (mp 110°C [27, 28]).

b. To a solution of 0.30 g (1.65 mmol) of 2-amino-1-(4-nitrophenyl)ethanol **5** in 10 mL of chloroform was added 0.27 g (1.7 mmol) of carbonyldiimidazole (CDI). The reaction mixture was stirred at 20°C for 24 h, washed with 33% water solution of citric acid (10 mL), then with water (2 × 15 mL). The organic layer was dried with sodium sulfate, evaporated in a vacuum, the residue was recrystallized from a mixture ethyl acetate–hexane. Yield 0.14 g (41%), mp 106–108°C (decomp.).

3-[(Bicyclo[2.2.1]hept-5-en-endo-2-yl)methyl]-5-(4-nitrophenyl)oxazolidin-2-one (15) was obtained by procedure *b* from 0.50 g (1.73 mmol) of aminoal-

cohol **12** [29] and 0.28 g (1.73 mmol) of carbonyldiimidazole. Yield 0.18 g (33%), mp 165–167°C. IR spectrum, cm^{-1} : 3445, 1750, 1515, 1445, 1350, 1255, 720. ^1H NMR spectrum (CDCl_3), δ , ppm: 0.55 m (1H, H^{3n}), 1.23 d (1H, H^{7a} , $^2J_{7s,7a}$ 8.1 Hz), 1.43 d (1H, H^{7s}), 1.85 m (1H, H^{3x}), 2.20 m (1H, H^2), 2.71 m (2H, H^8), 2.78 m (1H, H^4), 2.83 m (1H, H^1), 3.40 m (1H, NCH_2), 4.05 m (1H, NCH_2), 5.80 m (1H, OCH), 5.90 d.d (1H, H^5 , $^3J_{5,6}$ 6.0, $^3J_{4,5}$ 2.9 Hz), 6.15 d.d (1H, H^6 , $^3J_{5,6}$ 6.0, $^3J_{1,6}$ 3.1 Hz), 7.67 d (2H_{arom}, J 8.7 Hz), 8.20 d (2H_{arom}, J 8.7 Hz). Found, %: C 65.25; H 5.52; N 9.17. $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_4$. Calculated, %: C 64.96; H 5.77; N 8.91.

Bis[5-(4-nitrophenyl)oxazolidin-3-yl]methane (7). To 0.25 g (1.37 mmol) of aminoalcohol **5** was added 5 mL of 37% water solution of formaldehyde. The reaction mixture was stirred at 20°C for 12 h, solid sodium hydrogen carbonate was added to reach weak alkaline reaction, the reaction product was extracted into dichloromethane (3 × 20 mL). The organic layer was dried with sodium sulfate, evaporated in a vacuum, the residue was recrystallized from a mixture ethyl acetate–hexane. Yield 0.22 g (79%), mp 120–122°C. IR spectrum, cm^{-1} : 3030, 2880, 1595, 1345, 1190. ^1H NMR spectrum (CDCl_3), δ , ppm: 2.88 d.d (2H, H^{4A} , $^2J_{4A,4B}$ 11.8, $^3J_{5,4A}$ 7.5 Hz), 3.55 s (2H, H^6), 3.59 d.d (2H, H^{4B} , $^2J_{4A,4B}$ 11.8, $^3J_{5,4B}$ 6.5 Hz), 5.05 d (2H, H^{2A} , $^2J_{2A,2B}$ 6.0 Hz), 5.15 d (2H, H^{2B} , $^2J_{2A,2B}$ 6.0 Hz), 5.33 m (2H, H^5), 7.65 d (4H_{arom}, J 8.7 Hz), 8.20 d (4H_{arom}, J 8.7 Hz). Found, %: C 57.25; H 5.22; N 13.88. $\text{C}_{19}\text{H}_{20}\text{N}_4\text{O}_6$. Calculated, %: C 57.00; H 5.04; N 13.99.

3-[(Bicyclo[2.2.1]hept-5-en-endo-2-yl)methyl]-5-(4-nitrophenyl)oxazolidine (17) was similarly obtained from 0.50 g (1.73 mmol) of aminoalcohol **12** [29]. Yield 0.37 g (71%), mp 153–156°C. IR spectrum, cm^{-1} : 3045, 1715, 1525, 1420, 1125, 720. ^1H NMR spectrum (CDCl_3), δ , ppm: 0.75 m (1H, H^{3n}), 1.30 d (1H, H^{7a} , $^2J_{7s,7a}$ 8.1 Hz), 1.42 d (1H, H^{7s}), 1.71 m (1H, H^{3x}), 2.00 m (1H, H^2), 2.80–2.95 (4H, $\text{H}^{1,4,8}$), 4.01 m (2H, NCH_2CH), 4.50 d (1H, NCH_2O , 2J 6.1 Hz), 4.89 d (1H, NCH_2O , 2J 6.1 Hz), 5.15 m (1H, OCHCH_2), 6.05 d (1H, H^5 , $^3J_{5,6}$ 6.0 Hz), 6.20 d (1H, H^6 , $^3J_{5,6}$ 6.0 Hz), 7.69 d (2H_{arom}, J 8.7 Hz), 8.18 d (2H_{arom}, J 8.7 Hz). Found, %: C 68.21; H 6.50; N 9.11. $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_3$. Calculated, %: C 67.98; H 6.71; N 9.33.

6-(4-Nitrophenyl)morpholin-2-one (8). To a solution of 0.50 g (2.74 mmol) of aminoalcohol **5** in 30 mL of tetrahydrofuran was added 1.19 g (8.22 mmol) of 40% water glyoxal solution. The reaction mixture was

boiled for 6 h, poured into water (20 mL), the reaction product was extracted into dichloromethane (3 × 30 mL). Combined organic extracts were dried with sodium sulfate, evaporated, the residue was chromatographed on a column packed with silica gel (eluent hexane–ethyl acetate, 1 : 1). Yield 0.40 g (66%), mp 156–159°C (decomp.). IR spectrum, cm^{-1} : 3240, 3060, 1750, 1600, 1340, 1150. ^1H NMR spectrum (CDCl_3), δ , ppm: 1.65 br.s (1H, NH), 3.25–3.35 m (4H, $\text{H}^{3,5}$), 5.71 m (1H, H^6), 7.64 d (2H_{arom}, J 8.6 Hz), 8.21 d (2H_{arom}, J 8.6 Hz). Found, %: C 54.25; H 4.32; N 12.80. $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_4$. Calculated, %: C 54.05; H 4.54; N 12.61.

6-(4-Nitrophenyl)morpholin-3-one (9). To a solution of 0.50 g (2.74 mmol) of aminoalcohol **5** in 30 mL of acetonitrile was added at cooling to -10°C 0.28 g (0.38 mL, 2.74 mmol) of triethylamine, 0.31 g (0.22 mL, 2.74 mmol) of chloroacetyl chloride, and the reaction mixture was stirred at this temperature for 1 h and then at room temperature for 18 h. The reaction mixture was concentrated in a vacuum and subjected to column chromatography on silica gel (eluent hexane–ethyl acetate, 1 : 1). Yield of *N*-acyl derivative 0.57 g (80%), light-yellow oil. A solution of 0.50 g (1.93 mmol) of *N*-acyl derivative in 15 mL of *tert*-butanol was added dropwise to a solution of 0.54 g (4.83 mmol) of potassium *tert*-butylate in 10 mL of *tert*-butanol at 40°C . The reaction mixture was stirred at this temperature for 3 h. The solution was acidified to pH 2–3 by adding 2 M solution of HCl and then it was concentrated in a vacuum. The residue was dispersed in water (100 mL), the reaction product was extracted into ethyl acetate (4 × 70 mL), The extract was dried with sodium sulfate and concentrated in a vacuum. After column chromatography on silica gel (eluent ethyl acetate) we obtained 0.37 g of compound **9** (61% calculated for 2 stages), mp 230–233°C. IR spectrum, cm^{-1} : 3240, 3060, 1665, 1340, 1150. ^1H NMR spectrum (DMSO), δ , ppm: 3.35 d.d (1H, $\text{H}^{5\text{B}}$, $^2J_{5\text{A},5\text{B}}$ 8.0, $^3J_{6,5\text{B}}$ 7.0 Hz), 4.05 m (1H, $\text{H}^{5\text{A}}$), 4.33 m (2H, H^2), 4.71 d.d (1H, H^6 , $^3J_{6,5\text{A}}$ 8.1, $^3J_{6,5\text{B}}$ 7.0 Hz), 7.60 d (2H_{arom}, J 8.7 Hz), 8.00 br.s (1H, NH), 8.20 d (2H_{arom}, J 8.7 Hz). Found, %: C 54.31; H 4.60; N 12.33. $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_4$. Calculated, %: C 54.05; H 4.54; N 12.61.

6-(4-Nitrophenyl)morpholine-2,3-dione (10). To a solution of 0.50 g (2.74 mmol) of aminoalcohol **5** in 20 mL of ethanol was added 0.80 g (0.75 mL, 5.49 mmol) of diethyl oxalate. The reaction mixture was boiled for 8.5 h (TLC monitoring), evaporated in a vacuum, the residue was treated with ethyl ether, the formed crystals were filtered off. Yield 0.61 g (94%), mp 205–

208°C (decomp.). IR spectrum, cm^{-1} : 3240, 3060, 1750, 1690, 1440, 1120. ^1H NMR spectrum (DMSO), δ , ppm: 3.35 m (2H, H^5), 4.87 m (1H, H^6), 7.59 d (2H_{arom}, J 8.5 Hz), 8.19 d (2H_{arom}, J 8.5 Hz), 8.60 br.s (1H, NH). Found, %: C 50.71; H 3.60; N 12.07. $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_5$. Calculated, %: C 50.85; H 3.41; N 11.86.

Ethyl 2-[(bicyclo[2.2.1]hept-5-en-endo-2-yl)-methyl][2-hydroxy-2-(4-nitrophenyl)ethyl]amino}-2-oxoacetate (13) was obtained by the same procedure from 0.50 g (1.73 mmol) of aminoalcohol **12** [29] and 0.51 g (0.47 mL, 3.47 mmol) of diethyl oxalate. Yield 0.42 g (63%), mp 183–185°C (decomp.). IR spectrum, cm^{-1} : 3408, 3370, 3060, 1745, 1640, 1520, 1340, 1150, 725. ^1H NMR spectrum (CDCl_3), δ , ppm: 0.52 m (1H, $\text{H}^{3\text{n}}$), 1.23 d (1H, $\text{H}^{7\text{a}}$, $^2J_{7\text{s},7\text{a}}$ 8.1 Hz), 1.30 t (3H, CH_3 , 3J 7.5 Hz), 1.44 d (1H, $\text{H}^{7\text{s}}$), 1.85 m (1H, $\text{H}^{3\text{x}}$), 2.20 m (1H, H^2), 2.71 m (2H, H^8), 2.79 m (1H, H^4), 2.83 m (1H, H^1), 3.46 m (1H, NCH_2), 4.12 m (1H, NCH_2), 4.26 q (2H, CH_2 , 3J 7.0 Hz), 5.50 m (1H, OCH), 5.91 d.d (1H, H^5 , $^3J_{5,6}$ 5.6, $^3J_{4,5}$ 3.0 Hz), 6.16 d.d (1H, H^6 , $^3J_{5,6}$ 5.6, $^3J_{1,6}$ 3.0 Hz), 7.65 d (2H_{arom}, J 8.6 Hz), 8.19 d (2H_{arom}, J 8.6 Hz). Found, %: C 62.09; H 6.04; N 7.43. $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_6$. Calculated, %: C 61.85; H 6.23; N 7.21.

4-[(Bicyclo[2.2.1]hept-5-en-endo-2-yl)methyl]-6-(4-nitrophenyl)morpholine-2,3-dione (14). To a solution of 0.5 g (1.73 mmol) of aminoalcohol **12** [29] and 0.01 g (5 mol %) of 4-dimethylaminopyridine in dichloromethane (50 mL) at 0°C was added while stirring 0.70 g (0.96 mL, 6.92 mmol) of triethylamine. The mixture was stirred for 10 min, and then a solution of 0.33 g (2.60 mmol) of oxalyl chloride in dichloromethane (50 mL) was added dropwise within 4 h at 0°C . After adding the total amount of the reagent the reaction mixture was stirred additionally for 2 h at 0°C and warmed to the room temperature. The dichloromethane layer was washed with water (50 mL), dried with sodium sulfate, and evaporated in a vacuum. The reaction product was chromatographed on a column packed with silica gel (eluent hexane–ethyl acetate, 1 : 1). Yield 0.30 g (50%), mp 199–203°C. IR spectrum, cm^{-1} : 3030, 1750, 1690, 1515, 1340, 1150, 720. ^1H NMR spectrum (CDCl_3), δ , ppm: 0.52 m (1H, $\text{H}^{3\text{n}}$), 1.23 d (1H, $\text{H}^{7\text{a}}$, $^2J_{7\text{s},7\text{a}}$ 8.1 Hz), 1.44 d (1H, $\text{H}^{7\text{s}}$), 1.85 m (1H, $\text{H}^{3\text{x}}$), 2.20 m (1H, H^2), 2.70 m (2H, H^8), 2.81 m (1H, H^4), 2.83 m (1H, H^1), 3.46 m (1H, NCH_2), 4.12 m (1H, NCH_2), 5.60 m (1H, OCH), 5.93 m (1H, H^5), 6.19 m (1H, H^6), 7.65 d (2H_{arom}, J 8.6 Hz), 8.19 d (2H_{arom}, J 8.6 Hz). Found, %: C 62.99; H 5.04; N 7.91. $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_5$. Calculated, %: C 63.15; H 5.30; N 8.18.

3-{{Bicyclo[2.2.1]hept-5-en-endo-2-yl)methyl}-5-(4-nitrophenyl)-2-phenyloxazolidine (16). A slurry of 0.50 g (1.73 mmol) of aminoalcohol **12** [29], 0.18 g (1.73 mmol) of benzaldehyde, and 0.50 g of anhydrous sodium sulfate in 25 mL of anhydrous toluene was boiled for 6 h. The toluene solution was filtered while hot. On removing the solvent the reaction product was recrystallized from 2-propanol. Yield 0.48 g (73%), mp 133–135°C. IR spectrum, cm^{-1} : 3030, 1715, 1530, 1410, 1124, 720. ^1H NMR spectrum (CDCl_3), δ , ppm: 0.65 m (1H, H^{3n}), 1.30 d (1H, H^{7a} , $^2J_{7s,7a}$ 8.0 Hz), 1.43 d (1H, H^{7s}), 1.79 m (1H, H^{3x}), 2.10 m (1H, H^2), 2.85 m (1H, H^1), 2.95 m (1H, H^4), 3.21 m (2H, H^8), 4.41 m (2H, NCH_2), 5.15 m (1H, OCHCH_2), 5.70 m (1H, OCHN), 5.95 d (1H, H^5 , $^3J_{5,6}$ 6.0 Hz), 6.19 d (1H, H^6 , $^3J_{5,6}$ 6.0 Hz), 7.35 m (5 H_{arom}), 7.68 d (2 H_{arom} , J 8.7 Hz), 8.18 d (2 H_{arom} , J 8.7 Hz). Found, %: C 73.51; H 6.31; N 7.11. $\text{C}_{23}\text{H}_{24}\text{N}_2\text{O}_3$. Calculated, %: C 73.38; H 6.43; N 7.44.

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