The Synthesis of Fused Ring Systems Utilising the Intramolecular Addition of Alkenyl Radicals to Furans

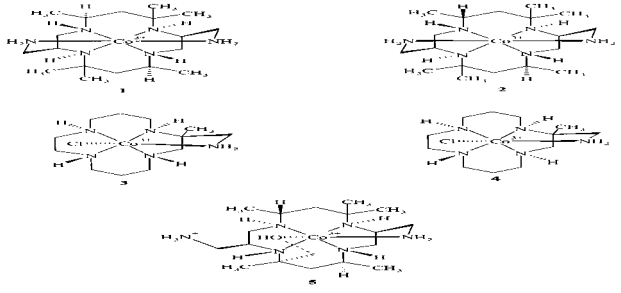
Aydin Demircan*[a] and Philip J. Parsons[b]

Keywords: Cyclization / Fused-ring systems / Radical reactions

Studies on the intramolecular addition of alkenyl radicals to furans are reported. The alkenyl radicals undergo a 5-exo-trig addition to furans and the resulting spirocyclic intermediate can fragment to give substituted cyclopentene rings.

Introduction

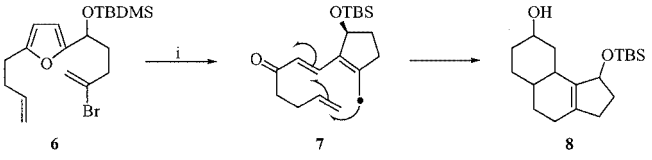
The use of radical reactions, and in particular radical cyclisation sequences, has been of great interest in organic chemistry for the last fifteen years.[1] In a previous paper[2] we demonstrated simple cyclopentenes could be formed by radical additions to furans and, subsequently, Pattenden et al. have applied this method in a synthesis of fused ring systems.[3] We have further examined reaction cascades resulting in the formation of substituted aromatic rings using a simple furan as the starting material.[4] The alkenyl bromide **1** was treated with tri-*n*-butyltin hydride to generate the alkenyl radical **2**. The alkenyl radical **2** reacted with the tethered furan ring to give the intermediate spirocyclic radical **3**, which underwent fragmentation to give the cyclopentenol intermediate **4**. When the intermediate **4** was trapped with a hydrogen atom donor, such as tri-*n*-butyltin hydride, the cyclopentenol **5** was isolated (Scheme 1).



Scheme 1. Reagents: i) Bu_3SnH, AlBN, PhH, 80 °C

[a] Nigde University, Faculty of Arts and Science, Department of Chemistry,
51700 Nigde, Turkey
Fax: (internat.) + 90-388/2250181
E-mail: ademircan@nigde.edu.tr
[b] The Chemical Laboratories, School of Chemistry, Physics & Environmental Science, University of Sussex,
Falmer, Brighton, East Sussex BN1 9QJ, U.K.
Fax: (internat.) + 44-1273/677196
E-mail: p.j.parsons@sussex.ac.uk

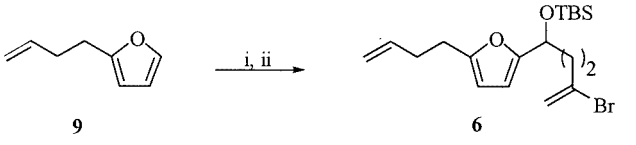
We wondered if it would be possible to trap the intermediate radical **4** with an alkene attached to the furan moiety (Scheme 2).



Scheme 2. Reagents: i) Bu_3SnH, AlBN, PhH, ΔT

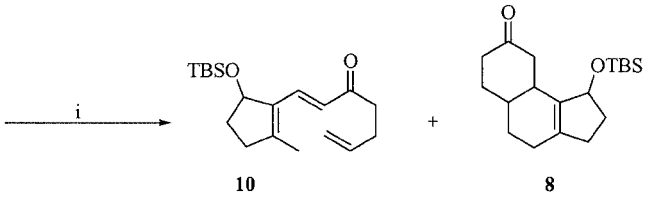
Results and Discussion

The bromoalkene **6** was synthesised as shown in Scheme 3.



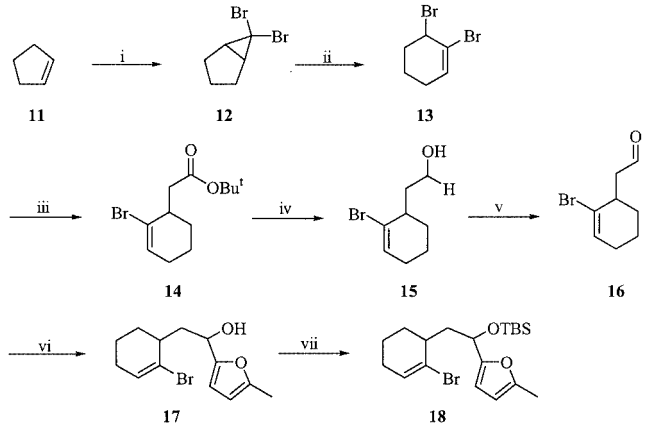
Scheme 3. Reagents: i) *n*BuLi, Et_2O, then 4-bromo-4-pentenal, −70 °C. ii) TBDMSCl, Imidazole, DMAP, CH_2Cl_2

Addition of *n*-butyllithium to the furan **9** resulted in smooth metallation.[5] Reaction of the anion derived from **9** with 4-bromo-4-pentenal[6] followed by protection of the resulting alcohol with a silyl group gave the bromide **6**. Treatment of **6** with tri-*n*-butyltin hydride in boiling toluene containing AlBN gave the enone **10** as the major product (50%) (Scheme 4).



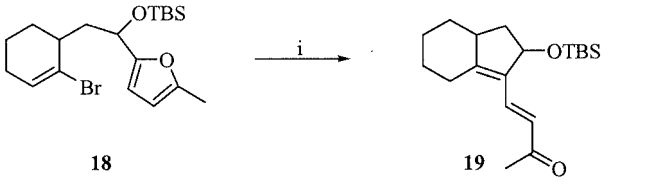
Scheme 4. Reagents: i) Bu_3SnH, AlBN, toluene, ΔT

Hitherto we have been unable to tune the reaction conditions to furnish the ketone **8** in more than trace quantities. To utilise our chemistry for the synthesis of fused rings, we synthesised the cyclohexenyl bromide **18** by the route outlined in Scheme 5.



Scheme 5. Reagents: i) $CHBr_3$, *t*BuOK (90%); ii) ΔT (70%); iii) *i*PCHA, *n*BuLi, *t*BuOAc, DME, −50 °C (53%); iv) $LiAlH_4$, Et_2O; v) Dess−Martin periodinane, DCM, RT (82%); vi) *n*BuLi, 2-methylfuran, then **16** (70%); vii) 2,6-lutidine, TBSOTf, DCM, −25 °C (75%)

Addition of dibromocarbene to cyclopentene, followed by thermal ring expansion, gave the known dibromide **13**. Displacement of the allylic bromide in **13** with lithium-*tert*-butyl acetate, followed by reduction of the resulting ester **14**[7] and oxidation of the resulting alcohol **15**, gave the aldehyde **16**. Reaction of **16** with 2-lithio-5-methylfuran, and then silyl protection of the resulting alcohol **17**, gave the desired bromide **18**. Exposure of the furan **18** to tri-*n*-butyltin hydride in boiling toluene gave the desired indene ring system **19** (Scheme 6).



Scheme 6. Reagents: i) Bu_3SnH, AIBN, toluene, reflux, 14 h (31%)

Conclusions

A new cascade reaction has been developed for the synthesis of fused ring systems that have potential applications in natural-product synthesis. These reactions will be utilised for the synthesis of prostaglandins and steroidal structures and our results will be published in the near future.

Experimental Section

Reactions were conducted in flame-dried glassware, under a nitrogen atmosphere except when noted otherwise. Solvents and reagents were freshly distilled as follows: tetrahydrofuran (THF) and diethyl ether (E) were distilled from over sodium benzophenone ketyl radical under a nitrogen atmosphere until anhydrous. Dichloromethane (CH_2Cl_2) and toluene were distilled from calcium hydride immediately prior to use. Reactions were monitored by thin-layer chromatography (TLC) using pre-coated silica plates (Macherey−Nagel Sil G UV254). Compounds were visualized using ultraviolet fluorescence, an alkaline potassium permanganate solution, or an acidic cerium(IV) sulfate solution. Column chromatography was carried out on Macherey−Nagel Kieselgel 60 (230−240 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker 300 MHz DPX 300 spectrometer. The chemical shifts are quoted in ppm as δ values downfield of tetramethylsilane (TMS) or relative to the residual solvent resonance. Infrared (IR) spectra were recorded on a Perkin−Elmer 1720 spectrophotometer; solid samples were recorded using potassium bromide discs, and liquid samples were recorded as thin films. High-resolution mass spectra (HRMS) and electron ionisation mass spectra (EI) were obtained on a Fisons VG Autospec mass spectrometer.

1-[5-(*tert*-Butyldimethylsilanyloxy)-2-methylcyclopent-1-enyl]oct-1-en-3-one (5): [4-Bromo-1-(5-pentyl-furan-2-yl)pent-4-enyloxy]-*tert*-butyldimethylsilane (**1**) (0.2 g, 0.48 mmol) and toluene (40 mL) were heated to 100 °C. A solution of tri-*n*-butyltin hydride (0.36 mL, 1.2 mmol) and AIBN (24 mg, 0.14 mmol) in toluene (6 mL) was slowly added over a period of 12 h. The reaction mixture was then heated for further 3 h at 100 °C. The mixture was then cooled, the solvents were evaporated under reduced pressure, and the residue was subjected by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (4:1), to give the title compound as a pale yellow oil (46 mg, 28%). TLC: R_f = 0.27 (40% PE/EtOAc). IR (film): $\tilde{\nu}_{max}$ = 2958 (s), 1665, 1626, 1592, 836 cm^{-1}. ^1H NMR ($CDCl_3$): δ_H = −0.48 (s, 6 H), 0.78 (s, 9 H), 1.16−1.12 (m, 3 H), 1.22−1.18 (m, 6 H), 1.49 (s, 3 H), 1.83−1.79 (m, 2 H), 2.12−2.08 (m, 2 H), 2.41 (t, J = 7.6 Hz, 2 H), 4.93 (t, J = 6.5 Hz, 1 H), 6.24 (d, J = 15.8 Hz, 1 H), 7.14 (d, J = 15.8 Hz, 1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ_C = 204, 153, 136, 135, 127, 42, 38.2, 33.0, 31.5, 26.0, 24.0, 22.0, 19.0, 18.5, 15.2, 14.4, −3.8 ppm.GC-MS: m/z = 337 ([M^+], 10%), 279 ([M^+ − *t*Bu], 100%), 205 ([M^+ − OTBDMS], 35%), 134. HRMS: calcd. for $C_{20}H_{36}O_2Si$ m/z = 336.2485; found m/z = 336.2476.

1-[5-(*tert*-Butyldimethylsilanyloxy)-2-methylcyclopent-1-enyl]hepta-1,6-dien-3-one (7): A solution of tri-*n*-butyltin hydride (1.48 mL, 4.9 mmol) and AIBN (52 mg, 0.30 mmol) in toluene (19 mL) was added to a boiling solution of [4-bromo-1-(5-but-3-enylfuran-2-yl)pent-4-enyloxy]-*tert*-butyldimethylsilane (**6**) (0.4 g, 1.0 mmol) and toluene (43 mL) over a period of 14 h. The reaction mixture was heated at 100 °C for a further 3 h. The reaction mixture was cooled to room temperature and was concentrated under reduced pressure. The resulting liquid was diluted with ethyl acetate

(52 mL), and then potassium fluoride (2.6 g) was added, followed by water (2.2 mL). The mixture was stirred for 4 h. Potassium carbonate was then added to the reaction mixture, which was then filtered. The filtrate was concentrated under reduced pressure and the residue was subjected to flash column chromatography to afford the title compound as a yellow oil (0.16 g, 50%). TLC: R_f = 0.5 (40% PE/EtOAc). IR: $\tilde{\nu}_{max}$ = 2929, 1631, 1594, 1361, 1078 cm^{-1}. ^1H NMR ($CDCl_3$): δ_H = 0.00 (s, 6 H), 0.93 (s, 9 H), 1.72−1.68 (m, 2 H), 2.00 (s, 3 H), 2.22−2.18 (m, 2 H), 2.42−2.38 (m, 2 H), 2.50 (t, J = 7.6 Hz, 2 H), 4.84−4.80 (m, 3 H), 5.74 (m, 1 H), 6.29 (d, J = 15.7 Hz, 1 H), 7.33 (d, J = 15.7 Hz, 1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 200.4, 153.2, 137.0, 135.7, 135.1, 126.3, 115.5, 78.1, 40.9, 37.4, 33.4, 28.7, 26.3, 18.5, 15.7, −3.4, −4.2 ppm. GC-MS: m/z = 263 ([M^+ − tBu], 100%), 207 ([M^+ − (TBDMS-H)], 26%), 189. HRMS: calcd. for $C_{15}H_{23}O_2Si$ m/z = 263.1467; found: m/z = 263.1458.

***tert*-Butyl (2-Bromocyclohex-2-enyl)acetate (14):** *n*BuLi (1.7 M in hexane, 1.55 mL, 15.2 mmol) was added dropwise to a solution of *iso*-propylcyclohexylamine (2.14 g, 15.2 mmol) in DME (50 mL) at −60 °C. The reaction mixture was stirred for 30 min, and then *tert*-butyl acetate (1.8 g, 15.2 mmol) was added dropwise. After further stirring for 15 min at 60 °C, a solution of 1,6-dibromocyclohexene (**13**) (4 g, 18.2 mmol) in DME (15 mL) was added dropwise. The suspension was warmed to ambient temperature and stirred for 4 h. The reaction mixture was then poured into icecold 10% HCl (75 mL) and extracted with diethyl ether (3 × 60 mL). The combined organic layers were washed with brine (60 mL), dried over $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure and the residue was subjected to flash column chromatography. The title compound was afforded as a pale yellow oil (2.2 g, 53%). TLC: R_f = 0.6 (80% PE/E). IR: $\tilde{\nu}_{max}$ = 2933, 1719, 734, 650 cm^{-1}. ^1H NMR ($CDCl_3$): δ_H = 1.38 (s, 9 H), 1.58 (m, 4 H), 1.98 (m, 2 H), 2.12−2.17 (dd, J = 16.0, 11.0 Hz, $1H_b$), 2.60 (m, $1H_a$), 2.73−2.77 (dd, J = 16.0, 3.3 Hz, 1 H), 6.01 (dt, J = 4.1, 1.1 Hz, 1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 171.9, 131.2, 126.7, 80.8, 40.0, 39.9, 29.2, 28.5, 28.1, 18.7 ppm. GC-MS): m/z = 276 ([M^+(^{81}Br)], 4%), 274 ([M^+(^{79}Br)], 4%), 261 ([M^+(^{81} Br) − Me], 27%), 259 ([M^+(^{79}Br) − Me], 26%), 220 ([M^+(^{81}Br) − tBu], 15%), 218 ([M^+(^{79}Br) − tBu], 16%), 203 ([M^+(^{81}Br) − OtBu], 56%), 201 ([M^+(^{79}Br) − OtBu], 100%), 195. HRMS: calcd. for $C_8H_{10}{}^{79}BrO$ [M − OtBu] m/z = 200.9910; found m/z = 200.9926.

2-(2-Bromocyclohex-2-enyl)ethanol (15): Lithium aluminum hydride (0.9 g, 24 mmol) was placed in a nitrogen-flushed two-neck flask equipped with a magnetic stirring bar. Dry diethyl ether (40 mL) was slowly injected onto the solid stirring at 0 °C. A solution of *tert*-butyl (2-bromocyclohex-2-enyl)acetate (**14**) (2.2 g, 8 mmol) in diethyl ether (10 mL) was added dropwise. The cooling bath was then removed and the suspension was left overnight at ambient temperature. When the reaction was complete by TLC, the flask was cooled to 0 °C and potassium sodium tartrate was added slowly to destroy excess lithium aluminum hydride. After filtration, the filtrate was dried over $MgSO_4$ and concentrated under reduced pressure. Purification of the residue by flash column chromatography afforded the title compound as a pale yellow oil (1.3 g, 80%). TLC: R_f = 0.22 (80% PE/E). IR: $\tilde{\nu}_{max}$ = 3340, 2924, 2855, 1644 cm^{-1}. ^1H NMR ($CDCl_3$): δ_H = 1.63−1.52 (m, $1H_b$), 1.75−1.65 (m, 4 H), 1.86−1.80 (m, $1H_a$), 2.12−2.04 (m, 3 H), 2.44−2.35 (br., 1 H), 3.70−3.64 (m, 2 H), 6.08 (dt, J = 1.2, J = 4.0 Hz,1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ_c = 130.5, 127.7, 61.0, 39.7, 36.6, 32.2, 31.6, 18.6 ppm. GC-MS: m/z = 188 ([M^+(^{81}Br) − OH], 52%), 186 ([M^+(^{79}Br) − OH], 53%), 107 ([M^+ − Br], 100%). HRMS: calcd. for $C_8H_{11}{}^{79}Br$ m/z = 186.0046; found m/z = 186.0044.

(2-Bromocyclohex-2-enyl)acetaldehyde (16): A solution of 2-(2-bromocyclohex-2-enyl)ethanol (**15**) (1.1 g, 5.5 mmol) in DCM (5 mL) was added to a stirred solution of Dess−Martin periodinane (3 g, 7.1 mmol) in DCM (30 mL). After 3 h of stirring, the solution was poured into a separating funnel containing NaOH (1 N, 30 mL) and diethyl ether (30 mL). The layers were separated and the diethyl ether layer was washed with water (2 × 30 mL) and brine (30 mL), dried over $MgSO_4$, and then concentrated under reduced pressure. The residue was subjected to flash column chromatography to give the title compound as a colourless oil (0.92 g, 82%). TLC: R_f = 0.43 (75% PE/E). IR: $\tilde{\nu}_{max}$ = 2933, 28639, 1642, 1448 cm^{-1}. ^1H NMR($CDCl_3$): δ_H = 1.59−1.51 (m, 4 H), 2.14−2.10 (m, 2 H), 2.50 (dq, J = 9.4, 2.16 Hz, $1H_b$), 2.89 (dd, J = 3.3, 16.9 Hz, $1H_a$), 2.94−2.90 (m, 1 H), 6.12 (dt, J = 1.3, 4.0 Hz, 1 H), 9.82 (d, J = 1.0 Hz, 1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 201.8, 131.8, 126.3, 48.2, 37.4, 30.5, 28.0, 19.0 ppm. GC-MS: m/z = 204 ([M^+(^{81}Br)], 11%), 202 ([M^+(^{79}Br)], 8%), 103 ([M^+(^{81}Br) − CHO], 21%); 171 ([M^+(^{79}Br) − CHO], 18%), 160 ([M^+(^{81}Br) − CH_2CHO], 51%), 158 ([M^+(^{79}Br) − CH_2CHO], 51%), 123 ([M^+ −Br], 78%).

2-(2-Bromocyclohex-2-enyl)-1-(5-methylfuran-2-yl)ethanol (17): *n*BuLi (6.3 mmol, 2.7 mL of 2.35 M solution in hexane) was added dropwise to a solution of 2-methylfuran (0.86 g, 10.5 mmol) in THF (30 mL) cooled to −78 °C under a nitrogen atmosphere. The solution was warmed to ambient temperature and stirred for 2 h before being cooled to −78 °C. A solution of (2-bromocyclohex-2-enyl)acetaldehyde (**16**) (1.7 g, 5.3 mmol) in THF (10 mL) was added slowly. The reaction mixture was warmed to ambient temperature and stirred for a further 3 h before being quenched with icecold water (30 mL). The aqueous layer was washed with diethyl ether (3 × 25 mL) and the combined organic layers were washed with brine (25 mL), dried over $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure. The residue was subjected to flash column chromatography to afford the title compound as a colourless oil (1.05 g, 70%). TLC: R_f = 0.19 (88% PE/EtOAc). IR: $\tilde{\nu}_{max}$ = 3463, 2931, 1738, 1447, 1246, 657 cm^{-1}. ^1H NMR ($CDCl_3$): δ_H = 1.54−1.45 (m, 4 H), 1.96−1.90 (m, 4 H), 2.10 (s, 3 H), 2.89−2.83 (br., 1 H), 4.73 (t, J = 6.7 Hz, 1 H), 5.84 (d, J = 3.9 Hz, 1 H), 6.03 (d, J = 3.1 Hz, 1 H), 6.08 (d, J = 3.1 Hz, 1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ_H = 154.0, 153.2, 131.8, 125.0, 109.6, 108.4, 66.9, 48.0, 31.7, 29.0, 22.3, 19.0, 14.8 ppm. GC-MS: m/z = 269 ([M^+(^{81}Br) − OH], 73%), 267 ([M^+(^{79}Br) − OH], 75%), 187 ([M^+ − (Br+OH+H)], 100%). HRMS: calcd. for $C_{13}H_{16}{}^{79}BrO$ m/z = 267.0385; found m/z = 267.0364.

[2-(2-Bromocyclohex-2-enyl)-1-(5-methylfuran-2-yl)ethoxy]-*tert*-butyldimethylsilane (18): *tert*-Butyldimethylsilyl trifluoromethanesulfonate (0.8 g, 3.06 mmol) was added dropwise to a stirred solution of 2-(2-bromocyclohex-2-enyl)-1-(5-methylfuran-2-yl)ethanol (**17**) (0.73 g, 2.55 mmol) and dry 2,6-lutidine (0.55 g, 5.1 mmol) in DCM (3 mL) at −25 °C. The reaction mixture was stirred for 2 h and then methanol (0.3 mL), DCM (20 mL) and water (25 mL) were added. The mixture was extracted with DCM (3 × 20 mL) and the combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure and the residue was subjected to flash column chromatography to afford the title compound as a pale yellow oil (0.76 g, 75%). TLC: R_f = 0.6 (88% PE/EtOAc). IR: $\tilde{\nu}_{max}$ = 2930, 2856, 1618, 1254 cm^{-1}. ^1H NMR ($CDCl_3$): δ_H = 0.00 (s, 6 H), 0.81 (s, 9 H), 1.64−1.53 (m, 6 H), 2.00−1.96 (m, 2 H), 2.19 (s, 3 H), 2.65−2.63 (br., 1 H), 4.73−4.69 (m, 1 H), 5.87−5.83 (m, 1 H), 6.03 (d, J = 3.1 Hz, 1 H), 6.14 (t, J = 3.1 Hz,1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ_C = 152.0, 151.6, 131.1, 125.0, 109.0, 107.6,

69.7, 46.8, 40.1, 31.1, 29.5, 26.1 (3 C), 23.0, 18.4, 14.1, −2.5, −3.2 ppm. GC-MS: *m*/*z* = 398 ([M^+(^{79}Br)], 3%), 343 ([M^+(^{81}Br) − *t*Bu], 59%), 341 ([M^+(^{79}Br) − *t*Bu], 60%), 269 ([M^+(^{81}Br) − OTBDMS], 100%). HRMS: calcd. for $C_{15}H_{22}{}^{79}BrO_2Si$ *m*/*z* = 341.0572; found *m*/*z* = 341.0594.

4-[2-(*tert*-Butyldimethylsilanyloxy)-3,3a,4,5,6,7-hexahydro-2H-inden-1-yl]-but-3-en-2-one (19): A solution of tri-*n*-butyltin hydride (0.53 mL, 1.98 mmol) and AIBN (26 mg, 0.15 mmol) in toluene (19 mL) was added over a period of 14 h to a boiling solution of [2-(2-bromocyclohex-2-enyl)-1-(5-methylfuran-2-yl)ethoxy]-*tert*-butyldimethylsilane (**18**) (0.36 g, 0.9 mmol) and AIBN (26 mg, 0.15 mmol) in toluene (125 mL). The reaction mixture was heated at reflux for an additional 4 h, cooled, and concentrated under reduced pressure. The resulting liquid was diluted with ethyl acetate (95 mL), potassium fluoride (2.8 g) and water (3.5 mL) were added, and the mixture was stirred for 3 h. Potassium carbonate was then added to the mixture and the solids filtered. The filtrate was concentrated under reduced pressure and the residue was subjected to flash column chromatography to afford the title compound as a pale yellow oil (88 mg, 31%). TLC: R_f = 0.32 (90% PE/EtOAc). IR: $\tilde{\nu}_{max}$ = 2928, 2855, 1718, 1666, 1626, 1256 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ_H = 0.00 (s, 6 H), 0.82 (s, 9 H), 1.69−1.30 (m, 6 H), 1.96−1.84 (m, 4 H), 2.30 (s, 3 H), 2.40 (m, 1 H), 4.94 (t, *J* = 6.2 Hz, 1 H), 6.32 (d, *J* = 16.0 Hz, 1 H), 7.28 (d, *J* = 16.0 Hz,1 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ_C = 198.0, 158.5, 134.3, 131.4, 125.5, 76.5, 47.5, 40.0, 35.0, 30.2, 28.6, 28.0, 27.0, 25.6 (3 C), 24.5, −4.6, −5.5 ppm. GC-MS: *m*/*z* = 320 ([M^+], 2.3%), 263 ([M^+ − *t*Bu], 100%), 205 ([M^+ − TBDMS], 9%), 189 ([M^+ − OTBDMS], 16%), 147 ([M^+ − (OTBDMS + CH_3CO − H)], 29%), 119 ([M^+ − OTBDMS − $CH_3COCHCH$)], 12%). HRMS: calcd. for $C_{15}H_{23}O_2Si$ [M − *tBu*] *m*/*z* = 263.1467; found *m*/*z* = 263.1450.

Acknowledgments

We thank Drs. Z. Gultekin and M. D. Mat for their interest in this work, and Nigde University for financial support (to A. D.)

[1] [1a] M. Malacria, *Chem. Rev.* **1996**, *96*, 289. [1b] P. J. Parsons, C. S. Penkett, A. J. Shell, *Chem. Rev.* **1996**, 96, 195. [1c] A. J. McCarroll, J. Walton, *Angew. Chem. Int. Ed.* **2001**, *40*, 2224.
[2] P. J. Parsons, M. Penverne, I. L. Pinto, *Synlett* **1994**, 721.
[3] P. Jones, W. S. Li, G. Pattenden, N. M. Thomson, *Tetrahedron Lett.* **1997**, 38, 9069.
[4] A. Demircan, P. J. Parsons, *Synlett* **1998**, 1215.
[5] R. Levine, V. Ramanathan, *J. Org. Chem.* **1962**, *27*, 1216.
[6] S. Huang, A. J. Manuiso, D. Swern, *J. Org. Chem.* **1978**, 43, 2480.
[7] F. Orsini, F. Pelizzoni, *Synth. Commun.* **1984**, 805.

Received June 18, 2002

