UV spectrum, λ_{max}: 283 and 342 nm. IR spectrum: 1790 (C=O) and 1660 cm^{-1}. Found: C 74.8; H 3.9; N 10.4%. $C_{17}H_{10}N_2O_2$. Calculated: C 74.5; H 3.6; N 10.2%. Compounds XIIIb, c, XV, and XVII (Table 2) were similarly obtained.

5-Azaquinophthalone [2-(2-Quinolyl)-5-azaindan-1,3-dione] (XIXa). A mixture of 1.15 g (4.2 mmole) of XIIIa, 46 ml of methanol, and a solution of sodium methoxide (0.1 g of Na was dissolved in 12 ml of methanol) was heated at 50-60°C for 1 h, after which the precipitate was removed by filtration to give 0.68 g of salt XVIIIa, which was a red infusible substance. The salt was refluxed with 120 ml of water to give 0.62 g (54%) of azaindandione XIXa. Successive recrystallization of the latter from water and benzene gave a bright-yellow product with mp 268°C and R_f 0.68. UV spectrum, λ_{max}: 291, 316, 454, and 478 nm. IR spectrum: 1685 cm^{-1} (C=O). Found: C 74.3; H 3.7; N 10.6%. $C_{17}H_{10}N_2O_2$. Calculated: C 74.5; H 3.6; N 10.2%. Compounds XIXb-e (Table 2) were similarly obtained.

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MACROHETEROCYCLES.

12.* SYNTHESIS AND PROPERTIES OF MACROCYCLIC TETRAAMIDES

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The N,N'-bis(methoxycarbonylmethyl)diamides of the corresponding acids were obtained by the reaction of glycine methyl ester hydrochloride with oxalic, succinic, adipic, diglycolic, and triglycolic acid dichlorides. Fourteen new macrocyclic tetraamides were obtained by the reaction of these diamides with various diamines. The structures of the synthesized compounds were proved by means of their IR, PMR, and mass spectra.

Polyfunctional macroheterocycles display interesting complexing properties and biological activity [2]. The least amount of study in this respect has been devoted to macrocyclic polyamides.

The synthesis of these compounds is generally accomplished by acylation of diamines (or polyamines with partially protected amino groups) by the corresponding dicarboxylic acid

*See [1] for Communication 11.

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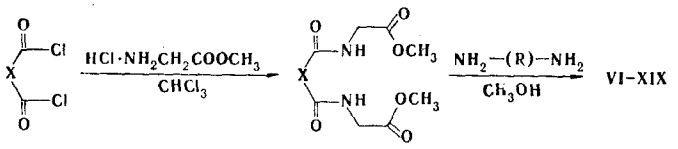
derivatives [3, 4]. In most cases this reaction is carried out under high-dilution conditions, and this creates certain experimental difficulties.

In a continuation of our research on the synthesis, structure, and properties of macroheterocycles [5-7] we developed a convenient method for the preparation of macrocyclic tetraamides from glycine that does not require high dilution of the reagents.

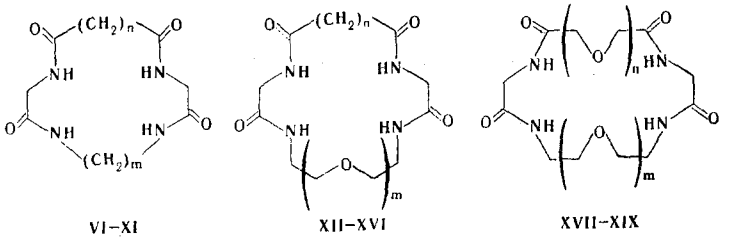
By acylation of glycine methyl ester hydrochloride with the dichlorides of the corresponding acids we obtained the N,N'-bis(methoxycarbonylmethyl-diamides of oxalic (I), succinic (II), adipic (III), diglycolic (IV), and triglycolic (V) acids.



I-V

I X = O; II X = $(CH_2)_2$; III X = $(CH_2)_4$; IV X = CH_2OCH_2; V X = $(CH_2OCH_2)_2$

Intense bands of stretching vibrations of ester and amide carbonyl groups at 1735-1755 and 1670-1680 cm^{-1}, respectively, as well as bands of NH stretching vibrations at 3220-3460 cm^{-1}, are present in the IR spectra of I-V. In addition to these bands, bands of an ether bond at 1130-1140 cm^{-1} are observed in the spectra of IV and V. The signal of the methylene group of glycine shows up in the form of a doublet at 3.87 ± 0.11 ppm in the PMR spectra of I-V in trifluoroacetic acid. The signal of an α-methylene group of an acyl fragment is observed at 2.43 ± 0.20 ppm for II and III and at 3.90 ± 0.10 ppm for IV and V. Singlet signals of the OCH_3 groups in I-V and of the OCH_2 group in V appear at 3.35 ± 0.05 ppm (see the experimental section).



VI n=2, m=2; VII n=2, m=4; VIII n=2, m=6; IX n=4, m=2; X n=4, m=4; XI n=4, m=6; XII n=0, m=2; XIII n=2, m=1; XIV n=2, m=2; XV n=4, m=1; XVI n=4, m=2; XVII n=1, m=1; XVIII n=2, m=1; XIX n=2, m=2

New macrocyclic tetraamides VI-XIX were obtained by reaction of amido esters I-V with the corresponding diamines in methanol. The reaction was carried out at 0.3 mole/liter concentrations of the reactants. A decrease in the concentrations of the reagents and an increase in the reaction temperature did not have a substantial effect on the yields of the desired products. However, the use of the N,N'-bis(ethoxycarbonylmethyl)diamides of oxalic and succinic acids results in appreciably lower yields of the cyclic tetraamides. Cyclic tetraamides VI-XIX were isolated and purified by liquid chromatography and (or) crystallization. Tetraamides VI-XIX are colorless crystalline substances; their properties are described in Table 1. The molecular masses of these compounds were determined by elementary analysis and IR and PMR spectroscopy. The crystal and molecular structures of cyclic tetraamides VI and VII were established by x-ray diffraction analysis [8, 9].

The IR spectra of solid samples of VI-XIX contain intense bands of stretching vibrations of a carbonyl group at 1640-1690 cm^{-1} and bands of deformation vibrations of an NH group at 1510-1570 cm^{-1}. The stretching vibrations of the NH group show up in the form of a number of bands at 3070-3460 cm^{-1}. The spectra of XII-XIX also contain bands of a C—O—C bond at 1100-1135 cm^{-1}.

X-ray diffraction analysis showed the presence of one transannular N—H···O=C hydrogen bond in tetraamide VI and two in tetraamide VII; in VI the remaining amide groups participate in the formation of intermolecular H bonds, whereas in VII one carbonyl group is unassociated [9]. This makes it possible to assume that the narrow bands at 3370 cm^{-1} in the spectra of tetraamides VI and VII are due to vibrations of NH groups that participate in the formation of intramolecular H bonds. The presence of a doublet of bands of a carbonyl

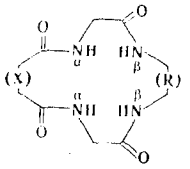
TABLE 1. Characteristics of Macrocyclic Tetraamides VI-XIX

Compound	mp, °C	IR spectrum, cm^{-1}				Found, %			Empirical formula	Calc., %			M^+	Yield, %
		νN—H	νC=O	δN—H	νC—O—C	C	H	N		C	H	N		
VI	348—349	3070 3260 3310 3370	1645 1660	1550	—	46,7	6,5	21,7	$C_{10}H_{16}N_4O_4$	46,9	6,3	21,9	256	15
VII	261—262	3070 3260 3300 3365	 1645 1680	1545	—	50,3	7,5	19,5	$C_{12}H_{20}N_4O_4$	50,7	7,0	19,7	284	20
VIII	244—245	3080 3220 3270 3330	1640 1660 1680	1545 1565	—	53,6	8,0	17,8	$C_{14}H_{24}N_4O_4$	53,8	7,7	17,9	312	15
IX	239—240	3090 3280 3320 3400	1645 1675	1530 1560	—	50,4	7,3	19,5	$C_{12}H_{20}N_4O_4$	50,7	7,0	19,7	284	10
X	280—281	3080 3290 3320 3400	1645 1665	1520 1545	—	53,5	7,9	17,8	$C_{14}H_{24}N_4O_4$	53,8	7,7	17,9	312	15
XI	214—215	3090 3320 3370	1645 1680	1530 1565	—	56,3	7,9	16,3	$C_{16}H_{28}N_4O_4$	56,5	8,2	16,5	340	12
XII	275—276	3080 3290 3390 3420	1665 1680	1525 1555	1100	45,5	6,5	17,6	$C_{12}H_{20}N_4O_6$	45,6	6,3	17,7	316	8
XIII	215—216	3080 3260 3300 3400 3490	 1645 1665	 1540 1570	1100 1115	47,8	6,9	18,6	$C_{12}H_{20}N_4O_5$	48,0	6,7	18,7	300	16
XIV	209—210	3100 3280 3320 3420	1645 1670 1690	1510 1540 1575	1100	48,6	7,5	16,1	$C_{14}H_{24}N_4O_6$	48,8	7,0	16,3	344	12
XV	223—224	3070 3270 3300 3315 3360	1655 1675	1535 1550	1135	51,0	7,5	16,9	$C_{14}H_{24}N_4O_5$	51,2	7,3	17,1	328	14
XVI	195—196	3100 3280 3290 3310	1650 1675	 1565	1120	51,4	7,8	14,9	$C_{16}H_{28}N_4O_6$	51,6	7,5	15,0	372	12
XVII	252—253	3090 3310 3390 3460	1640 1655 1685	1540 1550	1135	45,4	6,6	17,5	$C_{12}H_{20}N_4O_6$	45,6	6,3	17,7	316	8
XVIII	202—203	3100 3300 3370 3400	1650 1660 1690	1545 1570 1545	1115 1135	46,4	7,0	15,4	$C_{14}H_{24}N_4O_7$	46,7	6,7	15,6	360	9
XIX	189—190	3100 3290 3390 3420	1655 1660 1685	 1560 	1100 1135	47,4	7,1	13,7	$C_{16}H_{28}N_4O_8$	47,5	6,9	13,9	404	10

group in the spectrum of tetraamide VI is evidently associated with the difference in the energies of the intra- and intermolecular H bonds. The band at 1680 cm^{-1} in the spectrum of tetraamide VII can be assigned to the vibrations of an unassociated carbonyl group. Similar spectral principles are observed in the IR spectra of all cyclic tetraamides VI-XIX (Table 1).

The PMR spectra of tetramides VI-XIX are similar to the spectra of starting amido esters I-V (Table 2). The signal of the methylene group of glycine shows up in the form of a doublet at 3.90 ± 0.11 ppm ($^3J_{NHCH_2}$ = 3.5-6.5 Hz). The α-methylene groups of the acid

TABLE 2. PMR Spectra of Tetraamides VI-XIX



Compound	Chemical shifts, δ, ppm (CF_3COOH)
VI	7,71 (2H, t, $N^{\alpha}H$); 7,31 (2H s, $N^{\beta}H$); 3,82 (4H, d, $N^{\alpha}CH_2$); 3,21 (4H, s, $N^{\beta}CH_2$); 2,62 (4H, s, $N^{\alpha}COCH_2$)
VII	7,60 (4H, s, $N^{\alpha}H$, $N^{\beta}H$); 3,89 (4H, d, $N^{\alpha}CH_2$); 3,22 (4H, m, $N^{\beta}CH_2$); 2,69 (4H, s, $N^{\alpha}COCH_2$); 1,41 (4H, m, CH_2)
VIII	8,00 (2H, s, $N^{\alpha}H$); 7,52 (2H, s, $N^{\beta}H$); 3,97 (4H, d, $N^{\alpha}CH_2$); 3,29 (4H, m, $N^{\beta}CH_2$); 2,70 (4H, s, $N^{\alpha}COCH_2$); 1,40 (8H, m, CH_2)
IX	7,60 (4H, s, $N^{\alpha}H$, $N^{\beta}H$); 3,81 (4H, d, $N^{\alpha}CH_2$); 3,23 (4H, m, $N^{\beta}CH_2$); 2,19 (4H, m $N^{\alpha}COCH_2$); 1,40 (4H, m, CH_2)
X	7,60 (4H, s, $N^{\alpha}H$, $N^{\beta}H$); 3,80 (4H, d, $N^{\alpha}CH_2$); 3,11 (4H, m, $N^{\beta}CH_2$); 2,20 (4H, m, $N^{\alpha}COCH_2$); 1,31 (8H, m, CH_2)
XI	7,61 (4H, s, $N^{\alpha}H$, $N^{\beta}H$); 3,80 (4H, d, $N^{\alpha}CH_2$); 3,12 (4H, m, $N^{\beta}CH_2$); 2,21 (4H, m, $N^{\alpha}COCH_2$); 1,25 (12H, m, CH_2)
XII	8,50 (2H, s, $N^{\alpha}H$); 7,31 (2H, s, $N^{\beta}H$); 3,98 (4H d, $N^{\alpha}CH_2$); 3,33 (12H, d, $N^{\beta}CH_2$, CH_2OCH_2)
XIII	7,38 (2H, s, $N^{\alpha}H$); 7,29 (2H, s, $N^{\beta}H$); 3,81 (4H, d, $N^{\alpha}CH_2$); 3,31 (8H, s, $N^{\beta}CH_2$, CH_2OCH_2); 2,61 (4H, s, $N^{\alpha}COCH_2$)
XIV	7,60 (4H, s, $N^{\alpha}H$, $N^{\beta}H$); 3,82 (4H, d, $N^{\alpha}CH_2$), 3,39 (12H, s, $N^{\beta}CH_2$, CH_2OCH_2); 2,70 (4H, s, $N^{\alpha}COCH_2$)
XV	7,80 (2H, s, $N^{\alpha}H$); 7,41 (2H, s, $N^{\beta}H$); 3,79 (4H, d, $N^{\alpha}CH_2$); 3,30 (8H, s, $N^{\beta}CH_2$, CH_2OCH_2); 2,20 (4H, s $N^{\alpha}COCH_2$); 1,39 (4H, m, CH_2)
XVI	7,80 (2H, s, $N^{\alpha}H$); 7,40 (2H, s, $N^{\beta}H$); 3,90 (4H, d, $N^{\alpha}CH_2$); 3,39 (12H, s, $N^{\beta}CH_2$, CH_2OCH_2); 2,25 (4H, s, $N^{\alpha}COCH_2$); 1,40 (4H, m, CH_2)
XVII	7,92 (2H, s, $N^{\alpha}H$); 7,63 (2H, s, $N^{\beta}H$); 4,00 (8H, s $N^{\alpha}COCH_2$, $N^{\alpha}CH_2$); 3,29 (8H, s, $N^{\beta}CH_2$, CH_2OCH_2)
XVIII	7,80 (2H, s, $N^{\alpha}H$); 7,45 (2H, s, $N^{\beta}H$); 4,00 (8H, s, $N^{\alpha}COCH_2$, $N^{\alpha}CH_2$); 3,30 (10H, s, $N^{\beta}CH_2$, CH_2OCH_2, OCH_2CH_2O)
XIX	7,51 (4H, s, $N^{\alpha}H$, $N^{\beta}H$); 3,95 (8H, s, $N^{\alpha}COCH_2$, $N^{\alpha}CH_2$); 3,40 (16H, s, $N^{\beta}CH_2$, CH_2OCH_2, OCH_2CH_2O)

fragment resonate at 3.10 ± 0.91 ppm. The signals of the CH_2N and CH_2OCH_2 groups are observed at 3.26 ± 0.15 ppm. The NH group in VI, VIII, XII, XIII, and XV-XVIII shows up in the form of two signals of equal intensity (2H each) at 7.94 ± 0.56 and 7.46 ± 0.17 ppm, whereas in the spectra of VII, IX-XI, XIV, and XIX it shows up in the form of a broad signal at 7.56 ± 0.05 ppm. The presence of two signals of an NH group in the spectra of tetraamides VII, IX-XI, XIV, and XIX is probably due to the presence of intramolecular H bonds. Spectral features of terminal groups are absent in both the IR and PMR spectra of tetraamides VI-XIX, and this confirms the cyclic structure of these substances. A shift of all of the groups of signals to weak field is observed in the PMR spectra when salts of alkali and alkaline earth metals are added to solutions of XII-XIX in deuteromethanol. The protons of the methylene group of glycine and of the $OCH_2C(O)N$ group in the spectra of XVII and XIX experience the greatest shift (∿0.10 ppm).

These spectral changes are similar to those observed for crown ethers [10] and are evidently associated with the formation of complexes of cyclic tetraamides XII-XIX with the ions of the alkali and alkaline earth metals.

The metal cation is evidently included in the inner cavity of the macroring. The possibility of the formation of such complexes is confirmed by an analysis of Corey–Pauling–Koltun (CPK) molecular models.

EXPERIMENTAL

The IR spectra of KBr pellets of the compounds were recorded with a Perkin–Elmer 580B spectrometer. The PMR spectra were recorded with a Tesla BS-467 spectrometer (60 MHz) with hexamethyldisiloxane as the internal standard. The mass spectra were obtained with a Varian MAT CH-5 spectrometer. Silufol UV-254 plates were used for thin-layer chromatography (TLC). Column chromatography was carried out on L 100/160 silica gel.

3-Oxa-1,5-diamine, 3,6-Dioxa-1,8-diamine, and Diglycolic Acid. These compounds were obtained by the method in [11].

Triglycolic Acid. A 6-g (0.04 mole) sample of triethylene glycol was added to 120 g (1.38 g/cm^3) of HNO_3 heated to 60°C, and the reaction mixture was heated at 75°C until the evolution of nitrogen oxides ceased. Another 18 g (0.12 mole) of triethylene glycol was then added in such a way that the temperature did not exceed 60°C, and the mixture was maintained at this temperature for 2 h, after which it was heated at 80°C to 30 min. It was then evaporated at reduced pressure at 70°C, and the residue was dried by azeotropic distillation with benzene to give 21 g (70%) of a product with mp 75-76°C.

Oxalic Acid N,N'-Bis(methoxycarbonylmethyl)diamide (I). A 30.3-g (0.30 mole) sample of dry triethylamine was added with stirring to a cooled (to −10°C) suspension of 19.0 g (0.15 mole) of methyl glycinate hydrochloride in 250 ml of absolute chloroform, and 7.3 g (0.065 mole) of oxalic acid dichloride was then added slowly at 0°C. The mixture was stirred at room temperature for 3 h, after which the solvent was evaporated, the residue was treated with 100 ml of alcohol, and the solid material was removed by filtration and recrystallized from alcohol to give 9.1 g (60%) of a product with mp 158-159°C. IR spectrum: 3375 (NH); 1740 (ester C=O); 1685 (amide C=O); 1210, 1245 cm^{-1} (COC). PMR spectrum: 8.50 (2H, t, NH), 3.95 (4H, d, NCH_2), and 3.32 ppm (6H, s, OCH_3). Found: C 43.2; H 4.6; N 11.9%. $C_8H_{12}N_2O_6$. Calculated: C 43.3; H 4.3; N 12.1%.

Succinic Acid N,N'-Bis(methoxycarbonylmethyl)diamide (II). This compound, with mp 134-135°C, was similarly obtained in 50% yield by the reaction of succinic acid dichloride with methyl glycinate hydrochloride. IR spectrum: 3370 (NH); 1730 (ester C=O); 1660 (amide C=O); 1220, 1240 cm^{-1} (COC). PMR spectrum: 8.10 (2H, t, NH), 3.90 (4H, d NCH_2), 3.30 (6H, s, OCH_3), and 2.60 ppm (4H, s, $NCOCH_2$). Found: C 45.9; H 6.4; N 10.7%. $C_{10}H_{16}N_2O_6$. Calculated: C 46.1; H 6.1; N 10.8%.

Adipic Acid N,N'-Bis(methoxycarbonylmethyl)diamide (III). This compound was similarly synthesized by the reaction of adipic acid dichloride with methyl glycinate hydrochloride. After removal of the solvent by distillation, the residue was treated with hot ethyl acetate, and the mixture was filtered. The precipitated crystals were recrystallized from acetone–hexane to give a product with mp 132-133°C in 50% yield. IR spectrum: 3200, 3290 (NH); 1750, 1760 (ether C=O); 1645 (amide C=O); 1205 cm^{-1} (COC). PMR spectrum: 7.62 (2H, s, NH), 3.80 (4H, d, NCH_2), 3.30 (6H, s, OCH_3), 2.23 (4H, m, $NCOCH_2$), and 1.41 ppm (4H, m, CH_2). Found: C 49.8; H 7.1; N 9.6%. $C_{12}H_{10}N_2O_6$. Calculated: C 50.0; H 6.9; N 9.7%.

Diglycolic Acid N,N'-Bis(methoxycarbonylmethyl)diamide (IV). This compound was obtained by the reaction of diglycolic acid dichloride with glycine methyl ester hydrochloride. After removal of the solvent by distillation, the reaction product was isolated by column chromatography on silica gel [acetone–hexane (2:1)] and recrystallized from acetone–hexane to give a product with mp 88-89°C in 40% yield. IR spectrum: 3420 (NH); 1740 (ester C=O); 1635, 1670 (amide C=O); 1130, 1230 cm^{-1} (COC). PMR spectrum: 7.60 (2H, s, NH), 3.98 (4H, s, $COCH_2O$), 3.80 (4H, d, NCH_2), 3.39 ppm (6H, s, OCH_3). Found: C 43.4; H 6.0; N 10.0%. $C_{10}H_{16}N_2O_7$. Calculated: C 43.5; H 5.8; N 10.1%.

Triglycolic Acid N,N'-Bis(methoxycarbonylmethyl)diamide (V). This compound was obtained by the reaction of triglycolic acid dichloride with glycine methyl ester hydrochloride. After removal of the solvent by distillation, the desired product was isolated by column chromatography on silica gel [acetone–hexane (2:1)], as a result of which an oil was obtained in 40% yield. IR spectrum: 3380 (NH); 1750 (ester C=O); 1680 (amide C=O); 1135, 1215 cm^{-1} (COC). PMR spectrum: 7.45 (2H, s, NH), 3.90 (4H, s, $COCH_2O$), 3.82 (4H, d, NCH_2), and 3.40 ppm (10H, s, OCH_2, OCH_3). Found: C 44.8; H 6.5; N 8.7%. $C_{12}H_{20}N_2O_8$. Calculated: C 45.0; H 6.3; N 8.8%.

1,4,7,10-Tetraazacyclotetradecyl-3,8,11,14-tetraone (VI). A 1.8-g (0.03 mole) sample of ethylenediamine was added with stirring to 7.8 g (0.03 mole) of II in 100 ml of methanol, and the mixture was stirred at 40°C for 3 days. The solvent was removed by distillation, and the residue was treated with 100 ml of hot water and filtered. The precipitated crystals were dried *in vacuo*.

1,4,9,12-Tetraazacyclohexadecyl-3,10,13,16-tetraone (VII). This compound was similarly obtained from 7.8 g (0.03 mole) of II and 2.6 g (0.03 mole) of tetramethylenediamine. After removal of the solvent, the residue was treated with hot methanol, and the mixture was filtered. The precipitated crystals were recrystallized from methanol.

1,4,11,14-Tetraazacyclooctadecyl-3,12,15,18-tetraone (VIII), 1,4,7,10-tetraazacyclohexadecyl-3,8,11,16-tetraone (IX), 1,4,9,12-tetraazacyclooctadecyl-3,10,13,18-tetraone (X), 1,4,11,14-tetraazacycloeicosyl-3,12,15,18-tetraone (XI), 7,10-dioxa-1,4,13,16-tetraazacyclooctadecyl-3,14,17,18-tetraone (XII), 7-oxa-1,4,10,13-tetraazacycloheptadecyl-3,11,14,-17-tetraone (XIII), 7,10-dioxa-1,4,13,16-tetraazacycloeicosyl-3,14,17,20-tetraone (XIV), 7-oxa-1,4,10,13-tetraazacyclononadecyl-3,11,14,19-tetraone (XV), 7,10-dioxa-1,4,13,16-tetraazacyclodocosyl-3,14,17,22-tetraone (XVI), 7,16-dioxa-1,4,10,13-tetraazacyclooctadecyl-3,11,14,18-tetraone (XVII), 7,16,19-trioxa-1,4,10,13-tetraazacycloheneicosyl-3,11,14,21-tetraone (XVIII), and 7,10,19,22-tetraoxa-1,4,13,16-tetraazacyclotetracosyl-3,14,17,24-tetraone (XIX) were similarly obtained by the reaction of the N,N'-bis(methoxycarbacylmethyl)diamines of the corresponding acids with diamines. After removal of the solvent, the desired products were isolated by column chromatography (elution with methanol).

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