Direct C-Arylation of β -enamino Esters and Ketones with Arynes

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Supporting Information

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General Methods: All reactions were performed under a stream of nitrogen unless otherwise stated. Solvents and solutions were transferred with syringes and cannulae using standard inert atmosphere techniques. All solvents were purchased anhydrous over molecular sieves in Sure-Seal bottle and used as received. Nuclear magnetic resonance spectra were recorded on a Bruker (¹H, 600 MHz, 500 MHz or 400 MHz; ¹³C, 126 MHz; ²H, 92 MHz) spectrometer. Chemical shifts (δ) are reported in ppm relative to residual deuterated solvent peaks with tetramethylsilane (¹H) as internal standard. Spectral features are tabulated in the following order: chemical shift (δ , ppm); multiplicity (s-singlet, d-doublet, t-triplet, q-quartet, sept-septet, m-complex multiplet); coupling constants (*J*, Hz); number of protons. High resolution mass spectrometry was performed by the McGill University Mass Spectrometry Laboratory. Analytical TLC was done using EM Separations precoated silica gel 0.2-mm layer UV 254 fluorescent glass plates and visualized with UV light. Flash chromatography was performed using Isco Combi-Flash Sq16x system or by chromatography column with the indicated solvents using silica gel 60 (particle size 0.040-0.063 mm). The β -enamino ketones, (3*Z*)-4-anilinopent-3-en-2-one,¹ (3*Z*)-4-(allylamino)pent-3-en-2-one,¹ (3*Z*)-4-[(2-hydroxyethyl)amino]pent-3-en-2-one¹ and 3-aminocyclopent-2-en-1-one² were prepared according to literature procedures. The *o*-silyl aryltriflate, 4,5-difluoro-2-(trimethylsilyl)phenyl triflate,³ 10-(trimethylsilyl)phenanthren-9-yl triflate,³ 6-(trimethylsilyl)-1,3-benzodioxol-5-yl triflate,⁴ 6-(trimethylsilyl)-2,3-dihydro-1*H*-inden-5-yl triflate⁵ and 3,6-dimethyl-2-(trimethylsilyl)phenyl triflate,⁵ were prepared using reported literature procedures.

General procedure for the C-arylation of β -enamino esters and ketones with arynes.

To a mixture of the β -enamino ester or ketone (0.4 mmol, 1 eq.) and CsF (152 mg, 1 mmol, 2.5 eq.) in acetonitrile (2 mL, 0.2 M) in an oven dried 4 mL glass vial was added the *o*-silyl aryltriflate (0.5 mmol, 1.25 eq.). The vial was capped under nitrogen and the mixture was stirred at room temperature and monitored by TLC. After completion of the reaction, the solvent was evaporated, the residue dissolved in H₂O (2 mL) and extracted with EtOAc (3 x 2 mL). The combined EtOAc extracts were dried over Na₂SO₄ and concentrated. The residue was then dissolved in dichloromethane to precipitate out any insoluble salts, filtered and concentrated. The crude product was purified by combi-flash on silica gel column using a gradient of EtOAc/hexanes or EtOAc/MeOH as eluent to afford the desired product.

Ethyl (2Z)-3-[(4-Fluorophenyl)amino]-2-phenylbut-2-enoate (3a). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-8% EtOAc/hexanes) afforded the title compound as a yellowish solid (103.2 mg, 87%). ¹H NMR (400 MHz, acetone-*d*₆): δ 11.30 (br s, 1 H); 7.37-7.12 (m, 9 H); 4.08 (q, *J* = 7.07 Hz, 2 H); 1.80 (s, 3 H); 1.12 (t, *J* = 7.07 Hz, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 14.7, 18.5, 59.6, 101.0, 116.5 (d, ²*J* = 22.9 Hz), 126.9, 127.6 (d, ³*J* = 8.1 Hz), 128.6, 132.7, 136.9 (d, ⁴*J* = 2.6 Hz), 140.3, 158.2, 161.0 (d, ¹*J* = 242.8 Hz), 170.5. HRMS (ESI, MH⁺) calculated for C₁₈H₁₉FO₂ 300.1394, found 300.1394.

Ethyl (2Z)-3-anilino-2-phenylbut-2-enoate (3b). Reaction time, 6 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-10% EtOAc/hexanes) afforded the title compound as a yellowish oil (97.4mg, 87%). ¹H NMR (400 MHz, acetone-*d*₆): δ 11.43 (br s, 1 H); 7.42 (t, *J* = 7.72 Hz, 2 H); 7.35 (t, *J* = 7.41 Hz, 2 H); 7.30-7.18 (m, 6 H); 4.09 (q, *J* = 7.05 Hz, 2 H); 1.86 (s, 3 H); 1.12 (t, *J* = 7.08 Hz, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 14.2, 18.6, 59.5, 101.1, 125.3, 125.5, 126.9, 128.6, 129.9, 132.7, 139.3, 140.6, 158.6, 170.5. HRMS (ESI, MH⁺) calculated for C₁₈H₂₀NO₂ 282.1490, found 282.1489.

Dimethyl (2Z)-2-[(4-butyl-2-methylphenyl)amino]-3-phenylbut-2-enedioate (3c). Reaction time, 5 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-8% EtOAc/hexanes) afforded the title compound as a yellow solid (75 mg, 50%). ¹H NMR (400 MHz, acetone-*d*₆): δ 10.48 (br s, 1 H); 7.36-7.18 (m, 5 H); 7.13 (s, 1 H); 7.00 (d, *J* = 8.24 Hz, 1 H); 6.91 (d, *J* = 8.10 Hz, 1 H); 3.69 (s, 3 H); 3.26 (s, 3 H); 2.58 (t, *J* = 7.71 Hz, 2 H); 2.38 (s, 3 H); 1.59 (p, *J* = 7.65 Hz, 2 H); 1.43-1.30 (m, 2 H); 0.94 (t, *J* = 7.35 Hz, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 14.1, 18.0, 23.0, 34.3, 35.5, 51.6, 52.7, 101.4, 123.0, 127.3, 127.5, 128.3, 131.5, 132.0, 132.2, 136.5, 136.7, 140.9, 151.9, 164.7, 170.8. HRMS (ESI, MH⁺) calculated for C₂₃H₂₈NO₄ 382.2012, found 382.2013.

4-Ethyl 1-methyl (2Z)-2-(isopropylamino)-3-phenylbut-2-enedioate (3d). Reaction time, 5 hrs; Purification by Combi-Flash (SiO₂, gradient elution 1-8% EtOAc/hexanes) afforded the title compound as an off-white solid (64.5 mg, 58%). ¹H NMR (400 MHz, acetone-*d*₆): δ 8.77 (br s, 1 H); 7.31-7.13 (m, 5 H); 3.59 (s, 3 H); 3.42-3.51 (m, 4 H); 1.27 (s, 6 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 24.4, 48.2, 51.0, 52.2, 96.6, 127.2, 128.2, 132.4, 137.1, 154.6, 165.0, 170.5. HRMS (ESI, MH⁺) calculated for C₁₅H₂₀NO₄ 278.1386, found 278.1387.

Dimethyl (2Z)-2-amino-3-phenylbut-2-enedioate (3e). Reaction time, 6 hrs; Purification by Combi-Flash (SiO₂, gradient elution 2-15% EtOAc/hexanes) afforded the title compound as a white solid (54.9 mg, 72%). ¹H NMR (400 MHz, acetone-*d*₆): δ 8.64 (s, 1 H); 7.34-7.23 (m, 2 H); 7.19 (t, *J* = 7.33 Hz, 1 H); 7.12 (d, *J* = 7.46 Hz, 2 H); 6.27 (s, 1 H); 3.47 (s, 3 H); 1.73 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.0, 50.3, 97.5, 126.5, 128.4, 132.9, 139.7, 159.9, 170.2. HRMS (ESI, MH⁺) calculated for C₁₁H₁₄NO₂ 192.1018, found 192.1019.

Methyl (2Z)-3-amino-2-phenyl-3-[4-(trifluoromethyl)phenyl]acrylate (3f). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-15% EtOAc/hexanes) afforded the title compound as a yellow solid (108 mg, 84%). ¹H NMR (500 MHz, acetone-*d*₆): δ 7.50 (d, *J* = 8.01 Hz, 2 H); 7.38 (d, *J* = 7.99 Hz, 2 H); 7.05-6.95 (m, 5 H); 3.58 (s, 3 H). ¹³C NMR (126 MHz, Acetone): δ 50.8, 125.4, 125.4 (q, ³*J* = 4 Hz), 126.3, 127.1 (q, ¹*J* = 268 Hz), 128.0, 130.5, 133.7, 138.5, 143.3, 160.3, 170.7. HRMS (ESI, MH⁺) calculated for C₁₇H₁₅F₃NO₂ 322.1048, found 322.1049.

Ethyl (2Z)-3-amino-3-(4-fluorophenyl)-2-phenylacrylate (3g). Reaction time, 5 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-15% EtOAc/hexanes) afforded the title compound as a yellow solid (103.3 mg, 91%). ¹H NMR (400 MHz, DMSO-*d*₆): δ 7.06 (t, *J* = 8.46 Hz, 2 H); 7.00-6.87 (m, 5 H); 6.83 (d, *J* = 7.42 Hz, 2 H); 3.97 (q, *J* = 7.06 Hz, 2 H); 1.02 (t, *J* = 7.06 Hz, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 14.7, 59.4, 98.8, 115.3 (d, ²*J* = 21.5 Hz), 125.8, 127.7, 131.8 (d, ³*J* = 8.6 Hz), 133.7, 135.7, 140.0, 160.9, 163.1 (d, ¹*J* = 158.6 Hz), 170.2. HRMS (ESI, MH⁺) calculated for C₁₇H₁₇FNO₂ 286.1236, found 286.1238.

Ethyl (2Z)-3-amino-3-(3-furyl)-2-phenylacrylate (3h). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-15% EtOAc/hexanes) afforded the title compound as a white solid (73 mg, 71%). ¹H NMR (500 MHz, acetone-*d*₆): δ 7.34 (t, *J* = 1.75 Hz, 1 H); 7.26-7.18 (m, 4 H); 7.14-7.10 (m, 2 H); 6.00 (d, *J* = 1.85 Hz, 1 H); 4.08 (q, *J* = 7.08 Hz, 2 H); 1.12 (t, *J* = 7.07 Hz, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 14.7, 59.2, 98.5, 111.0, 124.2, 126.7, 128.2, 133.3, 139.5, 143.2, 144.0, 153.0, 170.2. HRMS (ESI, MH⁺) calculated for C₁₅H₁₆NO₃ 258.1123, found 258.1125.

(3Z)-4-Anilino-3-phenylpent-3-en-2-one (5a). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-10% EtOAc/hexanes) afforded the title compound as a yellow oil (81.6 mg, 82%) . ¹H NMR (400 MHz, CDCl₃): δ 13.77 (br s, 1 H); 7.61-7.31 (m, 10 H); 2.09 (s, 3 H); 1.94 (s, 3 H). ¹³C NMR (126 MHz, CDCl₃): δ 18.4, 29.4, 111.5, 125.3, 125.6, 126.8, 128.7, 129.1, 131.9, 139.0, 140.5, 159.4, 196.1. HRMS (ESI, MH⁺) calculated for C₁₇H₁₈NO 252.1381, found 252.1383.

(3Z)-4-[(Cyclopropylmethyl)amino]-3-phenylpent-3-en-2-one (5b). Reaction time, 4 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-10% EtOAc/hexanes) provided the title compound as a yellow oil (86.6 mg, 87%). ¹H NMR (500 MHz, acetone-*d*₆): δ 12.12 (br s, 1 H); 7.37 (dd, *J* = 7.50, 7.50 Hz, 2 H); 7.28 (dd, *J* = 7.40, 7.40 Hz, 1 H); 7.18 (d, *J* = 7.52 Hz, 2 H); 3.24 (t, *J* = 6.15 Hz, 2 H); 1.72 (s, 3 H); 1.71 (s, 3H) 1.14-1.04 (m, 1 H); 0.58 (q, *J* = 4.61, 2 H); 0.30 (q, *J* = 4.97, 2 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 2.9, 10.8, 15.8, 28.3, 47.5, 108.7, 126.3, 128.4, 132.3, 141.6, 161.3, 192.7. HRMS (ESI, MH⁺) calculated for C₁₅H₂₀NO 230.1538, found 230.1539.

(3Z)-4-(Allylamino)-3-phenylpent-3-en-2-one (5c). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-16% EtOAc/hexanes) provided title compound as an orange oil (75.3 mg, 88%). ¹H NMR (400 MHz, acetone-*d*₆): δ 12.03 (s, 1 H); 7.32-7.27 (m, 2 H); 7.23-7.18 (m, 1 H); 7.11 (dd, *J* = 8.00, 1.44 Hz, 2 H); 5.92 (ddt, *J* = 17.21, 10.27, 5.08 Hz, 1 H); 5.22 (dq, *J* = 17.21, 1.71 Hz, 1 H); 5.12 (dq, *J* = 10.38, 1.60 Hz, 1 H); 3.95-3.90 (m, 2 H); 1.65 (s, 3 H); 1.64 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 16.5, 29.2, 45.9, 110.1, 115.8, 127.2, 129.2, 133.0, 135.9, 142.2, 162.6, 194.0. HRMS (ESI, MH⁺) calculated for C₁₄H₁₈NO 216.1382, found 216.1383.

(3Z)-4-[(2-Hydroxyethyl)amino]-3-phenylpent-3-en-2-one (5d). Reaction time, 5 hrs; Purification by Combi-Flash (SiO₂, gradient elution 15-50% EtOAc/hexanes) afforded the title compound as a white solid (78.5 mg, 90%). ¹H NMR (400 MHz, acetone-*d*₆): δ 12.12 (s, 1 H); 7.38 (t, *J* = 7.47 Hz, 2 H); 7.30 (t, *J* = 3.72 Hz, 2 H); 7.19 (d, *J* = 7.51 Hz, 4 H); 4.17 (br s, 1 H); 3.76 (m, 2 H); 3.48 (q, *J* = 5.62 Hz, 2 H); 1.76 (s, 3 H); 1.71 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 16.9, 29.1, 46.4, 62.0, 109.7, 127.1, 129.2, 133.1, 142.5, 162.8, 193.4. HRMS (ESI, MH⁺) calculated for C₁₃H₁₈NO₂ 220.1332, found 220.1332.

(3Z)-4-Amino-3-phenylpent-3-en-2-one (5e). Reaction time, 6 hrs; Purification by Combi-Flash (SiO₂, gradient elution 20-50% EtOAc/hexanes) afforded the title compound as a white solid (65.4 mg, 92%). ¹H NMR (400 MHz, acetone-*d*₆): δ 10.56 (s, 1 H); 7.34 (t, *J* = 7.43 Hz, 2 H); 7.25 (t, *J* = 7.37 Hz, 1 H); 7.19-7.14 (m, 2 H); 6.63 (s, 1 H); 1.69 (s, 3 H); 1.66 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.4, 29.2, 109.9, 127.1, 129.1, 132.8, 142.1, 160.7, 195.1. HRMS (ESI, MH⁺) calculated for C₁₁H₁₄NO 176.1069, found 176.1070.

(2Z)-3-Amino-1, 2-diphenylbut-2-en-1-one (5f). Reaction time, 4 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-16% EtOAc/hexanes) afforded the title compound as a white solid (88.6 mg, 93%). ¹H NMR (400 MHz, acetone-*d*₆): δ 7.21-7.06 (m, 10 H); 1.92 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 22.8, 109.2, 126.6, 127.7, 128.6, 128.8, 133.8, 141.4, 144.3, 164.3. HRMS (ESI, MH⁺) calculated for C₁₆H₁₆NO 238.1225, found 238.1226.

3-Amino-2-phenylcyclopent-2-en-1-one (5g). Reaction time, 8 hrs; Purification by Combi-Flash (SiO₂, 5% MeOH/ EtOAc) afforded the title compound as a solid (57 mg,

86%) solid. ^1H NMR (500 MHz, CD_3OD): δ 7.39-7.31 (m, 4 H); 7.21 (t, J = 7.25 Hz, 1 H); 2.72-2.68 (m, 2 H); 2.50-2.46 (m, 2 H). ^{13}C NMR (126 MHz, CD_3OD): δ 27.4, 34.2, 127.4, 129.6, 129.8, 133.8, 204.2. HRMS (ESI, MH^+) calculated for $\text{C}_{11}\text{H}_{12}\text{NO}$ 174.0913, found 174.0913.

3-Amino-2-phenylcyclohex-2-en-1-one (5h). Reaction time, 7 hrs; Purification by Combi-Flash (SiO_2 , 5% MeOH/ EtOAc) afforded the title compound as a white solid (65.3 mg, 87%). ^1H NMR (500 MHz, CD_3OD): δ 7.37 (t, J = 7.62 Hz, 2 H); 7.25 (t, J = 7.47 Hz, 1 H); 7.12 (d, J = 7.53 Hz, 2 H); 2.58 (t, J = 6.30 Hz, 2 H); 2.40 (t, J = 6.45 Hz, 2 H); 2.01 (p, J = 6.36 Hz, 2 H). ^{13}C NMR (126 MHz, CD_3OD): δ 22.5, 29.9, 37.3, 112.6, 127.9, 129.8, 132.3, 136.2, 167.3, 196.6. HRMS (ESI, MH^+) calculated for $\text{C}_{12}\text{H}_{14}\text{NO}$, 188.1069 found 188.1070.

3-Anilino-2-phenylcyclohex-2-en-1-one (5i). Reaction time, 6 hrs; Purification by Combi-Flash (SiO_2 , gradient elution 10-100% EtOAc/hexanes) afforded the title compound as a white solid (80.8 mg, 77%). ^1H NMR (500 MHz, acetone- d_6): δ 7.12-7.37 (m, 10 H); 2.68 (m, 2 H); 2.39 (t, J = 6.26 Hz, 2 H); 2.02 (m, 2H). ^{13}C NMR (126 MHz, acetone- d_6): δ 22.7, 28.1, 38.0, 116.0, 125.7, 125.8, 127.3, 129.1, 129.7, 132.3, 136.3, 140.3, 159.2, 193.6. HRMS (ESI, MH^+) calculated for $\text{C}_{18}\text{H}_{18}\text{NO}$ 264.1380, found 264.1383.

5,5-Dimethyl-3-(methylamino)-2-phenylcyclohex-2-en-1-one (5j). Reaction time, 5 hrs; Purification by Combi-Flash (SiO_2 , gradient elution 10-100% EtOAc/hexanes) afforded the title compound as a white solid (53.2 mg, 58%). ^1H NMR (400 MHz, acetone- d_6): δ 7.31 (t, J = 7.47 Hz, 2 H); 7.20 (t, J = 7.32 Hz, 1 H); 7.11 (d, J = 7.44 Hz, 2 H); 5.49 (br s, 1H), 2.88 (s, 3H); 2.55 (s, 2 H); 2.19 (s, 2 H); 1.17 (s, 6 H). ^{13}C NMR (126 MHz, acetone- d_6): δ 28.7, 29.3, 32.1, 39.5, 51.2, 111.0, 126.8, 129.0, 132.5, 137.0, 161.1, 191.5. HRMS (ESI, MH^+) calculated for $\text{C}_{15}\text{H}_{20}\text{NO}$ 230.1538, found 230.1539.

Methyl (2Z)-3-amino-2-(1,3-benzodioxol-5-yl)but-2-enoate (7a). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-12% EtOAc/hexanes) afforded the title compound as a yellow oil (81.4 mg, 86%). ¹H NMR (400 MHz, acetone-*d*₆): δ 6.74-6.63 (m, 1 H); 6.58 (d, *J* = 1.52 Hz, 1 H); 6.53 (dd, *J* = 7.89, 1.65 Hz, 1 H); 5.97-5.86 (m, 2 H); 3.48-3.39 (m, 3 H); 1.73 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.1, 50.3, 97.0, 101.7, 108.2, 113.2, 126.7, 133.5, 146.7, 148.0, 160.2, 170.4. HRMS (ESI, MH⁺) calculated for C₁₂H₁₄NO₄ 236.0919, found 236.0917.

(3Z)-4-Amino-3-(1,3-benzodioxol-5-yl)pent-3-en-2-one (7b). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 10-50% EtOAc/hexanes) afforded the title compound as a brown oil (80.1 mg, 91%). ¹H NMR (500 MHz, acetone-*d*₆): δ 10.51 (br s, 1 H); 6.82 (d, *J* = 7.86 Hz, 1 H); 6.67 (s, 1 H); 6.63 (dd, *J* = 7.84, 1.76 Hz, 1 H); 5.99 (s, 2 H); 1.73 (s, 3H); 1.72 (s, 3H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.4, 29.0, 101.8, 108.8, 109.4, 112.9, 125.9, 135.7, 147.0, 148.5, 161.0, 195.4. HRMS (ESI, MH⁺) calculated for C₁₂H₁₄NO₃ 220.0967, found 220.0968.

Methyl (2Z)-3-amino-2-(2,3-dihydro-1H-inden-5-yl)but-2-enoate (7c). Reaction time, 8 hrs; Purification by Combi-Flash (SiO₂, gradient elution 2-15% EtOAc/hexanes) afforded the title compound as a white solid (61.1 mg, 66%). ¹H NMR (400 MHz, acetone-*d*₆): δ 7.11 (d, *J* = 7.65 Hz, 1 H); 6.95 (s, 1 H); 6.85 (d, *J* = 7.69 Hz, 1 H); 3.47 (s, 3 H); 2.87-2.81 (m, 4 H); 2.05-1.99 (m, 2H); 1.73 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.1, 26.1, 33.0, 33.2, 50.2, 97.6, 124.2, 128.4, 130.7, 137.2, 142.1, 144.0, 159.8, 170.5. HRMS (ESI, MH⁺) calculated for C₁₄H₁₈NO₂ 232.1330, found 232.1332.

(3Z)-4-Amino-3-(2,3-dihydro-1H-inden-5-yl)pent-3-en-2-one (7d). Reaction time, 6 hrs; Purification by Combi-Flash (SiO₂, gradient elution 5-25% EtOAc/hexanes) afforded the title compound as a white solid (69.1 mg, 80%). ¹H NMR (400 MHz, CDCl₃): δ 7.18 (d, *J* = 7.64 Hz, 1 H); 7.00 (s, 1 H); 6.90 (d, *J* = 7.64 Hz, 1 H); 2.91 (q, *J* = 6.48 Hz, 4 H); 2.09 (p, *J* = 7.44 Hz, 2 H); 1.86 (s, 3 H); 1.71 (s, 3 H). ¹³C NMR (126 MHz, CDCl₃): δ

22.3, 25.4, 32.6, 32.8, 110.4, 124.2, 127.7, 129.6, 138.1, 142.5, 144.5, 159.4, 197.3. HRMS (ESI, MH^+) calculated for $\text{C}_{14}\text{H}_{18}\text{NO}$ 216.1381, found 216.1383.

Methyl (2Z)-3-amino-2-(3,4-difluorophenyl)but-2-enoate (7e). Reaction time, 8 hrs; Purification by Combi-Flash (SiO_2 , gradient elution 0-15% EtOAc/hexanes) afforded the title compound as a white solid (69.5 mg, 77%). ^1H NMR (400 MHz, acetone- d_6): δ 8.68 (br s, 1 H); 7.19 (dt, $J = 10.93, 8.62$ Hz, 1 H); 7.06 (ddd, $J = 11.96, 7.98, 2.13$ Hz, 1 H); 6.95-6.90 (m, 1 H); 6.41 (br s, 1 H); 3.48 (s, 3 H); 1.76 (s, 3 H). ^{13}C NMR (126 MHz, acetone- d_6): δ 20.9, 50.4, 95.6, 117.0 (d, $^2J = 17.4$ Hz), 121.5 (d, $^2J = 15.4$ Hz), 129.5 (q, $^3J = 4.3$ Hz), 137.3, 148.2 (dd, $J = 248, 12$ Hz), 149.5 (dd, $J = 244, 13$ Hz), 160.5, 169.7. HRMS (ESI, MH^+) calculated for $\text{C}_{11}\text{H}_{12}\text{F}_2\text{NO}_2$ 228.0829, found 228.0831.

(3Z)-4-Amino-3-(3,4-difluorophenyl)pent-3-en-2-one (7f). Reaction time, 8 hrs; Purification by Combi-Flash (SiO_2 , gradient elution 10-50% EtOAc/hexanes) afforded the title compound as a white solid (73 mg, 87%). ^1H NMR (400 MHz, acetone- d_6): δ 10.57 (br s, 1 H); 7.35-7.23 (m, 1 H); 7.20-7.07 (m, 1 H); 7.02 (s, 1 H); 6.78 (br s, 1 H); 1.72 (s, 3 H); 1.71 (s, 3 H). ^{13}C NMR (126 MHz, acetone- d_6): δ 21.4, 29.1, 108.1, 117.8, 117.9 (d, $^2J = 17.9$ Hz), 121.6 (d, $^2J = 15.2$ Hz), 129.7 (q, $^3J = 3.2$ Hz), 139.5 (d, $^3J = 4.9$ Hz), 149.9 (dd, $J = 246, 12$ Hz), 150.6 (dd, $J = 248, 13$ Hz), 161.4. HRMS (ESI, MH^+) calculated for $\text{C}_{11}\text{H}_{12}\text{F}_2\text{NO}$ 212.0880, found 212.0882.

Methyl (2Z)-3-amino-2-(2,5-dimethylphenyl)but-2-enoate (7g). Reaction time, 7 hrs; Purification by Combi-Flash (SiO_2 , gradient elution 0-12% EtOAc/hexanes) afforded the title compound as a an oil (80.3 mg, 92%). ^1H NMR (400 MHz, acetone- d_6): δ 8.55 (s, 1 H); 7.01 (d, $J = 7.69$ Hz, 1 H); 6.89 (d, $J = 7.85$ Hz, 1 H); 6.80 (s, 1 H); 6.13 (br s, 1 H); 3.42 (s, 3 H); 2.20 (s, 3 H); 2.02 (s, 3 H); 1.58 (s, 3 H). ^{13}C NMR (126 MHz, Acetone): δ 19.5, 20.6, 20.9, 50.3, 96.0, 128.0, 130.1, 133.8, 135.4, 136.0, 138.8, 159.6, 170.1. HRMS (ESI, MH^+) calculated for $\text{C}_{13}\text{H}_{18}\text{NO}_2$ 220.1331, found 220.1332.

(3Z)-4-Amino-3-(2,5-dimethylphenyl)pent-3-en-2-one (7h). Reaction time, 7 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-12% EtOAc/hexanes) afforded the title compound as a clear oil (78.3 mg, 96%). ¹H NMR (500 MHz, acetone-*d*₆): δ 10.50 (s, 1 H); 7.09 (d, *J* = 7.71 Hz, 1 H); 6.97 (d, *J* = 7.85 Hz, 1 H); 6.89 (s, 1 H); 6.61 (br s, 1 H); 2.24 (s, 3 H); 2.06 (s, 3 H); 1.59 (s, 3 H); 1.56 (s, 3 H). ¹³C NMR (126 MHz, Acetone): δ 19.6, 20.9, 20.9, 28.6, 108.1, 128.5, 130.6, 133.6, 135.8, 136.0, 140.9, 160.4, 195.1. HRMS (ESI, MH⁺) calculated for C₁₃H₁₈NO 204.1382, found 204.1383.

Methyl (2Z)-3-amino-2-(9-phenanthryl)but-2-enoate (7i). Reaction time, 6 hrs; Purification by Combi-Flash (SiO₂, gradient elution 0-15% EtOAc/hexanes) afforded the title compound as a white solid (105.7 mg, 91%). ¹H NMR (500 MHz, acetone-*d*₆): δ 8.90 (br s, 1H); 8.85 (dd, *J* = 14.15, 8.22 Hz, 1 H); 7.92-7.88 (m, 2 H); 7.67-7.55 (m, 5 H); 6.50 (br s, 1H); 3.37 (s, 3 H); 1.67 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 20.7, 50.3, 94.1, 123.2, 123.7, 127.0, 127.0, 127.1, 127.3, 127.3, 129.5, 130.7, 130.8, 131.4, 133.1, 134.3, 135.7, 161.0, 170.5. HRMS (ESI, MH⁺) calculated for C₁₉H₁₈NO₂ 292.1333, found 292.1332.

(3Z)-4-Amino-3-(9-phenanthryl)pent-3-en-2-one (7j). Reaction time, 5 hrs; Purification by Combi-Flash (SiO₂, gradient elution 10-50% EtOAc/hexanes) afforded the title compound as a white solid (99.7 mg, 91%). ¹H NMR (400 MHz, acetone-*d*₆): δ 10.81 (br s, 1 H); 8.84 (dd, *J* = 18.04, 8.15 Hz, 2 H); 7.95 (dd, *J* = 14.84, 7.87 Hz, 2 H); 7.72-7.58 (m, 5 H); 6.90 (br s, 1 H); 1.66 (s, 3 H); 1.62 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.1, 28.6, 106.3, 123.4, 124.0, 126.8, 127.4, 127.4, 127.6, 127.7, 129.3, 130.8, 131.3, 131.7, 133.0, 133.9, 137.5, 161.9, 195.6. HRMS (ESI, MH⁺) calculated for C₁₉H₁₈NO 276.1383, found 276.1383.

Methyl (2Z)-3-amino-2-phenylbut-2-enoate-*d*₁ (3e-d). Reaction time, 6 hrs; Purification by Combi-Flash (SiO₂, gradient elution 2-15% EtOAc/hexanes) afforded the title compound as a white powder (61.6 mg, 80%). ¹H NMR (500 MHz, acetone-*d*₆): δ 7.26 (t, *J* = 6.85 Hz, 2 H); 7.18 (t, *J* = 7.39 Hz, 1 H); 7.11 (d, *J* = 7.70 Hz, 1 H); 3.46 (s, 3 H); 1.73-1.68 (m, 3 H). ²H NMR (92 MHz, 500 μL acetone + 2.5 μL acetone-*d*₆): δ 7.15 (s, 1H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 22.0, 50.3, 97.5, 126.5, 128.4 (d, *J* = 14 Hz), 132.9, 139.7, 159.9, 170.2. HRMS (ESI, MH⁺) calculated for C₁₁H₁₃DNO₂ 193.1081, found 193.1082.

(3Z)-4-Amino-3-phenylpent-3-en-2-one-*d*₁ (5e-d). Reaction time, 5 hrs; Purification by Combi-Flash (SiO₂, gradient elution 10-40% EtOAc/hexanes) afforded the title compound as a white powder (64.1 mg, 91%). ¹H NMR (400 MHz, acetone-*d*₆): δ 7.35 (t, *J* = 6.81 Hz, 2 H); 7.28-7.23 (m, 1 H); 7.20-7.16 (m, 1 H); 1.70 (s, 3 H); 1.67 (s, 3 H). ²H NMR (92 MHz, 500 μL acetone + 2.5 μL acetone-*d*₆): δ 7.23 (s, 1H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.4, 29.2, 109.9, 127.1, 129.1 (d, *J* = 14 Hz), 132.8, 142.1, 160.7, 195.1. HRMS (ESI, MH⁺) calculated for C₁₁H₁₃DNO 177.1132, found 177.1133.

Ethyl (2Z)-3-amino-3-(4-fluorophenyl)acrylate (2a). A mixture of 4-fluorobenzoyl acetic acid ethyl ester (1 g, 4.76 mmol) and NH₄OAc (1.83 g, 23.80 mmol) in MeOH (10 mL) was heated at 70 °C for 5 hrs. The mixture was cooled to room temperature and the solvent evaporated. The resulting residue was diluted with water (15 mL), extracted with EtOAc (3 x 15 mL) and the combined EtOAc fractions dried over Na₂SO₄. Evaporation of the solvent followed by purification by Combi-Flash (SiO₂, gradient elution 5-20% EtOAc/hexanes) afforded the title compound as an oil (662 mg, 67%). ¹H NMR (400 MHz, acetone-*d*₆): δ 8.12 (br s, 1 H); 7.69 (dd, *J* = 8.43, 5.33 Hz, 2 H); 7.21 (t, *J* = 8.60 Hz, 2 H); 6.48 (s, 1 H); 4.83 (s, 1 H); 4.09 (q, *J* = 7.09 Hz, 2 H); 1.22 (t, *J* = 7.10 Hz, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 14.9, 58.9, 83.8, 116.3 (d, ²*J* = 23 Hz), 129.5 (d, ³*J* = 10 Hz), 134.7, 160.8, 164.6 (d, ¹*J* = 248 Hz), 170.7. HRMS (ESI, MH⁺) calculated for C₁₁H₁₃FNO₂ 210.0925, found 210.0925.

Methyl (2Z)-3-amino-3-[4-(trifluoromethyl)phenyl]acrylate (2f). A mixture of methyl-4-trifluoromethyl benzoylacetate (1 g, 4.06 mmol) and NH_4OAc (1.6 g, 20.3 mmol) in MeOH (10 mL) was heated at 70 °C for 4 hr. The mixture was cooled to room temperature. The solid was filtered and washed with water then MeOH to give the title product as a white solid (987 mg, 99 %). ^1H NMR (400 MHz, acetone- d_6): δ 7.89-7.77 (m, 4 H); 4.92 (s, 1 H); 3.64 (s, 3 H). ^{13}C NMR (126 MHz, acetone- d_6): δ 50.3, 84.5, 126.4 (q, $^3J = 4$ Hz), 128.2, 132.2, 142.2, 160.4, 170.9. HRMS (ESI, MH^+) calculated for $\text{C}_{11}\text{H}_{11}\text{F}_3\text{NO}_2$ 246.0736, found 246.0736.

Ethyl (2Z)-3-amino-3-(3-furyl)acrylate (2h). A mixture of ethyl β -oxo-3-furanpropionate (1 g, 5.49 mmol) and NH_4OAc (2.12 g, 27.45 mmol) in MeOH (10 mL) was heated at 70 °C for 1 hr. The mixture was cooled to room temperature and the solvent was evaporated. The resulting residue was diluted with water (20 mL), extracted with EtOAc (3 x 20 mL) and the combined EtOAc fractions dried over Na_2SO_4 . Evaporation of the solvent followed by purification by Combi-Flash (SiO_2 , gradient elution 5-15% EtOAc/hexanes) afforded the title compound as an oil (702 mg, 71%). ^1H NMR (500 MHz, acetone- d_6): δ 8.10 (br s, 1 H); 7.63 (s, 1 H); 6.80 (s, 1 H); 4.91 (s, 1 H); 4.07 (q, $J = 7.08$ Hz, 2 H); 1.21 (t, $J = 7.13$ Hz, 3 H). ^{13}C NMR (126 MHz, acetone- d_6): δ 14.9, 58.8, 82.6, 109.0, 124.7, 142.6, 145.0, 153.7, 170.8. HRMS (ESI, MH^+) calculated for $\text{C}_9\text{H}_{12}\text{NO}_3$ 182.0811, found 182.0812.

(3Z)-4-Anilinopent-3-en-2-one (4a). To a solution of aniline (1.8 mL, 20 mmol) in H_2O (50 mL) was added 2,4-pentadione (1 g, 10 mmol) and the mixture stirred at room temperature. After 31 hrs, the reaction mixture was extracted with CH_2Cl_2 (3 x 15 mL). The combined CH_2Cl_2 fractions were washed with brine (20 mL) and dried over Na_2SO_4 . Evaporation of the solvent followed by purification by Combi-Flash (SiO_2 , gradient elution 0-3% EtOAc/hexanes) afforded the title compound as an oil (1.03 g, 59%). ^1H NMR (400 MHz, CDCl_3): δ 12.45 (br s, 1 H); 7.35-7.28 (m, 2 H); 7.17 (t, $J = 7.44$ Hz, 1 H); 7.09 (d, $J = 7.89$ Hz, 2 H); 5.17 (s, 1 H); 2.08 (s, 3 H); 1.97 (s, 3 H). ^{13}C NMR (126 MHz, CDCl_3): δ 19.9, 29.2, 97.6, 124.8, 125.6, 129.1, 138.8, 160.3, 196.2. HRMS (ESI, MH^+) calculated for $\text{C}_{11}\text{H}_{14}\text{NO}$ 176.1069, found 176.1070.

(3Z)-4-[(Cyclopropylmethyl)amino]pent-3-en-2-one (4b). To a solution of cyclopropanemethyl amine (1.73 g, 20 mmol) in H₂O (50 mL) was added 2,4-pentadione (1 g, 10 mmol) and the mixture stirred at room temperature. After 4 hrs, the reaction mixture was extracted with CH₂Cl₂ (3 x 15 mL). The combined CH₂Cl₂ fractions were washed with brine (20 mL) and dried over Na₂SO₄. Evaporation of the solvent followed by purification by Combi-Flash (SiO₂, gradient elution 1-15% EtOAc/hexanes) afforded the title compound as an oil (876 mg, 72%). ¹H NMR (500 MHz, acetone-*d*₆): δ 11.16-10.57 (br s, 1 H); 4.92 (s, 1 H); 3.16 (t, *J* = 6.29 Hz, 2 H); 1.92 (s, 3 H); 1.86 (s, 3 H); 1.06-0.96 (m, 1 H); 0.55-0.47 (m, 2 H); 0.25-0.20 (m, 2 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 3.5, 11.7, 18.8, 28.6, 47.7, 95.1, 163.1, 194.1. HRMS (ESI, MH⁺) calculated for C₉H₁₆NO 154.1226, found 154.1226.

(2Z)-3-Amino-1-phenylbut-2-en-1-one (4f). A mixture of 1-phenyl-1,3-butanedione (1 g, 6.17 mmol), NH₄OAc (2.38 g, 30.8 mmol) and MeOH (10 mL) was heated at 70 °C for 2 hrs. The mixture was cooled to room temperature. The solid was filtered and washed with water then MeOH to give the title product as a white solid (882 mg, 91%). ¹H NMR (400 MHz, acetone-*d*₆): δ 10.26 (s, 1 H); 7.90 (dd, *J* = 7.64, 1.84 Hz, 2 H); 7.46-7.37 (m, 3 H); 6.91 (s, 1 H); 5.77 (d, *J* = 1.85 Hz, 1 H); 2.06 (s, 3 H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 22.3, 91.6, 91.7, 127.8, 128.9, 131.3, 141.6, 188.6. HRMS (ESI, MH⁺) calculated for C₁₀H₁₂NO 162.0911, found 162.0913.

Methyl (2Z)-3-aminobut-2-enoate-*d*₃ (2e-*d*₃). Methyl-3-methylamino crotonate (0.4 g, 0.35 mmol) was dissolved in methyl alcohol-*d*₄ and heated at 50 °C for 15 hours. The solvent was evaporated and dried under high vacuum to give the title product as a solid (quantitative yield). ¹H NMR (400 MHz, acetone-*d*₆): δ 3.62 (s, 3 H); 2.01 (s, 3 H). ²H NMR (92 MHz, 500 μL acetone + 2.5 μL acetone-*d*₆): δ 7.89 (s, 1H), 6.14 (s, 1H), 4.43 (s, 1H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 20.8, 20.9, 48.9, 160.5, 169.9.

(3Z)-4-Aminopent-3-en-2-one-*d*₃ (4e-*d*₃). 4-Amino-3-pent-2-one (0.4 g, 4 mmol), was dissolved in methyl alcohol-*d*₄ and heated at 50 °C for 15 hours. The solvent was evaporated and the product dried under high vacuum to give the title product as a solid (quantitative yield). ¹H NMR (400 MHz, acetone-*d*₆): δ 1.87 (s, 3 H); 1.88 (s, 3 H). ²H NMR (92 MHz, 500 μL acetone + 2.5 μL acetone-*d*₆): δ 9.61 (s, 1H), 6.55 (s, 1H), 4.98 (s, 1H). ¹³C NMR (126 MHz, acetone-*d*₆): δ 21.4, 29.0, 95.1, 161.8, 195.3.

Observed proton nOe correlations for selected compounds

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carbon

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carbon

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carbon

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product

USER: ramtohum
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carbon

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product

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carbon

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before carbon

USER: ramtohul
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carbon nmr of product

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carbon

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product

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carbon

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7c

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7c

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 SOLVENT: CDCl3
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0084/3/fid
 product

7d

USER: ramtohu
 BOOK: 95729-0084
 EXPNO: 4
 ACQUIRED: 11-Jul-2006 13:34:40
 SOLVENT: CDCl3
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0084/4/fid

carbon

USER: ramtohu
 BOOK: 95729-0089
 EXPNO: 1
 ACQUIRED: 13-Jul-2006 16:51:06
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0089/1/fid

product

USER: ramtohu
 BOOK: 95729-0089
 EXPNO: 2
 ACQUIRED: 17-Jul-2006 13:04:32
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0089/2/fid

carbon

USER: ramtohui
 BOOK: 95729-0088
 EXPNO: 1
 ACQUIRED: 13-Jul-2006 13:25:37
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0088/1/fid
 product

USER: ramtohui
 BOOK: 95729-0088
 EXPNO: 2
 ACQUIRED: 13-Jul-2006 14:39:56
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0088/2/fid
 carbon

USER: ramtohui
 BOOK: 95729-0148
 EXPNO: 1
 ACQUIRED: 22-Aug-2006 14:18:53
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0148/1/fid
 product

USER: ramtohui
 BOOK: 95729-0148
 EXPNO: 2
 ACQUIRED: 22-Aug-2006 17:27:16
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0148/2/fid

carbon

USER: ramtohui
 BOOK: 95729-0147
 EXPNO: 1
 ACQUIRED: 23-Aug-2006 08:17:43
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0147/1/fid

product

USER: ramtohui
 BOOK: 95729-0147
 EXPNO: 2
 ACQUIRED: 23-Aug-2006 08:23:20
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0147/2/fid

carbon

USER: ramtohul
 BOOK: 95729-0104
 EXPNO: 3
 ACQUIRED: 24-Jul-2006 15:00:08
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohul/nmr/95729-0104/3/fid
 product

USER: ramtohul
 BOOK: 95729-0104
 EXPNO: 4
 ACQUIRED: 24-Jul-2006 15:13:38
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohul/nmr/95729-0104/4/fid
 carbon

USER: ramtohul
 BOOK: 95729-0092
 EXPNO: 2
 ACQUIRED: 17-Jul-2006 15:58:27
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohul/nmr/95729-0092/2/fid
 product

USER: trimble
 BOOK: 95729-0092
 EXPNO: 2
 ACQUIRED: 17-Jul-2006 16:15:16
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/trimble/nmr/95729-0092/2/fid

carbon

USER: ramtohu
 BOOK: 95729-0125
 EXPNO: 1
 ACQUIRED: 03-Aug-2006 11:17:51
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0125/1/fid

product

USER: trimble_71711
 BOOK: 71711-0141
 EXP: 95729_125_H2
 ACQUIRED: 11-Aug-2006 11:04:09
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/trimble_71711/nmr/71711-0141/95729_125_H2.fid

Inova 600 (Mahogany) - Cryoprobe
 2H spectrum of 95729-125 + 2.5 uL acetone-d6
 Yeeman Ramtohu

²H NMR

USER: ramtohui
 BOOK: 95729-0125
 EXPNO: 2
 ACQUIRED: 24-Aug-2006 14:35:59
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0125/2/fid

carbon

3e-d

USER: ramtohui
 BOOK: 95729-0110
 EXPNO: 1
 ACQUIRED: 26-Jul-2006 16:52:44
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohui/nmr/95729-0110/1/fid

product

5e-d

USER: trimble_71711
 BOOK: 71711-0141
 EXP: 95729_110_H2
 ACQUIRED: 11-Aug-2006 10:56:22
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/trimble_71711/nmr/71711-0141/95729_110_H2.fid

Inova 600 (Mahogany) - Cryoprobe
 2H spectrum of 95729-110 + 2.5 uL acetone-d6
 Yeeman Ramtohui

5e-d

²H NMR

USER: ramtohum
 BOOK: 95729-0110
 EXPNO: 2
 ACQUIRED: 24-Aug-2006 10:30:37
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohum/nmr/95729-0110/2/fid

carbon

USER: ramtohum
 BOOK: 95729-0080
 EXPNO: 4
 ACQUIRED: 10-Jul-2006 09:16:28
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohum/nmr/95729-0080/4/fid

product after purification

USER: ramtohum
 BOOK: 95729-0080
 EXPNO: 6
 ACQUIRED: 10-Jul-2006 11:52:04
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohum/nmr/95729-0080/6/fid

carbon

USER: ramtohum
 BOOK: 95729-0109
 EXPNO: 2
 ACQUIRED: 26-Jul-2006 15:20:32
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohum/nmr/95729-0109/2/fid
 product

USER: ramtohum
 BOOK: 95729-0109
 EXPNO: 6
 ACQUIRED: 27-Jul-2006 11:36:03
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohum/nmr/95729-0109/6/fid
 carbon

USER: ramtohum
 BOOK: 95729-0097
 EXPNO: 3
 ACQUIRED: 19-Jul-2006 11:22:37
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohum/nmr/95729-0097/3/fid
 product

USER: ramtohu
 BOOK: 95729-0097
 EXPNO: 5
 ACQUIRED: 20-Jul-2006 10:44:45
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0097/5/fid

carbon

USER: ramtohu
 BOOK: 95729-0127
 EXPNO: 1
 ACQUIRED: 08-Aug-2006 09:36:18
 SOLVENT: CDCl3
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0127/1/fid

product

USER: ramtohu
 BOOK: 95729-0127
 EXPNO: 2
 ACQUIRED: 08-Aug-2006 13:23:54
 SOLVENT: CDCl3
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0127/2/fid

carbon

USER: ramtohu
 BOOK: 95729-0124
 EXPNO: 3
 ACQUIRED: 04-Aug-2006 09:20:43
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0124/3/fid

4b

product

USER: ramtohu
 BOOK: 95729-0124
 EXPNO: 4
 ACQUIRED: 04-Aug-2006 09:26:39
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0124/4/fid

4b

carbon

USER: ramtohu
 BOOK: 95729-0064
 EXPNO: 2
 ACQUIRED: 22-Jun-2006 14:24:57
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0064/2/fid

4f

product

USER: ramtohul
 BOOK: 95729-0064
 EXPNO: 5
 ACQUIRED: 22-Jun-2006 16:51:35
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohul/nmr/95729-0064/5/fid
 carbon- 100 scans

USER: ramtohul
 BOOK: 95729-0120
 EXPNO: 1
 ACQUIRED: 31-Jul-2006 16:40:25
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohul/nmr/95729-0120/1/fid
 after 2 X MeOD

USER: trimble_71711
 BOOK: 71711-0141
 EXP: 95729_120_H2
 ACQUIRED: 11-Aug-2006 11:00:11
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/trimble_71711/nmr/71711-0141/95729_120_H2.fid
 Inova 600 (Mahogany) - Cryoprobe
 2H spectrum of 95729-120 + 2.5 uL acetone-d6
 Yeeman Ramtohul

USER: ramtohu
 BOOK: 95729-0120
 EXPNO: 3
 ACQUIRED: 24-Aug-2006 14:25:41
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0120/3/fid

product

USER: ramtohu
 BOOK: 95729-0106
 EXPNO: 3
 ACQUIRED: 26-Jul-2006 10:51:11
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/nmr/95729-0106/3/fid

after overnight

USER: trimble_71711
 BOOK: 71711-0141
 EXP: 95729_106_H2
 ACQUIRED: 11-Aug-2006 10:48:13
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/trimble_71711/nmr/95729_106_H2.fid

Inova 600 (Mahogany) - Cryoprobe
 2H spectrum of 95729-106 + 2.5 uL acetone-d6
 Yeeman Ramtohu

USER: ramtohu
 BOOK: 95729-0106
 EXPNO: 4
 ACQUIRED: 24-Aug-2006 10:20:38
 SOLVENT: Acetone
 FILENAME: /nmr_archive/data/ramtohu/95729-0106/4/fid

carbon

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- ⁴ Sato, Y.; Tamura, T.; Mori, M. *Angew. Chem. Int. Ed.* **2004**, 43, 2436-2440.
- ⁵ Yoshida, H.; Terayama, T.; Ohshita, J.; Kunai, A. *Chem. Commun.* **2004**, 1980-1981.