Synthesis of β-Lactones *via* a Spontaneous Intramolecular Cyclization of *O*-Lithiated Phenyl β-Hydroxyalkanoates Obtained by Aldolization of Ketones or Aldehydes with Lithium Enolates of Phenyl Esters

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The interest in β-lactones has steadily grown during the last years due to their versatile applicability as intermediates for the synthesis of more elaborate targets.[1] This development is mainly based on the progress made in the decarboxylation of β-lactones to olefins,[2–4] in the stereoselective reactions of β-lactone enolates with a variety of electrophiles,[4,5] and in the control of the regioselective fission of the β-lactone ring by many different nucleophiles.[6] Independently, much attention has been focused on this class of strained four-membered rings, at least after the detection of the β-lactone moiety as a constituent in a series of biologically active natural products like lipstatin,[7] lupeolactone,[8] or valilactone.[9] Although there exist several methods for the preparation of β-lactones, most of them are now synthesized by [2 + 2] cycloaddition of carbonyl compounds to ketenes or by intramolecular acylation of the hydroxy group of activated β-hydroxyalkanoic acid derivatives.[1] The cyclization of β-hydroxyalkanoic acids with benzenesulfonyl chloride in pyridine has proved to be one of the most efficient β-lactone syntheses.[2] This method has been supplemented in recent years by the spontaneous intramolecular cyclization of lithiated *S*-phenyl β-hydroxyalkanethioates prepared by the aldolization of aldehydes or ketones with the lithium enolates of *S*-phenyl alkanethioates.[4] This method provides not only tetra- and trisubstituted β-lactones in good to excellent yields, but also disubstituted ones.

In connection with a recently published synthesis of tetrasubstituted β-lactones by a Reformatsky reaction of ethyl α-bromoisobutyrate and ketones with zinc or indium in *N,N*-dimethylformamide it has been found that *O*-metalated ethyl β-hydroxyalkanoates can cyclize by an intramolecular acylation to β-lactones if all hydrogen atoms at C_α and C_β are replaced by alkyl or aryl substituents.[10] This has been explained by the *gem*-dialkyl effect.[11,12] In connection with attempts to broaden the scope of this synthesis to trisubstituted β-lactones the question arose, whether the expected decrease of the *gem*-dialkyl effect could be compensated by a proper choice of the ester group, which has to be eliminated as metal alkoxide in the course of the β-lactone formation. In this paper we describe the results obtained with *O*-lithiated β-hydroxyalkanoates.

Table 1. β-Lactones 7 Prepared from the Phenyl Alkanoates 1a–d

entry	β-lactone 7	R^1	R^2	R^3	R^4	yield of 7[a] (%)	yield of 5[b] (%)
1	**7aa**	Me	Me	$-(CH_2)_5-$		86	<1
2	**7ab**	Me	Me	Et	Et	75	3
3	**7ac**	Me	Me	*n*-Bu	H	75	<1
4	**7ad**	Me	Me	*t*-Bu	H	60	<1
5	**7ae**	Me	Me	Ph	H	66	4
6	**7ba**	H	Et	$-(CH_2)_5-$		73	6
7	**7bb**	H	Et	Et	Et	78	9
8	**7bc**	H	Et	*n*-Bu	H	32[c]	24
9	**7bd**	H	Et	*t*-Bu	H	35[d]	5
10	**7be**	H	Et	Ph	H	52[d]	<1
11	**7ca**	H	*n*-Hx[e]	$-(CH_2)_5-$		70	2
12	**7cb**	H	*n*-Hx	Et	Et	73	13
13	**7cc**	H	*n*-Hx	*n*-Bu	H	10[f]	<1
14	**7cd**	H	*n*-Hx	*t*-Bu	H	64[d]	16
15	**7ce**	H	*n*-Hx	Ph	H	20[d]	40
16	**7da**	H	*i*-Pr	$-(CH_2)_5-$		63	<1
17	**7db**	H	*i*-Pr	Et	Et	8[g]	<1
18	**7dc**	H	*i*-Pr	*n*-Bu	H	40[f]	<1
19	**7dd**	H	*i*-Pr	*t*-Bu	H	10[g]	4
20	**7de**	H	*i*-Pr	Ph	H	5[g]	60

[a] Yield of isolated β-lactones **7**, if not stated otherwise. [b] The yield was determined by HPLC of the crude reaction product. [c] A 3:1 mixture of *trans*- and *cis*-substituted β-lactone was isolated. [d] The isolated β-lactone was *trans*-substituted. [e] *n*-Hx = *n*-hexyl. [f] The isolated β-lactone was *cis*-substituted. [g] This β-lactone was not isolated in analytically pure form. The yield was determined by HPLC.

Deprotonation of phenyl isobutyrate (**1a**) with lithium diisopropylamide in THF at −78 °C and reaction of the formed phenyl ester enolate **2a** with cyclohexanone (**3a**) afforded the tetrasubstituted β-lactone **7aa**[13] in an isolated yield of 86% (Table 1, entry 1). This experiment clearly revealed that the *O*-lithiated phenyl β-hydroxyalkanoate **4aa** is sufficiently activated to cyclize to a β-lactone by elimination of lithium phenoxide. The β-hydroxy ester **5aa** was not formed in this reaction. The tetrasubstituted β-lactone **7ab** was obtained in a comparable yield, when diethyl ketone (**3b**) was used instead of cyclohexanone (entry 2).

In order to find out whether phenyl esters of *O*-lithiated β-hydroxyalkanoates are also suited for the preparation of less substituted β-lactones, phenyl isobutyrate (**1a**) was subjected to the aldolization with valeraldehyde (**3c**), pivalaldehyde (**3d**), and benzaldehyde (**3e**). In all three cases the α,α,β-trisubstituted β-lactones **7ac**, **7ad**, and **7ae** could be isolated in yields between 60 and 75% (entries 3–5). Moreover, also the corresponding

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(13) The meaning of R^1, R^2, R^3, and R^4 of the compounds **4**, **5**, **6**, and **7** is given in Table 1.

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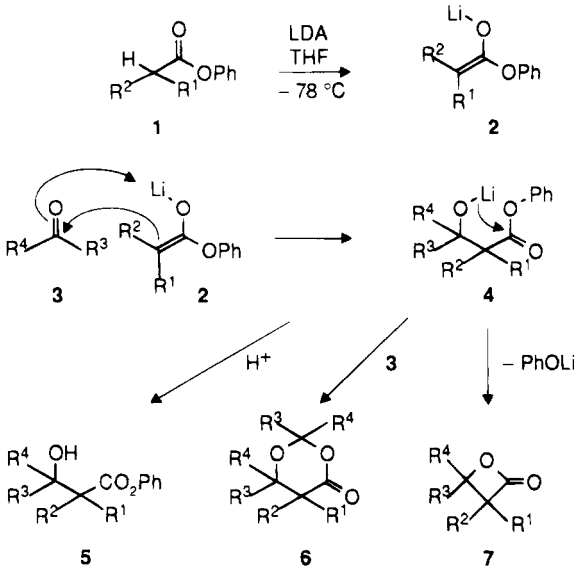
Scheme 1



1, 2	R^1	R^2
a	Me	Me
b	H	Et
c	H	*n*-Hex
d	H	*i*-Pr

3	R^3	R^4
a	$-(CH_2)_5-$	
b	Et	Et
c	*n*-Bu	H
d	*t*-Bu	H
e	Ph	H

α,β,β-trisubstituted β-lactones **7** were available by aldolization of cyclohexanone (**3a**) and diethyl ketone (**3b**) with the lithium ester enolates from phenyl butyrate (**1b**), phenyl octanoate (**1c**), and phenyl isovalerate (**1d**), respectively (entries 6, 7, 11, 12, 16, and 17). The yields of isolated β-lactones were in all these cases again in the order of 60–75%, except for **7db** (entry 17).[24]

However, the ability of the *O*-lithiated phenyl β-hydroxyalkanoates **4** to form β-lactones decreased when the number of the substituents at C_α and C_β was further reduced. The aldolization of phenyl butanoate (**1b**) with the aldehydes **3c–e** provided the corresponding α,β-disubstituted β-lactones **7** in yields of 32–52% (entries 8–10). The yields of β-lactones were still lower, when the β-branched phenyl isovalerate (**1d**) was used for the aldolization reaction (entries 18–20). In these attempts to prepare α,β-disubstituted β-lactones the reaction was much less selective and a greater number of side products was formed. Among them the phenyl β-hydroxyalkanoates **5** and the 2,5,6-trisubstituted 1,3-dioxan-4-ones **6** could be identified. The latter have been reported to be formed also to a considerable extent when the ester enolates of *S*-phenyl alkanethioates were reacted with aldehydes.[4] β-Keto phenyl esters formed by the self condensation of the ester enolates could also be isolated.

In all cases, where the α,β-disubstituted β-lactones could be separated from the byproducts by flash chromatography their structure was assigned on the basis of the coupling constants of the protons at C_α and C_β. According to the literature[14] the coupling constants $J_{\alpha,\beta}$ of the protons at C_α and C_β of *cis*- and *trans*-disubstituted β-lactones are about 6.5 and 4.5 Hz, respectively. On this basis the isolated product of the α,β-disubstituted β-lactone **7bc** was a 3:1 mixture of *trans*- and *cis*-compound. The β-lactones **7bd**, **7be**, **7cd**, and **7ce** derived from the straight-chain phenyl alkanoates **1b** and **1c** were *trans*-configured, whereas the isolated diastereoisomer of **7dc** derived from the β-branched phenyl alkanoate **1d** was *cis*-configured. The *cis*-configuration was also assigned to the isolated diastereoisomer of **7cc**.

The simple and efficient synthesis of tri-and tetrasubstituted β-lactones outlined above deserves some comment. The aldolization of aldehydes or ketones with alkyl ester enolates of alkanoic acids has found broad application for the synthesis of alkyl β-hydroxyalkanoates.[15] Methyl,[16] ethyl,[17] isopropyl,[18] and *tert*-butyl esters[19,20] have most often used as precursors for the ester enolates. To our knowledge, phenyl esters have so far not been included in this type of aldolization reactions. Their ability to form β-lactones under these conditions has therefore been overlooked. The activation of the carbonyl group by the phenoxy group revealed to be high enough to allow the intramolecular acylation of the *O*-lithiated phenyl β-hydroxyalkanoates **4** to the corresponding β-lactones **7** *via* the elimination of lithium phenoxide. Since phenyl alkanoates are easily prepared from phenol and the corresponding alkanoyl chlorides[20] and are not characterized by an unpleasant odor, they are more conveniently to handle than the analogous *S*-phenyl alkanethioates.[4] Therefore, the former should become the preferred starting material for the synthesis of tri- and tetrasubstituted β-lactones. In respect to β-lactones having this substitution pattern the described aldolization of phenyl ester enolates and the subsequent spontaneous cyclization of the *O*-lithiated phenyl β-hydroxyalkanoates competes well with other methods[1,2,4] for the preparation of β-lactones.

Experimental Section

General. The β-lactone syntheses were carried out under argon in flame-dried glassware. THF, dried over molecular sieves, was purchased from Fluka. The ketones and aldehydes, purchased from Merck and Fluka, were distilled prior to use. The phenyl alkanoates **1a**,[21] **1b**,[22] **1c**,[22] and **1d**[23] were prepared by refluxing the corresponding commercially available alkanoyl chlorides with phenol and a trace of H_2SO_4 in toluene following the literature.[21] ^{13}C NMR and ^1H NMR spectra were recorded in $CDCl_3$ at 75 and 300 MHz, respectively. The IR spectra were measured from films, if not stated otherwise.

General Procedure for the Preparation of the β-Lactones 7 from the Phenyl Ester Enolates 2 and the Carbonyl Compounds 3. A 1.0 N solution of *n*-butyllithium in hexane (6.0 mL, 6.0 mmol) was added by syringe at a tempera-

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(24) Note added in proof: Cyclopentanone failed to form the corresponding β-lactone in the reaction with the lithium ester enolate from phenyl butyrate (**1b**). Cycloheptanone, phenylacetone, and heptan-4-one, however, afforded the corresponding β-lactones with **1b** in yields of 72, 73, and 65%, respectively. 3-Methylbutan-2-one and 2-methylcyclohexanone provided the corresponding β-lactones in the reaction with the lithium ester enolate from phenyl propanoate in yields of 70 and 80%, respectively.

ture of −10 °C during 1 min to diisopropylamine (0.92 mL, 6.6 mmol) in THF (20 mL). The mixture was allowed to warm up to 22 °C within 15 min and thereafter cooled again to −78 °C. Then a phenyl ester **1** (6 mmol) dissolved in THF (5 mL) was added over 2 min. After stirring the mixture at this temperature for 30 min a carbonyl compound **3** (5 mmol) dissolved in THF (5 mL) was added from a dropping funnel equipped with a cooling jacket filled with acetone and dry ice. The mixture was kept at −70 °C for 30 min and then allowed to warm to 0 °C over 2 h. After addition of 1 N aqueous NaOH (15 mL, 15 mmol), water (50 mL), and diethyl ether (50 mL) the organic phase was separated, washed with 1 N NaOH (15 mL) and brine (2 × 20 mL), dried over Na_2SO_4, and filtered. Evaporation of the solvent under reduced pressure afforded a pale yellow oil, from which the corresponding β-lactone **7** was isolated by flash chromatography on silica gel with hexane/ethyl acetate (15:1) as eluent. The yields of isolated products are given in Table 1. According to this general procedure the following β-lactones have been prepared.

3,3-Dimethyl-1-oxaspiro[3.5]nonan-2-one (7aa).[2,4] Colorless crystals from hexane: mp 108−110 °C; IR ($CHCl_3$) 1810 cm^{-1}; ^{13}C NMR δ 18.1, 22.7, 24.8, 32.3, 54.3, 85.1, 176.2.

4,4-Diethyl-3,3-dimethyl-2-oxetanone (7ab). Colorless oil: IR 1817 cm^{-1}; ^{13}C NMR δ 8.2, 18.7, 25.0, 54.6, 88.3, 176.1. Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 68.79; H, 10.35.

(±)-4-Butyl-3,3-dimethyl-2-oxetanone (7ac). Colorless oil: IR 1823 cm^{-1}; ^{13}C NMR δ 13.9, 16.3, 22.5, 22.6, 27.7, 30.2, 53.3, 83.6, 175.6. Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 68.91; H, 10.33.

(±)-4-*tert*-Butyl-3,3-dimethyl-2-oxetanone (7ad). Colorless crystals: mp 33−35 °C; IR 1823 cm^{-1}; ^{13}C NMR δ 17.7, 25.5, 25.8, 33.6, 53.3, 90.4, 175.6. Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 68.91; H, 10.39.

(±)-3,3-Dimethyl-4-phenyl-2-oxetanone (7ae). Colorless oil: IR 1827 cm^{-1}; ^{13}C NMR δ 17.9, 22.4, 56.6, 82.7, 125.1, 128.4, 128.6, 135.3, 174.9. Anal. Calcd for $C_{11}H_{12}O_2$: C, 74.98; H, 6.86. Found: C, 74.95; H, 6.98.

(±)-3-Ethyl-1-oxaspiro[3.5]nonan-2-one (7ba). Colorless oil: IR 1814 cm^{-1}; ^{13}C NMR δ 12.3, 17.5, 22.2, 22.9, 24.9, 31.2, 37.5, 59.8, 82.1, 172.1. Anal. Calcd for $C_{10}H_{16}O_2$: C, 71.39; H, 9.59. Found: C, 71.34; H, 9.91.

(±)-3,4,4-Triethyl-2-oxetanone (7bb). Colorless oil: IR 1814 cm^{-1}; ^{13}C NMR δ 7.1, 7.8, 12.1, 17.5, 24.1, 29.0, 58.3, 84.5, 171.6. Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32 Found: C, 68.97; H, 10.48.

(±)-*trans*- and (±)-cis-4-Butyl-3-ethyl-2-oxetanone (7bc). Colorless oil: IR 1823 cm^{-1}; ^1H NMR δ 0.86 (3 H, t, J = 7 Hz), 0.98 (3 H, t, J = 7 Hz), 1.27−1.80 (8 H, m), 3.04−3.11 (0.76 H, m, *trans*), 3.42−3.52 (0.24 H, m, *cis*), 4.14−4.20 (0.76 H, m, *trans*), 4.42−4.51 (0.24 H, m, *cis*); ^{13}C NMR δ 11.3, 13.9, 21.1, 22.4, 27.1, 34.1, 57.5, 77.7, 171.5. Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 69.45; H, 10.54.

(±)-*trans*-4-*tert*-Butyl-3-ethyl-2-oxetanone (7bd). Colorless oil: IR 1826 cm^{-1}; ^1H NMR δ 0.93 (9 H, s), 1.63−1.85 (2 H, m), 3.15−3.21 (1 H, m), 3.89 (1 H, d, J = 4.1 Hz); ^{13}C NMR δ 11.4, 21.7, 24.3, 32.7, 52.3, 84.6, 171.4. Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 68.82; H, 10.01.

(±)-*trans*-3-Ethyl-4-phenyl-2-oxetanone (7be). Colorless oil: IR 1827 cm^{-1}; ^1H NMR δ 1.06 (3 H, t, J = 7 Hz), 1.80−2.00 (2 H, m), 3.40−3.47 (1 H, m), 5.16 (1H, d, J = 4.1 Hz), 7.20−7.40 (5 H, m); ^{13}C NMR δ 11.2, 21.3, 61.2, 77.2, 125.4, 128.9, 129.0, 137.3, 171.1. Anal. Calcd for $C_{11}H_{12}O_2$: C, 74.98; H, 6.86. Found: C, 75.15; H, 7.08.

(±)-3-Hexyl-1-oxaspiro[3.5]nonan-2-one (7ca). Colorless oil: IR 1815 cm^{-1}; ^{13}C NMR δ 14.0, 22.2, 22.6, 22.9, 24.0, 25.0, 27.7, 29.1, 31.3, 31.5, 37.4, 58.2, 82.1, 172.2. Anal. Calcd for $C_{14}H_{24}O_2$: C, 74.95; H, 10.78. Found: C, 74.77; H, 10.52.

(±)-4,4-Diethyl-3-hexyl-2-oxetanone (7cb). Colorless oil: IR 1817 cm^{-1}; ^{13}C NMR δ 7.4, 8.2, 14.0, 22.6, 24.4, 24.5, 27.9, 29.1, 29.3, 31.5, 57.0, 85.0, 172.2. Anal. Calcd for $C_{13}H_{24}O_2$: C, 73.54; H, 11.39. Found: C, 73.69; H, 11.43.

(±)-*cis*-4-Butyl-3-hexyl-2-oxetanone (7cc). Colorless oil: IR 1822 cm^{-1}; ^1H NMR δ 0.82−1.09 (6 H, m), 1.22−1.90 (16 H, m), 3.48−3.58 (1 H, dt, J = 7, 6 Hz), 4.41−4.51 (1H, m); ^{13}C NMR δ 13.9, 14.0, 22.4, 22.5, 23.9, 27.6, 27.7, 29.1, 29.9, 31.5, 52.6, 75.7, 172.4. Anal. Calcd for $C_{13}H_{24}O_2$: C, 73.54; H, 11.39. Found: C, 73.24; H, 11.81.

(±)-*trans*-4-*tert*-Butyl-3-hexyl-2-oxetanone (7cd). Colorless oil: IR 1823 cm^{-1}; ^1H NMR 0.82 (3 H, m), 0.92 (9 H, s), 1.01−1.83 (10 H, m), 3.15−3.25 (1 H, m), 3.87 (1H, d, J = 4.1 Hz); ^{13}C NMR δ 14.0, 22.5, 24.4, 26.9, 28.5, 29.0, 31.5, 32.8, 51.0, 85.1, 171.6. Anal. Calcd for $C_{13}H_{24}O_2$: C, 73.54; H, 11.39. Found: C, 73.69; H, 11.43.

(±)-*trans*-3-Hexyl-4-phenyl-2-oxetanone (7ce). Colorless oil: IR 1826 cm^{-1}; ^1H NMR δ 0.82 (3 H, m), 1.11−1.98 (10 H, m), 3.40−3.48 (1 H, m), 5.12 (1 H, d, J = 4.0 Hz), 7.20−7.35 (5 H, m); ^{13}C NMR δ 14.0, 22.5, 26.9, 28.1, 28.9, 31.5, 59.9, 77.7, 125.5, 128.9, 129.0, 137.4, 171.3. Anal. Calcd for $C_{15}H_{20}O_2$: C, 77.55; H, 8.68. Found: C, 77.78; H, 9.03.

(±)-3-Isopropyl-1-oxaspiro[3.5]nonan-2-one (7da). Colorless crystals: mp 43−45 °C; IR 1817 cm^{-1}; ^{13}C NMR δ 20.2, 21.8, 22.2, 22.7, 24.4, 25.1, 31.2, 37.6, 65.7, 82.4, 171.3. Anal. Calcd for $C_{11}H_{18}O_2$: C, 72.49; H, 9.95. Found: C, 72.58; H, 10.22.

(±)-4,4-Diethyl-3-isopropyl-2-oxetanone (7db). Colorless oil: IR 1816 cm^{-1}; ^{13}C NMR δ 7.3, 8.4, 20.4, 22.5, 24.3, 24.7, 29.3, 64.1, 85.3, 171.3.

(±)-*cis*-4-Butyl-3-isopropyl-2-oxetanone (7dc). Colorless oil: IR 1819 cm^{-1}; ^1H NMR δ 0.82−0.91 (6 H, m), 1.10 (3 H, d, J = 7 Hz), 1.25−1.80 (6 H, 3 m), 2.05−2.18 (1H, m), 3.20−3.26 (1 H, dd, J = 11, 6.4 Hz), 4.42−4.51 (1 H, m); ^{13}C NMR δ 13.9, 20.3, 22.1, 22.4, 24.6, 27.9, 29.8, 59.8, 76.0, 171.5. Anal. Calcd for $C_{10}H_{18}O_2$: C, 70.55; H, 10.66. Found: C, 70.56; H, 10.83.

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