Supporting Information

Organomediated Morita–Baylis–Hillman Cyclization Reactions

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Experimental Section

All oxygen- or moisture-sensitive reactions were carried out in oven-dried glassware under a positive pressure of argon. Sensitive liquids and solutions were transferred by oven dried glass syringes, or cannula and were introduced through rubber septa through which a positive pressure of argon was maintained. Concentration of solutions was accomplished using a Buchi rotary evaporator with a water aspirator followed by removal of residual solvents on a vacuum line held at 0.1–1 torr.

Unless otherwise noted, all reagents and solvents were used without additional purification. Exceptions include: chromatography grade hexane and ethyl acetate which were technical grade and were distilled before use; Et₂O and THF were distilled from sodium-benzophenone ketyl under argon; pyridine, triethylamine and *t*-BuOH were distilled from CaH₂ under argon atmosphere.

Analytical thin layer chromatography (TLC) was performed on Merck precoated silica gel 60 F₂₅₄ glass plates. Visualization on TLC was achieved by use of UV light (254 nm) or exposure to basic potassium permanganate solution, or acidic anisaldehyde, or 5% phosphomolybdic acid in ethanol stain followed by heating. Flash column chromatography was carried out using Merck 60, 230–400 mesh ASTM silica gel. Additional purification was achieved through use of a CombiFlash Graduate Medium Pressure LC unit. Proton nuclear magnetic resonance spectroscopy (¹H NMR) was recorded on a Varian Fourier Transform 500 (500 MHz) spectrometer. Chemical shifts are reported in units, parts per million (ppm) relative to the singlet at 7.26 ppm for chloroform-*d* or in ppm relative to the singlet at 7.15 ppm for benzene-*d*₆. The following abbreviations are used to describe peak patterns where appropriate: b = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, *J* are reported in Hertz (Hz). Carbon-13 nuclear magnetic resonance spectroscopy (¹³C NMR) was recorded on a Varian Fourier Transform 300 (75 MHz) and was fully decoupled by broad-band decoupling. Chemical shifts are reported in ppm with the center line of the triplet for chloroform-*d* set at 77.0 ppm. Infrared (IR) spectra were recorded as thin films on sodium chloride plates using a Perkin-Elmer FT-IR Paragon 1000 Fourier Transform spectrometer with frequencies given in reciprocal centimeters (cm⁻¹). Mass spectra were obtained on a Jeol model JMS600H mass spectrometer using either electron impact (EI) (70 eV) or chemical ionization (CI) (isobutane or methane). Elemental analyses were performed at Atlantic Microlab Inc. in Norcross, GA.

Experimental Procedures

Preparation of primary alcohols

* prepared by a cross-metathesis reaction as shown below

Cross-metathesis

To a mixture of 4-pentenol (1.68 g, 20 mmol) and allyl acetate (4.00 g, 40 mmol) in CH_2Cl_2 (100 mL, 0.2M) was added Grubbs' #2¹ (125 mg, 0.2 mmol, 1 mol%). The mixture was heated at reflux for 4 h and the solvent removed *in vacuo*. The resulting aldehyde could be used without further purification in the Wittig olefination reaction or purified by flash chromatography. Yield after purification, (2.81 g, 90%).

Wittig Olefination

A mixture of the aldehyde (1.56 g, 10 mmol) and methyl-(triphenylphosphoranylidene)-2-propanone (3.18 g, 10 mmol) in THF (50 mL, 0.2M) was heated at reflux for 12 h. The solvent was removed *in vacuo* and the residue purified by filtration through a plug of silica gel before flash chromatography. Yield after purification, (1.86 g, 95%).

¹ Connon, S. J.; Blechert, S. *Angew. Chem. Int. Ed.* **2004**, 42, 1900.

Acetate Hydrolysis

To the acetate (1.57 g, 8 mmol) in the THF–H₂O (1:1) (40 mL, 0.2M) at ambient temperature was added LiOH•H₂O (336 mg, 8 mmol). After stirring for 4 h, the mixture was diluted with Et₂O, and added to saturated brine. The mixture was extracted with Et₂O, dried over Na₂SO₄ and concentrated *in vacuo*. Purification by flash chromatography provided the alcohol **1** (924 mg, 75%).

Preparation of secondary alcohols

Wittig Olefination

A mixture of 4-pentenal (1.68 g, 20 mmol) and methyl (triphenylphosphoranylidene)-2-propanone (6.36 g, 20 mmol) in THF (100 mL, 0.2M) was heated at reflux for 12 h. The solvent was removed *in vacuo* and the residue purified by filtration through a plug of silica gel. The resulting olefin could be used without further purification in reduction reaction or further purified by flash chromatography. Yield of enone after purification (2.41 g, 97%)

Reduction

To a suspension of LiAlH₄ (474 mg, 12.5 mmol) in Et₂O (50 mL) at room temperature was added the enone (3.1 g, 25 mmol) in Et₂O (10 mL). After stirring for 15 minutes, the excess LiAlH₄ was quenched with H₂O–NaOH(aq)–H₂O following the procedure of Fieser.² The mixture was filtered through Celite[®], dried over Na₂SO₄ and concentrated *in vacuo*. The resulting alcohol could be used without further purification in the TBS protection reaction or purified by flash chromatography. Yield of alcohol after purification (3.15 g, 99%).

² Fieser, M. and Fieser, L. F. Reagents for Organic Synthesis, vol. 5, page 382. John Wiley and Sons.

TBS ether protection

To the alcohol (3.15 g, 25 mmol) in CH_2Cl_2 (50 mL, 0.5M) at room temperature was added imidazole (1.87 g, 27.5 mmol) and TBSCl (3.77 g, 25 mmol). The mixture was stirred for 16 h and water added. The layers were separated, extracted with CH_2Cl_2 , dried over Na_2SO_4 and concentrated *in vacuo*. The TBS ether was purified by flash chromatography to provide (5.59 g, 93%).

Cross-metathesis

To a mixture of the TBS ether (5.52 g, 23 mmol) and methyl vinyl ketone (3.22 g, 46 mmol) in CH_2Cl_2 (115 mL, 0.2M) was added Grubbs' #2¹ (195 mg, 0.23 mmol, 1 mol%). The mixture was heated at reflux for 16 h and the solvent removed *in vacuo*. The resulting enone could be purified by filtration through a plug of silica gel and used without further purification or alternatively could be further purified by flash chromatography. Yield after purification (3.89 g, 60%).

TBS Deprotection

To the TBS ether (2.82 g, 10 mmol) in CH_3CN (20 mL, 0.5M) at ambient temperature was added $\text{HF}\cdot\text{Py}$ complex (70% HF) (2 eq.) and the mixture stirred until complete (TLC analysis) (~90 min). The mixture was diluted with Et_2O and added to saturated aqueous NaHCO_3 . It was extracted with Et_2O , dried over Na_2SO_4 and concentrated *in vacuo*. The alcohol was purified by flash chromatography to provide (1.34 g, 80%).

Cyclization Reactions

To SOCl_2 (0.15 mL, 2 mmol) in Et_2O (10 mL) at ambient temperature was added dropwise over 5 min a 0.1M Et_2O solution of the alcohol **1** (154 mg, 1 mmol) and pyridine (0.16 mL, 2 mmol). After stirring for 20 min, the Et_2O layer was washed with saturated aqueous NaHCO_3 , dried over Na_2SO_4 and concentrated *in vacuo* to provide the allylic chloride which was used without further purification. Tributylphosphine (0.25 mL, 1 mmol) was then added to a 0.5M solution of the allylic chloride (1 mmol) in *t*-BuOH (2 mL) and the mixture was allowed to stir at ambient temperature until complete consumption of starting material (TLC analysis) (~5 h), at which point methylene chloride–water (1:1) was added to the mixture followed by addition of potassium hydroxide (112 mg, 200 mol %) and BnEt_3NCl (22 mg, 10 mol %) and stirred until complete (2 h). The layers were separated and the aqueous layer extracted with CH_2Cl_2 . The

combined CH_2Cl_2 extracted were dried over Na_2SO_4 and concentrated *in vacuo*. Purification of the cyclization adduct **8** was achieved by flash chromatography. Yield (109 mg, 80%).

Spectral Data

This compound was isolated as a mixture of alkene geometrical isomers (E:Z; 9:1). Spectral data for the major isomer is reported.

^1H NMR (500 MHz, CDCl_3): 6.78 (dt, $J = 16.1, 6.6$ Hz, 1H, $\text{COCH}=\text{CH}$), 6.09 (d, $J = 16.1$ Hz, 1H, COCH), 5.70–5.68 (m, 2H, $\text{HC}=\text{CHCH}_2\text{OH}$), 4.11 (dm, $J = 3.2$ Hz, 2H, CH_2OH), 2.33 (dt, $J = 6.6, 6.6$ Hz, 2H, $\text{COCH}=\text{CHCH}_2$), 2.24 (partly obscured s, 3H, CH_3) 2.26–2.23 (partly obscured m, 2H, $\text{COHC}=\text{CHCH}_2\text{CH}_2$).

^{13}C NMR (75 MHz, CDCl_3): 198.8, 147.5, 131.2, 130.2, 129.0, 62.7, 31.6, 30.3, 26.5.

HRMS (CI) Calcd. for $\text{C}_9\text{H}_{14}\text{O}_2$ ($\text{M} + \text{H}$)⁺, 155.1072; Found 155.1074.

FTIR (film): 3418, 2924, 1673, 1626, 1362, 1257, 973 cm^{-1} .

Anal. Calcd. For $\text{C}_9\text{H}_{14}\text{O}_2$: C, 70.10; H, 9.15. Found: C, 70.47; H, 9.01.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; 9:1). Spectral data for the major isomer is reported.

¹H NMR (500 MHz, CDCl₃): 7.93–7.91 (m, 2H, Ar–H), 7.57–7.53 (m, 1H, Ar–H), 7.48–7.44 (m, 2H, Ar–H), 7.04 (dt, *J* = 15.4, 6.8 Hz, 1H, COCH=CH), 6.90 (d, *J* = 15.4 Hz, 1H, COCH), 5.73–5.71 (m, 2H, HC=CHCH₂OH), 4.11 (dm, *J* = 3.7 Hz, 2H, CH₂OH), 2.42 (dt, *J* = 6.8, 6.8 Hz, 2H, COCH=CHCH₂), 2.30 (dt, *J* = 6.8, 6.8 Hz, 2H, COCH=CHCH₂CH₂).

¹³C NMR (75 MHz, CDCl₃): 190.8, 148.8, 137.5, 137.5, 132.5, 130.3, 130.2, 128.3, 126.0, 62.9, 32.0, 30.5.

HRMS (FAB) Calcd. for C₁₄H₁₆O₂ (M + Na)⁺, 239.1048; Found 239.1036.

FTIR (film): 3412, 2924, 1668, 1620, 1597, 1578, 1448, 1288, 1228, 972 cm⁻¹.

Anal. Calcd. For C₁₄H₁₆O₂: C, 77.75; H, 7.46. Found: C, 77.85; H, 7.49.

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¹H NMR (500 MHz, CDCl₃): 7.29–7.26 (m, 2H, Ar–H), 7.20–7.17 (m, 3H, Ar–H), 6.80 (dt, *J* = 15.9, 6.8 Hz, 1H, COCH=CH), 6.12 (dm, *J* = 15.9 Hz, 1H, COCH), 5.69–5.66 (m, 2H, HC=CHCH₂OH), 4.10 (dd, *J* = 3.2, 0.5 Hz, 2H, CH₂OH), 2.94 (t, *J* = 6.9 Hz, 2H, PhCH₂), 2.87 (td, *J* = 6.9, 1.7 Hz, 2H, CH₂CO), 2.30 (dt, *J* = 6.8, 6.8 Hz, 2H, COCH=CHCH₂), 2.24–2.20 (dt, *J* = 6.8, 6.8 Hz, 2H, COCH=CHCH₂CH₂).

¹³C NMR (75 MHz, CDCl₃): 199.5, 146.4, 141.1, 130.7, 130.5, 130.2, 128.4, 128.3, 126.0, 63.3, 41.6, 31.8, 30.5, 30.0.

HRMS (FAB) Calcd. for C₁₆H₂₀O₂ (M + Na)⁺, 267.1361; Found 267.1362.

FTIR (film): 2418, 3027, 2627, 2855, 1694, 1668, 1627, 1496, 1454, 1367, 1095, 973 cm⁻¹.

Anal. Calcd. For C₁₆H₂₀O₂: C, 78.65; H, 8.25. Found: C, 78.95; H, 8.33.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; 10:1). Spectral data for the major isomer is reported.

¹H NMR (500 MHz, CDCl₃): 7.93–7.91 (m, 2H, Ar–H), 7.57–7.54 (m, 1H, Ar–H), 7.48–7.45 (m, 2H, Ar–H), 7.06 (dt, *J* = 15.4, 7.1 Hz, 1H, COCH=CH), 6.89 (dt, *J* = 15.4, 1.2 Hz, 1H COCH), 5.73–5.65 (m, 2H, HC=CHCH₂OH), 4.11 (dm, *J* = 4.4 Hz, 2H, CH₂OH), 2.34 (dt, *J* = 7.1, 7.1 Hz, 2H, COCH=CHCH₂), 2.14 (dt, *J* = 7.1, 7.1 Hz, 2H, CH₂CH=CHCH₂OH), 1.64 (tt, *J* = 7.1, 7.1 Hz, 2H, CH₂CH₂CH₂).

¹³C NMR (75 MHz, CDCl₃): 190.7, 149.5, 137.6, 132.5, 131.4, 129.8, 128.4, 128.3, 125.8, 63.1, 32.0, 31.4, 27.3.

HRMS (EI) Calcd. for C₁₅H₁₈O₂ (M)⁺, 230.1307; Found 230.1312.

FTIR (film): 3412, 2929, 2858, 1668, 1619, 1448, 1290, 1226, 970, 695 cm⁻¹.

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^1H NMR (500 MHz, CDCl_3): 6.78 (dt, $J = 15.9, 6.8$ Hz, 1H, $\text{COCH}=\text{CH}$), 6.08 (d, $J = 15.9$ Hz, 1H, COCH), 5.63 (dt, $J = 15.6, 6.8$ Hz, 1H, $\text{HC}=\text{CHCHOH}$), 5.57 (dd, $J = 15.6, 6.3$ Hz, 1H, $\text{HC}=\text{CHCHOH}$), 4.27 (dq, $J = 6.3, 6.3$ Hz, 1H, CHOH), 2.32 (dt, $J = 6.8, 6.8$ Hz, 2H, $\text{COCH}=\text{CHCH}_2$), 2.24 (s, 3H, CH_3CO), 2.22 (dt, $J = 6.8, 6.8$ Hz, 2H, $\text{COCH}=\text{CHCH}_2\text{CH}_2$), 1.25 (d, $J = 6.3$ Hz, 3H, CH_3CHOH).

^{13}C NMR (75 MHz, CDCl_3): 198.6, 147.4, 135.4, 131.2, 127.9, 67.9, 31.6, 30.2, 26.5, 23.2.

HRMS (CI) Calcd. for $\text{C}_{10}\text{H}_{16}\text{O}_2$ ($\text{M} + \text{H}$) $^+$, 169.1229; Found 169.1230.

FTIR (film): 3410, 2970, 1673, 1626, 1366, 1255, 973 cm^{-1} .

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¹H NMR (500 MHz, CDCl₃): 7.93–7.91 (m, 2H, Ar–H), 7.57–7.54 (m, 1H, Ar–H), 7.48–7.45 (m, 2H, Ar–H), 7.02 (dt, *J* = 15.4, 6.8 Hz, 1H, COCH=CH), 6.89 (dm, *J* = 15.4 Hz, 1H, COCH), 5.66 (dt, *J* = 15.4, 6.8 Hz, 1H, HC=CHCHOH), 5.60 (dd, *J* = 15.4, 6.6 Hz, 1H, HC=CHCHOH), 4.28 (dq, *J* = 6.6, 6.6 Hz, 1H, CHOH), 2.42 (dt, *J* = 6.8, 6.8 Hz, 2H, COCH=CHCH₂), 2.27 (dt, *J* = 6.8, 6.8 Hz, 2H, COCH=CHCH₂CH₂), 1.26 (d, *J* = 6.6 Hz, 3H CH₃COH).

¹³C NMR (75 MHz, CDCl₃): 190.7, 148.7, 137.6, 135.4, 132.5, 128.5, 128.4, 128.3, 126.1, 68.3, 32.1, 30.5, 23.3.

HRMS (CI) Calcd. for C₁₅H₁₈O₂ (M + H)⁺, 231.1385; Found 231.1384.

FTIR (film): 3412, 2969, 2927, 1668, 1619, 1448, 1288, 971 cm⁻¹.

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¹H NMR (500 MHz, CDCl₃): 7.29–7.26 (m, 2H, Ar–H), 7.20–7.17 (m, 3H, Ar–H), 6.79 (dt, *J* = 15.9, 7.1 Hz, 1H, COCH=CH), 6.10 (d, *J* = 15.9 Hz, 1H, COCH), 5.62 (dt, *J* = 15.6, 7.1 Hz, 1H, HC=CHCHOH), 5.56 (dd, *J* = 15.6, 6.4 Hz, 1H, HC=CHCHOH), 4.27 (dq, *J* = 6.4, 6.4 Hz, 1H, CHOH), 2.94 (t, *J* = 7.5 Hz, 2H, PhCH₂), 2.87 (t, *J* = 7.5 Hz, 2H, CH₂CO), 2.29 (dt, *J* = 7.1, 7.1 Hz, 2H, COCH=CHCH₂), 2.19 (dt, *J* = 7.1, 7.1 Hz, 2H, COCH=CHCH₂CH₂), 1.25 (d, *J* = 6.4 Hz, 3H, CH₃CHOH).

¹³C NMR (75 MHz, CDCl₃): 199.4, 146.4, 141.2, 135.4, 130.5, 128.5, 128.4, 128.3, 126.0, 68.5, 41.6, 31.9, 30.4, 30.0, 23.4.

HRMS (EI) Calcd. for C₁₇H₂₂O₂ (M)⁺, 258.1620; Found 258.1618.

FTIR (film): 3422, 3027, 2969, 2927, 1670, 1628, 1453, 1367, 1063, 974 cm⁻¹.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; 4:1). Spectral data for the major isomer is reported.

¹H NMR (500 MHz, CDCl₃): 7.93–7.91 (m, 2H, Ar–H), 7.57–7.54 (m, 1H, Ar–H), 7.48–7.45 (m, 2H, Ar–H), 7.04 (dt, *J* = 15.4, 7.1 Hz, 1H, COCH=CH), 6.89 (dm, *J* = 15.4 Hz, 1H, COCH), 5.66–5.50 (m, 2H, HC=CHCHOH), 4.28 (dq, *J* = 6.4 Hz, 1H, CHOH), 2.33 (dt, *J* = 7.1, 7.1 Hz, 2H, COCH=CHCH₂), 2.12 (dt, *J* = 7.1, 7.1 Hz, 2H, COCH=CHCH₂CH₂), 1.63 (tt, *J* = 7.1, 7.1 Hz, 2H, CH₂CH₂CH₂), 1.27 (d, *J* = 6.4 Hz, 3H, CH₃CHOH).

¹³C NMR (75 MHz, CDCl₃): 190.3, 149.3, 137.3, 134.9, 132.2, 128.7, 128.0, 127.3, 125.7, 67.8, 31.7, 31.1, 27.1, 23.1.

HRMS (CI) Calcd. for C₁₆H₂₀O₂ (M + H)⁺, 245.1542; Found 245.1545.

FTIR (film): 3429, 2969, 2928, 1668, 1620, 1448, 1288, 971, 695 cm⁻¹.

Compounds **8**, **9** and **11** have been previously reported.³

8

9

11

10

¹H NMR (500 MHz, CDCl₃): 7.29–7.26 (m, 2H, Ar–H), 7.19–7.17 (m, 3H, Ar–H), 6.75–6.73 (br s, 1H, C=CH), 5.83 (ddd, $J = 17.3, 10.3, 7.3$ Hz, 1H, HC=CH₂), 5.01 (dm, $J = 17.1$ Hz, 1H, HC=CHH), 4.96 (dm, $J = 10.3$ Hz, 1H, HC=CHH), 3.65–3.61 (m, 1H, HC–CH=CH₂), 3.03–2.89 (m, 4H, PhCH₂CH₂), 2.57 (dddd, $J = 18.6, 8.1, 8.1, 2.4, 2.4$ Hz, 1H, C=CHCHH), 2.45 (dddd, $J = 18.6, 9.0, 3.7, 3.7, 1.0$ Hz, 1H, C=CHCHH), 2.15 (dddd, $J = 12.9, 9.0, 8.8, 8.8$ Hz, 1H, H₂C=CH–CH–CHH), 1.82 (dddd, $J = 12.9, 12.8, 3.7, 3.7$ Hz, 1H, H₂C=CH–CH–CHH).

¹³C NMR (75 MHz, CDCl₃): 197.4, 147.0, 143.9, 141.4, 139.9, 128.4, 128.3, 126.0, 113.5, 47.0, 41.1, 32.0, 30.3, 30.2.

HRMS (EI) Calcd. for C₁₆H₁₈O (M + H)⁺, 226.1358; Found 226.1355.

FTIR (film): 2930, 1668, 1453, 911 cm⁻¹.

Anal. Calcd. For C₁₆H₁₈O: C, 84.91; H, 8.02. Found: C, 84.60; H, 8.33.

³ Jellerichs, B. G.; Kong, J. -R.; Krische, M. J. *J. Am. Chem. Soc.* **2003**, *125*, 7758.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; >10:1). Spectral data for the major isomer is reported.

¹H NMR (500 MHz, CDCl₃): 6.74–6.72 (br s, 1H, C=CH), 5.48–5.39 (m, 2H, HC=CH), 3.58–3.55 (m, 1H, HC–CH=CH), 2.56 (dddd, *J* = 18.4, 8.3, 8.3, 2.2, 2.2 Hz, 1H, C=CHCHH), 2.44 (dddd, *J* = 18.4, 8.4, 3.7, 3.2, 1.0 Hz, 1H, C=CHCHH), 2.29 (s, 3H, CH₃CO), 2.12 (dddd, *J* = 12.9, 9.0, 9.0, 9.0 Hz, 1H, HC=CH–CH–CHH), 1.78–1.76 (m, 1H, HC=CH–CH–CHH), 1.63 (d, *J* = 4.9 Hz, 3H, HC–CH₃).

¹³C NMR (75 MHz, CDCl₃): 196.3, 148.0, 144.3, 132.5, 124.1, 45.8, 31.9, 31.0, 27.2, 17.8.

HRMS (CI) Calcd. for C₁₀H₁₄O (M + H)⁺, 151.1123; Found 151.1121.

FTIR (film): 2934, 1668, 1612, 1367, 963 cm⁻¹.

Anal. Calcd. For C₁₀H₁₄O: C, 79.96; H, 9.39. Found: C, 79.68; H, 9.46.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; >10:1). Spectral data for the major isomer is reported.

¹H NMR (500 MHz, CDCl₃): 7.78–7.75 (m, 2H, Ar–H), 7.53–7.50 (m, 1H, Ar–H), 7.44–7.41 (m, 2H, Ar–H), 6.47–6.45 (br s, 1H, C=CH), 5.56–5.45 (m, 2H, HC=CH), 3.84–3.78 (m, 1H, HC–CH=CH), 2.66 (dddd, *J* = 19.3, 8.3, 5.9, 2.4, 2.4 Hz, 1H, C=CHCHH), 2.48 (dddd, *J* = 19.3, 9.0, 5.6, 2.7, 2.7 Hz, 1H, C=CHCHH), 2.24 (dddd, *J* = 14.9, 9.0, 5.9, 5.9 Hz, 1H, HC=CH–CH–CHH), 1.82 (dddd, *J* = 14.9, 8.3, 5.6, 5.6 Hz, 1H, HC=CH–CH–CHH), 1.63 (d, *J* = 5.4 Hz, 3H, CH₃).

¹³C NMR (75 MHz, CDCl₃): 193.7, 146.6, 145.5, 138.8, 132.4, 131.9, 128.9, 128.1, 124.9, 47.5, 32.6, 30.7, 17.9.

HRMS (CI) Calcd. for C₁₅H₁₆O (M + H)⁺, 213.1280; Found 213.1276.

FTIR (film): 2934, 1645, 1598, 1447, 960 cm^{-1} .

Anal. Calcd. For $\text{C}_{15}\text{H}_{16}\text{O}$: C, 84.87; H, 7.60. Found: C, 84.63; H, 7.66.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; >10:1). Spectral data for the major isomer is reported.

^1H NMR (500 MHz, CDCl_3): 7.29–7.25 (m, 2H, Ar-H), 7.20–7.16 (m, 3H, Ar-H), 6.71–6.70 (br s, 1H, C=CH), 5.47–5.38 (m, 2H, HC=CH), 3.60–3.54 (m, 1H, HC-CH=CH), 3.00–2.88 (m, 4H, PhCH_2CH_2), 2.56 (dddd, $J = 18.6, 8.3, 8.3, 2.4, 2.4$ Hz, 1H, C=CHCHH), 2.42 (dddd, $J = 18.6, 9.3, 3.9, 3.9, 1.0$ Hz, 1H, C=CHCHH), 2.11 (dddd, $J = 12.9, 9.0, 7.8, 7.8$ Hz, HC=CHCHCHH), 1.76 (dddd, $J = 12.9, 8.3, 3.9, 3.9$ Hz, 1H, HC=CHCHCHH), 1.63 (d, $J = 4.9, 3\text{H}$).

^{13}C NMR (75 MHz, CDCl_3): 197.6, 147.6, 143.4, 141.5, 132.6, 128.4, 125.9, 124.2, 46.1, 41.1, 32.0, 30.9, 30.2, 17.9.

HRMS (EI) Calcd. for $\text{C}_{17}\text{H}_{20}\text{O}$ ($\text{M} + \text{H}$)⁺, 240.1514; Found 240.1512.

FTIR (film): 2933, 1670, 1607, 1453, 963 cm^{-1} .

Anal. Calcd. For $\text{C}_{17}\text{H}_{20}\text{O}$: C, 84.96; H, 8.39. Found: C, 84.57; H, 8.64.

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This compound was isolated as a mixture of alkene geometrical isomers (E:Z; 10:1). Spectral data for the major isomer is reported.

^1H NMR (500 MHz, CDCl_3): 7.70–7.67 (m, 2H, Ar-H), 7.51–7.48 (m, 1H, Ar-H), 7.43–7.40 (m, 2H, Ar-H), 6.50 (t, $J = 3.7$ Hz, 1H, C=CH), 5.48–5.37 (m, 2H, HC=CH), 3.62–3.57 (m, 1H, HC=CHCH), 2.34–2.27 (m, 1H, C=CHCHH), 2.20–2.11 (m, 1H, C=CHCHH), 1.75–1.60 (m, 7H).

^{13}C NMR (75 MHz, CDCl_3): 197.4, 142.5, 140.9, 138.7, 133.0, 131.5, 129.3, 127.9, 125.7, 35.4, 28.2, 26.0, 17.8.

HRMS (EI) Calcd. for $\text{C}_{16}\text{H}_{18}\text{O}$ ($\text{M} + \text{H}$)⁺, 226.1358; Found 226.1353.

FTIR (film): 2933, 2856, 1650, 1578, 1446, 1264, 967, 720 cm^{-1} .

Anal. Calcd. For $\text{C}_{16}\text{H}_{18}\text{O}$: C, 84.91; H, 8.02. Found: C, 84.50; H, 7.63.