An Efficient Perkin Synthesis of ¹³C-Labelled Cinnamic Acids From Acetic Acid as the Source of the Rare Isotope

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NMR spectroscopic product characterization 1



Figure S 1. ¹H NMR spectra of 1a in (CD₃)₂CO recorded at 270 MHz: a. natural isotope abundance; b. 1-¹³C. Note the additional splitting in the signals of the olefinic protons due to the coupling with 1^{-13} C (7 and 3 Hz, respectively). The Lorentzian lineshape analysis in the inset reveals that a doublet of doublets describes the spectral shape accurately (i.e. the fraction of 1^{-12} C is below detection).

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Figure S 2. ¹³C NMR spectra of **1a** in (CD₃)₂CO recorded at 75 MHz: a. DEPT-135; b. APT. Note the splitting of signals at 131.85 and 117.15 ppm due to a *J*-coupling to C-1 of 7 and 77 Hz, resp.; these signals likely correspond to C-4 and C-2.

1.1 Spectral listing



Figure S 3.Structures of products.

1a: ¹H-NMR (250 MHz, (CD₃)₂CO): δ = 7.65 (dd, $J_1(^{1}H^{-1}H)$ = 16 Hz, $J_2(^{1}H^{-13}C)$ = 7 Hz, 1H), 7.55 (d, 8 Hz, 2H), 7.25 (d, 8 Hz, 2H), 6.45 (dd, $J_1(^{1}H^{-1}H)$ = 16 Hz, $J_2(^{1}H^{-13}C)$ = 3 Hz, 1H, 2.4 ppm (s, 3H); {¹H}¹³C NMR (75 MHz, (CD₃)₂CO): δ = 167.09, 144.66, 140.54, 131.85 (d, J = 7 Hz), 129.55, 128.12, 117.15 (d, J = 77 Hz), 20.47 ppm.

1b: ¹H NMR (300 MHz, (CD₃)₂CO): δ = 7.66 (dd, $J_1(^{1}H^{-1}H)$ = 16 Hz, $J_2(^{1}H^{-13}C)$ = 7 Hz, 1H), 7.60 (d, J = 8 Hz, 2H), 7.29 (d, J = 8 Hz, 2H), 6.48 (dd, $J_1(^{1}H^{-1}H)$ = 16 Hz, $J_2(^{1}H^{-13}C)$ = 3 Hz, 1H), 2.67 (q, J = 8 Hz, 2H), 1.22 ppm (t, J = 8 Hz, 3H); {¹H}¹³C NMR (75 MHz, (CD₃)₂CO): δ = 166.94, 146.85, 144.59, 128.31 (d, J = 13 Hz), 117.25 (d, J = 73 Hz), 28.40, 14.92 ppm.

1c: ¹H NMR (300 MHz, (CD₃)₂CO): δ = 7.67 (dd, $J_1(^{1}H^{-1}H)$ = 16 Hz, $J_2(^{1}H^{-13}C)$ = 7 Hz, 1H), 7.61 (d, J = 8 Hz, 2H), 7.48 (d, J = 8 Hz, 2H), 6.49 (dd, $J_1(^{1}H^{-1}H)$ = 16 Hz, $J_2(^{1}H^{-13}C)$ = 3 Hz, 1H), 1.33 (s, 9H); {¹H}¹³C NMR (75 MHz, (CD₃)₂CO): 167.14, 144.56, 131.82 (d, J = 7 Hz), 128.00, 125.82, 117.34 (d, J = 73 Hz), 35.14, 30.52 ppm.

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Figure S 4. ¹H NMR spectra of **1a** to **1c** in (CD₃)₂CO. Note that close inspection reveals the couplings (as in Figure S 1) that indicate ¹³C labelling for all cases.