Facile Synthesis of α-Hydroxy Amides and Esters by Direct Autoxidation of Their Titanium Enolates

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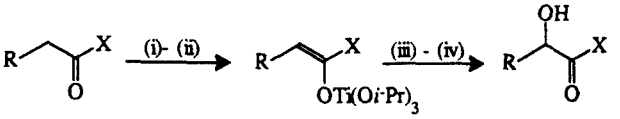
The autoxidation of titanium enolates **2**, derived from amides and esters **1**, afforded either directly the corresponding α-hydroxy carbonyl compounds **3** or, by variation of the reaction conditions, the γ-hydroxy β-keto ester **4** from ester **1g** in good yields (64–85%). Unfortunately, the chiral titanium enolate **2d** exhibited a low diastereoselectivity (d.r. 67:33) for this oxygen-transfer process.

The α-hydroxy carbonyl functionality plays an important role in organic chemistry because it is wide-spread in natural products and is frequently used as a building block in organic synthesis.[2] A most direct way to such α-hydroxy carbonyl compounds is the oxidation of their enolates, most recently by MoO_5–peroxide complexes,[3] oxaziridines,[4] or dimethyldioxirane.[5] In this context, among the first oxidative routes are the base-catalyzed autoxidations of enolates;[6] however, this approach is not frequently used, presumably because of the low yields and such oxidations are often complicated by α-carbon cleavage of the hydroperoxy intermediates.

During our investigations on the oxidation of titanium enolates by dimethyldioxirane,[7] we became interested in using such metal enolates also in autoxidation reactions. In fact, coordination of dioxygen at the metal was postulated in the autoxidation of titanium and zirconium alkyl complexes.[8] If chiral enolates are employed, such coordination could be very helpful for stereoselective oxygen-transfer reactions. Indeed, herein we demonstrate that titanium enolates react selectively with molecular oxygen to afford without further need of reduction directly the corresponding α-hydroxy carbonyl compounds.

The diisopropylamides **1a** (72%) and the hitherto unknown **1c** (64%) were prepared by reaction of the corresponding acyl chlorides with an excess of diisopropylamine. The structure of these new compounds was confirmed by the spectral data and by elemental analyses.

The titanium enolates were obtained by a transmetalation of the lithium analogs with the appropriate chlorotitanium reagents.[9] The lithium enolates were formed by deprotonation of **1a–g** with lithium diisopropylamide (LDA), in the case of the amides **1a–d** at 0°C, for the ester derivatives **1e–g** at −78°C (Eq. 1). These lithium enolates were allowed to react at −78°C with chlorotitanium triisopropoxide, either as a solution in pentane or in THF to form the corresponding titanium enolates **2** (Eq. 1). These orange colored reaction mixtures were cooled to the required temperatures, diluted by addition of dichloromethane, and dry oxygen gas was passed through the enolate solution for 30 minutes. During this time the color of the solution changed to pale yellow. After hydrolysis, the α-hydroxy amides and esters **3a–g** were obtained as the only products in good yields. The results are summarized in the Table, in which the yields and the spectral properties of the products **3a–g** are given. The structural assignment of the new derivatives **3a,c** was made by comparison of their spectral data with the literature-known derivative **3b** and by elemental analyses.



(1)

1 **2** **3**

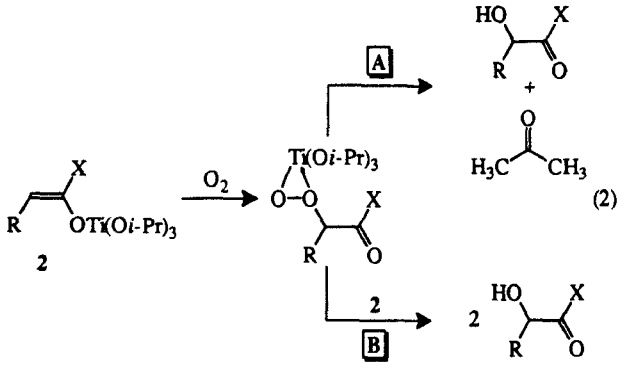
(i) : LDA, THF, 0 °C (**1a-d**) or -78 °C (**1e-g**) (for R and X cf. Table 1)

(ii) : $(i\text{-PrO})_3TiCl$, THF (or pentane), -78 to -30 °C, 3h

(iii) : O_2, -30 °C (**2a-d**) or -50 °C (**2e-g**), 30 min

(iv) : NH_4F, H_2O, 1 h

During the reaction or after workup, in no case could the corresponding α-hydroperoxy carbonyl compounds be detected either by thin layer chromatography (negative KI/AcOH peroxide test) or by spectroscopy. We suggest that either a Meerwein–Ponndorf reduction (**A**, Eq. 2) of the peroxytitanate intermediate to afford the α-hydroxylated products **3a–g** and acetone from the isopropoxy ligand or intermolecular oxygen transfer (**B**, Eq. 2) by the peroxytitanate intermediate to the yet not oxidized titanium enolate are responsible (Eq. 2).



(2)

The diastereoselectivity for this enolate oxidation was unfortunately low compared to other oxidants.[11] This is illustrated in the oxidation of the (*S*)-prolinol amide **1d**, for which the (2*S*):(2*R*) ratio was only 67:33, as determined by ^1H NMR analysis of the crude reaction mixture. Since the titanium moiety is fixed by intramolecular

chelation on the *re* side of the enolate, an attack of dioxygen by coordination to the metal center should lead to the (2*R*)-**3d** (Figure 1). However, attack of dioxygen occurs mainly from the less hindered *si* side of the enolate moiety (Figure 1) to form (2*S*)-**3d**. Thus, coordination of the dioxygen to the titanium metal is insignificant for the formation of the main diastereomer.

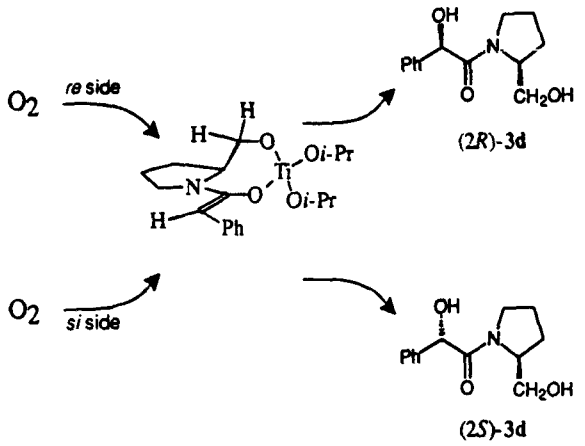
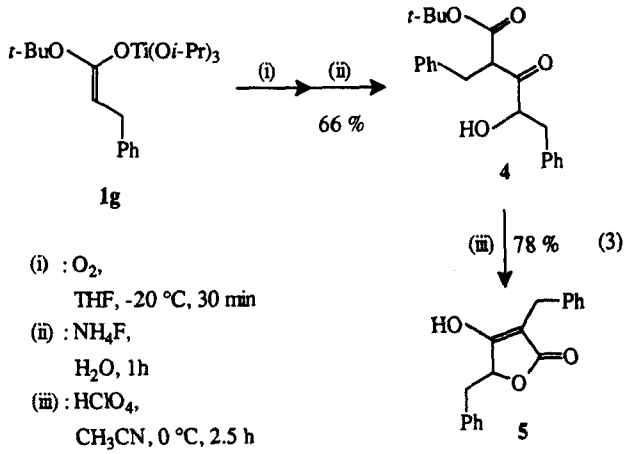


Figure 1. Dioxygen *re and si* side attack in the oxidation of the titanium enolate **2d**

If the autoxidation of the titanium enolate **2g** is performed at higher concentrations and at higher temperatures (−20 to 0 °C), the diastereomeric γ-hydroxy β-keto ester **4** is produced in 66% yield (d.r. = 50:50), besides traces of **3g** (5%) (Eq. 3). The product **4** resulted from aldol reaction of the oxidized with the nonoxidized titanium enolate **2g**. Under the same reaction conditions, the amide-derived titanium enolates **2a–d** did not result in the aldol product **4**. Since γ-hydroxy β-keto esters serve as convenient precursors for tetronic acids,[15] the alcohol product **4** could readily be transformed by acid-catalyzed lactonization to the derivative **5** in 78% yield (Eq. 3).



In summary, we have demonstrated that the autoxidation of titanium enolates **2** derived from amides and esters **1** afford in good yields selectively the corresponding α-hydroxy carbonyl compounds **3**. By variation of the reaction conditions, the ester titanium enolate **2g** resulted in the γ-hydroxy β-keto ester **4** by aldol reaction, which was cyclized to the tetronic acid derivative **5** by acid catalysis.

Melting points: Büchi 535. IR: Perkin-Elmer 1420. ^1H and ^{13}C NMR: Bruker AC 200 (200 MHz) or WM 250 (250 MHz); chemical shifts refer to TMS. All solvents were purified by standard literature methods; THF was distilled under an Ar gas atmosphere from K/benzophenone and CH_2Cl_2 was distilled from CaH_2. LDA was prepared and standardized according to the literature procedure[16] and used as a stock solution in THF. The amide **1d**[11] and $(i\text{-PrO})_3TiCl$[17] were prepared according to literature procedures. All glassware employed in the preparation of the organometallic compounds was vacuum-dried (heatgun/0.1 Torr) and all reactions of the enolate complexes were run under an Ar gas atmosphere. For the new compounds **1c**, **4** and **5** satisfactory microanalyses were obtained: C ± 0.44, H ± 0.25.

***N,N*-Bis(1-methylethyl)hexanamide (1a):**[18]
To a stirred solution of hexanoyl chloride (10.7 g, 79.5 mmol) in *t*-BuOMe (80 mL) was added at r. t. a solution of $i\text{-Pr}_2NH$ (17.7 g, 175 mmol) in *t*-BuOMe (50 mL). The mixture was allowed to stir for 12 h and heated under reflux for 1 h. After cooling to r.t., the precipitated $i\text{-Pr}_2NH \cdot HCl$ was removed by filtration, the filtrate was washed with sat. aq $NaHCO_3$ solution (2 × 50 mL) and brine (50 mL), and the organic phase was dried ($MgSO_4$). After removal of the drying agent, the solvent was evaporated (50 °C/400 Torr) and the residue was purified by distillation to give **1a** as a colorless oil; bp 60–64 °C/0.1 Torr; yield: 11.4 g (72%).

IR (neat): ν = 1670 (C=O) cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 0.91 (t, J = 6.8 Hz, 3H), 1.17 (d, J = 6.7 Hz, 6H), 1.26–1.35 (m, 4H), 1.38 (d, J = 6.8 Hz, 6H), 1.62 (quint, J = 7.5 Hz, 2H), 2.27 (t, J = 7.7 Hz, 2H), 3.50 (m, 1H), 3.97 (quint, J = 6.7 Hz, 1H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 14.2 (q), 20.9 (q), 21.2 (q), 22.8 (t), 25.4 (t), 31.9 (t), 35.6 (t), 45.7 (d), 48.4 (d), 172.3 (s).

N,N-Bis(1-methylethyl)-3-phenylpropanamide **(1c):** By following the above procedure, **1c** was obtained as a colorless oil starting from 3-phenylpropanoic acid chloride (4.43 g, 26.3 mmol) and diisopropylamine; bp 170 °C/0.8 Torr; yield: 3.92 g (64%).

IR (neat): ν = 1650 (C=O) cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 1.12 (d, J = 6.7 Hz, 6H), 1.39 (d, J = 6.8 Hz, 6H), 2.59 (m, 2H), 2.96 (m, 2H), 3.45 (m, 1H), 3.90 (quint, J = 6.7 Hz, 1H), 7.10–7.40 (m, 5H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 20.6 (q), 20.9 (q), 31.6 (t), 37.1 (t), 45.6 (d), 48.2 (d), 125.9 (d), 128.4 (d), 128.5 (d), 141.7 (s), 170.9 (s).

Autoxidation of the Titanium Enolates 1a–g; General Procedure:
To a cold 0.50–0.75 M solution of LDA in THF (in the case of the amides **1a–d** 1.50 equiv of LDA cooled to 0 °C, for the esters **1e–g** 1.10 equiv of LDA cooled to −78 °C was employed) was added dropwise under an Ar gas atmosphere a solution of the carbonyl compound **1a–g** (1 equiv) in THF (2 mL per mmol). After stirring for 1 h at the temperature given above, the mixture was treated with $(i\text{-PrO})_3TiCl$ (1.1 equiv, either as a 1.05 M solution in pentane or as a 0.55 M solution in THF) at −78 °C. The stirring was continued for 3 h during which time the temperature was allowed to reach −30 °C. The solution was cooled to the required oxidation temperature (in the case of the enolates **2a–d** to −30 °C, for the enolates **2e–g** to −50 °C) and diluted with CH_2Cl_2 (20 mL per mmol). A gentle stream of dry O_2 was bubbled through the mixture. After 30 min, the cold mixture was poured into aq sat. NH_4F solution (50 mL per mmol) and stirred for 1 h at r.t. The organic layer was separated and the aqueous phase extracted twice with *t*-BuOMe (20 mL per mmol). The combined organic layers were washed with brine and dried ($MgSO_4$). After removal of the drying agent, the solvent was evaporated (40 °C/400 Torr) and the residue purified by column chromatography [**3a–c**: silica gel, 3:1 petroleum ether (bp 50–60 °C)/*t*-BuOMe; **3d**: silica gel, 10:1 CH_2Cl_2/MeOH; **3e–g**; silica gel, 6:1 petroleum ether (bp 50–60 °C)/*t*-BuOMe]. The yields of the α-hydroxy carbonyl products **3a–g** and their spectral data are summarized in the Table.

Table. Autoxidation of the Titanium Enolates **2** to the α-Hydroxy Carbonyl Compounds **3**

Product	R	X	Yield[a] (%)	mp[b] (°C)	Lit. mp (°C)	IR (cm^{-1}) $\nu_{C=O}$	^1H NMR (200 or 250 MHz, $CDCl_3$/TMS) δ, J (Hz)	^{13}C NMR (50 or 63 MHz, $CDCl_3$/TMS) δ
3a	Bu	NPr_2-*i*	85	oil	–	1640 (neat)	0.89 (t, *J* = 6.9, 3H), 1.19 (d, *J* = 6.7, 3H), 1.21 (d, *J* = 6.7, 3H), 1.38 (d, *J* = 6.9, 3H), 1.43 (d, *J* = 6.8, 3H), 1.00–1.60 (m, 6H), 3.43 (sept, *J* = 6.9, 1H), 3.83 (sept, *J* = 6.6, 1H), 4.05 (d, *J* = 7.2, 1H, O*H*), 4.25 (ddd, *J* = 6.7, 6.6, 2.4, 1H)	13.9 (q), 20.2 (q), 20.6 (2q), 20.9 (q), 22.5 (t), 26.8 (t), 34.8 (t), 46.2 (d), 47.7 (d), 68.2 (d), 172.9 (s)
3b	Ph	NPr_2-*i*	79	92–93	95[10]	1615 (KBr)	0.44 (d, *J* = 6.6, 3H), 1.13 (d, *J* = 6.7, 3H), 1.39 (d, *J* = 6.8, 3H), 1.46 (d, *J* = 6.8, 3H), 3.34 (sept, *J* = 6.8, 1H), 3.79 (sept, *J* = 6.6, 1H), 5.11 (m, 2H), 7.21–7.41 (m, 5H)	18.5 (q), 19.5 (q), 20.4 (2q), 46.3 (d), 47.9 (d), 71.8 (d), 127.4 (d), 128.2 (d), 128.9 (d), *140.1* (s), 170.5 (s)
3c	$PhCH_2$	NPr_2-*i*	74	70–71	–	1685 (CCl_4)	1.15 (d, *J* = 6.6, 3H), 1.20 (d, *J* = 6.6, 3H), 1.38 (d, *J* = 6.8, 3H), 1.43 (d, *J* = 6.8, 3H), 2.79 (dd, *J* = 13.9, *J* = 7.2, 1H), 2.92 (dd, *J* = 13.9, 4.3, 1H), 3.45 (sept, *J* = 6.8, 1H), 3.90 (q, *J* = 6.7, 1H), 4.10 (d, *J* = 7.9, 1H, O*H*), 4.55 (ddd, *J* = 7.9, 7.2, 4.4, 1H), 7.15–7.34 (m, 5H)	19.9 (q), 20.1 (q), 20.5 (q), 21.0 (q), 41.9 (t), 46.3 (d), 48.0 (d), 69.1 (d), 126.6 (d), 128.3 (d), 129.4 (d), 137.1 (s), 172.2 (s)
3d	Ph	(*S*)-prolinol	69[c]	oil	oil[11]	1615 (CCl_4)	1.01–2.08 (m, 4H), 2.75–3.78 (m, 4H), 4.06–4.18 (m, 1H), 4.58 (s, 1H, O*H*), 5.09, 5.11 (s, 1H), 7.25–7.50 (m, 5H)	(2*S*)-**3d**: 24.3 (t), 27.6 (t), 47.2 (t), 62.0 (d), 66.0 (t), 72.7 (d), 127.6 (d), 128.6 (d), 129.0 (d), 138.6 (s), 172.9 (s) (2*R*)-**3d**: 24.0 (t), 27.8 (t), 46.9 (t), 62.0 (d), *66.0* (t), 72.9 (d), 127.5 (d), 128.7 (d), 129.0 (d), 138.2 (s), 172.6 (s)
3e	Bu	OBu-*t*	67	oil	oil[12]	1710 (CCl_4)	0.90 (m, 3H), 1.15–1.95 (m, 6H), 1.48 (s, 9H), 2.81 (d, *J* = 5.7, 1H, O*H*), 4.04 (ddd, *J* = 6.5, 5.7, 4.3, 1H)	13.9 (q), 22.4 (t), 26.7 (t), 28.0 (q), 34.1 (t), 70.5 (d), 82.2 (s), 174.7 (s)
3f	Ph	OBu-*t*	78	65–66	66–67[13]	1705 (CCl_4)	1.40 (s, 9H), 3.63 (d, *J* = 6.0, 1H, O*H*), 5.85 (d, *J* = 5.9, 1H), 7.25–7.51 (m, 5H)	27.7 (q), 72.9 (d), 90.0 (s), 126.3 (d), 128.0 (d), 128.3 (d), *138.9* (s), 172.8 (s)
3g	$PhCH_2$	OBu-*t*	64	oil	oil[14]	1710 (CCl_4)	1.42 (s, 9H), 2.86 (d, *J* = 5.9, 1H, O*H*), 2.92 (dd, *J* = 13.9, 6.5, 1H), 3.08 (dd, *J* = 13.9, 4.8, 1H), 4.31 (ddd, *J* = 6.4, 6.0, 4.8, 1H), 7.20–7.35 (m, 5H)	27.9 (q), 40.5 (t), 71.2 (d), 82.7 (s), 126.7 (d), 128.2 (d), 129.6 (d), 136.6 (s), 173.4 (s)

[a] Yield of isolated material after chromatography on silica gel.
[b] For new compounds satisfactory microanalyses obtained: C ± 0.35, H ± 0.16, N ± 0.26.
[c] Ratio of (2*S*) : (2*R*) = 67 : 33, as determined by ^1H NMR analyses of the crude reaction mixture (error ± 5%).

***tert*-Butyl 4-Hydroxy-3-oxo-5-phenyl-2-phenylmethylpentanoate (4):**
To a cold (−78 °C) 0.55 M solution of LDA in THF (10 mL, 5.50 mmol) was added dropwise under an Ar gas atmosphere a solution of **1g** (1.03 g, 5.00 mmol) in THF (10 mL). After stirring for 1 h at this temperature, a solution of $(i\text{-PrO})_3TiCl$ (1.45 g, 5.57 mmol) in THF (10 mL) was added. The stirring was continued for 3 h, during which time the mixture was allowed to reach −20 °C. Dried O_2 was passed through this mixture. After 30 min, the cold mixture was poured into aq sat. NH_4F solution (250 mL) and stirred for 1 h at r.t. The organic layer was separated and the aqueous phase extracted with *t*-BuOMe (3 × 100 mL). The combined organic layers were washed with brine (50 mL) and dried ($MgSO_4$). After removal of the drying agent, the solvent was evaporated (50 °C/400 Torr) and the residue purified by column chromatography [silica gel, 6:1 petroleum ether (bp 50–60 °C/*t*-BuOMe] to afford **4** as a colorless oil; yield: 585 mg (63%). Spectral data of the 1:1 mixture of the (*R**,*R**)-**4** and (*S**,*R**)-**4** diastereomers:

IR (CCl_4): ν = 1735 (C=O), 1705 cm^{-1} (C=O).

^1H NMR (200 MHz, $CDCl_3$): δ = 1.39, 1.40 (s, 9H), 2.53–2.66 (m, 1H), 2.89–3.17 (m, 4H), 3.96–4.19, 4.49 (m, 2H), 7.08–7.38 (m, 10H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 27.8, 27.8 (q), 33.4, 34.5 (t), 39.2, 39.5 (t), 56.7, 57.6 (d), 77.3, 78.2 (d), 82.6, 82.8 (s), 126.7 (d), 126.7 (d), 126.9 (d), 128.5 (d), 128.5 (d), 128.6 (d), 128.9 (d), 129.1 (d), 129.2 (d), 129.3 (d), 138.0, 138.1 (s), 167.4, 167.8 (s), 206.3, 207.3 (s).

3,5-Dibenzyltetrahydrofuran-2,4-dione (5):
A solution of **4** (300 mg, 0.850 mmol) in MeCN (2 mL) was cooled to 0 °C and treated with an aq 60% $HClO_4$ (0.5 mL) solution. After stirring for 2.5 h, sat. aq $NaHCO_3$ solution (pH 9) was added and the mixture was washed with *t*-BuOMe (5 mL). The aqueous phase was acidified with 12 N HCl (pH 2) and extracted with *t*-BuOMe (3 × 15 mL). The combined organic layers were washed with brine (10 mL) and dried ($MgSO_4$). After removal of the solvent (50 °C/400 Torr), a pale yellow, partially crystalline oil was obtained, which was recrystallized by dissolving in EtOAc (2 mL) and precipitation

with pentane (15 mL) to give **5** as a colorless amorphous solid; mp 159–162 °C; yield: 185 mg (78 %).

IR (KBr): ν = 1695 (C=O), 1640 cm^{-1} (br, C=O).

^1H NMR (200 MHz, CD_3CN): δ = 2.98 (dd, J = 14.5 Hz, 5.5 Hz, 1 H), 3.31 (dd, J = 14.5 Hz, 4.0 Hz, 1 H), AB system: δ_A = 3.30, δ_B = 3.43, ($J_{A,B}$ = 16.0 Hz, 2 H), 5.03 (dd, J = 5.5 Hz, 4.0 Hz, 1 H), 6.78 (d, 2 H), 7.13–7.31 (m, 8 H), 8.90 (s, 1 H, OH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 27.6 (t), 37.7 (t), 78.3 (d), 102.3 (s), 126.9 (d), 127.9 (d), 128.8 (d), 129.2 (d), 129.2 (d), 130.8 (d), 136.1 (2), 139.6 (s), 174.3 (s), 174.6 (s).

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(1) Undergraduate research participants, Renz, M., Fall 1991; Metz, M., Spring 1992.
(2) Hanessian, S. *Total Synthesis of Natural Products: The Chiron Approach*, Pergamon: New York, 1983, Chap. 2.
Mori, K. In *The Total Synthesis of Natural Products*; Apsimon, J., Ed.; Wiley Interscience: New York, 1981, Chap. 1.
Seebach, D.; Hungerbuhler, E. In *Modern Synthetic Methods*; Sheffold, R., Ed.; Otto Salle Verlag: Frankfurt, 1980.
(3) Vedejs, E.; Engler, D.A.; Telschow, J.E. *J. Org. Chem.* **1978**, *43*, 188.
Anderson, J.C.; Smith, S.C. *Synlett* **1990**, 107.
(4) Davis, F.A.; Haque, M.S.; Ulatowski, T.G.; Towson, J.C. *J. Org. Chem.* **1986**, *51*, 2402.
Davis, F.A.; Sheppard, A.C. *Tetrahedron* **1989**, *45*, 5703.
Davis, F.A.; Sheppard, A.C.; Chen, B.-C.; Haque, M.S. *J. Am. Chem. Soc.* **1990**, *112*, 6679.
Davis, F.A.; Weismiller, M.C. *J. Org. Chem.* **1990**, *55*, 3715.
Davis, F.A.; Chen, B.-C. *Chem. Rev.* **1992**, *92*, 919.
(5) Guertin, K.R.; Chan, T.-H. *Tetrahedron Lett.* **1991**, *32*, 715.
Adam, W.; Prechtl, F. *Chem. Ber.* **1991**, *124*, 2369.
(6) v.E. Doering, W.; Haines, R.M. *J. Am. Chem. Soc.* **1954**, *76*, 482.
Bailey, E.J.; Barton, D.H.R.; Elks, J.; Templeton, J.F. *J. Chem. Soc.* **1962**, 1578.
Konen, D.A.; Silbert, L.S.; Pfeffer, P.E. *J. Org. Chem.* **1975**, *40*, 3253.
Wassermann, H.H.; Lipshutz, B.H. *Tetrahedron Lett.* **1975**, 1731.
Adam, W.; Cueto, O.; Ehrig, V. *J. Org. Chem.* **1976**, *41*, 370.
Adam, W.; Cueto, O. *J. Org. Chem.* **1977**, *42*, 38.
(7) Adam, W.; Müller, M.; Prechtl, F. *J. Org. Chem.*, in press.
Adam, W.; Prechtl, F. *Chem. Ber.*, in press.
(8) Lubben, T.V.; Wolczanski, P.T. *J. Am. Chem. Soc.* **1987**, *109*, 424.
(9) Reetz, M.T.; Peter, R. *Tetrahedron Lett.* **1981**, *22*, 4691.
Reetz, M.T. *Organotitanium Reagents in Organic Synthesis*; Springer: Berlin, 1986.
(10) Fletcher, A.S.; Smith, K.; Swaminathan, K. *J. Chem. Soc., Perkin Trans. 1* **1977**, 1881.
(11) Davis, F.A.; Vishwakarma, L.C. *Tetrahedron Lett.* **1985**, *26*, 3539.
(12) Plaquevent, J.C.; Danvy, D.; Monteil, T.; Greciet, H.; Duhamel, L.; Duhamel, P.; Gross, G.; Schwartz, J.C.; Lecomte, J.M. Eur. Pat. Appl. EP 419327 (1991); *Chem. Abstr.* **1991**, *115*, 136779.
(13) Brown, H.C.; Pai, G.G. *J. Org. Chem.* **1985**, *50*, 1384.
(14) Kunz, H.; Lerchen, H.-G. *Tetrahedron Lett.* **1987**, *28*, 1873.
Terashima, S.; Tseng, C.C.; Koga, K. *Chem. Pharm. Bull.* **1979**, *27*, 747.
(15) Damon, R.E.; Luo, T.; Schlessinger, R.H. *Tetrahedron Lett.* **1976**, 2749.
(16) Vedejs, E.; Larsen, S. *Org. Synth.* **1986**, *64*, 127.
(17) Reetz, M.T.; Steinbach, R.; Keßeler, K. *Angew. Chem. Suppl* **1982**, 1899.
(18) Cooke, M.P. Jr.; Houpis, J.N. *Tetrahedron Lett.* **1985**, *26*, 4987