Facial-Selective Allylation of Methyl Ketones for the Asymmetric Synthesis of Tertiary Homoallylic Ethers

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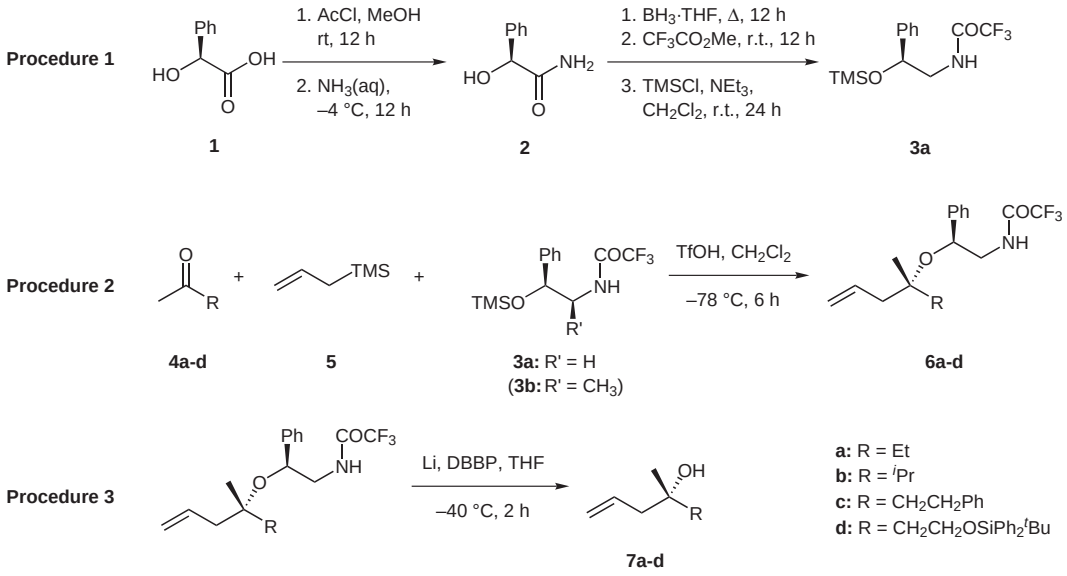
Received 7 April 2004

Dedicated to Professor Axel Zeeck on the occasion of his 65th birthday

Abstract: The stereoselective allylation of methyl ketones is described to give tertiary homoallylic ethers, which can easily be transformed into homoallylic alcohols by a Birch reduction. Reaction of methyl ketones **4** with allylsilane **5** in the presence of the chiral TMS ether **3a** and a catalytic amount of trifluoromethanesulfonic acid led to homoallylic ethers **6** in high yield with a selectivity of 9:1 to >20:1. The TMS ether **3a** was prepared from inexpensive mandelic acid, which is commercially available in both enantiomeric forms, in four steps.

Key words: allylations, allylsilannes, amino alcohols, asymmetric synthesis, Birch reduction, homoallylic alcohols



Scheme 1

Introduction

The facial-selective addition of allylmetals to carbonyl groups to furnish homoallylic alcohols is a fundamental transformation in chemistry and a widely used process in organic synthesis.[1] Typically, either chiral acetals, chiral catalysts or an equimolar amount of a chiral allyl reagent such as allylboranes or allyltitanium compounds are employed.[2] Most of these reagents give excellent selectivities with aldehydes but fail when applied to simple ketones.[3] Some progress has been made using 20–30 mol% of chiral titanium binol-based catalysts. However, the yields for the allylation of simple aliphatic ketones are not satisfying.[4]

Recently we have demonstrated that the reaction of aliphatic aldehydes with allylsilanes in the presence of the trimethylsilyl ether of *N*-trifluoroacetylnorpseudoephedrine (**3b**) and a catalytic amount of trimethyl trifluoromethanesulfonate (TMSOTf) led to the corresponding secondary homoallylic ethers with a >99:1 selectivity.[5] The procedure using trifluoromethanesulfonic acid (TfOH) instead of TMSOTf could also be applied to methyl ketones **4** to give tertiary homoallylic ethers, with a selectivity of 1:9 to >20:1 in a domino fashion,[6] as the only products (Scheme 1).[7] The homoallylic ethers can easily be transformed into the corresponding tertiary alcohols by cleavage of the benzyl ether moiety using either lithium in liquid ammonia or – as a much milder

SYNTHESIS 2004, No. 13, pp 2236–2239
Advanced online publication: 14.07.2004
DOI: 10.1055/s-2004-829153; Art ID: E10904SS

method – lithium in THF in the presence of 4,4′-di-(*tert*-butyl)-1,1′-biphenyl (DBBP).[8]

However, the general use of this procedure has so far been hampered by the fact that only one of the enantiomers of norpseudoephedrine is commercially available. We herein wish to report the synthesis of a modified auxiliary **3a** for the allylation of methyl ketones derived from inexpensive mandelic acid (**1**) in four reaction steps. Since (*R*)- and (*S*)-mandelic acid are commercially available, both enantiomeric forms of the auxiliary **3a** can be prepared. It was shown that the influence of the methyl group in the 2-position of **3b** is negligible; thus, the use of **3a** allows the allylation of methyl ketones with the same facial selectivity as with **3b**.

In this article we report the synthesis of the auxiliary **3a** as well as a general method for the facial selective allylation of aliphatic methyl ketones **4** to give enantiopure tertiary homoallylic ethers **6** in a highly diastereoselective fashion and their transformation into enantiopure homoallylic alcohols **7**.

Scope and Limitations

The preparation of (*S*)-2,2,2-trifluoro-*N*-(2-phenyl-2-trimethylsilyloxyethyl)acetamide (**3a**) is described in detail in Procedure 1. (*S*)-Mandelic acid (**1**) was transferred into the amide **2** using acetyl chloride followed by addition of ammonia. Reduction of the amide **2** employing the borane-THF complex gave the corresponding amine, which without purification was protected at the nitrogen as *N*-trifluoroacetyl group using CF_3CO_2Et and at the hydroxyl group as TMS ether with TMSCl (Scheme 1). The obtained product **3a** was purified by a simple column chromatography and then used for the allylation reaction.

The allylation reaction is described in Procedure 2. While the auxiliary **3a** works excellently with aliphatic methyl ketones it is not suitable for the diastereoselective allylation of α,β-unsaturated and aromatic methyl ketones. However, methyl ketones containing a double bond or an aromatic substituent, which is not in conjugation with the carbonyl moiety give excellent results; also other functional groups such as esters or ethers are tolerated. Hydroxy ketones are unreactive probably due to an inhibition caused by the hydroxyl group; in contrast protected hydroxy ketones react smoothly within 6 hours with high selectivities. The type of the protecting group in the substrate can have some influence on the selectivity; the TBDPS (*tert*-butyldiphenylsilyl) group was found to give good results.

As the allylation of the TBDPS-protected 4-hydroxybutanone **4d** leads to very interesting substrates **6d** as well as **7d** for further use in natural product synthesis,[7] and shows an excellent selectivity of >20:1 (unpurified), this compound was chosen as substrate for the general experimental procedure. Further typical results employing other aliphatic ketones are listed in Table 1 including butan-2-one as the most difficult substrate, which still allows a remarkable facial selectivity of 11.9:1 at –96 °C (unpurified). Attention should be drawn to the temperature of the reaction mixture. Having an external acetone bath, cooled by a cryostat, the solvent volume should not exceed 250 mL. In general, two equivalents of the ketone and the allylsilane are necessary, but it is possible to recover the unused ketone after the reaction by column chromatography nearly quantitatively. As an alternative, one can also employ one equivalent of the ketone to be transformed and one equivalent of ethyl isopropyl ketone, which does not react under the used reaction conditions, but is necessary to obtain good yields. The reason for this unusual behavior is not known yet. Although the starting materials, the auxiliary and the products show similar R_f values, a simple purification by column chromatography on silica gel is sufficient to obtain pure products in most cases. It should be noted that the obtained products are diastereomers, which can be further enriched using achiral adsorbents.

Table 1 Synthesis of Tertiary Homoallylic Ethers **6a–d** from Different Ketones **4a–d** in the Presence of **3a** (1 equiv)

Entry	R in **4**, **6** and **7**	T (°C)	Yield (%)	dr (%)
1: **a**	Et	–78	89	8.8:1
2: **a**	Et	–96	72	11.9:1
3: **b**	^{i}Pr	–78	82	13.6:1
4: **c**	CH_2CH_2Ph	–78	68	10.7:1
5: **d**	$CH_2CH_2OSiPh_2{}^{t}Bu$	–78	87	>20:1

Cleavage of the benzyl ether moiety is detailed in Procedure 3. The earlier used Birch conditions (lithium in liquid ammonia) described previously[7] gave no reliable results of the reaction outcome. In several cases the allylic double bond in **6** was partly or totally reduced, while using lithium in THF with DBBP, no reduction of the allylic double bond was observed.[8] The commercially available, but expensive DBBP can be prepared in a single reaction step starting from biphenyl, *tert*-butyl chloride and catalytic amounts of aluminum chloride as reported by Timberlake et al.[9]

Procedures

Herein we present the synthesis of the chiral auxiliary (*S*)-2,2,2-trifluoro-*N*-(2-phenyl-2-trimethylsilanyloxyethyl)acetamide (**3a**), the allylation of the methyl ketones **4a–d** and the removal of the chiral auxiliary in the products **6a–d** to give the homoallylic alcohols **7a–d**. In Procedure 1 the four step synthesis of **3a** starting from (*S*)-mandelic acid (**1**) with formation of the amide, followed by reduction and protection of the amino and hydroxyl group is described, which takes place in an overall yield of 68%. Procedure 2 illustrates as an example, the facial

selective allylation of 4-*tert*-butyldiphenylsilyloxybutanone (**4d**) to give **6d** in a yield of 87% and a selectivity of >20:1. Procedure 3 describes the cleavage of the benzyl ether moiety in **6d** to afford the enantiopure homoallylic alcohol **7d** in 82% yield.

4,4′-Di-(*tert*-butyl)-1,1′-biphenyl (DBBP)

Anhyd $AlCl_3$ (3.00 g, 22.5 mmol) was added to a 250 mL dry three-neck round-bottom flask, equipped with a magnetic stirring bar, condenser, addition funnel and a stopper, charged with nitromethane (75 mL) and biphenyl (13.2 g, 85.0 mmol). The color of the solution changed to deep violet. A solution of 2-chloro-2-methylpropane (17.4 g, 190 mmol) in nitromethane (20 mL) was then added dropwise to the stirred mixture over 30 min. At the end of the addition, the reaction started with vigorous evolution of HCl gas. The reaction mixture was stirred overnight, poured onto crushed ice in a 500 mL beaker and allowed to warm up to r.t. The organic layer was extracted with a mixture of nitromethane–pentane (1:1, 3 × 50 mL), dried ($MgSO_4$), and then filtered. The yellowish filtrate was treated with silica gel (100 g) to provide a colorless solution, which was concentrated in vacuo. The white powder was recrystallized from nitromethane to give white needles of 4,4′-di-(*tert*-butyl)-1,1′-biphenyl; yield: 21.0 g (92%); mp 128 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.44–7.55 (m, 8 H_{arom}), 1.37 [s, 18 H, 2 × $C(CH_3)_3$].

^{13}C NMR (50 MHz, $CDCl_3$): δ = 149.9 (C_{arom}), 139.2 (C_{arom}), 126.7 (CH_{arom}), 125.6 (CH_{arom}), 34.5 [2 *C*$(CH_3)_3$], 31.4 [2 C(*C*$H_3)_3$].

MS (EI, 70 eV): *m*/*z* (%) = 266.2 (41), 251.3 (100).

Procedure 1

(*S*)-Mandelic Acid Amide (2)

A 1 L oven dried two neck round-bottom flask, equipped with a magnetic stirring bar, and a three-way stopcock attached to a balloon filled with argon was charged with (*S*)-mandelic acid (**1**; 25.0 g, 164 mmol) and anhyd MeOH (670 mL) and cooled to 0 °C with an ice-water bath. Acetyl chloride (31.5 mL, 443 mmol) was added dropwise to the flask via syringe at 0 °C. After stirring 12 h at r.t., the solvent was evaporated on a rotary evaporator. The residue was dissolved in MeOH (125 mL), and aq ammonia solution (310 mL) was added. The flask was stored in the fridge overnight. After evaporation of the solvent a white solid was obtained, which was recrystallized from EtOH–pentane to afford white crystals; yield: 22.0 g (89%); mp 120 °C; $[\alpha]_D^{20}$ +78.0 (*c* = 1.7, acetone).

IR (NaCl): 3357, 3187, 2927. 1681, 1495, 1452, 1422, 1295, 1190, 1102, 1056, 928, 895, 859, 763, 710 cm^{-1}.

^{1}H NMR (300 MHz, DMSO): δ = 7.22–7.43 (m, 6 H, 5 H_{arom}, NH), 7.13 (s, 1 H, NH), 5.95 (d, 1 H, *J* = 4.7 Hz, 2-H), 4.83 (d, 1 H, *J* = 4.7 Hz, OH).

^{13}C NMR (50 MHz, DMSO): δ = 174.5 (C-1), 141.3 (C_{arom}), 127.8 (2 CH_{arom}), 127.2 (CH_{arom}), 126.4 (2 CH_{arom}), 73.4 (C-2).

MS (EI, 70 eV): *m*/*z* (%) = 151.1 (10), 107.0 (100).

HRMS (EI, 70 eV): *m*/*z* calcd: 151.0633; found: 151.0633.

(*S*)-2,2,2-Trifluoro-*N*-(2-phenyl-2-trimethylsilyloxyethyl)acetamide (3)

An oven-dried 500 mL dry three-neck round-bottom flask, equipped with a magnetic stirring bar, addition funnel, a stopper, and a reflux condenser with a three-way stopcock attached to a balloon filled with argon was charged with (*S*)-mandelic acid amide (**2**; 22.0 g, 146 mmol). A solution of borane-THF complex (1 M, 300 mL, 300 mmol) was added via an addition funnel (gas formation!). The mixture was refluxed for 12 h and cooled to r.t., before MeOH (60 mL) was carefully added (gas formation!) via an addition funnel. After evaporation of the solvent, the crude product was diluted with MeOH (185 mL). CF_3CO_2Et (14.4 mL, 143 mmol) was added dropwise and the mixture was stirred for 12 h at r.t. The solvent was removed under reduced pressure, and the residue was diluted in CH_2Cl_2 (230 mL). Et_3N (37.9 mL, 274 mmol) was added, and the mixture was cooled to 0 °C with an ice-water bath followed by addition of TMSCl (17.4 mL, 143 mmol). After stirring the mixture for 3 d at r.t., it was quenched with H_2O (150 mL), and the crude product was extracted with CH_2Cl_2 (3 × 250 mL). The combined organic layers were washed with brine (200 mL), dried ($MgSO_4$), and concentrated. The residue was purified by silica gel column chromatography using pentane–*tert*-butyl methyl ether (5:1) as eluent to afford **3a** (27.5 g, 76% over three steps) as a colorless oil; $[\alpha]_D^{20}$ +53.5 (*c* = 1.0, $CHCl_3$).

IR (NaCl): 3317, 3090, 3067, 3032, 2959, 1708, 1556, 1494, 1454, 1366, 1254, 1166, 1103, 1027, 963, 842, 756, 700 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.18–7.31 (m, 5 H, 5 H_{arom}), 6.60 (s, 1 H, NH), 4.74 (dd, 1 H, *J* = 8.4, 3.8 Hz, 1-H), 3.66 (ddd, 1 H, *J* = 13.5, 7.5, 3.8 Hz, 2-H_a), 3.19 (ddd, 1 H, *J* = 13.5, 8.4, 4.5 Hz, 2-H_b), –0.02 [s, 9 H, $Si(CH_3)_3$].

^{13}C NMR (50 MHz, $CDCl_3$): δ = 140.9 (C_{arom}), 128.5 (2 CH_{arom}), 128.1 (CH_{arom}), 125.8 (2 CH_{arom}), 72.7 (C-1), 47.4 (C-2), –0.16 [$Si(CH_3)_3$].

MS (DCI, 200 eV): *m*/*z* (%) = 628.5 (2), 340.3 (50), 323.3 (100).

Anal. Calcd for $C_{13}H_{18}F_3NO_2Si$: C, 51.13; H, 5.94. Found: C, 51.38; H, 5.96.

Procedure 2

(3*S*,1′*S*)-1-*tert*-Butyldiphenylsiloxy-3-methyl-3-(1′-phenyl-2′-trifluoroacetamido-1′-ethoxy)hex-5-ene (6d)

An oven-dried 500 mL dry two-neck round-bottom flask, equipped with a magnetic stirring bar, a stopper, and a three-way stopcock attached to a balloon filled with argon was charged with **3a** (13.8 g, 42.3 mmol), 4-*tert*-butyldiphenylsilyloxybutanone (**4d**; 25.8 g, 84.5 mmol) and CH_2Cl_2 (250 mL). The mixture was cooled to –78 °C using a cryostat with an acetone bath. Allyltrimethylsilane (14.4 mL, 84.5 mmol) was added via syringe at –78 °C, followed by addition of TfOH (790 μL, 8.45 mmol). After stirring for 6 h at –78 °C, the reaction was quenched with Et_3N (18 mL) and MeOH (200 mL). The crude product was extracted with CH_2Cl_2 (4 × 250 mL), dried (Na_2SO_4), and concentrated. The residue was purified by silica gel column chromatography using pentane–Et_2O (9:1) as eluent to afford **6d** as a colorless oil; yield: 21.5 g (87%), $[\alpha]_D^{20}$ +26.0 (*c* = 1.0, $CHCl_3$).

IR (NaCl): 3331, 3072, 2932, 1716, 1548, 1428, 1168, 1111, 917, 823, 739, 702 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.58–7.64 (m, 4 H, 4 H_{arom}), 7.33–7.45 (m, 6 H, 6 H_{arom}), 7.20–7.30 (m, 5 H, 5 H_{arom}), 6.62 (s, 1 H, NH), 5.75 (ddt, 1 H, *J* = 16.9, 10.2, 7.3 Hz, 5-H), 5.00–5.08 (m, 2 H, 6-H_2), 4.60 (dd, 1 H, *J* = 8.3, 4.1 Hz, 1′-H), 3.53–3.74 (m, 3 H, 1-H_2, 2′-H_a), 3.17 (ddd, 1 H, *J* = 13.1, 8.3, 4.5 Hz, 2′-H_b), 2.23 (d, 2 H, *J* = 7.1 Hz, 4-H_2), 1.70 (t, 2 H, *J* = 7.0 Hz, 2-H_2), 1.00 [s, 9 H, $C(CH_3)_3$], 0.95 (s, 3 H, 3-CH_3).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 156.9 (q, $^2J_{CF}$ = 36.2 Hz, C=O), 141.6 (C_{arom}), 135.5*, 133.7*, 129.6*, 127.9*, 127.6*, 126.1* (C-5, 2 C_{arom}, 15 CH_{arom}), 118.3 (C-6), 78.0 (C-1′), 71.8 (C-3), 60.0 (C-4), 46.7 (C-2′), 44.1 (C-2), 42.2 (C-4), 26.8 [C(*C*$H_3)_3$], 23.9 (3-CH_3), 19.1 [*C*$(CH_3)_3$].

MS (DCI, 200 eV): *m*/*z* (%) = 601.6 (100), 584.6 (5).

HRMS (ESI): *m*/*z* calcd for 606.2622; found: 606.2623.

Procedure 3

(*S*)-1-(*tert*-Butyldiphenylsilyloxy)-3-methylhex-5-en-3-ol (7d)

An oven-dried 1 mL dry two-neck round-bottom flask, equipped with a magnetic stirring bar, a stopper and a three-way stopcock attached to a ballon filled with argon was charged with THF (420 mL), and the solvent was degassed three times by freezing with a dry ice acetone bath in vacuo. DBBP (13.3 g, 49.9 mmol) and Li granulates (approx. size 1 mm) were added, and the mixture was vigorously stirred at r.t. until the solution showed a deep blue colour. After cooling down to 0 °C with an ice-water bath, the mixture was stirred for an additional 2 h before the solution was cooled to –78 °C using a cryostat with an acetone bath. A solution of **6d** (36.4 g, 62.3 mmol) in THF (100 mL) was added dropwise. After stirring for 1 h at –78 °C, the mixture was allowed to warm to –50 to –40 °C and was stirred for an additional 2 h. The reaction was quenched by adding solid NH_4Cl at –40 °C (until the black solution turned yellow), and allowed to warm to r.t. The solid residues were filtered off before adding a half-saturated solution of NH_4Cl (400 mL), and the crude product was extracted with Et_2O (3 × 300 mL). The combined organic layers were washed with brine (200 mL), dried (Na_2SO_4), filtered, and concentrated. The residue was purified by silica gel chromatography using first pentane to elute DBBP, and then a gradient of pentane–Et_2O (15:1 to 3:1) as eluent to afford **7d** (18.8 g, 82%) as a yellow oil; $[\alpha]_D^{20}$ –5.8 (*c* = 0.5, $CHCl_3$).

IR (NaCl): 3444, 3072, 2928, 1640, 1429, 1112, 913, 822, 737, 702 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.64–7.73 (m, 4 H, 4 H_{arom}), 7.35–7.48 (m, 6 H, 6 H_{arom}), 5.86 (ddt, 1 H, *J* = 17.0, 10.4, 7.4 Hz, 5-H), 5.01–5.10 (m, 2 H, 6-H_2), 3.89 (t, 2 H, *J* = 6.0 Hz, 1-H_2), 2.24–2.37 (m, 2 H, 4-H_2), 1.80 (dt, 1 H, *J* = 14.5, 6.1 Hz, 2-H_a), 1.68 (dt, 1 H, *J* = 14.5, 5.6 Hz, 2-H_b), 1.23 (s, 3 H, 3-CH_3), 1.05 [s, 9 H, $C(CH_3)_3$].

^{13}C NMR (50 MHz, $CDCl_3$): δ = 135.5 (2 CH_{arom}), 134.5 (C-5), 132.7 (C_{arom}), 129.9 (CH_{arom}), 127.8 (2 CH_{arom}), 117.8 (C-6), 72.4 (C-3), 61.7 (C-1), 47.0 (C-4), 41.1 (C-2), 26.8 [$C(CH_3)_3$], 26.6 (3-CH_3), 19.0 [$C(CH_3)_3$].

MS (DCI, 200 eV): *m*/*z* (%) = 369.4 (100), 386.4 (18).

Anal. Calcd for $C_{23}H_{32}O_2Si$: C, 74.95; H, 8.75. Found: C, 75.12; H, 8.66.

Acknowledgment

This work was supported by the DFG (Sonderforschungsbereich 416) and the Fonds der Chemischen Industrie. We are grateful to Wacker-Chemie GmbH for supplying chlorotrimethylsilane and *tert*-butyldiphenylchlorosilane.

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