Generation of reactive ketenes under flow conditions via Zinc mediated dehalogenation

Andreas Hafner, Steven V. Ley*

Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge, CB2 1EW (UK)

General experimental section. $^1$H-NMR spectra were recorded on a Bruker Avance DPX-400 DRX-500 Cryo or DRX-600 spectrometer with the residual solvent peak as the internal reference ($\text{CDCl}_3 = 7.26 \text{ ppm}$). $^1$H resonances are reported to the nearest 0.01 ppm. $^{13}$C-NMR spectra were recorded on the same spectrometers with the central resonance of the solvent peak as the internal reference ($\text{CDCl}_3 = 77.16 \text{ ppm}$). All $^{13}$C resonances are reported to the nearest 0.1 ppm. The multiplicity of $^1$H signals are indicated as: s = singlet, d = doublet, t = triplet, m = multiplet, br. = broad, or combinations of thereof. Coupling constants ($J$) are quoted in Hz and reported to the nearest 0.1 Hz. Where appropriate, averages of the signals from peaks displaying multiplicity were used to calculate the value of the coupling constant. Infrared spectra were recorded neat on a PerkinElmer Spectrum One FT-IR spectrometer using Universal ATR sampling accessories. Unless stated otherwise, reagents were obtained from commercial sources and used without purification. The removal of solvent under reduced pressure was carried out on a standard rotary evaporator. All the flow reactions were performed using a Vapourtec R2+R4 platform. In-line IR spectroscopy was performed using the Mettler Toledo FlowIR® device.

Procedures

All starting materials were purchased from commercial suppliers and used without further purification unless otherwise stated. Yields refer to isolated compounds estimated to be <95% pure as determined by $^1$H NMR.

General procedure 1 (GP1): In-flow generation of ketenes

A solution of $\alpha$-bromo acyl halide (0.05M) in Et$_2$O was passed through an omnifit glass column$^1$, loaded with a mixture of activated zinc (500 mg) and glass beads (1.00 g) using a Vapourtec R2 platform$^2$. The column output was monitored by using a Mettler Toledo Flow-IR® device$^3$. When freshly activated zinc was used the formation of the corresponding ketene could be directly observed in the IR spectra. When the zinc was not activated enough ketene formation sometimes did not occur directly. However, the formation could be easily initiated by heating the zinc column for a few seconds. Once the reaction was initiated the ketene formation was stable for about 30 min. CAUTION: Although the work was conducted using low ketene concentrations special precautions has to be taken as ketenes are highly toxic. All
work has been done in a fumehood and the outcoming ketene stream was directly quenched by either the respective coupling partner or a NaOH-solution.

**General procedure 2 (GP2): General procedure for the synthesis of β-lactams:**

0.20 mmol of the respective imine was dissolved in 1 mL of Et₂O in a vial. The ketene was synthesised according to GP1 and 6 mL of the outcoming ketene stream (0.05M, 3.00 mmol, 1.50 equiv.) were directly added to the imine solution. The reaction mixture was stirred for another 4 minutes and then quenched by adding 5 mL of sat. K₂CO₃-solution and stirred for another 5 minutes. The organic phase was separated and the aqueous phase was extracted another time with Et₂O. The combined organic phases were dried over MgSO₄ and the solvent removed under reduced pressure. The crude product was purified by flash column chromatography.

4-(4-chlorophenyl)-1-(4-methoxyphenyl)-3-methylazetidin-2-one (6a)
The product was obtained following GP2 as a 1:1.4 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4:1) as a yellowish oil. Rₕ = 0.27 and 0.18 (hexane/ethyl acetate = 4:1). 55.0 mg (91%).

**trans-isomer:** \(^1\)H NMR (400 MHz, CDCl₃) δ 7.34 (d, J = 8.0 Hz, 2H), 7.31–7.13 (m, 4H), 6.85–6.73 (m, 2H), 4.52 (d, J = 2.2 Hz, 1H), 3.73 (s, 3H), 3.07 (dq, J = 7.4, 2.2 Hz, 1H), 1.47 (d, J = 7.4 Hz, 3H) ppm. \(^13\)C NMR (151 MHz, CDCl₃) δ 167.4, 155.9, 136.5, 134.2, 131.1, 129.3, 127.1, 118.1, 114.3, 62.0, 55.4, 55.4, 13.1 ppm. *Analytical data is identical with the literature.*

**cis-isomer:** \(^1\)H NMR (400 MHz, CDCl₃) δ 7.34 (d, J = 8.0 Hz, 2H), 7.30–7.13 (m, 4H), 6.84–6.72 (m, 2H), 5.13 (d, J = 5.8 Hz, 1H), 3.75 (s, 3H), 3.67 (dq, J = 7.6, 5.8 Hz, 1H), 0.87 (d, J = 7.6 Hz, 3H) ppm. \(^13\)C NMR (151 MHz, CDCl₃) δ 167.5, 155.9, 133.9, 133.7, 131.0, 128.9, 128.3, 118.2, 114.3, 57.8, 55.4, 49.2, 9.8 ppm. HRMS (C₁₇H₁₇ClNO₂ [M+H]): calcd. 302.0948; found 302.0958. IR (neat): ν~ = 1738, 1509, 1492, 1385, 1294, 1150, 1089, 1029, 1013, 909, 826, 797, 727 cm⁻¹.

3-methyl-1,4-diphenylazetidin-2-one (6b)
The product was obtained following GP2 as a 1:4.7 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4:1) as a white solid. Rₕ = 0.42 and 0.36 (hexane/ethyl acetate = 4:1). 46.2 mg (97%).

**trans-isomer:** \(^1\)H NMR (400 MHz, CDCl₃) δ 7.43–7.21 (m, 9H), 7.09–6.98 (m, 1H), 4.59 (d, J = 2.4 Hz, 1H), 3.13 (dq, J = 7.4, 2.4 Hz, 1H), 1.49 (d, J = 7.4 Hz, 3H) ppm. \(^13\)C NMR (151 MHz, CDCl₃) δ 168.3, 137.9, 137.8, 129.1, 129.0, 128.4, 125.8, 123.7, 116.9, 62.7, 55.3, 13.1 ppm. *Analytical data is identical with the literature.*

**cis-isomer:** \(^1\)H NMR (400 MHz, CDCl₃) δ 7.38–7.22 (m, 9H), 7.07–7.03 (m, 1H), 5.20 (d, J = 5.9 Hz, 1H), 3.69 (dt, J = 7.6, 5.9 Hz, 1H), 0.89 (d, J = 7.6 Hz, 3H) ppm. \(^13\)C NMR (151 MHz, CDCl₃) δ 168.5, 137.7, 134.9, 129.0, 128.7, 128.1, 126.9, 123.7, 117.1, 58.3, 49.2, 9.7 ppm. *Analytical data is identical with the literature.* HRMS (C₁₆H₁₆NO [M+H]): calcd. 238.1232; found 238.1234. IR (neat): ν~ = 1734, 1597, 1496, 1453, 1384, 1375, 1355, 1156, 1149, 747, 725, 699, 687 cm⁻¹.

1-benzyl-3-methyl-4-phenylazetidin-2-one (6c)
The product was obtained following GP2 as a 1:2.1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4:1) as a colorless oil. Rf = 0.22 and 0.17 (hexane/ethyl acetate = 4:1). 34.3 mg (68%).

trans-isomer: 1H NMR (600 MHz, CDCl3) δ 7.43–7.22 (m, 8H), 7.16–7.14 (m, 2H), 4.85 (d, J = 15.0 Hz, 1H), 3.98 (d, J = 2.0 Hz, 1H), 3.77 (d, J = 15.0 Hz, 1H), 3.08 (dq, J = 7.4, 2.0 Hz, 1H), 1.36 (d, J = 7.4 Hz, 3H) ppm. 13C NMR (151 MHz, CDCl3) δ 170.9, 137.7, 135.7, 128.9, 128.7, 128.4, 128.4, 127.6, 126.4, 62.1, 55.3, 12.9 ppm. cis-isomer: 1H NMR (600 MHz, CDCl3) δ 7.43–7.23 (m, 6H), 7.21–7.17 (m, 4H), 4.92 (d, J = 14.9 Hz, 1H), 4.61 (d, J = 5.6 Hz, 1H), 3.92 (d, J = 14.9 Hz, 1H), 3.51 (dq, J = 7.6, 5.6 Hz, 1H), 0.85 (d, J = 7.6 Hz, 3H) ppm. 13C NMR (151 MHz, CDCl3) δ 171.1, 135.6, 135.3, 128.7, 128.5, 128.4, 128.0, 127.6, 127.2, 57.9, 49.9, 44.4, 9.7 ppm. HRMS (C17H18NO [M+H]+): calcd. 252.1388; found 252.1394. IR (neat): ν = 1742, 1495, 1377, 1364, 1337, 1285, 1276, 1276, 1276, 1276, 970 cm−1. Analytical data is identical with literature.

3,4-dimethyl-1,4-diphenylyzazetidin-2-one (6d)
The product was obtained following GP2 after flash column chromatography (hexane/ethyl acetate = 9:1–4:1) as a white solid. Rf = 0.24 (hexane/ethyl acetate = 4:1). 30.2 mg (70%).

cis-isomer: 1H NMR (400 MHz, CDCl3) δ 7.39–7.26 (m, 5H), 4.77 (d, J = 5.7 Hz, 1H), 3.33 (dq, J = 7.6, 5.7 Hz, 1H), 1.30 (s, 9H), 0.77 (d, J = 7.6 Hz, 3H) ppm. 13C NMR (151 MHz, CDCl3) δ 171.5, 138.1, 128.2, 127.8, 127.4, 57.9, 54.1, 48.1, 28.2, 9.6 ppm. HRMS (C17H18NO [M+H]+): calcd. 218.1545; found 218.1544. IR (neat): ν = 1720, 1455, 1377, 1364, 1337, 1277, 1102, 749, 702 cm−1. Analytical data is identical with the literature.

1-isopropyl-3-methyl-4-phenylyzazetidin-2-one (6f)
The product was obtained following GP2 as a 14.7:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4:1) as a colorless oil. Rf = 0.15 (hexane/ethyl acetate = 4:1). 24.8 mg (61%). NMR were only solved for the major isomer.

cis-isomer: 1H NMR (600 MHz, CDCl3) δ 7.42–7.36 (m, 2H), 7.35–7.30 (m, 1H), 7.29–7.26 (m, 2H), 4.77 (d, J = 5.6 Hz, 1H), 3.77 (hept, J = 6.7 Hz, 1H), 3.41 (qd, J = 7.6, 5.6 Hz, 1H), 1.34 (d, J = 6.7 Hz, 3H), 1.15 (d, J = 6.7 Hz, 3H), 0.82 (d, J = 7.6 Hz, 3H) ppm. 13C NMR (151 MHz, CDCl3) δ 171.3, 136.9, 128.3, 127.9, 127.4, 57.6, 48.8, 45.2, 21.3, 20.4, 9.7 ppm.
HRMS (C₁₃H₁₈NO [M⁺+H]): calcd. 204.1388; found 204.1382. IR (neat): ν = 1736, 1455, 1383, 1366, 1335, 789, 743, 702 cm⁻¹.

3-ethyl-1-isopropyl-4-phenylazetidin-2-one (6g)
The product was obtained following GP2 as a 8.9:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4.1) as a colorless oil. Rᵣ = 0.20 (hexane/ethyl acetate = 4:1). 36.4 mg (84%). NMR were only solved for the major isomer.
cis-isomer: ¹H NMR (600 MHz, CDCl₃) δ 7.38–7.33 (m, 2H), 7.30 (m, 3H), 4.75 (d, J = 5.5 Hz, 1H), 3.74 (hept, 6.8 Hz, 1H), 3.20 (ddd, J = 8.6, 7.4, 5.5 Hz, 1H), 1.51 (d,quin, J =14.1, 7.5, 1H), 1.31 (d, J = 6.8 Hz, 3H), 1.16–1.06 (m, 1H), 1.11 (d, J = 6.8 Hz, 3H), 0.67 (t, J = 7.4 Hz, 3H) ppm. ¹³C NMR (151 MHz, CDCl₃) δ 170.9, 137.1, 128.3, 127.9, 127.5, 57.4, 55.9, 45.1, 21.4, 20.3, 18.7, 11.7 ppm. HRMS (C₁₄H₂₀NO [M⁺]+H): calcd. 218.1545; found 218.1542. IR (neat): ν ≈ 1735, 1455, 1381, 1365, 1323, 782, 730, 702 cm⁻¹.

ethyl-1,4-diphenylazetidin-2-one (6h)
The product was obtained following GP2 as a 1:15.7 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4.1) as a white solid. Rᵣ = 0.49 (hexane/ethyl acetate = 4:1). 49.2 mg (98%). NMR were only solved for the major isomer.
trans-isomer: ¹H NMR (600 MHz, CDCl₃) δ 7.45–7.22 (m, 9H), 7.05 (t, J = 7.4 Hz, 1H), 3.03 (t, J = 8.0 Hz, 1H), 2.08 (s, 3H), 1.98–1.87 (m, 1H), 1.84–1.74 (m, 1H), 1.12 (t, J = 7.4 Hz, 3H) ppm. cis-isomer: ¹H NMR (600 MHz, CDCl₃) δ 7.44–7.31 (m, 9H), 7.04 (t, J = 7.5 Hz, 1H), 3.03 (t, J = 8.0 Hz, 1H), 2.08 (s, 3H), 1.98–1.87 (m, 1H), 1.84–1.74 (m, 1H), 1.12 (t, J = 7.4 Hz, 3H) ppm. ¹³C NMR (151 MHz, CDCl₃) δ 170.9, 137.1, 128.3, 127.9, 127.5, 57.4, 55.9, 45.1, 21.4, 20.3, 18.7, 11.7 ppm. HRMS (C₁₇H₁₈NO [M⁺]+H): calcd. 252.1388; found 252.1386. IR (neat): ν ≈ 1733, 1596, 1491, 1453, 1432, 1425, 1382, 1351, 1342, 1327, 1156, 1117, 907, 730, 698, 688, 658 cm⁻¹. Analytical data is identical with the literature.

3-ethyl-4-methyl-1,4-diphenylazetidin-2-one (6i)
The product was obtained following GP2 as a 1:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4.1) as a slightly yellowish oil. Rᵣ = 0.53 and 0.47 (hexane/ethyl acetate = 4:1). 44.3 mg (84%). NMR were only solved for the major isomer.
trans-isomer: ¹H NMR (600 MHz, CDCl₃) δ 7.45–7.31 (m, 9H), 7.04 (t, J = 7.5 Hz, 1H), 3.03 (t, J = 8.0 Hz, 1H), 2.08 (s, 3H), 1.98–1.87 (m, 1H), 1.84–1.74 (m, 1H), 1.12 (t, J = 7.4 Hz, 3H) ppm. cis-isomer: ¹H NMR (600 MHz, CDCl₃) δ 7.44–7.22 (m, 9H), 7.05 (t, J = 7.5 Hz, 1H), 3.11 (t, J = 8.0 Hz, 1H), 1.96 (s, 3H), 1.43–1.34 (m, 1H), 1.14–1.06 (m, 1H), 0.85 (t, J = 7.4 Hz, 3H). ¹³C NMR (151 MHz, CDCl₃, cis/trans mixture) δ 168.2, 167.9, 143.2, 138.8, 137.2, 137.2, 129.0, 128.9, 128.5, 127.7, 127.6, 126.7, 124.8, 123.4, 65.7, 62.8, 64.4, 63.5, 23.9, 19.5, 18.6, 18.2, 12.5, 11.9 ppm. HRMS (C₁₈H₂₀NO [M⁺]+H): calcd. 266.1545; found 266.1547. IR (neat): ν ≈ 1737, 1599, 1495, 1446, 1371, 1343, 1205, 753, 732, 692 cm⁻¹.

3-butyl-4-(4-chlorophenyl)-1-(4-methoxyphenyl)azetidin-2-one (6j)
The product was obtained following GP2 as a 1.2:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4.1) as a yellowish oil. Rᵣ = 0.54 and 0.45 (hexane/ethyl acetate = 4:1). 49.5 mg (72%).
cis-isomer: $^1$H NMR (600 MHz, CDCl$_3$) δ 7.38–7.31 (m, 2H), 7.23–7.15 (m, 4H), 6.81–6.75 (m, 2H), 5.11 (d, $J = 5.7$ Hz, 1H), 3.74 (s, 3H), 3.52 (td, $J = 7.9, 5.7$ Hz, 1H), 1.53–1.04 (m, 6H), 0.74 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) δ 137.3, 22.4, 25.1, 29.3, 54.7, 55.4, 60.8, 114.3, 118.3, 128.6, 128.9, 131.0, 133.8, 134.0, 155.9, 167.0 ppm. trans-isomer: $^1$H NMR (600 MHz, CDCl$_3$) δ 7.37–7.31 (m, 2H), 7.30–7.26 (m, 2H), 7.22–7.17 (m, 2H), 6.81–6.76 (m, 2H), 4.59 (d, $J = 2.2$ Hz, 1H), 3.73 (s, 3H), 3.03 (ddd, $J = 8.7, 6.0, 2.2$ Hz, 1H), 1.98–1.91 (m, 1H), 1.87–1.76 (m, 1H), 1.50–1.07 (m, 4H), 0.91 (t, $J = 7.3$ Hz, 3H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) δ 13.8, 22.6, 28.5, 29.3, 55.4, 57.8, 60.6, 114.3, 118.1, 127.2, 129.4, 131.2, 134.1, 136.9, 155.9, 167.3 ppm. HRMS (C$_{20}$H$_{25}$ClNO$_2$ [M$^+$+H$^+$]): calcd. 344.1417; found 344.1409. IR (neat): $\nu^\prime = 1738, 1509, 1492, 1385, 1297, 1243, 1178, 1150, 1111, 1089, 1031, 1013, 825, 802$ cm$^{-1}$.

3-buty1-4-methyl-1,4-diphenylazetidin-2-one (6k)

The product was obtained following GP2 as a 1:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 9:1–4:1) as a colorless oil. $R_f = 0.62$ and 0.54 (hexane/ethyl acetate = 4:1). 44.9 mg (77%).

cis-isomer: $^1$H NMR (600 MHz, CDCl$_3$) δ 7.42–7.28 (m, 7H), 7.28–7.19 (m, 2H), 7.04 (t, $J = 7.2$ Hz, 1H), 3.16 (t, $J = 7.6$ Hz, 1H), 1.94 (s, 3H), 1.62–1.50 (m, 1H), 1.43–0.99 (m, 5H), 0.72 (t, $J = 7.0$ Hz, 3H) ppm. trans-isomer: $^1$H NMR (600 MHz, CDCl$_3$) δ 7.42–7.28 (m, 7H), 7.27–7.21 (m, 2H), 7.03 (t, $J = 7.2$ Hz, 1H), 3.08 (t, $J = 7.9$ Hz, 1H), 2.07 (s, 3H), 1.91–1.80 (m, 1H), 1.74 (dd, $J = 7.9, 4.8$ Hz, 1H), 1.43–0.99 (m, 4H), 0.92 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$), cis/trans mixture δ 168.3, 167.9, 143.2, 138.8, 137.2, 137.2, 129.0, 128.9, 128.5, 127.7, 127.6, 126.7, 124.8, 123.4, 123.4, 117.7, 117.6, 64.8, 64.2, 63.5, 62.8, 30.1, 29.4, 25.6, 24.9, 23.9, 22.8, 22.4, 18.3, 13.9, 13.7 ppm. HRMS (C$_{20}$H$_{24}$NO [M$^+$+H$^+$]): calcd. 294.1858; found 294.1863. IR (neat): $\nu^\prime = 1740, 1600, 1495, 1446, 1374, 1362, 1330, 1266, 1201, 1028, 752, 692$ cm$^{-1}$.

1,3,4-triphenylazetidin-2-one (6l)

The product was obtained following GP2 as a 1:7.8 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 15.1–5.1) as a slightly yellow solid. $R_f = 0.49$ (hexane/ethyl acetate = 4:1). 46.3 mg (77%). NMR were only solved for the major isomer.

trans-isomer: $^1$H NMR (600 MHz, CDCl$_3$) δ 7.48–7.26 (m, 13H), 7.16–7.04 (m, 2H), 4.99 (d, $J = 2.6$ Hz, 1H), 4.31 (d, $J = 2.6$ Hz, 1H). $^{13}$C NMR (151 MHz, CDCl$_3$) δ 165.6, 137.5, 137.4, 134.7, 129.4, 129.1, 129.0, 128.6, 127.9, 127.4, 125.9, 124.0, 117.2, 65.1, 63.7 ppm. HRMS (C$_{21}$H$_{18}$NO [M$^+$+H$^+$]): calcd. 300.1388; found 300.1394. IR (neat): $\nu^\prime = 1736, 1597, 1495, 1452, 1383, 1356, 1148, 747, 698$ cm$^{-1}$.

4-(4-chlorophenyl)-1-(4-methoxyphenyl)-3-phenylazetidin-2-one (6m)

The product was obtained following GP2 as a 5.6:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 15.1–5.1) as a slightly brownish solid. $R_f = 0.22$ (hexane/ethyl acetate = 4:1). 60.8 mg (84%).

$^1$H NMR (600 MHz, CDCl$_3$) δ 7.36–7.32 (m, 2H), 7.16–7.05 (m, 7H), 7.01 (d, $J = 8.1$ Hz, 2H), 6.84 (d, $J = 8.4$ Hz, 2H), 5.40 (d, $J = 5.9$ Hz, 1H), 5.01 (d, $J = 5.9$ Hz, 1H), 3.78 (s, 3H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) δ 164.7, 156.2, 133.6, 133.2, 131.9, 131.0, 129.5, 128.8,
128.5, 128.2, 127.4, 118.4, 114.3, 60.3, 59.7, 55.4 ppm. HRMS (C\textsubscript{22}H\textsubscript{19}ClNO\subscript{2} [M\textsuperscript{+}+H]): calcd. 364.1104; found 364.1116. IR (neat): υ\textasciitilde = 1744, 1512, 1493, 1386, 1247, 827, 698 cm\textsuperscript{-1}.

3-(2-bromoethyl)-1,4-diphenylazetidin-2-one (6n)
The product was obtained following GP2 as a 1:5.6 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 16.1–5.1) as a white solid. R\textsubscript{f} = 0.48 (hexane/ethyl acetate = 4:1). 41.1 mg (63%).

Trans-isomer: \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}) δ 7.43–7.23 (m, 9H), 7.10–7.04 (m, 1H), 4.78 (d, J = 2.4 Hz, 1H), 3.66–3.53 (m, 2H), 3.30 (ddd, J = 8.5, 7.1, 2.4 Hz, 1H), 2.62–2.55 (m, 1H), 2.47–2.39 (m, 1H) ppm. cis-isomer: \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}) δ 7.45–7.22 (m, 9H), 7.10–7.03 (m, 1H), 5.27 (d, J = 5.9 Hz, 1H), 3.88 (td, J = 7.8, 5.9 Hz, 1H), 3.40–3.35 (m, 1H), 3.28–3.23 (m, 1H), 2.09–1.99 (m, 1H), 1.74–1.66 (m, 1H) ppm. \textsuperscript{13}C NMR (151 MHz, CDCl\textsubscript{3}) δ 166.4, 137.5, 137.3, 129.2, 129.0, 128.6, 125.9, 123.9, 116.9, 60.9, 58.9, 32.3, 29.8 ppm. HRMS (C\textsubscript{17}H\textsubscript{17}BrNO [M\textsuperscript{+}+H]): calcd. 330.0494; found 330.0499. IR (neat): υ\textasciitilde = 1732, 1593, 1491, 1455, 1356, 1133, 750, 698 cm\textsuperscript{-1}.

3-(2-bromoethyl)-4-methyl-1,4-diphenylazetidin-2-one (6o)
The product was obtained following GP2 as a 1:1 mixture (cis:trans) after flash column chromatography (hexane/ethyl acetate = 16.1–5.1) as a colorless oil. R\textsubscript{f} = 0.49 and 0.42 (hexane/ethyl acetate = 4:1). 62.0 mg (90%).

Trans-isomer: \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}) δ 7.45–7.22 (m, 9H), 7.06 (t, J = 7.5 Hz, 1H), 3.56–3.48 (m, 2H), 3.31–3.26 (m, 1H), 2.53–2.42 (m, 1H), 2.34–2.24 (m, 1H), 2.12 (s, 3H) ppm. cis-isomer: \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}) δ 7.45–7.22 (m, 9H), 7.06 (t, J = 7.5 Hz, 1H), 3.68–3.63 (m, 1H), 3.40–3.35 (m, 1H), 3.27–3.23 (m, 1H), 1.97 (s, 3H), 1.94–1.84 (m, 1H), 1.64–1.56 (m, 1H) ppm. \textsuperscript{13}C NMR (151 MHz, CDCl\textsubscript{3}, cis/trans mixture) δ 18.6, 23.7, 29.0, 29.3, 30.2, 30.6, 60.3, 62.2, 63.2, 64.5, 117.6, 117.6, 123.7, 123.7, 124.8, 126.4, 127.9, 128.0, 128.8, 129.0, 129.0, 129.1, 136.8, 136.8, 138.3, 142.2, 166.5, 166.8 ppm. HRMS (C\textsubscript{18}H\textsubscript{19}BrNO [M\textsuperscript{+}+H]): calcd. 344.0650; found 344.0645. IR (neat): υ\textasciitilde = 1737, 1599, 1495, 1446, 1374, 1346, 1273, 1257, 1208, 906, 753, 730, 691 cm\textsuperscript{-1}.
References

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(2) http://www.vapourtec.co.uk/products/rsrieressystem
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