N-(α-Chloroalkyloxycarbonyl)pyrrolidines as a Source of Oxygenated d^1-Reagents

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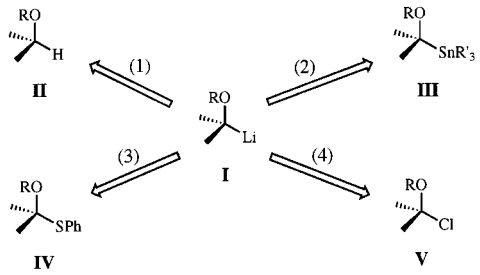
Dedicated to Prof. Dr. José Elguero on the occasion of his 65th birthday

Keywords: Organolithium compounds / d^1-Reagents / Lithiation / Carbamates / 1,2-Diols

Reaction of the *N*-(α-chloroalkyloxycarbonyl)pyrrolidines **1** with lithium powder and a catalytic amount of 4,4′-di-*tert*-butylbiphenyl (DTBB, 2.5 mol-%) in the presence of different electrophiles [*i*BuCHO, *t*BuCHO, PhCHO, Et_2CO, $(CH_2)_5CO$, PhCOMe, Ph_2CO, Me_3SiCl], in THF at temperatures ranging between –78 and –60°C leads, after hydrolysis with water, to the expected functionalized carbamates **2**. Deprotection of compounds **2**, derived from carbonyl compounds, with lithium hydroxide in a mixture of ethanol and water at 80°C affords the correponding 1,2-diols **3**.

Introduction

Functionalized organolithium compounds[1] are interesting intermediates in synthetic organic chemistry because, in a reaction with electrophiles, they are able to transfer their functionality to the other reagent giving polyfunctionalized molecules in only one reaction step. Among these lithium compounds, the α-oxygenated alkyl derivatives of type **I** (R = alkyl: α-alkoxy carbanionic building blocks of the type RO−C$^-$) belong to the family of so-called d^1-reagents, following Seebach's nomenclature,[2] and can be prepared by four different routes: (1) Direct deprotonation of ethers of type **II** (only in special cases *e.g.* alkyl benzyl ethers[3a] or *tert*-butyl methyl ether[3b]) with an alkyllithium and a coreagent [tetrametylethylenediamine (TMEDA) or potassium *tert*-butoxide]; (2) Tin-lithium transmetallation of compounds of type **III** with *n*-butyllithium;[4] (3) Sulfur-lithium exchange in compounds of type **IV** with activated lithium (*e.g.* lithium naphthalenide);[5] (4) Chlorine-lithium exchange in chloroethers of type **V** with lithium and either a stoichiometric[6] or a catalytic[7][8] amount of an arene, naphthalene or 4,4′-di-*tert*-butylbiphenyl (DTBB) being the most commonly used[9] (Scheme 1). To the best of our knowledge the alcohol derivatives of type **I** (R = H, Me or COX: α-hydroxy carbanion building blocks of the type HO−C$^-$)have only been prepared following routes (1)[10] and (2)[11] with, in the first case, very hindered arylic esters[10a] or *O*-benzyl carbamates.[10b,10c,11] In the last few years we have applied the above-mentioned arene-catalyzed lithiation[8] (route 4) for the generation of organolithium reagents starting from nonhalogenated materials,[13] very reactive functionalized organolithium compounds from chlorinated precursors[14] and heterocycles[15] or polylithiated building blocks[16] from polychlorinated materials and working under Barbier-type reaction conditions.[17] In this paper we apply this methodology to the transformation of *O*-chloroalkyl carbamates into the corresponding α-functionalized organolithium compounds, which have been trapped in situ with different electrophilic reagents.[18]

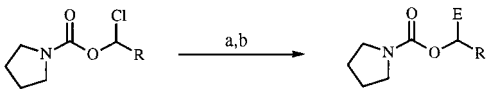


Scheme 1. Preparation of d^1-reagents **I**

Results and Discussion

The reaction of *O*-(α-chloroalkyl) carbamates **1a−c** with an excess of lithium powder (*ca.* 1:7 molar ratio) and a catalytic amount of DTBB (1:0.05 molar ratio; 2.5 mol-%) in the presence of different electrophiles [*i*BuCHO, *t*BuCHO, PhCHO, Et_2CO, $(CH_2)_5CO$, PhCOMe, Ph_2CO, Me_3SiCl] in THF, under the reaction conditions (temperature and reaction time) given in Table 1 gave, after hydrolysis, the corresponding reaction products **2aa−cf** (Scheme 2 and Table 1).

It should be noted that the process shown in Scheme 1 has to be performed under Barbier-type reaction conditions



1a : R = H
1b : R = Me
1c : R = cC_6H_{11}

2aa-cf

Scheme 2. a: Li, DTBB cat. (2.5 mol%), E^+ = *i*BuCHO, *t*BuCHO, PhCHO, Et_2CO, $(CH_2)_5CO$, PhCOMe, Ph_2CO, Me_3SiCl, THF; b: H_2O

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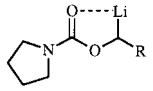
Table 1. Preparation of carbamates **2**

Entry	Starting material	Electrophile E^+	Conditions[a] T (°C) / t	Product[b] No.	R	E	Yield(%)[c]
1	**1a**	*i*BuCHO	−78 to −60 / 2 h	**2aa**	H	*i*BuCHOH	68
2	**1a**	*t*BuCHO	−78 to −60 / 2 h	**2ab**	H	*t*BuCHOH	63
3	**1a**	PhCHO	−78 / 1 h	**2ac**	H	PhCHO	72
4	**1a**	Et_2CO	−78 to −60 / 2 h	**2ad**	H	Et_2COH	80
5	**1a**	$(CH_2)_5CO$	−78 to −60 / 2 h	**2ae**	H	$(CH_2)_5COH$	73
6	**1a**	PhCOMe	−78 / 1 h	**2af**	H	PhC(OH)Me	64
7	**1a**	Ph_2CO	−78 / 1 h	**2ag**	H	Ph_2COH	64
8	**1a**	Me_3SiCl	−78 to −60 / 2 h	**2ah**	H	Me_3Si	81
9	**1b**	*t*BuCHO	−78 / 40 min	**2bb**	Me	*t*BuCHOH	57[d]
10	**1b**	Et_2CO	−78 / 40 min	**2bd**	Me	Et_2COH	62
11	**1b**	$(CH_2)_5CO$	−78 / 40 min	**2be**	Me	$(CH_2)_5COH$	50
12	**1b**	PhCOMe	−78 / 40 min	**2bf**	Me	PhC(OH)Me	61[e]
13	**1c**	Et_2CO	−78 / 2 h	**2cd**	Cy[f]	Et_2COH	64
14	**1c**	$(CH_2)_5CO$	−78 / 2 h	**2ce**	Cy[f]	$(CH_2)_5COH$	61
15	**1c**	PhCOMe	−78 / 2 h	**2cf**	Cy[f]	PhC(OH)Me	45[g]

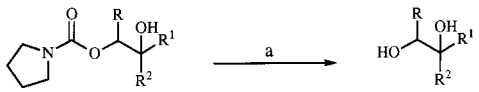
[a] Corresponding to the lithiation step in the presence of the electrophile. — [b] All products **2** were ≥95% pure (300 MHz ^{1}H NMR and/or GLC). — [c] Isolated yield after column chromatography (silica gel, hexane/ethyl acetate) based on the starting material **1**. — [d] A 3.5:1 diastereomeric mixture was obtained (300 MHz ^{1}H NMR). — [e] A 1.8:1 diastereomeric mixture was obtained (300 MHz ^{1}H NMR). — [f] Cyclohexyl. — [g] A 1.2:1 diastereomeric mixture was obtained (300 MHz ^{1}H NMR).

in order to avoid decomposition of the intermediate of type **VI**, generated in situ, even at low temperature. This intermediate, which can decompose either by reaction with the excess lithium or by nucleophilic addition to the amide moiety, is stabilized by intramolecular coordination of the lithium atom to the carbonylic oxygen, through the so-called CIPE ("complex-induced polarity effect").[19] Once the intermediates **VI** are formed, they react mainly with the electrophile present in the reaction medium to give the expected reaction products. As a confirmation of the low stability of this type of intermediate, we performed the step-by-step process starting from the chlorocarbamate **1b** and carrying out the DTBB-catalyzed lithiation at −90°C followed by deuterolysis with deuterium oxide, obtaining a very poor yield (<10%) of the expected product **2a** (R = Me, E = D) with a very low deuterium content (<5%, from mass spectrometry). Unexpectedly, when the same two-step process was carried out with the starting material **1c**, the corresponding product **2c** (R = *c*-C_6H_{11}, E = D) was isolated in 81% yield and with 70% deuterium incorporation (mass spectrometry). The starting chlorocarbamates **1** were prepared from the corresponding chlorinated chloroformates and pyrrolidine in the presence of pyridine.



VI

In the last part of this study we performed the deprotection of carbamates **2** derived from carbonyl compounds. Although this process could be carried out by reduction with DIBAL in refluxing THF for 6 h (Table 2, entry 6 and footnote [c]),[18] we found that hydrolysis with lithium hydroxide at 80°C for 1.5 h worked just as well, so we used this methodology to obtain the corresponding 1,2-diols **3** (Scheme 3 and Table 2). After this deprotection it is apparent that we can use the methodology described in this paper to introduce the nucleophilic $HOCH(R)^-$ unit into carbonyl compounds, which act as electrophilic components.



2aa-ag, 2bb-cf **3aa-cf**

Scheme 3. a: LiOH, $EtOH/H_2O$, 80°C, 1.5 h

Conclusions

From the results described in this paper we can conclude that this methodology is a new and versatile way to generate a synthetic equivalent of α-lithiated alcohols. They readily trap electrophiles in situ to give functionalized alcohols, mainly 1,2-diols.

Table 2. Preparation of diols **3**

Entry	Starting material	Product[a] No.	R	R1		R2	Yield (%)[b]
1	**2aa**	**3aa**	H	H		*i*Bu	92
2	**2ab**	**3ab**	H	H		*t*Bu	80
3	**2ac**	**3ac**	H	H		Ph	91
4	**2ad**	**3ad**	H	Et		Et	86
5	**2ae**	**3ae**	H		$(CH_2)_5$		86
6	**2af**	**3af**	H	Ph		Me	88 (96[c])
7	**2ag**	**3ag**	H	Ph		Ph	95
8	**2bb**[d]	**3bb**	Me	H		*t*Bu	85[d]
9	**2bd**	**3bd**	Me	Et		Et	81
10	**2be**	**3be**	Me		$(CH_2)_5$	77	
11	**2bf**[d]	**3bf**	Me	Ph		Me	91[d]
12	**2bf'**[e]	**3bf'**	Me	Ph		Me	88[e]
13	**2cd**	**3cd**	Cy[f]	Et		Et	86
14	**2ce**	**3ce**	Cy[f]		$(CH_2)_5$		90
15	**2cf**[e]	**3cf**	Cy[f]	Ph		Me	95[d]
16	**2cf'**[e]	**3cf'**	Cy[f]	Ph		Me	88[e]

[a] All products **2** were ≥92% pure (300 MHz ^{1}H NMR and/or GLC). — [b] Isolated crude yield based on the starting material **2**. — [c] Yield corresponding to the deprotection of compound **2af** using DIBAL (see text). — [d] The major diastereomer was used. — [e] The minor diastereomer was used.

Experimental Section

General: All reactions were performed in oven-dried glassware under an argon atmosphere. − Melting points: Reichert Thermovar Apparatus. − ^{1}H- and ^{13}C-NMR spectroscopy: Bruker AC-300 with $CDCl_3$ as a solvent; chemical shifts are quoted relative to TMS as internal standard; δ in ppm, *J* in Hz. − FTIR: Nicolet Impact 400D spectrophotometer recorded on films between NaCl plates and reported in cm^{-1}. − GC-LRMS: Shimazu QP-5000 Mass Spectrometer at 70 eV using electronic impact; relative intensities for signals ≥10% in parentheses. − GC: Hewlett Packard HP-5890 instrument equipped with a flame ionization detector and a 12 m HP-1 capillary column (0.2 mm diam., 0.33 mm film thickness), with nitrogen (2 mL/min) as the carrier gas, $T_{injector}$ = 275°C, $T_{detector}$ = 300°C, T_{column} = 60°C (3 min) and 60−270°C (15°C/min). − TLC: Schleicher & Schuell F1400/LS 254 plates coated with a 0.2 mm layer of silica gel; R_f values are given under these conditions. − Column chromatography: Merk silica gel 60 of 63−200 mesh. − HRMS: Finigan MAT 955. − Microanalysis: Microanalysis Service at the University of Alicante. − Commercially available reagents were purchased (Aldrich, Acros) of the highest grade and used without further purification. Chloromethyl (Fluka) and α-chloroethyl (Aldrich) chloroformates are commercially available. α-Chloro-α-cyclohexylmethyl chloroformate was prepared from cyclohexanecarboxaldehyde and triphosgene following the literature procedure.[20] [**CAUTION**: α−Chloroalkyl chloroformates are toxic and should be handled carefully]. Solvents were dried by standard procedures.[21]

Preparation of the Chlorocarbamates 1: To a stirred solution of pyrrolidine (1.42 g, 20 mmol), and pyridine (1.58 g, 20 mmol), in dichloromethane (15 mL) was added dropwise a solution of the corresponding chloroformate (20 mmol) in dichloromethane (5 mL) for 15 min at 0°C under an argon atmosphere. The resulting mixture was allowed to warm to room temperature (2 h for **1a,b** and 40 min for **1c**) and then dichloromethane (100 mL) was added. The resulting solution was sucessively washed with 0.2 M HCl (3 × 10 mL), water (3 × 10 mL) and brine (2 × 10 mL). The organic layer was dried with Na_2SO_4, the solvent evaporated, and the resulting residue distilled at reduced pressure to give the title compounds **1b** (65% yield; b.p. 50°C/0.1 Torr) and **1c** (60% yield; b.p. 140°C/0.1 Torr). Compound **1a** was used without purification for the lithiation reaction (≈ 100% crude yield).

***N*-(Chloromethyloxycarbonyl)pyrrolidine (1a):** oil. − IR (film): $\tilde{\nu}$ = 1732 (C=O), 1121, 1093 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.85−1.95 (m, 4 H, $CH_2CH_2CH_2N$), 3.40−3.50 (m, 4 H, CH_2NCH_2), 5.79 (s, 2 H, CH_2Cl). − ^{13}C NMR: δ = 24.7, 25.55 ($CH_2CH_2CH_2N$), 46.4, 46.9 (CH_2NCH_2), 70.55 (CH_2Cl), 152.0 (CO_2). − GC-LRMS: *m/z* (%) = 165 (M^+ + 2, 8), 163 (M^+, 24), 114 (80), 98 (10), 70 (50), 69 (10), 56 (56), 55 (70), 43 (32), 42 (100). − HRMS: *m/z* = 163.0399 (M^+); calcd. for $C_6H_{10}ClNO_2$: 163.0400.

***N*-[(1-Chloro)ethyloxycarbonyl]pyrrolidine (1b):** oil. − IR (film): $\tilde{\nu}$ = 1731 (C=O), 1100, 1071 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.81 (d, *J* = 5.8 Hz, 3 H, CH_3), 1.85−1.95 (m, 4 H, $CH_2CH_2CH_2N$), 3.40−3.50 (m, 4 H, CH_2NCH_2), 6.61 (q, *J* = 5.8 Hz, 1 H, CH). − ^{13}C NMR: δ = 24.7, 25.5 ($CH_2CH_2CH_2N$), 25.45 (CH_3), 45.8, 46.25 (CH_2NCH_2), 82.8 (CH), 151.85 (CO_2). − GC-LRMS: *m/z* (%) = 179 (M^+ + 2, 1.4), 177 (M^+, 4.2), 114 (39), 98 (61), 87 (11), 70 (55), 65 (35), 63 (100), 56 (53), 55 (68), 44 (18), 43 (71), 42 (53), 41 (62). − HRMS: *m/z* = 177.0537 (M^+); calcd. for $C_7H_{12}ClNO_2$: 177.0556.

***N*-[(Chlorocyclohexyl)methyloxycarbonyl]pyrrolidine (1c):** oil. − IR (film): $\tilde{\nu}$ = 1728 (C=O), 1100, 1080 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.10−1.35 (m, 4 H, 4 × CH cyclohexyl ring), 1.63−2.05 (m, 11

H, 7 × CH cyclohexyl ring, $CH_2CH_2CH_2N$), 3.30−3.50 (m, 4 H, CH_2NCH_2), 6.32 (d, J = 4.8 Hz, 1 H, CHCl). − ^{13}C NMR: δ = 24.8, 25.6 (3C), 26.1 (5 × CH_2 cyclohexyl ring), 27.85, 28.25 ($CH_2CH_2CH_2N$), 44.95 ($CHCH_2$), 45.85, 46.3 (CH_2NCH_2), 90.25 (CHCl), 152.2 (CO_2). − GC-LRMS: m/z (%) = 209 (M^+ − HCl, 6), 99 (5), 98 (100), 56 (23), 55 (76). − HRMS: m/z = 209.1404 (M^+ − HCl); calcd. for $C_{12}H_{19}NO_2$: 209.1416.

Preparation of Compounds 2 by DTBB-Catalyzed Lithiation of Chlorocarbamates 1 under Barbier Conditions: To a blue suspension of lithium powder (100 mg, 14 mmol) and DTBB (26 mg, 0.1 mmol) in THF (6 mL) was slowly added (*ca.* 15 min) a solution of the starting material **2** (2 mmol) and the corresponding electrophile (2 mmol) in THF (4 mL) at −78 °C under an argon atmosphere. The reaction mixture was stirred for 40 min or 2 h (see Table 1) at temperatures ranging between −78 and −60 °C (see Table 1), and was then hydrolyzed with water (10 mL). After warming to room temperature the crude mixture was extracted with diethyl ether (3 × 20 mL), the organic layer was dried with Na_2SO_4 and the solvents evaporated. The residue was purified by column chromatography [silica gel (flash chromatography for compounds **2bb**, **bf**, **cf**), hexane/ethyl acetate] to give pure compounds **2**. Yields are reported in Table 1.

***N*-[(2-Hydroxy-4-methyl)pentyloxycarbonyl]pyrrolidine (2aa):** oil, R_f = 0.31 (hexane/ethyl acetate, 1:1). − IR (film): ṽ = 3430 (OH), 1683 (C=O), 1181, 1130, 1101 (CO) cm^{-1}. − ^1H NMR: δ = 0.86 (t, J = 6.7 Hz, 6 H, 2 × CH_3), 1.70−1.79 [m, 5 H, $CH_2CH_2CH_2N$, $(CH_3)_2CH$], 2.90 (br s, 1 H, OH), 3.26- 3.40 (m, 4 H, CH_2NCH_2), 3.78−3.88 (m, 1 H, *CH*OH), 3.92 (dd, J = 11.3, 7.0 Hz, 1 H, C*H*HO), 4.07 (dd, J = 11.3, 3.1 Hz, CH*H*O). − ^{13}C NMR: δ = 21.9, 23.2 (2 × CH_3), 24.2 [$CH(CH_3)_2$], 24.8, 25.55 ($CH_2CH_2CH_2N$), 42.2 [$CH_2CH(CH_3)_2$], 45.7, 46.15 (CH_2NCH_2), 68.5 (CHOH), 69.8 (CH_2O), 155.35 (CO_2). − GC- LRMS: m/z (%) = 215 (M^+, 0.2), 158 (12), 129 (16), 116 (65), 114 (100), 98 (75), 70 (33), 56 (33), 55 (80). − HRMS: m/z = 215.1534 (M^+); calcd. for $C_{11}H_{21}NO_3$: 215.1521.

***N*-[(3,3-Dimethyl-2-hydroxy)butyloxycarbonyl]pyrrolidine (2ab):** oil, R_f = 0.44 (hexane/ethyl acetate, 1:1). − IR (film): ṽ = 3447 (OH), 1684 (C=O), 1132, 1113, 1095 (CO) cm^{-1}. − ^1H NMR: δ = 0.96 [s, 9 H, $(CH_3)_3C$], 1.80−1.95 [m, 4 H, $CH_2CH_2CH_2N$], 2.84 (br d, J = 3 Hz, 1 H, OH), 3.22−3.35 (m, 4 H, CH_2NCH_2), 3.45 (m, 1 H, *CH*OH), 4.08 (dd, J = 11.6, 8.5 Hz, 1 H, C*H*HCH), 4.28 (dd, J = 11.6, 2.0 Hz, CH*H*CH). − ^{13}C NMR: δ = 24.9, 25.7 ($CH_2CH_2CH_2N$), 25.85 [$(CH_3)_3C$], 33.9 [$(CH_3)_3C$], 45.8, 46.25 (CH_2NCH_2), 67.3 (CH_2O), 78.0 (CH), 155.65 (CO_2). − GC-LRMS: m/z (%) = 158 [M^+ − $(CH_3)_3C$, 27], 116 (53), 114 (67), 98 (100), 70 (31), 57 (32), 56 (40), 55 (70), 44 (21). − HRMS: m/z = 215.1532 (M^+); calcd. for $C_{11}H_{21}NO_3$: 215.1521.

***N*-[(2-Hydroxy-2-phenyl)ethyloxycarbonyl]pyrrolidine (2ac)**: white solid (m.p. 79−80 °C, toluene), R_f = 0.29 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3411 (OH), 1681 (C=O), 1131, 1111, 1066, 1028 (CO) cm^{-1}. − ^1H NMR: δ = 1.70−1.93 [m, 4 H, $CH_2CH_2CH_2N$], 3.20−3.33 (m, 4 H, CH_2NCH_2), 3.70 (br s, 1 H, OH), 4.10−4.16 (m, 2 H, CH_2CH), 4.85 (dd, J = 6.5, 4.4 Hz, 1 H, C*H*OH), 7.18−7.30 (m, 5 H, ArH). − ^{13}C NMR: δ = 24.8, 25.55 ($CH_2CH_2CH_2N$), 45.75, 46.15 (CH_2NCH_2), 70.35 (CH_2O), 73.0 (CHO), 126.1, 127.7, 128.25, 140.35 (4 × ArC), 155.45 (CO_2). − GC-LRMS: m/z (%) = 217 [M^+ − H_2O, 3], 129 (21), 116 (23), 114 (56), 98 (80), 91 (35), 79 (12), 77 (27), 71 (10), 70 (28), 65 (14), 56 (45), 55 (100). − $C_{13}H_{17}NO_3$ (235.28): calcd. C 66.36, H 7.28, N 5.95; found C 66.43, H 7.35, N 5.79.

***N*-[(2-Ethyl-2-hydroxy)butyloxycarbonyl]pyrrolidine (2ad)**: oil, R_f = 0.36 (hexane/ethyl acetate, 1:1). − IR (film): ṽ = 3435 (OH), 1684 (C=O), 1182, 1130, 1110 (CO) cm^{-1}. − ^1H NMR: δ = 0.90 (t, J = 7.4 Hz, 6 H, 2 × CH_3CH_2), 1.53 (q, J = 7.4 Hz, 4 H, 2 × CH_3CH_2), 1.80−1.95 (m, 4 H, $CH_2CH_2CH_2N$), 2.50 (s, 1 H, OH), 3.35−3.45 (m, 4 H, CH_2NCH_2), 4.03 (s, 2 H, CH_2O). − ^{13}C NMR: δ = 7.57 (2 × CH_3CH_2), 24.85, 25.65 ($CH_2CH_2CH_2N$), 28.5 (2 × CH_3CH_2), 45.75, 46.2 (CH_2NCH_2), 69.75 (CH_2O), 73.85 (COH), 155.35 (CO_2). − GC-LRMS: m/z (%) = 186 (M^+ − CH_3CH_2, 8), 129 (21), 114 (100), 98 (82), 71 (10), 70 (33), 57 (14), 56 (37), 55 (71), 45 (29), 44 (12), 43 (36).

***N*-[(1-Hydroxycyclohexyl)methyloxycarbonyl]pyrrolidine (2ae)**: oil, R_f = 0.24 (hexane/ethyl acetate, 1:1). − IR (film): ṽ = 3421 (OH), 1683 (C=O), 1130, 1107, 1071 (CO) cm^{-1}. − ^1H NMR: δ = 1.40−1.70 (m, 10 H, 5 × CH_2 cyclohexyl ring), 1.83−1.95 (m, 4 H, $CH_2CH_2CH_2N$), 2.53 (br s, 1 H, OH), 3.35−3.45 (m, 4 H, CH_2NCH_2), 4.01 (s, 2 H, CH_2O). − ^{13}C NMR: δ = 21.6, 25.7 (2 C), 34.25 (2 C) (5 × CH_2 cyclohexyl ring), 24.8 ($CH_2CH_2CH_2N$), 45.7, 46.15 (CH_2NCH_2), 70.6 (CH_2O) 72.15 (COH), 155.25 (CO_2). − GC-LRMS: m/z (%) = 227 (M^+, 0.7), 130 (13), 129 (31), 114 (100), 98 (48), 81 (13), 70 (29), 56 (22), 55 (52), 44 (10), 43 (52), 42 (20), 41 (40). − HRMS: m/z = 227.1512 (M^+); calcd. for $C_{12}H_{21}NO_3$: 227.1521.

***N*-[(2-Hydroxy-2-phenyl)propyloxycarbonyl]pyrrolidine (2af):** oil, R_f = 0.36 (hexane/ethyl acetate, 1:1). − IR (film): ṽ = 3427 (OH), 1683 (C=O), 1181, 1129, 1108 (CO) cm^{-1}. − ^1H NMR: δ = 1.56 (s, 3 H, CH_3), 1.80−1.90 (m, 4 H, $CH_2CH_2CH_2N$), 3.22−3.38 (m, 4 H, CH_2NCH_2), 3.53 (s, 1 H, OH), 4.22, 4.30 (2 d, J = 11.6 Hz, 2 H, CH_2O), 7.20−7.50 (m, 5 H, ArH). − ^{13}C NMR: δ = 24.85, 25.6 ($CH_2CH_2CH_2N$), 26.35 (CH_3), 45.7, 46.2 (CH_2NCH_2), 72.85 (CH_2O), 74.0 (COH), 125.15, 126.95, 128.1, 144.8 (4 × ArC), 155.35 (CO_2). − GC-LRMS; m/z (%): = 234 (M^+ − CH_3, 0.2), 129 (32), 121 (20), 115 (10), 114 (100), 98 (30), 77 (16), 71 (11), 70 (36), 56 (31), 55 (48), 44 (17), 43 (68). − HRMS: m/z = 234.1146 (M^+ − CH_3); calcd. for $C_{13}H_{16}NO_3$: 234.1130.

***N*-[(2,2-Diphenyl-2-hydroxy)ethyloxycarbonyl]pyrrolidine (2ag):** white solid (m.p. 153−155 °C, hexane), R_f = 0.48 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3486 (OH), 1683 (C=O), 1216, 1185, 1129 (CO) cm^{-1}. − ^1H NMR: δ = 1.75−1.85 (m, 4 H, $CH_2CH_2CH_2N$), 3.10−3.20, 3.30−3.40 (2 m, 4 H, CH_2NCH_2), 3.90 (s, 1 H, OH), 4.69 (s, 2 H, CH_2O), 7.20−7.50 (m, 10 H, ArH). − ^{13}C NMR: δ = 24.8, 25.5 ($CH_2CH_2CH_2N$), 45.7, 46.25 (CH_2NCH_2), 70.9 (CH_2O), 77.95 (COH), 126.55, 127.25, 128.1, 143.95 (4 × ArC), 155.15 (CO_2). − GC-LRMS: m/z (%) = 294 (M^+ − H_2O, 1.4), 196 (25), 184 (11), 183 (74), 165 (14), 114 (50), 105 (70), 98 (98), 77 (44), 72 (10), 70 (16), 56 (44), 55 (100). − $C_{19}H_{21}NO_3$ (311.38): calcd. C 73.29, H 6.80, N 4.50; found C 73.05, H 6.84, N 4.46.

***N*-(Trimethylsilylmethyloxycarbonyl)pyrrolidine (2ah):** oil, R_f = 0.68 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 1705 (C=O), 1178, 1128, 1102 (CO), 857 (CSi) cm^{-1}. − ^1H NMR: δ = 0.04 [s, 9 H, $(CH_3)_3Si$], 1.75−1.85 (m, 4 H, $CH_2CH_2CH_2N$), 3.25−3.40 (m, 4 H, CH_2NCH_2), 3.68 (s, 2 H, CH_2Si). − ^{13}C NMR: δ = −3.2 [$(CH_3)_3Si$], 24.95, 25.7 ($CH_2CH_2CH_2N$), 46.2, 45.6 (CH_2NCH_2), 57.9 (CH_2O), 156.35 (CO_2). − GC-LRMS: m/z (%) = 201 (M^+, 0.2), 186 (40), 142 (10), 114 (50), 98 (30), 73 (100), 56 (45), 55 (78), 45 (27). − HRMS: m/z = 201.1188 (M^+); calcd. for $C_9H_{19}NO_2Si$: 201.1185.

***N*-[(2-Hydroxy-1,3,3-trimethyl)butyloxycarbonyl]pyrrolidine (2bb), First diastereomer**: white solid (m.p. 65−66 °C, hexane), R_f = 0.38 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3455 (OH), 1678 (C=O), 1129, 1106 (CO) cm^{-1}. − ^1H NMR: δ = 0.98 [s, 9 H, $(CH_3)_3C$], 1.29 (d, J = 6.5 Hz, 3 H, CH_3CH), 1.75−1.92 (m, 4 H, $CH_2CH_2CH_2N$), 2.15 (s, 1 H, OH), 3.25−3.40 (m, 4 H,

CH_2NCH_2), 3.47 (d, J = 3.1 Hz, 1 H, C*H*OH), 4.97 (qd, J = 6.5, 3.1 Hz, 1 H, CH_3CH). − ^{13}C NMR: δ = 16.0 (CH_3CH), 25.55, 25.65 ($CH_2CH_2CH_2N$), 26.6 [$(CH_3)_3C$], 34.3 [$(CH_3)_3C$], 45.95, 46.1 (CH_2NCH_2), 72.75 (CH_3CH), 80.75 (CHOH), 154.5 (CO_2). − GC-LRMS: *m*/*z* (%) = 172 [M^+ − $(CH_3)_3C$, 8], 143 (11), 116 (52), 114 (70), 99 (20), 98 (100), 71 (11), 70 (18), 57 (50), 56 (25), 55 (51), 44 (20), 43 (36). − $C_{12}H_{23}NO_3$ (229.32): calcd. C 62.85, H 10.11, N 6.11; found C 62.53, H 9.86, N 5.98. **Second diastereomer**: IR (KBr): ṽ = 3486 (OH), 1678 (C=O), 1180, 1132, 1109 (CO) cm^{-1}. − ^{1}H NMR: δ = 0.94 [s, 9 H, $(CH_3)_3C$], 1.34 (d, J = 6.4 Hz, 3 H, CH_3CH), 1.80−1.95 (m, 4 H, $CH_2CH_2CH_2N$), 2.12 (br s, 1 H, OH), 3.23−3.45 (m, 5 H, CH_2NCH_2, CHOH), 5.10 (qd, J = 6.4, 1.2 Hz, 1H, CH_3CH). − ^{13}C NMR: δ = 19.5 (CH_3CH), 24.75, 24.8 ($CH_2CH_2CH_2N$), 26.25 [$(CH_3)_3C$], 35.0 [$(CH_3)_3C$], 45.5, 45.6 (CH_2NCH_2), 69.75 (CH_3CH), 81.15 (CHOH), 153.8 (CO_2). − GC-LRMS: *m*/*z* (%) = 172 [M^+ − $(CH_3)_3C$, 16], 116 (15), 114 (37), 98 (175), 70 (26), 57 (100), 56 (21), 55 (47), 44 (40), 43 (56).

***N*-[(2-Ethyl-2-hydroxy-1-methyl)butyloxycarbonyl]pyrrolidine (2bd)**: white solid (m.p. 85−86°C, hexane), R_f = 0.40 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3475 (OH), 1675 (C=O), 1131, 1100 (CO) cm^{-1}. − ^{1}H NMR: δ = 0.91 (t, J = 7.5 Hz, 6 H, 2 × CH_3CH_2), 1.23 (d, J = 6.4 Hz, 3 H, CH_3CH), 1.35−1.68 (m, 4 H, 2 × CH_3CH_2), 1.80−1.95 (m, 4 H, $CH_2CH_2CH_2N$), 1.98 (s, 1 H, OH), 3.30−3.45 (m, 4 H, CH_2NCH_2), 4.84 (q, J = 6.4 Hz, 1 H, CH). − ^{13}C NMR: δ = 7.45, 7.5 (2 × CH_3CH_2), 14.95 (CH_3CH), 24.85, 25.65 ($CH_2CH_2CH_2N$), 26.65, 28.1 (2 × CH_3CH_2), 45.75, 46.2 (CH_2NCH_2), 75.1 (CH), 75.8 (COH), 154.85 (CO_2). − GC-LRMS: *m*/*z* (%) = 200 (M^+ − CH_3CH_2, 4), 143 (19), 116 (16), 114 (10), 98 (70), 71 (14), 70 (23), 57 (27), 56 (28), 55 (50), 45 (29), 44 (12), 43 (36). − $C_{12}H_{23}NO_3$ (229.32): calcd. C 62.85, H 10.11, N 6.11; found C 63.26, H 10.34, N 6.04.

***N*-[1-(1′-Hydroxycyclohexyl)ethyloxycarbonyl]pyrrolidine (2be)**: white solid (m.p. 72−74°C, hexane), R_f = 0.38 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3448 (OH), 1681 (C=O), 1129, 1104 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.23 (d, J = 6.1 Hz, 3 H, CH_3), 1.16−1.78 (m, 11 H, 5 × CH_2 cyclohexyl ring, OH), 1.82−1.94 (m, 4 H, $CH_2CH_2CH_2N$), 3.30−3.45 (m, 4 H, CH_2NCH_2), 4.71 (q, J = 6.1 Hz, 1 H, CH). − ^{13}C NMR: δ = 14.4 (CH_3), 21.25, 21.4, 25.7, 32.5, 34.05 (5 × CH_2 cyclohexyl ring), 24.8, 25.55 ($CH_2CH_2CH_2N$), 45.6, 46.05 (CH_2NCH_2), 72.6 (COH), 76.8 (CH), 154.7 (CO_2). − GC-LRMS: *m*/*z* (%) = 212 (M^+ − CH_3CH_2, 0.1), 143 (22), 115 (11), 114 (100), 99 (15), 98 (52), 81 (17), 71 (26), 70 (41), 69 (10), 57 (12), 56 (42), 55 (83), 53 (11), 45 (25), 44 (18), 43 (52), 42 (26), 41 (56). − $C_{13}H_{23}NO_3$ (241.33): calcd. C 64.70, H 9.61, N 5.80; found C 64.67, H 9.79, N 5.72.

***N*-[(2-Hydroxy-1-methyl-2-phenyl)propyloxycarbonyl]pyrrolidine (2bf), First diastereomer:** white solid (m.p. 123−125°C, hexane), R_f = 0.40 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3436 (OH), 1667 (C=O), 1198, 1095 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.04 (d, J = 6.4 Hz, 3 H, CH_3CH), 1.58 (s, 3 H, CH_3C), 1.80−1.93 (m, 4 H, $CH_2CH_2CH_2N$), 2.62 (s, 1 H, OH), 3.30−3.45 (m, 4 H, CH_2NCH_2), 5.05 (q, J = 6.4 Hz, 1 H, CH), 7.15−7.47 (m, 5 H, ArH). − ^{13}C NMR: δ = 15.1 (CH_3CH), 24.9, 25.65 ($CH_2CH_2CH_2N$), 27.55 (CH_3C), 45.75, 46.25 (CH_2NCH_2), 76.0 (COH), 77.4 (CH_3CH), 125.35, 126.8, 127.95, 144.2 (4 × ArC), 154.7 (CO_2). − GC-LRMS: *m*/*z* (%) = 246 (M^+ − OH, 0.1), 143 (37), 121 (28), 115 (10), 114 (88), 99 (14), 98 (72), 77 (18), 71 (38), 70 (33), 56 (44), 55 (77), 44 (15), 43 (100), 42 (14), 41 (23). − $C_{15}H_{21}NO_3$ (263.34): calcd. C 68.42, H 8.04, N 5.32; found C 68.63, H 8.15, N 5.28. **Second diastereomer:** white solid (m.p. 96−98°C, hexane), R_f = 0.36 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3427 (OH), 1678 (C=O), 1129, 1104 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.16 (d, J = 6.1 Hz, 3 H, CH_3CH), 1.55 (s, 3 H, CH_3C), 1.75−1.90 (m, 4 H, $CH_2CH_2CH_2N$), 3.10−3.35 [m, 5 H, CH_2NCH_2, OH], 5.05 (q, J = 6.1 Hz, 1 H, CH), 7.18−7.57 (m, 5 H, ArH). − ^{13}C NMR: δ = 15.4 (CH_3CH), 24.1 (CH_3C), 24.7, 25.5 ($CH_2CH_2CH_2N$), 45.55, 46.0 (CH_2NCH_2), 76.2 (COH), 77.3 (CH_3CH), 125.5, 126.8, 127.85, 145.2 (4 × ArC), 154.7 (CO_2). − GC-LRMS: *m*/*z* (%) = 246 (M^+ − OH, 0.2), 143 (47), 121 (42), 115 (13), 114 (99), 99 (20), 98 (90), 77 (24), 72 (12), 70 (45), 56 (57), 55 (95), 44 (20), 43 (100), 42 (24), 41 (29). − $C_{15}H_{21}NO_3$ (263.34): calcd. C 68.42, H 8.04, N 5.32; found C 68.75, H 8.22, N 5.26.

***N*-[(Deuterocyclohexyl)methyloxycarbonyl]pyrrolidine (2c, E = D):** oil, R_f = 0.63 (hexane/ethyl acetate, 1:1). − IR (film): ṽ = 1703 (C=O), 1132, 1115, 1099 (CO) cm^{-1}. − ^{1}H NMR: δ = 0.80−2.00 [m, 15 H, 11 H cyclohexyl ring, $CH_2CH_2CH_2N$], 3.32−3.42 (m, 4 H, CH_2NCH_2), 3.89 (d, J = 6.4 Hz, 1 H, CHD). − ^{13}C NMR: δ = 24.9, 25.7 (3 C), 26.45 (5 × CH_2 cyclohexyl ring), 29.65 (2 C, $CH_2CH_2CH_2N$), 37.4 ($CHCH_2$), 45.65, 46.0 (CH_2NCH_2), 69.75 (t, J = 22 Hz, 1 C, CHD), 155.35 (CO_2). − GC-LRMS: *m*/*z* (%) = 212 (M^+, 0.1), 116, (90), 98 (12), 70 (23), 68 (13), 67 (16), 56 (58), 55 (100), 54 (12).

***N*-[(1-Cyclohexyl-2-ethyl-2-hydroxy)butyloxycarbonyl]pyrrolidine (2cd):** white solid (m.p. 96−98°C, hexane), R_f = 0.49 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3462 (OH), 1676 (C=O), 1130, 1127, 1115 (CO) cm^{-1}. − ^{1}H NMR: δ = 0.88, 0.90 (2 t, J = 7.5, 7.5 Hz, 6 H, 2 × CH_3), 1.05−2.10 (m, 20 H, 11 H cyclohexyl ring, 2 × CH_2CH_3, $CH_2CH_2CH_2N$, OH), 3.35- 3.50 (m, 4 H, CH_2NCH_2), 4.64 (d, J = 2.4 Hz, 1 H, CHO). − ^{13}C NMR: δ = 7.65 (2 C, 2 × CH_3), 24.85, 25.65 ($CH_2CH_2CH_2N$), 26.2, 26.3, 26.6, 26.85, 27.7, 28.8, 32.25 (5 × CH_2 cyclohexyl ring, 2 × CH_2CH_3), 38.45 ($CHCH_2$), 45.7, 46.25 (CH_2NCH_2), 76.95 (COH), 80.95 (CHO), 155.2 (CO_2). − GC-LRMS: *m*/*z* (%) = 268 (M^+ − CH_3CH_2, 1), 211 (11), 116 (63), 115 (21), 114 (100), 98 (55), 71 (16), 70 (14), 57 (24), 56 (18), 55 (58), 45 (14), 43 (18). − $C_{17}H_{31}NO_3$ (297.44): calcd. C 68.65, H 10.51, N 4.71; found C 68.30, H 10.57, N 4.55.

***N*-[Cyclohexyl-(1′-hydroxycyclohexyl)methyloxycarbonyl]-pyrrolidine (2ce):** white solid (m.p. 113−115°C, hexane), R_f = 0.51 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3505 (OH), 1682 (C=O), 1177, 1134, 1107 (CO) cm^{-1}. − ^{1}H NMR: δ = 1.02−2.05 (m, 26 H, 10 × CH_2 cyclohexyl ring, $CH_2CH_2CH_2N$, CHCHO, OH), 3.30- 3.50 (m, 4 H, CH_2NCH_2), 4.53 (d, J = 3.0 Hz, 1 H, CHCHO). − ^{13}C NMR: δ = 21.5 (2 C), 24.85, 25.7 (2 C), 26.2, 26.3, 26.55, 27.55, 32.3, 33.25, 35.5 (12 C, $CH_2CH_2CH_2N$, 10 × CH_2 cyclohexyl rings), 38.4 (CHCHO), 45.65, 46.25 (CH_2NCH_2), 73.8 (COH), 82.85 (CHCHO), 155.2 (CO_2). − GC-LRMS: *m*/*z* (%) = 211 [M^+ − $(CH_2)_5CO$, 20], 116 (63), 115 (27), 115 (27), 114 (100), 99 (18), 98 (51), 71 (20), 70 (17), 56 (20), 55 (62).

***N*-[(1-Cyclohexyl-2-hydroxy-2-phenyl)propyloxycarbonyl]-pyrrolidine (2cf), First diastereomer:** white solid (m.p. 160−162°C, hexane), R_f = 0.42 (hexane/ethyl acetate, 1:1). − IR (KBr): ṽ = 3443, 3129 (OH), 1685 (C=O), 1128, 1108, 1067 (CO) cm^{-1}. − ^{1}H NMR: δ = 0.97−1.93 [m, 16 H, 11 H cyclohexyl ring, $CH_2CH_2CH_2N$, OH], 1.51 (s, 3 H, CH_3), 3.40−3.55 (m, 4H, CH_2NCH_2), 4.91 (d, J = 3.3 Hz, 1 H, CHO), 7.24−7.51 (m, 5 H, ArH). − ^{13}C NMR: δ = 24.9, 25.75 ($CH_2CH_2CH_2N$), 26.1 (2 C), 26.3, 27.65, 31.95 (5 × CH_2 cyclohexyl ring), 29.4 (CH_3), 38.45 ($CHCH_2$), 45.8, 46.4 (CH_2NCH_2), 77.1 (COH), 83.25 (CHO), 125.15, 126.7, 128.0, 144.55 (4 × ArC), 155.15 (CO_2). − GC-LRMS: *m*/*z* (%) = 211 (M^+ − CH_3COPh, 15), 121 (30), 116 (31), 115 (15), 144 (60), 99 (13), 98 (97), 71 (23), 70 (14), 56 (27), 55 (76), 43 (100). − $C_{20}H_{29}NO_3$ (331.46): calcd. C 72.47, H 8.82, N

4.23; found C 72.40, H 8.91, N 4.34. **Second diastereomer:** white solid (m.p. 143−145°C, hexane), R_f = 0.34 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3410 (OH), 1678 (C=O), 1139, 1120, 1111, (CO) cm^{-1}. − ^1H NMR: δ = 1.07−1.90 [m, 15 H, 11 H cyclohexyl ring, $CH_2CH_2CH_2N$], 1.58 (s, 3 H, CH_3), 2.99 (s, 1 H, OH), 3.35−3.50 (m, 4 H, CH_2NCH_2), 4.85 (d, J = 4.0 Hz, 1 H, CHO), 7.26−7.51 (m, 5 H, ArH). − ^{13}C NMR: δ = 24.8, 25.65 ($CH_2CH_2CH_2N$), 25.05 (CH_3), 26.1, 26.2, 26.3, 27.85, 32.3 (5 × CH_2 cyclohexyl ring), 38.5 ($CHCH_2$), 45.65, 46.25 (CH_2NCH_2), 76.9 (COH), 84.05 (CHO), 125.6, 126.9, 127.9, 145.35 (4 × ArC), 155.35 (CO_2). − GC-LRMS: *m/z* (%) = 211 (M^+ − CH_3COPh, 13), 121 (27), 116 (28), 115 (13), 99(12), 98 (90), 71 (21), 70 (13), 56 (24), 55 (74), 43 (100). − $C_{20}H_{29}NO_3$ (331.46): calcd. C 72.47, H 8.82, N 4.23; found C 72.82, H 8.84, N 4.31.

Preparation of 1,2-Diols 3 by Hydrolysis of Carbamates 2: A mixture of the corresponding compound **2** (0.5 mmol) in ethanol (5 mL) and 3 M LiOH aqueous solution (2.5 mL) was warmed at 80°C for 1.5 h. The resulting mixture was cooled to room temperature, acidified with 2 M hydrochloric acid and extracted with ethyl acetate (4 × 10 mL). The organic layer was neutralized with saturated aqueous $NaHCO_3$, dried with Na_2SO_4 and the solvents evaporated. The resulting residue contained essentially pure (>95% from NMR spectroscopy) 3. For analytical purposes they were distilled (kugelrohr) at reduced pressure (0.1 Torr). Yields are reported in Table 2.

4-Methyl-1,2-pentanediol (3aa):[22] oil, R_f = 0.29 (hexane/ethyl acetate, 3:7). − IR (film): $\tilde{\nu}$ = 3385 (OH), 1076, 1025 (CO) cm^{-1}. − ^1H NMR: δ = 0.91, 0.93 [2 d, J = 5.5, 5.5 Hz, 6 H, $(CH_3)_2CH$], 1.16−1.37, 1.70−1.84 [2 m, 2 H, $CH_2CHC(CH_3)_2$], 1.71−1.84 [m, 1 H, $(CH_3)_2CH$], 2.50, 3.28 (2 × br s, 2 H, 2 × OH), 3.39 (dd, J = 11.6, 7.9 Hz, 1 H, CHHOH), 3.61 (dd, J = 11.6, 2.4 Hz, 1 H, CHHOH), 3.77 (m, 1 H, CHOH). − ^{13}C NMR: δ = 22.05, 23.3 [$(CH_3)_2CH$], 24.4 [$(CH_3)_2CH$], 41.95 [$(CH_3)_2CHCH_2$], 67.1 (CH_2OH), 70.4 (CHOH). − GC-LRMS: *m/z* (%) = 118 (M^+, 0.4), 87 (30), 69 (82), 61 (28), 57 (14), 45 (67), 43 (100), 41 (69).

3,3-Dimethyl-1,2-butanediol (3ab):[11b] oil, R_f = 0.26 (hexane/ethyl acetate, 3:7). − IR (film): $\tilde{\nu}$ = 3404 (OH), 1090, 1044, 1020 (CO) cm^{-1}. − ^1H NMR: δ = 0.92 [s, 9 H, $(CH_3)_3C$], 3.36 (dd, J = 10.0, 2.4 Hz, 1 H, CHHOH), 3.44 (br s, 1 H, OH), 3.48 (t, J = 10.0 Hz, 1 H, CHHOH), 3.73 (dd, J = 10.0, 2.4 Hz, 1 H, CHHOH). − ^{13}C NMR: δ = 25.9 [$(CH_3)_3C$], 33.5 [$(CH_3)_3C$], 63.1 (CH_2OH), 79.7 (CHOH). − GC-LRMS: *m/z* (%) = 100 (M^+ − H_2O, 0.9), 87 (67), 69 (33), 61 (11), 57 (85), 56 (27), 55 (13), 44 (27), 43 (50).

1-Phenyl-1,2-ethanediol (3ac):[11a] white solid (m.p. 64°C, toluene), R_f = 0.26 (hexane/ethyl acetate, 3:7). − IR (KBr): $\tilde{\nu}$ = 3315 (OH), 1110, 1088, 1077, 1054, 1026 (CO) cm^{-1}. − ^1H NMR: δ = 3.36 (br s, 2 H, 2 × OH), 3.55−3.76 (m, 2 H, CH_2O), 4.72−4.78 (m, 1 H, CHO), 7.20−7.30 (m, 5 H, ArH). − ^{13}C NMR: δ = 67.95 (CH_2OH), 74.65 (CHOH), 126.0, 127.85, 128.45, 140.45 (4 × ArC). − GC-LRMS: *m/z* (%) = 138 (M^+, 6.5), 107 (100), 79 (80), 77 (54), 51 (20).

2-Ethyl-1,2-butanediol (3ad):[23] white solid (m.p. 42−43°C, toluene), R_f = 0.29 (hexane/ethyl acetate, 3:7). − IR (KBr): $\tilde{\nu}$ = 3404 (OH), 1073, 1055 (CO) cm^{-1}. − ^1H NMR: δ = 0.87 (t, J = 7.6 Hz, 6 H, 2 × CH_3CH_2), 1.51 (2 q, J = 7.3 Hz, 4 H, 2 × CH_2CH_3), 2.17, 2.35 (s, br s, 2 H, 2 × OH), 3.46 (s, 2 H, CH_2O). − ^{13}C NMR: δ = 7.6 (2 × CH_3CH_2), 27.55 (2 × CH_2CH_3), 67.3 (CH_2OH), 75.05 (COH). − GC-LRMS: *m/z* (%) = 101 (M^+ − OH, 0.3), 87 (45), 71 (125), 69 (13), 57 (15), 45 (100), 43 (83), 41 (60).

1-Hydroxymethylcyclohexanol (3ae):[11a] white solid (m.p. 75°C, toluene), R_f = 0.25 (hexane/ethyl acetate, 3:7). − IR (KBr): $\tilde{\nu}$ = 3287 (OH), 1081, 1044 (CO) cm^{-1}. − ^1H NMR: δ = 1.20−1.70 (m, 10 H, 5 × CH_2 cyclohexyl ring), 1.95, 2.15 (2 × br s, 2 H, 2 × OH), 3.45 (s, 2 H, CH_2O). − ^{13}C NMR: δ = 21.8 (2 C), 25.85, 34.0 (2 C) (5 × CH_2 cyclohexyl ring), 70.0 (CH_2OH), 71.9 (COH). − GC-LRMS: *m/z* (%) = 130 (M^+, 0.4), 99 (100), 87 (10), 81(79), 79 (17), 69 (10), 57 (16), 55 (40), 53 (11), 43 (44).

2-Phenyl-1,2-propanediol (3af):[24] white solid (m.p. 106−108°C, toluene), R_f = 0.30 (hexane/ethyl acetate, 3:7). − IR (KBr): $\tilde{\nu}$ = 3398 (OH), 1603, 1493 (Ph), 1069, 1044, 1027 (CO) cm^{-1}. − ^1H NMR: δ = 1.42 (s, 3 H, CH_3C), 1.96, 2.68 (2 s, 2 H, 2 × OH), 3.51 (d, J = 11.3 Hz, 1 H, CHHO), 3.67 (d, J = 11.3 Hz, 1 H, CHHO), 7.15−7.40 (5 H, m, ArH). − ^{13}C NMR: δ = 25.95 (CH_3), 70.95 (CH_2), 74.8 (COH), 125.05, 127.05, 128.15, 145.05 (4 × ArC). − GC-LRMS: *m/z* (%) = 152 (M^+, 0.4), 121 (47), 105 (21), 77 (14), 51 (12), 43 (100).

1,1-Diphenyl-1,2-ethanediol (3ag):[25] white solid (m.p. 120−121°C, toluene), R_f = 0.55 (hexane/ethyl acetate, 3:7). − IR (KBr): $\tilde{\nu}$ = 3377, 3311 (OH), 3083, 3057, 3023 (Ph), 1104, 1070, 1044 (CO) cm^{-1}. − ^1H NMR: δ (CD_3OD) = 4.14 (s, 2 H, CH_2), 7.23−7.48 (m, 10 H, ArH). − ^{13}C NMR: δ = 70.75 (CH_2), 80.35 (COH), 128.65, 128.7, 129.75, 147.3 (8 × ArC). − GC-LRMS: *m/z* (%) = 196 (M^+ − H_2O, 3.2), 168 (77), 167 (15), 166 (36), 165 (43), 152 (27), 82 (15), 82 (11), 51 (10).

4,4-Dimethyl-2,3-pentanediol (3bb).[26] **First diastereomer**: white solid (m.p. 71−72°C, hexane), R_f = 0.34 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3381 (OH), 1073, 1040, 1013, 1001 (CO) cm^{-1}. − ^1H NMR: δ = 0.97 [s, 9 H, $(CH_3)_3C$], 1.23 (d, J = 6.1 Hz, 3 H, CH_3CH), 1.96, 2.10 (2 br s, 2 H, 2 × OH), 3.40 [d, J = 3.1 Hz, 1 H, $CHC(CH_3)_3$], 3.95 (qd, J = 6.1, 3.1 Hz, 1 H, $CHCH_3$). − ^{13}C NMR: δ = 18.55 (CH_3CH), 26.65 [$(CH_3)_3C$], 34.0 [$(CH_3)_3C$], 68.5 [$CHC(CH_3)_3$], 82.55 ($CHCH_3$). − GC-LRMS: *m/z* (%) = 114 (M^+ − H_2O, 0.15), 87 (40), 75 (16), 73 (13), 69 (41), 57 (100), 45 (37), 43 (42), 41 (60). **Second diastereomer:** oil, R_f 0.31 (hexane/ethyl acetate, 1:1). − IR (film): $\tilde{\nu}$ = 3416 (OH), 1132, 1067, 1016 (CO) cm^{-1}. − ^1H NMR: δ = 0.95 [s, 9 H, $(CH_3)_3C$], 1.25 (d, J = 6.1 Hz, 3 H, CH_3CH), 2.50−2.90 (br s, 2 H, 2 × OH), 2.98 [d, J = 1.2 Hz, 1 H, $CHC(CH_3)_3$], 3.98 (qd, J = 6.1, 1.2 Hz, 1 H, $CHCH_3$). − ^{13}C NMR: δ = 22.55 (CH_3CH), 25.8 [$(CH_3)_3C$], 33.5 [$(CH_3)_3C$], 65.2 [$CHC(CH_3)_3$], 80.9 ($CHCH_3$). − GC-LRMS: *m/z* (%) = 114 (M^+ − H_2O, 0.2), 87 (36), 75 (26), 73 (14), 69 (37), 57 (100), 45 (55).

3-Ethyl-2,3-pentanediol (3bd):[27] oil, R_f = 0.33 (hexane/ethyl acetate, 1:1). − IR (film): $\tilde{\nu}$ = 3486 (OH), 1100, 1086 (CO) cm^{-1}. − ^1H NMR: δ = 0.88 (t, J = 7.6 Hz, 6 H, 2 × CH_3CH_2), 1.15 (d, J = 6.1 Hz, 3 H, CH_3CH), 1.31−1.69 (m, 4 H, 2 × CH_2), 2.44 (br s, 2 H, 2 × OH), 3.73 (q, J = 6.1 Hz, 1 H, CH_3CH). − ^{13}C NMR: δ = 7.5, 7.6 (2 × CH_3CH_2), 17.1 (CH_3CH), 25.95, 27.5 (2 × CH_2), 70.95 (CH), 76.25 (COH). − GC-LRMS: *m/z* (%) = 117 (M^+ − CH_3, 0.16), 103 (15), 87 (57), 85 (13), 69 (31), 57 (75), 55 (15), 45 (100), 43 (99), 41 (61).

1-(1′-Hydroxyethyl)-1-cyclohexanol (3be):[28] oil, R_f = 0.29 (hexane/ethyl acetate, 1:1). − IR (film): $\tilde{\nu}$ = 3404 (OH), 1097, 1075, 1048 (CO) cm^{-1}. − ^1H NMR: δ = 1.16 (d, J = 6.7 Hz, 3 H, CH_3), 1.20−1.70 (m, 10 H, 5 × CH_2), 2.00−2.45 (2 × br s, 2 H, 2 × OH), 3.57 (q, J = 6.7 Hz, 1 H, CH). − ^{13}C NMR: δ = 16.95 (CH_3), 21.4, 21.6, 25.85, 31.25, 34.15 (5 × CH_2), 73.4 (COH), 73.75 (CH). − GC-LRMS: *m/z* (%) = 126 (M^+ − H_2O, 0.2), 99 (77), 81 (100), 79 (15), 57 (20), 55 (65), 45 (19), 43 (70), 42 (18), 41 (39).

2-Phenyl-2,3-butanediol (3bf):[26] white solid (m.p. 46°C, hexane), R_f = 0.30 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3416 (OH), 1602, 1493, 1447 (Ph), 1080, 1067, 1058, 1050 (CO) cm^{-1}. − ^1H NMR: δ = 0.94 (d, J = 6.1 Hz, 3 H, CH_3CH), 1.59 (s, 3 H, CH_3C), 2.50 (s, 2 H, 2 × OH), 3.87 (q, J = 6.1 Hz, 1 H, CH), 7.21−7.44 (m, 5H, ArH). − ^{13}C NMR: δ = 17.6 (*C*H_3CH), 26.6 (*C*H_3C), 74.25 (CH), 76.45 (COH), 125.35, 126.8, 128.05, 144.54 (4 × ArC). − GC-LRMS: *m/z* (%) = 148 (M^+ − H_2O, 0.25), 122 (18), 121 (43), 104 (12), 77 (23), 51 (19), 45 (11), 43 (100).

2-Phenyl-2,3-butanediol (3bf′):[26] oil, R_f = 0.36 (hexane/ethyl acetate, 1:1). − IR (film): $\tilde{\nu}$ = 3420 (OH), 1606, 1499 (Ph), 1034, 1112 (CO) cm^{-1}. − ^1H NMR: δ = 1.09 (d, J = 6.1 Hz, 3 H, CH_3CH), 1.48 (s, 3 H, CH_3C), 2.74, 3.25 (2 s, 2 H, 2 × OH), 3.93 (q, J = 6.1 Hz, 1 H, CH), 7.21−7.44 (m, 5 H, ArH). − ^{13}C NMR: δ = 16.6 (*C*H_3CH), 22.75 (*C*H_3C), 74.05 (CH), 76.55 (COH), 125.55, 126.85, 128.05, 146.0 (4 × ArC). − GC-LRMS: *m/z* (%) = 148 (M^+ − H_2O, 0.3), 121 (18), 105 (6), 77 (8), 43 (100).

1-Cyclohexyl-2-ethyl-1,2-butanediol (3cd):[29] white solid (m.p. 144−150°C, hexane), R_f = 0.70 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3421, 3345 (OH), 1134, 11095, 1086 (CO) cm^{-1}. − ^1H NMR: δ = 0.86, 0.89 (2 t, J = 7.9, 7.9 Hz, 6 H, 2 × CH_3), 1.10−2.02 (m, 17 H, 7 × CH_2, C*H*CHO, 2 × OH), 3.26 (d, J = 4.3 Hz, 1 H, CHC*H*O). − ^{13}C NMR: δ = 7.75, 7.9 (2 × CH_3), 26.3 (2 C), 26.6 (2 C), 26.85 (5 × CH_2 cyclohexyl ring), 28.5, 32.1 (2 × *C*H_2CH_3), 39.0 (*C*HCHO), 77.1 (COH), 77.95 (CH*C*HO). − GC-LRMS: *m/z* (%) = 171 (M^+ − CH_2CH_3, 1), 87 (100), 86 (18), 69 (14), 57 (45), 55 (28), 45 (78).

1-(Cyclohexylhydroxymethyl)cyclohexanol (3ce): white solid (m.p. 90−92°C, hexane), R_f = 0.58 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3415 (OH), 1098, 1085 (CO) cm^{-1}. − ^1H NMR: δ = 1.05−1.95 (m, 22 H, 10 × CH_2, C*H*CHO, OH), 2.04 (d, J = 6.7 Hz, 1 H, OH) 3.26 (dd, J = 6.7, 1.8 Hz, 1 H, CHC*H*O). − ^{13}C NMR: δ = 21.8, 21.85, 25.75, 26.2, 26.3, 26.55, 26.8, 32.25, 33.0, 36.0 (10 × CH_2), 38.75 (*C*HCHO), 74.0 (COH), 80.6 (CH*C*HO). − GC-LRMS: *m/z* (%) = 212 (M^+, 0.1), 99 (100), 98 (66), 83 (13), 81 (76), 67 (16), 57 (16), 55 (51), 53 (11), 43 (36). − HRMS: *m/z* = 212.1774 (M^+); calcd. for $C_{13}H_{24}O_2$: 212.1776.

1-Cyclohexyl-2-phenyl-1,2-propanediol (3cf): white solid (m.p. 113−115°C, hexane), R_f = 0.65 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3462, 3341 (OH), 1603, 1494 (Ph), 1120, 1098, 1063 (CO) cm^{-1}. − ^1H NMR: δ = 0.95−1.80 (m, 11 H, 5 × CH_2, C*H*CHO), 1.62 (s, 3 H, CH_3) 2.07 (d, J = 5.8 Hz, 1 H, OH) 2.37 (s, 1 H, OH), 3.52 (d, J = 5.8 Hz, 1 H, CHC*H*O), 7.20−7.45 (m, 5 H, ArH). − ^{13}C NMR: δ = 25.95, 26.15 (2 C), 26.5, 32.1 (5 × CH_2), 29.1 (CH_3), 39.15 (*C*HCHO), 77.4 (COH), 81.6 (CH*C*HO), 124.9, 126.65, 128.1, 145.35 (4 × ArC). − GC-LRMS: *m/z* (%) = 216 (M^+ −H_2O, 1.5), 111 (25), 105 (20), 83 (100), 55 (52), 41 (26). − HRMS: *m/z* = 216.1517 (M^+ − H_2O); calcd. for $C_{15}H_{20}O$: 216.1514. − $C_{15}H_{22}O_2$ (234.33): calcd. C 76.88, H 9.46; found C, 77.19, H 9.56.

1-Cyclohexyl-2-phenyl-1,2-propanediol (3cf′): white solid (m.p. 90−91°C, hexane), R_f = 0.66 (hexane/ethyl acetate, 1:1). − IR (KBr): $\tilde{\nu}$ = 3345 (OH), 1600, 1493 (Ph), 1082, 1074, 1045 (CO) cm^{-1}. − ^1H NMR: δ = 1.10−1.78 (m, 10 H, 5 × CH_2), 1.54 (s, 3 H, CH_3), 1.79 (d, J = 4.2 Hz, 1 H, OH), 1.96 (m, 1 H, C*H*CHO), 2.71 (s, 1 H, OH), 3.61 (t, J = 3.9 Hz, 1 H, CHC*H*O), 7.20−7.50 (m, 5 H, ArH). − ^{13}C NMR: δ = 24.35 (CH_3), 26.2 (2 C), 26.6, 26.8, 32.05 (5 × CH_2), 38.9 (*C*HCHO), 76.9 (COH), 81.55 (CH*C*HO), 125.35, 126.85, 128.1, 147.05 (4 × ArC). − GC-LRMS: *m/z* (%) = 216 (M^+ − H_2O, 0.5), 187 (24), 134 (34), 105 (100), 91 (25), 77 (12), 55 (23), 41 (29). − HRMS: *m/z* = 216.1536 (M^+ − H_2O); calcd. for $C_{15}H_{20}O$: 216.1514.

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