# Macrocyclic Polyesters. II.
# The Preparation of Diester Diamide Macrocycles Containing the 2-Aminobutanol Sub-units as Potential Antimicrobial Agents

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This paper reports the synthesis of a novel class of diester diamide macrocycles containing 2-aminobutanol sub-units, by reacting diacyl dichloride with the aminoalcohol or its oxazastannolane derivatives. The structure of the possible isomers was assigned by spectroscopic and analytical data and, when possible by comparison with samples prepared otherwise.

The ms spectra of these compounds have been studied and the fragmentation patterns and structures of the main ions are described.

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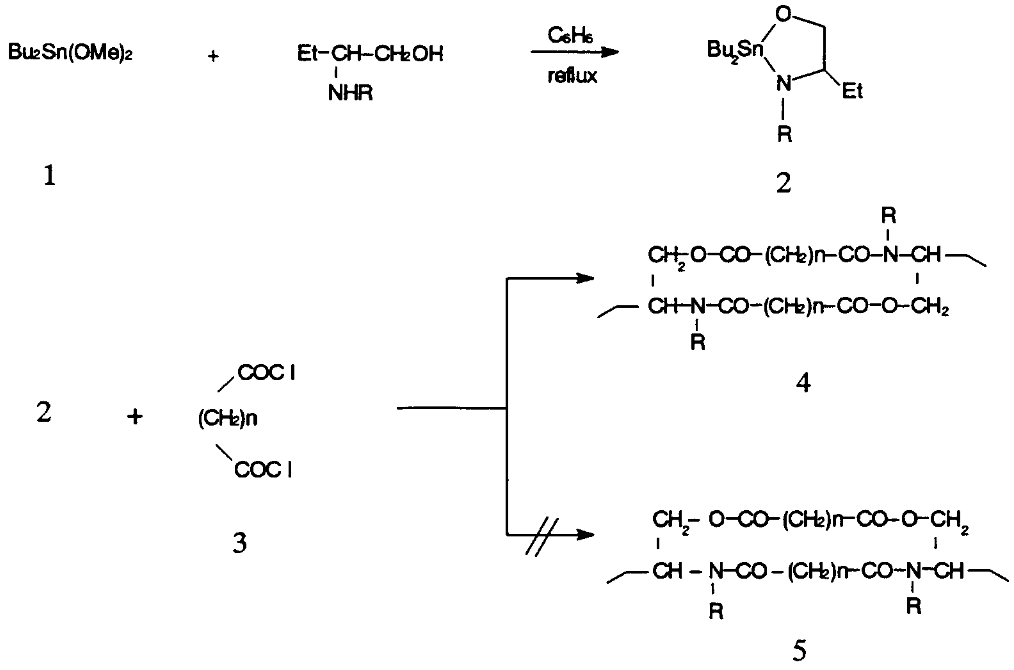
Recently macrocyclic ligands with amide units in the macroring have been of interest because they often complex with metal ions which replace the amide proton. Some macrocyclic diamides, when incorporated into membranes, show a remarkable selectivity for lithium ions over potassium, calcium and magnesium [1,2].

Macrocyclic tetraamides have also been prepared which complex such certain ions as Fe (IV) and other metal ions [3-7]. This kind of macrocyclic compound is interesting both from the biological point of view and as a potential catalyst for phase transfer reactions. In the last 20 years, some macrocyclic polyamides were reported; most of them were synthesized *via* a condensation reaction of diacyl dichlorides or diesters and amines or diamines under high dilution conditions [6,8-12].

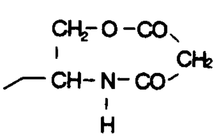
We have previously prepared and studied a few macrocyclic tetraesters some of which showed an appreciable activity against gram-positive bacteria (*Staphylococcus aureus*) and fungi (*Candida albicans*) [13]. The complexation properties of these new macrocycles have not been determined.

We now report the preparation of diamide diester macrocycles containing substituted alkanolamine sub-units with 14, 18, 22, 26, 28 ring members. The synthesis was

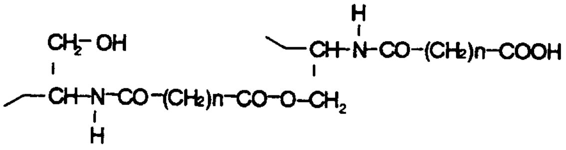
Scheme 1



a) n=1 R=H; b) n=3 R=H; c) n=5 R=H; d) n=7 R=H; e) n=8 R=H; f) n=3 R=$COCH_3$

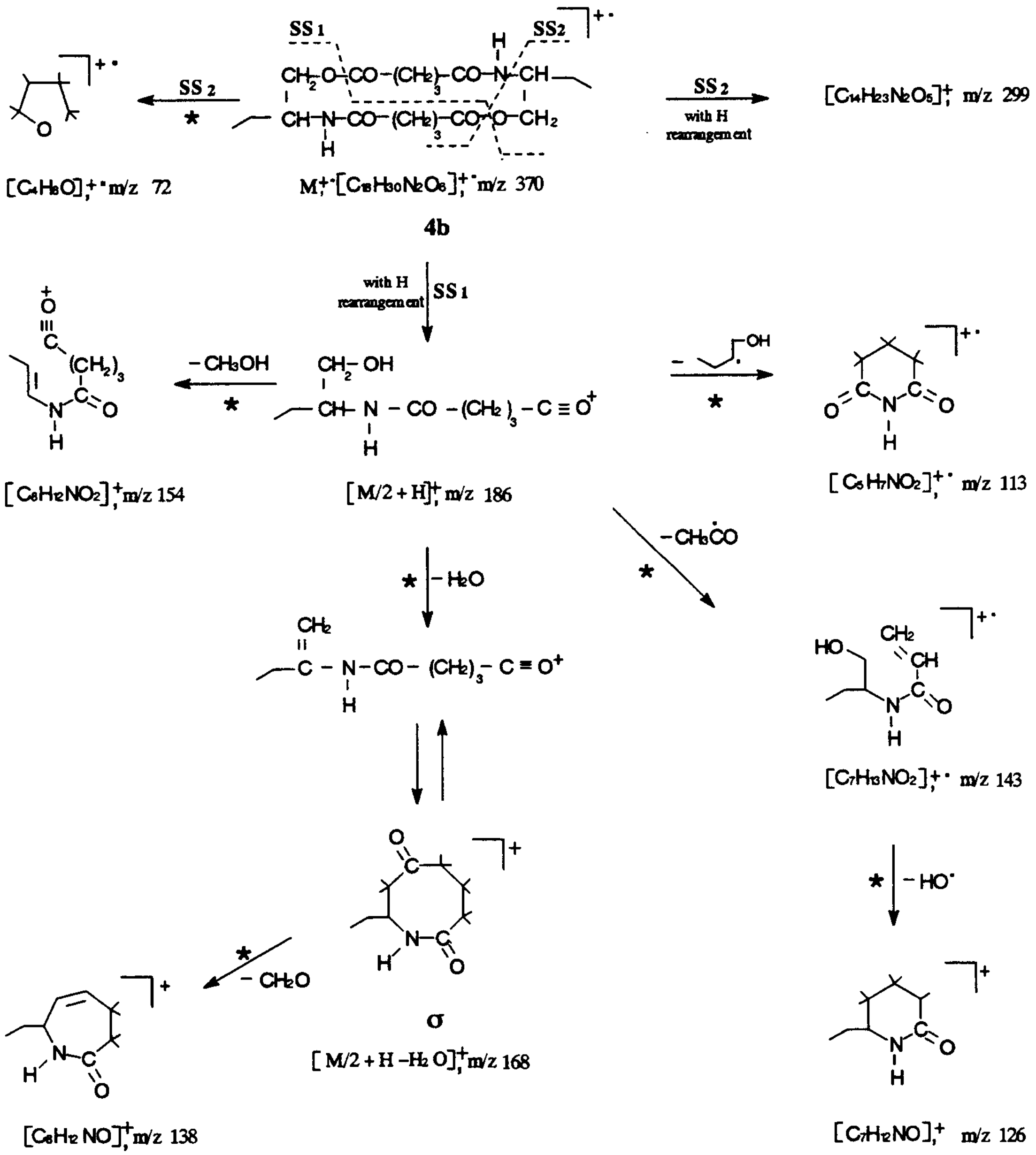


**6a**



**7a-b**

Scheme 2



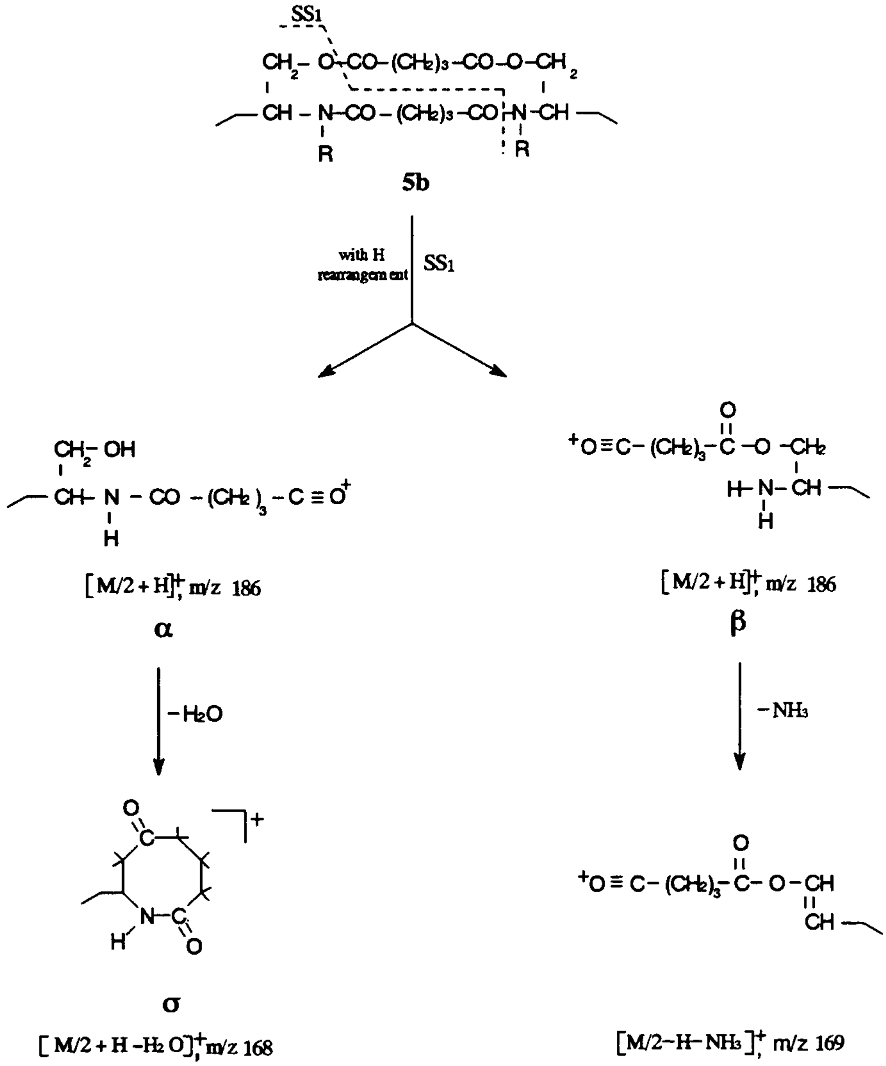
carried out by reacting different diacyl dichlorides **3** with 2,2-dialkyl-1,3,2-oxazastannolane derivatives **2**, which were in turn obtained by reactions of dialkyltin dialkoxides with alkanolamines having free as well as substituted amino groups [14].

The advantage in using stannolane compounds is their good reactivity toward diacyl dichlorides under homogeneous as well as heterogeneous conditions. Thus, oxazastannolane **2** is an excellent intermediate for the preparation of different macrocyclic derivatives by cleavage reactions with diacyl chlorides (Scheme 1). We obtained good yields (20-50%) of the dimeric macrocyclic derivatives, whose structures were assigned on the basis of analytical and spectroscopic data (ir, nmr and ms) and, for some of them, by comparison with authentic samples prepared otherwise. Adduct **6** was obtained in a low yield together with the dimeric adduct when diacyl dichloride **3** (n = 1) was used.

The direct reaction of diacyl dichloride with the aminoalcohol in the presence of a proton acceptor and in a highly diluted chloroform solution gave a mixture of **4, 5, 6** and **7**. The main reaction products were dimeric **4** and the open structure **7** together with small amounts of other products, whose structure was not established in some cases. The formation of **5** was strongly correlated to the concentration and was in an appreciable amount only when n = 1, while it was completely absent in high dilute solutions or with diacyl dichlorides **3** where n ≥ 3.

The $^1$H nmr spectrum of **5** (n = 1) exhibited two well defined singlets related to the methylenes between the two amidic groups and the two ester groups. The two signals appear at 3.33 and 3.13 ppm respectively, while in the spectrum of compound **4** (n = 1), where the two methylene groups are equivalent, only one singlet appears at 3.21 ppm. The structure of compound **7** was assigned either by spectroscopy or by chemistry: *i.e.* by cyclization, working

Scheme 3



in Dean and Stark apparatus at prolonged reflux in benzene solution, it formed compound **4**.

Working *via* the stannolanic derivative, the formation of isomers **4** and **5** or a mixture of both should be possible. Chromatographic (tlc [15]; hplc) and spectroscopic data showed that only one product had been formed. Structure **4** was confirmed unequivocally by mass spectrometry and when possible by chemical reactions.

The fragmentation pattern of compound **4** (n = 3) by mass measurements in the EI source and in the first and second free field regions (B/E, MIKE) is shown in Scheme 2. Molecular ions $M^{+\cdot}$, $[C_{18}H_{30}N_2O_6]^{+\cdot}$, m/z 370, give further confirmation of the dimeric structure of this compound. Formation of $[M/2+H]^+$, $[C_9H_{16}NO_3]^+$, m/z 186 ionic species, obtained by $SS_1$ fragmentation with H rearrangement, is very important in determining the structure of **4**. Successively, loss of $H_2O$ leads to $[C_9H_{14}NO_2]^+$ ions, m/z 168, which are stabilized by the fact that they can have cyclic structure $\sigma$ and, therefore, they became the base peak in the spectrum. Due to the subsequent loss of $CH_2O$, these ions give rise to $[C_8H_{12}NO]^+$, m/z 138 ions. The loss of acetyl from the $[M/2+H]^+$ ions leads to $[C_7H_{13}NO_2]^{+\cdot}$, m/z 143 ions, which in turn lose $HO^\cdot$ giving rise to the very stable $[C_7H_{12}NO]^+$, m/z 126 ions.

To confirm the cyclic structure of $\sigma$, it is very important to observe the variation of the relative abundance ratios of m/z 138 and m/z 143, measured in the three different zones of the instrument. In fact the $[138]^+/[143]^{+\cdot}$ ratio varies from 4 in the EI spectrum to 2 in the B/E spectrum to 0.8 in the MIKE spectrum, respectively, suggesting that the process leading to m/z 138 ions is faster than the one that gives rise to m/z 143 ions. Therefore, the loss of the acetyl group is slower than the two sequential losses of $H_2O$ and $CH_2O$ possibly because loss of $H_2O$ and $CH_2O$ is kinetically preferred in the intermediate formation of the $\sigma$ cyclic structure.

Table 1

m/z ( Relative abundance, %) of most important ions in EI mass spectra of compounds **4a–f**

| Compounds | 4a | 4b | 4c | 4d | 4e |
|---|---|---|---|---|---|
| Ions | | | | | |
| $M^{+\cdot}$ | 314 (21) | 370 (31) | 426 (12) | 482 (13) | 510 (13) |
| $[M-C_4H_7O]^+$ | 243 (9) | 299 (11) | 355 (14) | 411 (11) | 439 (10) |
| $[M-{}_2HNCO(CH_2)_nCO]^+$ | / | 256 (3) | 284 (8) | 312 (3) | 326 (2) |
| $[M/2+H]^+$ | 158 (22) | 186 (24) | 214 (29) | 242 (10) | 256(8) |
| $[M/2]^{+\cdot}$ | 157 (15) | 185 (9) | 213 (8) | 241 (3) | 255 (3) |
| $[M/2+H-H_2O]^+$ | 140 (100) | 168 (100) | 196 (100) | 224 (100) | 238 (100) |
| $[M/2+H-CH_3OH]^+$ | / | 154 (20) | 183 ( 7) | 211 (4) | 225 (3) |
| $[M/2+H-CH_2OH]^{+\cdot}$ | 128 (7) | / | / | / | / |
| $[M/2+H-CH_3CO]^{+\cdot}$ | / | 143 (2) | 171 (3) | 199 (3) | 213 (4) |
| $[M/2+H-CH_2CO]^{+\cdot}$ | 116 (26) | / | / | / | / |
| $[M/2+H-H_2O-CH_2O]^+$ | / | 138 (12) | 166 (8) | 194 (11) | 208 (12) |
| $[M/2+H-CH_3CO-OH]^+$ | / | 126 (30) | 154 (20) | 182 (25) | 196 (28) |
| $[M/2+H-CH_2CO-OH]^+$ | 100 (8) | / | / | / | / |
| $[{}_2HNCO-(CH_2)n-CO^+]$ | 86 (7) | 114 (35) | 142 (9) | 170 (14) | 184 (13) |
| $[M/2+H-C_4H_9O]^{+\cdot}$ | 85 (14) | 113 (21) | 141 (6) | 169 (7) | 183 (8) |
| $[O=C=CH(CH_2)_nC{=}O^+]$ | 69 (51) | 97 (19) | 125 (25) | 153 (20) | 167 (12) |
| $[C_4H_7O]^+$ | 72 (28) | 72 (34) | 72 (55) | 72 (42) | 72 (30) |

For the hypothetical structure **5**, and $SS_1$ fragmentation would lead to formation of two$[M/2+H]^+$ ions, $\alpha$ and $\beta$, with identical composition and mass but different structure (Scheme 3). In fact this would form an $\alpha$ structure identical to the m/z 186 ions described above and a $\beta$ structure which would lose ammonia and give rise to m/z 169 ionic species. This last species is completely absent in the mass spectrum.

The study of B/E and MIKE spectra on the m/z 186 ions is further confirmation of the uniqueness of the $\alpha$ structure of these ionic species (Figure 1). For this purpose besides the m/z ions described above, m/z 154 and m/z 113 ions, that derive from a loss of methanol and butanol, respectively, are also present.

The mass spectra of compounds **4a-e** are reported in Table 1.

## EXPERIMENTAL

2-Aminobutanol, acetyl chloride, malonyl dichloride, glutaroyl dichloride, azeloyl dichloride, sebacoyl dichloride, and dibutyltin dimethoxide were commercial samples and used as received. Literature procedures were followed in the preparation of pimeloyl dichloride [16] and *N*-acetyl-2-aminobutanol [17]. Melting points are uncorrected and were determined using an electrothermal capillary point apparatus. All infrared spectra were obtained on a Perkin Elmer model 157G spectrophotometer. The proton nuclear magnetic resonance spectra were obtained on a Varian EM 360 L spectrometer in deuteriochloroform solution using tetramethylsilane as internal standard. The mass spectra were run on a VG ZAB 2F instrument operating at 70 eV (200$\mu$A). Microanalyses for CHN were carried out on a Fison model EA 1108 elemental analyzer. Precoated Merck F254 silica gel plates were used for thin layer chromatography; chromatographic separations were performed on columns packed with Merck silica gel (70-230 mesh). Hplc analyses were carried out on a Perkin Elmer series 4 liquid chromatography. A Spherisorb $C_{18}$ ODS-2 (5 $\mu$, 150 x 4.6 mm i.d.) column was used, mobile phase: 25:75 water-acetonitrile; flow-rate: 1 ml minute$^{-1}$; detector uv/visible Perkin Elmer LC85 B ($\lambda$ = 237 nm).

### General Procedure for the Synthesis of Oxazastannolane Derivatives **2a,f**.

Slightly modified literature procedures [14,18,19] were adopted. These compounds were prepared by distilling azeotropically equimolar amount of aminoalcohol and dibutyltin dimethoxide in 100 ml of benzene for 24 hours. Evaporation under vacuum of the solvent gave product **2**.

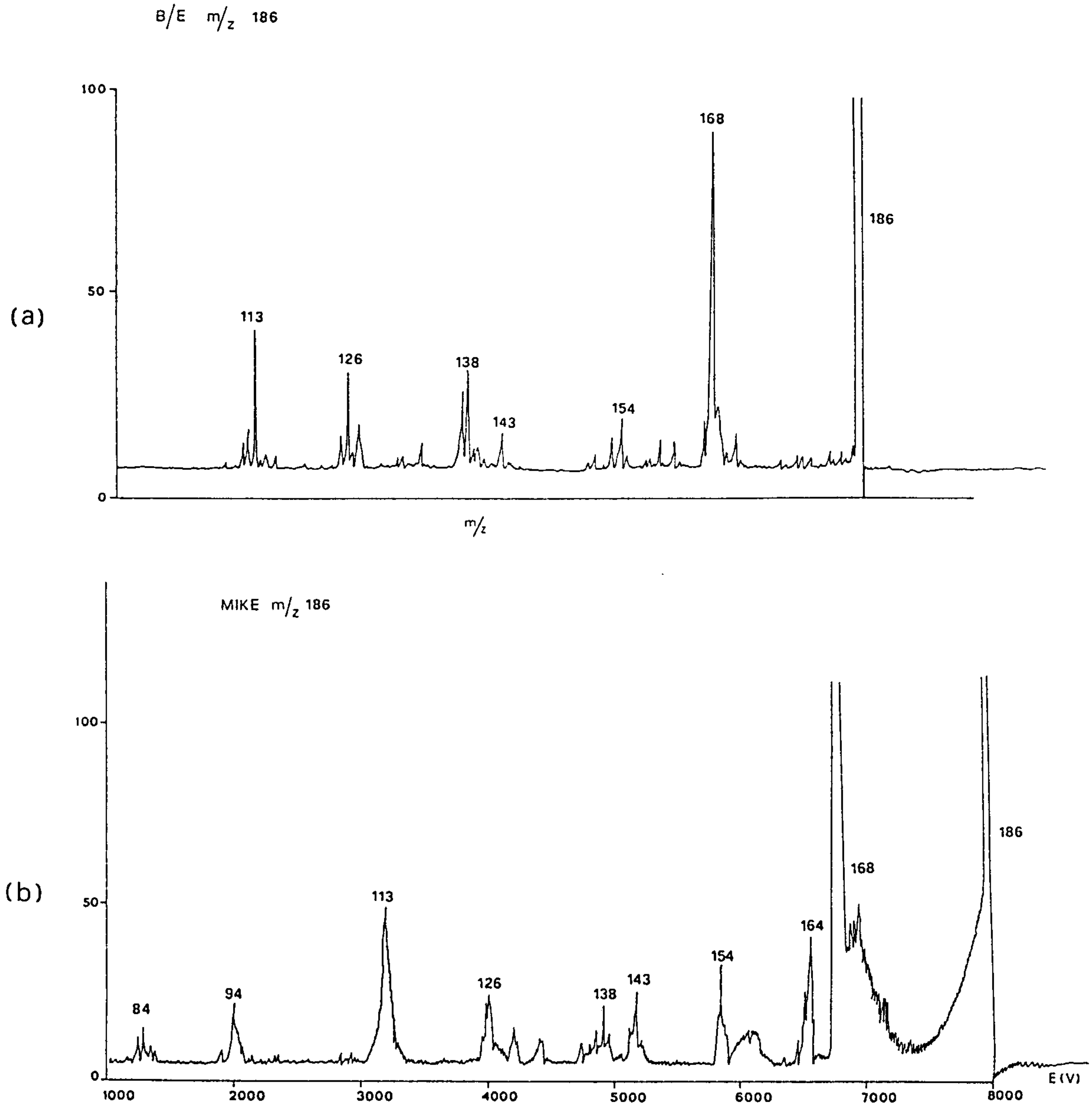


Figure 1. a) B/E linked-scan mass spectrum of [M/2 + H]$^+$, m/z 186; b) MIKE spectrum of [M/2 + H]$^+$, m/z 186.

### 2,2-Di-*n*-butyl-1,3,2-oxazastannolane (**2a**).

Racemic 2-aminobutanol (0.89 g, 0.01 mole) and dibutyltin dimethoxide **1** (2.94 g, 0.01 mole) were used. This compound was obtained (80% yield) as a yellow viscous liquid which slowly decomposed in the presence of air moisture, $n_D^{20}$ 1.4943; ir (film): $\nu$ 3220, 2969, 2890, 2860, 2800, 2700, 1600, 1460, 1375, 1290, 1250, 1170, 1150, 1060, 870, 770 and 675 cm$^{-1}$; $^1$H nmr (deuteriochloroform): $\delta$ 3.23 (m, 3H, CH+CH$_2$O), 2.5 (s, 1H, NH, D$_2$O exchanged), 1.5 (q, 2H, C*H*$_2$CH$_3$), 1.23 (m, 18H, 2C$_4$H$_9$) and 0.8 ppm (t, 3H, CH$_2$C*H*$_3$).

*Anal.* Calcd. for $C_{12}H_{27}NOSn$: C, 47.03; H, 8.50; N, 4.38. Found: C, 46.85; H, 8.54; N, 4.36.

### 2,2-Di-*n*-butyl-*N*-acetyl-1,3,2-oxazastannolane (**2f**).

2-Acetylamidobutanol (1.31 g, 0.01 mole) and dibutyltin dimethoxide (0.75 g, 0.01 mole) were used. This product was obtained (90% yield) as a yellow viscous liquid which decomposed in air moisture; ir (film): $\nu$ 2950, 2850, 1640, 1550, 1460, 1375, 1280, 1150, 1060, 1000, 875, 760 and 670 cm$^{-1}$; $^1$H nmr (deuteriochloroform): $\delta$ 4.03 (m, 1H, CH), 3.53 (d, 2H, CH$_2$O), 1.90 (s, 3H, COCH$_3$), 1.36 (m, 20H, C*H*$_2$CH$_3$ + 2C$_4$H$_9$) and 0.9 ppm (t, 3H, CH$_2$C*H*$_3$).

*Anal.* Calcd. for $C_{14}H_{29}NO_2Sn$: C, 46.43; H, 8.07; N, 3.86. Found: C, 46.26; H, 8.11; N, 3.84.

### General Procedure for the Synthesis of Macrocyclic Derivatives **4a-f**.

### Method A.

A solution of diacyl dichloride **3** (0.012 mole) in benzene (5 ml) was added dropwise to a stirred hot solution of compound **2** (0.01

mole) in dry benzene (100 ml). The resulting mixture was stirred under reflux for 24 hours. The solvent was then removed under vacuum and the residue was mixed with *n*-hexane (3 x 10 ml) to dissolve the dibutyltin dichloride. After decanting the *n*-hexane solution, the crude product was purified by column chromatography on silica gel using chloroform-methanol 30:1 or methylene dichloride-methanol 1:10 as eluants.

Method B.

A solution of diacyl dichloride **3** (0.011 mole) in chloroform (5 ml) was added dropwise at 0° to a well-stirred dispersion of racemic 2-aminobutanol (0.011 mole) and anhydrous sodium carbonate in chloroform (300 ml). The resulting mixture was then refluxed for 24 hours. After cooling, the mixture was filtered to remove the inorganic solid. The chloroform was evaporated under vacuum to give an oily residue which was chromatographed on a silica gel column. Following this procedure, compounds **5a**, **6a**, **7a** and **7b** were also obtained.

3,10-Diethyl-1,8-dioxa-4,11-diazacyclotetradecane-5,7,12,14-tetraone (**4a**).

This compound was obtained as a white solid, mp 243-245°, 20% (method A), 10% (method B); ir (nujol): $\nu$ 3275 (NH), 2915-2895 ($CH_2$), 1740 (COO), 1640 cm$^{-1}$ (CON); $^1H$ nmr (deuteriochloroform): $\delta$ 4.06 (m, 6H, 2CH + $2CH_2O$), 3.21 (s, 4H, $2OCOCH_2CON$), 1.66 (s, 2H, 2NH, deuterium oxide exchanged), 1.50 (q, 4H, $2C\mathit{H}_2CH_3$) and 0.91 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{14}H_{22}N_2O_6$: C, 53.49; H, 7.05; N, 8.91. Found: C, 53.30; H, 7.07; N, 8.88.

3,12-Diethyl-1,10-dioxa-4,13-diazacyclooctadecane-5,9,14,18-tetraone (**4b**).

This compound was obtained as a white powder, mp 245-247°, 35% (method A), 20% (method B); ir (nujol): $\nu$ 3270 (NH), 2910-2895 ($CH_2$), 1735 (COO), 1635 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 4.05 (m, 6H, 2CH + $2CH_2O$), 2.33 (t, 4H, $2CH_2CON$), 2.22 (t, 4H, $2CH_2COO$), 1.90 (m, 4H, $2CH_2C\mathit{H}_2CH_2$), 1.71 (s, 2H, 2NH, deuterium oxide exchanged), 1.51 (q, 4H, $2C\mathit{H}_2CH_3$) and 0.93 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{18}H_{30}N_2O_6$: C, 58.36; H, 8.16; N, 7.56. Found: C, 58.48; H, 8.19; N, 7.53.

3,14-Diethyl-1,12-dioxa-4,15-diazacyclodocosan-5,11,16,22-tetraone (**4c**).

This compound was obtained as a white powder, mp 212-215°, 45% (method A), 35% (method B); ir (nujol): $\nu$ 3280 (NH), 2920-2885 ($CH_2$), 1740 (COO), 1645 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 4.05 (m, 6H, 2CH + $2CH_2O$), 2.23 (t, 4H, $2CH_2CON$), 2.16 (t, 4H, $2CH_2COO$), 1.80 (s, 2H, 2NH, deuterium oxide exchanged), 1.56 (m, 12H, $2CH_2CH_2CH_2$), 1.43 (q, 4H, $2C\mathit{H}_2CH_3$) and 0.91 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{22}H_{38}N_2O_6$: C, 61.94; H, 8.98; N, 6.57. Found: C, 61.74; H, 9.02; N, 6.55.

3,6-Diethyl-1,4-dioxa-4,17-diazacycloesacosane-5,13,18,26-tetraone (**4d**).

This compound was obtained as a white powder, mp 170-172°, 48% (method A), 35% (method B); ir (nujol): $\nu$ 3220 (NH), 2920-2885 ($CH_2$), 1740 (COO) and 1645 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 4.02 (m, 6H, 2CH + $2CH_2O$), 2.22 (t, 4H, $2CH_2CON$), 2.13 (t, 4H, $2CH_2COO$), 1.70 (s, 2H, 2NH, deuterium oxide exchanged), 1.52 (q, 4H, $2C\mathit{H}_2CH_3$), 1.20 (m, 22H, $2(CH_2)_5$) and 0.89 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{26}H_{46}N_2O_6$: C, 64.70; H, 9.61; N, 5.80. Found: C, 64.45; H, 9.64; N, 5.77.

3,17-Diethyl-1,15-dioxa-4,18-diazacyclooctacosane-5,14,19,28-tetraone (**4e**).

This compound was obtained as a white powder, mp 165-167°, 50% (method A), 38% (method B); ir (nujol): $\nu$ 3296 (NH), 2910-2890 ($CH_2$), 1731 (COO) and 1638 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 4.03 (m, 6H, 2CH + $2CH_2O$), 2.21 (t, 4H, $2CH_2CON$), 2.11 (t, 4H, $2CH_2COO$), 1.60 (s, 2H, 2NH, deuterium oxide exchanged), 1.51 (q, 4H, $2C\mathit{H}_2CH_3$), 1.16 (m, 24H, $2(CH_2)_6$) and 0.86 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{28}H_{50}N_2O_6$: C, 65.85; H, 9.87; N, 5.49. Found: C, 65.72; H, 9.91; N, 5.46.

4,13-Diacetyl-3,12-diethyl-1,10-dioxa-4,13-diazacyclooctadecane-5,9,14,18-tetraone (**4f**).

This compound was obtained as a yellow oil, $n_D^{21}$ 1.4784, 32% (method A), 28% (method B); ir (nujol): $\nu$ 2980-2870 ($CH_2$), 1735 (COO) and 1670 cm$^{-1}$ (CON); $^1H$ nmr (deuteriochloroform) $\delta$ 4.76 (m, 2H, 2CH), 4.30 (d, 4H, $2CH_2O$), 2.56 (t, 4H, $2CH_2COO$), 1.96 (s, 6H, $2COCH_3$), 1.86 (t, 4H, $2CH_2CON$), 1.66 (m, 8H, $2C\mathit{H}_2CH_3$ + $2CH_2C\mathit{H}_2CH_2$) and 0.91 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{22}H_{34}N_2O_8$: C, 58.13; H, 7.54; N, 6.16. Found: C, 58.04; H, 7.57; N, 6.14.

3,11-Diethyl-1,13-dioxa-4,10-diazacyclooctadecane-5,9,14,18-tetraone (**5a**).

This compound was obtained as a white powder, mp 184-185°, 10% (only method B); ir (nujol): $\nu$ 3260 (NH), 2920-2890 ($CH_2$), 1740 (COO) and 1640 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 4.06 (m, 6H, 2CH + $2CH_2O$), 3.33 (s, 2H, $CH_2COO$), 3.13 (s, 2H, $CH_2CON$), 1.66 (s, 2H, 2NH, deuterium oxide exchanged) 1.43 (q, 4H, $2C\mathit{H}_2CH_3$) and 0.9 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{14}H_{22}N_2O_6$: C, 53.49; H, 7.05; N, 8.91. Found: C, 53.32; H, 7.08; N, 8.88.

3-Ethyl-1-oxa-4-azacycloheptane-5,7-dione (**6a**).

This compound was obtained as a white powder, mp 193-195°, 12% (only method B); ir (nujol): $\nu$ 3270 (NH), 2920-2890 ($CH_2$), 1740 (COO) and 1640 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 4.08 (m, 3H, CH + $CH_2O$), 2.91 (s, 2H, $CH_2$), 1.65 (s, 2H, 2NH, deuterium oxide exchanged), 1.50 (q, 2H, $C\mathit{H}_2CH_3$) and 0.90 ppm (t, 3H, $CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_7H_{11}NO_3$: C, 53.49; H, 7.05; N, 8.91. Found: C, 53.28; H, 7.07; N, 8.88.

$\alpha$-(1-Hydroxy-2-butyl)amido-$\alpha'$-(2-carboxyacetylamino-1-butyl)-malonic ester (**7a**).

This compound was obtained as a pale oil $n_D^{21}$ 1.4794, 25% (only method B); ir (film): $\nu$ 3280 (COOH + OH), 3050 (NH), 2940-2850 ($CH_2$), 1740 (COO) and 1640 cm$^{-1}$ (CON); $^1H$ nmr (deuteriochloroform): $\delta$ 9.03 (s, 1H, COOH, deuterium oxide exchanged), 4.06 (m, 4H, 2CH + $CH_2O$), 3.70 (s, OH, deuterium oxide exchanged), 3.53 (d, 2H, $CH_2OH$), 3.23 (s, 2H, 2NH, deuterium oxide exchanged), 3.10 (s, 4H, $2OCOCH_2CON$), 1.41 (q, 4H, $2C\mathit{H}_2CH_3$) and 0.83 ppm (t, 6H, $2CH_2C\mathit{H}_3$).

*Anal.* Calcd. for $C_{14}H_{24}N_2O_7$: C, 50.59; H, 7.28; N, 8.43. Found: C, 50.39; H, 7.31; N, 8.41.

$\alpha$-(1-Hydroxy-2-butyl)amido-$\gamma$-(2-carboxybutanoylamino-1-butyl)-glutaric Ester (**7b**).

This compound was obtained as a pale oil $n_D^{21}$ 1.4715, 30% (only method B); ir (film): $\nu$ 3360 (OH + COOH), 3300 (NH), 2970-2920 ($CH_2$), 1730 (COO) and 1640 cm$^{-1}$ (CONH); $^1H$ nmr (deuteriochloroform): $\delta$ 9.05 (s, 1H, COOH, deuterium oxide exchanged), 4.08 (m, 4H, 2CH + $CH_2O$), 3.66 (s, OH, deuterium oxide exchanged), 3.46 (s, 2H, 2NH, deuterium oxide exchanged), 3.40 (d, 2H, $CH_2OH$), 2.23 (t, 4H, $CH_2COO$ + $CH_2COOH$), 2.10 (t, 4H, $2CH_2CON$), 1.76 (m, 4H, $2CH_2CH_2CH_2$), 1.33 (q, 4H, $2CH_2CH_3$) and 0.83 ppm (t, 6H, $2CH_2CH_3$).

*Anal.* Calcd. for $C_{18}H_{32}N_2O_7$: C, 55.65; H, 8.30; N, 7.21. Found: C, 55.44; H, 8.33; N, 7.18.

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