Supporting information

Microfluidic synthesis of α-ketoesters via oxidative coupling of acetophenones with alcohols under metal-free conditions

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General experimental

All general reagents and solvents were commercially available and used as received. $^1$H and $^{13}$C NMR spectra were measured on magnet system 400’54 ascend instrument purchased from Bruker Biospin AG. Chemical data for protons are reported in parts per million (ppm) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent (CDCl$_3$, 7.26 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = penta, dd = doublet of doublets, dt = doublet of triplets, ddt = doublet of doublet of triplets, dtd = doublet of triplet of doublets, m = multiplet, br = broad), coupling constant (J) in Hertz (Hz), and integration. $^{13}$C NMR were recorded at 125 MHz or 100 MHz and chemical data for carbons are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the solvent. Melting points of compounds were recorded with BUCHI (B-545) instrument. Column chromatography was generally performed on Silicycle silica gel (200-300 mesh). Analytical thin-layer chromatography (TLC) was performed on 0.2 mm coated silica gel plates (HSGF 254) and visualized the course of the reactions using a UV light (254 nm or 365 nm).

Microfluidic chip reactor set-ups

Reactions are performed in a Labtrix® Start R2.2 system (Chemtrix BV, NL). This commercially available microreactor system can be fitted with different glass chip reactors, of which in this project a 10.0 µL microreactor chip (Chemtrix 3223 reactor, 3 inlets) was employed. This reactor chip employs staggered oriented ridge (SOR-2) static micromixers to assure fast mixing. Reaction temperatures are controlled via a MTTC1410 temperature controller (Melcor Thermal Solutions, temperature range –20 to 195°C), while the reactor pressure was maintained at 20 bar backpressure via a preset back pressure regulator (Upchurch Scientific). Reactant solutions are injected into the reactor via 1 mL gastight syringes (SGE). Flowrates vary between 0.1 and 40 µL/min, and are controlled via syringe pumps (Chemyx).

Figure 1 Microfluidic chip reactor for the preparation of α-ketoesters
Experimental procedures

2.0 mmol acetophenones 1, 8.0 mmol alcohols 2, 0.8 mmol iodine, and 3.0 mmol DBU were dissolved in 3 ml DMF, which was extracted to Syringe A. 8 mmol TBHP (70 wt% in water, 4 eq.) were dissolved in 3 ml DMF, which was extracted to Syringe B. The flow rate of Syringe A and B were both 7.5 μl/min and the residence time was 40s. The temperature of the chip was set in 110 °C. The outflow of the reaction mixture was collected, then quenched with saturated Na₂S₂O₃ solution and extracted with ethyl acetate. The organic layer was dried over anhydrous sodium sulfate and solvent was removed under vacuum. And the crude product was purified by flash chromatography on silica gel by gradient elution with ethyl acetate in petroleum (hexane/ethyl acetate 50:1), affording the desired product 3 in good yields (66-90%).
Optimization studies in Batch

![Chemical Reaction Diagram]

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Reaction conditions: 0.5 mmol 1a, 2.0 mmol 2a, promoter, 2.0 mmol oxidant and 0.75 mmol base were solved in 3 ml DMF and stirred at 90 ℃ for 10h in the sealed tube. Yields of the isolated product 3aa. No reaction.
Analytical data of products 3

**Product 3aa:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.83 – 7.72 (m, 2H), 7.58 – 7.53 (m, 1H), 7.40 – 7.35 (m, 2H), 7.27 – 7.17 (m, 5H), 4.54 (t, $J$ = 7.0 Hz, 2H), 3.01 (t, $J$ = 7.0 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.41, 163.84, 137.06, 135.01, 132.41, 130.15, 129.14, 128.97, 128.81, 126.99, 66.54, 35.05; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{14}$O$_3$ 277.0835 found 277.0886.

**Product 3ba:** Light yellow wax; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.26 – 8.13 (m, 2H), 8.13 – 8.02 (m, 2H), 7.28 – 7.17 (m, 5H), 4.51 (t, $J$ = 6.9 Hz, 2H), 3.03 (t, $J$ = 6.9 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 164.72, 150.64, 137.56, 135.76, 130.81, 129.04, 128.79, 126.94, 123.68, 66.45, 35.23; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$NO$_5$ 322.0686 found 322.0691.

**Product 3ca:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.81 (dd, $J$ = 8.9, 5.4 Hz, 2H), 7.28 – 7.17 (m, 5H), 7.04 (t, $J$ = 8.6 Hz, 2H), 4.55 (t, $J$ = 6.9 Hz, 2H), 3.02 (t, $J$ = 6.9 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 184.60, 168.17, 165.61, 163.45, 137.04, 133.14, 133.04, 129.16, 128.85, 127.04, 116.46, 116.24, 66.63, 35.05; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$O$_3$F 295.0741 found 295.0742.

**Product 3da:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.80 (dd, $J$ = 8.9, 5.4 Hz, 2H), 7.22 (dt, $J$ = 23.2, 7.5 Hz, 5H), 7.03 (t, $J$ = 8.6 Hz, 2H), 4.54 (t, $J$ = 6.9 Hz, 2H), 3.01 (t, $J$ = 6.9 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 184.93, 163.23, 141.70, 136.99, 131.53, 130.83, 129.37, 129.14,
Product 3ea: Light yellow wax; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (dd, $J$ = 6.8, 1.8 Hz, 2H), 7.31-7.24 (m, 5H), 6.91 (d, $J$ = 8.8 Hz, 2H), 4.60 (t, $J$ = 7.0 Hz, 2H), 3.87 (s, 3H), 3.08 (t, $J$ = 7.0 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 184.7, 164.9, 164.0, 137.0, 132.5, 129.0, 128.6, 126.7, 125.3, 114.1, 66.2, 55.6, 34.9; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$O$_3$Cl 311.0445 found 311.0477.

Product 3fa: Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.67 (d, $J$ = 8.1 Hz, 2H), 7.24 – 7.14 (m, 7H), 4.51 (t, $J$ = 6.9 Hz, 2H), 2.99 (t, $J$ = 7.0 Hz, 2H), 2.33 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.04, 164.02, 146.32, 137.09, 130.24, 129.67, 129.10, 128.76, 126.93, 66.40, 35.03, 22.00; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{17}$H$_{16}$O$_3$ 291.0992 found 291.0988.

Product 3ga: Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.62 – 7.51 (m, 2H), 7.35 (d, $J$ = 7.6 Hz, 1H), 7.26 – 7.13 (m, 6H), 4.52 (t, $J$ = 7.0 Hz, 2H), 2.99 (t, $J$ = 7.0 Hz, 2H), 2.29 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.54, 163.91, 138.82, 137.00, 135.77, 132.36, 130.27, 129.01, 128.76, 128.69, 127.41, 126.87, 66.38, 34.97, 21.27; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{17}$H$_{16}$O$_3$ 291.0992 found 291.0998.

Product 3ha: Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.28 (s, 1H), 7.88 (d, $J$ = 8.7 Hz, 1H), 7.83 – 7.75 (m, 3H), 7.55 (t, $J$ = 7.5 Hz, 1H), 7.47 (t, $J$ = 7.4 Hz, 1H), 7.26-7.15 (m, 5H), 4.59 (t, $J$ =
7.0 Hz, 2H), 3.04 (t, J = 6.9 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.31, 163.94, 137.17, 136.49, 133.65, 132.36, 130.18, 129.84, 129.69, 129.15, 129.04, 128.83, 128.02, 127.22, 127.02, 124.06, 66.62, 35.12; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{20}$H$_{16}$O$_3$ 327.0992 found 327.1008.

![3ia](image)

**Product 3ia:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.86 (dd, J = 3.8, 1.2 Hz, 1H), 7.74 – 7.67 (m, 1H), 7.26 – 7.14 (m, 5H), 7.06 (t, J = 4.4 Hz, 1H), 4.50 (t, J = 7.1 Hz, 2H), 3.02 (t, J = 7.1 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 176.41, 161.69, 139.19, 137.55, 137.39, 129.12, 128.80, 128.77, 126.99, 67.03, 34.98; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{14}$H$_{13}$O$_3$S 283.0399 found 283.0391.

![3ab](image)

**Product 3ab:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.76 (d, J = 7.3 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.39 (t, J = 7.9 Hz, 2H), 7.20 (t, J = 6.7 Hz, 2H), 7.11 (d, J = 8.3 Hz, 2H), 4.52 (t, J = 6.8 Hz, 2H), 2.98 (t, J = 6.8 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.27, 163.74, 135.62, 135.11, 132.90, 132.38, 130.52, 130.11, 129.00, 128.95, 66.13, 34.44; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$O$_3$Cl 311.0445 found 311.0454.

![3ac](image)

**Product 3ac:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.89 – 7.78 (m, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.40 (t, J = 7.8 Hz, 2H), 7.33 – 7.28 (m, 1H), 7.22 – 7.18 (m, 1H), 7.16 – 7.08 (m, 2H), 4.56 (t, J = 6.9 Hz, 2H), 3.16 (t, J = 6.9 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.20, 163.68, 135.02, 134.73, 134.37, 132.48, 131.48, 130.16, 129.84, 128.97, 128.59, 127.14, 64.86, 32.90; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$O$_3$Cl 311.0445 found 311.0460.

![3ad](image)

**Product 3ad:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.80 – 7.68 (m, 2H), 7.48 (t, J = 7.4 Hz, 1H), 7.37 (t, J = 7.8 Hz, 2H), 7.34 – 7.30 (m, 1H), 7.39 – 7.16 (m, 1H), 7.16 – 7.08 (m, 2H), 4.56 (t, J = 6.9 Hz, 2H), 3.16 (t, J = 6.9 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.20, 163.68, 135.02, 134.73, 134.37, 132.48, 131.48, 130.16, 129.84, 128.97, 128.59, 127.14, 64.86, 32.90; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$O$_3$Cl 311.0445 found 311.0460.
**Product 3ad:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.90 – 7.74 (m, 2H), 7.69 – 7.61 (m, 1H), 7.53 – 7.38 (m, 4H), 7.19 – 7.05 (m, 2H), 4.60 (ddd, $J = 9.5$, 5.9, 2.4 Hz, 2H), 3.04 (td, $J = 6.6$, 3.5 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.27, 163.75, 136.15, 135.12, 132.39, 131.93, 130.92, 130.13, 129.02, 120.96, 66.05, 34.5; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{13}$O$_3$Br 354.9940 found 354.9964.

![Chemical Structure](image)

**Product 3ae:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.91 (t, $J = 6.2$ Hz, 2H), 7.65 (t, $J = 6.9$ Hz, 1H), 7.48 (t, $J = 7.5$ Hz, 2H), 7.17 (dd, $J = 12.4$, 5.1 Hz, 4H), 4.59 (t, $J = 7.0$ Hz, 2H), 3.12 (t, $J = 7.2$ Hz, 2H), 2.38 (d, $J = 4.6$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.34, 163.79, 136.68, 135.08, 135.00, 132.50, 130.66, 130.18, 129.81, 128.98, 127.17, 126.38, 65.71, 32.35, 19.50; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{17}$H$_{16}$O$_3$ 291.0992 found 291.1001.

![Chemical Structure](image)

**Product 3af:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.87 (dd, $J = 8.3$, 1.1 Hz, 2H), 7.68 – 7.58 (m, 1H), 7.46 (t, $J = 7.8$ Hz, 2H), 7.24 – 7.10 (m, 2H), 6.94 – 6.78 (m, 2H), 4.59 (t, $J = 7.0$ Hz, 2H), 3.80 (s, 3H), 3.03 (t, $J = 7.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.44, 163.86, 158.66, 134.98, 132.46, 130.14, 129.04, 128.94, 114.20, 66.72, 55.36, 34.19; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{17}$H$_{16}$O$_4$ 307.0941 found 307.0949.

![Chemical Structure](image)

**Product 3ag:** Light yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.99 – 7.87 (m, 2H), 7.68 – 7.62 (m, 1H), 7.49 (td, $J = 7.6$, 1.7 Hz, 2H), 7.19 (dd, $J = 5.1$, 1.2 Hz, 1H), 7.01 – 6.88 (m, 2H), 4.62 (t, $J = 6.8$ Hz, 2H), 3.31 (t, $J = 6.7$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 186.22, 163.70, 138.98, 135.04, 132.44, 130.17, 128.99, 127.18, 126.14, 124.43, 66.21, 29.22; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{14}$H$_{12}$O$_3$S 283.0399 found 283.0417.
**Product 3ah:** Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.98 (d, $J = 7.2$ Hz, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.53 – 7.33 (m, 7H), 5.42 (s, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.17, 163.77, 135.06, 134.67, 132.56, 130.15, 128.94, 128.87, 67.88; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{15}$H$_{12}$O$_3$ 263.0679 found 263.0685.

**Product 3ai:** Light yellow solid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.97 (dd, $J = 13.8$, 6.5 Hz, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.8$ Hz, 2H), 7.47 – 7.29 (m, 4H), 5.37 (s, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 185.92, 163.56, 135.17, 134.94, 133.18, 132.48, 130.15, 129.10, 129.07, 66.99; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{15}$H$_{11}$O$_3$Cl 297.0829 found 297.0300.

**Product 3aj:** Light yellow solid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.02 – 7.89 (m, 2H), 7.68 – 7.59 (m, 1H), 7.53 – 7.44 (m, 2H), 7.33 (t, $J = 10.6$ Hz, 2H), 7.20 (d, $J = 7.9$ Hz, 2H), 5.38 (s, 2H), 2.37 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.27, 163.85, 138.91, 135.03, 132.61, 131.69, 130.16, 129.55, 129.02, 128.91, 67.92, 21.38; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{16}$H$_{14}$O$_3$ 277.0835 found 277.0845.

**Product 3ak:** Light yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.16 – 7.76 (m, 2H), 7.74 – 7.56 (m, 1H), 7.54 – 7.40 (m, 2H), 5.09 (dd, $J = 13.2$, 8.9, 4.0 Hz, 1H), 1.99 (d, $J = 4.1$ Hz, 2H), 1.78 (dd, $J = 8.7$, 4.2 Hz, 2H), 1.65 – 1.51 (m, 3H), 1.47 – 1.27 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.94, 163.80, 134.90, 132.71, 130.07, 129.00, 75.57, 31.57, 25.30, 23.76; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{14}$H$_{10}$O$_3$ 255.0992 found 255.0998.
**Product 3al:** Light yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.09 – 7.93 (m, 2H), 7.66 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.8$ Hz, 2H), 4.45 (q, $J = 7.1$ Hz, 2H), 1.42 (t, $J = 7.1$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.56, 163.96, 135.02, 132.62, 130.15, 129.02, 62.46, 14.24; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{10}$H$_{10}$O$_3$ 201.0522 found 201.0523.

**Product 3am:** Colourless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.92 (d, $J = 8.2$ Hz, 2H), 7.58 (t, $J = 7.8$ Hz, 1H), 7.44 (t, $J = 7.7$ Hz, 2H), 4.32 (t, $J = 6.7$ Hz, 2H), 1.73 – 1.64 (m, 2H), 1.43 – 1.32 (m, 2H), 0.89 (t, $J = 7.4$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.62, 164.13, 134.99, 132.63, 130.11, 129.01, 66.21, 30.59, 19.15, 13.74; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{12}$H$_{14}$O$_3$ 229.0835 found 229.0841.

**Product 3an:** Colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.92 (d, $J = 7.4$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 4.78 (s, 1H), 4.72 (s, 1H), 4.44 (t, $J = 6.9$ Hz, 2H), 2.42 (t, $J = 6.8$ Hz, 2H), 1.72 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 186.43, 163.92, 141.01, 135.01, 132.55, 130.16, 128.98, 113.13, 64.45, 36.66, 22.54; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{13}$H$_{14}$O$_3$ 241.0835 found 241.0834.

**Product 3ao:** Light yellow solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.00 (d, $J = 7.3$ Hz, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.8$ Hz, 2H), 5.45 (d, $J = 4.3$ Hz, 1H), 4.94 (ddd, $J = 16.4$, 10.5, 4.7 Hz, 1H), 2.50 (d, $J = 7.7$ Hz, 2H), 2.05 – 1.76 (m, 6H), 1.61 – 1.46 (m, 6H), 1.40 – 1.10 (m, 11H), 1.06 – 0.97 (m, 6H), 0.93 (d, $J = 6.5$ Hz, 3H), 0.87 (dd, $J = 6.6$, 2.1 Hz, 6H), 0.69 (s, 3H); $^{13}$C NMR (125MHz, CDCl$_3$) $\delta$ 186.68, 163.55, 139.14, 134.81, 132.68, 130.04, 128.92, 123.42, 76.65, 56.78, 56.26, 50.13, 42.41, 39.81, 39.60, 37.97, 37.03, 36.69, 36.27, 35.85, 32.00, 31.94, 28.28, 28.07, 27.73, 24.35, 23.91, 22.86, 22.61, 21.13, 19.34, 18.79, 11.93; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{35}$H$_{50}$O$_3$ 541.3652 found 541.3654.
$^1$H NMR and $^{13}$C NMR spectra of products 3

Product 3aa
Product 3da

Chemical structures and spectral data are shown for Product 3da, including proton nuclear magnetic resonance (1H NMR) spectra with peaks at various ppm values.

Additional spectral data includes carbon (13C NMR) and chlorine (Cl) peaks at specific ppm values.

The chemical structure of Product 3da is also depicted with relevant atoms and bonds.
Product 3ga

1H NMR (500 MHz, CDCl3) δ (ppm):
- 7.60
- 7.56
- 7.35
- 7.22
- 7.17

- 6.09
- 2.00
- 1.07
- 2.00

H2C₆H₃-CH2-CH2-CH2-CH₃

1H NMR (500 MHz, CDCl3) δ (ppm):
- 8.0
- 7.5
- 7.0
- 6.5
- 6.0
- 5.5
- 5.0
- 4.5
- 4.0
- 3.5
- 3.0
- 2.5
- 2.0
- 1.5
- 1.0
- 0.5
- 0.0
Product 3ae
Product 3af
Product 3ag
Product 3ah

$$\text{f1 (ppm)}$$

$$\begin{align*}
7.3 & \quad 7.4 \\
7.5 & \quad 7.6 \\
7.7 & \quad 7.8 \\
7.9 & \quad 8.0 \\
\end{align*}$$

$$\begin{align*}
5.4 & \\
\end{align*}$$

$$\text{f1 (ppm)}$$

$$\begin{align*}
128.73 & \quad 128.87 \\
128.94 & \quad 129.02 \\
130.15 & \quad 132.56 \\
134.67 & \quad 135.06 \\
\end{align*}$$

$$\text{f1 (ppm)}$$

$$\begin{align*}
67.88 & \quad 76.84 \\
77.16 & \quad 77.48 \\
\end{align*}$$
Product 3aj

O
O
C
H
3

O
O
C
H
3
Product 3ak

![Chemical Structure](image)

![NMR Spectrum](image)
Product 3al

\[
\begin{align*}
\text{O} & \quad \text{O} \\
\text{C} & \quad \text{H}_3
\end{align*}
\]

\[
\begin{align*}
\text{f1 (ppm)} & \quad 2.04 \\
\text{f1 (ppm)} & \quad 1.40 \\
\text{f1 (ppm)} & \quad 1.42 \\
\text{f1 (ppm)} & \quad 1.44 \\
\text{f1 (ppm)} & \quad 4.42 \\
\text{f1 (ppm)} & \quad 4.44 \\
\text{f1 (ppm)} & \quad 4.46 \\
\text{f1 (ppm)} & \quad 4.48
\end{align*}
\]

\[
\begin{align*}
\text{f1 (ppm)} & \quad 186.1 \\
\text{f1 (ppm)} & \quad 163.1 \\
\text{f1 (ppm)} & \quad 135.1 \\
\text{f1 (ppm)} & \quad 132.1 \\
\text{f1 (ppm)} & \quad 130.1 \\
\text{f1 (ppm)} & \quad 129.1 \\
\text{f1 (ppm)} & \quad 77.41 \\
\text{f1 (ppm)} & \quad 77.16 \\
\text{f1 (ppm)} & \quad 76.84 \\
\text{f1 (ppm)} & \quad 62.41
\end{align*}
\]

\[
\begin{align*}
\text{f1 (ppm)} & \quad 14.22
\end{align*}
\]