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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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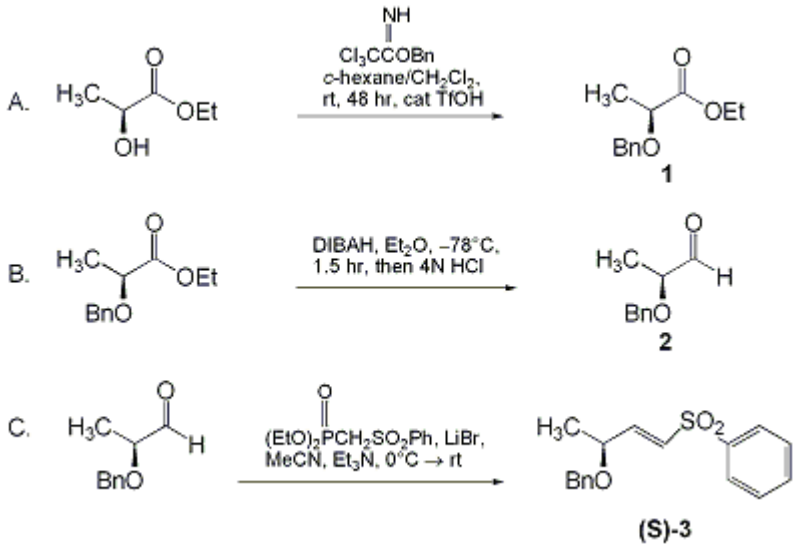
SYNTHESIS OF (−)-(E,S)-3-(BENZYLOXY)-1-BUTENYL PHENYL SULFONE VIA A HORNER-WADSWORTH-EMMONS REACTION OF (−)-(S)-2-(BENZYLOXY)PROPANAL

[Benzene, [[[1-methyl-3-(phenylsulfonyl)-2-propenyl]oxy]methyl]-, [S-(E)]-) from (Propanal, 2-(phenylmethoxy)-, (S)-)]



Submitted by D. Enders[1] , S. von Berg[1] , and B. Jandeleit[2] .
Checked by Brad M. Savall and William R. Roush.

1. Procedure

A. (−)-(S)-Ethyl 2-(benzyloxy)propanoate. As described in ref. 3, a flame-dried, 500-mL Schlenk flask equipped with a magnetic stirring bar, rubber septum, and an argon balloon is charged with 11.8 g (100 mmol) of (S)-ethyl 2-hydroxypropanoate [(S)-ethyl lactate] (Note 1) and 50.9 g (200 mmol) of O-benzyl-2,2,2-trichloroacetimidate (Note 2). The reagents are dissolved in 250 mL of a mixture of anhydrous cyclohexane (Note 3) and anhydrous dichloromethane (Note 4) (7:1 v/v) under an atmosphere of argon. Neat trifluoromethanesulfonic acid (Note 5) (0.4 mL, 4.53 mmol) is added dropwise by means of a syringe while the mixture is stirred rapidly (Note 6). The reaction mixture is stirred for 48 to 60 hr at room temperature (Note 7) and subsequently diluted with water (100 mL) and hexane (300 mL). Stirring is continued for an additional 3 hr at room temperature. The precipitated colorless trichloroacetamide is filtered off by means of a Büchner funnel. The aqueous phase is separated and extracted three times with 50-mL portions of hexane . The combined organic extracts are washed with 50 mL of aqueous saturated sodium bicarbonate ($NaHCO_3$) solution and finally with 50 mL of aqueous saturated sodium chloride (NaCl) solution. After drying over magnesium sulfate ($MgSO_4$), filtration and removal of the solvents under reduced pressure by means of a rotary evaporator, the residue is purified by fractional distillation using a Vigreux column (15-20 cm) to yield 18.7 g (90%) of a colorless slightly turbid liquid (Notes 8, 9).

B. (−)-(S)-2-(Benzyloxy)propanal . A flame-dried, 500-mL Schlenk flask equipped with a magnetic stirring bar, dropping funnel sealed with a rubber septum, and an argon balloon is loaded under an atmosphere of argon with 18.7 g (90 mmol) of (−)-(S)-ethyl 2-(benzyloxy)propanoate and the compound is dissolved in anhydrous diethyl ether (180 mL) (Note 10). The reaction mixture is cooled to −78°C by means of a cooling bath (dry ice/ethanol). A 1 M solution of diisobutylaluminum hydride (DIBAH) in hexane (126 mL, 126 mmol) (Note 11) is added very slowly dropwise to the solution of the ester and stirring is continued for at least 1 hr after the complete addition of the DIBAH solution (Note

12). Upon complete consumption of the ester, the crude reaction mixture is poured directly with vigorous stirring into 360 mL of ice cold 4 N hydrochloric acid (Note 13). The aqueous phase is extracted with diethyl ether (4 × 180 mL) and the combined organic extracts are washed with 50 mL of aqueous saturated NaCl solution. After drying over $MgSO_4$, filtration and removal of the solvents under reduced pressure by means of a rotary evaporator, 14.4 g (98%) of the crude aldehyde is obtained (Note 14).

C. (−)-(E,S)-3-(Benzyloxy)-1-butenyl phenyl sulfone . As described in ref. 4, a flame-dried, 500-mL Schlenk flask (or three-necked flask with a thermometer) equipped with a large magnetic stirring bar (Note 15), dropping funnel sealed with a rubber septum, and an argon balloon is charged under an atmosphere of argon with 13.1 g (151 mmol) of lithium bromide (Note 16) and 36.6 g (125 mmol) of diethyl [(phenylsulfonyl)methyl]phosphonate (Note 17). The reagents are suspended in 250 mL of anhydrous acetonitrile (Note 18) and 19.1 mL (13.9 g, 138 mmol) of triethylamine (Note 19) is then added. The reaction mixture is stirred at room temperature until it becomes homogeneous (Note 20) and is then cooled to 0°C (an ice-salt bath is used to maintain the internal temperature at 0°C). The dropping funnel is charged with a solution of 20.6 g (125 mmol) of (−)-(S)-2-(benzyloxy)propanal in 50 mL of anhydrous acetonitrile . The aldehyde solution is added dropwise at 0°C with vigorous stirring. After complete addition the reaction mixture is stirred for ca. 12 hr and allowed to warm to room temperature during this period. The reaction is monitored by TLC and is halted by the addition of 0.1 N hydrochloric acid (150 mL) and water (150 mL). The reaction mixture is diluted with diethyl ether (200 mL). After phase separation, the aqueous phase is reextracted with diethyl ether (4 × 200 mL) and the combined organic extracts are washed with aqueous saturated NaCl solution (200 mL). After drying over $MgSO_4$, filtration, and removal of the solvents under reduced pressure using a rotary evaporator, the crude product is purified by column chromatography using a 15 : 1 (w/w) ratio of silica gel to crude product and 1:2 → 1:1 (v/v) diethyl ether/light petroleum as eluent to yield 34.2 g (90%) of the pure (E)-isomer as a colorless viscous oil (Notes 21 and 22).

2. Notes

1. (S)-Ethyl lactate was purchased from Merck, Darmstadt, Germany, in enantiomerically pure form (ee >> 99%) and was used without further purification.
2. O-Benzyl 2,2,2-trichloroacetimidate was synthesized in 98% yield according to ref. 5 by addition of 1.0 equiv of benzyl alcohol (Aldrich Chemical Company, Inc.; previously distilled over calcium hydride (CaH_2) under an atmosphere of argon), to 1.0 equiv of trichloroacetonitrile (Aldrich Chemical Company, Inc.; no previous purification) in the presence of 0.1 equiv of sodium hydride in anhydrous diethyl ether .The resulting viscous, dark brown crude compound showed sufficient purity (> 97%) by ^1H NMR and GLC analysis and was used without further purification. The benzylating agent can be stored at 4°C under an atmosphere of argon for several weeks without decomposition or loss of quality. However, the compound can be further purified by distillation to yield a colorless viscous liquid, or, alternatively, can be purchased from Aldrich Chemical Company, Inc.
3. Cyclohexane was purified by distillation from CaH_2 under argon.
4. Dichloromethane was purified by distillation from CaH_2 under argon.
5. Trifluoromethanesulfonic acid was purchased from Aldrich Chemical Company, Inc. , stored and handled under an atmosphere of argon, and used without further purification.
6. During the addition of the catalyst an almost colorless precipitate of trichloroacetamide forms, which might dissolve again after a few minutes. If this happens or if the reaction mixture does not become turbid at all, more (0.4 mL) trifluoromethanesulfonic acid can be added.
7. The submitters indicated that extended reaction times are essential to obtain complete consumption of the starting material and good yields. However, the checkers observed that the reaction appeared to be complete within several hours according to TLC analysis.
8. The checkers observed that a crystalline solid (trichloroacetamide) formed in the condenser at the beginning of the distillation, and that the distillate contained small amounts of a precipitated crystalline solid (trichloroacetamide) that could be removed by filtration through a sintered glass funnel. The checkers obtained 78-83% yields of product, with material collected from 125 - 135° to maximize the yield of product.
9. The compound shows the following physical data: R_f = 0.40 (0.25 mm silica gel on glass, diethyl ether/light petroleum = 1:5); bp: 124-127°C/6 mm, α_D^{21} : −73.6° (neat), $\{[\alpha]_D^{20}$: −74.5 ($CHCl_3$, *c*

2.94)};[3] ^{31}H NMR (300 MHz, $CDCl_3$) δ: 1.30 (t, 3 H, J = 7.2), 1.44 (d, 3 H, J = 6.9), 4.05 (q, 1 H, J = 6.8), 4.22 (m, 2 H), 4.45 (d, 1 H, J = 11.8), 4.69 (d, 1 H, J = 11.8), 7.20-7.40 (m, 5 H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.2, 18.7, 60.8, 71.9, 74.0, 127.8, 127.9, 128.4, 136.6, 173.2 ; IR (film) cm^{-1}: 2985, 1746, 1455, 1200, 1143 ; HRMS calcd for $C_{12}H_{17}O_3$ [M+H] 209.1178, found 209.1183 . Anal. Calcd for $C_{12}H_{16}O_3$: C, 69.21; H, 7.73, Found: C, 69.17; H, 7.74. The O-Bn protected (S)-ethyl lactate can be stored without special precautions.

10. Diethyl ether was purified by distillation from sodium benzophenone ketyl under argon.

11. Diisobutylaluminum hydride (DIBAH) (1 M in hexane) was purchased from Aldrich Chemical Company, Inc. , and used without further purification. DIBAH should be handled with caution and all operations should be performed by employing the usual inert gas techniques (cannula, syringe, etc.).

12. To avoid reduction of the generated aldehyde that is more prone to reduction than the corresponding ester, the reducing agent should be added very slowly, avoiding any local temperature increase. The reaction mixture should be kept below −70°C. The checkers used a three-necked flask equipped with a low temperature thermometer to monitor the internal reaction temperature.

13. It is strongly recommended that the reaction mixture be added to the beaker containing the ice-cold acid solution with vigorous stirring because of the strong evolution of hydrogen gas. Reversing the order of addition causes freezing of the acid and effects a vigorous evolution of hydrogen gas on warming.

14. The resulting viscous colorless crude compound obtained by the submitters showed sufficient purity (> 97%) by ^{1}H NMR and GLC analysis to be used without further purification. The submitters report that the aldehyde can be stored at −20°C under an atmosphere of argon for several days without detectable racemization or loss of quality. However, the checkers observed that some crystalline material resembling aluminum salts formed when the crude aldehyde was stored in the refrigerator. The salts could be removed by dissolving the product in diethyl ether and washing with 1 N HCl (2 × 50 mL). The purity of the aldehyde obtained by the checkers was 80% by GC analysis. Nevertheless, this material gave acceptable results in the following Horner-Wadsworth-Emmons reaction. If desired, the aldehyde can be purified by distillation to yield a colorless viscous liquid.[3] The compound shows the following physical data: R_f = 0.27 (0.25 mm silica gel on glass, diethyl ether/light petroleum = 1:3); α: −65.3° (neat) {α: −65.9° (neat)};[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.31 (d, 3 H, J = 7.2), 3.88 (qd, 1 H, J = 6.9, 1.9), 4.57 (d, 1 H, J = 11.8), 4.67 (d, 1 H, J = 11.8), 7.35 (m, 5 H), 9.64 (d, 1 H, J = 1.6) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 15.5, 72.0, 79.4, 127.9, 128.6, 137.3, 203.4 ; IR (film) cm^{-1}: 3448, 2870, 1733, 1455, 1375, 1094 ; HRMS calcd for $C_{10}H_{16}NO_2$ [M+NH_4^+] 182.1181, found 182.1176 . Anal. Calcd for $C_{10}H_{12}O_2$: C, 73.14; H, 7.37. Found: C, 68.98; H, 7.52.

15. Because of precipitating lithium phosphates during the course of the reaction the mixture forms a sticky slurry and so the use of a large stirring bar is recommended.

16. Lithium bromide was purchased from Fluka Chemical Corp. and dried for ca. 12 hr at 100-110°C under reduced pressure using a high vacuum pump. The salt was handled and stored under an atmosphere of argon with the exclusion of moisture.

17. Diethyl [(phenylsulfonyl)methyl]phosphonate was synthesized according to the accompanying procedure: *Org. Synth.* **2002**, *78*, 169.

18. Acetonitrile was purified by distillation from CaH_2 under argon.

19. Triethylamine was purified by distillation from CaH_2 under argon.

20. To obtain good chemical yields and a very high (E)-selectivity in the olefination step it seems to be crucial that all of the phosphonate must be transformed into its chelated lithium derivative before the aldehyde is added. Sometimes the reaction mixture becomes turbid during cooling but this does not affect reactivity.

21. During the olefination step only very small amounts of the corresponding (Z)-isomer are formed, which are easily removed by column chromatography on silica gel. The (Z)-isomer has a slightly higher R_f-value than the (E)-isomer: R_f(Z) = 0.49 (0.25 mm silica gel on glass, diethyl ether/light petroleum (v/v) 1:1).

22. The compound shows the following physical data: R_f = 0.44 (0.25 mm silica gel on glass, dimethyl ether/light petroleum (v/v) 1:1); [α[: −31.9° ($CHCl_3$, *c* 1.10); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.31 (d, 3 H, J = 6.6), 4.18 (qdd, 1 H, J = 6.6, 4.8, 1.6), 4.44 (d, 1 H, J = 12.0), 4.48 (d, 1 H, J = 12.0), 6.57 (dd, 1 H, J = 15, 1.5), 6.94 (dd, 1 H, J = 15.3, 4.9), 7.21-7.34 (m, 5 H), 7.48-7.55 (m, 2 H), 7.57-7.64 (m, 1 H), 7.85-7.90 (m, 2 H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.0, 71.0, 72.9, 127.5, 127.6, 127.8, 128.4, 129.3, 130.3, 133.4, 137.6, 140.3, 147.0 ; IR (film) cm^{-1}: 3063, 1447, 1307, 1147, 1086, 834 ; HRMS calcd for

$C_{17}H_{22}O_3NS$ [M+NH_4^+] 320.1320, found 320.1310 . Anal. Calcd for $C_{17}H_{18}O_3S$: C, 67.43; H, 6.00. Found: C, 66.45; H, 6.09. The checkers found that the product solidified when stored at −20°C, cracking the glass bottle.

Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

(−)-(S)-2-(Benzyloxy)propanal (**2**) has attracted considerable interest since it is readily available in optically pure form from inexpensive starting materials. This useful aldehyde is widely used in organic synthesis[6] and has therefore been synthesized many times employing several approaches.[3, 7 8 9 10 11 12]

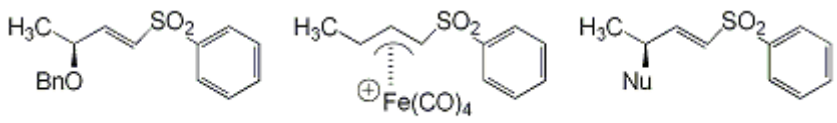
The most frequently used procedures to obtain aldehyde **2** employ commercially available ethyl (S)-lactate . The α-hydroxy ester is converted in the first step to ethyl (S)-2-(benzyloxy)propionate (**1**) by O-benzylation. Alkylation of the ester can be accomplished by using freshly prepared silver(I) oxide and benzyl bromide according to a procedure described by Mislow.[13] This method is high yielding, but it is not amenable to large scale preparation of **1**. Benzylation using benzyl bromide and sodium hydride is also described in the literature. It is reported that this approach affords the product in low yield[14] and that the alkylation results in considerable racemization.[7] However, Varelis and Johnson[15] reported that the latter method is also suited to large-scale preparation of virtually enantiopure **1**.

Using the acid-catalyzed benzylation of the ethyl lactate as described here avoids racemization during the reaction course and affords the product in high yield. In addition all reagents are commercially available and it is possible to carry out the reaction on a large scale.

Besides the one-step procedure described here, conversion of ester **1** to aldehyde **2** can be accomplished by reduction of the ester with lithium aluminum hydride , and subsequent oxidation of the alcohol produced using a Swern-oxidation protocol.[8, 14, 16]

The highly enantioenriched vinyl sulfone **3** has been used in the synthesis of the highly diastereomerically and enantiomerically enriched tetracarbonyl π-allyl iron(1+) complex **4**. Nucleophilic attack on this electrophilic organometal complex occurs regioselectively at the γ-position with respect to the sulfone functionality. In addition, the reaction proceeds with conservation of the double bond configuration allowing syntheses of highly enantioenriched (E)-alkenyl sulfones **5** with a wide range of substitution patterns at the allylic position[17,18,19] (Scheme 1). The value of **3** and the related methodology of the iron-mediated chirality transfer has also been demonstrated in the syntheses of methyl-branched natural products in high enantiomeric purity and in their naturally occurring absolute configuration.[20,21,22]

Scheme 1



3 (1R,2S,3R)-**4** **5**: ee > 96- > 97%

This preparation is referenced from:

- Org. Syn. Coll. Vol. 10, 289
- Org. Syn. Coll. Vol. 10, 672

References and Notes

1. Institut für Organische Chemie, Technical University of Aachen, Professor Pirlet-Straße 1, D-52074 Aachen, Germany.
2. Symyx Technologies, 3100 Central Expressway, Santa Clara, CA 95051.
3. Ito, Y.; Kobayashi, Y.; Kawabata, T.; Takase, M.; Terashima, S. *Tetrahedron* **1989**, *45*, 5767.
4. Rathke, M. W.; Nowak, M. *J. Org. Chem.* **1985**, *50*, 2624.
5. Wessel, H.-P.; Iversen, T.; Bundle, D. R. *J. Chem. Soc., Perkin Trans. I* **1985**, 2247.
6. Some recent publications include: (a) Ayerbe, M.; Arrieta, A.; Cossio, F. P.; Linden, A. *J. Org. Chem.* **1998**, *63*, 1795; (b) Yang, H. W.; Romo, D. *J. Org. Chem.* **1998**, *63*, 1344; (c) Reetz, M.; Haning, H. *J. Organomet. Chem.* **1997**, *541*, 117; (d) Almendros, P.; Thomas, E. J. *J. Chem. Soc., Perkin Trans. I* **1997**, 2561; (e) Jacobi, P. A.; Herradura, P. *Tetrahedron Lett.* **1997**, *38*, 6621; (f) Szymoniak, J.; Thery, N.; Moise, C. *Bull. Soc. Chim. Fr.* **1997**, *134*, 85; (g) Mikami, K.; Matsukawa, S.; Sawa, E.; Harada, A.; Koga, N. *Tetrahedron Lett*. **1997**, *38*, 1951; (h) Hoppe, D.; Tebben, P.; Reggelin, M.; Bolte, M. *Synthesis* **1997**, 183; (i) Cormick, R.; Lofstedt, J.; Perlmutter, P.; Westman, G. *Tetrahedron Lett.* **1997**, *38*, 2737; (j) Gennari, C.; Moresca, D.; Vulpetti, A.; Pain, G. *Tetrahedron* **1997**, *53*, 5593; (k) Enders, D.; Jandeleit, B.; von Berg, S. *J. Organomet. Chem.* **1997**, *533*, 219; (l) Enders, D.; Jandeleit, B.; von Berg, S. *Synlett* **1997**, 421; (m) Enders, D.; Frank, U.; Fey, P.; Jandeleit, B.; Lohray, B. B. *J. Organomet. Chem.* **1996**, *519*, 147; (n) Klute, W.; Kruger, M.; Hoffmann, R. W. *Chem. Ber.* **1996**, *129*, 633; (o) Evans, D. A.; Murry, J. A.; Kozlowski, M. C. *J. Am. Chem. Soc.* **1996**, *118*, 5814; (p) Peng, Z.-H.; Li, Y.-L.; Wu, W.-L.; Liu, C.-X.; Wu, Y.-L. *J. Chem. Soc., Perkin Trans. I* **1996**, 1057; (q) Marshall, J. A.; Jablonowski, J. A.; Welmaker, G. S. *J. Org. Chem.* **1996**, *61*, 2904; (r) Solladie-Cavallo, A.; Roche, D.; Fischer, J.; De Cian, A. *J. Org. Chem.* **1996**, *61*, 2690; (s) Jacobi, P. A.; Murphree, S.; Rupprecht, F.; Zheng, W. *J. Org. Chem.* **1996**, *61*, 2413; (t) Bernardi, A.; Marchionni, C.; Novo, B.; Karamfilova, K.; Potenza, D.; Scolastico, C.; Roversi, P. *Tetrahedron* **1996**, *52*, 3497; (u) Solladie-Cavallo, A.; Bonne, F. *Tetrahedron: Asymmetry* **1996**, *7*, 171; (v) Jain, N. F.; Cirillo, P. F.; Pelletier, R.; Panek, J. S. *Tetrahedron Lett.* **1995**, *36*, 8727; (w) Marshall, J. A.; Jablonowski, J. A.; Luke, G. P. *J. Org. Chem.* **1994**, *59*, 7825; (x) Roush, W. R.; VanNieuwenhze, M. S. *J. Am. Chem. Soc.* **1994**, *116*, 8536; (y) Shanmuganathan, K.; French, L. G.; Jensen, B. L. *Tetrahedron: Asymmetry* **1994**, *5*, 797; (z) Gennari, C.; Moresca, D.; Vulpetti, A.; Pain, G. *Tetrahedron Lett.* **1994**, *35*, 4623.
7. Kobayashi, Y.; Takase, M.; Ito, Y.; Terashima, S. *Bull. Chem. Soc. Jpn.* **1989**, *62*, 3038.
8. Takai, K.; Heathcock, C. H. *J. Org. Chem.* **1985**, *50*, 3247.
9. Baker, D. C.; Hawkins, L. D. *J. Org. Chem.* **1982**, *47*, 2179.
10. Mulzer, J.; Angermann, A. *Tetrahedron Lett.* **1983**, *24*, 2843.
11. Guanti, G.; Banfi, L.; Guaragna, A.; Narisano, E. *J. Chem. Soc., Chem. Commun.* **1986**, 138.
12. Bianchi, D.; Cesti, P.; Golini, P. *Tetrahedron* **1989**, *45*, 869.
13. Mislow, K.; O'Brien, R. E.; Schaefer, H. *J. Am. Chem. Soc.* **1962**, *84*, 1940.
14. Massad, S. K.; Hawkins, L. D.; Baker, D. C. *J. Org. Chem.* **1983**, *48*, 5180.
15. Varelis, P.; Johnson, B. L. *Aust. J. Chem.* **1995**, *48*, 1775.
16. Wuts, P. G. M.; Bigelow, S. S. *J. Org. Chem.* **1983**, *48*, 3489.
17. For accounts and reviews see: (a) Ref. 6l; (b) Enders, D.; Jandeleit, B.; von Berg, S. In "Organic Synthesis via Organometallics, OSM 5"; Helmchen, G., Ed.; Vieweg: Braunschweig, 1997, 279.
18. Enders, D.; Jandeleit, B.; Raabe, G. *Angew. Chem.* **1994**, *106*, 2033; *Angew. Chem., Int. Ed. Engl.* **1994**, *33*, 1949.
19. (a) Enders, D.; von Berg, S.; Jandeleit, B. *Synlett* **1996**, 18; (b) Jackson, R. F. W.; Turner, D.; Block, M. H. *Synlett* **1997**, 789.
20. Enders, D.; Jandeleit, B. *Synthesis* **1994**, 1327.
21. Enders, D.; Jandeleit, B. *Liebigs Ann.* **1995**, 1173.
22. Enders, D.; Jandeleit, B.; Prokopenko, O. F. *Tetrahedron* **1995**, *51*, 6273.

Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

(−)-(E,S)-3-(Benzyloxy)-1-butenyl phenyl sulfone:
Benzene, [[[1-methyl-3-(phenylsulfonyl)-2-propenyl]oxy]methyl]-, [S-(E)]- (13); (168431-27-4)

(−)-(S)-Ethyl 2-(benzyloxy)propanoate:
Propanoic acid, 2-(phenylmethoxy)-, ethyl ester, (S)- (9); (54783-72-1)

(−)-(S)-2-(Benzyloxy)propanal:
Propanal, 2-(phenylmethoxy)-, (S)- (11); (81445-44-5)

(S)-Ethyl hydroxypropanoate:
(S)-Ethyl lactate:
Lactic acic, ethyl ester, L- (8);
Propanoic acid, 2-hydroxy-, ethyl ester, (S)- (9); (687-47-8)

O-Benzyl-2,2,2-trichloroacetimidate:
Ethanimidic acid, 2,2,2-trichloro-, phenylmethyl ester (11); (81927-55-1)

Trifluoromethanesulfonic acid: HIGHLY CORROSIVE:
Methanesulfonic acid, trifluoro- (8,9); (1493-13-6)

Diisobutylaluminum hydride:
Aluminum, hydrodiisobutyl- (8);
Aluminum, hydrobis(2-methylpropyl)- (9); (1191-15-7)

Lithium bromide (8,9); (7550-35-8)

Diethyl[(phenylsulfonyl)methyl]phosphonate:
Phosphonic acid, [(phenylsulfonyl)methyl]-, diethyl ester (9); (56069-39-7)

Acetonitrile: TOXIC (8,9); (75-05-8)

Triethylamine (8);
Ethanamine, N,N-diethyl- (9); (121-44-8)

Benzyl alcohol (8);
Benzenemethanol (9); (100-51-6)

Trichloroacetonitrile:
Acetonitrile, trichloro- (8,9); (545-06-2)

Sodium hydride (8,9); (7646-69-7)