TMEDAO₂ Facilitates Atom Economical/Open Atmosphere Ley–Griffith (TPAP) Tandem Oxidation-Wittig Reactions

Supplementary Information

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1. General Experimental
Reactions were monitored using silica-60 F_{254} TLC plates, visualised first under UV light then developed using a KMnO\textsubscript{4} dip. Column chromatography was undertaken using silica gel (flash silica gel, 230-400 mesh) using distilled solvents. All reagents and solvents were purified prior to use according to literature methods (D. D. Perin, W. L. F. Armarego, Purification of Laboratory Chemicals, 3rd ed., Pergamon Press, Oxford, England, 1988.) tetrakis(tripropyl)ammonium perruthenate (97%) and fine chemicals were purchased from Sigma-Aldrich or Precious Metals Online. Dry DCM was freshly distilled from a CaH\textsubscript{2} still. NMR spectra were recorded using either a Bruker AV300 (300 MHz, 75 MHz), AV400 (400 MHz, 100 MHz) or AV500 (500 MHz, 125 MHz) instrument, with all data being processed using MestReNova software, version 9.1.0. Chemical shifts are given in parts per million (ppm) and referenced according to solvent; CDCl\textsubscript{3} (\textsuperscript{1}H \textsuperscript{δ}: 7.26 ppm; \textsuperscript{13}C \textsuperscript{δ}: 77.0 ppm), D\textsubscript{2}O (\textsuperscript{1}H \textsuperscript{δ}: 4.79 ppm, internal reference: dioxane \textsuperscript{13}C \textsuperscript{δ}: 67.1 ppm), d\textsubscript{6}-DMSO (\textsuperscript{1}H \textsuperscript{δ}: 2.50 ppm; \textsuperscript{13}C \textsuperscript{δ}: 39.5 ppm). Coupling constants (J) are given in Hz. Gas Chromatography-Mass Spectrometry (GC-MS) was recorded using a GCMS-QP2010 Ultra machine with autosampler and analysed using GCMSsolution, v4.20. Infra-red spectra were measured using a Perkin Elmer FT-IR spectrometer (Spectrum 2000). High resolution electrospray ionisation (HRESIMS) accurate mass measurements were recorded in positive mode on a Bruker MicroOTOF-Q (quadrupole-time of flight) instrument with a Bruker ESI source using sodium formate as a reference calibrant. Microanalyses were performed by the University of Queensland Microanalytical Service.

2. Synthesis of Reagents and Starting Material
2.1 TMEDAO\textsubscript{2}•4H\textsubscript{2}O (6)

\[
\begin{align*}
\text{H}_2\text{O}_2 (5 \text{ eq.}) & \quad \text{0 °C - r.t., 0.5 h} \\
\text{TMEDA} (2.7 \text{ mL, 18 mmol}) & \quad \text{(6)}
\end{align*}
\]

Hydrogen peroxide (9.0 mL, 90 mmol, 30% ) was added to a wide necked round bottom flask and cooled to 0°C. TMEDA (2.7 mL, 18 mmol) was added drop-wise over 5 minutes and then stirred at 0°C for 30 minutes. The solution was allowed to warm to room temperature before being concentrated under reduced pressure (60 °C, 20 Torr, behind a blast shield). Water (3.0 mL) was added and the reaction mixture again concentrated under reduced pressure, followed by drying under high vacuum, providing TMEDAO\textsubscript{2}•4H\textsubscript{2}O (6, 3.805 g, 95%) as a white solid. TMEDAO\textsubscript{2}•4H\textsubscript{2}O (6) was stored in a desiccator. IR and \textsuperscript{1}H NMR data matched that reported by Gelbard et al.\textsuperscript{1}

\textsuperscript{1}H NMR (300 MHz, D\textsubscript{2}O): \textsuperscript{δ} (ppm): 3.90(s, 4H), 3.30(s, 12H) \textsuperscript{13}C NMR (100 MHz, D\textsubscript{2}O, Dioxane reference): \textsuperscript{δ} (ppm): 63.0(CH\textsubscript{2}), 59.2(CH\textsubscript{3}). IR (neat, cm\textsuperscript{-1}): 1697 (w), 1483 (m), 1467 (m), 1458 (m), 1439 (m), 1399 (m), 1289 (w), 1182 (w), 969 (s), 940 (s), 759 (s). HRMS \textit{m}/\textit{z} C\textsubscript{6}H\textsubscript{16}N\textsubscript{2}O\textsubscript{2}H\textsuperscript{+} [M+H]\textsuperscript{+}, Calculated: 149.1285, Found: 149.1288.
2.2. O-Vanillin Alkylation (8b)

\[
\begin{align*}
\text{MeO} & \quad \text{K}_2\text{CO}_3, \text{DMF, } \text{tPrBr} \\
\text{OH} & \quad 110 \degree \text{C}, 3 \text{ h} \\
\text{MeO} & \quad \text{O}^{\text{Pr}} \\
\text{O} & \quad \text{Me} \\
\end{align*}
\]

O-vanillin was alkylated with 2-bromopropane following the method of Couture, et. al.\textsuperscript{2} A clear oil was obtained in 90% yield.

2.3 Sodium Borohydride Reduction Protocol

\[
\begin{align*}
\text{R}_1 & \quad \text{NaBH}_4 (1.5 \text{ eq.)} \\
\text{R}_2 & \quad \text{MeOH, 0 \degree \text{C to r.t., 0.5-2 h}} \\
\text{R}_3 & \quad \text{(7)}
\end{align*}
\]

Aldehyde or ketone (1.0 eq.) was dissolved in methanol (0.6 M solution) and cooled to 0 \degree \text{C}. \text{NaBH}_4 (1.5 eq.) was then added, and the reaction stirred for 5 minutes at 0 \degree \text{C} followed by warming to room temperature until deemed complete by TLC (0.5 - 2 hrs). The reaction was quenched with water, stirred for 10 minutes, and then concentrated under reduced pressure to half volume. The remaining solution was extracted with \text{Et}_2\text{O}, the organic fractions were combined, washed with brine, dried over \text{MgSO}_4 or \text{Na}_2\text{SO}_4 and filtered. The crude alcohol was purified by flash chromatography (ethyl acetate / petroleum spirit) as required.

Alcohols synthesised by this method: (2-isopropoxy-3-methoxyphenyl)methanol, 4-bromobenzyl alcohol, (E)-2-methyl-3-phenylprop-2-en-1-ol, (E)-3-(2-methoxyphenyl)prop-2-en-1-ol, 1-phenylethan-1-ol, 4-isopropylbenzyl alcohol, 4-(hydroxymethyl)benzonitrile, and 2-(trifluoromethyl)benzyl alcohol.
2.4 Synthesis of Diol (15)

The method of Lipshutz et al. was followed. However, the extraction method for 15 was modified such that the residue was dissolved in DCM and extracted using water. The aqueous layers were combined and washed with fresh DCM, then concentrated to afford 3-(4-(hydroxymethyl)phenyl)propan-1-ol (15) (53% yield) as a clear oil. The data of 19 and 15 matched that reported by Lipshutz B. H., et al.¹

19: ¹H NMR (300 MHz, CDCl₃): δ (ppm): 10.00(s, 1H), 7.83(d, J = 8.5 Hz, 2H), 7.57(d, J = 8.2 Hz, 2H), 4.53(d, J = 5.9 Hz, 2H), 1.86(t, J = 6.0 Hz, 1H); GC-MS m/z (ion, % relative intensity): 160([M]+, 41), 131([M-CHO]+, 100), 129([M-CH₃O]+, 5), 77([M-C₄H₄O₂]+, 43).

15: ¹H NMR (300 MHz, CDCl₃): δ (ppm): 7.29(d, J = 8.2 Hz, 2H), 8.17(d, J = 8.2 Hz, 2H), 4.65(s, 2H), 3.66(t, J = 6.4 Hz, 2H), 2.70(t, J = 6.4 Hz, 2H), 1.87(m, 3H), 1.46(s, br, 1H); GC-MS m/z (ion, % relative intensity): 166([M]+, 38), 148([M-O]+, 20), 147([M-HO]+, 9), 133([M-CH₃O]+, 28), 119([M-C₂H₅O]+, 13), 105([M-C₂H₇O]+, 37), 104([M-C₃H₄O₂]+, 18), 77([M-C₄H₆O₂]+, 44).

3. Alcohol Oxidation Protocol

Alcohol (1.0 mmol) was added to a dry flask containing anhydrous DCM (2.0 mL) and powdered 4Å molecular sieves (500 mg / mmol) under an argon atmosphere. TMEDAO₂ (Treated as TMEDAO₂•4H₂O, Mw = 220.27 g/mol, 165 mg, 0.75 mmol) was added and the reaction stirred for 5 minutes before addition of TPAP (17.6 mg, 5 mol%). The reaction was stirred for 4 hrs before the mixture was passed through a silica plug using Et₂O and concentrated under reduced pressure. The crude reaction products were purified by flash chromatography (solvent system ranging from 1:7 EtOAc:Pet. ether to 1:2 EtOAc:Pet. ether or 1:3 Et₂O:Pentane to 1:1 Et₂O:Pentane).
4. Tandem Protocol (Method A)

\[
\begin{array}{c}
\text{R-OH} \\
\text{TPAP (5 mol%),} \\
\text{TMEDAO\textsubscript{2} (0.75 eq.),} \\
\text{PPh\textsubscript{3}CHCO\textsubscript{2}Et (1.1 eq.),} \\
\text{DCM, r.t., 24 h}
\end{array}
\rightarrow
\begin{array}{c}
\text{R} \\
\text{OEt}
\end{array}
\]

Alcohol (1.0 mmol) was dissolved in DCM (2.0 mL), TMEDAO\textsubscript{2} (165 mg, 0.75 mmol) and (carbethoxymethylene)triphenylphosphorane (366 mg, 1.05 mmol) were added and the reaction was stirred for 5 minutes. TPAP (17.6 mg, 5 mol\%) was then added and the reaction mixture was left to stir for 24 hours. The reaction mixture was then passed through a silica plug using Et\textsubscript{2}O (75 mL) and concentrated. Purification by flash chromatography (solvent system ranging from 1:14 EtOAc:Pet. ether to 1:3 EtOAc:Pet. ether) provided the desired product.

5. Tandem Protocol (Method B)

\[
\begin{array}{c}
\text{R-OH} \\
1) \text{TPAP (5 mol%), TMEDAO\textsubscript{2} (0.75 eq.),} \\
\text{DCM, r.t., 8 h} \\
2) \text{PPh\textsubscript{3}CHR’ (2 eq.), reflux, 16 h}
\end{array}
\rightarrow
\begin{array}{c}
\text{R} \\
\text{R’}
\end{array}
\]

Alcohol (1.0 mmol) was dissolved in DCM (2.0 mL), TMEDAO\textsubscript{2} (165 mg, 0.75 mmol) was then added and the reaction was stirred for 5 minutes. TPAP (17.6 mg, 5 mol\%) was added and the reaction mixture was left to stir for 8 hrs. Phosphorane (697 mg, 2.0 mmol) was added and the reaction was refluxed for 16 hrs. The reaction mixture was then passed through a silica plug using Et\textsubscript{2}O (75 mL) and concentrated. Purification by flash chromatography (solvent system ranging from 1:7 EtOAc:Pet. ether to 1:2 EtOAc:Pet. ether) provided the desired product.

6. Tandem Protocol (Method C)

\[
\begin{array}{c}
\text{R-OH} \\
1) \text{TPAP (5 mol%), TMEDAO\textsubscript{2} (0.75 eq.),} \\
\text{DCM, r.t., 8 h} \\
2) \text{PPh\textsubscript{3}CHR’ (2 eq.), reflux, 16 h} \\
3) \text{PPh\textsubscript{3}CHR’ (2 eq.), reflux, 8 h}
\end{array}
\rightarrow
\begin{array}{c}
\text{R} \\
\text{R’}
\end{array}
\]

Alcohol (1.0 mmol) was dissolved in DCM (2.0 mL), TMEDAO\textsubscript{2} (165 mg, 0.75 mmol) was then added and the reaction was stirred for 5 minutes. TPAP (17.6 mg, 5 mol\%) was added and the reaction mixture was left to stir for 8 hours. Phosphorane (697 mg, 2.0 mmol) was added and the reaction was heated to reflux for 16 hrs. A further addition of phosphorane (697 mg, 2.0 mmol) was made and the reaction continued at reflux for a further 8 hrs. The reaction mixture was then passed through a silica plug using Et\textsubscript{2}O (75 mL) and concentrated. Purification by flash chromatography (solvent system ranging from 1:7 EtOAc:Pet. ether to 1:2 EtOAc:Pet. ether) provided the desired product.
7. Tandem Protocol (Method D)

3-(4-(Hydroxymethyl)phenyl)propan-1-ol (15) (166.05 mg, 1.0 mmol), TMEDAO₂ (110.1 mg, 0.50 mmol), (carbethoxymethylene) triphenylphosphorane (697 mg, 2.0 mmol), and DCM (2.0 mL) were added to a round bottom flask and stirred for 5 minutes. TPAP (17.6 mg, 5 mol%) was added and the reaction mixture was left to stir for 24 hours. The reaction mixture was then passed through a silica plug using Et₂O (75 mL) and concentrated. Purification by column chromatography (40% EtOAc:Petroleum Ether) gave the final product 16 (113.6 mg, 49% [78% BRSM]), and by-products 17 (22 mg, 7% [11% BRSM]) and 18 (9.9 mg, 4% [7% BRSM]).
8. Analytical Data of Isolated Products

4-Methoxybenzaldehyde (8a)

![Structure](image)

Clear oil, 88% yield. Data matched that reported by Chen et al.\(^4\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.89(s, 1H), 7.85(d, \(J = 9.0\) Hz, 2H), 7.01(d, \(J = 9.0\) Hz, 2H), 3.90 (s, 3H).

2-Isopropoxy-3-methoxybenzaldehyde (8b)

![Structure](image)

Clear oil, 90% yield. Data matched that reported by Couture et al.\(^2\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 10.46(s, 1H), 7.42(dd, \(J = 6.8, 2.6\) Hz, 1H), 7.14-7.08(m, 2H), 4.63(sep, \(J = 6.0, 3.0\) Hz, 1H), 3.88(s, 3H), 1.33(d, \(J = 6.2\) Hz, 6H).

4-Bromobenzaldehyde (8c)

![Structure](image)

White solid, 46% yield. Data matched that reported by Couture et al.\(^2\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.98(s, 1H), 7.75(d, \(J = 9.0\) Hz, 2H), 7.69(d, \(J = 9.0\) Hz, 2H).

Methyl 4-formylbenzoate (8d)

![Structure](image)

White solid, 69% yield. Data matched that reported by Pelletier et al.\(^5\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 10.11(s, 1H), 8.20(d, \(J = 8.0\) Hz, 2H), 7.96(d, \(J = 8.0\) Hz, 2H), 3.97(s, 3H).

Cinnamaldehyde (8e)

![Structure](image)

Yellow oil, 78% yield. Data matched that reported by Chen et al.\(^4\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.71(d, \(J = 7.9\) Hz, 1H), 7.60-7.41(m, 6H), 6.75(dd, \(J = 15.8, 7.6\) Hz, 1H).

(E)-2-Methyl-3-phenylacrylaldehyde (8e)

![Structure](image)

Pale yellow oil, 85% yield. Data matched that reported by Dohi et al.\(^6\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.60(s, 1H), 7.54(d, \(J = 8.0\) Hz, 2H), 7.48-7.38(m, 3H), 7.28(s, 1H), 2.09(d, \(J = 1.3\) Hz, 3H).

(E)-2-Methoxycinnamaldehyde (8g)

![Structure](image)

Yellow solid, 77% yield. Data matched that reported by Zhu et al.\(^7\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.69(d, \(J = 8.0\) Hz, 1H), 7.84(d, \(J = 16.0\) Hz, 1H), 7.55(m, 1H), 7.41(m, 1H), 7.00(t, \(J = 8.0\) Hz, 1H), 6.95(d, \(J = 8.0\) Hz, 1H), 6.79(dd, \(J = 16.0, 8.0\) Hz, 1H), 3.92(s, 3H).
(1S,5R)-6,6-dimethylbicyclo[3.1.1]hept-2-ene-2-carbaldehyde (Myrtenal, 8h)

Clear oil, 67% yield. Data matched that reported by dos Santos et al.\(^8\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.44(s, 1H), 6.71(m, 1H), 2.87(t, \(J = 6.0\) Hz, 1H), 2.59-2.55(m, 2H), 2.49(m, 1H), 2.19(m, 1H), 1.34(s, 3H), 1.05(d, \(J = 9.0\) Hz, 1H), 0.74(s, 3H).

(S)-(−)-Perillaldehyde (8i)

Clear oil, 75% yield. Data matched that reported by Lu.\(^9\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.44(s, 1H), 6.83(m, 1H), 4.76(d, \(J = 14.5\) Hz, 2H), 2.52-2.43(m, 2H), 2.31-2.22(m, 2H), 2.15(m, 1H), 1.93(m, 1H), 1.77(s, 3H), 1.46(m, 1H).

Acetophenone (8j)

Clear oil, 75% yield. Data matched that reported by Yuan et al.\(^10\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.98-7.95(m, 2H), 7.57(m, 1H), 7.49-7.44(m, 2H), 2.61(s, 3H).

Benzophenone (8k)

White solid, 61% yield. Data matched that reported by Yuan, et al.\(^10\)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.83-7.80(m, 4H), 7.62-7.57(m, 2H), 7.52-7.47(m, 4H).

Citronellal (3,7-Dimethyloct-6-enal, 8l)

Clear oil, 47% yield. Data matched that reported by He et al.\(^11\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 9.77(t, \(J = 4.0\) Hz, 1H), 5.10(t, \(J = 8.0\) Hz, 1H), 2.42(ddd, \(J = 16.0, 5.6, 1.9\) Hz, 1H), 2.24(ddd, \(J = 15.9, 8.0, 2.5\) Hz, 1H), 2.11-1.98(m, 3H), 1.70(s, 3H), 1.62(s, 3H), 1.42-1.26(m, 2H), 0.99(d, \(J = 8.0\) Hz, 2H), 0.90(m, 1H).

Ethyl cinnamate (10a)

Method A: Clear oil, 89% yield \([E:Z, 16:1]\). Data matched that reported by Lebel et al.\(^12\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.69(d, \(J = 16.0\) Hz, 1H), 7.54-7.52(m, 2H), 7.39-7.37(m, 3H), 6.44(d, \(J = 16.0\) Hz, 1H), 4.27(q, \(J = 7.0\) Hz, 2H), 1.33(t, \(J = 8.0\) Hz, 3H); \(^13\)C NMR (75 MHz, CDCl\(_3\)): \(\delta\) (ppm): 167.1, 144.7, 134.6, 130.4, 129.0, 128.1, 118.5, 60.7, 14.5; GC-MS \(m/z\) (ion, % relative intensity): 176([M]\(^+\), 31), 131([M-C\(_2\)H\(_5\)O]\(^+\), 100), 103(M\(^+\)-C\(_3\)H\(_5\)O\(_2\), 45), 77(M\(^+\)-C\(_6\)H\(_7\)O\(_2\), 24).
Ethyl (E)-3-(4-isopropylphenyl)acrylate (10b)

Method A: Clear oil, 83% yield [E:Z 40:1]. Data matched that reported by Ohkawa et al.\(^{13}\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.68(d, \(J = 16.1 \text{ Hz}, 1\)H), 7.47(d, \(J = 7.9 \text{ Hz}, 2\)H), 7.26(d, \(J = 8.2 \text{ Hz}, 2\)H), 6.41(d, \(J = 15.8 \text{ Hz}, 1\)H), 4.27(q, \(J = 7.3 \text{ Hz}, 2\)H), 2.94(sep, \(J = 6.8 \text{ Hz}, 1\)H), 1.36(t, \(J = 7.0 \text{ Hz}, 3\)H), 1.27(d, \(J = 7.0 \text{ Hz}, 6\)H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (ppm): 167.2, 151.5, 144.6, 132.1, 128.1, 127.0, 117.2, 60.4, 34.1, 23.8, 14.3; GC-MS: m/z (ion, % relative intensity): 218([M]+, 64), 203([M–CH\(_3\)]+), 175([M–C\(_3\)H\(_7\)]+, 13), 173([M–C\(_2\)H\(_6\)O]+, 20).

Ethyl (E)-(4-methoxyacrylate (10c)

Method A: Clear oil, 83% yield [E:Z, 20:1]. Data matched that reported by Hyotanishi et al.\(^{14}\)

\(^1\)H NMR (300MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.64(d, \(J = 16.0 \text{ Hz}, 1\)H), 7.47(d, \(J = 9.0 \text{ Hz}, 2\)H), 6.89(d, \(J = 8.8 \text{ Hz}, 2\)H), 6.30(d, \(J = 16.0 \text{ Hz}, 1\)H), 4.25(q, \(J = 9.0 \text{ Hz}, 2\)H), 3.83(s, 3H), 1.33(t, \(J = 9.0 \text{ Hz}, 3\)H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta\) (ppm): 167.5, 161.5, 144.4, 129.8, 127.4, 115.9, 114.5, 60.5, 55.5, 14.5; GC-MS m/z (ion, % relative intensity): 206([M]+, 67), 161([M–C\(_2\)H\(_6\)O]+, 100), 133([M–C\(_3\)H\(_5\)O\(_2\)]+), 41.

Ethyl (E)-(2-isopropoxy-3-methoxyphenyl)acrylate (10d)

Method A: Clear oil, 63% yield [E:Z, 4:1].

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 8.08(d, \(J = 16.2 \text{ Hz}, 1\)H), 7.17(dd, \(J = 8.0, 1.2 \text{ Hz}, 1\)H), 7.03(t, \(J = 8.0 \text{ Hz}, 1\)H), 6.92(dd, \(J = 8.0, 1.2 \text{ Hz}, 1\)H), 6.43(d, \(J = 16.2 \text{ Hz}, 1\)H), 4.45(septet, \(J = 8.0 \text{ Hz}, 1\)H), 4.26(q, \(J = 8.0 \text{ Hz}, 2\)H), 3.85(s, 3H), 1.34(t, \(J = 8.0 \text{ Hz}, 3\)H), 1.30(d, \(J = 8.0 \text{ Hz}, 6\)H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta\) (ppm): 167.1, 153.4, 146.4, 140.3, 129.7, 123.6, 118.9, 118.8, 113.7, 76.1, 60.3, 55.8, 22.4, 14.3; GC-MS: m/z (ion, % relative intensity): 264([M]+, 11), 222([M–C\(_3\)H\(_7\)]+), 21), 176([M–C\(_4\)H\(_6\)O\(_2\)]+), 100), 134([M–C\(_9\)H\(_12\)O\(_3\)]+), 6), 77([M–C\(_9\)H\(_17\)O\(_4\)]+), 7); IR (neat, cm\(^{-1}\)): 2976 (w), 1709 (m), 1632 (w), 1577 (w), 1262 (s), 1214 (m), 1105 (m), 776 (m), 737(m); Anal. Calcd. for C\(_{15}\)H\(_{20}\)O\(_4\): C, 68.16; H, 7.63 O, 24.21. Found: C, 68.11; H, 7.71; O, 24.18; HRMS: m/z C\(_{15}\)H\(_{20}\)O\(_4\)Na\(^{+}\) [M+Na]+, Calculated: 287.1254, Found: 287.1262.

Ethyl (E)-(3-(benzo[d][1,3]dioxol-5-yl)acrylate (10e)

Method A: Clear oil, 90% yield [E:Z 11:1]. Data matched that reported by Leung et al.\(^{15}\)

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.60(d, \(J = 15.8 \text{ Hz}, 1\)H), 7.04(d, \(J = 1.8 \text{ Hz}, 1\)H), 7.01(ddd, \(J = 7.9, 1.8, 0.5 \text{ Hz}, 1\)H), 6.82(d, \(J = 7.9 \text{ Hz}, 1\)H), 6.27(d, \(J = 16.1 \text{ Hz}, 1\)H), 6.02(s, 2H), 4.26(q, \(J = 7.2 \text{ Hz}, 2\)H), 1.34(t, \(J = 7.2 \text{ Hz}, 3\)H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (ppm): 167.2, 149.5, 148.3, 144.3, 128.9, 124.4, 116.2, 108.5, 106.5,
Ethyl (E)-(4-bromophenyl)acrylate (10f)

Method A: Clear oil, 95% yield [E:Z, 11:1]. Data matched that reported by Peñafiel et al.\textsuperscript{16}

\[ \text{\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 7.61(d, J = 16.0 \text{ Hz}, 1H), \]
\[ 7.52(d, J = 8.4 \text{ Hz, 2H}), 7.39(d, J = 8.4 \text{ Hz, 2H}), 6.42(d, J = 16.0 \text{ Hz, 1H}), 4.27(q, J = 10.0, 5.0 \text{ Hz, 2H}), 1.34(t, J = 10.0, 5.0 \text{ Hz, 3H}); \text{\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 166.7, 143.2, 133.4, 132.1, 129.4, 124.5, 119.0, 60.6, 14.3; GC-MS \text{ m/z (ion, % relative intensity): } 256/254([M]\textsuperscript{+}, 79Br/\textsuperscript{81}Br, 41/40), 211/209([M-C\textsubscript{2}H\textsubscript{5}O]\textsuperscript{+}, 79Br/\textsuperscript{81}Br, 91/90), 183/181([M-C\textsubscript{3}H\textsubscript{2}O\textsubscript{2}]\textsuperscript{+}, 79Br/\textsuperscript{81}Br, 24/21), 102([M-C\textsubscript{3}H\textsubscript{5}O\textsubscript{2}Br]\textsuperscript{+}, 100). \]

Ethyl (E)-(4-nitrophenyl)acrylate (10g)

Method A: White solid, 80% yield [E:Z, 16:1]. Data matched that reported by Sharma et al.\textsuperscript{17}

\[ \text{\textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 8.25(d, J = 9.0 \text{ Hz, 2H}), \]
\[ 7.73(d, J = 16.0 \text{ Hz, 1H}), 7.70(d, J = 8.5 \text{ Hz, 2H}), 6.56(d, J = 16.0 \text{ Hz, 1H}), 4.29(q, J = 8.0 \text{ Hz, 2H}), 1.35(t, J = 8.0 \text{ Hz, 3H}); \text{\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 166.0, 148.5, 141.6, 140.6, 128.6, 124.2, 122.6, 61.0, 14.3; GC-MS \text{ m/z (ion, % relative intensity): } 221([M]\textsuperscript{+}, 25), 192([M-C\textsubscript{2}H\textsubscript{5}]\textsuperscript{+}, 8), 176([M-C\textsubscript{2}H\textsubscript{5}O]\textsuperscript{+}, 100). \]

Ethyl (E)-3-(3-methyl-4-nitrophenyl)acrylate (10h)

Method A: Clear oil, 77% yield [E:Z 11:1]

\[ \text{\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 166.1, 149.5, 141.7, 138.9, 134.3, 132.3, 125.9, 125.4, 122.0, 60.9, 20.6, 14.2; GC-MS: \text{ m/z (ion, % relative intensity): } 235([M]\textsuperscript{+}, 24), 219([M-O]\textsuperscript{+}, 13), 190([M-C\textsubscript{2}H\textsubscript{5}O]\textsuperscript{+}, 100), 144([M-C\textsubscript{2}H\textsubscript{5}O\textsubscript{2}N]\textsuperscript{+}, 72); IR (neat, cm\textsuperscript{-1}): 3117(w), 3045(w), 2992(w), 1717(s), 16419(m), 1609(m), 1512(s), 1327(s), 1291(s), 1159(s), 1032(s), 981(s), 826(s), 760(s), 676(m); Anal. Calcd for C\textsubscript{12}H\textsubscript{13}NO\textsubscript{4}: C, 61.27; H, 5.57; N, 5.95. Found: C, 61.07; H, 5.64; N, 6.00; HRMS: \text{ m/z C}_{12}H_{13}NO_4Na\textsuperscript{+} [M+Na]\textsuperscript{+}, Calcd. 258.0737, Found 258.0747. \]

Ethyl (E)-3-(4-cyanophenyl)acrylate (10i)

Method A: Clear oil, 80% yield [E:Z 10:1]. Data matched that reported by Leung et al.\textsuperscript{15}

\[ \text{\textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 7.71-7.61(m, 5H), 6.52(d, J = 15.8 \text{ Hz, 1H}), 4.29(q, J = 7.1 \text{ Hz, 2H}), 1.36(t, J = 7.2 \text{ Hz, 3H}); \text{\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (\text{ppm}): 166.1, 142.1, 138.7, 132.6, 128.3, 121.8, 118.4, 113.3,
Ethyl (E)-(4-methylbenzanoate)acrylate (10j)

Method A: White solid, 87% yield [E:Z, 14:1]. Data matched that reported by Chintareddy et al.\textsuperscript{18} 

\[ ^1H \text{NMR (300 MHz, CDCl}_3\]: } \delta (ppm): 8.05(d, J = 8.0, 2H), 7.70 (d, J = 16.0 Hz, 1H) 7.58(d, J = 8.0, 2H), 6.52(d, J = 16.0 Hz, 1H), 4.28(q, J = 7.0 Hz, 2H), 3.93(s, 3H), 1.35(t, J = 7.0 Hz, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (ppm): 166.1, 166.5, 143.2, 138.8, 131.4, 130.2, 128.0, 120.8, 60.9, 52.4, 14.4; GC-MS m/z (ion, % relative intensity): 234([M]+, 86), 189([M-C\textsubscript{2}H\textsubscript{5}O]+, 100), 175([M-C\textsubscript{2}H\textsubscript{5}O\textsubscript{2}]+, 60), 161([M-C\textsubscript{3}H\textsubscript{5}O\textsubscript{2}]+, 7).

Ethyl (E)-3-(2-(trifluoromethyl)phenyl)acrylate (10k)

Method A: Clear oil, 77% yield [E:Z, 12:1]. Data matched that reported by Wang et al.\textsuperscript{19}

\[ ^1H \text{NMR (400 MHz, CDCl}_3\]: } \delta (ppm): 8.07(dq, J = 15.6, 2.3 Hz, 1H), 7.73-7.70(m, 2H), 7.58(m, 1H), 7.49(t, J = 7.3 Hz, 1H), 6.42(d, J = 15.8 Hz, 1H), 4.30(q, J = 7.0 Hz, 2H), 1.36(t, J = 7.0 Hz, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (ppm): 166.1, 140.0, 133.5, 132.1, 129.5, 127.9, 126.1 (q, J = 5.7 Hz), 125.3, 122.6, 60.8, 14.3; GC-MS: m/z (ion, % relative intensity): 244([M]+, 18), 199([M-C\textsubscript{2}H\textsubscript{5}O]+, 100), 175([M-CF\textsubscript{3}]+, 62), 171([M-C\textsubscript{3}H\textsubscript{5}O\textsubscript{2}]+, 26).

Ethyl (E)-(furan-2-yl)acrylate (10l)

Method A: Orange oil, 70% [E:Z, 7:1] yield. Data matched that reported by Chintareddy et al.\textsuperscript{18}

\[ ^1H \text{NMR (300 MHz, CDCl}_3\]: } \delta (ppm): 7.48(m, 1H), 7.43(d, J = 18.0 Hz, 1H), 6.60(d, J = 3.0 Hz, 1H), 6.47(dd, J = 3.0, 1.9 Hz, 1H), 6.32(d, J = 18.0 Hz, 1H), 4.23(q, J = 6.0 Hz, 2H), 1.32(t, J = 6.0 Hz, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): } \delta (ppm): 167.1, 151.0, 144.7, 131.0, 116.0, 114.6, 112.2, 60.4, 14.3; GC-MS m/z (ion, % relative intensity): 166([M]+, 22), 121([M-C\textsubscript{2}H\textsubscript{5}O]+, 100), 93([M-C\textsubscript{2}H\textsubscript{5}O\textsubscript{2}]+, 13).

Ethyl (2E,4E)-5-(2-methoxyphenyl)penta-2,4-dienoate (10m)

Method A: Clear oil, 82% yield [EE;EZ, 6:1]. Data matched that reported by Magrioti et al.\textsuperscript{20}

\[ ^1H \text{NMR (300 MHz, CDCl}_3\]: } \delta (ppm): 7.52-7.47(m, 2H), 7.31-7.23(m, 2H), 6.98-6.88(m, 3H), 5.96(d, J = 15.0 Hz, 1H), 4.32(q, J = 7.0 Hz, 2H), 3.88(s, 3H), 1.31(t, J = 7.0 Hz, 3H); \textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}): } \delta (ppm): 167.2, 157.4, 145.5, 135.5, 130.1, 127.3, 126.8, 125.1, 120.7, 120.6, 111.1, 60.2, 55.5, 14.3; GC-MS m/z (ion, % relative intensity): 232([M]+, 5), 159([M-C\textsubscript{3}H\textsubscript{5}O\textsubscript{2}]+, 24).
**Ethyl (E)-7-phenylhept-2-enoate (10n)**

Method A: Clear oil, 42% yield [E:Z, 9:1]. Data matched that reported by Ghogare et al. 21

^1^H NMR (400 MHz, CDCl₃): δ (ppm): 7.31-7.24 (m, 2H), 7.21-7.16 (m, 3H), 6.96 (dt, J = 15.6, 7.0 Hz, 1H), 5.81 (dt, J = 15.6, 1.5 Hz, 1H), 4.19 (q, J = 7.0 Hz, 2H), 2.63 (t, J = 7.6 Hz, 2H), 2.23 (qd, J = 7.3, 1.5 Hz, 2H), 1.70-1.62 (m, 2H), 1.57-1.45 (m, 2H), 1.29 (t, J = 7.0 Hz, 3H); ^1^3^C NMR (100 MHz, CDCl₃): δ (ppm): 166.7, 149.0, 142.2, 128.4, 128.3, 125.7, 121.4, 60.1, 35.7, 32.0, 31.0, 27.6, 14.3. GC-MS: m/z (ion, % relative intensity): 232 ([M]^+, 8), 159 ([M−C₃H₅O₂]^+, 12), 158 ([M−C₃H₅O₂]^+, 29), 91 ([M−C₆H₁₃O₂]^+, 100).

**Ethyl (E)-5,9-dimethyldeca-2,8-dienoate (10o)**

Method A: Clear oil, 48% yield [E:Z, 6:1]. Data matched that reported by Lebel et al. 12

^1^H NMR (300 MHz, CDCl₃): δ (ppm): 6.94 (dt, J = 15.0, 7.5 Hz, 1H), 5.81 (d, J = 15.0 Hz, 1H), 5.08 (m, 1H), 4.19 (q, J = 6.0 Hz, 2H), 2.20 (m, 1H), 2.09-1.94 (m, 3H), 1.68 (s, 3H), 1.64 (m, 1H), 1.60 (s, 3H), 1.37 (m, 1H), 1.29 (t, J = 7.0 Hz, 3H), 1.17 (m, 1H), 0.91 (d, J = 7.0 Hz, 3H); ^1^3^C NMR (75 MHz, CDCl₃): δ (ppm): 166.7, 148.2, 131.5, 124.4, 122.4, 60.1, 39.6, 36.7, 32.0, 25.7, 25.5, 19.5, 17.7, 14.3; GC-MS m/z (ion, % relative intensity): 224 ([M]^+, 2), 209 ([M−CH₃]^+, 3), 179 ([M−C₂H₅O]^+, 6), 155 ([M−C₅H₅]^+, 4), 151 ([M−C₃H₅O₃]^+, 5), 141 ([M−C₆H₁₁]^+, 14), 125 ([M−C₅H₇O₂]^+, 10), 83 ([M−C₆H₁₃O₂]^+, 8), 69 ([M−C₆H₁₅O₂]^+, 100), 55 ([M−C₁₀H₁₇O₂]^+, 40).

**Benzyl (E)-(4-methoxyphenyl)acrylate (14a)**

Method B: Clear oil, 84% yield [E:Z, 14:1]. Data matched that reported by El-Batta et al. 22

^1^H NMR (300 MHz, CDCl₃): δ (ppm): 7.73 (d, J = 15.9 Hz, 1H), 7.47 (d, J = 8.8 Hz, 2H), 7.44-7.33 (m, 5H), 6.91 (d, J = 8.8 Hz, 2H), 6.39 (d, J = 15.9 Hz, 1H), 5.28 (s, 2H), 3.81 (s, 3H); ^1^3^C NMR (75 MHz, CDCl₃): δ (ppm): 166.7, 161.1, 144.5, 135.9, 129.4, 128.2, 127.9, 127.8, 126.7, 115.0, 114.0, 65.8, 55.0; GC-MS m/z (ion, % relative intensity): 268 ([M]^+, 52), 177 ([M−C₁₁H₁₂]^+, 10), 161 ([M−C₇H₉O]^+, 60), 135 ([M−C₉H₅O]^+, 9), 133 ([M−C₉H₇O₂]^+, 24), 107 ([M−C₁₀H₉O₂]^+, 3), 77 ([M−C₁₁H₁₂O₃]^+, 16).

**Benzyl (E)-(4-nitrophenyl)acrylate (14b)**

Method B: White solid, 54% yield [E:Z, 10:1]. Data matched that reported by Echavarren et al. 23

^1^H NMR (400 MHz, CDCl₃): δ (ppm): 8.25 (d, J = 8.7 Hz, 2H), 7.75 (d, J = 16.0 Hz, 1H), 7.67 (d, J = 8.6 Hz, 2H), 7.44-7.35 (m, 5H), 6.60 (d, J = 16.0 Hz, 1H), 5.28 (s, 2H); ^1^3^C NMR (100 MHz, CDCl₃): δ (ppm): 165.8, 148.5, 142.1, 140.4, 135.6, 128.7, 128.6, 128.5, 128.4, 124.2, 122.2, 66.8; GC-MS m/z (ion, % relative intensity): 283 ([M]^+, 2), 266 ([M−O]^+, 11), 238 ([M−O₂N]^+, 24),...
192([M–C₇H₇]⁺, 18), 176([M–C₇H₇O]⁺, 35), 107([M–C₆H₁₀N₃]⁺, 6), 91([M–C₆H₁₆O₄N]⁺, 100), 77([M–C₁₀H₁₆O₃N]⁺, 10).

(E)-1-(4-Methoxyphenyl)but-1-en-3-one (14c)

Method B: White Solid, 75% yield [E:Z, >99%E]. Data matched that reported by Solin et al.²⁴

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.53-7.45(m, 3H), 6.95-6.90(m, 2H), 6.61(d, J = 16.3 Hz, 1H), 3.85(s, 3H), 2.36(s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 198.5, 161.7, 143.4, 130.1, 127.2, 125.2, 114.6, 55.6, 27.6; GC-MS m/z (ion, % relative intensity): 176([M]⁺, 49), 161([M–CH₃]⁺, 100), 145([M–CH₂O]⁺, 14), 133([M–C₂H₅O]⁺, 45), 77([M–C₅H₈O₂]⁺, 12).

(E)-1-(4-Nitrophenyl)but-1-en-3-one (14d)

Method B: Pale Yellow Solid, 45% yield [E:Z, 11:1]. Data matched that reported by Leung et al.¹⁵

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.29-8.22(m, 2H), 7.72-7.66(m, 2H), 7.53(d, J = 16.3 Hz, 1H), 6.82(d, J = 16.3 Hz, 1H), 2.42(s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 197.6, 148.7, 140.8, 140.2, 130.5, 128.9, 124.4, 28.2; GC-MS m/z (ion, % relative intensity): 191([M]⁺, 25), 176([M–CH₃]⁺, 100), 146([M–NO₂]⁺, 9), 77([M–C₄H₅NO₃]⁺, 6).

(E)-4-Methoxycinnamonic acid (14e)

Method C: Clear oil, 88% yield [E:Z, 3:1]. Data matched that reported by Qin et al.²⁵

¹H NMR (300 MHz, CDCl₃): δ (ppm): 7.42-7.37(m, 2H), 7.33(d, J = 16.5 Hz, 1H), 6.98-6.88(m, 2H), 5.71(d, J = 16.6 Hz, 1H), 3.85(s, 3H). ¹³C NMR (75 MHz, CDCl₃): δ (ppm): 162.0, 150.0, 129.0, 126.3, 118.7, 114.5, 93.4, 55.4; GC-MS m/z (ion, % relative intensity): 159([M]⁺, 100), 144([M–CH₃]⁺, 31), 129([M–CH₂O]⁺, 11), 77([M–C₄H₅NO]⁺, 4).

(E)-4-Nitrocinnamic acid (14f)

Method B: Pale Yellow Solid, 21% yield [E:Z, 3:1]. Data matched that reported by Zhou et al.²⁶

¹H NMR (300 MHz, d₆-DMSO) δ (ppm): 8.32-8.24(m, 2H), 7.96-7.89(m, 2H), 7.82(d, J = 16.8 Hz, 1H), 6.73(d, J = 16.7 Hz, 1H). ¹³C NMR (75 MHz, d₆-DMSO) δ (ppm): 148.2, 146.7, 139.8, 128.9, 124.1, 118.1, 101.4; GC-MS m/z (ion, % relative intensity): 174([M]⁺, 100), 128([M–NO₂]⁺, 69), 77([M–C₃H₂N₂O₂]⁺, 51).
Ethyl (E)-3-(4-(3-hydroxypropyl)phenyl)acrylate (16)

Method D: Pale Yellow oil, 49% [77% BRSM] yield.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 7.67(d, \(J = 16.0\) Hz, 1H), 7.45(d, \(J = 8.1\) Hz, 2H), 7.22(d, \(J = 8.0\) Hz, 2H), 6.40(d, \(J = 16.0\) Hz, 1H), 4.26(q, \(J = 7.1\) Hz, 2H), 3.73-3.64(m, 2H), 2.77-2.70(m, 2H), 1.94-1.85(m, 2H), 1.34(t, \(J = 7.1\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 167.1, 144.5, 144.4, 132.2, 129.0, 128.2, 117.4, 62.1, 60.4, 33.9, 31.9, 14.3; GC-MS m/z (ion, % relative intensity): 234([M]\(^+\), 100), 205([M−C\(_2\)H\(_5\)]\(^+\), 8), 203([M−CH\(_3\)O]\(^+\), 58), 189([M−C\(_2\)H\(_5\)O]\(^+\), 50), 175([M−C\(_3\)H\(_6\)O]\(^+\), 22), 161([M−C\(_3\)H\(_5\)O\(_2\)]\(^+\), 36), 77([M−C\(_11\)H\(_11\)O\(_2\)]\(^+\), 14); IR (neat, cm\(^{-1}\)): 3352 (br, w), 2936 (w), 1706 (s), 1634 (s), 1310 (m), 1173 (s), 1036(m); Anal. Calcd. for C\(_{14}\)H\(_{18}\)O\(_3\): C, 71.77; H, 7.74; O, 20.49. Found: C, 71.09; H, 7.82; O, 21.09; HRMS: m/z C\(_{14}\)H\(_{18}\)O\(_3\)Na\(^+\) [M+Na]\(^+\), Calculated: 257.1148, Found: 257.1150.

Ethyl (E)-5-(4-((E)-3-ethoxy-3-oxoprop-1-en-1-yl)phenyl)pent-2-enoate (17)

Method D: Clear oil, 7% [11% BRSM] yield. Data matched that reported by Panther \textit{et al.}\textsuperscript{27}

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 7.66(d, \(J = 16.1\) Hz, 1H), 7.46(d, \(J = 7.6\) Hz, 2H), 7.20(d, \(J = 7.7\) Hz, 2H), 6.97(dt, \(J = 15.6, 6.8\) Hz, 1H), 6.40(d, \(J = 16.0\) Hz, 1H), 5.84(dt, \(J = 15.6, 1.6\) Hz, 1H), 4.26(q, \(J = 7.1\) Hz, 2H), 4.18(q, \(J = 7.0\) Hz, 2H), 2.83-2.73(m, 2H), 2.57(m, 2H), 1.34(t, \(J = 7.1\) Hz, 3H), 1.28(t, \(J = 7.1\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 167.1, 166.4, 147.5, 144.3, 143.3, 133.6, 132.5, 128.9, 128.2, 122.1, 117.6, 60.4, 60.2, 34.2, 33.5, 14.3; GC-MS m/z (ion, % relative intensity): 302([M]\(^+\), 7), 257([M−C\(_2\)H\(_5\)O]\(^+\), 8), 211([M−C\(_4\)H\(_{10}\)O\(_2\)]\(^+\), 4), 189([M−C\(_6\)H\(_9\)O\(_2\)]\(^+\), 100).
9. NMR Spectra

300 MHz, D$_2$O

100 MHz, D$_2$O (Dioxane)
$\text{(8g)}$

400 MHz, CDCl$_3$

$\text{(8h)}$

300 MHz, CDCl$_3$
NMO vs. TMEDAO₂ % Weight Gain

% Weight Gain

Time (hours)

0.0 2.0 4.0 6.0 8.0 10.0

0.0 0.5 1.0 1.5 2.0 2.5 3.0

Water Absorption

NMO vs. TMEDAO₂ Water Absorption
11. References


