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Diastereomerically Pure 3-Alkoxy- and 3-Acyloxyoxetanes from 3-Silyloxyoxetanes. A Case Study

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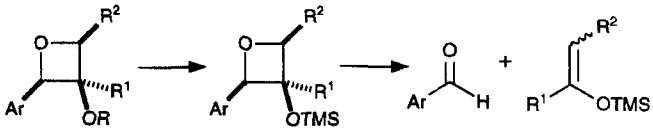
3-Alkoxy- or 3-acyloxysubstituted oxetanes may be obtained by photocycloaddition of a carbonyl compound with an enol ether [1] or an enol alkanoate [2], respectively. However, the variety of enol ethers which can be employed for this purpose is somewhat limited by their synthetic availability. They can, for example, not be obtained from the corresponding enolates due to competitive *C*-alkylation. Enol acetates or benzoates are more readily synthesized, but the yields of photocycloaddition products with carbonyl compounds are low [2] and the diastereoselectivity is often insufficient. Since we have recently shown that the readily available silyl enol ethers are excellent substrates in the Paternò-Büchi reaction with aromatic aldehydes [3], we wanted to demonstrate that a facile access to various other 3-alkoxy- and 3-acyloxyoxetanes can be achieved by protective group replacement reactions. The strategy is outlined in Scheme 1.



Scheme 1

The racemic 3-silyloxyoxetane **1** [3a] could be deprotected most conveniently and quantitatively by treatment with K_2CO_3 in methanol as previously reported for related systems [4]. This protocol proved superior to an acidic cleavage (HOAc in MeOH) [5] of the silyl ether which yielded only 79% of the desired oxetanol **2** under optimized condition.

Although there are plenty of methods in the literature for alcohol alkylation and acylation [6] we were well aware that only a few had a chance to be successful for this particular case. The hydroxyl group in **2** is very difficult to access for an electrophile. Moreover, the 2-phenyl substituent is a potential source for side reactions stabilizing both a negative or a positive charge adjacent to itself. If very reactive electrophiles are to be employed an attack at the oxetane ether oxygen may for example readily initiate a pinacol type rearrangement as has been observed in related systems [7]. The severe steric hindrance and the presence of the 2-aryl substituent make the described system different from other sterically less demanding 3-oxetanols, in particular from those related to the Taxol D-ring [8]. This fact was also reflected by the relative stability of the products. In table 1 the results of our investigations are summarized.

For the formation of 3-alkoxymethoxyoxetanes deprotonation of the oxetanol with *n*-butyllithium (*n*-BuLi) was mandatory. Upon subsequent treatment with methoxymethyl (MOM) or methoxyethoxymethyl (MEM) chloride a smooth substitution was observed which yielded the desired compounds **7** and **8** (entries 5 and 6). Contrary to that, other reported methods could not be employed successfully in this case. With $NEt{i}Pr_2$ as base [13] MOMCl reacted slowly (CH_2Cl_2, *r.t.*) and gave only a 28% yield of oxetane **7** after seven days. MEMCl showed a 14% conversion according to ^{1}H NMR under the same conditions. Conversions with NaH as base [12] (THF) were at 50% after five days. Moreover, an epimerisation was observed in the course of the reaction. Although the starting oxetanol was diastereomerically pure the crude reaction product showed two diastereomeric products in a ratio of 86:14 (according to ^{1}H NMR and GLC). A similar effect was observed in the benzylation series (entry 2). With NaH as base and benzyl bromide (BnBr) as the alkylating agent (TBAI as catalyst) [14] two diastereomeric benzyl ethers were obtained in a ratio of 65:35. We have not yet been able to establish the mechanism of the epimerisation, but one may consider either a deprotonation/protonation sequence at C-2 of the oxetanol or an alkoxide driven ring opening/ring closure. In the latter case fragmentation to the aldehyde and the enolate should be a competitive process which we found no indication for. The epimerisation was circumvented by the use of *n*-BuLi as initially mentioned. The *n*-BuLi protocol (procedure **B**) was less effective in the desired methylation (entry 1). After two days of stirring (THF, *r.t.*) there was still more than half of the starting material left. We therefore applied the KOH based method [9] although a

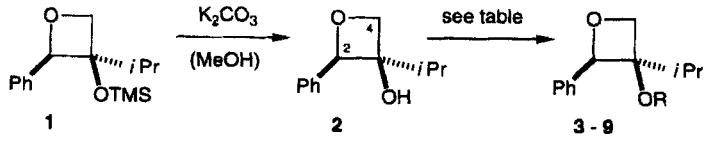


Table 1 Preparation and Acylation/Alkylation of 3-Oxetanol **2**

Entry	Conditions	Procedure	Time (h)	Temp. (°C)	Solvent	Prod.	R	Yield (%)
1	KOH (4 equiv.), MeI (2 equiv.) [9]	A	1	55	DMSO	**3**	Me	99
2	*n*-BuLi (1 equiv.), BnBr (2 equiv.) [a]	B	72	60	THF	**4**	Bn [b]	75
3	*n*-BuLi (1 equiv.), BzCl (1.7 equiv.) [10]	B	3	*r.t.* [c]	THF	**5**	Bz [d]	74
4	Ac_2O (2.5 equiv.), NEt_3 [e] (3.5 equiv.) [11]	C	48	*r.t.* [c]	CH_2Cl_2	**6**	Ac	80
5	*n*-BuLi (1 equiv.) MOMCl (1.7 equiv.)	B	48	*r.t.* [c]	THF	**7**	MOM [f]	84
6	*n*-BuLi (1 equiv.), MEMCl (1.7 equiv.) [12]	B	48	60	DME	**8**	MEM [g]	83
7	Boc_2O (5 equiv.), NEt_3 [e] (7 equiv.)	C	24	*r.t.* [c]	CH_2Cl_2	**9**	Boc [h]	99

[a] TBAI (tetrabutylammonium iodide) as catalyst; [b] Bn: benzyl; [c] *r.t.*: room temperature (25°C); [d] Bz: benzoyl; [e] DMAP (*N,N*-dimethylaminopyridine) as catalyst; [f] MOM: methoxymethyl; [g] MEM: methoxyethoxymethyl; [h] Boc: *t*-butyloxycarbonyl.

minor amount (≤ 5%) of epimerisation product was detected in some instances (entry 1, procedure **A**). An uncomplete reaction caused problems in the benzylation case (entry 2), too. With dimethoxyethane (DME) instead of the successfully employed THF as solvent only 25% of the oxetanol **2** were converted to its benzylether after four days at 60 °C. The benzoylation [10] (entry 3) and acetylation [11] (entry 4) proceeded smoothly according to published procedures. In terms of stability the acylated oxetanols **5**, **6** and **9** are highly superior to the alkylated analogues **3**, **4**, **7** and **8**. In particular, the MEM and the MOM protected derivatives decomposed rapidly upon standing at room temperature. A major decomposition product could unfortunately not be detected, rather there was a variety of products both on GLC and on TLC.

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Experimental

All reactions involving water sensitive chemicals were carried out in flame-dried glassware with magnetic stirring under Ar. Chemicals and solvents for this kind of reactions were distilled from an appropriate drying agent. Common solvents (cyclohexane, ethyl acetate) used for chromatography were distilled prior to use. The starting oxetane **1** was prepared as described previously [3a]. All other reagents and solvents were used as received. – Melting points: Reichert hot bench (uncorrected). – IR: Perkin Elmer 1605 FT or Perkin Elmer 298. – MS: Varian Saturn II ion trap instrument (GC/MS), Finnigan MAT 8230 (GC/MS) or Finnigan MAT 312. – ^1H and ^{13}C NMR: Bruker AM-360, or Bruker WM-300. Chemical shifts are reported in ppm relative to tetramethylsilane as an internal reference. $CDCl_3$ was used as solvent unless noted otherwise. The multiplicities of the ^{13}C NMR signals were determined with DEPT pulse sequences. – Elemental Analyses: Perkin Elmer 240. – TLC: glass-backed plates (Merck 0.25 mm silica gel 60-F); eluent given in brackets, a cyclohexane (CH)/ethyl acetate (EA) mixture was used unless stated otherwise; detection by UV or by coloration with ceric ammonium molybdate (CAM). – Flash chromatography [15] (FC): Merck silica gel 60 (230–400 mesh) (50 g for 1 g of material to be separated).

3-Hydroxy-3-(methylethyl)-2-phenyloxetane (**2**)

13.5 mmol of oxetane **1** (3.57 g) were dissolved in 100 ml of methanol which was saturated with K_2CO_3, and the solution was stirred for 4 h at room temperature. Upon dilution with 250 ml of ether the mixture was washed with water. The aqueous layer was extracted twice with ether. The combined organic layers were washed with brine, dried with $MgSO_4$ and filtered. After removal of the solvents *in vacuo* the desired oxetanol **2** was obtained (purity according to GLC: >95%). Yield: 2.43 g (94%). *m.p.* = 63–64 °C. – R_f = 0.18 (90/10). – IR (KBr): $\tilde{\nu}$ = 970 cm^{-1} (s, COC). – ^1H NMR (300 MHz):

δ 0.97 (d, 3 H, $^3J_{HH}$ = 6.7 Hz, C$\underline{H}_3$), 1.01 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.60 (s, 1 H, O$\underline{H}$), 2.24 [virt. sept, 1 H, $^3J_{HH} \cong$ 6.7 Hz, C$\underline{H}$(CH$_3$)$_2$], 4.45 (d, 1 H, $^2J_{HH}$ = 6.9 Hz, C$\underline{H}$H), 4.66 (d, 1 H, $^2J_{HH}$ = 6.9 Hz, CH$\underline{H}$), 5.57 (s, 1 H, PhC$\underline{H}$), 7.30–7.50 [m, 5 H, C$\underline{H}$(arom.)]. – ^{13}C NMR (75.5 MHz): δ 15.5 (q), 15.7 (q), 35.1 (d), 78.3 (s), 81.7 (t), 91.0 (d), 126.3 (d), 128.2 (d), 128.7 (d), 137.1 (s). – Anal. Calcd. for $C_{12}H_{16}O_2$ (192.257): C 74.97 H 8.39. Found: C 74.81 H 8.28.

3-Methoxy-3-(methylethyl)-2-phenyloxetane (**3**)

Procedure **A**.
2 mmol powdered KOH (112 mg) were dissolved in 1 ml of DMSO. Subsequently, 0.5 mmol of oxetanol **2** (96 mg) and 1 mmol methyl iodide (142 mg, 60 µl) were added rapidly. The mixture was warmed to 55 °C and stirred for 1 h. Upon quenching with 10 ml of H_2O the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with water, dried with $MgSO_4$ and filtered. After removal of the solvents *in vacuo* the desired oxetane **3** was obtained (purity according to GLC: 94%). Yield: 110 mg (quant.). R_f = 0.32 (90/10). – IR (film): $\tilde{\nu}$ = 975 cm^{-1} (vs, COC). – ^{1}H NMR (300 MHz): δ 1.06 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.12 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 2.23 [sept, 1 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}$(CH$_3$)$_2$], 2.87 (s, 3 H, OC$\underline{H}_3$), 4.59 (d, 1 H, $^2J_{HH}$ = 7.0 Hz, C$\underline{H}$H), 4.71 (d, 1 H, $^2J_{HH}$ = 7.0 Hz, CH$\underline{H}$), 5.56 (s, 1 H, PhC$\underline{H}$), 7.20–7.43 [m, 5 H, C$\underline{H}$(arom.)]. – ^{13}C NMR (75.5 MHz): δ 16.3 (q), 16.6 (q), 30.8 (d), 51.1 (q), 75.2 (t), 83.3 (s), 88.1 (d), 126.5 (d), 127.4 (d), 127.8 (d), 138.6. – Anal. Calcd. for $C_{13}H_{18}O_2$ (206.285): C 75.69 H 8.79. Found: C 75.59 H 8.98.

3-Benzyloxy-3-(methylethyl)-2-phenyloxetane (**4**)

According to the protocol described in procedure **B** (*vide supra*) 5 mmol of oxetanol **2** (960 mg) were transformed into the desired product employing the conditions indicated in table 1. Purification by flash chromatography (CH/EA = 99/1). Yield: 1.06 g (75%). R_f = 0.30 (90/10). – IR (film): $\tilde{\nu}$ = 975 cm^{-1} (vs, COC). – ^{1}H NMR (300 MHz): δ 1.14 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.18 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 2.36 [sept, 1 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}$(CH$_3$)$_2$], 4.12 (s, 2 H, OC$\underline{H}_2$Ph), 4.65 (d, 1 H, $^2J_{HH}$ = 6.9 Hz, C$\underline{H}$H), 4.81 (d, 1 H, $^2J_{HH}$ = 6.9 Hz, CH$\underline{H}$), 5.66 (s, 1 H, PhC$\underline{H}$), 6.80–6.86 [m, 2 H, C$\underline{H}$(arom.)], 7.11–7.18 [m, 3 H, C$\underline{H}$(arom.)], 7.24–7.37 [m, 3 H, C$\underline{H}$ (arom.)], 7.46 [d, 2 H, $^3J_{HH}$ = 7.4 Hz, C$\underline{H}$(arom.)]. – ^{13}C NMR (75.5 MHz): δ 16.5 (q), 16.5 (q), 32.1 (d), 65.5 (t), 75.8 (t), 83.4 (s), 88.8 (d), 126.5 (d), 126.8 (d), 127.0 (d), 127.4 (d), 127.6 (d), 128.0 (d), 138.4 (s), 138.8 (s). – HRMS Calcd. for $C_{18}H_{22}O + NH_4^+$: 270.1858. Found: 270.1835.

3-Benzoyloxy-3-(methylethyl)-2-phenyloxetane (**5**)

Procedure **B**
3 mmol of oxetanol **2** (576 mg) were dissolved in 20 ml of THF and the solution was cooled to 0 °C. A solution of *n*-BuLi (1.3 ml of a 2.37 M solution in *n*-hexane, 3 mmol) was added dropwise. Upon complete addition the mixture was stirred for 0.5 h at 0 °C and then 5.2 mmol benzoyl chloride (730 mg, 600 µl) were added by syringe. The mixture was slowly warmed to ambient temperature and stirred for 3 h. Upon dilution with ether the mixture was washed with saturated $NaHCO_3$ solution (aq) and with brine, dried with $MgSO_4$ and filtered. After removal of the solvents *in vacuo* the residue (1.2 g) was purified by flash chromatography (CH/EA= 97/3). Yield: 660 mg (74%). R_f = 0.27 (90/10). – IR (film): $\tilde{\nu}$ = 1705 cm^{-1} (vs, C=O), 975 (s, COC). – ^{1}H NMR (300 MHz): δ 1.04 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.37 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 2.73 [sept, 1 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}$(CH$_3$)$_2$], 5.03 (s, 2 H, C$\underline{H}_2$), 5.77 (s, 1 H, PhC$\underline{H}$), 7.20–7.50 [m, 10 H, C$\underline{H}$(arom.)]. – ^{13}C NMR (75.5 MHz): δ 16.1 (q), 16.9 (q), 31.0 (d), 77.0 (t), 84.4 (s), 86.2 (d), 126.0 (d), 127.4 (d), 127.9 (d), 127.9 (d), 129.2 (d), 129.7 (s), 132.7 (d), 137.7 (s), 164.3 (s). – Anal. Calcd. for $C_{19}H_{20}O_3$ (296.365): C 77.00 H 6.80. Found: C 77.13 H 6.92.

3-Acetoxy-3-(methylethyl)-2-phenyloxetane (**6**)

Procedure **C**.
3.2 mmol of oxetanol **2** (608 mg) were dissolved in 20 ml of CH_2Cl_2. To the stirred solution 8 mmol Ac_2O (820 mg, 755 µl), 100 mg DMAP and 11.2 mmol NEt_3 (1.13 g, 1.56 ml) were added successively at room temperature. The mixture was stirred for 2 d. Upon quenching with 2 ml of methanol the solvents and excess reagents were removed *in vacuo*. The residue was diluted with ether and washed successively with water, 1.5 M HCl (aq), saturated $NaHCO_3$ solution (aq) and with brine. Upon drying with $MgSO_4$ and filtration the solvents were removed *in vacuo* and the residue (0.9 g) was purified by flash chromatography (CH/EA = 95/5). Yield: 610 mg (80 %). R_f = 0.25 (90/10). – IR (film): $\tilde{\nu}$ = 1725 cm^{-1} (vs, C=O), 975 (s, COC). – ^{1}H NMR (300 MHz): δ 1.04 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.25 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.72 (s, 3 H, OOCC$\underline{H}_3$), 2.60 [sept, 1 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}$(CH$_3$)$_2$], 4.86 (d, 1 H, $^2J_{HH}$ = 8.1 Hz, C$\underline{H}$H), 4.92 (d, 1 H, $^2J_{HH}$ = 8.1 Hz, CH$\underline{H}$), 5.61 (s, 1 H, PhC$\underline{H}$), 7.20–7.40 [m, 5 H, C$\underline{H}$(arom.)]. – ^{13}C NMR (75.5 MHz): δ 16.3 (q), 16.9 (q), 20.8 (q), 31.1 (d), 76.9 (t), 84.3 (s), 86.6 (d), 126.4 (d), 127.6 (d), 127.9 (d), 137.7 (s), 169.3 (s). – Anal. Calcd. for $C_{14}H_{18}O_3$ (234.294): C 71.77 H 7.74. Found: C 71.57 H 7.71.

3-Methoxymethoxy-3-(methylethyl)-2-phenyloxetane (**7**)

According to the protocol described in procedure **B** (*vide infra*) 1.0 mmol of oxetanol **2** (192 mg) were transformed into the desired product employing the conditions indicated in table 1. Purification by flash chromatography (CH/EA= 98/2). Yield: 199 mg (84%). R_f = 0.30 (90/10). – IR (film): $\tilde{\nu}$ = 980 cm^{-1} (s, COC). – ^{1}H NMR (300 MHz): δ 1.03 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 1.16 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}_3$), 2.31 [sept, 1 H, $^3J_{HH}$ = 6.9 Hz, C$\underline{H}$(CH$_3$)$_2$], 3.09 (s, 3 H, OC$\underline{H}_3$), 4.18 (d, 1 H, $^2J_{HH}$ = 7.6 Hz, OC$\underline{H}$HO), 4.45 (d, 1 H, $^2J_{HH}$ = 7.6 Hz, OCH$\underline{H}$O), 4.62 (d, 1 H, $^2J_{HH}$ = 7.4 Hz, C$\underline{H}$H), 4.96 (d, 1 H, $^2J_{HH}$ = 7.4 Hz, CH$\underline{H}$), 5.58 (s, 1 H, PhC$\underline{H}$), 7.20–7.40 [m, 5 H, C$\underline{H}$(arom.)] – ^{13}C NMR (75.5 MHz): δ 16.1 (q), 16.2 (q), 35.0 (d), 55.4 (q), 76.5 (t), 83.2 (s), 89.6 (d), 92.4 (t), 125.8 (d), 127.2 (d), 128.0 (d), 138.6 (s). – HRMS Calcd. for $C_{14}H_{20}O_3 + NH_4^+$: 254.1756. Found: 254.1736.

3-Methoxyethoxymethoxy-3-(methylethyl)-2-phenyloxetane (**8**)

According to the protocol described in procedure **B** (*vide infra*) 1.0 mmol of oxetanol **2** (192 mg) were transformed into the desired product employing the conditions indicated in table

1. Purification by flash chromatography (CH/EA = 95/5). Yield: 233 mg (83%). R_f = 0.37 (70/30). – IR (film): $\tilde{\nu}$ = 980 cm^{-1} (vs, COC). – ^1H NMR (300 MHz): δ 1.02 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, CH_3), 1.16 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, CH_3), 2.31 [sept, 1 H, $^3J_{HH}$ = 6.9 Hz, $CH(CH_3)_2$], 3.25–3.48 (m, 4 H, OCH_2CH_2O), 3.31 (s, 3 H, OCH_3), 4.30 (d, 1 H, $^2J_{HH}$ = 7.6 Hz, OCHHO), 4.53 (d, 1 H, $^2J_{HH}$ = 7.6 Hz, OCHHO), 4.61 (d, 1 H, $^2J_{HH}$ = 7.4 Hz, CHH), 5.02 (d, 1 H, $^2J_{HH}$ = 7.4 Hz, CHH), 5.57 (s, 1 H, PhCH), 7.20–7.50 [m, 5 H, CH(arom.)]. – ^{13}C NMR (75.5 MHz): δ 16.0 (q), 16.1 (q), 34.9 (d), 58.8 (q), 66.9 (t), 71.5 (t), 76.4 (t), 83.1 (s), 89.5 (d), 91.3 (t), 125.7 (d), 127.1 (d), 127.9 (d), 138.5 (s). – HRMS Calcd. for $C_{16}H_{24}O_4 + NH_4^+$: 298.2018. Found: 298.2031.

3-t-Butyloxycarbonyloxy-3-(methylethyl)-2-phenyloxetane (**9**)

According to the protocol described in procedure **C** (*vide infra*) 3.0 mmol of oxetanol **2** (576 mg) were transformed into the desired product employing the conditions indicated in table 1. Purification by flash chromatography (CH/EA = 98/2). Yield: 870 mg (99%). *m.p.* 61–62 °C. – R_f = 0.50 (70/30). – IR (KBr): $\tilde{\nu}$ = 1720 cm^{-1} (vs, C=O), 980 (s, COC). – ^1H NMR (300 MHz): δ 1.15 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, $CHCH_3$), 1.17 (d, 3 H, $^3J_{HH}$ = 6.9 Hz, $CHCH_3$), 1.23 [s, 9 H, $C(CH_3)_3$], 2.71 [sept., 1 H, $^3J_{HH}$ = 6.9 Hz, $CH(CH_3)_2$], 4.79 (d, 1 H, $^2J_{HH}$ = 7.9 Hz, CHH), 4.90 (d, 1 H, $^2J_{HH}$ = 7.9 Hz, CHH), 5.63 (s, 1H, PhCH), 7.26–7.46 [m, 5 H, CH(arom.)]. – ^{13}C NMR (75.5 MHz): δ 16.7 (q), 16.8 (q), 27.5 (q), 30.5 (d), 76.2 (t), 82.0 (s), 85.0 (s), 87.2 (d), 127.3 (d), 127.9 (d), 128.0 (d), 137.6 (s), 151.1 (s). – Anal. Calcd. for $C_{17}H_{24}O_4$ (292.374): C 69.84 H 8.27. Found: C 69.66 H 8.42.

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