Reactions of methoxycarbonylcarbene with 3-ethyl-2-phenyl- and 2,3-diphenyloxazolidines

*A. P. Molchanov,[a] A. V. Stepakov,[a] J. Kopf,[b] I. G. Zenkevich,[a] and R. R. Kostikov[a]**

[a]St. Petersburg State University,
2 Universitetsky prosp., 198504 St. Petersburg, Russian Federation.
E-mail: rkost@rk1198.spb.edu
[b]Institute of Inorganic Chemistry,
*6 Martin Luther King Platz, D-20146 Hamburg, Germany.**
E-mail: kopf@xray.chemie.uni-hamburg.de

The reactions of methoxycarbonylcarbene, which was generated by catalytic thermal decomposition of methyl diazoacetate, with 3-ethyl-2-phenyl- or 2,3-diphenyloxazolidines resulted in the insertion of the former predominantly at the C—N bond of the oxazolidine ring to produce substituted esters of morpholine-3-carboxylic acid.

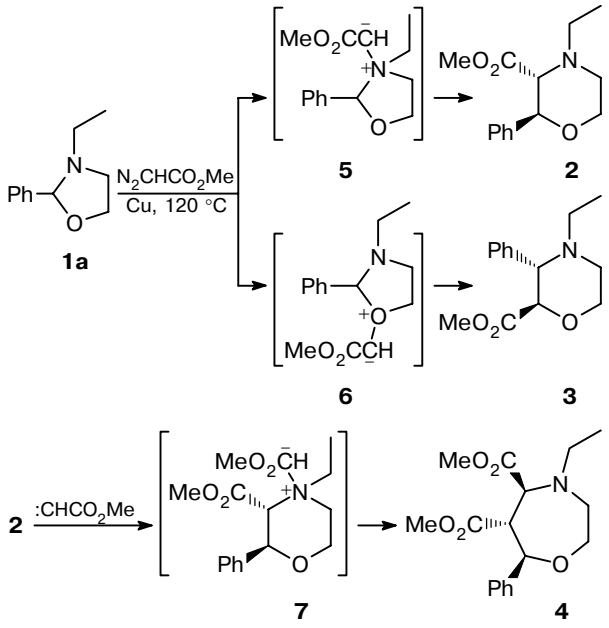
Key words: carbenes, diazo compounds, oxazolidines, insertion reactions.

Previously,[1,2] it has been noted that the reactions of ethoxycarbonylcarbene with 2-substituted 1,3-dioxolanes resulted in the insertion of the carbene at the C—O bond of the dioxolane to form esters of 3-substituted 1,4-dioxane-2-carboxylic acids.

In the present study, we established that the reaction of methoxycarbonylcarbene, which was generated by catalytic thermal decomposition of methyl diazoacetate, (120 °C, copper bronze), with 3-ethyl-2-phenyloxazolidine (**1a**) afforded a complex mixture of products. According to the GLC data, this mixture contained dimethyl maleate, dimethyl fumarate, methyl cinnamate, methyl 4-ethyl-2-phenylmorpholine-3-carboxylate (**2**), methyl 4-ethyl-3-phenylmorpholine-2-carboxylate (**3**), and dimethyl 4-ethyl-7-phenyl-1,4-oxazepane-5,6-dicarboxylate (**4**) (Scheme 1). The ratio of esters **2**, **3**, and **4** in the resulting reaction mixture depended on the ratio of the starting reagents (diazo ester : oxazolidine). The ratio of the esters changed from 3 : 1 : 0.3 to 0.6 : 1 : 1.3 as the above-mentioned ratio of the reagents was increased from 0.7 : 1 to 1.8 : 1. This result agrees with the fact that ester **4** was generated from ester **2**. It should be noted that attempts to obtain products of the insertion of carbene at the C—N or C—O bond with the use of other catalysts for decomposition of diazoacetate ($Rh_2(OAc)_4$, benzene, 80 °C; $Pd(OAc)_2$, CH_2Cl_2, *ca.* 20 °C; or $CuSO_4$, benzene, 80 °C) failed.

The reaction mixture was separated by column chromatography. After chromatography, an inseparable mixture of monoesters **2** and **3** was obtained. The structures of these esters were confirmed by the mass spectra and the chromatographic retention indices (I_r) on a standard nonpolar phase. In the latter case, the assignment of

Scheme 1



isomers **2** and **3** was made based on the correlation between the order in which chromatographic elution of the isomers occurred and their intramolecular dynamic parameters E_{dyn} (the sums of the vibrational and rotational energies).[3] The higher energy E_{dyn} for ester **3** (78.2±1.8 kcal mol^{-1}) compared to that for ester **2** (76.3±1.5 kcal mol^{-1}) corresponds to the lower I_r value (1711±2 and 1736±2, respectively). The ^{1}H NMR spectrum of a mixture of compounds **2** and **3** has doublet signals (δ 4.19 and 4.98; δ 4.31 and 4.79, respectively; J = 7 Hz for all siganls) belonging to the methine

* Institut für anorganische Chemie, 6 Martin Luther King Platz, D-20146 Hamburg, Germany.

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Table 1. Selected bond lengths (*d*) in compound **4**

Bond	*d*/Å	Bond	*d*/Å
O(1)—C(7)	1.417(9)	C(71)—C(76)	1.391(10)
O(1)—C(2)	1.436(5)	C(72)—C(73)	1.389(3)
O(51)—C(51)	1.204(9)	C(41)—C(42)	1.516(8)
O(52)—C(51)	1.332(6)	C(74)—C(75)	1.390(6)
O(52)—C(52)	1.453(8)	C(75)—C(76)	1.387(3)
O(62)—C(61)	1.341(9)	C(5)—C(51)	1.534(8)
O(62)—C(62)	1.443(7)	C(6)—C(61)	1.520(8)
N(4)—C(3)	1.462(10)	C(6)—C(7)	1.553(10)
N(4)—C(5)	1.462(8)	C(7)—C(71)	1.518(2)
N(4)—C(41)	1.476(10)	C(73)—C(74)	1.383(10)
C(2)—C(3)	1.513(10)	C(5)—C(6)	1.553(13)
C(71)—C(72)	1.390(5)	O(61)—C(61)	1.207(3)

protons at the C(2) and C(3) atoms of esters **2** and **3**, respectively. The spin-spin coupling constants are indicative of the *trans* arrangement of the substituents at the adjacent C atoms.

Diester **4** was isolated in the individual state in 30% yields (I_r = 2077±3). Its composition and structure were established based on the results of elemental analysis,

Table 2. Selected bond angles (ω) in compound **4**

Angle	ω/deg
C(7)—O(1)—C(2)	113.57(11)
C(51)—O(52)—C(52)	115.32(12)
C(61)—O(62)—C(62)	115.41(11)
C(3)—N(4)—C(5)	115.58(11)
C(61)—C(6)—C(5)	107.37(11)
C(5)—N(4)—C(41)	113.30(12)
O(1)—C(2)—C(3)	111.55(12)
O(61)—C(61)—O(62)	123.25(13)
C(61)—C(6)—C(7)	111.90(11)
C(7)—C(6)—C(5)	115.03(11)
C(3)—N(4)—C(41)	110.87(11)
O(61)—C(61)—C(6)	126.25(13)
O(62)—C(61)—C(6)	110.49(11)
N(4)—C(3)—C(2)	110.49(11)
N(4)—C(41)—C(42)	113.23(13)
O(1)—C(7)—C(6)	112.13(11)
O(1)—C(7)—C(71)	108.38(11)
C(71)—C(7)—C(6)	112.72(12)
C(72)—C(71)—C(76)	119.00(13)
C(72)—C(71)—C(7)	121.54(13)
C(76)—C(71)—C(7)	119.46(13)
C(73)—C(72)—C(71)	120.14(14)
N(4)—C(5)—C(51)	114.78(12)
N(4)—C(5)—C(6)	112.35(11)
C(72)—C(73)—H(73)	119.60(16)
C(73)—C(74)—C(75)	119.18(15)
C(74)—C(73)—C(72)	120.82(15)
C(51)—C(5)—C(6)	113.18(11)
O(51)—C(51)—O(52)	123.91(14)
O(51)—C(51)—C(5)	124.29(13)
C(76)—C(75)—C(74)	120.18(15)
C(75)—C(76)—C(71)	120.67(14)
O(52)—C(51)—C(5)	111.69(12)

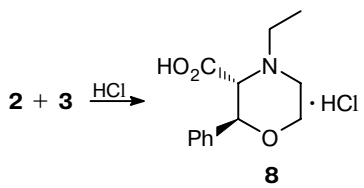
Table 3. Selected dihedral angles (α) in compound **4**

Angle	α/deg
O(1)—C(2)—C(3)—N(4)	−65.8(2)
C(3)—N(4)—C(5)—C(6)	−79.8(1)
N(4)—C(5)—C(6)—C(7)	77.9(1)
N(4)—C(5)—C(6)—C(61)	−47.4(1)
N(4)—C(5)—C(51)—O(52)	−167.5(1)
C(5)—C(6)—C(7)—O(1)	−13.8(2)
C(5)—C(6)—C(7)—C(71)	108.8(1)
C(6)—C(7)—O(1)—C(2)	−66.0(1)
C(7)—O(1)—C(2)—C(3)	99.0(1)
C(41)—N(4)—C(5)—C(51)	−78.1(1)
C(51)—C(5)—C(6)—C(61)	−179.0(1)
C(52)—O(52)—C(51)—C(5)	179.4(1)
O(52)—C(51)—C(5)—N(4)	−167.5(1)
C(61)—C(6)—C(7)—C(71)	−128.0(1)
C(62)—O(62)—C(61)—O(61)	−4.6(2)
C(72)—C(71)—C(7)—C(6)	−122.4(1)

spectral data, and X-ray diffraction analysis (Tables 1—3). The ^{1}H NMR spectrum of diester **4** has doublet signals for the methine protons at the C(5) and C(7) atoms and a triplet signal for the methine H(6) proton (δ 4.16, 5.17, and 3.59, respectively; J = 7 Hz for all signals). The spin-spin coupling constants are indicative of the *trans* arrangement of the substituents at the adjacent C atoms. The assignments of the signals for the protons at the C(5) and C(7) atoms in compound **4** (as well as for analogous protons in substituted morpholines) were made based on comparison with the results of calculations using additive schemes[4] according to which the protons at the C(5) and C(7) atoms should give signals at δ *ca.* 4 and *ca.* 5, respectively. In all cases, only the relative configurations of the substituents in the ring were established. The structure of diester **4** was additionally confirmed by X-ray diffraction analysis (Fig. 1).

Hydrolysis of a fraction containing monoesters **2** and **3** in the presence of HCl afforded hydrochloride of 4-ethyl-2-phenylmorpholine-3-carboxylic acid (**8**) (Scheme 2). The composition and the structure of acid **8** were established based on the results of elemental analysis and spectral data. The ^{1}H NMR spectrum of **8** has a signal for the proton of the COOH group (δ 12.22), doublet signals for the methine protons at the C(2) and C(3) atoms (δ 4.65 and 4.18, respectively; J = 10 Hz for both signals), and signals for the protons of the methylene and ethyl groups and the aromatic fragment.

Scheme 2



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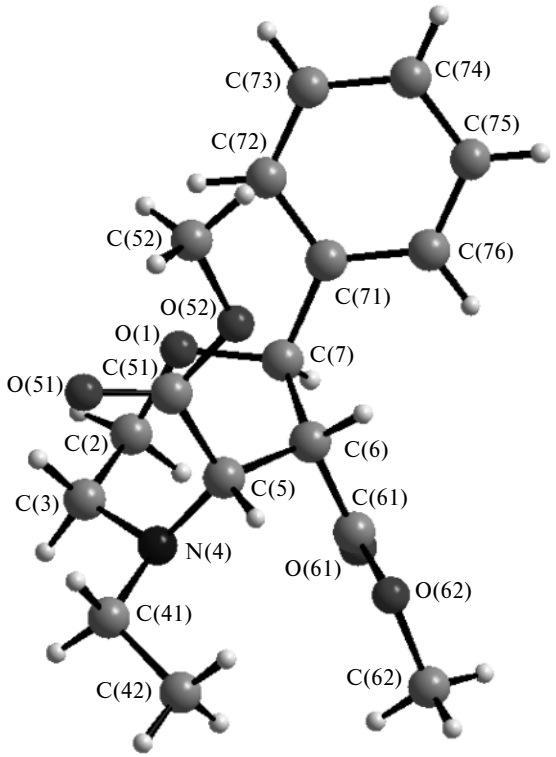
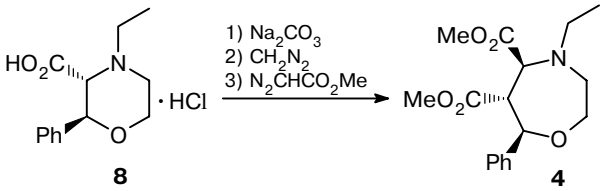


Fig. 1. Overall view of molecule **4** according to the results of X-ray diffraction study.

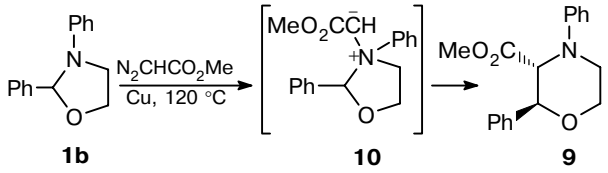
The spin-spin coupling constants for the protons at the C(2) and C(3) atoms are indicative of their *trans* arrangement. In addition, hydrochloride salt **8** was converted into free amino acid and was subjected to esterification with diazomethane. The reaction of the resulting ester with methoxycarbonylcarbene afforded ester whose spectral data are identical with those of diester **4** isolated previously (Scheme 3).

Scheme 3



The reaction of 2,3-diphenyloxazolidine (**1b**) with methoxycarbonylcarbene, which was generated under the same conditions, was accompanied by substantial resinification of the reaction mixture. Column chromatography of this mixture made it possible to isolate only methyl 2,4-diphenylmorpholine-3-carboxylate (**9**) in 13% yield (Scheme 4). The composition and the structure of ester **9** were established based on the results of elemental analysis and spectral data. The ^{1}H NMR spectrum of ester **9** has doublet signals for the methine protons at the C(2) and C(3) atoms (δ 5.03 and 4.32, respectively; J = 6 Hz for both signals).

Scheme 4



Hence, the results obtained in the present study provide evidence that methoxycarbonylcarbene reacted with oxazolidines **1a,b** to form *N*-ylide **5**, *N*-ylide **10**, or *O*-ylide **6**, which underwent the Stevens rearrangement with the ring expansion to produce morpholine derivatives. The results of quantum-chemical MNDO/1 calculations for ylides **5** and **6** confirmed that the formation of *N*-ylide **5** is thermodynamically more favorable compared to *O*-ylide **6** (by 3—5 kcal mol^{-1}). The subsequent reaction of ester **2** with methoxycarbonylcarbene also afforded *N*-ylide **7**, which was rearranged into diester **4**.

Experimental

The IR spectra were recorded on a UR-20 spectrophotometer (2% solutions in $CHCl_3$ for compounds **4** and **9**; a KBr pellet for compound **8**). The ^{1}H NMR spectra were measured on a Bruker AM-300 instrument (300 MHz). The resulting compounds were tested for purity and the reaction mixtures were analyzed by TLC on Silufol UV-254 plates and by GLC on a Tsvet-126 chromatograph equipped with a flame ionization detector (nitrogen as the carrier gas; a 1500×3-mm glass column; 5% SE-30 on Chromaton N-AW as the stationary phase). The retention indices (I_r) were determined on a Biokhrom-1 chromatograph equipped with a flame ionization detector and a quartz capillary column (25 m × 0.20 mm) with the OV-101 phase (4300 t.p. m^{-1}; t.p. are theoretical plates) in the temperature-programming mode from 60 to 240 °C at a rate of 6 deg min^{-1}. To determine the indices I_r, a sample (0.6 μL) was dosed together with a mixture of the reference C_6—C_{18} *n*-alkanes. The parameters of the chromatographic peaks were recorded using a TR 2213 integrator. The linear-logarithmic I_r indices were calculated on a programmed CASIO PB100 microcalculator using a program available in the manual.[5]

The mass spectra of the products obtained in the reaction of 3-ethyl-2-phenyloxazolidine with methoxycarbonylcarbene were measured on an LKB-2091 GLC-mass spectrometer equipped with a glass packed column (3 m × 2 mm; 3 % SE-30 on Chromaton-W-HMDS) in the temperature-programming mode from 60 to 250 °C at a rate of 6 deg min^{-1}. The energy of ionizing electrons was 70 eV, the current of emission was 25 μA, the accelerating voltage was 3.5 kV, and the temperature of the ion source was 280 °C.

The intramolecular energies were calculated by molecular dynamics using the HyperChem 5.1 program package with the following parameters for simulation: the temperature was 300 K, the energy was averaged with a step of 0.0005 ps, the relax-

ation time was 0.1 ps, the total time of simulation (no larger than 20 ps) was chosen depending on the rate of the attainment of the reasonable accuracy (~2%) of the estimation of the E_{dyn} parameters. The initial period of simulation, which was equivalent to heating of the molecule to a given temperature (~1.5 ps), was excluded and calculations were resumed using the command RESTART. The molecular geometry was initially optimized using the MM+ and AM1 methods (simultaneously, the relative thermodynamical stabilities of the isomers were estimated).

Methyl diazoacetate,[6] 3-ethyl-2-phenyloxazolidine (**1a**),[7] and 2,3-diphenyloxazolidine (**1b**)[7] were synthesized according to known procedures.

The reaction of 3-ethyl-2-phenyloxazolidine (1a) with methoxycarbonylcarbene. Methyl diazoacetate (1.5 g, 15 mmol) was added with intense stirring to a mixture of oxazolidine **1a** (0.8 g, 4.5 mmol) and copper bronze (0.15 g) in octane (15 mL) at 120 °C during 3 h. The reaction mixture was stirred at this temperature for 30 min and then cooled. The solvent was distilled off *in vacuo*. According to the GLC data, the mixture contained dimethyl maleate, dimethyl fumarate, methyl cinnamate, methyl morpholinecarboxylates **2** and **3**, and methyl oxazepanecarboxylate **4**.

Separation of the reaction mixture on a column with silica gel using a 1 : 1 hexane—Et_2O mixture as the eluent afforded a fraction (0.15 g, 13%), which contained primarily a mixture of methyl *trans*-2-phenylmorpholine-3-carboxylate (**2**) (I_r = 1736) and methyl *trans*-3-phenylmorpholine-2-carboxylate (**3**) (I_r = 1711) as well as the minor components with I_r = 1726 and 1756. The latter were, apparently, geometric isomers of the above-mentioned regioisomers. MS, *m/z* (I_{rel} (%)): compound **2** — 249 $[M]^+$ (0.6), 191 (14), 190 $[M - CO_2Me]^+$ (100), 143 (6), 128 (11), 115 (7), 105 (8), 91 (8), 84 (8), 83 (11), 77 (11), 59 (7), 56 $[CHNEt]^+$ (68), 55 (7), 54 (5); compound **3** — 249 $[M]^+$ (19), 191 (5), 190 $[M - CO_2Me]^+$ (33), 162 (9), 161 (54), 160 $[M - CO_2Me - CHOH]^+$ (94), 146 (14), 133 $[PhCHNEt]^+$ (35), 132 $[PhCNEt]^+$ (100), 131 (13), 130 (5), 121 (6), 119 (7), 118 $[PhCHNCH_2]^+$ (66), 117 (5), 105 (18), 104 $[PhCNH]^+$ (37), 103 (12), 100 (5), 91 (36), 78 (6), 77 (15), 70 (6), 65 (6), 59 (7), 55 (29).

The solvent was evaporated from the fraction (0.38 g) containing diester **4** and the residue was recrystallized from 95% EtOH to isolate **dimethyl (5*S*,6*R*,7*R*)-4-ethyl-7-phenyl-1,4-oxazepane-5,6-dicarboxylate (4)** in a yield of 0.29 g (30%), m.p. 61—62 °C. Found (%): C, 63.56; H, 7.02; N, 4.18. $C_{17}H_{23}NO_5$. Calculated (%): C, 63.54; H, 7.21; N, 4.36. IR, ν/cm^{-1}: 2960 (C—H), 1730 (C=O), 1600, 1520, 1450, 1360, 1250, 1120, 1080, 1010. ^{1}H NMR ($CDCl_3$), δ: 1.03 (t, 3 H, Me, *J* = 7 Hz); 2.72 (dt, 1 H, H(3), *J* = 14 Hz, *J* = 3 Hz); 2.85 (m, 2 H, CH_2); 3.37 (ddd, 1 H, H(3), *J* = 14 Hz, *J* = 12 Hz, *J* = 3 Hz); 3.44 and 3.62 (both s, 3 H each, 2 OMe); 3.59 (t, 1 H, H(6), *J* = 7 Hz); 3.73 (td, 1 H, H(2), *J* = 12 Hz, *J* = 3 Hz); 4.02 (dt, 1 H, H(2), *J* = 12 Hz, *J* = 3 Hz); 4.16 and 5.17 (both d, 1 H each, H(5), H(7), *J* = 7 Hz); 7.24—7.33 (m, 5 H, Ar). ^{13}C NMR, δ: 14.1 (q), 50.6 (t), 51.4 (q), 51.7 (q), 57.3 (d), 65.1 (d), 72.0 (t), 80.9 (d), 126.1 (d), 127.3 (d), 128.0 (d), 142.3 (s), 172.4 (s), 172.7 (s). MS, *m/z* (I_{rel} (%)): 321 $[M]^+$ (1.6), 263 (11), 262 $[M - CO_2Me]^+$ (63), 234 (11), 232 (14), 200 (5), 188 (7), 156 (10), 155 $[M - CO_2Me - PhCHOH]^+$ (100), 131 (8), 129 (17), 128 (14), 116 (10), 115 (14), 104 (5), 100 (29), 98 (17), 97 (5), 96 (10), 91 (7), 82 (5), 77 (12), 72 (11), 70 (5), 69 (6), 68 (6), 59 (28), 58 (5), 56 (24).

Hydrochloride of *trans*-4-ethyl-2-phenylmorpholine-3-carboxylic acid (8). A 10% HCl solution (10 mL) was added to a fraction (0.14 g) containing a mixture of monoesters **2** and **3**. The mixture was kept at *ca*. 20 °C for 48 h. Water was removed by azeotropic distillation with benzene. Then dry ether (8 mL) and several drops of MeOH were added to the residue until crystallization started. The crystalline precipitate was filtered off and washed with ether. Salt **8** was obtained in a yield of 47 mg (6%), m.p. 234—236 °C (decomp.). Found (%): C, 57.18; H, 6.72; N, 5.11. $C_{13}H_{18}ClNO_3$. Calculated (%): C, 57.46; H, 6.68; N, 5.15. IR, ν/cm^{-1}: 3240 (N—H), 2960, 2680, 1750 (C=O), 1610, 1490, 1460, 1370, 1250, 1130, 1070, 1010, 980, 950. ^{1}H NMR (DMSO-d_6), δ: 1.14 (t, 3 H, Me, *J* = 7 Hz); 2.85 (m, 2 H, CH_2); 3.35 and 3.61 (both m, 1 H each, NCH_2); 4.18 (d, 1 H, H(3), *J* = 10 Hz); 4.44 (m, 2 H, OCH_2); 4.65 (d, 1 H, H(2), *J* = 10 Hz); 7.46—7.81 (m, 5 H, Ar); 12.22 and 13.20 (both br.s, 1 H each, NH, OH).

Diester 4 from salt 8. A 1% Na_2CO_3 solution was added dropwise to a solution of salt **8** (30 mg, 0.1 mmol) in water (1.5 mL) until a white amorphous precipitate formed. The precipitate was extracted with ether. The extract was dried with $MgSO_4$, ether was evaporated to the volume of *ca*. 2 mL, and an ethereal solution of diazomethane was added dropwise until liberation of nitrogen ceased and the solution turned pale-yellow. The solvent was evaporated at *ca*. 20 °C and then octane (3 mL) and copper bronze (10 mg) were added to the residue. The reaction mixture was heated to 120 °C and methyl diazoacetate was slowly added until the initial compound was consumed (TLC control). The mixture was cooled, the solvent was distilled off *in vacuo*, and the residue was separated on a column with silica gel using a 1 : 1 hexane—Et_2O mixture as the eluent. Diester **4** was isolated in a yield of 5.8 mg (16%). Its NMR spectral data were identical with those of the specimen obtained previously.

Methyl *trans*-2,4-diphenylmorpholine-3-carboxylate (9). Methyl diazoacetate (0.8 g, 8 mmol) was added with intense stirring to a mixture of oxazolidine **1b** (0.45 g, 2 mmol) and copper bronze (0.1 g) in octane (10 mL) at 120 °C for 2.5 h. Then the reaction mixture was stirred at this temperature for 45 min and cooled. The solvent was distilled off *in vacuo*. The residue was separated on a column with silica gel using a 2 : 1 hexane—Et_2O mixture as the eluent. After recrystallization from 95% EtOH, ester **9** was obtained in a yield of 52 mg (13%), m.p. 106—107 °C. Found (%): C, 72.78; H, 6.29; N, 4.53. $C_{18}H_{19}NO_3$. Calculated (%): C, 72.71; H, 6.44; N, 4.71. IR, ν/cm^{-1}: 2950 (C—H), 1740 (C=O), 1600, 1510, 1460, 1380, 1350, 1250, 1120, 1030, 980, 940. ^{1}H NMR ($CDCl_3$), δ: 3.21 and 3.56 (both m, 1 H each, NCH_2); 3.46 (s, 3 H, OMe); 3.99 (t, 2 H, OCH_2, *J* = 5 Hz); 4.32 and 5.03 (both d, 1 H each, H(3), H(2), *J* = 6 Hz); 6.98—7.08 and 7.28—7.48 (both m, 3 H and 7 H, Ar).

X-ray diffraction analysis of compound 4. Single crystals of diester **4** were obtained by crystallization from EtOH. X-ray diffraction data were collected on a Hilger & Watts-Y290 diffractometer (graphite monochromator, λ(Cu-Kα) = 1.54178 Å, 173 K, θ/2θ scanning technique) using a crystal of dimensions 0.8×0.5×0.4 mm. The crystals are triclinic, *a* = 6.9538(9) Å, *b* = 10.0732(12) Å, *c* = 12.256(2) Å, α = 75.55(1)°, β = 87.45(1)°, γ = 83.63(1)°, *V* = 819.35(20) Å^3, *Z* = 2, M = 321.36, space group *P*1, d_{calc} = 1.302 g cm^{-3}, *F*(000) = 344.0, μ = 0.790, R_{int} = 3441, the number of reflections with $I \geq 2\sigma(I)$ was 3378, the number of the parameters in the refinement was 235, $R_1(I \geq 2\sigma(I))$ = 0.071, wR_2 = 0.1251. The absorption correction was applied using experimental azimuth scanning curves (T_{min}/T_{max}). The structure was solved by the direct method. The positional and thermal parameters of the nonhydrogen atoms were refined by

the full-matrix least-squares method first isotropically and then anisotropically. All calculations were carried out using the SHELXL-97 program package.

The principal bond lengths, bond angles, and dihedral angles are given in Tables 1—3, respectively. The complete crystallographic parameters for compound **4** were deposited with the Cambridge Structural Database (CCDC-163325).

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