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FULL PAPER

Synthesis of Macrocyclic Lactones *via* Ring Transformation of 4-(ω-Hydroxyalkyl)-1,3-oxazol-5(4*H*)-ones

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The synthesis of α-benzamido-α-benzyl lactones **23** of various ring size was achieved either *via* 'direct amide cyclization' by treatment of 2-benzamido-2-benzyl-ω-hydroxy-*N*,*N*-dimethylalkanamides **21** in toluene at 90 – 110° with HCl gas or by 'ring transformation' of 4-benzyl-4-(ω-hydroxyalkyl)-2-phenyl-1,3-oxazol-5(4*H*)-ones under the same conditions. The precursors were obtained by C-alkylations of 4-benzyl-2-phenyl-1,3-oxazol-5(4*H*)-one (**15**) with THP- or TBDMS-protected ω-hydroxyalkyl iodides. Ring opening of the THP-protected oxazolones by treatment with Me_2NH followed by deprotection of the OH group gave the diamides **21**, whereas deprotection of the TBDMS series of oxazolones **25** with TBAF followed by treatment with HCl gas led to the corresponding lactones **23** in a one-pot reaction.

Keywords: Lactones, 1,3-Oxazol-5(4*H*)-ones, Ring transformation, Direct amide cyclization, Alkylation, X-Ray crystallography.

Introduction

Since the pioneering work of *Erlenmeyer* on the synthesis of azlactones [1], 1,3-oxazol-5(4*H*)-ones have been recognized as an important class of heterocycles [2]. Their synthesis, reactivity, and use as intermediates in organic synthesis have been described in several reviews [3], and the continuing interest in the chemistry of 1,3-oxazol-5(4*H*)-ones is documented by a large series of recent publications (*e.g.*, [4]).

We became interested in 1,3-oxazol-5(4*H*)-ones as intermediates in the 'azirine/oxazolone method' for the synthesis of peptides containing α,α-disubstituted α-amino acids [5][6]. It has been shown that peptides of type **1** with a C-terminal Aib-amide on treatment with HCl in toluene at elevated temperature form 1,3-oxazol-5(4*H*)-ones **2**, which in the presence of nucleophiles spontaneously react under ring opening to give, *e.g.*, esters **3** [6a][6c][6d] (*Scheme 1*). Furthermore, it was proposed that the DCC-coupling of peptides containing a terminal Aib-acid with amino acid esters **4** leads to peptides **5** *via* an intermediate 1,3-oxazol-5(4*H*)-one **2** [6b – 6e] (*cf.* also [4e][7]). The smooth and selective cyclization to give 1,3-oxazol-5(4*H*)-ones may be explained by the *Thorpe–Ingold* (gem-dimethyl) effect [8].

In Aib-amides of type **1** with an alcohol function in R^1, the intermediate **2** undergoes a spontaneous ring enlargement reaction *via* nucleophilic attack of the OH group onto C(5)=O of **2** to give lactones of type **6** (*Scheme 1*) [9]. This reaction, called 'direct amide cyclization', has been used extensively for the synthesis of Aib-containing cyclodepsipeptides [9 – 11]. The same concept, under different reaction conditions, proved to be successful for the preparation of Aib-containing cyclopeptides [11][12].

It was also shown that amino acid derivatives of type **8** under the conditions of the 'direct amide cyclization' react to give 13- to 15-membered lactams **10**, albeit in low yield (11 – 27%) [13] (*Scheme 2*). Most likely, 1,3-oxazol-5(4*H*)-ones **9** are formed as intermediates, which undergo a ring transformation *via* nucleophilic attack of the amino group onto C(5)=O of **9**. An analogous ring transformation has been described recently by *Rai et al.* [4g] (*Scheme 2*): although 2-phenyl-1,3-oxazol-5(4*H*)-one (**11**) and the tosylated aziridine **12** in the ionic liquid [bmim]OH at room temperature react to give the oxazolone derivative **13**, the corresponding pyrrolidine **14** was formed in the presence of I_2 as a 96:4 mixture of *cis*/*trans*-isomers. It has been shown that **13** is an intermediate, which undergoes ring transformation to yield **14** by treatment with I_2 in [bmim]OH at room temperature.

Based on the results described above, it was of interest to study the 'direct amide cyclization' of compounds of type **8** bearing an OH instead of the NH_2 group in the side chain as well as the corresponding ring transformation of analogues of **9** with a $(CH_2)_nOH$ group at C(4). Preliminary experiments with compounds **8** with a $(CH_2)_nOH$ side chain were reported many years ago [14] (*cf.* also [11]). In the present report, we describe the

[‡]) Part of the Ph.D. thesis of *S. P. F.*, Universität Zürich, 1995.

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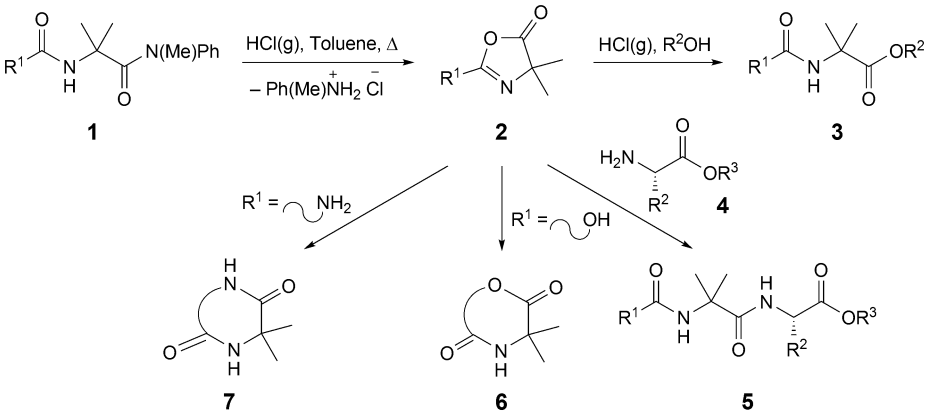
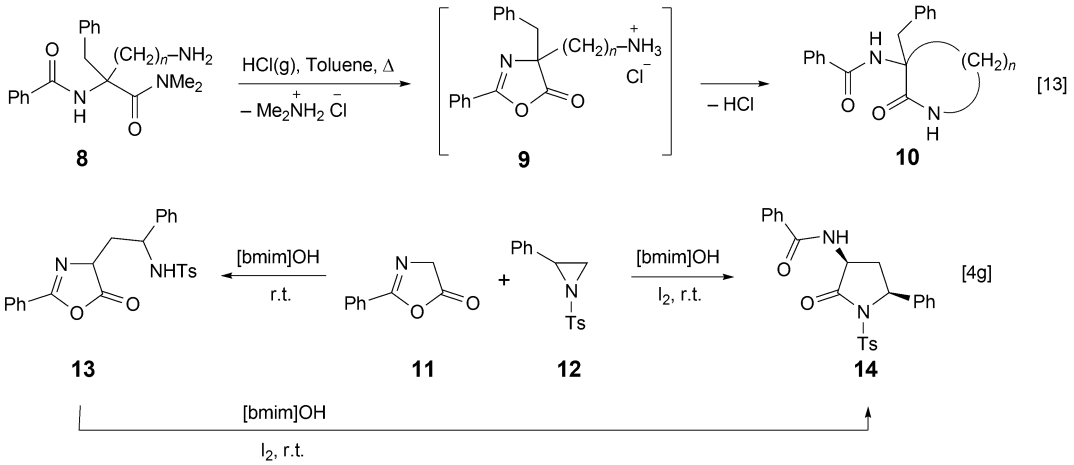
Scheme 1



Scheme 2



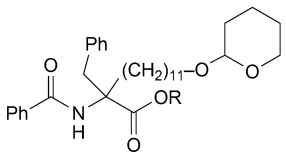
extension of the study in detail together with the crystal structures of some of the prepared macrocyclic lactones.

Results and Discussion

Motivated by the results of the preliminary study [14], we planned to prepare 1,3-oxazol-5(4*H*)-ones of type **17** with THP-protected alkanol substituents at C(4) (*Scheme 3*). The alkylation of 4-benzyl-2-phenyl-1,3-oxazol-5(4*H*)-one (**15**) [15] with 2-[(ω-iodoalkyl)oxy]tetrahydropyrans **16** [16] in THF/HMPT with LDA at 15 – 20° led to mixtures of the desired C(4)-alkylated 1,3-oxazol-5(4*H*)-ones **17** and the isomeric O-alkylated 1,3-oxazoles **18** (ratio *ca.* 2:1 to 4:1)[1]). Unfortunately, all attempts to deprotect selectively the alcohol function were unsuccessful, and complex mixtures of products were formed. For this reason, the mixtures of **17** and **18** were treated with Me_2NH in MeCN at room temperature. After chromatographic purification, the diamides **20** were obtained and subsequently deprotected by treatment with pyridinium tosylate (Py · TsOH) in boiling EtOH [17] to give compounds **21** in good yields. Finally, the 'direct amide cyclization' of **21b** (n = 11), *i.e.*, treatment of a solution in toluene at reflux with HCl gas for *ca.* 2 h, gave the lactone **23b** in 86% yield (with respect to **21b**). Under the same reaction conditions, **23a** (n = 9) was obtained in only 43% yield. In addition, 4-benzyl-4-(9-hydroxynonyl)-2-phenyl-1,3-oxazol-5(4*H*)-one, the proposed intermediate in the 'direct amide cyclization' is the corresponding hydrochloride **22a** (*Scheme 3*) – was isolated in 55% yield. Obviously, in this case, the reaction time was too short for complete conversion.

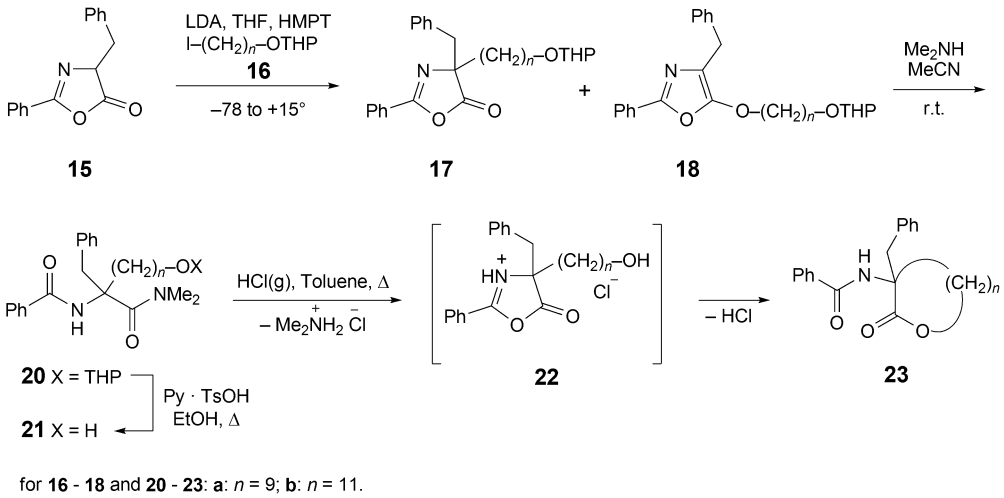
The structures of the products **23** were confirmed from their spectroscopic data and by X-ray crystallography (*Fig. 1*). Since for both molecules the space group is centrosymmetric ($P2_1/c$), the compounds in the crystals are racemic. Furthermore, in the case of **23a**, the benzyl

[1]) In the case of the alkylation with 2-[(11-iodoundecyl)oxy] tetrahydropyran (**16b**), butyl 2-benzamido-2-benzyl-13-[(tetrahydropyran-2-yl)oxy]tridecanoate (**19**, 11%) was isolated in addition to **17b**/**18b**.



19 R = $(CH_2)_3Me$

Scheme 3



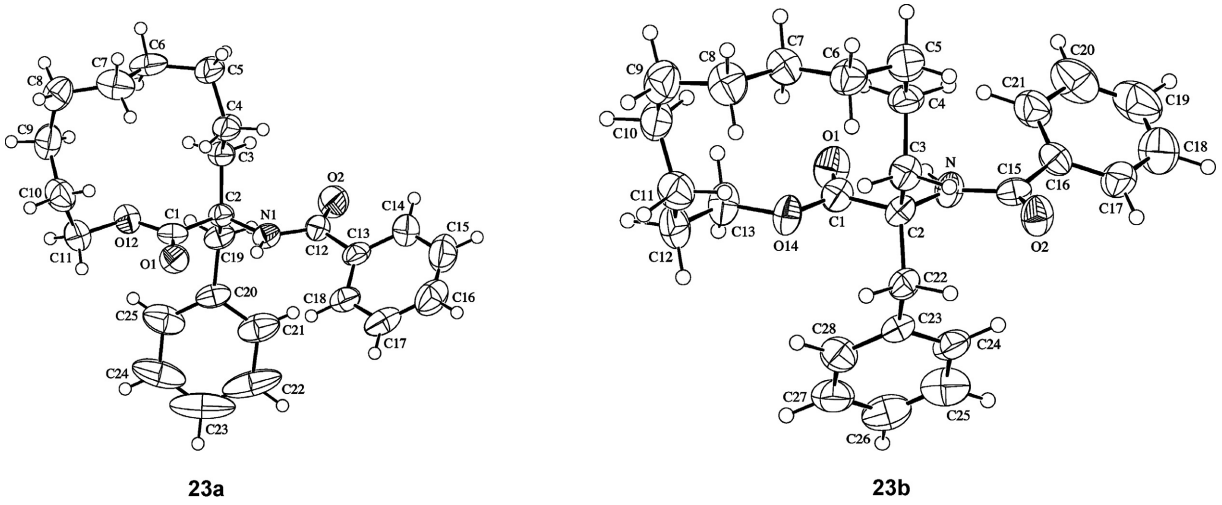


Fig. 1. ORTEP Plot [18] of the molecular structures of the 12- and 14-membered lactones **23a** (major conformation) and **23b** (50% probability ellipsoids, arbitrary numbering of atoms).

group is disordered over two nearly equally occupied conformations.

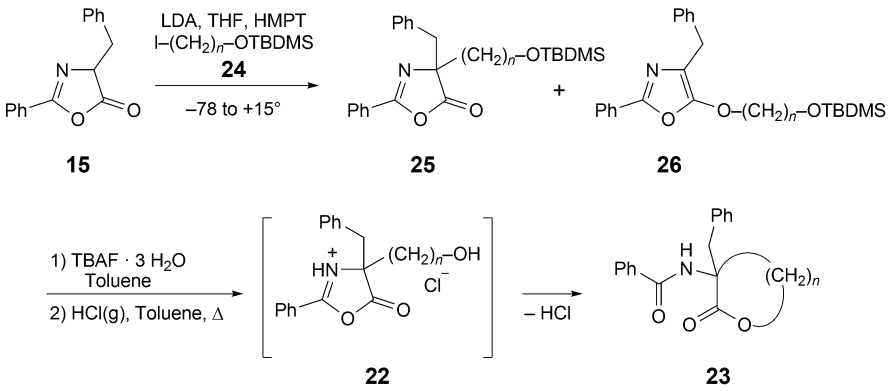
In summary, although the described protocol allows the synthesis of 14- and 12-membered lactones **23**, the reaction sequence *via* ring opening of **17**, subsequent deprotection of **20**, and cyclization *via* the oxazolone intermediate **22** is not very straightforward. Deprotection of the alcohol function in **17** followed by ring transformation to give the lactone would be a more attractive approach. Therefore, a different OH-protecting group was desired, which could be cleaved without destroying the oxazolone moiety in compounds of type **17**.

A suitable choice was the (*tert*-butyl)dimethylsilyl (TBDMS) group, which allows deprotection by treatment with F^- ions [19]. The ω-bromoalkanols in DMF were treated with TBDMSCl and imidazole at 25° for 1.5 h leading to the O-protected derivatives in 81 – 99% yield [16][2]). The latter were transformed into the corresponding iodo compounds **24** by treatment with excess NaI in boiling acetone (*Finkelstein* reaction) [20][3]). The subsequent alkylation of the 1,3-oxazolone **15** using pure 12-{[(*tert*-butyl)dimethylsilyl]oxy}-1-iodododecane (**24c**, $n = 12$) under the conditions described for the THP-

[2]) The desired O-protected ω-bromoalkanols were, in some cases, contaminated with variable amounts of the corresponding O-protected ω-chloroalkanols.

[3]) For the halogen exchange, the obtained mixtures of bromo and chloro derivatives were used. Under the chosen reaction conditions, only the bromo compounds were transformed to the desired O-protected ω-iodoalkanols [16]. For the subsequent alkylations of oxazolone **15**, mixtures of iodo and chloro derivatives were used.

Scheme 4



for **22** – **26**: **b**: n = 11; **c**: n = 12; **d**: n = 10; **e**: n = 8; **f**: n = 7; **g**: n = 6; **h**: n = 3; **i**: n = 2.

Table 1. Alkylations of 1,3-oxazol-5(4*H*)-one **15** with ω-{[(*tert*-butyl)dimethylsilyl]oxy}-1-iodoalkanes **24**[a]) (*Scheme 4*)

Iodoalkane	n	Products			Yield [%][b])	Ratio
24		Oxazolone **25**	Oxazole **26**	Esters **27** + **28**	**25/26**	**25/26**
b	11	**b**	**b**	–	52	9:1
c	12	**c**	–	**c** (14%)	35	
d	10	**d**	**d**	**d** (9%)	60	10:1
e	8	**e**	**e**	–	58	16:2
f	7	**f**	**f**	**f** (14%)[c])	42	23:4
g	6	**g**	**g**	**g** (13%)[c])	30	20:3
h	3	**h**	**h**	**h** (16%)[c])	40	13:4
i	2	**i** (61%)	**i** (10%)	–	71	6:1

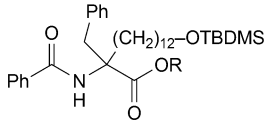
[a]) LDA, THF/HMPT (*ca.* 2:1 – 4:1), –78° – +15°. [b]) Yield of isolated products. [c]) Only butyl ester of type **27**.

protected iodoalkanols **16** gave, after chromatographic workup, the azlactone **25c** as a viscous oil in 35% yield (*Scheme 4*, *Table 1*)[4]). In a similar manner, **15** was alkylated using the obtained ω-{[(*tert*-butyl)dimethylsilyl]oxy}-1-iodoalkanes (**24**, n = 11, 10, 8 – 6, 3, 2)[2]). In all cases, mixtures of the corresponding C- and O-alkylated products **25** and **26** were formed, with the desired **25** as the major product, in some cases together with the corresponding butyl ester of type **27** (*Table 1*). Separation of the products by flash chromatography (FC; [21]) gave, in general, a mixture of **25** and **26**, which was subsequently used for the ring transformation reaction. Only in the case of **24i**, the two products **25i** and **26i** with the $(CH_2)_2$ side chain, were obtained as pure compounds.

The first experiments to deprotect the silylated primary alcohol group in compounds **25** by treatment with pyridinium fluoride were unsuccessful, but the deprotection was achieved with tetrabutylammonium fluoride (TBAF). Thus, bubbling HCl gas through a solution of **25c** and TBAF · 3 H_2O in toluene at 90 – 110° for 1.5 – 2 h led directly to the 15-membered lactone **23c**, which was isolated as a colorless crystalline material in 65% yield (*Scheme 4*, *Table 2*). In one of the repetitions of the experiment, 35% of **23c** were obtained side by side with 43% of 4-benzyl-4-(12-hydroxydodecyl)-2-phenyl-1,3-oxazol-5(4*H*)-one, the proposed deprotected intermediate. Under acidic conditions (HCl), the corresponding hydrochloride **22c** spontaneously underwent ring transformation to give **23c**. The expected structure of **23c** was supported by the spectroscopic and analytical data, and finally established by an X-ray crystal structure determination (*Fig. 2*).

The space group of **23c** (*Pbca*) is centrosymmetric and therefore the compound in the crystal is racemic. The 15-membered ring is disordered in such a way that both enantiomers are present at the same site. In this arrangement, all atoms of the enantiomers occupy identical positions with the only detectable difference being the

[4]) In addition, a nonseparable mixture of butyl and but-3-enyl 2-benzamido-2-benzyl-14-{[(*tert*-butyl)dimethylsilyl]oxy} tetradecanoate (**27c** and **28c**, resp., *ca.* 4:1) was obtained. Their formation has been rationalized *via* nucleophilic ring opening of azlactone **25a** by Li butanolate (BuOLi) and Li but-3-enolate, respectively. Small amounts of the latter are formed by the reaction of THF with BuLi/HMPT at –30 to 20° [16].



27c R = $(CH_2)_3Me$
28c R = $(CH_2)_2CH{=}CH_2$

presence of the lactone carbonyl O-atom on both sides of the quaternary C-atom of the 15-membered ring. The ratio of the site occupation factor of the disordered sites is approximately 10:1. The NH group of each molecule acts as a donor for intermolecular H-bonds. The corresponding acceptor atom is the O-atom of the amide group of a neighboring molecule [N(1)–H···O(3′); H···O = 2.12(2) Å, N–H···O 172(2)°]. These H-bonds link the molecules into extended chains which run parallel to the [001] direction and can be described by a graph set motif of C(4) [22].

Under the same reaction conditions, oxazolones **25b** and **25d** – **25h**, used as mixtures with the corresponding oxazoles **26**, as well as the pure oxazolone **25i**, were transformed into the 14-, 13-, 11-, 10-, 9-, 6-, and 5-membered lactones **23** (*Scheme 4*, *Table 2*). The 6- and 5-membered lactones **23h** and **23i** were obtained in very good yields, and also the yields of the large-ring lactones **23b** and **23c** (14- and 15-membered, resp.) were good. As expected, the formation of the medium-sized rings was more difficult, and the products were isolated in fair-to-low yields, with the lowest one (10%) for the 11-membered lactone **23e**. Furthermore, a comparison of the formation of the 14-membered lactone **23b** *via* 'direct amide cyclization' of diamide **21b** (*Scheme 3*) and *via* 'ring transformation' of 4-(11-hydroxyundecyl)-1,3-oxazol-5(4*H*)-one **25b** indicate that the first type of reaction is more efficient (86 *vs.* 69% yield).

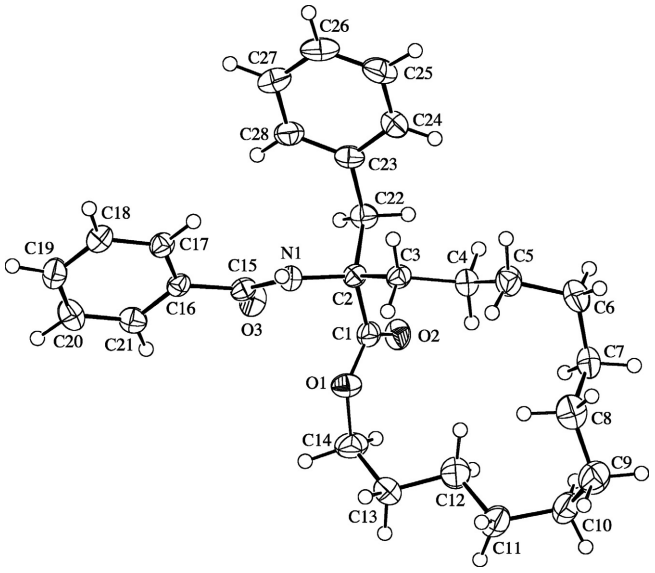


Fig. 2. ORTEP Plot [18] of the molecular structure of the 15-membered lactone **23c** (major conformation; 50% probability ellipsoids, arbitrary numbering of atoms).

Table 2. Synthesis of lactones **23** *via* ring transformation of oxazolones **25**[a]) (*Scheme 4*)

Oxazolone **25**	*n*	Lactone **23**	Ring size [*n* + 3]	Yield [%][b])
b	11	**b**	14	69
c	12	**c**	15	65[c])
d	10	**d**	13	51
e	8	**e**	11	10
f	7	**f**	10	20
g	6	**g**	9	51
h	3	**h**	6	86
i	2	**i**	5	100

[a]) *ca.* 1.9 mM solution of **25** in toluene, *ca.* 1.7 equiv. of TBAF · 3 H_2O, 90 – 110°, bubbling HCl gas through the solution for 1.5 – 2 h. [b]) Yield of isolated product; calculated on the amount of **25** in the used mixture of **25**/**26**. [c]) In one experiment (*ca.* 1 h), the reaction was not complete. In addition to lactone **23c** (35%), 43% of the corresponding deprotected 1,3-oxazol-5(4*H*)-one was isolated.

The structures of the racemic lactones **23d** – **23f** were also established by X-ray crystallography (*Fig. 3*). In **23f**, the amide NH group forms an intermolecular H-bond with the C=O O-atom of the lactone group of a neighboring molecule [N(1)–H···O(1′); H···O = 2.32(2) Å, N–H···O = 150(2)°]. These interactions link pairs of molecules into centrosymmetric dimers. It is worth mentioning that in the crystal structures of **23a**, **23b**, **23d**, and **23e**, the amide NH group is not involved in intermolecular H-bonds. In all cases, the H-atom of the NH group is located close to the O-atom of the lactone C=O group, most likely resulting from geometric restriction rather than being real H-bonds. Furthermore, in all lactones except **23e**, the Ph ring of the benzamido group is not coplanar with the NH–C=O group.

Conclusions

This study demonstrates that macrocyclic lactones of type **23** can be prepared in fair-to-good yields from 2-benzamido-2-benzyl-*N*,*N*-dimethyl-ω-hydroxyalkanamides **21** by treatment with HCl gas in boiling toluene, *i.e.*, *via* 'direct amide cyclization'. This reaction occurs *via* formation of a 4-(ω-hydroxyalkyl)-substituted 1,3-oxazol-5(4*H*)-one hydrochloride **22** as an intermediate, in analogy to the reaction of the corresponding ω-aminoalkanamides to give macrocyclic lactams [13]. The precursors **21** are accessible by C-alkylation of 4-benzyl-2-phenyl-1,3-oxazol-5(4*H*)-one (**15**), subsequent ring opening with Me_2NH, and deprotection of the THP-protected OH group. Changing the protecting group of the primary alcohol to the TBDMS-group allows the direct transformation of the 1,3-oxazol-5(4*H*)-ones **25** to the lactones **23**. In this case, the intermediate **22** is formed *via* F^- mediated deprotection of the primary alcohol followed by treatment with HCl gas in boiling toluene.

These results extend the use of 1,3-oxazol-5(4*H*)-ones as reactive building blocks and intermediates in organic synthesis. Intramolecular reactions with a nucleophilic group attached to the amino acid residue at C(2) are the key step in the preparation of cyclopeptides [12] and cyclodepsipeptides [9][10] containing α,α-disubstituted α-amino acids, in which the 1,3-oxazol-5(4*H*)-ones are formed as intermediates. On the other hand, the 'direct amide cyclization' of 2-acylamino acid *N*,*N*-dimethylamides of type **21** with a ω-amino- or ω-hydroxyalkyl substituent in the α-position leads to lactams and lactones, respectively, *via* an intermediate 1,3-oxazol-5(4*H*)-one bearing the nucleophile-containing side chain at C(4). In this study,

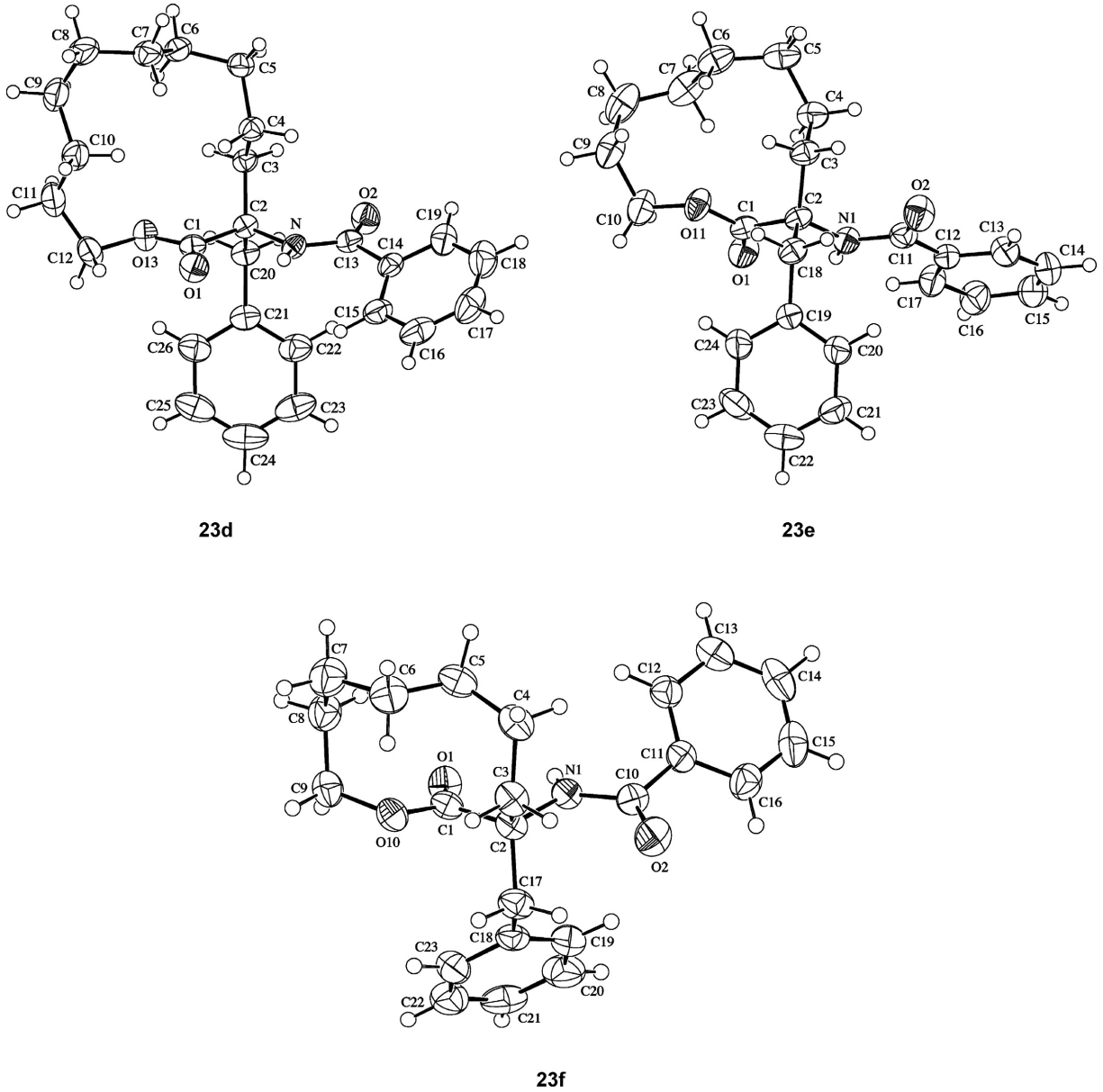


Fig. 3. ORTEP Plots [18] of the molecular structures of the lactones **23d** – **23f** (50% probability ellipsoids, arbitrary numbering of atoms).

1,3-oxazol-5(4*H*)-ones with a protected OH group in the side chain at C(4) are used as starting materials for the first time.

We thank the analytical sections of our institute for spectra and analyses, and *F. Hoffmann-La Roche AG,* Basel, for financial support.

Experimental Part

1. *General*. See [13][16]. Solvents were purified by standard procedures. TLC: *Merck* (Darmstadt, Germany) TLC glass plates, silica gel $60F_{254}$. Flash column chromatography (FC [21]): silica gel *60* (*Merck*), 0.040 – 0.060 mm. M.p.: *Mettler-FP-5* apparatus (Greifensee, Switzerland); uncorrected. IR Spectra: *Perkin Elmer-297* or *781* spectrometer (Schwerzenbach, Switzerland), in $CHCl_3$ (3% soln.) if not otherwise indicated; $\tilde{\nu}$ in cm^{-1}. ^1H- and ^{13}C-NMR spectra: *Bruker AC-300*, *Bruker ARX-300* or *Bruker AM-400* spectrometer (Fällanden, Switzerland) at 300 or 400 (^1H) and 75.5 or 100 MHz (^{13}C), resp., in $CDCl_3$; multiplicities of ^{13}C signals determined by the DEPT technique; δ in ppm rel. to Me_4Si as internal standard, *J* in Hz. ESI-MS: *Finnigan TSQ-700* instrument (Langenselbold, Germany); EI (70 eV) and CI (with isobutane): *Finnigan SSQ-700* instrument; *m*/*z* (rel. %).

General Procedure 1 (*GP 1; Alkylation of 4-Benzyl-2-phenyl-1,3-oxazol-5(4*H*)-one* (**15**)). To a stirred soln. of LDA[5]) in THF/HMPT (*ca.* 2:1 – 4:1) at *ca.* −78°, a THF

[5]) Either freshly prepared from BuLi and $^iPr_2NH$ in THF or purchased from *Fluka AG* (Buchs, Switzerland).

soln. of **15** was added, followed by the O-protected ω-iodoalkanol[6]). Then, the temp. was increased to 15 – 20° and the mixture stirred for 5 – 16 h. The mixture was extracted with cold H_2O (0°, 2×) and Et_2O (2×), and dried ($MgSO_4$). Separation of the products by FC (hexane/Et_2O 98:2 – 3:2) gave mixtures of the desired oxazolones **17** or **25** (major product) and the O-alkylated oxazoles **18** or **26**, as well as butyl and but-3-enyl esters of type **19**, **27**, and **28**.

General Procedure 2 (*GP 2; Ring Opening of Oxazolones* **17** *with Me2NH*). To a soln. of a mixture of 1,3-oxazol-5(4*H*)-one **17** and 1,3-oxazole **18** in MeCN at 24°, condensed Me_2NH was added. After stirring for 2 – 5 h at 22 – 25°, the crude mixture of **20** and **18** was purified by FC (Et_2O/hexane 3:2) to give THP-protected diamide **20**.

General Procedure 3 (*GP 3; Deprotection of the THP-ethers* **20**). A soln. of the protected alcohol **20** (1 equiv.) and pyridinium *p*-toluenesulfonate (Py · TsOH, 0.17 equiv.) in EtOH was heated at reflux for 2 h. After cooling to 24°, the solvent was evaporated, the residue was dissolved in CH_2Cl_2, the soln. washed with sat. aq. Na_2CO_3 soln. (2×), and dried ($MgSO_4$). After filtration and evaporation of the solvent, the product **21** was purified by FC (SiO_2; Et_2O).

General Procedure 4 (*GP 4; Direct Amide Cyclization of* **21**). Dry HCl gas[7]) was bubbled through a 1.9 mM soln. of **21** in toluene at 90 – 110° for 1.5 – 2 h. Then, the solvent was evaporated, and the crude product was purified by FC (SiO_2; hexane/Et_2O 4:1 – 1:1) to give lactone **23**.

General Procedure 5 (*GP 5; Ring Transformation of* **25**). Dry HCl gas[7]) was bubbled through a 1.9 mM soln. of a mixture of **25** and **26** in toluene containing 1.7 equiv. of TBAF · 3 H_2O at 90 – 110° for 1.5 – 2 h. Evaporation of the solvent and FC of the residue (SiO_2, hexane/Et_2O 16:9 or 4:1) gave lactone **23**.

2. *Alkylations of 4-Benzyl-2-phenyl-1,3-oxazol-5(4*H*)-one* (**15**) [15]. 2.1. *With Tetrahydro-2-[(9-iodononyl)oxy]pyran* (**16a**) [16]. According to *GP1*, a mixture of **15** (0.578 g, 2.30 mmol) in THF (5 ml), LDA (2.80 mmol) in THF (6 ml) and HMPT (5 ml), and **16a** (0.980 g, 2.77 mmol) in THF (5 ml) gave an oily mixture of **4-benzyl-2-phenyl-4-[9-(tetrahydro-2*H*-pyran-2-yloxy)nonyl]-1,3-oxazol-5(4*H*)-one** (**17a**) and **4-benzyl-2-phenyl-5-{[9-(tetrahydro-2*H*-pyran-2-yloxy)nonyl]oxy}-1,3-oxazole** (**18a**; 0.366 g, 33%).

Data of the mixture of **17a**/**18a** (2.2:1): R_f (Et_2O/hexane 1:1): 0.48. IR ($CHCl_3$): 3060*w*, 3020*w*, 2930*s*, 2860*s*, 1815*s*, 1655*s*, 1450*m*, 1440*m*, 1320*m*, 1290*m*, 1135*m*, 1120*m*, 1075*m*, 1060*m*, 1025*s*, 975*m*, 785*s*, 730*s*, 700*s*, 670*s*. CI-MS (isobutane): 478 (12, $[M + 1]^+$), 394 (11), 133 (100), 85 (19). Anal. calc. for $C_{30}H_{39}NO_4$ (477.65): C 75.12, H 8.62, N 2.92; found: C 74.99, H 8.52, N 2.74.

Data for **17a**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.90 – 7.80 (*m*, 2 arom. H); 7.55 – 7.45 (*m*, arom. H); 7.45 – 7.35 (*m*, 3 arom. H); 7.35 – 7.25 (*m*, arom. H); 7.20 – 7.10 (*m*, 3 arom. H); 4.57 (*dd*-like, J = 6.9, 3.8, OCHO); 3.95 – 3.80 (*m*, OCH_{eq}); 3.71 (*td*, J = 6.9, 9.5, 1 H of CH_2O); 3.55 – 3.45 (*m*, OCH_{ax}); 3.36 (*dt*, J = 6.7, 9.6, 1 H of CH_2O); 3.21, 3.13 (*AB*, J = 13.3, PhCH_2); 2.05 – 1.95 (*m*, 2 H); 1.90 – 1.75 (*m*, 2 H); 1.80 – 1.65 (*m*, 2 H); 1.60 – 1.45 (*m*, 8 H); 1.45 – 1.15 (*m*, 8 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.6 (*s*, C=O); 159.6 (*s*, C=N); 139.3, 134.3 (2*s*, 2 arom. C); 132.3 (*d*, arom. CH); 130.0 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 127.6 (*d*, 2 arom. CH); 126.9 (*d*, arom. CH); 98.6 (*d*, OCHO); 74.7 (*s*, C(4)); 67.4, 62.1 (2*t*, 2 CH_2O); 43.7 (*t*, Ph*C*H_2); 37.3, 30.6, 29.6 (3*t*, 3 CH_2); 29.2 (*t*, 2 CH_2); 29.1, 29.0, 26.0, 25.4, 23.9, 19.5 (6*t*, 6 CH_2).

Selected data of **18a**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 7.60 – 7.55 (*m*, arom. H); 4.10 (*t*, J = 6.6, CH_2O); 3.84 (*s*, PhCH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 129.3 (*s*, arom. C); 128.4 (*d*, 2 arom. CH); 128.2 (*d*, 2 arom. CH); 128.1, 127.7, 125.6 (3*d*, 5 arom. CH); 98.6 (*d*, OCHO); 74.6 (*t*, CH_2O); 31.0, 29.5, 29.4, 28.9, 26.1, 25.53 (6*t*, CH_2); 25.45 (*t*, 2 CH_2); 25.3 (*t*, CH_2).

2.2. *With Tetrahydro-2-[(11-iodoundecyl)oxy]pyrane* (**16b**) [16]. According to *GP1*, a mixture of **15** (2.000 g, 7.96 mmol) in THF (20 ml), LDA (9.60 mmol) in THF (30 ml) and HMPT (20 ml), and **16b** (3.670 g, 9.60 mmol) in THF (20 ml) gave an oily mixture of **4-benzyl-2-phenyl-4-[11-(tetrahydro-2*H*-pyran-2-yloxy)undecyl]-1,3-oxazol-5(4*H*)-one** (**17b**) and **4-benzyl-2-phenyl-5-{[11-(tetrahydro-2*H*-pyran-2-yloxy)undecyl]oxy}-1,3-oxazole** (**18b**; 1.584 g, 39%) as well as **butyl 2-benzamido-2-benzyl-13-(tetrahydro-2*H*-pyran-2-yloxy)tridecanoate** (**19**, 0.631 g, 11%).

Data of the mixture of **17b**/**18b** (15:4): R_f (Et_2O/hexane 1:1): 0.51. IR ($CHCl_3$): 3060*w*, 3010*m*, 2930*s*, 2860*s*, 1815*s*, 1655*s*, 1495*m*, 1465*m*, 1455*m*, 1440*m*, 1350*m*, 1320*m*, 1290*m*, 1135*m*, 1120*m*, 1075*s*, 1025*s*, 970*m*, 895*m*, 700*s*. CI-MS (isobutane): 506 (100, $[M + 1]^+$), 422 (23), 85 (13). Anal. calc. for $C_{32}H_{43}NO_4$ (505.70): C 76.00, H 8.57, N 2.77; found: C 76.01, H 8.63, N 2.90.

Data for **17b**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.90 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.25 – 7.10 (*m*, 5 arom. H); 4.57 (*dd*-like, J = 4.3, 2.6, OCHO); 3.90 – 3.85 (*m*, 1H of CH_2O); 3.72 (*dtd*, J = 11.7, 6.9, 4.1, OCH_{eq}); 3.55 – 3.45 (*m*, 1 H of CH_2O); 3.45 – 3.30 (*m*, OCH_{ax}); 3.21, 3.13 (*AB*, J = 13.4, PhCH_2); 2.05 – 1.10 (*m*, 13 CH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.6 (*s*, C=O); 159.6 (*s*, C=N); 134.3 (*s*, arom. C); 132.8 (*d*, arom. CH); 130.0 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 127.6 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 125.3 (*s*, arom. C); 98.6 (*d*, OCHO); 74.7 (*s*, C(4)); 67.5, 62.1 (2*t*, 2 CH_2O); 43.7 (*t*, Ph*C*H_2); 37.3, 30.7, 29.6 (3*t*, 3 CH_2); 29.3 (*t*, 5 CH_2); 29.1, 26.1, 25.4, 23.9, 19.5 (5*t*, 5 CH_2).

Selected data of **18b**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 7.35 – 7.25 (*m*, 2 arom. H);

[6]) In some cases, mixtures of ω-iodo and ω-chloro derivatives were used [16].

[7]) HCl gas was bubbled through conc. H_2SO_4.

4.11 (*t*, *J* = 6.6, CH_2O); 3.84 (*s*, PhCH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 139.5 (*s*, arom. C); 129.3 (*d*, 2 arom. CH); 128.2 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 126.0 (*d*, 2 arom. CH); 125.6 (*d*, arom. CH); 98.6 (*d*, OCHO); 74.6 (*t*, CH_2O); 31.0, 25.5 (2*t*, CH_2).

Data of **19**: IR ($CHCl_3$): 3410*w*, 3005*m*, 2930*s*, 2860*m*, 1725*m*, 1660*s*, 1515*s*, 1485*m*, 1465*m*, 1455*m*, 1440*m*, 1350*m*, 1240*m*, 1200*m*, 1130*m*, 1075*m*, 1030*m*, 705*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.65 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H); 6.97 (*s*, NH); 4.57 (*dd*, *J* = 4.3, 2.8, OCHO); 4.27, 4.13 (2*td*, *J* = 6.6, 10.8, and 6.5, 10.8, resp., CH_2OC=O); 3.93, 3.17 (*AB*, *J* = 13.5, PhCH_2); 3.90 – 3.80, 3.55 – 3.45 (2*m*, CH_2O); 3.72, 3.37 (2*td*, *J* = 6.9, 9.6 and 6.7, 9.6, resp., CH_2O); 2.85 – 2.75 (*m*, 1 H); 2.00 – 1.85 (*m*, 1 H); 1.85 – 1.15 (*m*, 14 CH_2); 0.99 (*t*, *J* = 7.4, Me). ^{13}C-NMR (50 MHz, $CDCl_3$): 173.5 (*s*, OC=O); 166.5 (*s*, NC=O); 136.4, 135.3 (2*s*, 2 arom. C); 131.2 (*d*, arom. CH); 129.6 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 126.7 (*d*, 3 arom. CH); 98.7 (*d*, OCHO); 67.6 (*t*, CH_2O); 66.4 (*s*, C–N); 65.7 (*t*, *C*H_2OC=O); 62.2 (*t*, CH_2O); 40.6 (*t*, Ph*C*H_2); 35.3, 30.7, 30.5, 29.7 (4*t*, 4 CH_2); 29.43 (*t*, 2 CH_2); 29.38 (*t*, 3 CH_2); 29.3 (*t*, 2 CH_2); 26.1, 25.4, 24.3, 19.6, 19.1 (5*t*, 5 CH_2); 13.6 (*q*, Me). CI-MS (isobutane): 580 (5, $[M + 1]^+$), 538 (3), 496 (100).

2.3. *With 12-[(*tert-*Butyl)dimethylsilyloxy]-1-iodododecane* (**24c**) [16]. According to *GP1*, a mixture of **15** (0.395 g, 1.57 mmol) in THF (3.8 ml), LDA (1.57 mmol) in THF (2.5 ml) and HMPT (2.5 ml), and **24c** (0.836 g, 1.96 mmol) in THF (2.5 ml) gave **4-benzyl-4-(12-{[*tert*-butyl(dimethyl)silyl]oxy}dodecyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25c**; 0.300 g, 35%) as an oil and 0.141 g (*ca.* 14%) of an inseparable mixture of **butyl** and **but-3-en-1-yl 2-benzamido-2-benzyl-14-{[*tert*-butyl(dimethyl)silyl]oxy} tetradecanoate** (**27c** and **28c**, resp.).

Data of **25c**: R_f (Et_2O/hexane 1:1): 0.67. IR ($CHCl_3$): 3060*w*, 2930*s*, 2860*s*, 1810*s*, 1655*s*, 1470*m*, 1460*m*, 1450*m*, 1320*m*, 1290*m*, 1255*m*, 1095*m*, 1045*m*, 970*m*, 895*m*, 840*s*, 700*s*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.85 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, arom. H); 7.45 – 7.40 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 5 arom. H); 3.59 (*t*, *J* = 6.6, CH_2O); 3.22, 3.14 (*AB*, *J* = 13.3, PhCH_2); 2.05 – 1.95, 1.55 – 1.45 (2*m*, 2 CH_2); 1.25 – 1.10 (*m*, 9 CH_2); 0.89 (*s*, Me_3C); 0.04 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.7 (*s*, C=O); 159.7 (*s*, C=N); 134.3 (*s*, arom. C); 132.3 (*d*, arom. CH); 130.1 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 127.7 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 125.7 (*s*, arom. C); 74.8 (*s*, C(4)); 63.2 (*t*, CH_2O); 43.8 (*t*, Ph*C*H_2); 37.4, 32.8, 29.6 (3*t*, 3 CH_2); 29.5 (*t*, 2 CH_2); 29.4 (*t*, 3 CH_2); 29.2 (*t*, CH_2); 25.9 (*q*, *Me*$_3$C); 25.7, 24.0 (2*t*, 2 CH_2); 18.3 (*s*, Me_3CSi); −5.4 (*q*, Me_2Si). CI-MS (isobutane): 551 (100, $[M + 1]^+$), 493 (17). Anal. calc. for $C_{34}H_{51}NO_3Si$ (549.88): C 74.27, H 9.35, N 2.55; found: C 74.26, H 9.62, N 2.71.

Data of the mixture of **27c**/**28c** (3.8:1): R_f (Et_2O/hexane 1:1): 0.55. IR ($CHCl_3$): 3410*w*, 3000*w*, 2930*s*, 2860*s*, 1725*m*, 1660*s*, 1515*s*, 1485*m*, 1470*m*, 1460*m*, 1440*m*, 1255*m*, 1200*m*, 1095*m*, 840*s*, 705*m*.

Data of **27c**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H); 6.97 (br. *s*, NH); 4.35 – 4.30, 4.30 – 4.10 (2*m*, CH_2O); 3.93, 3.17 (*AB*, *J* = 13.5, PhCH_2); 3.59 (*t*, *J* = 6.6, CH_2OSi); 2.85 – 2.75 (*m*, 1 H); 2.00 – 1.90 (*m*, 1 H); 1.75 – 1.65 (*m*, 2 H); 1.60 – 1.40 (*m*, 5 H); 1.40 – 1.15 (*m*, 17 H); 0.99 (*t*, *J* = 7.3, Me); 0.89 (*s*, Me_3C); 0.04 (*s*, Me_2Si). CI-MS (isobutane): 625 (100, $[M + 1]^+$), 567 (18), 105 (44).

Selected data of **28c**: ^{1}H-NMR (300 MHz, $CDCl_3$): 6.94 (*s*, NH); 5.90 – 5.70 (*m*, CH=); 5.50 – 5.51 (*m*, CH_2=); 3.92 (*A* of *AB*, *J* = 13.5, 1 H of PhCH_2); 2.55 – 2.45 (*m*, 1 H). CI-MS (isobutane): 623 (29, $[M + 1]^+$).

2.4. *With 11-[(*tert-*Butyl)dimethylsilyloxy]-1-iodoundecane* (**24b**) [16]. According to *GP1*, a mixture of **15** (0.330 g, 1.31 mmol) in THF (2 ml), LDA (1.31 mmol) in THF (3 ml) and HMPT (2 ml), and **24b** (0.650 g, 1.58 mmol) in THF (2 ml) gave **4-benzyl-4-(11-{[*tert*-butyl(dimethyl)silyl]oxy}undecyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25b**) and **4-benzyl-5-[(11-{[*tert*-butyl(dimethyl)silyl]oxy}undecyl)oxy]-2-phenyl-1,3-oxazole** (**26b**) (0.360 g, 52%).

Data of the mixture of **25b**/**26b** (9:1): R_f (Et_2O/hexane 1:1): 0.64. IR ($CHCl_3$): 3010*w*, 2935*s*, 2860*s*, 1815*s*, 1660*m*, 1455*m*, 1290*m*, 1255*m*, 1095*m*, 1045*m*, 840*s*, 700*s*. CI-MS (isobutane): 536 (100, $[M + 1]^+$). Anal. calc. for $C_{33}H_{49}NO_3Si$ (535.85): C 73.93, H 9.22, N 2.61; found: C 73.87, H 9.29, N 2.79.

Data for **25b**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.85 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, 3 arom. H); 7.25 – 7.15 (*m*, 5 arom. H); 3.59 (*t*, *J* = 6.6, CH_2O); 3.22, 3.14 (*AB*, *J* = 13.4, PhCH_2); 2.05 – 1.95, 1.55 – 1.45 (2*m*, 2 CH_2); 1.30 – 1.20 (*m*, 8 CH_2); 0.89 (*s*, Me_3C); 0.04 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.7 (*s*, C=O); 159.7 (*s*, C=N); 134.4 (*s*, arom. C); 132.3 (*d*, arom. CH); 130.1 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 127.7 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 125.8 (*s*, arom. C); 74.8 (*s*, C(4)); 63.2 (*t*, CH_2O); 43.8 (*t*, Ph*C*H_2); 37.4, 32.8, 29.5 (3*t*, 3 CH_2); 29.4 (*t*, 4 CH_2); 29.2 (*t*, CH_2); 25.9 (*q*, *Me*$_3$C); 25.7, 24.0 (2*t*, 2 CH_2); 18.3 (*s*, Me_3CSi); −5.3 (*q*, Me_2Si).

Selected data of **26b**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 7.35 – 7.25 (*m*, 3 arom. H); 4.11 (*t*, *J* = 6.6, CH_2O); 3.84 (*s*, PhCH_2).

2.5. *With 10-[(*tert-*Butyl)dimethylsilyloxy]-1-iododecane* (**24d**) [16]. According to *GP1*, a mixture of **15** (0.542 g, 2.09 mmol) in THF (3.2 ml), LDA (2.09 mmol) in THF (5 ml) and HMPT (3.2 ml), and **24d** (1.000 g, 2.51 mmol) in THF (3.22 ml) gave an oily mixture of **4-benzyl-4-(10-{[*tert*-butyl(dimethyl)silyl]oxy}decyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25d**) and **4-benzyl-5-[(10-{[*tert*-butyl(dimethyl)silyl]oxy}decyl)oxy]-2-phenyl-1,3-oxazole** (**26d**; 0.662 g, 60%), as well as **but-3-en-1-yl 2-benzamido-2-**

benzyl-12-{[(*tert*-butyl)dimethylsilyl]oxy}dodecanoate (**28d**; 0.110 g, 9%).

Data of the mixture of **25d**/**26d** (10:1): R_f (Et_2O/hexane 1:1): 0.63. IR ($CHCl_3$): 3065*w*, 2940*s*, 2860*s*, 1815*s*, 1660*s*, 1500*m*, 1470*m*, 1465*m*, 1455*m*, 1325*m*, 1290*m*, 1260*m*, 1045*m*, 1025*m*, 840*s*, 700*s*. CI-MS (isobutane): 523 (100, $[M + 1]^+$). Anal. calc. for $C_{32}H_{47}NO_3Si$ (521.81): C 73.66, H 9.08, N 2.68; found: C 73.46, H 9.14, N 2.44.

Data for **25d**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.90 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.10 (*m*, 5 arom. H); 3.58 (*t*, *J* = 3.6, CH_2O); 3.21, 3.14 (*AB*, *J* = 13.4, PhCH_2); 2.05 – 1.95, 1.55 – 1.40 (2*m*, 2 CH_2); 1.35 – 1.10 (*m*, 7 CH_2); 0.88 (*s*, Me_3C); 0.04 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.7 (*s*, C=O); 159.7 (*s*, C=N); 134.4 (*s*, arom. C); 132.3 (*d*, arom. CH); 130.1 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 127.7 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 125.7 (*s*, arom. C); 74.8 (*s*, C(4)); 63.2 (*t*, CH_2O); 43.8 (*t*, Ph*C*H_2); 38.4, 37.4, 32.8, 29.5, 29.4 (5*t*, 5 CH_2); 29.3 (*t*, 2 CH_2); 25.9 (*q*, *Me*$_3$C); 25.7, 24.0 (2*t*, 2 CH_2); 18.3 (*s*, Me_3*C*Si); −5.3 (*q*, Me_2Si).

Selected data of **26d**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 4.11 (*t*, *J* = 6.6, CH_2O); 3.83 (*s*, PhCH_2); 3.60 (*t*, *J* = 6.6, CH_2OSi). ^{13}C-NMR (50 MHz, $CDCl_3$): 129.3 (*d*, arom. CH); 128.3 (*d*, 2 arom. CH); 127.8 (*d*, arom. CH); 125.7 (*d*, 2 arom. CH); 125.4 (*d*, arom. CH); 29.65, 29.59 (2*t*, CH_2).

Data of **28d**: R_f (Et_2O/hexane 1:1): 0.57. IR ($CHCl_3$): 3060*w*, 3010*m*, 2940*s*, 2860*s*, 1730*m*, 1660*s*, 1520*s*, 1485*s*, 1470*m*, 1255*m*, 1200*m*, 1095*m*, 835*s*, 705*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.50 – 7.35 (*m*, 3 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.10 – 7.00 (*m*, 2 arom. H); 6.94 (br. *s*, NH); 5.90 – 5.75 (*m*, CH=); 5.20 – 5.10 (*m*, CH_2=); 4.35 – 4.25, 4.20 – 4.10 (2*m*, CH_2OC=O); 3.92, 3.16 (*AB*, *J* = 13.5, PhCH_2); 3.58 (*t*, *J* = 6.6, CH_2OSi); 2.85 – 2.75 (*m*, 1 H); 2.50 – 2.45 (*m*, CH_2); 2.00 – 1.90 (*m*, 1 H); 1.55 – 1.15 (*m*, 16 H); 0.89 (*s*, Me_3C); 0.04 (*s*, Me_2Si). CI-MS (isobutane): 595 (100, $[M + 1]^+$).

2.6. *With 8-[(*tert*-Butyl)dimethylsilyloxy]-1-iodooctane* (**24e**) [16]. According to *GP1*, a mixture of **15** (0.389 g, 1.55 mmol) in THF (3.5 ml), LDA (1.9 mmol) in THF (3.5 ml) and HMPT (2.4 ml), and **24e** (0.700 g, 1.89 mmol) in THF (2 ml) gave **4-benzyl-4-(8-{[*tert*-butyl(dimethyl)silyl]oxy}octyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25e**) and **4-benzyl-5-[(8-{[*tert*-butyl(dimethyl)silyl]oxy}octyl)oxy]-2-phenyl-1,3-oxazole** (**26e**) (0.445 g, 58%) as an oily mixture.

Data of the mixture of **25e**/**26e** (8:1): R_f (Et_2O/hexane 1:1): 0.60. IR ($CHCl_3$): 3060*w*, 2930*s*, 2860*s*, 1815*s*, 1655*s*, 1470*m*, 1465*m*, 1450*m*, 1320*m*, 1290*m*, 1255*m*, 1095*s*, 1050*m*, 1020*m*, 1010*m*, 970*m*, 840*s*, 700*s*. CI-MS (isobutane): 495 (5, $[M + 1]^+$), 302 (100). Anal. calc. for $C_{30}H_{43}NO_3Si$ (493.76): C 72.98, H 8.78, N 2.84; found: C 73.21, H 8.98, N 2.91.

Data for **25e**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.85 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.10 (*m*, 5 arom. H); 3.57 (*t*, *J* = 6.6, CH_2O); 3.21, 3.14 (*AB*, *J* = 13.3, PhCH_2); 2.05 – 1.95, 1.55 – 1.45 (2*m*, 2 CH_2); 1.45 – 1.20 (*m*, 5 CH_2); 0.88 (*s*, Me_3C); 0.03 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.7 (*s*, C=O); 159.7 (*s*, C=N); 134.4 (*s*, arom. C); 132.3 (*d*, arom. CH); 130.1 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 127.7 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 125.7 (*s*, arom. C); 74.8 (*s*, C(4)); 63.1 (*t*, CH_2O); 43.8 (*t*, Ph*C*H_2); 37.4, 32.7, 29.3 (3*t*, 3 CH_2); 29.2 (*t*, 2 CH_2); 25.9 (*q*, *Me*$_3$C); 25.6, 24.0 (2*t*, 2 CH_2); 18.3 (*s*, Me_3*C*Si); −5.3 (*q*, Me_2Si).

Selected data of **26e**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 7.35 – 7.25 (*m*, 2 arom. H); 4.10 (*t*, *J* = 6.6, CH_2O); 3.84 (*s*, PhCH_2); 3.60 (*t*, *J* = 6.5, CH_2O).

2.7. *With 7-[(*tert*-Butyl)dimethylsilyloxy]-1-iodoheptane* (**24f**) [16]. According to *GP1*, a mixture of **15** (0.588 g, 2.34 mmol) in THF (3 ml), LDA (3.5 mmol) in THF (8 ml) and HMPT (5 ml), and **24f** (1.000 g, 2.81 mmol) in THF (3 ml) gave an oily mixture of **4-benzyl-4-(7-{[*tert*-butyl(dimethyl)silyl]oxy}heptyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25f**) and **4-benzyl-5-[(7-{[*tert*-butyl(dimethyl)silyl]oxy}heptyl)oxy]-2-phenyl-1,3-oxazole** (**26f**; 0.464 g, 42%) as well as **butyl 2-benzamido-2-benzyl-9-{[(*tert*-butyl)dimethylsilyl]oxy}nonanoate** (**27f**; 0.173 g, 14%).

Data of the mixture of **25f**/**26f** (23:4): R_f (Et_2O/hexane 1:1): 0.62. IR ($CHCl_3$): 3060*w*, 2935*s*, 2860*s*, 1815*s*, 1655*s*, 1495*m*, 1470*m*, 1460*m*, 1450*m*, 1320*m*, 1290*m*, 1255*m*, 1095*s*, 1050*m*, 1005*m*, 970*m*, 890*m*, 840*s*, 700*s*. EI-MS: 479 (12, M^+), 251 (19), 115 (13), 105 (100). Anal. calc. for $C_{29}H_{41}NO_3Si$ (479.73): C 72.61, H 8.61, N 2.92; found: C 72.71, H 8.49, N 2.88.

Data for **25f**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.85 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.15 – 7.10 (*m*, 5 arom. H); 3.56 (*t*, *J* = 6.6, CH_2O); 3.21, 3.13 (*AB*, *J* = 13.3, PhCH_2); 2.10 – 1.95 (*m*, CH_2); 1.50 – 1.00 (*m*, 5 CH_2); 0.89 (*s*, Me_3C); 0.02 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.8 (*s*, C=O); 159.8 (*s*, C=N); 139.6, 134.6 (2*s*, 2 arom. C); 132.4 (*d*, arom. CH); 130.1 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 128.1 (*d*, 2 arom. CH); 127.8 (*d*, 2 arom. CH); 127.1 (*d*, arom. CH); 74.9 (*s*, C(4)); 63.2 (*t*, CH_2O); 43.8 (*t*, Ph*C*H_2); 37.4, 32.7, 29.4, 29.1 (4*t*, 4 CH_2); 26.0 (*q*, *Me*$_3$C); 25.6, 24.0 (2*t*, 2 CH_2); 18.3 (*s*, Me_3*C*Si); −5.3 (*q*, Me_2Si).

Selected data of **26f**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 4.10 (*t*, *J* = 6.6, CH_2O); 3.81 (*s*, PhCH_2); 3.60 (*t*, *J* = 6.4, CH_2O); 0.89 (*s*, Me_3C); 0.05 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 129.4 (*s*, arom. C); 128.6 (*s*, 2 arom. C); 128.3 (*s*, arom. C); 125.4 (*s*, C=N); 74.7 (*t*, CH_2O); 31.1, 29.4, 29.1, 26.1, 25.7, 25.0 (6*t*, 6 CH_2).

Data of **27f**: R_f (Et_2O/hexane 1:1): 0.55. IR ($CHCl_3$): 3410*w*, 3060*w*, 3020*w*, 3005*w*, 2930*s*, 2860*s*, 1725*m*, 1660*s*, 1515*m*, 1485*m*, 1470*m*, 1320*m*, 1255*m*, 1095*s*, 840*s*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H);

7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H); 6.97 (br. *s*, NH); 4.30 – 4.20, 4.15 – 3.95 (2*m*, CH_2O); 3.93, 3.16 (*AB*, *J* = 13.5, PhCH_2); 3.65 – 3.55 (*m*, CH_2OSi); 2.85 – 2.80 (*m*, 1 H); 2.00 – 1.90 (*m*, 1 H); 1.70 – 1.60 (*m*, CH_2); 1.55 – 1.25 (*m*, 6 CH_2); 0.96 (*t*, *J* = 7.4, Me); 0.88 (*s*, Me_3C); 0.03 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 173.5 (*s*, C=O); 166.4 (*s*, NC=O); 136.4, 135.3 (2*s*, 2 arom. C); 131.2 (*d*, arom. CH); 129.6 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 128.4 (*d*, 2 arom. CH); 126.7 (*d*, 3 arom. CH); 66.4 (*t*, CH_2O); 65.7 (*s*, C–N); 63.1 (*t*, CH_2O); 40.6 (*t*, PhCH_2); 35.3, 32.7, 29.3, 29.2, 28.9, 28.4 (6*t*, 6 CH_2); 25.9 (*q*, Me_3C); 25.6, 24.3 (2*t*, 2 CH_2); 18.2 (*s*, Me_3CSi); −5.4 (*q*, Me_2Si). EI-MS: 497 (12, $[M - 57]^+$), 463 (8), 105 (75). Anal. calc. for $C_{33}H_{51}NO_4Si$ (553.86): C 71.57, H 9.28, N 2.53; found: C 70.62, H 9.89, N 2.32.

2.8. *With 6-[(*tert*-Butyl)dimethylsilyloxy]-1-iodohexane* (**24g**) [16]. According to *GP1*, a mixture of **15** (0.490 g, 1.95 mmol) in THF (5 ml), LDA (1.95 mmol) in THF (5 ml) and HMPT (3 ml), and **24g** (0.800 g, 2.34 mmol) in THF (5 ml) gave an oily mixture of **4-benzyl-4-(6-{[*tert*-butyl(dimethyl)silyl]oxy}hexyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25g**) and **4-benzyl-5-[(6-{[*tert*-butyl(dimethyl)silyl]oxy}hexyl)oxy]-2-phenyl-1,3-oxazole** (**26g**; 0.270 g, 30%) as well as **butyl 2-benzamido-2-benzyl-8-{[(*tert*-butyl)dimethylsilyl]oxy}octanoate** (**27g**; 0.133 g, 13%).

Data of the mixture of **25g**/**26g** (20:3): R_f (Et_2O/hexane 1:1): 0.64. IR ($CHCl_3$): 3060*w*, 3020*w*, 3000*w*, 2935*s*, 2860*s*, 1810*m*, 1655*s*, 1495*m*, 1470*m*, 1460*m*, 1450*m*, 1320*m*, 1290*m*, 1255*s*, 1095*s*, 1050*m*, 1025*m*, 1005*m*, 990*m*, 965*m*, 840*s*, 810*m*, 700*s*, 665*m*, 630*m*. CI-MS: 466 (100, $[M + 1]^+$). Anal. calc. for $C_{28}H_{39}NO_3Si$ (465.71): C 72.22, H 8.44, N 3.01; found: C 72.18, H 8.66, N 3.22.

Data for **25g**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.90 – 7.80 (*m*, 2 arom. H); 7.60 – 7.45 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.10 (*m*, 5 arom. H); 3.55 (*t*, *J* = 6.5, CH_2O); 3.21, 3.13 (*AB*, *J* = 13.3, PhCH_2); 2.05 – 1.95, 1.55 – 1.50 (2*m*, 2 CH_2); 1.50 – 1.10 (*m*, 3 CH_2); 0.87 (*s*, Me_3C); 0.02 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.6 (*s*, C=O); 159.7 (*s*, C=N); 134.3 (*s*, arom. C); 132.2 (*d*, arom. CH); 130.0 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.0 (*d*, 2 arom. CH); 127.7 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 125.7 (*s*, arom. C); 74.8 (*s*, C(4)); 63.0 (*t*, CH_2O); 43.7 (*t*, PhCH_2); 37.3, 32.6, 29.2 (3*t*, 3 CH_2); 25.9 (*q*, Me_3C); 25.4, 23.9 (2*t*, 2 CH_2); 18.2 (*s*, Me_3CSi); −5.4 (*q*, Me_2Si).

Selected data of **26g**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 7.35 – 7.25 (*m*, 2 arom. H); 4.10 (*t*, *J* = 6.6, CH_2O); 3.83 (*s*, PhCH_2); 3.60 (*t*, *J* = 6.5, CH_2O); 0.89 (*s*, Me_3C); 0.05 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 129.3 (*s*, arom. C); 128.3, 127.84, 127.78, 126.7, 126.1, 125.3 (6*d*, 10 arom. CH); 74.5 (*t*, CH_2O); 31.1, 29.3 (2*t*, 2 CH_2).

Data of **27g**: R_f (Et_2O/hexane 1:1): 0.56. IR ($CHCl_3$): 3410*w*, 3060*w*, 3000*m*, 2935*s*, 2860*s*, 1730*m*, 1660*s*, 1530*m*, 1517*s*, 1487*s*, 1470*m*, 1460*m*, 1390*m*, 1350*m*, 1285*m*, 1255*s*, 1200*m*, 1100*s*, 840*s*, 705*s*, 670*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H); 6.97 (br. *s*, NH); 4.30 – 4.20, 4.15 – 4.10 (2*m*, CH_2O); 3.93, 3.16 (*AB*, *J* = 13.5, PhCH_2); 3.56 (*t*, *J* = 6.6, CH_2OSi); 2.90 – 2.75 (*m*, 1 H); 2.05 – 1.95 (*m*, 1 H); 1.80 – 1.70 (*m*, 2 H); 1.55 – 1.40 (*m*, 4 H); 1.40 – 1.25 (*m*, 6 H); 0.99 (*t*, *J* = 7.3, Me); 0.88 (*s*, Me_3C); 0.03 (*s*, Me_2Si). CI-MS: 541 (100, $[M + 1]^+$), 105 (25).

2.9. *With 3-[(*tert*-Butyl)dimethylsilyloxy]-1-iodopropane* (**24h**) [16]. According to *GP1*, a mixture of **15** (0.709 g, 2.82 mmol) in THF (4.5 ml), LDA (2.70 mmol) in THF (6.8 ml) and HMPT (4.5 ml), and **24h** (1.049 g, 3.49 mmol) in THF (4.5 ml) gave an oily mixture of **4-benzyl-4-(3-{[*tert*-butyl(dimethyl)silyl]oxy}propyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25h**) and **4-benzyl-5-[(3-{[*tert*-butyl(dimethyl)silyl]oxy}propyl)oxy]-2-phenyl-1,3-oxazole** (**26h**; 0.477 g, 40%), as well as **butyl 2-benzamido-2-benzyl-5-{[(*tert*-butyl)dimethylsilyl]oxy}pentanoate** (**27h**; 0.222 g, 16%).

Data of the mixture of **25h**/**26h** (13:4): R_f (Et_2O/hexane 1:1): 0.59. IR ($CHCl_3$): 3060*w*, 3005*w*, 2960*s*, 2930*s*, 2890*m*, 2860*m*, 1815*s*, 1657*s*, 1495*m*, 1470*m*, 1460*m*, 1450*m*, 1320*m*, 1290*m*, 1260*s*, 1040*m*, 1025*m*, 970*m*, 945*m*, 890*m*, 840*s*, 810*m*, 700*s*, 670*m*. CI-MS: 424 (100, $[M + 1]^+$), 366 (20). Anal. calc. for $C_{25}H_{33}NO_3Si$ (423.63): C 70.88, H 7.85, N 3.31; found: C 70.84, H 8.04, N 3.51.

Data for **25h**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.85 – 7.80 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, arom. H); 7.45 – 7.30 (*m*, 2 arom. H); 7.20 – 7.05 (*m*, 5 arom. H); 3.60 (*t*, *J* = 6.3, CH_2O); 3.23, 3.15 (*AB*, *J* = 13.4, PhCH_2); 2.10 – 2.00, 1.60 – 1.35 (2*m*, 2 CH_2); 0.87 (*s*, Me_3C); 0.02 (*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.7 (*s*, C=O); 159.8 (*s*, C=N); 134.4 (*s*, arom. C); 132.5 (*d*, arom. CH); 130.1 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 128.1 (*d*, 2 arom. CH); 127.8 (*d*, 2 arom. CH); 127.1 (*d*, arom. CH); 125.4 (*s*, arom. C); 74.6 (*s*, C(4)); 62.5 (*t*, CH_2O); 43.7 (*t*, PhCH_2); 33.9, 27.4 (2*t*, 2 CH_2); 25.9 (*q*, Me_3C); 18.3 (*s*, Me_3CSi); −5.4 (*q*, Me_2Si).

Selected data of **26h**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.85 (*m*, 2 arom. H); 4.24 (*t*, *J* = 6.3, CH_2O); 3.83 (*s*, PhCH_2); 3.75 (*t*, *J* = 6.0, CH_2O). ^{13}C-NMR (50 MHz, $CDCl_3$): 129.6 (*s*, arom. C); 129.2, 128.8, 128.5, 128.4, 127.9, 125.7 (6*d*, 10 arom. CH); 125.5 (*s*, arom. C); 71.6, 59.1 (2*t*, 2 CH_2O); 45.9 (*t*, PhCH_2).

Data of **27h**: R_f (Et_2O/hexane 1:1): 0.55. IR ($CHCl_3$): 3405*w*, 3005*m*, 2960*s*, 2935*s*, 2860*m*, 1725*s*, 1660*s*, 1520*s*, 1470*m*, 1450*m*, 1390*m*, 1350*m*, 1285*m*, 1255*s*, 1200*s*, 1100*s*, 840*s*, 705*w*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.60 (*m*, 2 arom. H); 7.60 – 7.35 (*m*, 3 arom. H); 7.30 – 7.00 (*m*, 5 arom. H); 6.95 (br. *s*, NH); 4.35 – 4.20, 4.20 – 4.05 (2*m*, CH_2OC=O); 3.89, 3.20 (*AB*, *J* = 13.4, PhCH_2); 3.75 – 3.50 (*m*, CH_2OSi); 2.85 – 2.70 (*m*, 1 H); 2.20 – 2.00 (*m*, 1 H); 1.80 – 1.65 (*m*, CH_2); 1.65 – 1.40 (*m*, 1 H); 1.40 – 1.20 (*m*, 1 H); 0.96 (*t*, *J* = 7.4, Me); 0.85 (*s*, Me_3C); 0.00 (*s*, Me_2Si). CI-MS: 498 (100, $[M + 1]^+$), 440 (18), 105 (27).

2.10. *With 2-[(*tert*-Butyl)dimethylsilyloxy]-1-iodoethane* (**24i**) [16]. According to *GP1*, a mixture of **15** (0.309 g, 1.23 mmol) in THF (2 ml), LDA (1.20 mmol) in THF (3 ml) and HMPT (2 ml), and **24i** (0.414 g, 1.45 mmol) in THF (2 ml) gave pure **4-benzyl-4-(2-{[*tert*-butyl(dimethyl)silyl]oxy}ethyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (**25i**; 0.305 g, 61%), and pure **4-benzyl-5-[(2-{[*tert*-butyl(dimethyl)silyl]oxy}ethyl)oxy]-2-phenyl-1,3-oxazole** (**26i**; 0.048 g, 10%) as viscous oils.

Data of **25i**: R_f (Et_2O/hexane 1:1): 0.48. IR ($CHCl_3$): 3060*w*, 3020*w*, 2935*w*, 2860*w*, 1820*m*, 1655*m*, 1470*w*, 1450*w*, 1320*w*, 1290*w*, 1255*m*, 1225*m*, 1205*s*, 1110*m*, 1095*m*, 1020*m*, 980*w*, 895*w*, 835*m*, 780*s*, 670*s*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.95 – 7.90 (*m*, 2 arom. H); 7.60 – 7.55 (*m*, arom. H); 7.55 – 7.45 (*m*, 2 arom. H); 7.25 – 7.20 (*m*, 5 arom. H); 3.75 – 3.65 (*m*, CH_2O); 3.26, 3.19 (*AB*, *J* = 13.3, PhCH_2); 2.60 – 2.45 (*m*, 1 H); 2.23 (*dt*, *J* = 2.9, 13.9, 1 H); 0.85 (*s*, Me_3C); 0.00, –0.11 (2*s*, Me_2Si). ^{13}C-NMR (50 MHz, $CDCl_3$): 179.5 (*s*, C=O); 160.3 (*s*, C=N); 134.1 (*s*, arom. C); 132.2 (*d*, arom. CH); 130.3 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 127.6 (*d*, 2 arom. CH); 127.0 (*d*, arom. CH); 126.2 (*s*, arom. C); 71.6 (*s*, C(4)); 58.8 (*t*, CH_2O); 44.4 (*t*, Ph*C*H_2); 39.9 (*t*, CH_2); 25.6 (*q*, *Me*$_3$C); 18.0 (*s*, Me_3*C*Si); –6.0 (*q*, Me_2Si). CI-MS: 410 (100, $[M + 1]^+$), 352 (77), 324 (39), 289 (27), 250 (10). Anal. calc. for $C_{24}H_{31}NO_3Si$ (409.60): C 70.38, H 7.63, N 3.42; found: C 70.12, H 7.30, N 3.19.

Data of **26i**: R_f (Et_2O/hexane 1:1): 0.58. IR ($CHCl_3$): 3060*w*, 3030*w*, 2960*m*, 2935*m*, 1740*m*, 1680*m*, 1480*m*, 1470*m*, 1260*s*, 1225*m*, 1205*s*, 1130*m*, 1105*m*, 930*m*, 840*m*, 755*s* (br.), 670*s*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.85 – 7.80 (*m*, 2 arom. H); 7.35 – 7.25 (*m*, 3 arom. H); 7.25 – 7.15 (*m*, 4 arom. H); 7.15 – 7.05 (*m*, arom. H); 4.10 (*t*-like, *J* = 5.0, CH_2O); 3.80 (*t*-like, *J* = 5.0, CH_2OSi); 3.76 (*s*, PhCH_2); 0.82 (*s*, Me_3C); 0.00 (*s*, Me_2Si). CI-MS: 410 (100, $[M + 1]^+$). Anal. calc. for $C_{24}H_{31}NO_3Si$ (409.60): C 70.38, H 7.63, N 3.42; found: C 70.21, H 7.50, N 3.29.

3. *Ring Opening of Oxazolones* **17** *with Me*$_2$*NH*. 3.1. ***N*-[1-Benzyl-1-(*N*,*N*-dimethylcarbamoyl)-10-(tetrahydro-2*H*-pyran-2-yloxy)decyl]benzamide** (**20a**). A mixture of oxazolone **17a** and oxazole **18a** (0.200 g, 0.42 mmol of **17a**) dissolved in MeCN (13 ml) was treated with Me_2NH according to *GP 2* to give 0.193 g (89%) of **20a** as a colorless oil. R_f (Et_2O/hexane 1:1): 0.12. IR ($CHCl_3$): 3250*w*, 3060*w*, 3005*m*, 2930*s*, 2860*m*, 1655*m*, 1625*s*, 1505*s*, 1480*s*, 1455*m*, 1440*m*, 1395*m*, 1120*m*, 1075*m*, 1030*m*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 8.00 (br. *s*, NH); 7.60 – 7.80 (*m*, 2 arom. H); 7.50 – 7.35 (*m*, 3 arom. H); 7.20 – 7.10 (*m*, 3 arom. H); 7.05 – 6.95 (*m*, 2 arom. H); 4.55 (*dd*-like, *J* = 4.4, 2.7, OCHO); 4.12, 3.21 (*AB*, *J* = 14.3, PhCH_2); 3.90 – 3.80 (*m*, OCH_{eq}); 3.71 (*td*, *J* = 6.9, 9.6, 1 H of CH_2O); 3.55 – 3.40 (*m*, OCH_{ax}); 3.36 (*dt*, *J* = 6.7, 9.6, 1 H of CH_2O); 3.40 – 2.90 (*m*, 2 H, Me_2N); 2.05 – 1.00 (*m*, 10 CH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 171.0 (*s*, O=*C*NMe_2); 165.7 (*s*, PhC=O); 136.6, 135.6 (2*s*, 2 arom. C); 131.0 (*d*, arom. CH); 129.5 (*d*, 2 arom. CH); 128.3 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 126.7 (*d*, 2 arom. CH); 126.6 (*d*, arom. CH); 98.7 (*d*, OCHO); 67.4, 62.1 (2*t*, 2 CH_2O); 65.9 (*s*, C–N); 38.6 (*t*, Ph*C*H_2); 38.4 (*q*, Me_2N); 33.6, 30.7, 29.6, 29.4 (4*t*, 4 CH_2); 29.3 (*t*, 2 CH_2); 29.2, 26.1, 25.4, 24.2, 19.6 (5*t*, 5 CH_2). CI-MS (isobutane): 523 (<5, $[M + 1]^+$), 439 (24), 349 (100), 347 (10), 85 (14). Anal. calc. for $C_{32}H_{46}N_2O_4$ (522.73): C 73.53, H 8.87, N 5.36; found: C 73.30, H 8.65, N 5.16.

3.2. ***N*-[1-Benzyl-1-(*N*,*N*-dimethylcarbamoyl)-12-(tetrahydro-2*H*-pyran-2-yloxy)dodecyl]benzamide** (**20b**). A mixture of oxazolone **17b** and oxazole **18b** (1.026 g, 2.03 mmol of **17b**) dissolved in MeCN (50 ml) was treated with Me_2NH according to *GP 2* to give 1.110 g (88%) of **20b** as a colorless oil. R_f (Et_2O/hexane 1:1): 0.12. IR ($CHCl_3$): 3350*m*, 3060*w*, 3005*s*, 2930*s*, 2860*s*, 1660*s*, 1630*s*, 1580*m*, 1505*s*, 1480*s*, 1455*m*, 1445*m*, 1400*s*, 1250*m*, 1120*m*, 1075*m*, 1030*s*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.99 (br. *s*, NH); 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.35 (*m*, 3 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H); 4.57 (*dd*-like, *J* = 4.4, 2.8, OCHO); 4.13, 3.22 (*AB*, *J* = 14.2, PhCH_2); 3.90 – 3.85 (*m*, OCH_{eq}); 3.72 (*td*, *J* = 6.9, 9.6, 1 H of CH_2O); 3.55 – 3.45 (*m*, OCH_{ax}); 3.37 (*td*, *J* = 6.7, 9.6, 1 H of CH_2O); 3.45 – 2.85 (*m*, Me_2N); 2.00 – 1.15 (*m*, 13 CH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 171.0 (*s*, O=*C*NMe_2); 165.6 (*s*, PhC=O); 136.5, 135.6 (2*s*, 2 arom. C); 131.0 (*d*, arom. CH); 129.4 (*d*, 2 arom. CH); 128.3 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 126.6 (*d*, 2 arom. CH); 126.5 (*d*, arom. CH); 98.6 (*d*, OCHO); 67.5, 62.1 (2*t*, 2 CH_2O); 66.0 (*s*, C–N); 38.6 (*t*, Ph*C*H_2); 38.4 (*q*, Me_2N); 33.6, 30.6, 29.6 (3*t*, 3 CH_2); 29.4 (*t*, 5 CH_2); 29.2, 26.0, 25.3, 24.2, 19.5 (5*t*, 5 CH_2). CI-MS (isobutane): 552 (47, $[M + 1]^+$), 467 (100), 422 (53), 105 (44), 85 (35). Anal. calc. for $C_{34}H_{50}N_2O_4$ (550.80): C 74.14, H 9.15, N 5.09; found: C 74.04, H 9.29, N 5.31.

4. *Deprotection of the THP-ethers* **20**. 4.1. ***N*-[1-Benzyl-1-(*N*,*N*-dimethylcarbamoyl)-10-hydroxydecyl]benzamide** (**21a**). According to *GP 3*, a soln. of **20a** (0.170 g, 0.33 mmol) in EtOH (4 ml) was treated with Py · TsOH (0.014 g, 0.05 mmol) to give 0.101 g (70%) of **21a** as a colorless wax. R_f (Et_2O/hexane 1:1): 0.13. IR ($CHCl_3$): 3620*w*, 3350*w*, 3060*w*, 3005*m*, 2930*s*, 2860*m*, 1655*m*, 1625*s*, 1505*s*, 1480*s*, 1400*m*, 715*m*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 8.01 (br. *s*, NH); 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.35 (*m*, 3 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.00 – 6.95 (*m*, 2 arom. H); 4.12, 3.22 (*AB*, *J* = 14.3, PhCH_2); 3.61 (*t*, *J* = 6.5, CH_2O); 3.50 – 2.80 (br. *s*, 1 H, Me_2N); 2.05 – 1.90 (*m*, 1 H); 1.70 – 1.45 (*m*, 3 H); 1.45 – 1.20 (*m*, 12 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 171.0 (*s*, O=*C*NMe_2); 165.7 (*s*, PhC=O); 136.4, 135.4 (2*s*, 2 arom. C); 131.0 (*d*, arom. CH); 129.4 (*d*, 2 arom. CH); 128.3 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 126.6 (*d*, 2 arom. CH); 126.5 (*d*, arom. CH); 65.9 (*s*, C–N); 62.5 (*t*, CH_2O); 38.6 (*t*, Ph*C*H_2); 38.4 (*q*, Me_2N); 33.5, 32.5, 29.3, 29.2, 29.1, 29.0, 25.5, 24.1 (8*t*, 8 CH_2). CI-MS (isobutane): 439 (72, $[M + 1]^+$), 394 (100), 366 (8), 347 (22), 317 (6).

4.2. ***N*-[1-Benzyl-1-(*N*,*N*-dimethylcarbamoyl)-12-hydroxydodecyl]benzamide** (**21b**). According to *GP 3*, a soln. of **20b** (0.130 g, 0.24 mmol) in EtOH (3 ml) was treated

with Py · TsOH (0.010 g, 0.04 mmol) to give 0.080 g (71%) of **21b** as a colorless wax. R_f (Et_2O/hexane 1:1): 0.17. IR ($CHCl_3$): 3620*w*, 3350*w*, 3005*m*, 2930*s*, 2860*m*, 1655*m*, 1625*s*, 1505*s*, 1480*s*, 1400*m*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 8.00 (br. *s*, NH); 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.35 (*m*, 3 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H); 4.12, 3.22 (*AB*, J = 14.3, PhCH_2); 3.63 (*t*, J = 6.6, CH_2O); 3.45 – 2.85 (br. *s*, Me_2N); 2.05 – 1.90 (*m*, 1 H); 1.63 (br. *s*, OH); 1.60 – 1.50 (*m*, 2 H); 1.45 – 1.00 (*m*, 17 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 171.0 (*s*, O=*C*NMe$_2$); 165.7 (*s*, PhC=O); 136.4, 135.5 (2*s*, 2 arom. C); 131.0 (*d*, arom. CH); 129.4 (*d*, 2 arom. CH); 128.3 (*d*, 2 arom. CH); 127.9 (*d*, 2 arom. CH); 126.7 (*d*, 2 arom. CH); 126.6 (*d*, arom. CH); 66.1 (*s*, C–N); 62.6 (*t*, CH_2O); 38.7 (*t*, Ph*C*H$_2$); 38.4 (*q*, Me_2N); 33.6, 32.6 (2*t*, 2 CH_2); 29.3, 29.28, 29.2 (3*t*, 6 CH_2); 25.6, 24.2 (2*t*, 2 CH_2). CI-MS (isobutane): 467 (94, $[M + 1]^+$), 422 (100), 375 (8). Anal. calc. for $C_{29}H_{42}N_2O_3$ (466.67): C 74.64, H 9.07, N 6.00; found: C 74.23, H 9.09, N 6.16.

5. *Lactone-Formation* via *Direct Amide Cyclization of Diamides* **21**. 5.1. ***N*-(3-Benzyl-2-oxo-1-oxacyclododecan-3-yl)benzamide** (**23a**). The reaction of **21a** (0.085 g, 0.19 mmol) in toluene (101 ml) with HCl gas according to *GP 4* gave **23a** (0.032 g, 43%) as colorless crystals and **4-benzyl-4-(9-hydroxynonyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (0.041 g, 55%).

Data of **23a**: R_f (Et_2O/hexane 1:1): 0.47. M.p. 137.5 – 138.5°. IR ($CHCl_3$): 3410*w*, 3060*w*, 3010*w*, 2935*m*, 1725*m*, 1660*s*, 1520*s*, 1490*s*, 1470*m*, 1455*m*, 1350*m*, 1235*m*, 1200*m*, 720*s*, 705*s*, 670*s*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.50 – 7.35 (*m*, 3 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H, NH); 4.80 – 4.75, 4.00 – 3.90 (2*m*, CH_2O); 3.96, 3.22 (*AB*, J = 13.5, PhCH_2); 2.85 – 2.75, 2.25 – 2.05 (2*m*, 2 H); 1.70 – 1.60 (*m*, 3 H); 1.55 – 0.90 (*m*, 11 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 173.9 (*s*, C=O); 166.6 (*s*, NC=O); 136.6, 135.3 (2*s*, 2 arom. C); 131.4 (*d*, arom. CH); 129.6 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 128.2 (*d*, 2 arom. CH); 126.8 (*d*, 3 arom. CH); 67.2 (*s*, C(3)); 65.5 (*t*, CH_2O); 40.5 (*t*, Ph*C*H$_2$); 32.8, 26.3 (2*t*, 2 CH_2); 25.8 (*t*, 2 CH_2); 22.9, 22.1, 21.4, 20.7 (4*t*, 4 CH_2). CI-MS (isobutane): 395 (100, $[M + 1]^+$), 303 (94). Anal. calc. for $C_{25}H_{31}NO_3$ (393.53): C 76.30, H 7.94, N 3.56; found: C 76.28, H 7.98, N 3.42.

Suitable crystals for the X-ray crystal structure determination were grown from Et_2O/hexane.

Data of **4-benzyl-4-(9-hydroxynonyl)-2-phenyl-1,3-oxazol-5(4*H*)-one**: IR ($CHCl_3$): 3620*w*, 3060*w*, 3010*w*, 2930*s*, 2860*m*, 1815*s*, 1660*s*, 1455*m*, 1320*m*, 1290*m*, 1050*m*, 1025*m*, 980*m*, 700*s*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.90 – 7.80 (*m*, 2 arom. H); 7.60 – 7.50 (*m*, 1 arom. H); 7.50 – 7.40 (*m*, 2 arom. H); 7.20 – 7.10 (*m*, 5 arom. H); 3.62 (*t*, J = 6.6, CH_2O); 3.21, 3.14 (*AB*, J = 13.4, PhCH_2); 2.05 – 1.95, 1.60 – 1.50 (2*m*, 2 CH_2); 1.25 (br. *s*, 6 CH_2).

5.2. ***N*-(3-Benzyl-2-oxo-1-oxacyclotetradecan-3-yl)benzamide** (**23b**). The reaction of **21b** (0.250 g, 0.54 mmol) in toluene (165 ml) with HCl gas according to *GP 4* gave **23b** (0.196 g, 86%) as colorless crystals. R_f (Et_2O/hexane 1:1): 0.52. M.p. 115.8 – 116.0°. IR ($CHCl_3$): 3410*w*, 3020*w*, 3005*w*, 2935*s*, 2860*m*, 1725*m*, 1660*s*, 1515*s*, 1485*s*, 1460*m*, 1345*m*, 1235*m*, 1200*m*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, 1 arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.07 (br. *s*, NH); 7.05 – 7.00 (*m*, 2 arom. H); 4.45 (*td*-like, J = 10.7, 2.2, 1 H of CH_2O); 4.10 – 4.00 (*m*, 1 H of CH_2O); 3.99, 3.16 (*AB*, J = 13.4, PhCH_2); 2.86 (*td*-like, J = 13.4, 4.5, 1 H); 2.00 – 1.95 (*m*, 1 H); 1.75 – 1.55 (*m*, 2 H); 1.55 – 1.20 (*m*, 14 H); 1.20 – 0.95 (*m*, 2 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 174.0 (*s*, C=O); 166.5 (*s*, NC=O); 136.6, 135.4 (2*s*, 2 arom. C); 131.4 (*d*, arom. CH); 129.6 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 128.2 (*d*, 2 arom. CH); 127.0 (*d*, 3 arom. CH); 67.0 (*s*, C(3)); 65.0 (*t*, CH_2O); 41.0 (*t*, Ph*C*H$_2$); 34.7, 27.9, 26.1, 25.9, 25.8, 24.1, 24.0, 22.6, 22.5, 21.3 (10*t*, 10 CH_2). CI-MS (isobutane): 422 (100, $[M + 1]^+$), 330 (13). Anal. calc. for $C_{27}H_{35}NO_3$ (421.58): C 76.93, H 8.37, N 3.32; found: C 76.82, H 8.21, N 3.36.

Suitable crystals for the X-ray crystal structure determination were grown from Et_2O/hexane.

6. *Lactone-Formation* via *Ring Transformations of Oxazolones* **25**. 6.1. ***N*-(3-Benzyl-2-oxo-1-oxacyclopentadecan-3-yl)benzamide** (**23c**). Treatment of a soln. of **25c** (0.208 g, 0.35 mmol) in toluene (190 ml) with TBAF (0.194 g, 0.62 mmol) according to *GP 5* gave **23c** (0.108 g, 65%) as colorless crystals[8]). R_f (Et_2O/hexane 1:1): 0.55. M.p. 120.0 – 121.6°. IR ($CHCl_3$): 3410*w*, 3060*w*, 3010*w*, 2935*s*, 2860*m*, 1725*m*, 1660*s*, 1515*s*, 1487*s*, 1455*m*, 1390*m*, 1347*m*, 1285*m*, 1240*m*, 1200*m*, 705*s*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.65 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, 1 arom. H); 7.45 – 7.40 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H, NH); 4.60 – 4.50, 4.10 – 4.00 (2*m*, CH_2O); 3.95, 3.16 (*AB*, J = 13.4, PhCH_2); 2.85 – 2.80, 2.10 – 1.95 (2*m*, 2 H); 1.80 – 1.60 (*m*, CH_2); 1.50 – 1.05 (*m*, 9 CH_2). CI-MS (isobutane): 436 (100, $[M + 1]^+$), 344 (7). Anal. calc. for $C_{28}H_{37}NO_3$ (435.61): C 77.20, H 8.56, N 3.22; found: C 77.08, H 8.54, N 3.16.

Suitable crystals for the X-ray crystal structure determination were grown from Et_2O/hexane.

6.2. ***N*-(3-Benzyl-2-oxo-1-oxacyclotetradecan-3-yl)benzamide** (**23b**). Treatment of a soln. of the mixture **25b**/**26b** (0.200 g, 0.37 mmol, *i.e.*, 0.180 g, 0.34 mmol **25b**) in toluene (213 ml) with TBAF (0.221 g, 0.70 mmol) according to *GP 5* gave **23b** (0.099 g, 69%) as colorless crystals (see *5.2*).

6.3. ***N*-(3-Benzyl-2-oxo-1-oxacyclotridecan-3-yl)benzamide** (**23d**). Treatment of a soln. of the mixture **25d**/**26d** (0.199 g, 0.38 mmol, *i.e.*, 0.189 g, 0.36 mmol **25d**) in toluene (189 ml) with TBAF (0.212 g, 0.67 mmol)

[8]) In an experiment with incomplete transformation of **25c** (0.200 g, 0.36 mmol), in addition to 0.055 g (35%) of **23c**, was obtained **4-benzyl-4-(12-hydroxydodecyl)-2-phenyl-1,3-oxazol-5(4*H*)-one** (0.068 g, 43%).

according to *GP 5* gave **23d** (0.075 g, 51%) as colorless crystals. R_f (Et_2O/hexane 1:1): 0.49. M.p. 139.5 – 140.5°. IR ($CHCl_3$): 3410*w*, 3060*w*, 3000*w*, 2930*s*, 2860*m*, 1720*s*, 1660*s*, 1515*s*, 1485*s*, 1460*m*, 1385*m*, 1345*m*, 1285*m*, 1250*m*, 1195*m*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, 1 arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.05 – 7.00 (*m*, 2 arom. H, NH); 4.65 (*ddd*, *J* = 10.8, 9.3, 3.2, 1 H of CH_2O); 4.03 (*ddd*, *J* = 10.9, 4.9, 3.3, 1 H of CH_2O); 3.96, 3.18 (*AB*, *J* = 13.5, PhCH_2); 2.85 – 2.75, 2.15 – 2.05 (2*m*, 2 H); 1.80 – 1.50 (*m*, CH_2); 1.40 – 1.05 (*m*, 7 CH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 173.9 (*s*, C=O); 166.5 (*s*, NC=O); 136.5, 135.3 (2*s*, 2 arom. C); 131.4 (*d*, arom. CH); 129.6 (*d*, 2 arom. CH); 128.8 (*d*, arom. CH); 128.5 (*d*, 2 arom. CH); 128.1 (*d*, 2 arom. C); 126.8 (*d*, 2 arom. CH); 67.0 (*s*, C(3)); 66.2 (*t*, CH_2O); 40.9 (*t*, PhCH_2); 34.4, 27.9 (2*t*, 2 CH_2); 26.6 (*t*, 2 CH_2); 25.6, 24.9, 24.5, 24.0, 21.6 (5*t*, 5 CH_2). CI-MS (isobutane): 408 (100, $[M + 1]^+$). Anal. calc. for $C_{26}H_{33}NO_3$ (407.56): C 76.62, H 8.16, N 3.44; found: C 76.88, H 8.09, N 3.53.

Suitable crystals for the X-ray crystal structure determination were grown from Et_2O/hexane.

6.4. ***N*-(3-Benzyl-2-oxo-1-oxacycloundecan-3-yl)benzamide** (**23e**). Treatment of a soln. of the mixture **25e**/**26e** (0.348 g, 0.70 mmol, *i.e.*, 0.331 g, 0.67 mmol **25e**) in toluene (373 ml) with TBAF (0.383 g, 1.21 mmol) according to *GP 5* gave **23e** (0.026 g, 10%) as colorless crystals. R_f (Et_2O/hexane 1:1): 0.49. M.p. 150.5 – 151.5°. IR ($CHCl_3$): 3410*w*, 3050*w*, 3000*w*, 2940*m*, 2930*m*, 2855*w*, 1720*s*, 1660*s*, 1530*s*, 1485*s*, 1470*m*, 1395*m*, 1385*m*, 1360*m*, 1340*m*, 1230*m*, 1195*m*, 700*w*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, 1 arom. H); 7.45 – 7.40 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.08 (br. *s*, NH); 7.00 – 6.95 (*m*, 2 arom. H); 4.80 (*td*-like, *J* = 10.8, 1.3, 1 H of CH_2O); 4.00 (*ddd*, *J* = 11.2, 5.2, 1.8, 1 H of CH_2O); 3.90, 3.10 (*AB*, *J* = 13.4, PhCH_2); 3.00 – 2.90, 2.15 – 2.05 (2*m*, 2 H); 1.95 – 1.85 (*m*, 1 H); 1.70 – 1.50 (*m*, 5 H); 1.35 – 1.15 (*m*, 6 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 174.0 (*s*, C=O); 166.6 (*s*, NC=O); 136.3, 135.3 (2*s*, 2 arom. C); 131.3 (*d*, arom. CH); 129.5 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.1 (*d*, 2 arom. CH); 126.8 (*d*, 3 arom. CH); 67.0 (*s*, C(3)); 66.1 (*t*, CH_2O); 41.2 (*t*, PhCH_2); 30.6, 25.6, 25.2, 25.0, 23.9, 21.4, 20.3 (7*t*, 7 CH_2). CI-MS (isobutane): 380 (100, $[M + 1]^+$), 289 (18), 105 (53). Anal. calc. for $C_{24}H_{29}NO_3$ (379.51): C 75.96, H 7.70, N 3.69; found: C 75.88, H 7.93, N 3.53.

Suitable crystals for the X-ray crystal structure determination were grown from Et_2O/hexane.

6.5. ***N*-(3-Benzyl-2-oxo-1-oxacyclodecan-3-yl)benzamide** (**23f**). Treatment of a soln. of the mixture **25f**/**26f** (0.267 g, 0.56 mmol, *i.e.*, 0.227 g, 0.47 mmol **25f**) in toluene (295 ml) with TBAF (0.307 g, 0.97 mmol) according to *GP 5* gave **23f** (0.034 g, 20%) as colorless crystals. R_f (Et_2O/hexane 1:1): 0.43. M.p. 85.0 – 87.4°. IR ($CHCl_3$): 3405*s*, 3240*w*, 3060*w*, 3020*w*, 3005*w*, 2940*w*, 2860*w*, 1720*m*, 1660*s*, 1520*s*, 1490*s*, 1470*m*, 1455*m*, 1390*m*, 1370*m*, 1240*w*, 705*w*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.70 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, 1 arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.20 – 7.15 (*m*, 3 arom. H); 7.03 (br. *s*, NH); 7.00 – 6.95 (*m*, 2 arom. H); 4.83 (*ddd*, *J* ≈ 12.4, 5.3, 3.0, 1 H of CH_2O); 4.15 – 4.05 (*m*, 1 H of CH_2O); 3.93, 3.05 (*AB*, *J* = 13.5, PhCH_2); 3.00 – 2.95, 2.40 – 2.30 (2*m*, 2 H); 2.20 – 2.05 (*m*, 1 H); 1.85 – 1.25 (*m*, 8 H); 1.05 – 0.85 (*m*, 1 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 174.2 (*s*, C=O); 166.7 (*s*, NC=O); 136.0, 135.4 (2*s*, 2 arom. C); 131.3 (*d*, arom. CH); 129.6 (*d*, 2 arom. CH); 128.5 (*d*, 2 arom. CH); 128.1 (*d*, 2 arom. CH); 126.8 (*d*, 3 arom. CH); 68.0 (*t*, CH_2O); 65.5 (*s*, C(3)); 41.5 (*t*, PhCH_2); 29.9, 25.8, 24.9, 23.8, 23.1, 22.2 (6*t*, 6 CH_2). EI-MS: 365 (5, M^+), 274 (100), 105 (83), 91 (29), 77 (63). Anal. calc. for $C_{23}H_{27}NO_3$ (365.48): C 75.58, H 7.45, N 3.83; found: C 75.56, H 7.63, N 3.97.

Suitable crystals for the X-ray crystal structure determination were grown from Et_2O/hexane.

6.6. ***N*-(3-Benzyl-2-oxo-1-oxacyclononan-3-yl)benzamide** (**23g**). Treatment of a soln. of the mixture **25g**/**26g** (0.172 g, 0.37 mmol, *i.e.*, 0.160 g, 0.23 mmol **25g**) in toluene (200 ml) with TBAF (0.210 g, 1.21 mmol) according to *GP 5* gave **23g** (0.075 g, 51%) as colorless crystals. R_f (Et_2O/hexane 1:1): 0.56. M.p. 107.0 – 111.0°. IR ($CHCl_3$): 3410*w*, 3000*m*, 2930*m*, 2855*w*, 1730*s*, 1660*s*, 1515*s*, 1485*s*, 1460*m*, 1380*m*, 1345*m*, 1290*m*, 1240*m*, 1195*s*, 1085*m*, 970*m*, 925*m*, 880*m*, 700*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.50 – 7.45 (*m*, 1 arom. H); 7.45 – 7.35 (*m*, 2 arom. H); 7.25 – 7.15 (*m*, 3 arom. H); 7.15 – 7.05 (*m*, 2 arom. H); 6.83 (br. *s*, NH); 4.85 – 4.75 (*m*, 1 H of CH_2O); 4.15 (*dt*, *J* = 10.8, 6.7, 1 H of CH_2O); 3.81, 3.37 (*AB*, *J* = 13.6, PhCH_2); 2.65 – 2.55 (*m*, 1 H); 2.13 (*ddd*, *J* = 14.4, 6.6, 3.5, 1 H); 1.80 – 1.70 (*m*, 2 H); 1.70 – 1.55 (*m*, 2 H); 1.55 – 1.30 (*m*, 4 H). CI-MS (isobutane): 352 (100, $[M + 1]^+$). Anal. calc. for $C_{22}H_{25}NO_3$ (351.45): C 75.19, H 7.17, N 3.99; found: C 74.92, H 7.42, N 4.23.

6.7. ***N*-[3-Benzyltetrahydro-2-oxo-2*H*-pyran-3-yl]benzamide** (**23h**). Treatment of a soln. of the mixture **25h**/**26h** (0.181 g, 0.43 mmol, *i.e.*, 0.112 g, 0.26 mmol **25h**) in toluene (230 ml) with TBAF (0.238 g, 0.75 mmol) according to *GP 5* gave **23h** (0.069 g, 86%) as colorless crystals and **4-benzyl-5-[(3-hydroxypropyl)oxy]-2-phenyl-1,3-oxazole** (0.016 g, 30% with respect to **26h**).

Data of **23h**: R_f (Et_2O/hexane 1:1): 0.48. M.p. 155.7 – 156.7°. IR ($CHCl_3$): 3440*w*, 3060*w*, 3010*m*, 2970*w*, 2860*w*, 1730*s*, 1660*s*, 1510*s*, 1480*s*, 1455*m*, 1400*m*, 1280*m*, 1260*s*, 1170*s*, 1120*m*, 1100*m*, 1075*m*, 975*m*, 705*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.70 – 7.65 (*m*, 2 arom. H); 7.55 – 7.50 (*m*, 1 arom. H); 7.50 – 7.30 (*m*, 5 arom. H); 7.25 – 7.20 (*m*, 2 arom. H); 6.09 (br. *s*, NH); 4.65 – 4.55, 4.45 – 4.35 (2*m*, CH_2O); 3.52, 3.16 (*AB*, *J* = 13.3, PhCH_2); 2.45 – 2.40 (*m*, CH_2); 1.90 – 1.80 (*m*, CH_2). ^{13}C-NMR (50 MHz, $CDCl_3$): 172.4 (*s*, C=O); 166.6 (*s*, NC=O); 134.3, 133.6 (2*s*, 2 arom. C); 131.7 (*d*, arom. CH); 130.2 (*d*, 2 arom. CH); 128.7 (*d*, 2 arom. CH); 128.4 (*d*, 2 arom. CH); 127.6 (*d*, arom. C); 126.9 (*d*, 2 arom. CH); 69.8 (*t*, CH_2O); 58.9 (*s*, C(3)); 43.4 (*t*, PhCH_2); 31.2, 21.5 (2*t*, 2

Table 3. Crystallographic data for compounds **23a** – **23f**

	23a	**23b**	**23c**	**23d**	**23e**	**23f**
Crystallized from	Et_2O/hexane	Et_2O/hexane	Et_2O/hexane	Et_2O/hexane	Et_2O/hexane	Et_2O/hexane
Empirical formula	$C_{25}H_{31}NO_3$	$C_{27}H_{35}NO_3$	$C_{28}H_{37}NO_3$	$C_{26}H_{33}NO_3$	$C_{24}H_{29}NO_3$	$C_{23}H_{27}NO_3$
Formula weight	393.52	421.56	435.59	407.53	379.50	365.46
Crystal color, habit	Colorless, prism	Colorless, prism	Colorless, needle	Colorless, prism	Colorless, prism	Colorless, prism
Crystal dimensions [mm]	0.16 × 0.38 × 0.44	0.30 × 0.32 × 0.54	0.12 × 0.15 × 0.40	0.24 × 0.36 × 0.43	0.25 × 0.42 × 0.45	0.34 × 0.40 × 0.45
Temperature [K]	213 (1)	295 (1)	173 (1)	213 (1)	213 (1)	213 (1)
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$	*Pbca*	$P2_1/c$	$P2_1/c$	$P2_1/c$
Z	4	4	8	4	4	4
Reflections for cell determination	25	17	25	25	25	25
2θ range for cell determination [°]	20 – 22	24 – 30	27 – 37	27 – 30	28 – 33	30 – 32
Unit cell parameters:						
a [Å]	17.008 (4)	8.828 (3)	18.147 (5)	17.917 (2)	10.836 (1)	9.990 (1)
b [Å]	8.391 (2)	15.678 (5)	26.831 (5)	8.205 (1)	9.216 (1)	19.808 (3)
c [Å]	15.683 (4)	17.963 (5)	10.054 (5)	16.168 (3)	21.011 (3)	9.968 (2)
β [°]	100.25 (2)	96.29 (3)	90	107.32 (1)	97.47 (1)	99.89 (1)
V [Å^3]	2202.5 (9)	2471 (1)	4896 (3)	2269.1 (6)	2080.3 (5)	1943.4 (5)
D_x [g/cm^3]	1.187	1.133	1.182	1.193	1.212	1.249
μ(MoK_α) [mm^{-1}]	0.077	0.073	0.076	0.072	0.079	0.082
Scan type	ω	ω	$\omega/2\theta$	ω	ω	ω
2θ(max) [°]	50	50	55	55	55	55
Total reflections measured	4655	4997	7177	6604	4821	5106
Symmetry independent reflections	3874	4323	5601	5227	3778	4451
Reflections with $I > 2\sigma(I)$	1830	1946	2818	3082	2570	2883
Reflections used in refinement	3874	4303	5601	5226	3778	4451
Parameters refined; restraints	321; 181	284; 0	309; 0	276; 0	258; 0	248; 0
Final $R(F)$ [$I > 2\sigma(I)$ reflections]	0.0809	0.1152	0.0613	0.0496	0.0421	0.0498
$wR(F^2)$ (all data)	0.2540	0.4048	0.1503	0.1168	0.1014	0.1154
Weighting parameters [a; b][a]	0.0916; 2.1728	0.2; 0	0.0476; 0	0.2293; 0	0.0382; 0.4213	0.0368; 0.4747
Goodness of fit	1.034	1.207	1.003	1.043	1.023	1.016
Secondary extinction coefficient	–	–	0.0013 (4)	0.0046 (8)	0.0049 (7)	–
Final Δ_{max}/σ	0.036	0.001	0.001	0.001	0.001	0.001
$\Delta\rho$(max; min) [e Å^{-3}]	0.40; −0.17	0.44; −0.51	0.34; −0.22	0.16; −0.16	0.17; −0.13	0.19; −0.19

[a]) $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$ where $P = (F_o^2 + 2\,F_c^2)/3$.

CH_2). CI-MS (isobutane): 310 (100, $[M + 1]^+$). Anal. calc. for $C_{19}H_{19}NO_3$ (309.37): C 73.77, H 6.19, N 4.53; found: C 74.02, H 6.38, N 4.65.

Data of **4-Benzyl-5-[(3-hydroxypropyl)oxy]-2-phenyl-1,3-oxazole**: ^{1}H-NMR (300 MHz, $CDCl_3$): 7.90 – 7.85 (*m*, 2 arom. H); 7.50 – 7.30 (*m*, 3 arom. H); 7.30 – 7.20 (*m*, 4 arom. H); 7.20 – 7.10 (*m*, 1 arom. H); 4.19 (*t*, *J* = 6.1, CH_2O); 3.80 (*s*, PhCH_2); 3.72 (*t*, *J* = 6.0, CH_2O); 1.89 (*pent*, *J* = 6.0, CH_2); 1.80 – 1.40 (br. *s*, OH).

6.8. ***N*-[3-Benzyltetrahydro-2-oxofuran-3-yl]benzamide** (**23i**). Treatment of a soln. of **25i** (0.109 g, 0.27 mmol) in toluene (140 ml) with TBAF (0.151 g, 0.48 mmol) according to *GP 5* gave **23i** (0.080 g, 100%) as colorless crystals. R_f (Et_2O/hexane 1:1): 0.50. M.p. 170.2 – 171.2°. IR ($CHCl_3$): 3420*w*, 3070*w*, 3020*w*, 2930*w*, 2860*w*, 1775*s*, 1670*s*, 1515*s*, 1485*s*, 1290*m*, 1185*m*, 1165*m*, 1025*m*, 710*m*. ^{1}H-NMR (300 MHz, $CDCl_3$): 7.75 – 7.70 (*m*, 2 arom. H); 7.60 – 7.50 (*m*, 1 arom. H); 7.50 – 7.40 (*m*, 2 arom. H); 7.40 – 7.30 (*m*, 3 arom. H); 7.30 – 7.25 (*m*, 2 arom. H); 6.63 (br. *s*, NH); 4.36 (*td*, *J* = 9.3, 2.4, 1 H of CH_2O); 3.61 (*td*, *J* = 9.5, 7.3, 1 H of CH_2O); 3.33, 3.25 (*AB*, *J* = 13.2, PhCH_2); 2.87 (*ddd*, *J* = 13.4, 7.3, 2.5, 1 H); 2.77 (*td*, *J* = 9.7, 13.4, 1 H). ^{13}C-NMR (50 MHz, $CDCl_3$): 177.0 (*s*, C=O); 166.8 (*s*, NC=O); 133.8, 133.3 (2*s*, 2 arom. C); 132.0 (*d*, arom. CH); 130.0 (*d*, 2 arom. CH); 128.8 (*d*, 2 arom. CH); 128.6 (*d*, 2 arom. CH); 127.8 (*d*, arom. CH); 127.0 (*d*, 2 arom. CH); 65.9 (*t*, CH_2O); 60.1 (*s*, C(3)); 41.6 (*t*, PhCH_2); 33.2 (*t*, CH_2). CI-MS (isobutane): 296 (100, $[M + 1]^+$), 250 (21). Anal. calc. for $C_{18}H_{17}NO_3$ (295.34): C 73.20, H 5.80, N 4.74; found: C 73.13, H 5.69, N 4.62.

7. *X-Ray Crystal Structure Determination of* **23a** – **f** (see *Table 3* and *Figs. 1 – 3*)[9]). The measurements for **23a**, **23b**, **and 23d** – **23f** were made on a *Nicolet-R3* diffractometer using graphite-monochromated Mo$K\alpha$ radiation (λ 0.71073 Å), and those for **23c** on a *Rigaku AFC5R* diffractometer using graphite-monochromated MoK_α radiation and a 12 kW rotating anode generator. The intensities were corrected for *Lorentz* and polarization effects, but not for absorption. Equivalent reflections were merged. The data collection and refinement parameters are given in *Table 3*, and views of the molecules are shown in *Figs 1 – 3*[10]). The structures were solved by direct methods using SHELXS86 [23], which revealed the positions of all non-H-atoms. The benzyl group of **23a** is disordered over two conformations as a result of a small rotation about the C(2)–C(19) bond. Two sets of positions were defined for the atoms of the Ph ring and the site occupation factor of the major conformation refined to 0.510(12). Similarity restraints were applied to the chemically equivalent bond lengths and angles involving all disordered C-atoms, while neighboring atoms within and between each disordered conformation were restrained to have similar atomic displacement parameters. In the case of **23c**, the 15-membered ring is disordered in such a way that both enantiomers are present at the same site. In this arrangement, all atoms occupy identical positions, with the only detectable difference being the presence of the lactone C=O O-atom of the 15-membered ring on both sides of the quaternary C-atom. This requires that O(1) and C(4) are similarly disordered across common sites. Atoms C(4) and O(1A) were constrained to have identical atomic coordinates and atomic displacement parameters. Similar constraints were applied to C(4A) and O(1). The site occupation factors of the disordered sites was refined and converged at 0.912(4) for the major conformer. For all compounds, the non-H-atoms were refined anisotropically. The amide H-atoms were placed in the positions indicated by difference electron density maps and their positions were allowed to refine together with individual isotropic displacement parameters. All remaining H-atoms were placed in geometrically calculated positions and refined using a riding model where each H-atom was assigned a fixed isotropic displacement parameter with a value equal to $1.2U_{eq}$ of its parent C-atom. The refinement of each structure was carried out on F^2 using full-matrix least-squares procedures, which minimized the function $\sigma w (F_o^2 - F_c^2)^2$. A correction for secondary extinction was applied in the cases of **23c** – **23e**. In the cases of **23b** and **23d**, twenty and one reflection, resp., whose intensities were considered to be extreme outliers, were omitted from the final refinement. Neutral atom scattering factors for non-H-atoms were taken from [24], and the scattering factors for H-atoms were taken from [25]. Anomalous dispersion effects were included in F_c [26]; the values for f' and f'' were those of [27]. The values of the mass attenuation coefficients are those of [28]. The SHELXL97 program [29] was used for all calculations.

[9]) CCDC-1447397 – 1447402 contain the supplementary crystallographic data for this article. These data can be obtained free of charge from *The Cambridge Crystallographic Data Centre via* www.ccdc.cam.ac.uk/getstructures.

[10]) For **23a** and **23b**, the obtainable crystal quality was suboptimal, and this is reflected in the quality of the crystal structure refinement results, although both structures are quite unambiguous.

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