

Supporting Information

Non-enzymatic Acylative Kinetic Resolution of Primary Allylic Amines

Amandine Kolleth, Martin Cattoen, Stelios Arseniyadis,* and Janine Cossy*

Laboratoire de Chimie Organique, ESPCI ParisTech, CNRS, 10 rue Vauquelin, 75231 Paris Cedex 05, France.

Tel: +33 (0)1 40 79 46 62; Fax: +33 (0)1 40 79 46 60

E-mail: stelios.arseniyadis@espci.fr; E-mail: janine.cossy@espci.fr

General: The reactions were run under argon atmosphere in oven-dried glassware unless otherwise specified. Dichloromethane was distilled from calcium hydride. THF and Et₂O were distilled from sodium/benzophenone. Analytical thin layer chromatography (TLC) was performed on silica gel plates (Merck 60F₂₅₄) visualized either with a UV lamp (254 nm) or by using solutions of *p*-anisaldehyde/sulfuric acid/acetic acid in EtOH or KMnO₄/K₂CO₃/AcOH in water followed by heating. Flash chromatographies were performed on silica gel (60-230 mesh mesh). All the reactions were carried out under Ar atmosphere. Organic extracts were dried over anhydrous Na₂SO₄. Infrared spectra (IR) were recorded on a Bruker TENSOR™ 27 (IRTF) and wave-numbers are indicated in cm⁻¹. ¹H NMR spectra were recorded on a Bruker AVANCE 400 at 400 MHz in CDCl₃ and data are reported as follows: chemical shift in parts per million from tetramethylsilane as an internal standard, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of nonequivalent resonances), integration. ¹³C NMR spectra were recorded at 100 MHz in CDCl₃ (unless otherwise specified) and data were reported as follows: chemical shift in parts per million from tetramethylsilane with the solvent as an internal indicator (CDCl₃ δ 77.0 ppm), multiplicity with respect to proton (deduced from DEPT experiments, s = quaternary C, d = CH, t = CH₂, q = CH₃). Mass spectra (MS) were recorded using a Hewlett-Packard tandem 5890A/5971 GCMS (70 eV). High-Resolution Mass Spectra were performed by "Groupe de Spectrométrie de masse de l'Université Pierre et Marie Curie (Paris)". The enantiomeric excesses were determined by supercritical fluid chromatography (SFC) analysis on chiral phase.

General procedure for the synthesis of the allylic alcohol precursors (Procedure A):

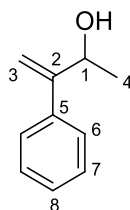
Magnesium chips (6.14 mmol, 1.1 equiv.) and one crystal of I₂ were added in THF (2 mL) followed by a few drops of vinyl bromide in order to initiate the reaction. Once discoloration of the medium occurred, the rest of the THF was added followed by the vinyl bromide (5.58 mmol, 1 equiv.), which was added slowly. After stirring for 1 h at room temperature, the medium was cooled to 0 °C and a solution of aldehyde (8.37 mmol, 1.5 equiv.) in THF (5 mL) was added. The mixture was stirred for an additional 30 min at the same temperature before a saturated aqueous solution of NH₄Cl (50 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with Et₂O (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel to afford the pure allylic alcohol.

General procedure for the synthesis of the phthalimide precursors (Procedure B): To a solution of allylic alcohol (4.65 mmol, 1 equiv.) in THF (50 mL) at 0 °C were added triphenylphosphine (5.1 mmol, 1.1 equiv.), phthalimide (5.1 mmol, 1.1 equiv.) and a solution of DEAD (40 % wt in toluene, 6.0 mmol, 1.3 equiv.). The reaction mixture was then stirred for 20 h at room temperature before the solvent was evaporated and the triphenylphosphine oxide precipitated by addition of a 1:1 mixture of Et₂O/PE (40 mL). The precipitate was filtered through a plug of Celite[®] and the solvent was evaporated under reduced pressure to afford a crude residue which was purified by flash column chromatography over silica gel.

General procedure for the cleavage of the phthalimide (Procedure C): To a solution of phthalimide (3 mmol, 1 equiv.) in EtOH (26 mL) was added hydrazine hydrate (18 mmol, 6 equiv.) drop-wise and the resulting mixture was stirred at reflux for 3 h. The formed pasty precipitate was then filtered through a plug of Celite[®], washed with Et₂O (20 mL) and the solvent was evaporated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel to afford the desired pure allylic amine.

General procedure for the kinetic resolution of allylic amines (Procedure D): To a solution of allylic amine (0.142 mmol, 1 equiv.) in THF (3.4 mL) at -20 °C was added the supported salt (319 mg, 2 equiv.). A solution of (1*S*,2*S*)-1 (0.071 mmol, 0.5 equiv.) in THF (0.6 mL) was then slowly added during a period of 1 h using a syringe pump and the resulting mixture was stirred overnight at the same temperature. Filtration of the resin followed by evaporation of the solvent and purification of the crude residue by flash column chromatography over silica gel gave the corresponding enantio-enriched acetamide along with the unreacted and enantio-enriched allylic amine.

3-Phenylbut-3-en-2-ol¹

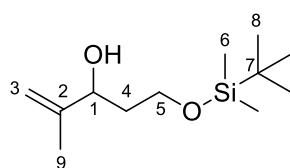


MW (g/mol): 148.20

Molecular formula: C₁₀H₁₂O

This compound was prepared following general procedure A. **IR** (neat): 3437, 2930, 1715, 1600, 1449, 1274, 1070, 761, 702 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.46–7.28 (m, 5H, H_{Ph}), 5.39 (t_{app}, J = 1.3 Hz, 1H, H₃), 5.30 (s, 1H, H₃), 4.84 (q, J = 6.1 Hz, 1H, H₁), 1.92 (s, 1H, OH), 1.34 (d, J = 6.4 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 153.2 (s, C₂), 140.0 (s, C₅), 128.5 (d, C_{Ph}), 127.8 (d, C_{Ph}), 126.9 (d, C_{Ph}), 111.7 (t, C₃), 69.60 (d, C₁), 22.7 (q, C₄).

5-((*tert*-Butyldimethylsilyl)oxy)-2-methylpent-1-en-3-ol



MW (g/mol): 230.42

Molecular formula: C₁₂H₂₆O₂Si

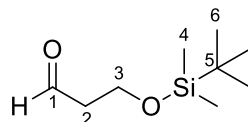
To a solution of 3-((*tert*-butyldimethylsilyl)oxy)propanal (1.0 g, 5.3 mmol, 1.0 equiv.) in THF (10 mL) at 0 °C was added a solution of isopropenylmagnesium bromide (0.5M in THF, 12 mL, 6 mmol, 1.1 equiv.) drop-wise. After stirring for 30 min at the same temperature, a saturated aqueous solution of NH₄Cl (20 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with Et₂O (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (Petroleum ether/EtOAc = 90:10) to afford the pure 5-((*tert*-butyldimethylsilyl)oxy)-2-methylpent-1-en-3-ol in 91% yield.

IR (neat): 3421, 2953, 2929, 2857, 1472, 1254, 1084, 898, 832, 774 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.02 (m, 1H, H₃), 4.85 (m, 1H, H₃), 4.26 (dd, J = 7.6, 3.8 Hz, 1H, H₁), 3.88 (m, 1H, H₅),

¹ García Ruano, L. G.; Fernández-Ibáñez, M. A.; Fernández-Salas, J. A.; Maestro, M. C.; Márquez-López, P.; Rodríguez-Fernández, M. M. *J. Org. Chem.*, **2009**, 74, 1200.

3.80 (m, 1H, H₅), 1.84–1.75 (m, 2H, H₄), 1.73 (s, 3H, H₉), 0.91 (s, 9H, H₈), 0.08 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 147.2 (s, C₂), 110.5 (t, C₃), 75.3 (d, C₁), 62.3 (t, C₅), 36.8 (t, C₄), 26.0 (q, C₈), 18.5 (q, C₉), 18.3 (s, C₇), –5.4 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₂H₂₆NaO₂Si [M + Na]⁺: 253.1594, found: 253.1595.

3-((*tert*-Butyldimethylsilyl)oxy)propanal²

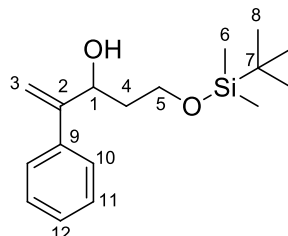


MW (g/mol): 188.34

Molecular formula: C₉H₂₀O₂Si

IR (neat): 2955, 2930, 2857, 1726, 1472, 1254, 1096, 832, 775 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 9.78 (t, J = 2.3 Hz, 1H, H₁), 3.97 (t, J = 6.0 Hz, 2H, H₃), 2.57 (td, J = 6.0, 2.1 Hz, 2H, H₂), 0.86 (s, 9H, H₆), 0.05 (s, 6H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 202.1 (d, C₁), 57.5 (t, C₃), 46.6 (t, C₂), 25.9 (q, C₆), 18.3 (s, C₅), –5.4 (q, C₄).

5-((*tert*-Butyldimethylsilyl)oxy)-2-phenylpent-1-en-3-ol³



MW (g/mol): 292.49

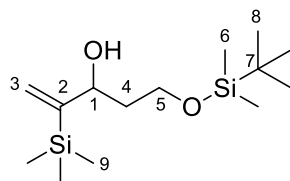
Molecular formula: C₁₇H₂₂O₂Si

This compound was prepared following general procedure A. **IR** (neat): 3431, 2954, 2928, 2857, 1471, 1254, 1077, 834, 774, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.40–7.24 (m, 5H, H_{Ph}), 5.48 (t_{app}, J = 1.6 Hz, 1H, H₃), 5.35 (s, 1H, H₃), 4.93–4.87 (m, H₁), 3.90–3.81 (m, 2H, OH + H₅), 3.76 (ddd, J = 10.2, 7.3, 3.8 Hz, 1H, H₅), 1.85 (m, 1H, H₄), 1.64 (m, 1H, H₄), 0.92 (s, 9H, H₈), 0.08 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 151.2 (s, C₂), 140.4 (s, C₉), 128.5 (d, C_{Ph}), 127.6 (d, C_{Ph}), 126.9 (d, C_{Ph}), 112.7 (t, C₃), 73.4 (d, C₁), 62.3 (t, C₅), 37.0 (t, C₄), 26.0 (q, C₈), 18.3 (s, C₇), –5.5 (q, C₆), –5.4 (q, C₆).

² Smith, A. B.; Fox, R. J.; Vanecko, J. A. *Org. Lett.*, **2005**, 7, 3099.

³ Zhang, D.; Ready, J. M. *J. Am. Chem. Soc.*, **2007**, 129, 12088.

5-((*tert*-Butyldimethylsilyl)oxy)-2-(trimethylsilyl)pent-1-en-3-ol

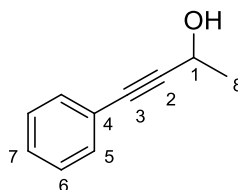


MW (g/mol): 288.57

Molecular formula: C₁₄H₃₂O₂Si₂

This compound was prepared following general procedure A. **IR** (neat): 3352, 2954, 2858, 1407, 1248, 1052, 934, 833, 757 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.86 (dd, *J* = 2.7, 1.5 Hz, 1H, H₃), 5.44 (dd, *J* = 2.7, 1.2 Hz, 1H, H₃), 4.53 (m, 1H, H₁), 3.87 (m, 1H, H₅), 3.81 (m, 1H, H₅), 3.35 (s, 1H, OH), 1.85–1.62 (m, 2H, H₄), 0.91 (s, 9H, H₈), 0.13 (s, 9H, H₉), 0.08 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 154.8 (s, C₂), 123.7 (t, C₃), 75.4 (d, C₁), 62.4 (t, C₅), 39.1 (t, C₄), 26.0 (q, C₈), 18.3 (s, C₇), -0.5 (q, C₉), -5.4 (q, C₆). **HRMS (ESI)** *m/z*: calcd for C₁₄H₃₂NaO₂Si₂ [M + Na]⁺: 311.1833, found: 311.1832.

4-Phenylbut-3-yn-2-ol⁴



MW (g/mol): 146.18

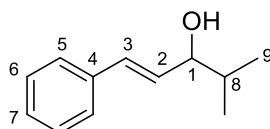
Molecular formula: C₁₀H₁₀O

To a solution of ethynylbenzene (10 mmol, 1 equiv.) in THF (28 mL) at -78 °C was added a solution of *n*-butyllithium (2.5 M in THF, 4 mL, 10 mmol, 1 equiv.) drop-wise. After stirring for 30 min at the same temperature, the acetaldehyde (12 mmol, 1.2 equiv.) was added and the mixture was stirred for an additional 30 min before a saturated aqueous solution of NH₄Cl (50 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with EtOAc (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel to afford the pure propargyl alcohol in 72% yield.

⁴ Rigby, J. H.; Laurent, S. B.; Kamal, Z.; Heeg, M. J. *Org. Lett.*, **2008**, *10*, 5609.

¹H NMR (400 MHz, CDCl₃) δ 7.43–7.36 (m, 2H, H_{Ph}), 7.30–7.22 (m, 3H, H_{Ph}), 4.73 (qd, J = 6.6, 1.7 Hz, 1H, H_I), 2.37 (s, 1H, OH), 1.52 (dd, J = 6.6, 1.8 Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 131.7 (d, C_{Ph}), 128.5 (d, C_{Ph}), 128.4 (d, C_{Ph}), 122.7 (s, C₄), 91.1 (s, C₂), 84.1 (s, C₃), 58.9 (d, C₁), 24.4 (q, C₈).

(*E*)-4-Methyl-1-phenylpent-1-en-3-ol⁵



MW (g/mol): 176.25

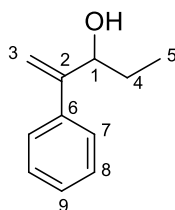
Molecular formula: C₁₂H₁₆O

To a solution of cinnamaldehyde (1.5 mL, 12 mmol, 1.5 equiv.) in THF (80 mL) at 0 °C was added isopropylmagnesium chloride solution (1.3M in THF, 2.6 mL, 8 mmol, 1.0 equiv.) drop-wise. After stirring for 30 min at the same temperature, a saturated aqueous solution of NH₄Cl (70 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with Et₂O (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (Petroleum ether/EtOAc = 90:10) to afford the pure (*E*)-4-methyl-1-phenylpent-1-en-3-ol in 71% yield.

IR (neat): 33423, 2962, 2931, 2874, 1720, 1610, 1451, 1264, 969, 750, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.53–7.13 (m, 5H, H_{Ph}), 6.59 (d, J = 15.9 Hz, 1H, H₃), 6.25 (dd, J = 15.9, 7.0 Hz, 1H, H₂), 4.05 (ddd, J = 7.1, 6.0, 1.2 Hz, 1H, H_I), 1.85 (m, 1H, H₈), 1.70 (brs, 1H, OH), 1.01 (d, J = 6.8 Hz, 3H, H₉), 0.97 (d, J = 6.8 Hz, 3H, H₉). **¹³C NMR** (100 MHz, CDCl₃) δ 137.0 (s, C₄), 131.3 (d, C₃ or C₂), 131.0 (d, C₃ or C₂), 128.7 (d, C_{Ph}), 127.7 (d, C_{Ph}), 126.6 (d, C_{Ph}), 78.3 (d, C₁), 34.2 (d, C₈), 18.5 (q, C₉), 18.2 (q, C₉).

⁵ Liu, Z. Q.; Sun, L.; Wang, J. G.; Han, J.; Zhao, Y. K.; Zhou, B. *Org. Lett.*, **2009**, *11*, 1437.

2-Phenylpent-1-en-3-ol

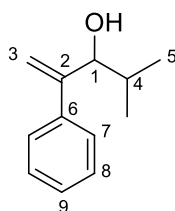


MW (g/mol): 162.23

Molecular formula: C₁₁H₁₄O

This compound was prepared following general procedure A. **IR** (neat): 3412, 2965, 2934, 1702, 1493, 1449, 978, 907, 778, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.52–7.27 (m, 5H, H_{Ph}), 5.34 (d, J = 14.9 Hz, 2H, H₃), 4.59 (m, 1H, H₁), 1.80–1.61 (m, 2H, OH + H₄), 1.53 (dq_{int}_{app}, J = 14.3, 7.3 Hz, 1H, H₄), 0.93 (t, J = 7.4 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 151.9 (s, C₂), 140.2 (s, C₆), 128.5 (d, C_{Ph}), 127.7 (d, C_{Ph}), 127.1 (d, C_{Ph}), 112.9 (t, C₃), 75.3 (d, C₁), 28.9 (t, C₄), 9.9 (q, C₅). **HRMS (ESI)** m/z : calcd for C₁₁H₁₅O [M + H]⁺: 163.1117, found: 163.1114.

4-Methyl-2-phenylpent-1-en-3-ol⁶



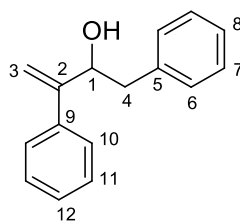
MW (g/mol): 176.25

Molecular formula: C₁₂H₁₆O

This compound was prepared following general procedure A. **IR** (neat): 3435, 2961, 2871, 1493, 1009, 909, 776, 697 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.56–7.15 (m, 5H, H_{Ph}), 5.34 (s, 2H, H₃), 4.42 (d, J = 5.2 Hz, 1H, H₁), 1.88–1.69 (m, 2H, OH + H₄), 0.93 (d, J = 6.8 Hz, 3H, H₅), 0.89 (d, J = 6.7 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 151.4 (s, C₂), 140.6 (s, C₆), 128.4 (d, C_{Ph}), 127.6 (d, C_{Ph}), 127.2 (d, C_{Ph}), 113.8 (t, C₃), 79.2 (d, C₁), 31.5 (d, C₄), 19.9 (q, C₅), 16.3 (q, C₅).

⁶ García Ruano, L. G.; Fernández-Ibáñez, M. A.; Fernández-Salas, J. A.; Maestro, M. C.; Márquez-López, P.; Rodríguez-Fernández, M. M. *J. Org. Chem.*, **2009**, *74*, 1200.

1,3-Diphenylbut-3-en-2-ol⁷

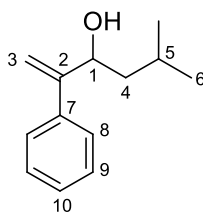


MW (g/mol): 224.30

Molecular formula: C₁₆H₁₆O

This compound was prepared following general procedure A. **IR** (neat): 3413, 3027, 1494, 1051, 1027, 908, 748, 696 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.51–7.19 (m, 10H, H_{Ph}), 5.40 (s, 1H, H₃), 5.37 (s, 1H, H₃), 4.89 (dd, J = 8.6, 3.6 Hz, 1H, H₁), 2.99 (dd, J = 13.9, 3.7 Hz, 1H, H₄), 2.72 (dd, J = 13.9, 8.6 Hz, 1H, H₄), 1.91 (s, 1H, OH). **¹³C NMR** (100 MHz, CDCl₃) δ 151.1 (s, C₂), 140.0 (s, C₉), 138.3 (s, C₅), 129.6 (d, C_{Ph}), 128.6 (d, C_{Ph}), 128.6 (d, C_{Ph}), 127.9 (d, C_{Ph}), 127.1 (d, C_{Ph}), 126.7 (d, C_{Ph}), 113.2 (t, C₃), 74.5 (d, C₁), 42.9 (t, C₄).

5-Methyl-2-phenylhex-1-en-3-ol



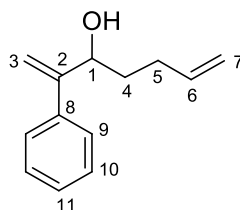
MW (g/mol): 190.28

Molecular formula: C₁₃H₁₈O

This compound was prepared following general procedure A. **IR** (neat): 3343, 2955, 2928, 1467, 1059, 898, 771, 712, 693 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.48–7.25 (m, 5H, H_{Ph}), 5.37 (t_{app}, J = 1.3 Hz, 1H, H₃), 5.30 (dd, J = 1.3, 0.6 Hz, 1H, H₃), 4.70 (dd, J = 8.7, 3.7 Hz, 1H, H₁), 1.84 (m, 1H, H₅), 1.65 (s, 1H, OH), 1.51–1.31 (m, 2H, H₄), 0.92 (d, J = 6.6 Hz, 3H, H₆), 0.90 (d, J = 6.7 Hz, 3H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 152.8 (s, C₂), 140.1 (s, C₇), 128.5 (d, C_{Ph}), 127.7 (d, C_{Ph}), 127.0 (d, C_{Ph}), 112.5 (t, C₃), 72.3 (d, C₁), 45.7 (t, C₄), 25.0 (d, C₅), 23.5 (q, C₆), 21.9 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₃H₁₈NaO [M + Na]⁺: 213.1250, found: 213.1251.

⁷ Kong, W.; Guo, B.; Fu, C.; Ma, S. *Eur. J. Org. Chem.* **2011**, 2278.

2-Phenylhepta-1,6-dien-3-ol⁸

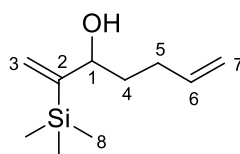


MW (g/mol): 188.27

Molecular formula: C₁₃H₁₆O

This compound was prepared following general procedure A. **IR** (neat): 3369, 3078, 2921, 1639, 1493, 1443, 1061, 1025, 907, 777, 967 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.53–7.28 (m, 5H, H_{Ph}), 5.81 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H, H₆), 5.38 (s, 1H, H₃), 5.33 (s, 1H, H₃), 5.03 (dd, J = 17.1, 1.6 Hz, 1H, H₇), 4.97 (d, J = 10.2 Hz, 1H, H₇), 4.67 (dd, J = 7.8, 4.3 Hz, 1H, H₁), 2.35–2.05 (m, 2H, H₅), 1.81–1.67 (m, 2H, OH + H₄), 1.60 (m, 1H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 152.1 (s, C₂), 140.1 (s, C₈), 138.4 (d, C₆), 128.5 (d, C_{Ph}), 127.8 (d, C_{Ph}), 127.1 (d, C_{Ph}), 115.1 (t, C₇), 112.8 (t, C₃), 73.4 (d, C₁), 35.2 (t, C₄), 30.1 (t, C₅).

2-(Trimethylsilyl)hepta-1,6-dien-3-ol 2-(trimethylsilyl)hepta-1,6-dien-3-ol



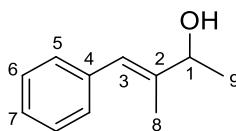
MW (g/mol): 184.35

Molecular formula: C₁₀H₂₀OSi

This compound was prepared following general procedure A. **IR** (neat): 2955, 1249, 1054, 912, 838, 618 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.87 (m, 1H, H₆), 5.79 (dd, J = 2.5, 1.3 Hz, 1H, H₃), 5.43 (dd, J = 2.5, 1.0 Hz, 1H, H₃), 5.05 (dq app, J = 17.1, 1.8 Hz, 1H, H₇), 4.99 (m, 1H, H₇), 4.31 (dd, J = 7.9, 4.7 Hz, 1H, H₁), 2.31–1.96 (m, 2H, H₅), 1.76–1.53 (m, 3H, H₄ + OH), 0.15 (s, 9H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 155.4 (s, C₂), 138.6 (d, C₆), 124.1 (t, C₃), 115.0 (t, C₇), 75.8 (d, C₁), 36.5 (t, C₄), 30.3 (t, C₅), -0.4 (q, C₈). **HRMS (ESI)** m/z : calcd for C₁₀H₂₀NaOSi [M + Na]⁺: 207.1176, found: 207.1175.

⁸ Kano, S.; Yokomatsu, T.; Yuasa, Y.; Shibuya, S. *J. Org. Chem.*, **1985**, *50*, 3449.

(*E*)-3-Methyl-4-phenylbut-3-en-2-ol⁹



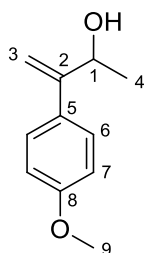
MW (g/mol): 162.23

Molecular formula: C₁₁H₁₄O

To a solution of α -methyl-trans-cinnamaldehyde (0.56 mL, 4 mmol, 1.0 equiv.) in THF (40 mL) at 0 °C was added a solution of MeMgBr (3M in Et₂O, 2.6 mL, 8 mmol, 2 equiv.) drop-wise. After stirring for 30 min at the same temperature, a saturated aqueous solution of NH₄Cl (50 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with Et₂O (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (Petroleum ether/EtOAc = 85:15) to afford the pure (*E*)-3-methyl-4-phenylbut-3-en-2-ol in quantitative yield.

IR (neat): 3392, 2975, 1704, 1656, 1444, 1368, 1072, 917, 744, 697 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.40–7.13 (m, 5H, H_{Ph}), 6.51 (s, 1H, H₃), 4.37 (q, *J* = 6.4 Hz, 1H, H₁), 1.87 (d, *J* = 1.3 Hz, 3H, H₈), 1.76 (s, 1H, OH), 1.36 (d, *J* = 6.4 Hz, 3H, H₉). **¹³C NMR** (100 MHz, CDCl₃) δ 141.7 (s, C₂ or C₄), 137.8 (s, C₂ or C₄), 129.1 (d, C_{Ph}), 128.2 (d, C_{Ph}), 126.5 (d, C_{Ph}), 124.5 (d, C₃), 73.8 (d, C₁), 21.9 (q, C₉), 13.5 (q, C₈).

3-(4-Methoxyphenyl)but-3-en-2-ol



MW (g/mol): 178.23

Molecular formula: C₁₁H₁₄O₂

Methyl lithium (3.2 mL, 1.6M, 5.0mmol) was added to a solution of butynol (393 μ L, 5.0 mmol, 1 equiv.) in THF (15 mL) at -78 °C. After stirring for 20 min at the same temperature, the solution was warmed up to rt and ready for use. During this time, ZnCl₂ (4.1 g, 30 mmol, 6.0 equiv.) was weighed to another flask and fused under vacuum. After the flask cooled to rt, Cp₂Zr(H)Cl (2.5 g,

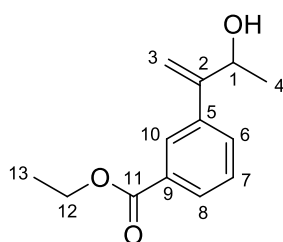
⁹ Liu, Z. Q.; Sun, L.; Wang, J. G.; Han, J.; Zhao, Y. K.; Zhou, B. *Org. Lett.*, **2009**, *11*, 1437.

10 mmol, 2.0 equiv.) and THF (10 mL) were added sequentially. The resulting mixture was stirred until all $\text{Cp}_2\text{Zr(H)Cl}$ dissolved (solid ZnCl_2 remained suspended). The prepared solution of alkoxide was then transferred via cannula into the mixture of ZnCl_2 and $\text{Cp}_2\text{Zr(H)Cl}$ in THF, followed by rinsing with THF (5 mL). The result clear solution was stirred for 2h and gave a mixture with some gray precipitate.

After the hydrozirconation, CH_3CN (1mL) and 4-iodoanisole (3.51 g, 15 mmol, 3 equiv.) were added. After 10 min, $\text{Pd(PPh}_3)_4$ (580 mg, 0.50 mmol, 0.1 equiv.) in THF (10 mL) was added and the reaction was stirred overnight. The reaction was quenched with aqueous NH_4Cl and extracted with Et_2O . The combined organic phases were dried over MgSO_4 , concentrated and purified by flash chromatography on silica gel to give the pure product (44% yield).

IR (neat): 3404, 2974, 2933, 1607, 11510, 1243, 1179, 1029, 833 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.35 (d, J = 8.9 Hz, 2H, H_6), 6.89 (d, J = 8.9 Hz, 2H, H_7), 5.30 (t_{app} , J = 1.2 Hz, 1H, H_3), 5.23 (m, 1H, H_3), 4.81 (q, J = 6.3 Hz, 1H, H_1), 3.83 (s, 3H, H_9), 1.74 (brs, 1H, OH), 1.34 (d, J = 6.4 Hz, 3H, H_4). **^{13}C NMR** (100 MHz, CDCl_3) δ 159.3 (s, C_8), 152.5 (s, C_2), 132.3 (s, C_5), 128.0 (d, C_6), 113.9 (d, C_7), 110.4 (t, C_3), 69.7 (d, C_1), 55.4 (q, C_9), 22.7 (q, C_4). **HRMS (ESI) m/z** : calcd for $\text{C}_{11}\text{H}_{15}\text{O}_2$ [$\text{M} + \text{H}$] $^+$: 179.1067, found: 179.1065.

Ethyl 3-(3-hydroxybut-1-en-2-yl)benzoate



MW (g/mol): 220.26

Molecular formula: $\text{C}_{13}\text{H}_{16}\text{O}_3$

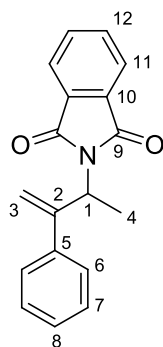
Methyl lithium (3.2 mL, 1.6M, 5.0mmol) was added to a solution of butynol (393 μL , 5.0 mmol, 1 equiv.) in THF (15 mL) at -78°C . After stirring for 20 min at the same temperature, the solution was warmed up to rt and ready for use. During this time, ZnCl_2 (4.1 g, 30 mmol, 6.0 equiv.) was weighed to another flask and fused under vacuum. After the flask cooled to rt, $\text{Cp}_2\text{Zr(H)Cl}$ (2.5 g, 10 mmol, 2.0 equiv.) and THF (10 mL) were added sequentially. The resulting mixture was stirred until all $\text{Cp}_2\text{Zr(H)Cl}$ dissolved (solid ZnCl_2 remained suspended). The prepared solution of alkoxide was then transferred via cannula into the mixture of ZnCl_2 and $\text{Cp}_2\text{Zr(H)Cl}$ in THF, followed by

rinsing with THF (5 mL). The result clear solution was stirred for 2h and gave a mixture with some gray precipitate.

After hydrozirconation, CH₃CN (1mL) and ethyl 3-iodobenzoate (4.14 g, 15 mmol, 3 equiv.) were added. After 10 min, Pd(PPh₃)₄ (580 mg, 0.50 mmol, 0.1 equiv.) in THF (10 mL) was added and the reaction was stirred overnight. The reaction was quenched with aqueous NH₄Cl and extracted with Et₂O. The combined organic phases were dried over MgSO₄, concentrated and purified by flash chromatography on silica gel to give the pure product (26% yield).

IR (neat): 3429, 2979, 1718, 1368, 11296, 1246, 1137, 1024, 910, 764 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 8.07 (td, J = 1.8, 0.5 Hz, 1H, H_{Ar}), 7.98 (ddd, J = 7.8, 1.7, 1.2 Hz, 1H, H_{Ar}), 7.60 (ddd, J = 7.7, 1.9, 1.2 Hz, 1H, H_{Ar}), 7.42 (td, J = 7.7, 0.5 Hz, 1H, H_{Ar}), 5.44 (m, 1H, H₃), 5.34 (m, 1H, H₃), 4.86 (q, J = 6.8 Hz, 1H, H₁), 4.40 (q, J = 7.1 Hz, 2H, H₁₂), 1.72 (brs, 1H, OH), 1.41 (t, J = 7.1 Hz, 3H, H₁₃), 1.33 (d, J = 6.4 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 166.7 (s, C₁₁), 152.4 (s, C₂), 140.3 (s, C₅), 131.4 (d, C_{Ar}), 130.8 (s, C₉), 128.9 (d, C_{Ar}), 128.6 (d, C_{Ar}), 128.1 (d, C_{Ar}), 112.8 (t, C₃), 69.6 (d, C₁), 61.2 (t, C₁₂), 22.7 (q, C₄), 14.5 (q, C₁₃). **HRMS (ESI)** m/z : calcd for C₁₃H₁₆NaO₃ [M + Na]⁺: 243.0992, found: 243.0990.

2-(3-Phenylbut-3-en-2-yl)isoindoline-1,3-dione

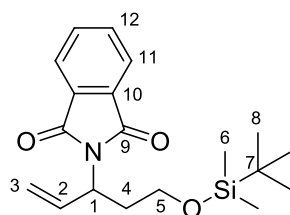


MW (g/mol): 277.32

Molecular formula: C₁₈H₁₅NO₂

This compound was prepared following general procedure B. **IR** (neat): 2984, 1775, 1704, 1383, 1330, 1015, 907, 778, 717 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.75 (dd, J = 5.6, 3.0 Hz, 2H, H₁₁ or H₁₂), 7.65 (dd, J = 5.4, 3.2 Hz, 2H, H₁₁ or H₁₂), 7.44–7.36 (m, 2H, H_{Ph}), 7.32–7.16 (m, 3H, H_{Ph}), 5.54 (m, 1H, H₁), 5.47 (d, J = 1.6 Hz, 1H, H₃), 5.45 (d, J = 1.8 Hz, 1H, H₃), 1.75 (d, J = 6.9 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 168.0 (s, C₉), 147.6 (s, C₂), 140.1 (s, C₅), 133.9 (d, C_{Ph}), 131.9 (s, C₁₀), 128.4 (d, C_{Ph}), 127.8 (d, C_{Ph}), 126.8 (d, C_{Ph}), 123.2 (d, C_{Ph}), 114.8 (t, C₃), 48.0 (d, C₁), 16.9 (q, C₄). **HRMS (ESI)** m/z : calcd for C₁₈H₁₆NO₂ [M + H]⁺: 278.1176, found: 278.1180.

2-(5-((*tert*-Butyldimethylsilyl)oxy)pent-1-en-3-yl)isoindoline-1,3-dione

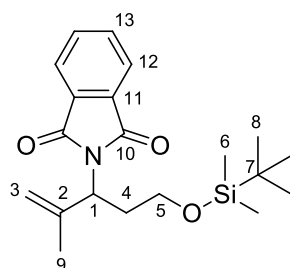


MW (g/mol): 345.51

Molecular formula: C₁₉H₂₇NO₃Si

This compound was prepared following general procedure B. **IR** (neat): 2953, 2928, 2856, 1710, 1382, 1254, 1096, 833, 775, 718 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.82 (dd, J = 5.5, 3.0 Hz, 2H, H₁₁ or H₁₂), 7.70 (dd, J = 5.5, 3.0 Hz, 2H, H₁₁ or H₁₂), 6.19 (ddd, J = 17.2, 10.2, 7.5 Hz, 1H, H₂), 5.25 (dt, J = 17.2, 1.2 Hz, 1H, H₃), 5.18 (dt, J = 10.2, 1.2 Hz, 1H, H₃), 4.99 (m, 1H, H₁), 3.69–3.58 (m, 2H, H₅), 2.34 (m, 1H, H₄), 2.13 (m, 1H, H₄), 0.84 (s, 9H, H₈), -0.05 (d, J = 1.6 Hz, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 168.2 (s, C₉), 135.7 (s, C₂), 134.0 (d, C₁₁ or C₁₂), 132.2 (s, C₁₀), 123.3 (d, C₁₁ or C₁₂), 117.5 (t, C₃), 60.1 (t, C₅), 51.2 (d, C₁), 34.8 (t, C₄), 26.0 (q, C₈), 18.3 (s, C₇), -5.4 (q, C₆), -5.5 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₉H₂₇NaNO₃Si [M + Na]⁺: 368.1652, found: 368.1658.

2-(5-((*tert*-Butyldimethylsilyl)oxy)-2-methylpent-1-en-3-yl)isoindoline-1,3-dione



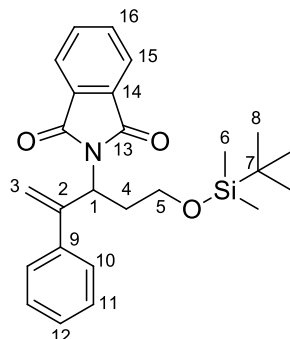
MW (g/mol): 359.53

Molecular formula: C₂₀H₂₉NO₃Si

This compound was prepared following general procedure B. **IR** (neat): 2953, 2929, 1713, 1385, 1255, 1101, 833, 719 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.83 (dd, J = 5.4, 3.1 Hz, 2H, H₁₂ or H₁₃), 7.71 (dd, J = 5.5, 3.0 Hz, 2H, H₁₂ or H₁₃), 5.04 (s, 1H, H₃), 4.99 (m, 1H, H₃), 4.93 (dd, J = 10.0, 5.3 Hz, 1H, H₁), 3.68 (m, 1H, H₅), 3.60 (m, 1H, H₅), 2.53 (m, 1H, H₄), 2.23 (ddt, J = 14.1, 7.4, 5.2 Hz, 1H, H₄), 1.78 (s, 3H, H₉), 0.82 (s, 9H, H₈), -0.08 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 168.6 (s, C₁₀), 142.6 (s, C₂), 134.0 (d, C₁₂ or C₁₃), 132.1 (s, C₁₁), 123.3 (d, C₁₂ or C₁₃), 113.4 (t, C₃), 60.5 (t, C₅), 53.0 (d, C₁), 32.1 (t, C₄), 26.0 (q, C₈), 20.8 (q, C₉), 18.4 (s, C₇), -5.4 (q,

C₆), -5.5 (q, C₆). **HRMS (ESI)** *m/z*: calcd for C₂₀H₂₉NaNO₃Si [M + Na]⁺: 382.1809, found: 382.1812.

2-(5-((*tert*-Butyldimethylsilyl)oxy)-2-phenylpent-1-en-3-yl)isoindoline-1,3-dione

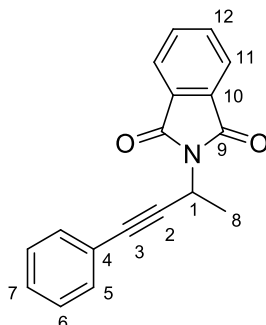


MW (g/mol): 421.60

Molecular formula: C₂₅H₃₁NO₃Si

This compound was prepared following general procedure B. **IR** (neat): 2927, 2855, 1774, 1710, 1383, 1254, 1099, 834, 776, 718 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.76 (dd, *J* = 5.5, 3.0 Hz, 2H, H₁₅ or H₁₆), 7.65 (dd, *J* = 5.4, 3.1 Hz, 2H, H₁₅ or H₁₆), 7.47–7.38 (m, 2H, H_{Ph}), 7.31–7.18 (m, 3H, H_{Ph}), 5.60 (dd, *J* = 10.4, 4.4 Hz, 1H, H₁), 5.43 (d, *J* = 1.0 Hz, 1H, H₃), 5.40 (d, *J* = 1.6 Hz, 1H, H₃), 3.77–3.57 (m, 2H, H₅), 2.61 (m, 1H, H₄), 2.24 (m, 1H, H₄), 0.83 (s, 9H, H₈), -0.08 (s, 3H, H₆), -0.10 (s, 3H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 168.3 (s, C₁₃), 147.2 (s, C₂), 140.3 (s, C₉), 133.9 (d, C₁₅ or C₁₆), 132.0 (s, C₁₄), 128.4 (d, C_{Ph}), 127.8 (d, C_{Ph}), 127.0 (d, C_{Ph}), 123.2 (d, C₁₅ or C₁₆), 114.8 (t, C₃), 60.5 (t, C₅), 50.6 (d, C₁), 32.9 (t, C₄), 25.9 (q, C₈), 18.3 (s, C₇), -5.5 (q, C₆). **HRMS (ESI)** *m/z*: calcd for C₂₅H₃₁NaNO₃Si [M + Na]⁺: 444.1965, found: 444.1968.

2-(4-Phenylbut-3-yn-2-yl)isoindoline-1,3-dione



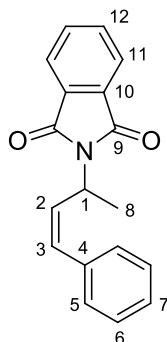
MW (g/mol): 275.30

Molecular formula: C₁₈H₁₃NO₂

This compound was prepared following general procedure B. **IR** (neat): 3064, 2926, 1777, 1703, 1488, 1384, 1337, 1147, 1065, 879, 760, 713 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.85 (dd, *J* = 5.5,

3.0 Hz, 2H, H₁₁ or H₁₂), 7.70 (dd, $J = 5.5, 3.0$ Hz, 2H, H₁₁ or H₁₂), 7.47–7.38 (m, 2H, H_{Ph}), 7.29–7.23 (m, 3H, H_{Ph}), 5.42 (q, $J = 7.1$ Hz, 1H, H₁), 1.78 (d, $J = 7.2$ Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 167.1 (s, C₉), 134.2 (s, C₁₁ or C₁₂), 132.1 (d, C_{Ph}), 132.0 (s, C₁₀), 128.5 (d, C_{Ph}), 128.3 (d, C_{Ph}), 123.5 (s, C₁₁ or C₁₂), 122.6 (s, C₄), 86.7 (s, C₂), 83.0 (s, C₃), 37.8 (s, C₁), 20.4 (s, C₈). **HRMS (ESI)** m/z : calcd for C₁₈H₁₃NaNO₂ [M + Na]⁺: 298.0838, found: 298.0842.

(Z)-2-(4-Phenylbut-3-en-2-yl)isoindoline-1,3-dione



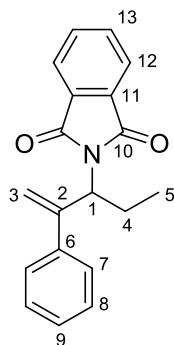
MW (g/mol): 277.32

Molecular formula: C₁₈H₁₅NO₂

To a solution of 2-(4-phenylbut-3-yn-2-yl)isoindoline-1,3-dione (363 mg, 1.32 mmol, 1 equiv.) in EtOAc (15 mL) was added quinoline (1.32 mmol, 156 μ L, 1 equiv.) and the Lindlar catalyst (84 mg). After degassing the mixture under vacuum several times, the solution was then stirred for 3 h under positive pressure of H₂. The catalyst was eventually filtered through a plug of Celite[®] and the solvent was evaporated under reduced pressure. Purification of the residue by flash column chromatography over silica gel gave the corresponding (Z)-2-(4-phenylbut-3-en-2-yl)isoindoline-1,3-dione in quantitative yield.

IR (neat): 3055, 3025, 2988, 1770, 1694, 1611, 1384, 1355, 1141, 1065, 1016, 869, 771, 717 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.82 (dd, $J = 5.5, 3.0$ Hz, 2H, H₁₁ or H₁₂), 7.70 (dd, $J = 5.5, 3.0$ Hz, 2H, H₁₁ or H₁₂), 7.40–7.32 (m, 2H, H_{Ph}), 7.31–7.22 (m, 3H, H_{Ph}), 6.57 (d, $J = 11.6$ Hz, 1H, H₃), 6.30 (dd, $J = 11.6, 9.6$ Hz, 1H, H₂), 5.46 (dq, $J = 9.5, 6.9, 1.1$ Hz, 1H, H₁), 1.56 (d, $J = 6.9$ Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 168.1 (s, C₉), 136.2 (s, C₄), 134.0 (d, C₁₁ or C₁₂), 132.2 (s, C₁₀), 131.2 (d, C₂), 129.9 (d, C₃), 128.7 (d, C_{Ph}), 128.5 (d, C_{Ph}), 127.4 (d, C_{Ph}), 123.2 (d, C₁₁ or C₁₂), 44.8 (d, C₁), 20.1 (q, C₈). **HRMS (ESI)** m/z : calcd for C₁₈H₁₅NaNO₂ [M + Na]⁺: 300.0995, found: 300.0997.

2-(2-Phenylpent-1-en-3-yl)isoindoline-1,3-dione

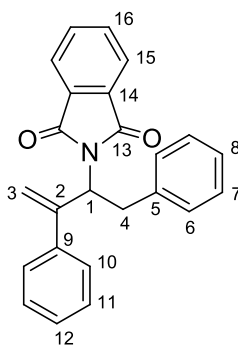


MW (g/mol): 291.34

Molecular formula: C₁₉H₁₇NO

This compound was prepared following general procedure B. **IR** (neat): 2969, 1770, 1708, 1382, 1330, 1037, 777, 717, 702 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.75 (dd, J = 5.5, 3.0 Hz, 2H, H₁₂ or H₁₃), 7.64 (dd, J = 5.4, 3.1 Hz, 2H, H₁₂ or H₁₃), 7.47–7.32 (m, 2H, H_{Ph}), 7.29–7.15 (m, 3H, H_{Ph}), 5.43 (s, 2H, H₃), 5.24 (dd, J = 10.7, 4.7 Hz, 1H, H₁), 2.36 (ddq, J = 14.6, 10.7, 7.3 Hz, 1H, H₄), 2.09 (m, 1H, H₄), 0.95 (t, J = 7.4 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 168.4 (s, C₁₀), 147.2 (s, C₂), 140.6 (s, C₆), 134.0 (d, C₁₂ or C₁₃), 131.7 (s, C₁₁), 128.4 (d, C_{Ph}), 127.8 (d, C_{Ph}), 126.9 (d, C_{Ph}), 123.3 (d, C₁₂ or C₁₃), 115.1 (t, C₃), 54.8 (d, C₁), 23.4 (t, C₄), 11.4 (q, C₅). **HRMS (ESI)** m/z : calcd for C₁₉H₁₇NaNO₂ [M + Na]⁺: 314.1152, found: 314.1154.

2-(1,3-Diphenylbut-3-en-2-yl)isoindoline-1,3-dione



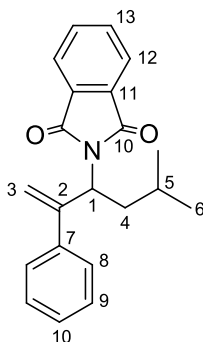
MW (g/mol): 353.41

Molecular formula: C₂₄H₁₉NO₂

This compound was prepared following general procedure B. **IR** (neat): 3028, 1771, 1706, 1382, 1330, 910, 871, 716, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.66 (dd, J = 5.6, 2.9 Hz, 2H, H₁₅ or H₁₆), 7.59 (dd, J = 5.6, 2.9 Hz, 2H, H₁₅ or H₁₆), 7.50–7.39 (m, 2H, H_{Ph}), 7.33–7.11 (m, 8H, H_{Ph}), 5.64 (ddt, J = 11.4, 4.6, 1.5 Hz, 1H, H₁), 5.55 (d, J = 1.8 Hz, 1H, H₃), 5.51 (d, J = 1.3 Hz, 1H, H₃), 3.66 (dd, J = 13.9, 11.4 Hz, 1H, H₄), 3.38 (dd, J = 13.9, 4.6 Hz, 1H, H₄). **¹³C NMR** (100 MHz,

CDCl_3) δ 168.1 (s, C_{13}), 146.9 (s, C_2), 140.3 (s, C_9), 138.0 (s, C_5), 133.8 (d, C_{15} or C_{16}), 131.5 (s, C_{14}), 129.0 (d, C_{Ph}), 128.5 (d, C_{Ph}), 128.5 (d, C_{Ph}), 127.9 (d, C_{Ph}), 126.9 (d, C_{Ph}), 126.7 (d, C_{Ph}), 123.2 (d, C_{15} or C_{16}), 115.4 (t, C_3), 54.5 (d, C_1), 36.2 (t, C_4). **HRMS (ESI)** m/z : calcd for $\text{C}_{24}\text{H}_{19}\text{NaNO}_2$ $[\text{M} + \text{Na}]^+$: 376.1308, found: 376.1312.

2-(5-Methyl-2-phenylhex-1-en-3-yl)isoindoline-1,3-dione

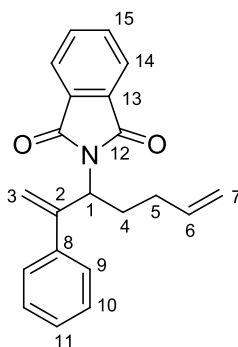


MW (g/mol): 319.40

Molecular formula: $\text{C}_{21}\text{H}_{21}\text{NO}_2$

This compound was prepared following general procedure B. **IR** (neat): 2955, 1764, 1702, 1379, 1327, 1069, 716, 705 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.82 (dd, $J = 5.5, 3.0$ Hz, 2H, H_{12} or H_{13}), 7.72 (dd, $J = 5.5, 3.0$ Hz, 2H, H_{12} or H_{13}), 7.49–7.43 (m, 2H, H_{Ph}), 7.36–7.24 (m, 3H, H_{Ph}), 5.53–5.44 (m, 3H, $\text{H}_1 + \text{H}_3$), 2.50 (ddd, $J = 14.2, 11.0, 3.9$ Hz, 1H, H_4), 1.79 (ddd, $J = 14.1, 9.9, 4.3$ Hz, 1H, H_4), 1.57 (m, 1H, H_5), 1.05 (d, $J = 6.5$ Hz, 3H, H_6), 0.99 (d, $J = 6.6$ Hz, 3H, H_6). **^{13}C NMR** (100 MHz, CDCl_3) δ 168.4 (s, C_{10}), 147.5 (s, C_2), 140.6 (s, C_7), 134.0 (d, C_{12} or C_{13}), 131.8 (s, C_{11}), 128.4 (d, C_{Ph}), 127.8 (d, C_{Ph}), 126.9 (d, C_{Ph}), 123.3 (d, C_{12} or C_{13}), 114.9 (t, C_3), 51.4 (d, C_1), 39.0 (t, C_4), 25.5 (d, C_5), 23.7 (q, C_6), 21.7 (q, C_6). **HRMS (ESI)** m/z : calcd for $\text{C}_{21}\text{H}_{21}\text{NaNO}_2$ $[\text{M} + \text{Na}]^+$: 342.1465, found: 342.1467.

2-(2-Phenylhepta-1,6-dien-3-yl)isoindoline-1,3-dione

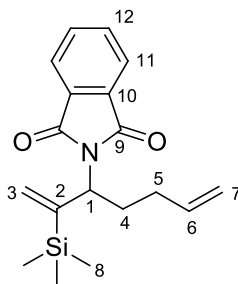


MW (g/mol): 317.38

Molecular formula: $\text{C}_{21}\text{H}_{19}\text{NO}_2$

This compound was prepared following general procedure B. **IR** (neat): 3061, 2934, 1774, 1708, 1384, 1358, 1069, 912, 718, 700 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.68 (dd, $J = 5.5, 3.0$ Hz, 2H, H_{14} or H_{15}), 7.57 (dd, $J = 5.5, 3.0$ Hz, 2H, H_{14} or H_{15}), 7.35–7.28 (m, 2H, H_{Ph}), 7.22–7.09 (m, 3H, H_{Ph}), 5.72 (ddt, $J = 16.7, 10.2, 6.5$ Hz, 1H, H_6), 5.35 (s, 2H, H_3), 5.27 (m, 1H, H_1), 4.91 (m, 1H, H_7), 4.85 (m, 1H, H_7), 2.43 (m, 1H, H_5), 2.10–1.98 (m, 3H, $\text{H}_4 + \text{H}_5$). **^{13}C NMR** (100 MHz, CDCl_3) δ 168.3 (s, C_{12}), 147.1 (s, C_2), 140.4 (s, C_8), 137.3 (d, C_6), 134.0 (d, C_{14} or C_{15}), 131.7 (s, C_{13}), 128.4 (d, C_{Ph}), 127.8 (d, C_{Ph}), 126.9 (d, C_{Ph}), 123.3 (d, C_{14} or C_{15}), 115.7 (t, C_7), 115.2 (t, C_3), 52.7 (d, C_1), 31.1 (t, C_4), 29.4 (t, C_5). **HRMS (ESI)** m/z : calcd for $\text{C}_{21}\text{H}_{19}\text{NaNO}_2$ $[\text{M} + \text{Na}]^+$: 340.1308, found: 340.1309.

2-(2-(Trimethylsilyl)hepta-1,6-dien-3-yl)isoindoline-1,3-dione

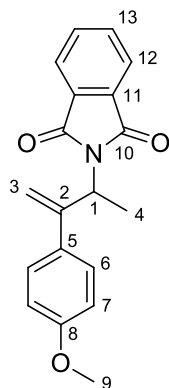


MW (g/mol): 313.47

Molecular formula: $\text{C}_{18}\text{H}_{23}\text{NO}_2\text{Si}$

This compound was prepared following general procedure B. **IR** (neat): 2956, 1711, 1383, 1352, 1249, 838, 718 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.83 (dd, $J = 5.4, 3.1$ Hz, 2H, H_{11} or H_{12}), 7.72 (dd, $J = 5.5, 3.0$ Hz, 2H, H_{11} or H_{12}), 5.94 (t_{app} , $J = 1.7$ Hz, 1H, H_3), 5.78 (m, 1H, H_6), 5.62 (t_{app} , $J = 1.4$ Hz, 1H, H_3), 5.01–4.89 (m, 3H, $\text{H}_1 + \text{H}_7$), 2.49 (m, 1H, H_5), 2.09–1.95 (m, 3H, $\text{H}_4 + \text{H}_5$), 0.09 (s, 9H, H_8). **^{13}C NMR** (100 MHz, CDCl_3) δ 168.6 (s, C_9), 148.8 (s, C_2), 137.5 (d, C_6), 134.1 (d, C_{11} or C_{12}), 132.0 (s, C_{10}), 126.7 (t, C_3), 123.3 (d, C_{11} or C_{12}), 115.5 (t, C_7), 53.4 (d, C_1), 31.2 (t, C_5), 29.8 (t, C_4), –1.1 (q, C_8). **HRMS (ESI)** m/z : calcd for $\text{C}_{18}\text{H}_{23}\text{NaNO}_2\text{Si}$ $[\text{M} + \text{Na}]^+$: 336.1390, found: 336.1394.

2-(3-(4-Methoxyphenyl)but-3-en-2-yl)isoindoline-1,3-dione

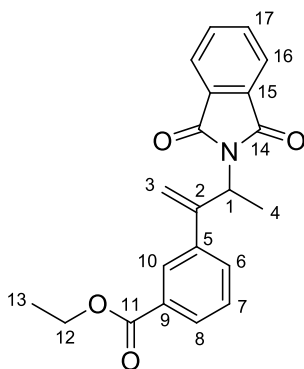


MW (g/mol): 307.34

Molecular formula: C₁₉H₁₇NO₃

This compound was prepared following general procedure B. **IR** (neat): 2937, 1704, 1511, 1383, 1330, 1245, 1031, 835, 716 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.73 (dd, J = 5.5, 3.0 Hz, 2H, H₁₂ or H₁₃), 7.63 (dd, J = 5.5, 3.0 Hz, 2H, H₁₂ or H₁₃), 7.31 (d, J = 8.9 Hz, 2H, H₆), 6.78 (d, J = 8.9 Hz, 2H, H₇), 5.48 (qt, J = 6.9, 1.8 Hz, 1H, H₁), 5.40 (d, J = 1.4 Hz, 1H, H₃), 5.36 (d, J = 1.8 Hz, 1H, H₃), 3.73 (s, 3H, H₉), 1.71 (d, J = 6.9 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 168.0 (s, C₁₀), 159.2 (s, C₈), 146.9 (s, C₂), 133.9 (d, C₁₂ or C₁₃), 132.5 (s, C₅), 131.9 (s, C₁₁), 127.8 (d, C₆), 123.2 (d, C₁₂ or C₁₃), 113.7 (d, C₇), 113.7 (t, C₃), 55.3 (q, C₉), 47.9 (d, C₁), 16.9 (q, C₄). **HRMS (ESI)** m/z : calcd for C₁₉H₁₇NaNO₃ [M + Na]⁺: 330.1100, found: 330.1099.

Ethyl 3-(3-(1,3-dioxoisindolin-2-yl)but-1-en-2-yl)benzoate



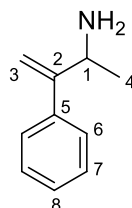
MW (g/mol): 349.38

Molecular formula: C₂₁H₁₉NO₄

This compound was prepared following general procedure B. **IR** (neat): 2972, 1772, 1703, 1470, 1367, 1291, 1238, 1090, 926, 735, 721, 711 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 8.06 (m, 1H, H_{Ar}), 7.88 (ddd, J = 7.8, 1.6, 1.2 Hz, 1H, H_{Ar}), 7.74 (dd, J = 5.6, 3.0 Hz, 2H, H₁₆ or H₁₇), 7.64 (dd, J = 5.6, 3.0 Hz, 2H, H₁₆ or H₁₇), 7.56 (ddd, J = 7.8, 1.9, 1.2 Hz, 1H, H_{Ar}), 7.33 (t, J = 7.7 Hz, 1H,

H_{Ar}), 5.58–5.49 (m, 2H, $H_1 + H_3$), 5.48 (d, $J = 1.9$ Hz, 1H, H_3), 4.35 (qd, $J = 7.1, 1.8$ Hz, 2H, H_{12}), 1.73 (d, $J = 6.9$ Hz, 3H, H_4), 1.39 (t, $J = 7.1$ Hz, 3H, H_{13}). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.9 (s, C_{14}), 166.5 (s, C_{11}), 146.8 (s, C_2), 140.3 (s, C_5), 134.0 (d, C_{16} or C_{17}), 131.8 (s, C_{15}), 131.0 (d, C_{Ar}), 130.8 (s, C_9), 129.0 (d, C_{Ar}), 128.4 (d, C_{Ar}), 128.0 (d, C_{Ar}), 123.3 (d, C_{16} or C_{17}), 115.8 (t, C_3), 61.2 (t, C_{12}), 48.0 (d, C_1), 16.9 (q, C_4), 14.4 (q, C_{13}). **HRMS (ESI)** m/z : calcd for $C_{21}H_{19}NaNO_4$ $[M + Na]^+$: 372.1206, found: 372.1206.

3-Phenylbut-3-en-2-amine (2a)

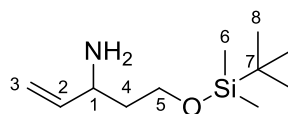


MW (g/mol): 147.22

Molecular formula: $C_{10}H_{13}N$

This compound was prepared following general procedure C. **IR** (neat): 2965, 1573, 1493, 1443, 1368, 901, 777, 697 cm^{-1} . **1H NMR** (400 MHz, $CDCl_3$) δ 7.43–7.22 (m, 5H, H_{Ph}), 5.26 (s, 1H, H_3), 5.20 (s, 1H, H_3), 4.03 (q, $J = 6.6$ Hz, 1H, H_1), 1.57 (s, 2H, NH), 1.23 (d, $J = 6.6$ Hz, 3H, H_4). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.1 (s, C_2), 141.3 (s, C_5), 128.4 (d, C_{Ph}), 127.5 (d, C_{Ph}), 126.9 (d, C_{Ph}), 110.6 (d, C_1), 50.0 (t, C_3), 23.0 (q, C_4). **HRMS (ESI)** m/z : calcd for $C_{10}H_{14}N$ $[M + H]^+$: 148.1121, found: 148.1120.

5-((tert-Butyldimethylsilyl)oxy)pent-1-en-3-amine (2b)

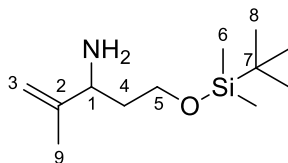


MW (g/mol): 215.41

Molecular formula: $C_{11}H_{25}NOSi$

This compound was prepared following general procedure C. **IR** (neat): 2954, 2929, 2857, 1472, 1254, 1094, 915, 832, 773 cm^{-1} . **1H NMR** (400 MHz, $CDCl_3$) δ 5.82 (ddd, $J = 17.1, 10.3, 6.7$ Hz, 1H, H_2), 5.13 (dt_{app}, $J = 17.2, 1.4$ Hz, 1H, H_3), 5.03 (m, 1H, H_3), 3.80–3.62 (m, 2H, H_5), 3.49 (q_{app}, $J = 6.7$ Hz, 1H, H_1), 1.70–1.59 (m, 2H, H_4), 1.56 (brs, 2H, NH), 0.90 (s, 9H, H_8), 0.05 (s, 6H, H_6). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.2 (s, C_2), 113.5 (t, C_3), 60.7 (t, C_5), 52.0 (d, C_1), 40.2 (t, C_4), 26.1 (s, C_7), 18.4 (q, C_8), -5.2 (q, C_6). **HRMS (ESI)** m/z : calcd for $C_{11}H_{26}NOSi$ $[M + H]^+$: 216.1778, found: 216.1779.

5-((*tert*-Butyldimethylsilyl)oxy)-2-methylpent-1-en-3-amine (2c)

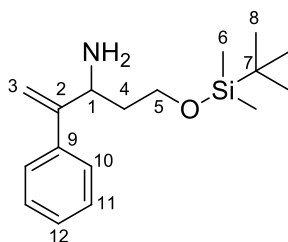


MW (g/mol): 229.43

Molecular formula: C₁₂H₂₇NOSi

This compound was prepared following general procedure C. **IR** (neat): 2953, 2929, 1472, 1253, 1092, 832, 773 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 4.87 (s, 1H, H₃), 4.76 (s, 1H, H₃), 3.75–3.59 (m, 2H, H₅), 3.45 (dd, J = 7.2, 5.9 Hz, 1H, H₁), 1.79–1.67 (m, 4H, H₉ + H₄), 1.64–1.38 (m, 3H, H₄ + NH), 0.89 (s, 9H, H₈), 0.05 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 149.1 (s, C₂), 110.2 (t, C₃), 61.0 (t, C₅), 55.0 (d, C₁), 38.5 (t, C₄), 26.1 (q, C₈), 18.4 (s, C₇), 18.2 (q, C₉), -5.2 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₂H₂₈NaNOSi [M + H]⁺: 230.1935, found: 230.1936.

5-((*tert*-Butyldimethylsilyl)oxy)-2-phenylpent-1-en-3-amine (2d)

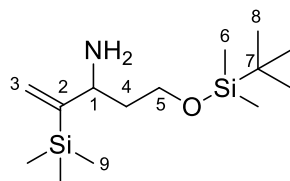


MW (g/mol): 291.50

Molecular formula: C₁₇H₂₉NOSi

This compound was prepared following general procedure C. **IR** (neat): 2953, 2928, 2856, 1471, 1253, 1092, 833, 774, 699 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.50–7.04 (m, 5H, H_{Ph}), 5.18 (s, 1H, H₃), 5.17 (s, 1H, H₁), 3.96 (dd, J = 8.2, 3.8 Hz, 1H, H₁), 3.72–3.51 (m, 2H, H₅), 1.73 (m, 1H, H₄), 1.55 (brs, 2H, NH), 1.40 (m, 1H, H₄), 0.79 (s, 9H, H₈), -0.07 (s, 3H, H₆), -0.08 (s, 3H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 154.0 (s, C₂), 141.2 (s, C₉), 128.4 (d, C_{Ph}), 127.6 (d, C_{Ph}), 127.0 (d, C_{Ph}), 111.6 (t, C₃), 61.1 (d, C₁), 52.4 (t, C₅), 39.3 (t, C₄), 26.1 (q, C₈), 18.4 (s, C₇), -5.3 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₇H₃₀NOSi [M + H]⁺: 292.2091, found: 292.2094.

5-((*tert*-Butyldimethylsilyl)oxy)-2-(trimethylsilyl)pent-1-en-3-amine (2e)

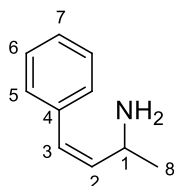


MW (g/mol): 287.59

Molecular formula: C₁₄H₃₃NOSi₂

This compound was prepared following general procedure C. **IR** (neat): 2954, 2858, 1472, 1249, 1092, 928, 832, 774 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.77 (dd, J = 2.4, 1.3 Hz, 1H, H₃), 5.40 (dd, J = 2.4, 0.7 Hz, 1H, H₃), 3.77–3.62 (m, 3H, H₁ + H₅), 1.78 (m, 1H, H₄), 1.55–1.42 (m, 3H, H₄ + NH), 0.90 (s, 9H, H₈), 0.13 (s, 9H, H₉), 0.06 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 157.1 (s, C₂), 123.2 (t, C₃), 61.1 (t, C₅), 53.7 (d, C₁), 40.5 (t, C₄), 26.1 (q, C₈), 18.4 (s, C₇), -0.4 (q, C₉), -5.2 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₄H₃₄NOSi₂ [M + H]⁺: 288.2173, found: 288.2176.

(*Z*)-4-Phenylbut-3-en-2-amine (2f)

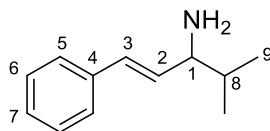


MW (g/mol): 147.22

Molecular formula: C₁₀H₁₃N

This compound was prepared following general procedure C. **IR** (neat): 3356, 3056, 2961, 1599, 1493, 1447, 1370, 1116, 1072, 793, 765, 697 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.39–7.31 (m, 2H, H_{Ph}), 7.30–7.21 (m, 3H, H_{Ph}), 6.40 (d, J = 11.6 Hz, 1H, H₃), 5.58 (dd, J = 11.6, 9.5 Hz, 1H, H₂), 4.05 (dq, J = 9.4, 6.4, 0.9 Hz, 1H, H₁), 1.61 (s, 2H, NH), 1.25 (d, J = 6.4 Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 138.6 (d, C₂), 137.3 (s, C₄), 128.8 (d, C₃), 128.4 (d, C_{Ph}), 128.0 (d, C_{Ph}), 127.0 (d, C_{Ph}), 44.5 (d, C₁), 24.0 (q, C₈). **HRMS (ESI)** m/z : calcd for C₁₀H₁₄N [M + H]⁺: 148.1121, found: 148.1117.

(*E*)-4-Methyl-1-phenylpent-1-en-3-amine (2h)



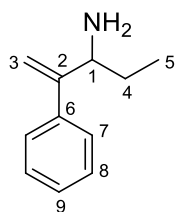
MW (g/mol): 175.27

Molecular formula: C₁₂H₁₇N

To a solution of (*E*)-4-methyl-1-phenylpent-1-en-3-ol (440 mg, 2.5 mmol, 1 equiv.) in CH₂Cl₂ (10 mL) were added TEMPO (75 mg, 0.5 mmol, 0.2 equiv.) and (diacetoxyiodo)benzene (944 mg, 2.87 mmol, 1.15 equiv.). The reaction mixture was stirred for 1.5 h at room temperature until the alcohol was totally oxidized. MeOH (5 mL) was then added followed by ammonium formate (1.61 g, 25 mmol, 10 equiv.) and NaBH₃CN (321 mg, 5.0 mmol, 2.0 equiv.) and the mixture was stirred overnight at rt. The solution was quenched with a 2 M aqueous solution of NaOH (15 mL) and the aqueous phase was extracted twice with CH₂Cl₂ (2 x 30 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (90:10 EtOAc/MeOH) to afford the pure (*E*)-4-methyl-1-phenylpent-1-en-3-amine in 26% yield.

IR (neat): 3295, 2960, 2929, 2870, 1665, 1449, 1373, 1103, 968, 748, 694 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.47–7.15 (m, 5H, H_{Ph}), 6.49 (d, *J* = 15.9 Hz, 1H, H₃), 6.18 (dd, *J* = 15.9, 7.4 Hz, 1H, H₃), 3.28 (t_{app}, *J* = 6.6 Hz, 1H, H₁), 1.83 (brs, 2H, NH), 1.75 (m, 1H, H₈), 0.98 (d, *J* = 6.8 Hz, 3H, H₉), 0.95 (d, *J* = 6.8 Hz, 3H, H₉). **¹³C NMR** (100 MHz, CDCl₃) δ 137.4 (s, C₄), 133.7 (d, C₂), 130.1 (d, C₃), 128.7 (d, C_{Ph}), 127.4 (d, C_{Ph}), 126.4 (d, C_{Ph}), 60.0 (d, C₁), 34.3 (d, C₈), 18.9 (q, C₉), 18.9 (q, C₉). **HRMS (ESI)** *m/z*: calcd for C₁₂H₁₅ [M + H – NH₃]⁺: 159.1168, found: 159.1168.

2-Phenylpent-1-en-3-amine (2i)

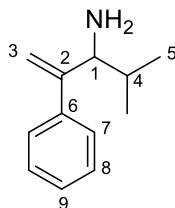


MW (g/mol): 161.24

Molecular formula: C₁₁H₁₅N

This compound was prepared following general procedure C. **IR** (neat): 2961, 2931, 1492, 1460, 902, 777, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.46–7.24 (m, 5H, H_{Ph}), 5.24 (s, 2H, H₃), 3.82 (m, 1H, H₁), 1.63 (m, 1H, H₄), 1.51 (s, 2H, NH), 1.39 (m, 1H, H₄), 0.92 (t, *J* = 7.4 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 153.7 (s, C₂), 141.5 (s, C₆), 128.4 (d, C_{Ph}), 127.5 (d, C_{Ph}), 127.0 (d, C_{Ph}), 111.9 (t, C₃), 56.3 (d, C₁), 29.3 (t, C₄), 10.4 (q, C₅). **HRMS (ESI)** *m/z*: calcd for C₁₁H₁₆N [M + H]⁺: 162.1277, found: 162.1277.

4-Methyl-2-phenylpent-1-en-3-amine (2j)

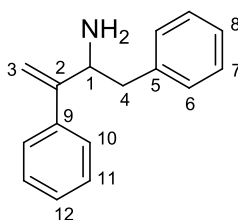


MW (g/mol): 175.27

Molecular formula: C₁₂H₁₇N

This compound was prepared following general procedure C. **IR** (neat): 3079, 3056, 2958, 1626, 1599, 1573, 1499, 1383, 1365, 902, 777, 969 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.46–7.19 (m, 5H, H_{Ph}), 5.27 (s, 1H, H₃), 5.24 (s, 1H, H₃), 3.71 (d, J = 5.0 Hz, 1H, H₁), 1.71 (m, 1H, H₄), 1.36 (s, 2H, NH), 0.95 (d, J = 6.8 Hz, 3H, H₅), 0.79 (d, J = 6.8 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 153.4 (s, C₂), 142.0 (s, C₆), 128.4 (d, C_{Ph}), 127.4 (d, C_{Ph}), 127.1 (d, C_{Ph}), 112.7 (t, C₃), 60.6 (d, C₁), 31.2 (d, C₄), 20.7 (q, C₅), 16.0 (q, C₅). **HRMS (ESI)** m/z : calcd for C₁₂H₁₈N [M + H]⁺: 176.1434, found: 176.1436.

2-(1,3-Diphenylbut-3-en-2-yl)isoindoline-1,3-dione (2k)¹⁰



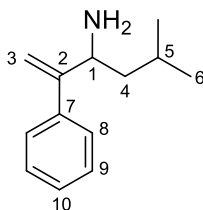
MW (g/mol): 223.31

Molecular formula: C₁₆H₁₇N

This compound was prepared following general procedure C. **IR** (neat): 3026, 1600, 1493, 1454, 1075, 905, 778, 696 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.47–7.17 (m, 10H, H_{Ph}), 5.32 (s, 2H, H₃), 4.17 (d, J = 8.4 Hz, 1H, H₁), 2.99 (d, J = 13.3 Hz, 1H, H₄), 2.52 (dd, J = 13.4, 9.0 Hz, 1H, H₄), 1.68 (s, 2H, NH). **¹³C NMR** (100 MHz, CDCl₃) δ 152.8 (s, C₂), 141.3 (s, C₉), 139.2 (s, C₅), 129.5 (s, C_{Ph}), 128.6 (d, C_{Ph}), 128.6 (d, C_{Ph}), 127.7 (s, C_{Ph}), 127.0 (s, C_{Ph}), 126.5 (s, C_{Ph}), 112.5 (s, C₃), 56.0 (s, C₁), 43.2 (s, C₄).

¹⁰ Anderson, L. L.; Woerpel, K. A. *Org. Lett.*, **2009**, *11*, 425.

5-Methyl-2-phenylhex-1-en-3-amine (2l)

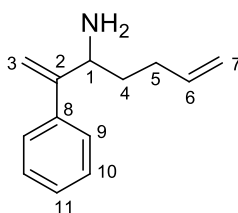


MW (g/mol): 189.30

Molecular formula: C₁₃H₁₉N

This compound was prepared following general procedure C. **IR** (neat): 2954, 1626, 1598, 1573, 1493, 1466, 1366, 903, 777, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.44–7.14 (m, 5H, H_{Ph}), 5.17 (s, 1H, H₃), 5.13 (s, 1H, H₃), 3.81 (dd, J = 8.7, 4.8 Hz, 1H, H₁), 1.70 (m, 1H, H₅), 1.43–1.23 (m, 3H, NH + H₁), 1.19 (m, 1H, H₁), 0.83 (d, J = 6.6 Hz, 3H, H₆), 0.81 (d, J = 6.7 Hz, 3H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 154.7 (s, C₂), 141.4 (s, C₇), 128.4 (d, C_{Ph}), 127.5 (d, C_{Ph}), 127.1 (d, C_{Ph}), 111.6 (t, C₃), 52.9 (d, C₁), 46.4 (t, C₄), 25.2 (d, C₅), 23.6 (q, C₆), 21.9 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₃H₂₀N [M + H]⁺: 190.1590, found: 190.1592.

2-Phenylhepta-1,6-dien-3-amine (2m)

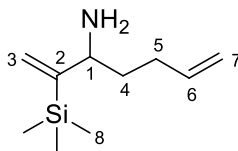


MW (g/mol): 187.28

Molecular formula: C₁₃H₁₇N

This compound was prepared following general procedure C. **IR** (neat): 3077, 2925, 1639, 1573, 1493, 1442, 997, 9905, 777, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.39–7.26 (m, 5H, H_{Ph}), 5.78 (ddt, J = 17.0, 10.2, 6.7 Hz, 1H, H₆), 5.27 (t_{app}, J = 1.2 Hz, 1H, H₃), 5.26 (s, 1H, H₃), 5.01 (m, 1H, H₇), 4.95 (m, 1H, H₇), 3.88 (dd, J = 7.7, 5.0 Hz, 1H, H₁), 2.31–2.02 (m, 2H, H₅), 1.76–1.65 (m, 3H, NH + H₄), 1.48 (m, 1H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 153.7 (s, C₂), 141.3 (s, C₈), 138.4 (d, C₆), 128.5 (d, C_{Ph}), 127.6 (d, C_{Ph}), 127.1 (d, C_{Ph}), 115.0 (t, C₇), 112.1 (t, C₃), 54.4 (d, C₁), 35.7 (t, C₄), 30.6 (t, C₅). **HRMS (ESI)** m/z : calcd for C₁₃H₁₈N [M + H]⁺: 188.1434, found: 188.1436.

2-(Trimethylsilyl)hepta-1,6-dien-3-amine (2n)

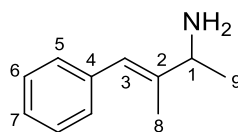


MW (g/mol): 183.37

Molecular formula: C₁₀H₂₁NSi

This compound was prepared following general procedure C. **IR** (neat): 2955, 1735, 1641, 11521, 1249, 1066, 838, 756 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.83 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H, H₆), 5.74 (dd, J = 2.4, 1.2 Hz, 1H, H₃), 5.41 (dd, J = 2.4, 0.7 Hz, 1H, H₃), 5.03 (m, 1H, H₇), 4.94 (m, 1H, H₇), 3.45 (m, 1H, H₁), 2.26–1.93 (m, 2H, H₅), 1.65 (m, 1H, H₄), 1.46 (m, 1H, H₄), 1.35–1.27 (m, 2H, NH), 0.14 (s, 9H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 155.9 (s, C₂), 138.5 (d, C₆), 124.2 (t, C₃), 114.9 (t, C₇), 56.1 (d, C₁), 36.6 (t, C₄), 30.7 (t, C₅), -0.4 (q, C₈). **HRMS (ESI)** m/z : calcd for C₁₀H₂₂NSi [M + H]⁺: 184.1516, found: 184.1519.

(*E*)-3-Methyl-4-phenylbut-3-en-2-amine (2o)¹¹



MW (g/mol): 161.24

Molecular formula: C₁₁H₁₅N

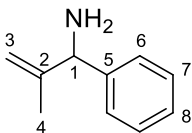
To a solution of (*E*)-3-methyl-4-phenylbut-3-en-2-ol (162 mg, 1.0 mmol, 1 equiv.) in CH₂Cl₂ (4 mL) were added TEMPO (30 mg, 0.2 mmol, 0.2 equiv.) and (diacetoxyiodo)benzene (370 mg, 1.15 mmol, 1.15 equiv.). The reaction mixture was stirred for 1.5 h at room temperature until the alcohol was totally oxidized. MeOH (2 mL) was then added followed by ammonium formate (630 mg, 10 mmol, 10 equiv.) and NaBH₃CN (125 mg, 2.0 mmol, 2.0 equiv.) and the mixture was stirred overnight at rt. The solution was quenched with a 2 M solution of NaOH (5 mL) and the aqueous phase was extracted twice with CH₂Cl₂ (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (90:10 EtOAc/MeOH) to afford the pure (*E*)-3-methyl-4-phenylbut-3-en-2-amine in 27% yield.

IR (neat): 2963, 1599, 1492, 1443, 1372, 1101, 917, 856, 740, 697 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.35–7.16 (m, 5H, H_{Ph}), 6.46 (s, 1H, H₃), 3.61 (q, J = 6.5 Hz, 1H, H₁), 1.91 (brs, 2H, NH),

¹¹ Klauber, E. G.; Mittal, N.; Shah, T. K.; Seidel, D. *Org. Lett.*, **2011**, *13*, 2464.

1.87 (d, $J = 1.3$ Hz, 3H, H₈), 1.26 (d, $J = 6.6$ Hz, 3H, H₉). **¹³C NMR** (100 MHz, CDCl₃) δ 143.2 (s, C₂ or C₄), 138.2 (s, C₂ or C₄), 129.1 (d, C_{Ph}), 128.2 (d, C_{Ph}), 126.3 (d, C_{Ph}), 123.7 (d, C₃), 54.9 (d, C₁), 22.28 (q, C₉), 14.0 (q, C₈). **HRMS (ESI)** m/z : calcd for C₁₁H₁₃ [M + H – NH₃]⁺: 145.1012, found: 145.1011.

2-Methyl-1-phenylprop-2-en-1-ol (2p)



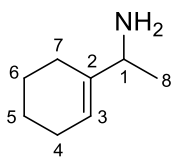
MW (g/mol): 147.22

Molecular formula: C₁₀H₁₃N

To a solution of freshly distilled benzonitrile (0.5 mL, 4.8 mmol, 1 equiv.) in THF (4 mL) was added isoprenylmagnesium bromide (0.5 M in THF, 11 mL, 5.5 mmol, 1.15 equiv.). The reaction was refluxed for 1h and MeOH (2 mL) was added at 0 °C followed by NaBH₄ (190 mg, 4.8 mmol, 1.0 equiv.). The mixture was stirred 30 min at the same temperature and quenched with water (5 mL). The aqueous phase was extracted twice with EtOAc (2 x 10 mL), the combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (90:10 EtOAc/MeOH) to afford the pure 2-methyl-1-phenylprop-2-en-1-ol in 65% yield.

IR (neat): 3062, 3026, 2970, 1645, 1491, 1450, 1372, 892, 756, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.38–7.30 (m, 4H, H_{Ph}), 7.25 (m, 1H, H_{Ph}), 5.16 (s, 1H, H₃), 4.93 (s, 1H, H₃), 4.47 (s, 1H, H₁), 1.61 (s, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 148.4 (s, C₂), 143.9 (s, C₅), 128.5 (d, C_{Ph}), 127.2 (d, C_{Ph}), 127.0 (d, C_{Ph}), 110.2 (t, C₃), 61.4 (d, C₁), 19.6 (q, C₄). **HRMS (ESI)** m/z : calcd for C₁₀H₁₄N [M + H]⁺: 148.1120, found: 148.1116.

1-(Cyclohex-1-en-1-yl)ethanamine (2q)¹²



MW (g/mol): 125.21

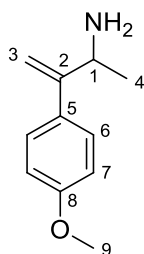
Molecular formula: C₈H₁₅N

¹² Pallavicini, M.; Valoti, E.; Villa, L.; Piccolo, O. *Tetrahedron: Asymmetry*, **2000**, *11*, 4017.

1-Acetyl-1-cyclohexene (0.77 mL, 6.0 mmol, 1.0 equiv.) was diluted in an ammonia solution (2 M in EtOH, 1.5 mL) and the titanium isopropoxide was added (3.64 mL, 12 mmol, 2 equiv.). After stirring for 12 h at room temperature, NaBH₄ (225 mg, 6 mmol, 1 equiv.) was added portion-wise at 0 °C. After stirring for an additional 12 h at room temperature, NaBH₄ (225 mg, 6 mmol, 1 equiv.) was added again at 0 °C. The reaction was quenched with a 2 M sol. of NH₄OH and the resulting precipitate was filtered through a pad of Celite[®]. The aqueous phase was extracted twice with EtOAc (2 x 10 mL), the combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography over silica gel (90:10 EtOAc/MeOH) to afford the pure 1-(cyclohex-1-en-1-yl)ethanamine.

IR (neat): 2924, 2855, 1561, 1447, 1373, 1096, 918, 802 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.54 (s, 1H, H₃), 3.32 (q, J = 6.6 Hz, 1H, H₁), 2.04–1.87 (m, 4H, H₄ + H₇), 1.65–1.52 (m, 4H, H₅ + H₆), 1.27 (s, 2H, NH), 1.12 (d, J = 6.6 Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 142.9 (s, C₂), 119.8 (d, C₃), 52.9 (d, C₁), 25.1 (t, C₄ or C₇), 24.5 (t, C₄ or C₇), 23.0 (t, C₅ or C₆), 22.9 (t, C₅ or C₆), 22.4 (q, C₈).

3-(4-Methoxyphenyl)but-3-en-2-amine (2r)

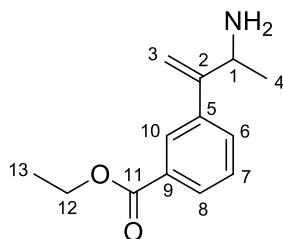


MW (g/mol): 177.24

Molecular formula: C₁₁H₁₅NO

This compound was prepared following general procedure C. **IR** (neat): 3371, 2961, 2835, 1607, 1509, 1243, 1179, 11031, 833, 794 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (d, J = 8.9 Hz, 2H, H₆), 6.88 (d, J = 8.9 Hz, 2H, H₇), 5.19 (t_{app}, J = 1.2 Hz, 1H, H₃), 5.16 (s, 1H, H₃), 4.00 (q, J = 6.6 Hz, 1H, H₁), 3.82 (s, 3H, H₉), 1.65 (s, 2H, NH), 1.23 (d, J = 6.6 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 159.2 (s, C₈), 154.3 (s, C₂), 133.6 (s, C₅), 128.0 (d, C₆), 113.8 (d, C₇), 109.5 (t, C₃), 55.4 (q, C₉), 50.0 (d, C₁), 23.0 (q, C₄). **HRMS (ESI)** m/z : calcd for C₁₁H₁₆NO [M + H]⁺: 178.1226, found: 178.1228.

Ethyl 3-(3-aminobut-1-en-2-yl)benzoate (2s)

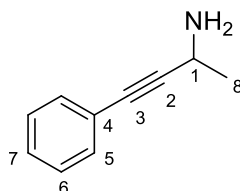


MW (g/mol): 219.28

Molecular formula: C₁₃H₁₇NO₂

This compound was prepared following general procedure C. **IR** (neat): 3375, 2978, 1715, 1367, 1291, 1246, 1108, 905, 764 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 8.03 (s, 1H, H_{Ar}), 7.97 (d, J = 7.7 Hz, 1H, H_{Ar}), 7.55 (d, J = 7.7 Hz, 1H, H_{Ar}), 7.41 (t, J = 7.7 Hz, 1H, H_{Ar}), 5.32 (s, 1H, H₃), 5.25 (s, 1H, H₃), 4.39 (q, J = 7.4 Hz, 2H, H₁₂), 4.06 (q, J = 6.6 Hz, 1H, H₁), 1.67 (brs, 2H, NH), 1.40 (t, J = 6.8 Hz, 3H, H₁₃), 1.22 (d, J = 6.6 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 166.7 (s, C₁₁), 154.2 (s, C₂), 141.6 (s, C₅), 131.4 (d, C_{Ar}), 130.7 (s, C₉), 128.7 (d, C_{Ar}), 128.5 (d, C_{Ar}), 128.0 (d, C_{Ar}), 111.7 (t, C₃), 61.2 (t, C₁₂), 50.1 (d, C₁), 23.1 (q, C₄), 14.5 (q, C₁₃). **HRMS (ESI) m/z :** calcd for C₁₃H₁₈NO₂ [M + H]⁺: 220.1332, found: 220.1333.

4-Phenylbut-3-yn-2-amine (2u)¹³



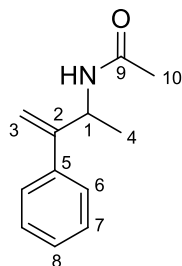
MW (g/mol): 145.20

Molecular formula: C₁₀H₁₁N

This compound was prepared following general procedure C. **¹H NMR** (400 MHz, CDCl₃) δ 7.44–7.38 (m, 2H, H_{Ph}), 7.32–7.26 (m, 3H, H_{Ph}), 3.92 (q, J = 6.8 Hz, 1H, H₁), 1.70 (s, 2H, NH₂), 1.44 (d, J = 6.8 Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 131.6 (s, C_{Ph}), 128.3 (s, C_{Ph}), 128.0 (s, C_{Ph}), 123.3 (s, C₄), 94.0 (s, C₂), 81.6 (s, C₃), 39.4 (s, C₁), 24.5 (s, C₈).

¹³ Blanchet, J.; Bonin, M.; Micouin, L.; Husson, H. P. *J. Org. Chem.*, **2000**, 65, 6423.

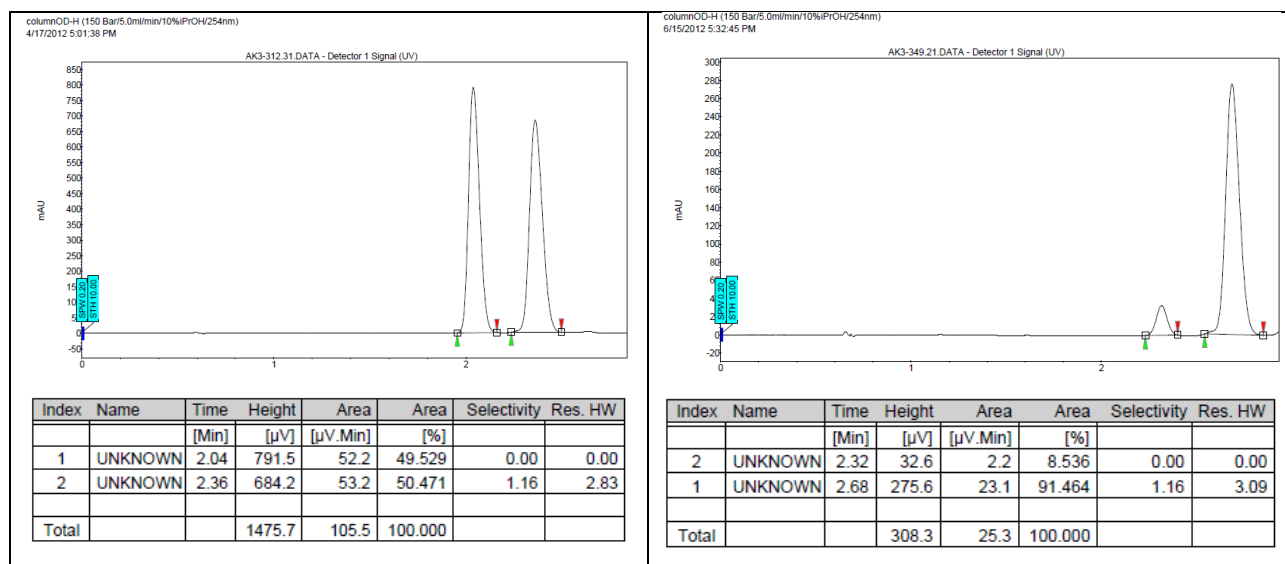
N-(3-Phenylbut-3-en-2-yl)acetamide (3a)



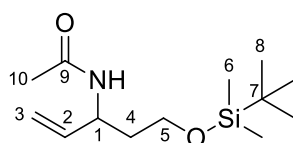
MW (g/mol): 189.25

Molecular formula: C₁₂H₁₅NO

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -30 (*c* 0.3, CHCl₃, 83% *ee*). **IR** (neat): 3274, 3058, 2975, 1639, 1543, 1372, 1279, 904, 778, 698 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.45–7.23 (m, 5H, H_{Ph}), 5.49 (s, 1H, NH), 5.34 (s, 1H, H₃), 5.23 (s, 1H, H₃), 5.12 (dq, *J* = 13.8, 7.0 Hz, 1H, H₁), 1.95 (s, 3H, H₁₀), 1.33 (d, *J* = 6.8 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 169.3 (s, C₉), 150.6 (s, C₂), 140.0 (s, C₅), 128.5 (d, C_{Ph}), 127.9 (d, C_{Ph}), 126.8 (d, C_{Ph}), 112.4 (t, C₃), 47.6 (d, C₁), 23.5 (q, C₄), 20.4 (q, C₁₀). **HRMS (ESI)** *m/z*: calcd for C₁₂H₁₅NaNO [M + Na]⁺: 212.1046, found: 212.1047. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t_R* = 2.3 min and *t_R* = 2.7 min.



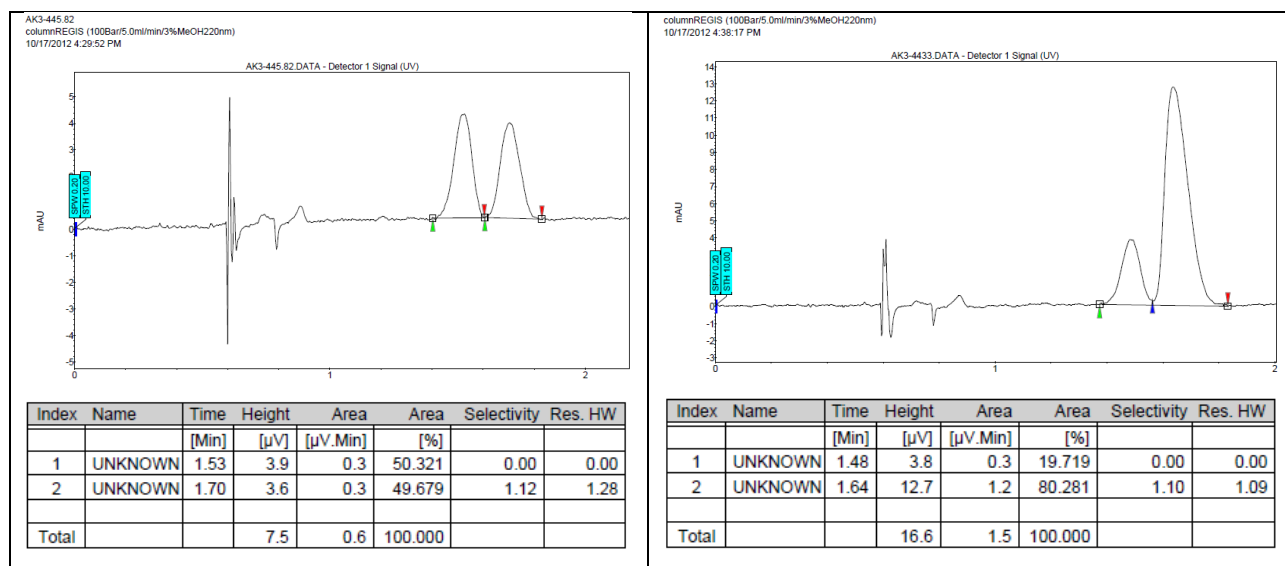
N-(5-((*tert*-Butyldimethylsilyl)oxy)pent-1-en-3-yl)acetamide (3b)



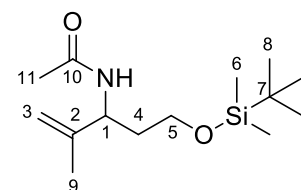
MW (g/mol): 257.44

Molecular formula: C₁₃H₂₇NO₂Si

This compound was prepared following general procedure D. $[\alpha]_D^{20} -7$ (c 0.89, CHCl_3 , 60% *ee*).
IR (neat): 3273, 3082, 2931, 11632, 1546, 1429, 1374, 1304, 1056, 919, 836, 776 cm^{-1} .
 ^1H NMR (400 MHz, CDCl_3) δ 6.63 (s, 1H, NH), 5.77 (ddd, $J = 17.1, 10.5, 5.0$ Hz, 1H, H_2), 5.16 (dt_{app}, $J = 7.5, 1.4$ Hz, 1H, H_3), 5.13 (d, $J = 1.6$ Hz, 1H, H_3), 4.62 (m, 1H, H_1), 3.79 (td, $J = 10.0, 3.7$ Hz, 1H, H_5), 3.70 (m, 1H, H_5), 1.97 (s, 3H, H_{10}), 1.89 (m, 1H, H_4), 1.68 (m, 1H, H_4), 0.91 (s, 9H, H_8), 0.06 (s, 6H, H_6).
 ^{13}C NMR (100 MHz, CDCl_3) δ 169.3 (s, C_9), 137.4 (d, C_2), 114.9 (t, C_3), 60.5 (t, C_5), 50.4 (d, C_1), 35.7 (t, C_4), 26.0 (q, C_8), 23.5 (q, C_{10}), 18.2 (s, C_7), -5.4 (q, C_6).
HRMS (ESI) m/z : calcd for $\text{C}_{13}\text{H}_{27}\text{NaNO}_2\text{Si}$ $[\text{M} + \text{Na}]^+$: 280.1703, found: 280.1705.
SFC: REGISPACK, Pressure = 100 bar, $\text{CO}_2/\text{MeOH} = 97:3$, Flow rate = 5 mL/min, UV = 220 nm, $t_R = 1.5$ min and $t_R = 1.6$ min.



N-(5-((*tert*-Butyldimethylsilyl)oxy)-2-methylpent-1-en-3-yl)acetamide (**3c**)

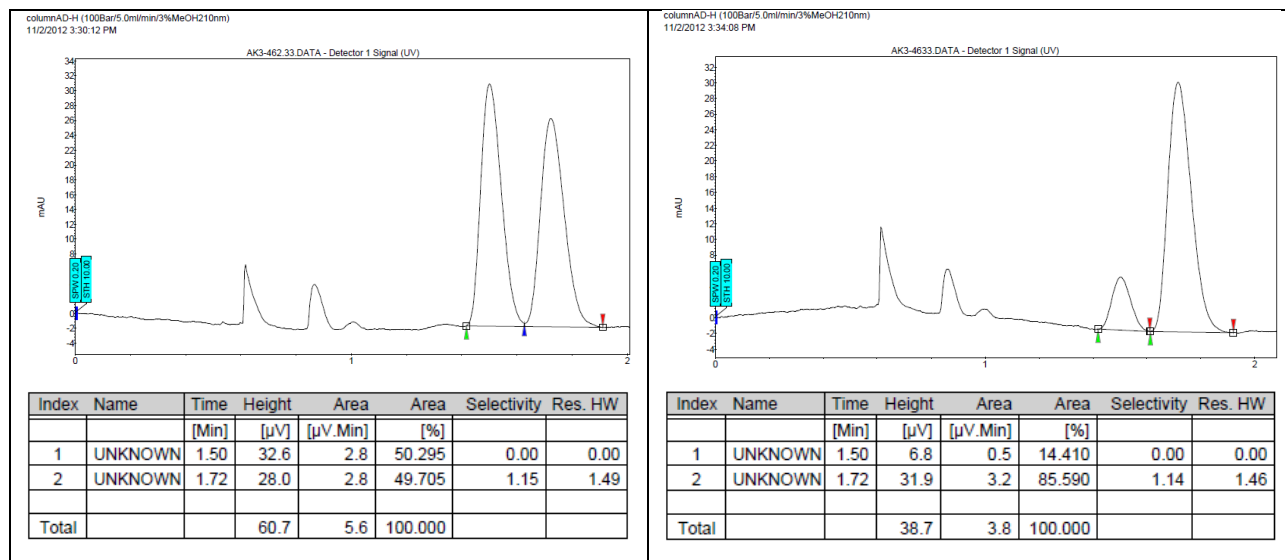


MW (g/mol): 271.47

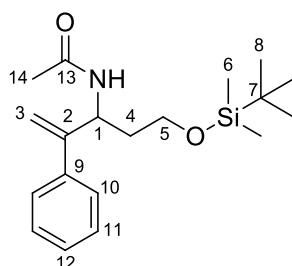
Molecular formula: $\text{C}_{14}\text{H}_{29}\text{NO}_2\text{Si}$

This compound was prepared following general procedure D. $[\alpha]_D^{20} +2$ (c 0.25, CHCl_3 , 71% *ee*).
IR (neat): 3278, 2954, 2929, 2857, 1645, 1550, 1373, 1254, 1095, 834, 775 cm^{-1} .
 ^1H NMR (400 MHz, CDCl_3) δ 6.79 (d, $J = 6.0$ Hz, 1H, NH), 4.88 (s, 1H, H_3), 4.86 (s, 1H, H_3), 4.46 (m, 1H, H_1), 3.75–3.63 (m, 2H, H_5), 1.96 (s, 3H, H_{11}), 1.86 (ddd, $J = 18.8, 9.3, 4.7$ Hz, 1H, H_4), 1.79–1.68 (m, 4H, $\text{H}_4 + \text{H}_9$), 0.90 (s, 9H, H_8), 0.05 (s, 6H, H_6).
 ^{13}C NMR (100 MHz, CDCl_3)

δ 169.1 (s, C₁₀), 143.7 (s, C₂), 111.0 (t, C₃), 60.5 (t, C₅), 53.4 (d, C₁), 33.9 (t, C₄), 26.0 (q, C₈), 23.5 (q, C₁₁), 20.1 (q, C₉), 18.2 (s, C₇), -5.5 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₄H₂₉NaNO₂Si [M + Na]⁺: 294.1860, found: 294.1855. **SFC**: AD-H, Pressure = 100 bar, CO₂/MeOH = 97:3, Flow rate = 5 mL/min, UV = 210 nm, t_R = 1.5 min and t_R = 1.7 min.



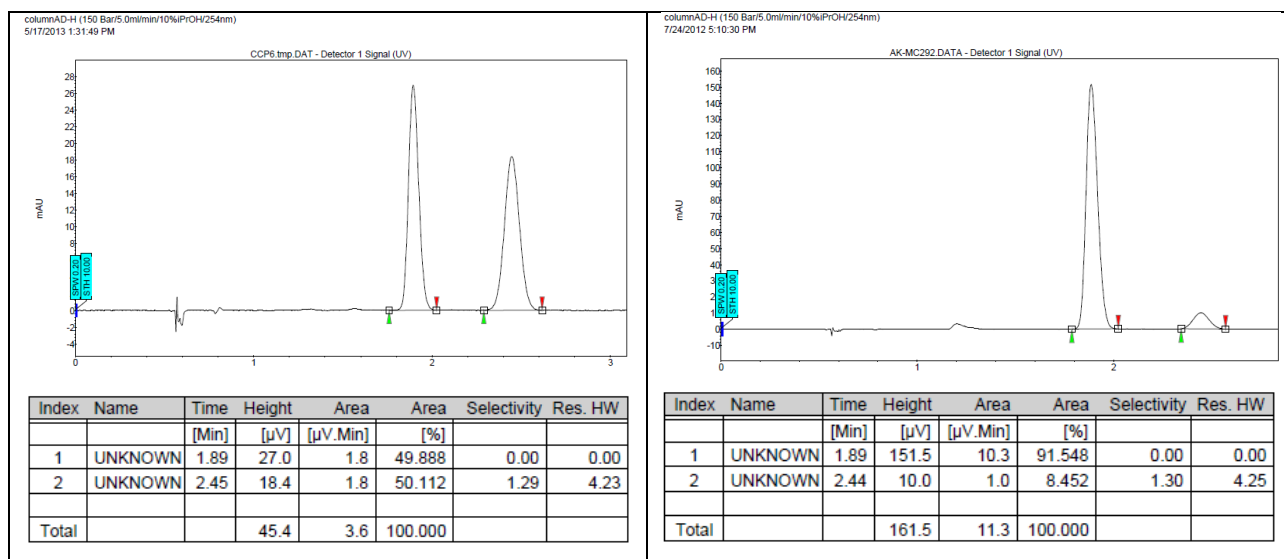
N-(5-((*tert*-Butyldimethylsilyloxy)-2-phenylpent-1-en-3-yl)acetamide (3d)



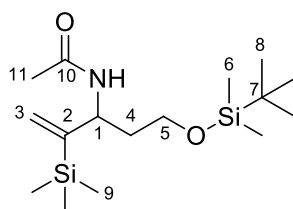
MW (g/mol): 333.54

Molecular formula: C₁₉H₃₁NO₂Si

This compound was prepared following general procedure D. [α]_D²⁰ +80 (*c* 0.8, CHCl₃, 83% *ee*). **IR** (neat): 3276, 2954, 2928, 2856, 1649, 1548, 1254, 1091, 835, 774, 706 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.38–7.32 (m, 2H, H_{Ph}), 7.30–7.19 (m, 3H, H_{Ph}), 6.97 (d, *J* = 6.7 Hz, 1H, NH), 5.24 (s, 1H, H₃), 5.14–5.05 (m, 2H, H₃ + H₁), 3.67 (td, *J* = 10.4, 3.0 Hz, 1H, H₅), 3.52 (dt, *J* = 10.3, 4.2 Hz, 1H, H₅), 1.95 (s, 3H, H₁₄), 1.79 (m, 1H, H₄), 1.51 (m, 1H, H₄), 0.86 (s, 9H, H₈), 0.01 (s, 3H, H₆), -0.01 (s, 3H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 169.1 (s, C₁₃), 148.5 (s, C₂), 140.6 (s, C₉), 128.5 (d, C_{Ph}), 127.8 (d, C_{Ph}), 127.0 (d, C_{Ph}), 112.8 (t, C₃), 60.6 (t, C₅), 52.0 (d, C₁), 33.7 (t, C₄), 26.0 (q, C₈), 23.6 (s, C₁₄), 18.2 (s, C₇), -5.5 (q, C₆). **HRMS (ESI)** m/z : calcd for C₁₉H₃₁NaNO₂Si [M + Na]⁺: 356.2016, found: 356.2013. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, t_R = 1.9 min and t_R = 2.4 min.



***N*-(5-((*tert*-Butyldimethylsilyl)oxy)-2-(trimethylsilyl)pent-1-en-3-yl)acetamide (3e)**

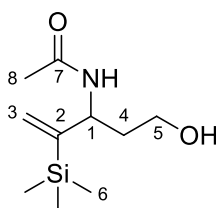


MW (g/mol): 329.63

Molecular formula: C₁₆H₃₅NO₂Si₂

This compound was prepared following general procedure D. The *ee* was determined by chiral SFC analysis after deprotection of the silyl ether. [α]_D²⁰ -22 (*c* 1.0, CHCl₃, 84% *ee*). **IR** (neat): 3274, 2955, 2857, 1649, 1552, 1250, 1098, 837, 775 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 6.71 (d, *J* = 6.3 Hz, 1H, NH), 5.71 (m, 1H, H₃), 5.47 (m, 1H, H₃), 4.72 (m, 1H, H₁), 3.78–3.60 (m, 2H, H₅), 1.96 (s, 3H, H₁₁), 1.90 (m, 1H, H₄), 1.64 (m, 1H, H₄), 0.92 (s, 9H, H₈), 0.13 (s, 9H, H₉), 0.07 (s, 6H, H₆). **¹³C NMR** (100 MHz, CDCl₃) δ 168.8 (s, C₁₀), 151.3 (s, C₂), 124.1 (t, C₃), 60.6 (t, C₅), 52.2 (d, C₁), 35.7 (t, C₄), 26.0 (q, C₈), 23.6 (q, C₁₁), 18.3 (s, C₇), -0.8 (q, C₉), -5.4 (q, C₆). **HRMS (ESI)** *m/z*: calcd for C₁₆H₃₅NaNO₂Si₂ [M + Na]⁺: 352.2098, found: 352.2095.

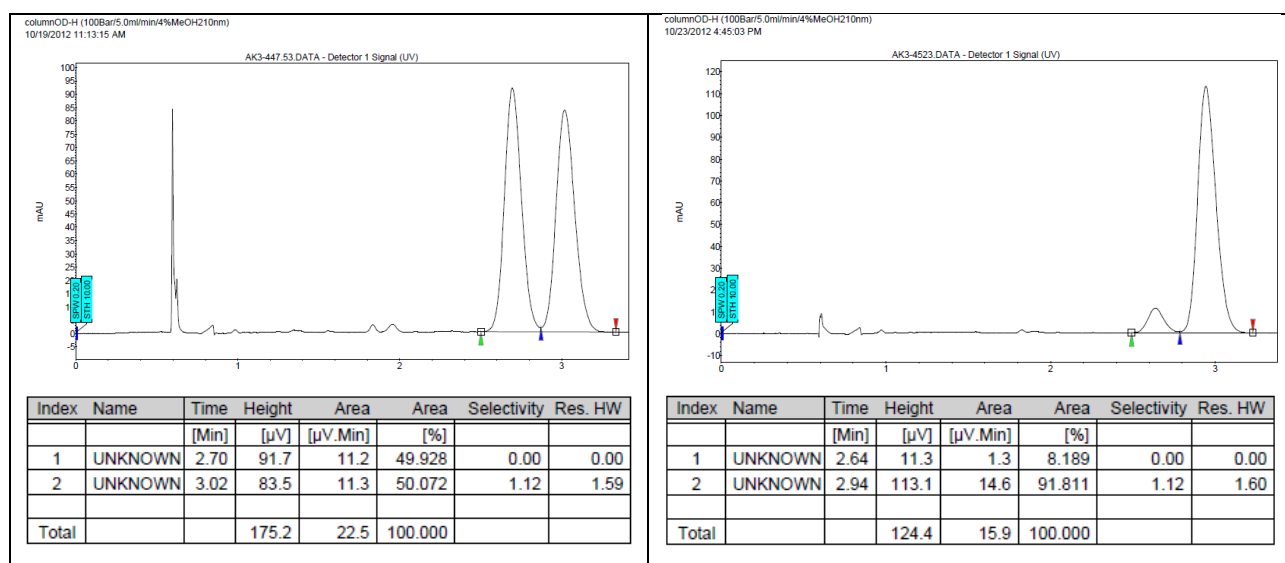
***N*-(5-Hydroxy-2-(trimethylsilyl)pent-1-en-3-yl)acetamide**



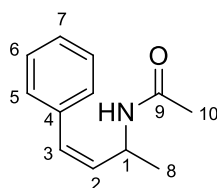
MW (g/mol): 215.36

Molecular formula: C₁₀H₂₁NO₂Si

$[\alpha]_D^{20}$ -70 (c 0.96, CHCl_3 , 84% ee). **IR** (neat): 3278, 1650, 1542, 1374, 1249, 838, 759 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 5.79 (t, J = 1.5 Hz, 1H, H_3), 5.64 (d, J = 7.9 Hz, 1H, NH), 5.54 (dd, J = 1.6, 0.9 Hz, 1H, H_3), 4.77 (m, 1H, H_1), 3.69 (ddd, J = 11.9, 5.2, 3.0 Hz, 1H, H_5), 3.57 (m, 1H, H_5), 2.04 (s, 3H, H_8), 1.93 (m, 1H, H_4), 1.55 (m, 1H, H_4), 0.14 (s, 9H, H_6). **^{13}C NMR** (100 MHz, CDCl_3) δ 170.8 (s, C_7), 152.2 (s, C_2), 125.0 (t, C_3), 58.8 (t, C_5), 48.8 (d, C_1), 37.0 (t, C_4), 23.4 (q, C_8), -1.1 (q, C_6). **HRMS (ESI)** m/z : calcd for $\text{C}_{10}\text{H}_{22}\text{NO}_2\text{Si}$ $[\text{M} + \text{H}]^+$: 216.1414, found: 216.1412. **SFC**: OD-H, Pressure = 100 bar, CO_2/MeOH = 96:4, Flow rate = 5 mL/min, UV = 210 nm, t_R = 2.6 min and t_R = 2.9 min.



(Z)-N-(4-Phenylbut-3-en-2-yl)acetamide (3f)

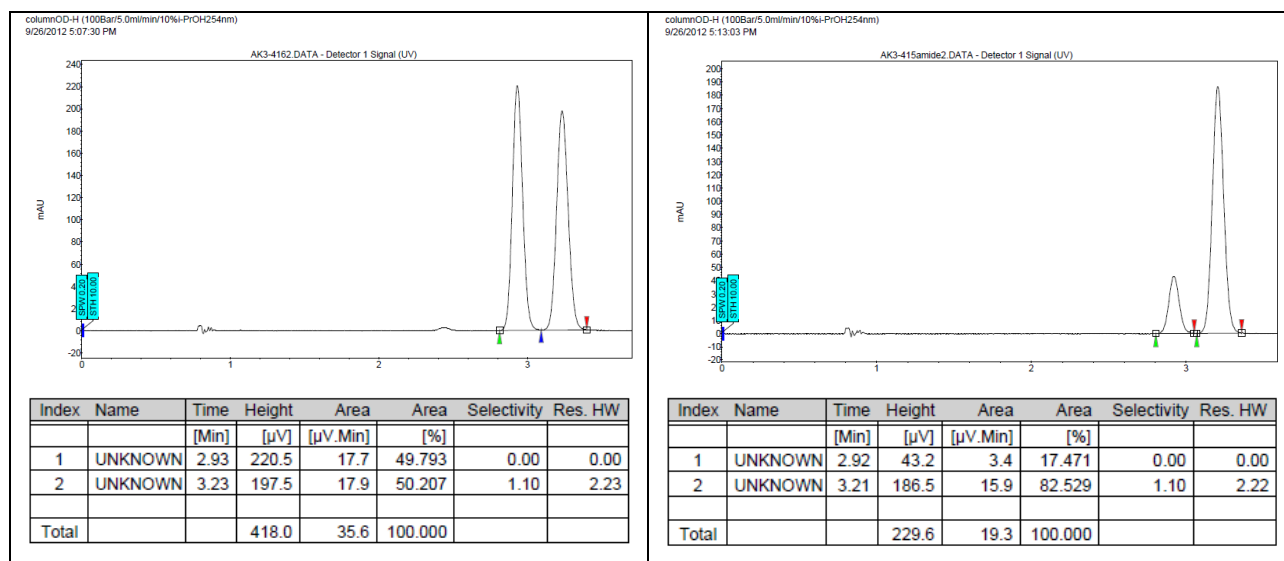


MW (g/mol): 189.25

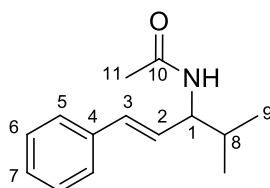
Molecular formula: $\text{C}_{12}\text{H}_{15}\text{NO}$

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ $+6$ (c 0.13, CHCl_3 , 64% ee). **IR** (neat): 3252, 3080, 2974, 2928, 1634, 1556, 1493, 1445, 1371, 1297, 1136, 799, 773, 700, 605 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.33–7.15 (m, 5H, H_{Ph}), 6.41 (d, J = 11.6 Hz, 1H, H_3), 5.53 (s, 1H, NH), 5.45 (dd, J = 11.5, 9.3 Hz, 1H, H_2), 4.94 (m, 1H, H_2), 1.86 (s, 3H, H_{10}), 1.22 (d, J = 6.6 Hz, 3H, H_8). **^{13}C NMR** (100 MHz, CDCl_3) δ 169.2 (s, C_9), 136.4 (s, C_4), 132.9 (d, C_2), 130.5 (d, C_3), 128.8 (d, C_{Ph}), 128.5 (d, C_{Ph}), 127.4 (d, C_{Ph}), 44.0 (d, C_1), 23.6 (q, C_{10}), 22.1 (q, C_8). **HRMS (ESI)** m/z : calcd for $\text{C}_{12}\text{H}_{15}\text{NaNO}$ $[\text{M} + \text{Na}]^+$: 212.1046, found: 212.1046. **SFC**: OD-H,

Pressure = 100 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 220 nm, *t*_R = 2.9 min and *t*_R = 3.2 min.



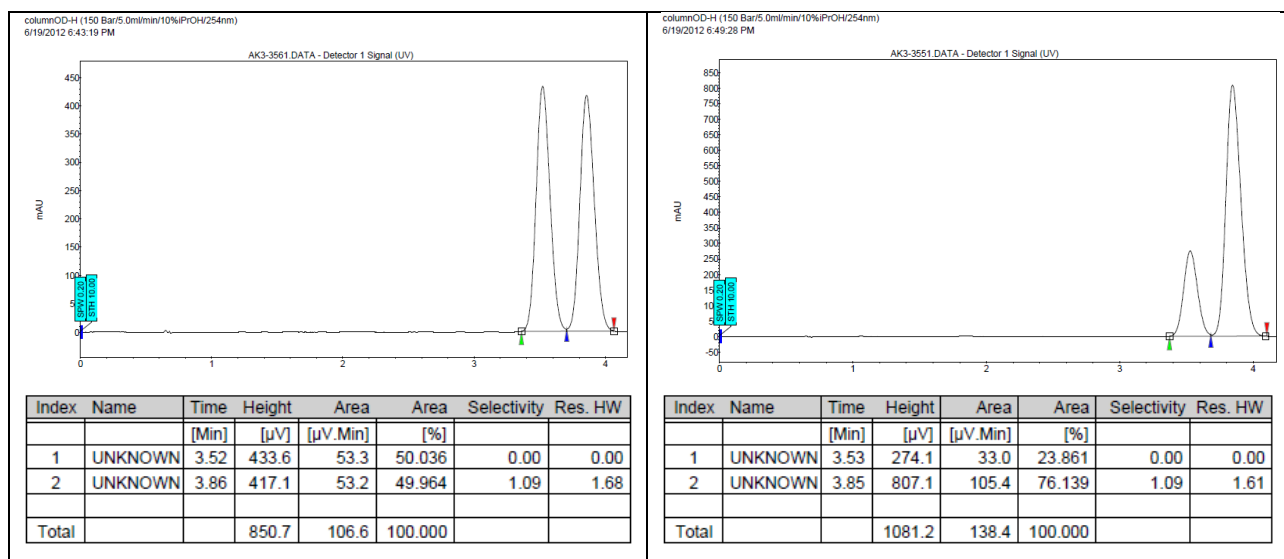
(*E*)-*N*-(4-Methyl-1-phenylpent-1-en-3-yl)acetamide (3h)



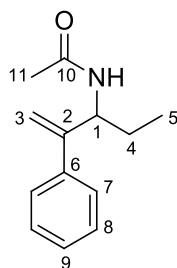
MW (g/mol): 217.31

Molecular formula: C₁₄H₁₉NO

This compound was prepared following general procedure D. [α]_D²⁰ −49 (*c* 0.37, CHCl₃, 52% *ee*). **IR** (neat): 3280, 2962, 2927, 1636, 1550, 1374, 1289, 967, 744, 691, 605 cm^{−1}. **¹H NMR** (400 MHz, CDCl₃) δ 7.41–7.19 (m, 5H, H_{Ph}), 6.52 (d, *J* = 15.9 Hz, 1H, H₃), 6.09 (dd, *J* = 15.9, 6.9 Hz, 1H, H₂), 5.56 (d, *J* = 8.1 Hz, 1H, NH), 4.51 (m, 1H, H₁), 2.06 (s, 3H, H₁₁), 1.90 (m, 1H, H₈), 0.97 (d, *J* = 6.8 Hz, 6H, H₉). **¹³C NMR** (100 MHz, CDCl₃) δ 169.5 (s, C₁₀), 136.9 (s, C₄), 131.6 (d, C₃), 128.7 (d, C₂), 128.4 (d, C_{Ph}), 127.7 (d, C_{Ph}), 126.5 (d, C_{Ph}), 56.6 (d, C₁), 32.7 (d, C₈), 23.7 (q, C₁₁), 18.9 (q, C₉), 18.7 (q, C₉). **HRMS (ESI)** *m/z*: calcd for C₁₄H₁₉NaNO [M + Na]⁺: 240.1359, found: 240.1353. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 3.5 min and *t*_R = 3.9 min.



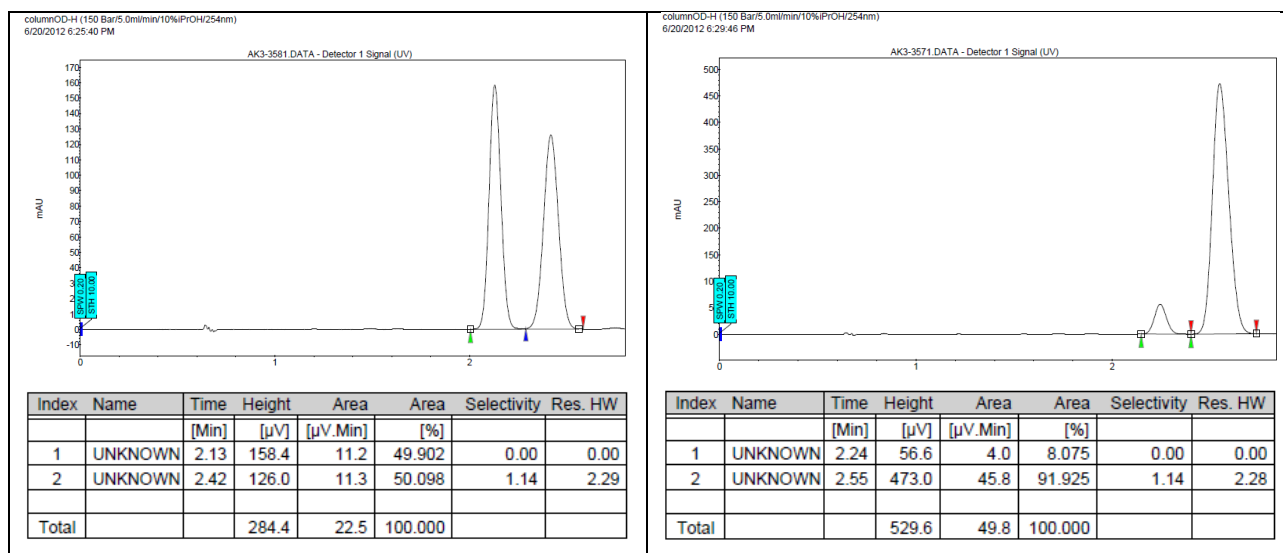
N-(2-Phenylpent-1-en-3-yl)acetamide (3i)



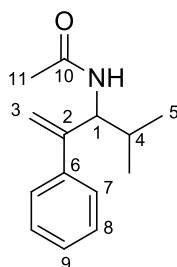
MW (g/mol): 203.28

Molecular formula: C₁₃H₁₇NO

This compound was prepared following general procedure D. [α]_D²⁰ +48 (*c* 0.60, CHCl₃, 84% *ee*). **IR** (neat): 3277, 2966, 2933, 1647, 1542, 1373, 1295, 777, 699 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.43–7.25 (m, 5H, H_{Ph}), 5.67 (d, *J* = 6.3 Hz, 1H, NH), 5.30 (s, 1H, H₃), 5.18 (s, 1H, H₃), 4.90 (m, 1H, H₁), 2.01 (s, 3H, H₁₁), 1.70 (m, 1H, H₄), 1.51 (m, 1H, H₄), 0.92 (t, *J* = 7.4 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 169.6 (s, C₁₀), 149.8 (s, C₂), 140.6 (s, C₆), 128.5 (d, C_{Ph}), 127.8 (d, C_{Ph}), 126.9 (d, C_{Ph}), 112.7 (t, C₃), 53.8 (d, C₁), 27.4 (t, C₄), 23.5 (q, C₁₁), 10.5 (q, C₅). **HRMS (ESI)** *m/z*: calcd for C₁₃H₁₇NaNO [M + Na]⁺: 226.1202, found: 226.1204. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 2.2 min and *t*_R = 2.5 min.



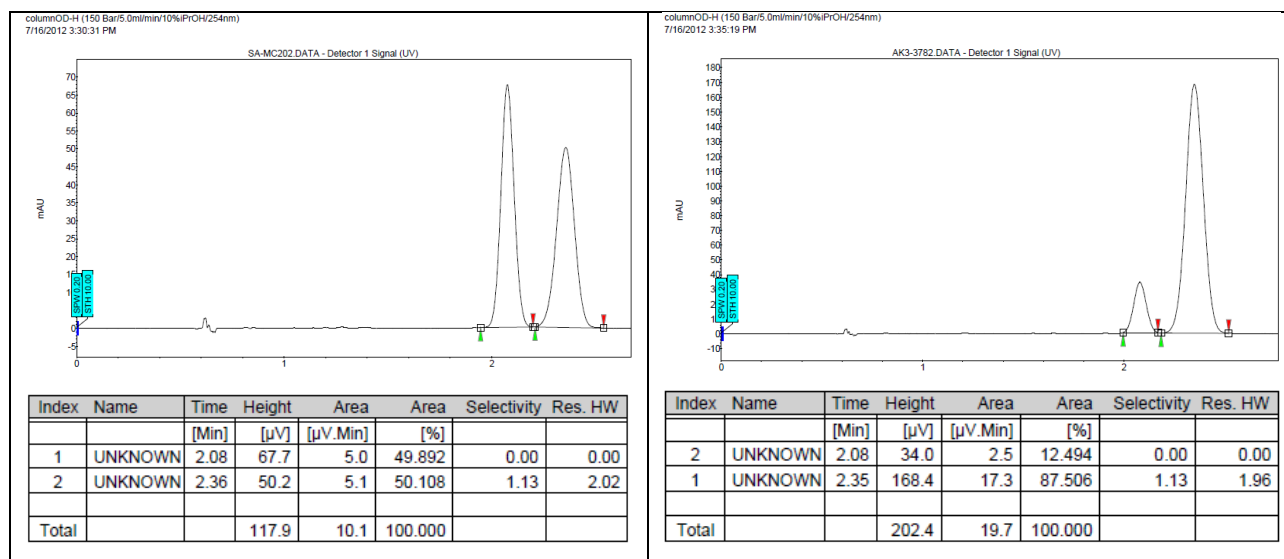
N-(4-Methyl-2-phenylpent-1-en-3-yl)acetamide (**3j**)



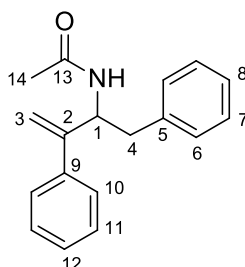
MW (g/mol): 217.31

Molecular formula: C₁₄H₁₉NO

This compound was prepared following general procedure D. $[\alpha]_D^{20} +89$ (*c* 1.1, CHCl₃, 75% *ee*). **IR** (neat): 3307, 2960, 2930, 1645, 1535, 1371, 1279, 897, 774, 693 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.43–7.27 (m, 5H, H_{Ph}), 5.61 (d, *J* = 7.9 Hz, 1H, NH), 5.27 (s, 1H, H₃), 5.12 (s, 1H, H₃), 4.88 (dd, *J* = 9.5, 5.8 Hz, 1H, H₁), 2.06 (s, 3H, H₁₁), 1.83 (m, 1H, H₄), 0.94 (d, *J* = 6.8 Hz, 3H, H₅), 0.83 (d, *J* = 6.8 Hz, 3H, H₅). **¹³C NMR** (100 MHz, CDCl₃) δ 169.6 (s, C₁₀), 149.8 (s, C₂), 141.1 (s, C₆), 128.5 (d, C_{Ph}), 127.7 (d, C_{Ph}), 127.1 (d, C_{Ph}), 113.1 (t, C₃), 57.6 (d, C₁), 30.5 (d, C₄), 23.6 (q, C₁₁), 20.2 (q, C₅), 17.0 (q, C₅). **HRMS (ESI)** *m/z*: calcd for C₁₄H₁₉NaNO [M + Na]⁺: 240.1359, found: 240.1352. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 2.0 min and *t*_R = 2.3 min.



N-(1,3-Diphenylbut-3-en-2-yl)acetamide (**3k**)¹⁴

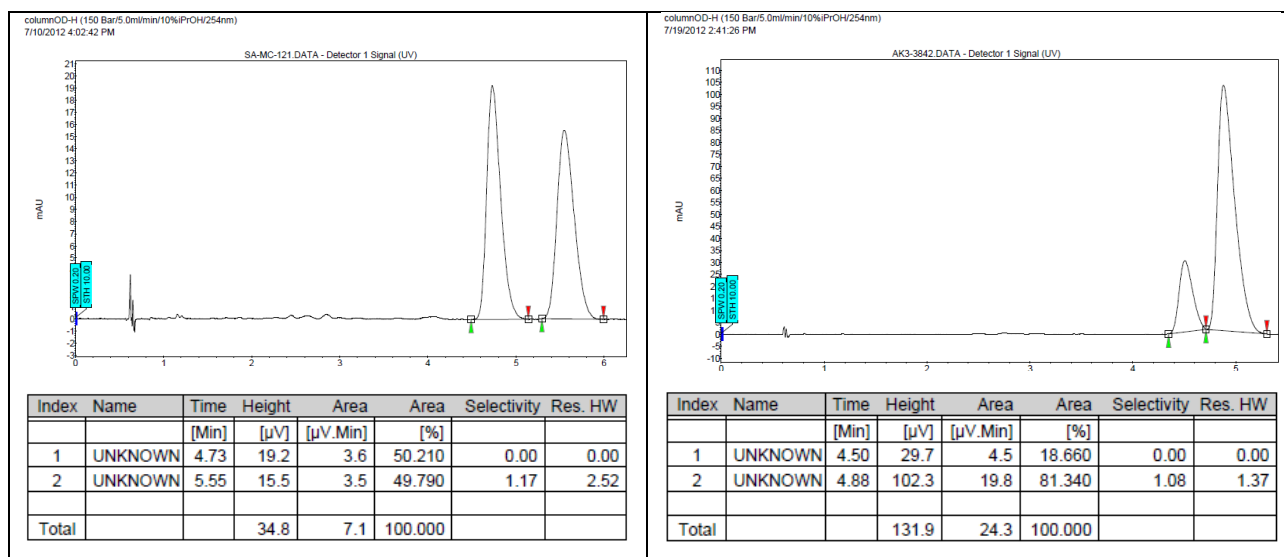


MW (g/mol): 265.35

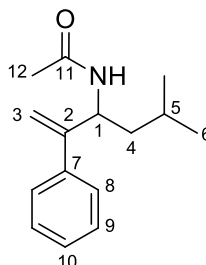
Molecular formula: C₁₈H₁₉NO

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ +58 (*c* 1.08, CHCl₃, 63% *ee*). **IR** (neat): 3309, 3027, 2926, 1651, 1547, 1375, 910, 778, 744, 697 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.52–7.44 (m, 2H, H_{Ph}), 7.39–7.20 (m, 6H, H_{Ph}), 7.17–7.10 (m, 2H, H_{Ph}), 5.59 (d, *J* = 8.4 Hz, 1H, NH), 5.35 (m, 1H, H₁), 5.31 (s, 1H, H₃), 5.07 (s, 1H, H₃), 3.00 (dd, *J* = 14.1, 5.8 Hz, 1H, H₄), 2.78 (dd, *J* = 14.1, 7.2 Hz, 1H, H₄), 1.94 (s, 3H, H₁₄). **¹³C NMR** (100 MHz, CDCl₃) δ 169.3 (s, C₁₃), 149.2 (s, C₂), 140.8 (s, C₉), 137.4 (s, C₅), 129.4 (d, C_{Ph}), 128.6 (d, C_{Ph}), 128.6 (d, C_{Ph}), 128.0 (d, C_{Ph}), 127.1 (d, C_{Ph}), 126.7 (d, C_{Ph}), 113.2 (t, C₃), 52.9 (d, C₁), 39.8 (t, C₄), 23.5 (q, C₁₄). **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 4.5 min and *t*_R = 4.9 min.

¹⁴ Anderson, L. L.; Woerpel, K. A. *Org. Lett.*, **2009**, *11*, 425.



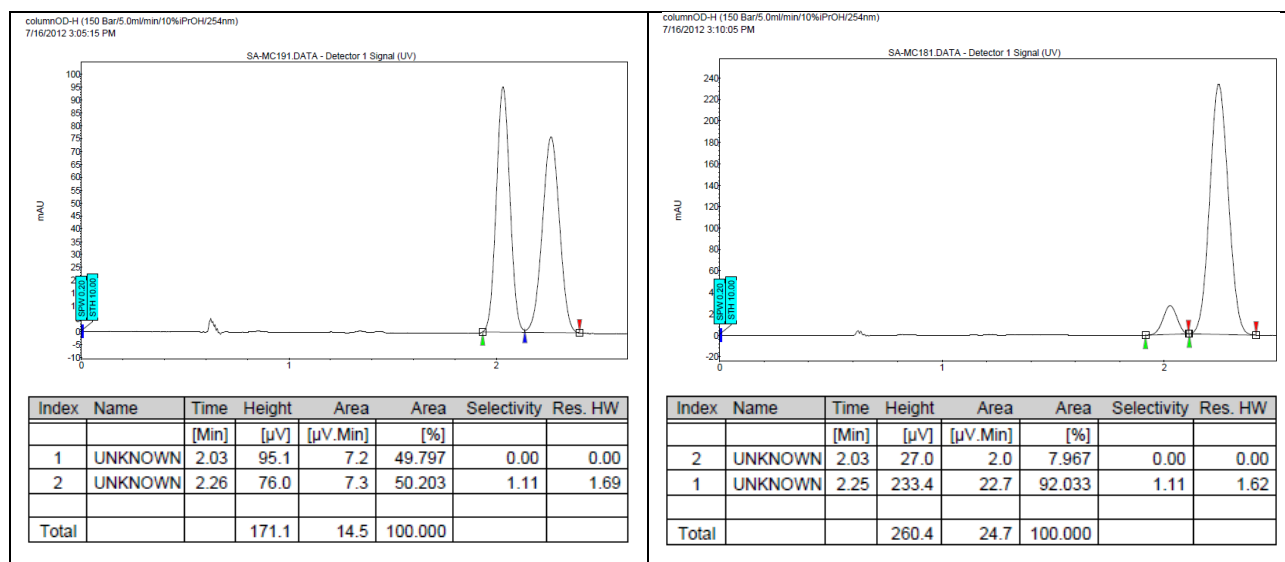
N-(5-Methyl-2-phenylhex-1-en-3-yl)acetamide (**3l**)



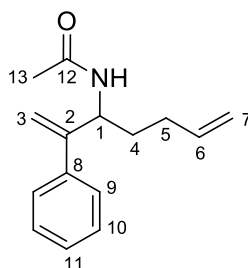
MW (g/mol): 231.33

Molecular formula: C₁₅H₂₁NO

This compound was prepared following general procedure D. $[\alpha]_D^{20} +29$ (*c* 1.15, CHCl₃, 84% *ee*).
IR (neat): 3272, 3059, 2955, 1644, 1546, 1370, 1296, 1160, 900, 776, 698 cm⁻¹.
¹H NMR (400 MHz, CDCl₃) δ 7.44–7.23 (m, 5H, H_{Ph}), 5.59 (d, *J* = 8.8 Hz, 1H, NH), 5.27 (s, 1H, H₃), 5.19 (s, 1H, H₃), 5.03 (td app, *J* = 9.1, 5.7 Hz, 1H, H₁), 2.00 (s, 3H, H₁₂), 1.66 (m, 1H, H₅), 1.51–1.32 (m, 2H, H₄), 0.92 (d, *J* = 6.6 Hz, 3H, H₆), 0.87 (d, *J* = 6.7 Hz, 3H, H₆).
¹³C NMR (100 MHz, CDCl₃) δ 169.4 (s, C₁₁), 150.8 (s, C₂), 140.5 (s, C₇), 128.5 (d, C_{Ph}), 127.8 (d, C_{Ph}), 127.0 (d, C_{Ph}), 112.3 (t, C₃), 50.8 (d, C₁), 44.2 (t, C₄), 25.3 (d, C₅), 23.5 (q, C₁₂), 23.2 (q, C₆), 22.1 (q, C₆).
HRMS (ESI) *m/z*: calcd for C₁₅H₂₁NaNO [M + Na]⁺: 254.1515, found: 254.1518.
SFC: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 2.0 min and *t*_R = 2.3 min.



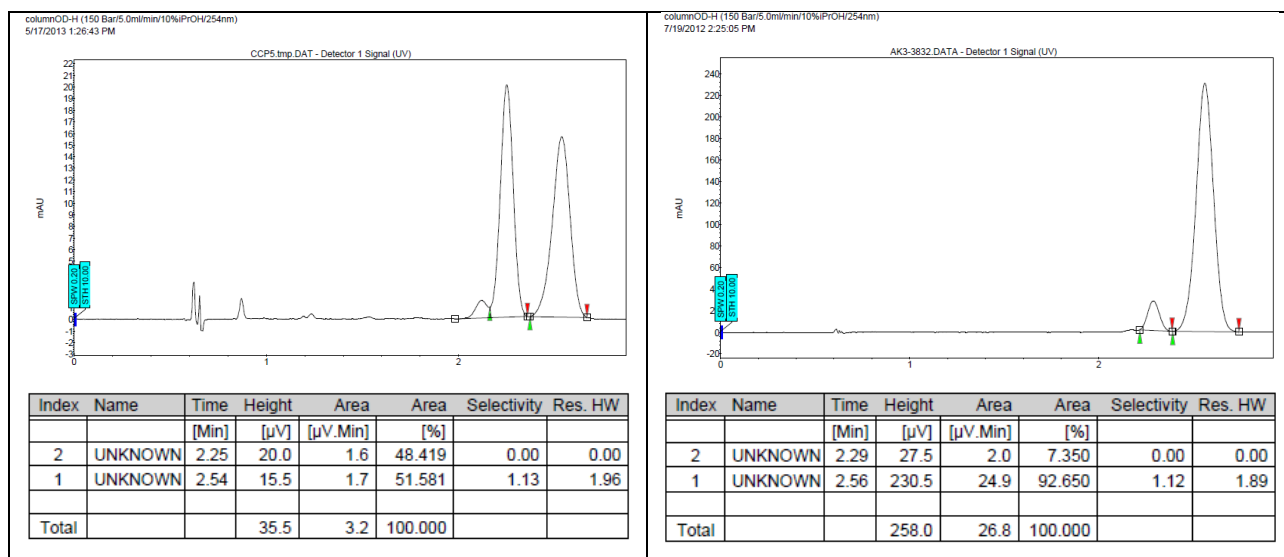
N-(2-Phenylhepta-1,6-dien-3-yl)acetamide (3m)



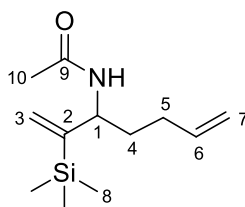
MW (g/mol): 229.32

Molecular formula: C₁₅H₁₉NO

This compound was prepared following general procedure D. [α]_D²⁰ +51 (*c* 1.03, CHCl₃, 85% *ee*).
IR (neat): 3273, 3077, 2930, 1643, 1543, 1442, 1373, 1298, 906, 777, 699 cm⁻¹.
¹H NMR (400 MHz, CDCl₃) δ 7.46–7.28 (m, 5H, H_{Ph}), 5.78 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H, H₆), 5.62 (d, *J* = 5.1 Hz, 1H, NH), 5.32 (s, 1H, H₃), 5.20 (s, 1H, H₃), 5.05–4.91 (m, 3H, H₇ + H₁), 2.16–2.06 (m, 2H, H₅), 2.01 (s, 3H, H₁₃), 1.76 (m, 1H, H₄), 1.61 (dt, *J* = 15.2, 7.9 Hz, 1H, H₄).
¹³C NMR (100 MHz, CDCl₃) δ 169.4 (s, C₁₂), 149.9 (s, C₂), 140.4 (s, C₈), 137.9 (d, C₆), 128.5 (d, C_{Ph}), 127.9 (d, C_{Ph}), 127.0 (d, C_{Ph}), 115.4 (t, C₇), 112.9 (t, C₃), 52.1 (d, C₁), 33.7 (t, C₄), 30.4 (t, C₅), 23.6 (q, C₁₃).
HRMS (ESI) *m/z*: calcd for C₁₅H₁₉NaNO [M + Na]⁺: 252.1360, found: 252.1362.
SFC: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 2.3 min and *t*_R = 2.5 min.



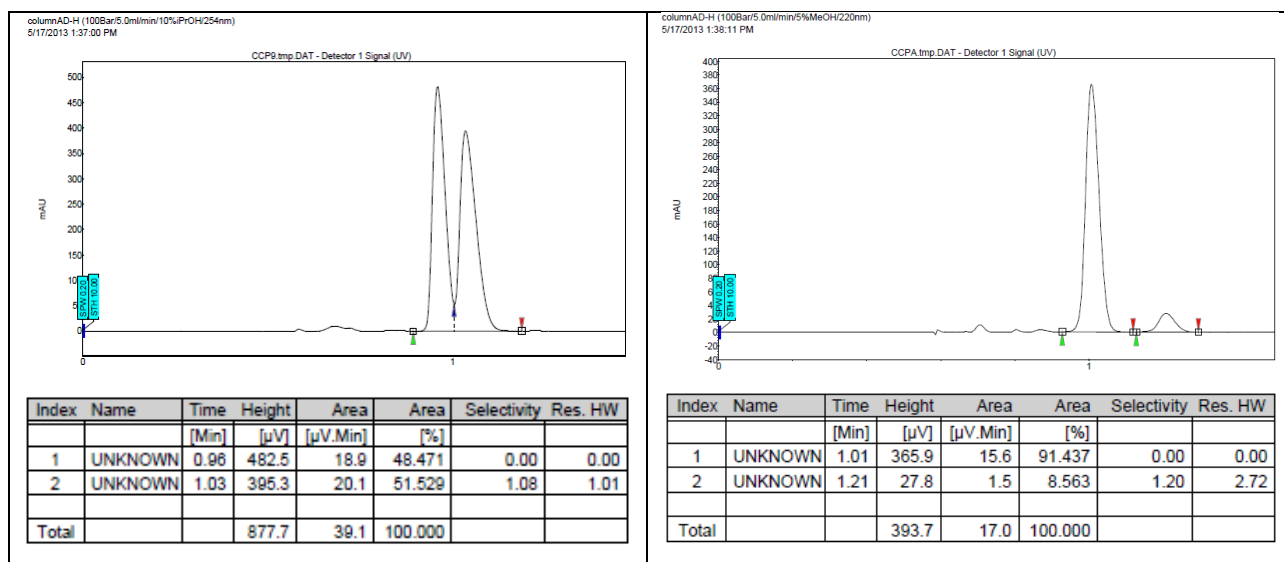
N-(2-(Trimethylsilyl)hepta-1,6-dien-3-yl)acetamide (**3n**)



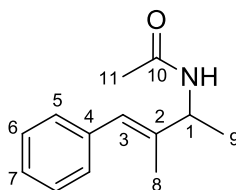
MW (g/mol): 225.40

Molecular formula: C₁₂H₂₃NOSi

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -53 (*c* 0.84, CHCl₃, 84% *ee*). **IR** (neat): 3275, 2954, 11643, 1545, 1373, 1249, 837, 758 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 5.80 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H, H₆), 5.72 (s, 1H, H₃), 5.56 (d, *J* = 8.1 Hz, 1H, NH), 5.46 (m, 1H, H₃), 5.05–4.91 (m, 2H, H₇), 4.58 (m, 1H, H₁), 2.09–2.01 (m, 2H, H₅), 1.97 (s, 3H, H₁₀), 1.71 (m, 1H, H₄), 1.57 (m, 1H, H₄), 0.12 (s, 9H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 169.1 (s, C₉), 152.4 (s, C₂), 138.0 (d, C₆), 124.6 (t, C₃), 115.1 (t, C₇), 52.5 (d, C₁), 34.1 (t, C₄), 30.5 (t, C₅), 23.6 (q, C₁₀), -0.9 (q, C₈). **HRMS (ESI)** *m/z*: calcd for C₁₂H₂₄NOSi [M + H]⁺: 226.1622, found: 226.1624. **SFC**: AD-H, Pressure = 100 bar, CO₂/MeOH = 95:5, Flow rate = 5 mL/min, UV = 220 nm, *t*_R = 1.8 min and *t*_R = 2.0 min.



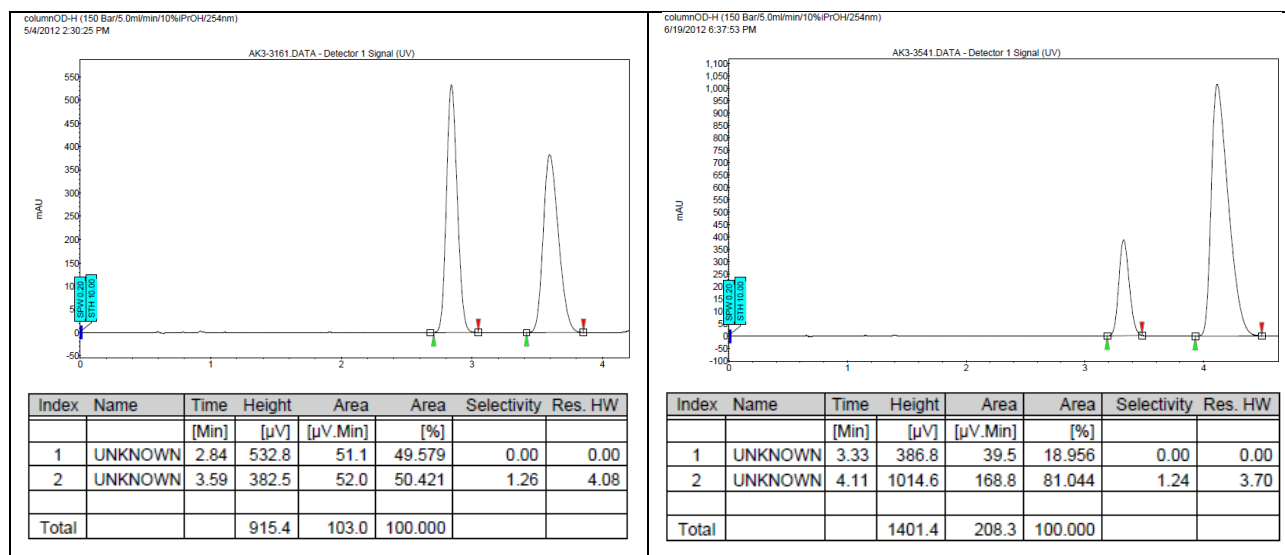
(E)-N-(3-Methyl-4-phenylbut-3-en-2-yl)acetamide (3o)



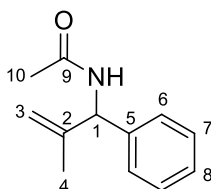
MW (g/mol): 203.28

Molecular formula: C₁₃H₁₇NO

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -77 (*c* 0.95, CHCl₃, 62% *ee*). **IR** (neat): 3277, 2974, 2930, 1646, 1545, 1444, 1372, 1301, 1132, 745, 699 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.37-7.29 (m, 2H, H_{Ph}), 7.27-7.18 (m, 3H, H_{Ph}), 6.46 (s, 1H, H₃), 5.65 (s, 1H, NH), 4.63 (quint_{app}, *J* = 7.4 Hz, 1H, H₁), 2.03 (s, 3H, H₁₁), 1.87 (d, *J* = 1.3 Hz, 3H, H₈), 1.35 (d, *J* = 6.9 Hz, 3H, H₉). **¹³C NMR** (100 MHz, CDCl₃) δ 169.4 (s, C₁₀), 138.8 (s, C₂ or C₄), 137.7 (s, C₂ or C₄), 129.1 (d, C_{Ph}), 128.2 (d, C_{Ph}), 126.6 (d, C_{Ph}), 125.1 (d, C₃), 51.7 (d, C₁), 23.6 (q, C₁₀), 19.9 (q, C₉), 15.4 (q, C₈). **HRMS (ESI)** *m/z*: calcd for C₁₃H₁₇NaNO [M + Na]⁺: 226.1202, found: 226.1204. **SFC**: OD-H, Pressure = 100 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 3.3 min and *t*_R = 4.1 min.



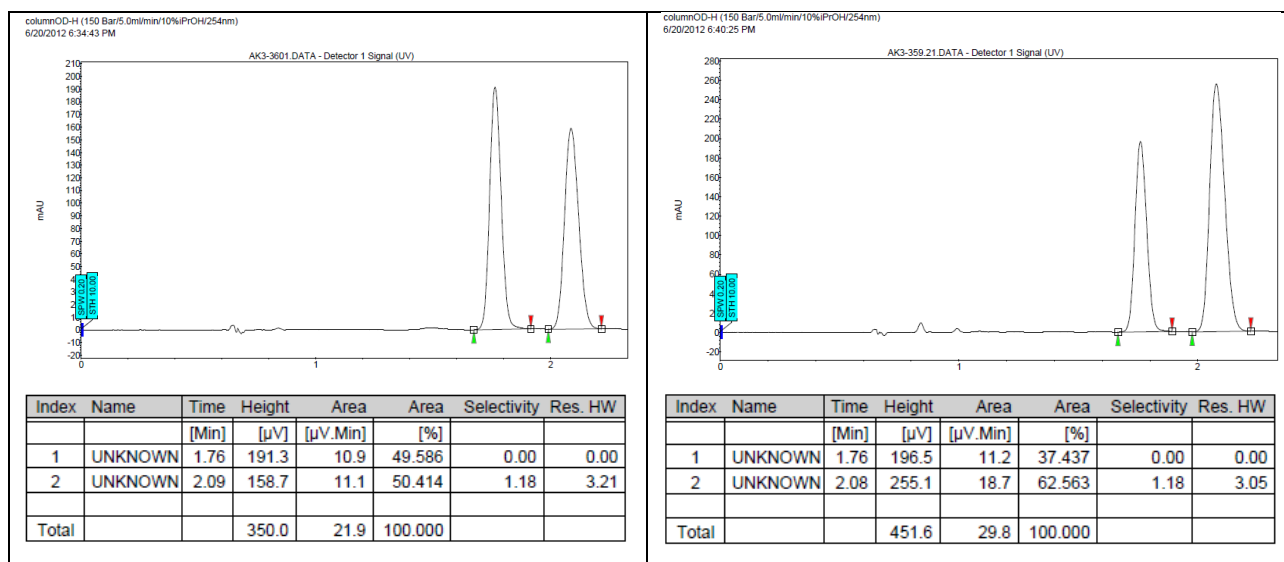
N-(2-Methyl-1-phenylallyl)acetamide (3p)



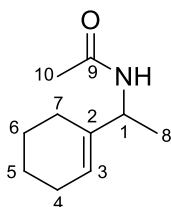
MW (g/mol): 189.25

Molecular formula: C₁₂H₁₅NO

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -56 (*c* 0.48, CHCl₃, 25% *ee*). **IR** (neat): 3265, 2934, 1630, 1543, 1370, 1286, 1104, 908, 757, 695 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.37–7.24 (m, 5H, H_{Ph}), 6.13 (s, 1H, NH), 5.47 (d, *J* = 8.3 Hz, 1H, H₁), 5.10–4.94 (m, 2H, H₃), 2.00 (s, 3H, H₁₀), 1.66 (s, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 169.2 (s, C₉), 144.1 (s, C₂), 140.0 (s, C₅), 128.8 (d, C_{Ph}), 127.8 (d, C_{Ph}), 127.5 (d, C_{Ph}), 111.6 (t, C₃), 58.5 (d, C₁), 23.3 (q, C₁₀), 20.5 (q, C₄). **HRMS (ESI)** *m/z*: calcd for C₁₂H₁₆NO [M + H]⁺: 190.1226, found: 190.1229. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 1.7 min and *t*_R = 2.0 min.



***N*-(1-(Cyclohex-1-en-1-yl)ethyl)acetamide (3q)¹⁵**

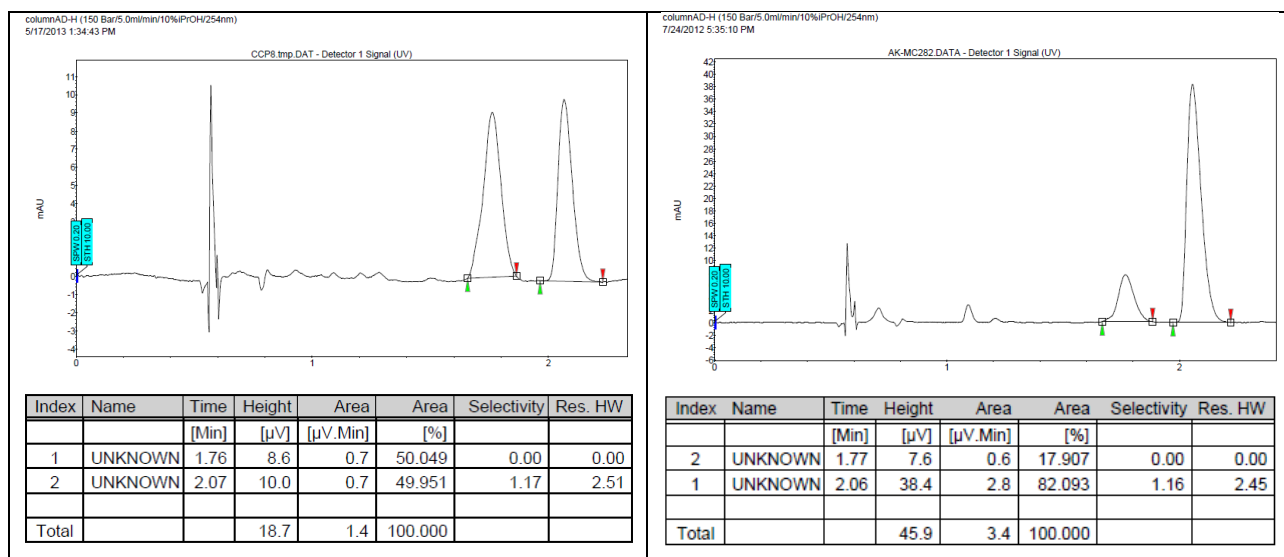


MW (g/mol): 167.25

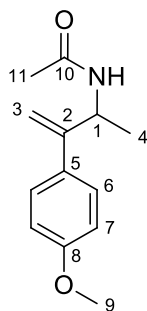
Molecular formula: C₁₀H₁₇NO

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -48 (*c* 1.0, CHCl₃, 64% *ee*). ¹H NMR (400 MHz, CDCl₃) δ 5.60 (s, 1H, H₃), 5.43 (brs, 1H, NH), 4.39 (m, 1H, H₁), 2.04-1.91 (m, 7H, H₄ + H₇ + H₁₀), 1.66-1.52 (m, 4H, H₅ + H₆), 1.21 (d, *J* = 6.9 Hz, 3H, H₈). ¹³C NMR (100 MHz, CDCl₃) δ 169.3 (s, C₉), 138.5 (s, C₂), 121.7 (d, C₃), 49.9 (d, C₁), 25.9 (t, C₄ or C₇), 25.1 (t, C₄ or C₇), 23.7 (q, C₁₀), 22.8 (t, C₅ or C₆), 22.5 (t, C₅ or C₆), 19.6 (q, C₈). **SFC:** AD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t*_R = 1.8 min and *t*_R = 2.0 min.

¹⁵ Hengge, A. C.; Cleland, W. W. *J. Org. Chem.*, **1991**, *56*, 1972.



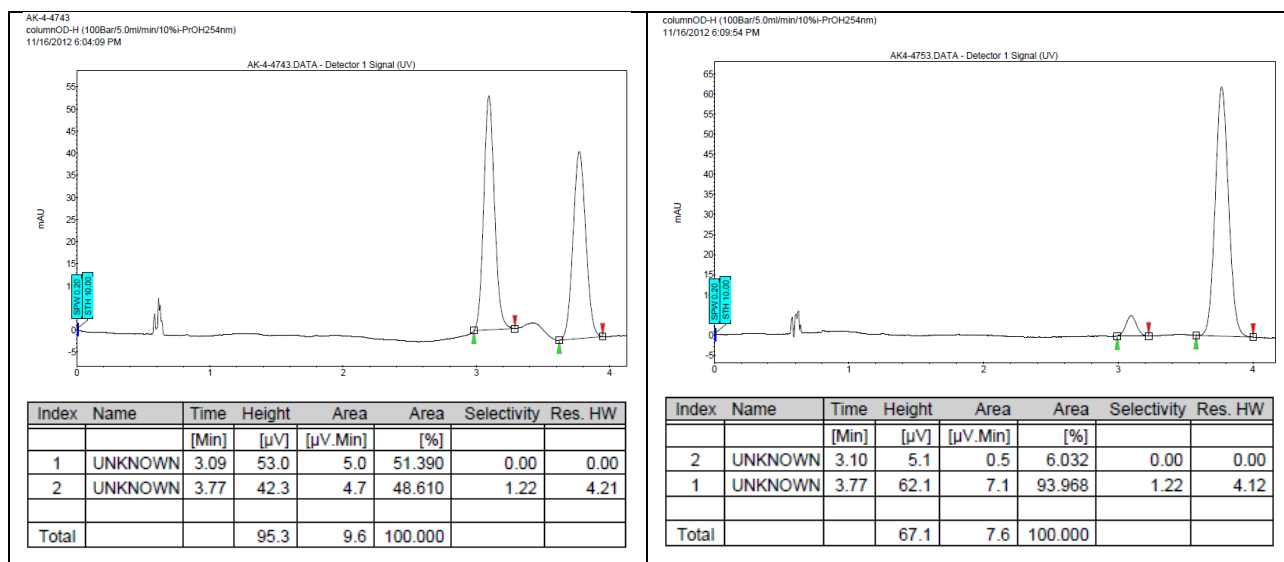
N-(3-(4-Methoxyphenyl)but-3-en-2-yl)acetamide (**3r**)



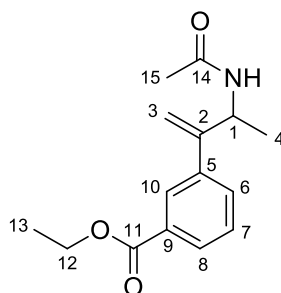
MW (g/mol): 219.28

Molecular formula: C₁₃H₁₇NO₂

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -37 (*c* 1.1, CHCl₃, 88% *ee*). **IR** (neat): 3310, 2977, 2933, 1631, 1518, 1369, 1282, 1252, 1188, 1023, 904, 835, 713, 606 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.35 (d, *J* = 8.9 Hz, 2H, H₆), 6.87 (d, *J* = 8.9 Hz, 2H, H₇), 5.45 (d br, *J* = 6.3 Hz, 1H, NH), 5.29 (s, 1H, H₃), 5.16 (s, 1H, H₃), 5.09 (m, 1H, H₁), 3.82 (s, 3H, H₉), 1.95 (s, 3H, H₁₁), 1.33 (d, *J* = 6.7 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 169.3 (s, C₁₀), 159.4 (s, C₈), 149.8 (s, C₂), 132.3 (s, C₅), 127.8 (d, C₆), 113.9 (d, C₇), 111.0 (t, C₃), 55.4 (q, C₉), 47.4 (d, C₁), 23.6 (q, C₁₁), 20.4 (q, C₄). **HRMS (ESI)** *m/z*: calcd for C₁₃H₁₇NaNO₂ [M + Na]⁺: 242.1151, found: 242.1145. **SFC**: OD-H, Pressure = 100 bar, CO₂/MeOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t_R* = 3.1 min and *t_R* = 3.7 min.



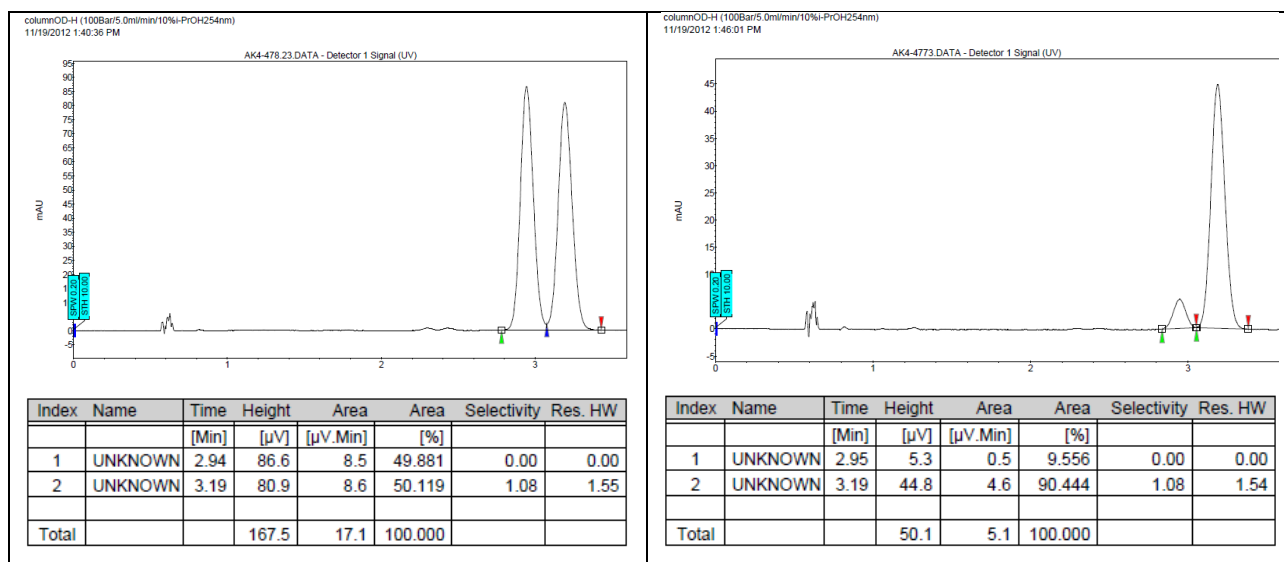
Ethyl 3-(3-acetamidobut-1-en-2-yl)benzoate (3s)



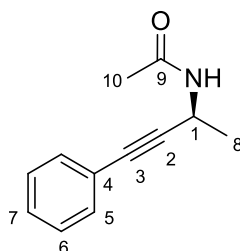
MW (g/mol): 261.32

Molecular formula: C₁₅H₁₉NO₃

This compound was prepared following general procedure D. $[\alpha]_D^{20}$ -18 (*c* 1.1, CHCl₃, 81% *ee*). **IR** (neat): 3276, 2979, 1718, 1650, 1543, 1369, 1293, 11244, 1166, 1022, 908, 763 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 8.06 (t, *J* = 1.6 Hz, 1H, H_{Ar}), 7.96 (m, 1H, H_{Ar}), 7.59 (ddd, *J* = 7.8, 1.9, 1.2 Hz, 1H, H_{Ar}), 7.40 (t, *J* = 7.8 Hz, 1H, H_{Ar}), 5.66 (d br, *J* = 7.5 Hz, 1H, NH), 5.36 (s, 1H, H₃), 5.28 (s, 1H, H₃), 5.10 (quint_{app}, *J* = 7.0 Hz, 1H, H₁), 4.38 (q, *J* = 7.1 Hz, 2H, H₁₂), 1.95 (s, 3H, H₁₅), 1.40 (t, *J* = 7.1 Hz, 3H, H₁₃), 1.31 (d, *J* = 6.8 Hz, 3H, H₄). **¹³C NMR** (100 MHz, CDCl₃) δ 169.3 (s, C₁₄), 166.6 (s, C₁₁), 149.9 (s, C₂), 140.5 (s, C₅), 131.1 (d, C_{Ar}), 130.8 (s, C₉), 128.9 (d, C_{Ar}), 128.6 (d, C_{Ar}), 128.0 (d, C_{Ar}), 113.2 (t, C₃), 61.2 (t, C₁₂), 47.6 (d, C₁), 23.4 (q, C₁₅), 20.4 (q, C₄), 14.4 (q, C₁₃). **HRMS (ESI)** *m/z*: calcd for C₁₅H₁₉NaNO₃ [M + Na]⁺: 284.1257, found: 284.1257. **SFC**: OD-H, Pressure = 100 bar, CO₂/MeOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, *t_R* = 2.9 min and *t_R* = 3.2 min.



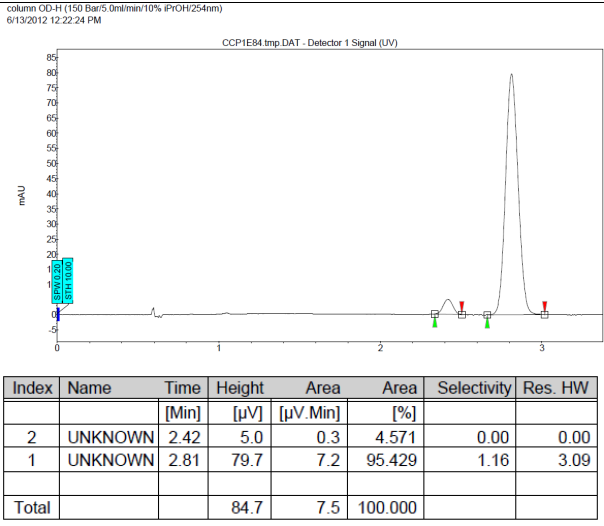
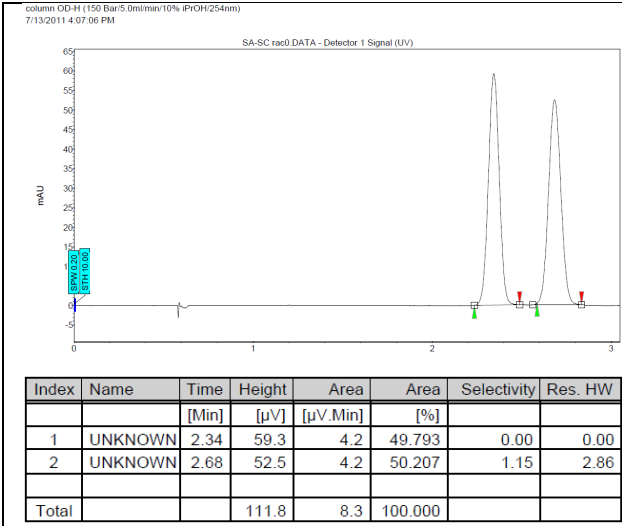
N-(4-Phenylbut-3-yn-2-yl)acetamide (**3u**)

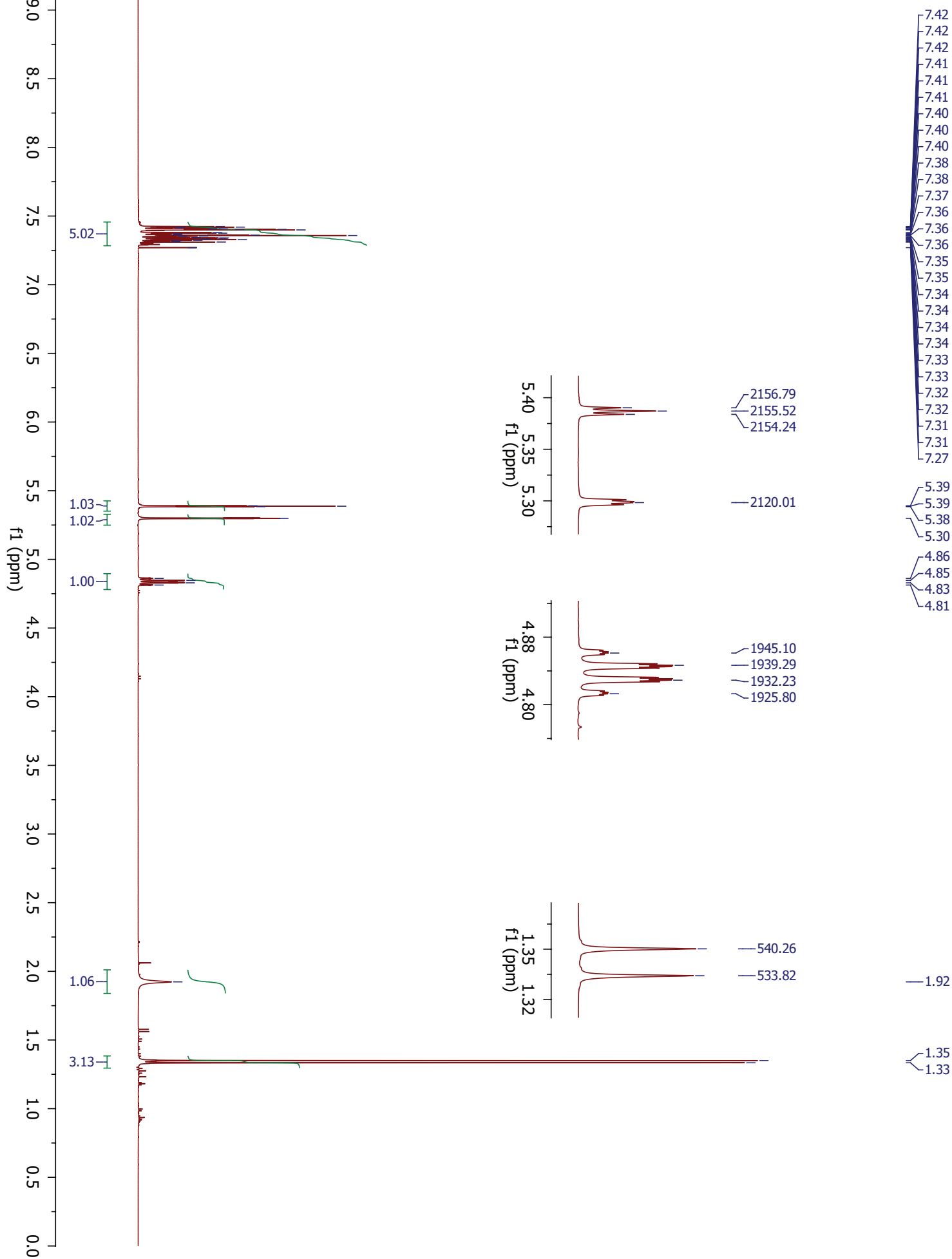
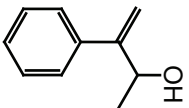


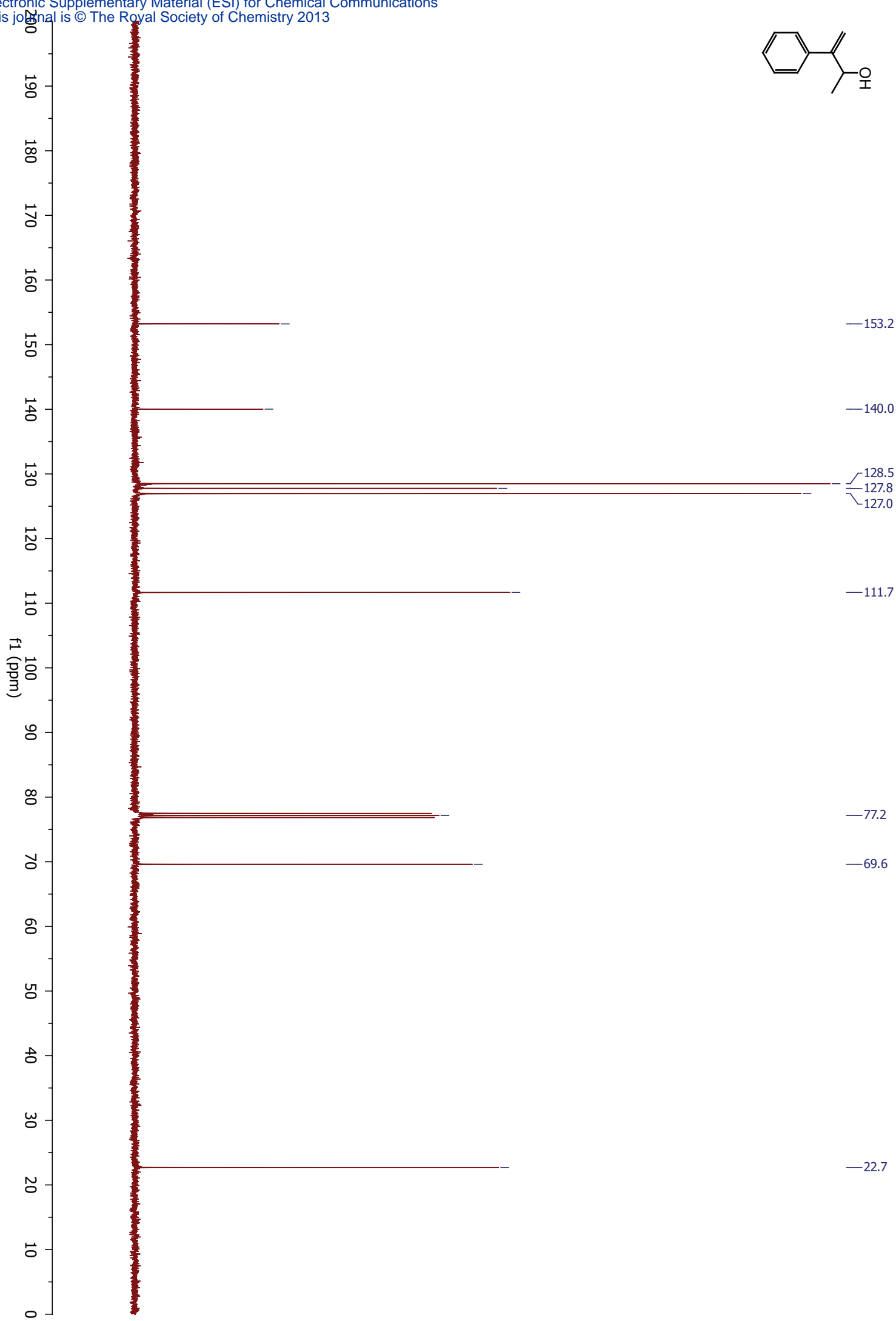
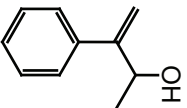
MW (g/mol): 187.24

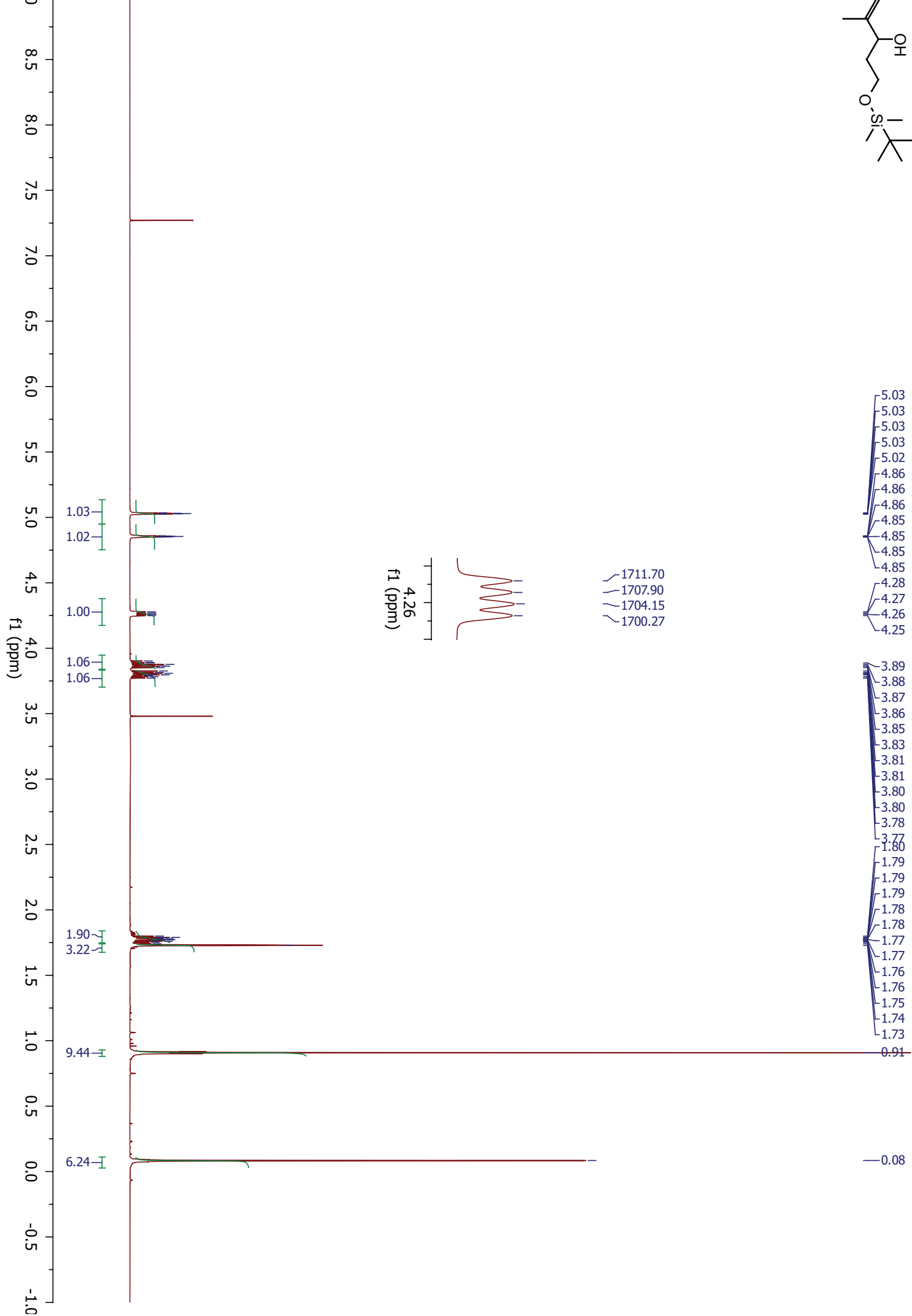
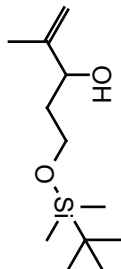
Molecular formula: C₁₂H₁₃NO

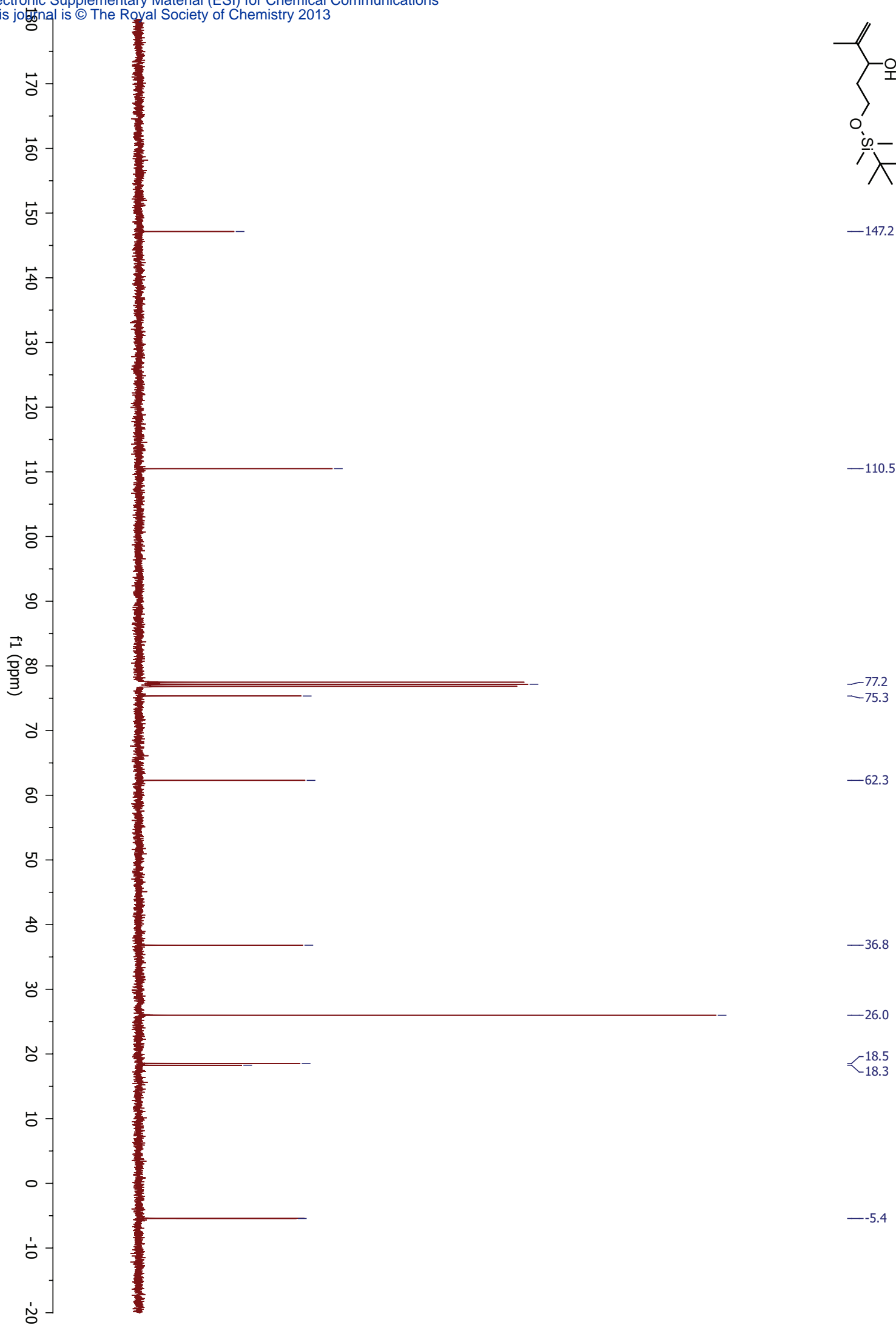
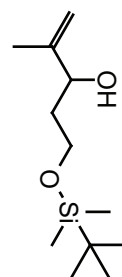
This compound was prepared following general procedure D. **IR** (neat): 3290, 3057, 2981, 2932, 1643, 1542, 1372, 1134, 757, 691 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.46–7.38 (m, 2H, H_{Ph}), 7.35–7.28 (m, 3H, H_{Ph}), 5.84 (s, 1H, NH), 5.05 (dq, *J* = 13.8, 6.9 Hz, 1H, H₁), 2.02 (s, 3H, H₁₀), 1.49 (d, *J* = 6.8 Hz, 3H, H₈). **¹³C NMR** (100 MHz, CDCl₃) δ 169.15 (s, C₉), 131.76 (d, C_{Ph}), 128.4 (d, C_{Ph}), 128.3 (d, C_{Ph}), 122.7 (s, C₄), 89.5 (s, C₂), 82.2 (s, C₃), 37.7 (d, C₁), 23.3 (q, C₈ or C₁₀), 22.6 (q, C₈ or C₁₀). [**α**]_D²⁰ −163 (*c* 0.6, CHCl₃, 91% *ee*). **HRMS (ESI)** *m/z*: calculated for C₁₂H₁₃NaNO [M + Na]⁺: 210.0889, found: 210.0887. **SFC**: OD-H, Pressure = 150 bar, CO₂/*i*-PrOH = 90/10, Flow rate = 5 mL/min, UV = 254 nm, t_R = 2.4 min and t_R = 2.8 min (major).

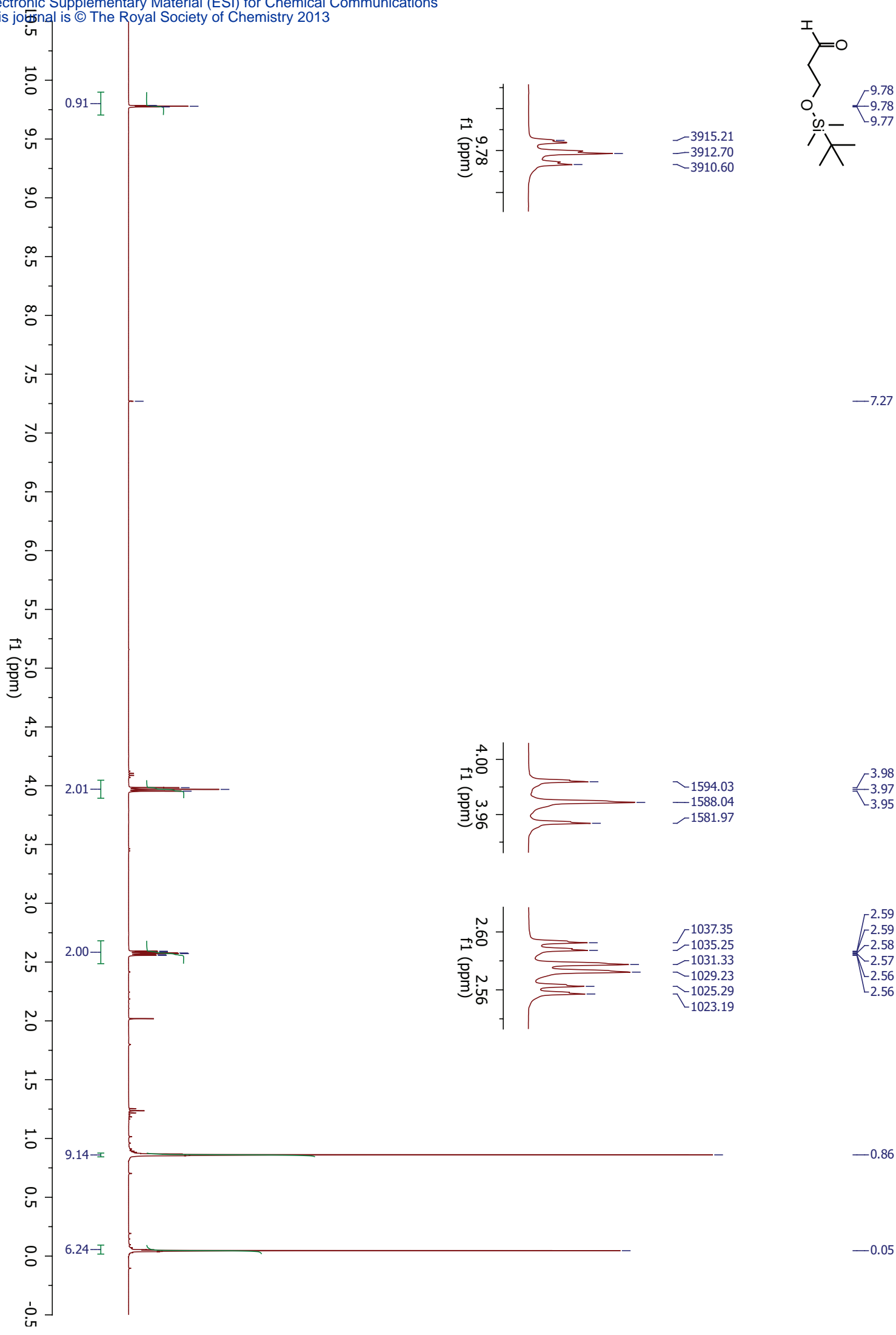


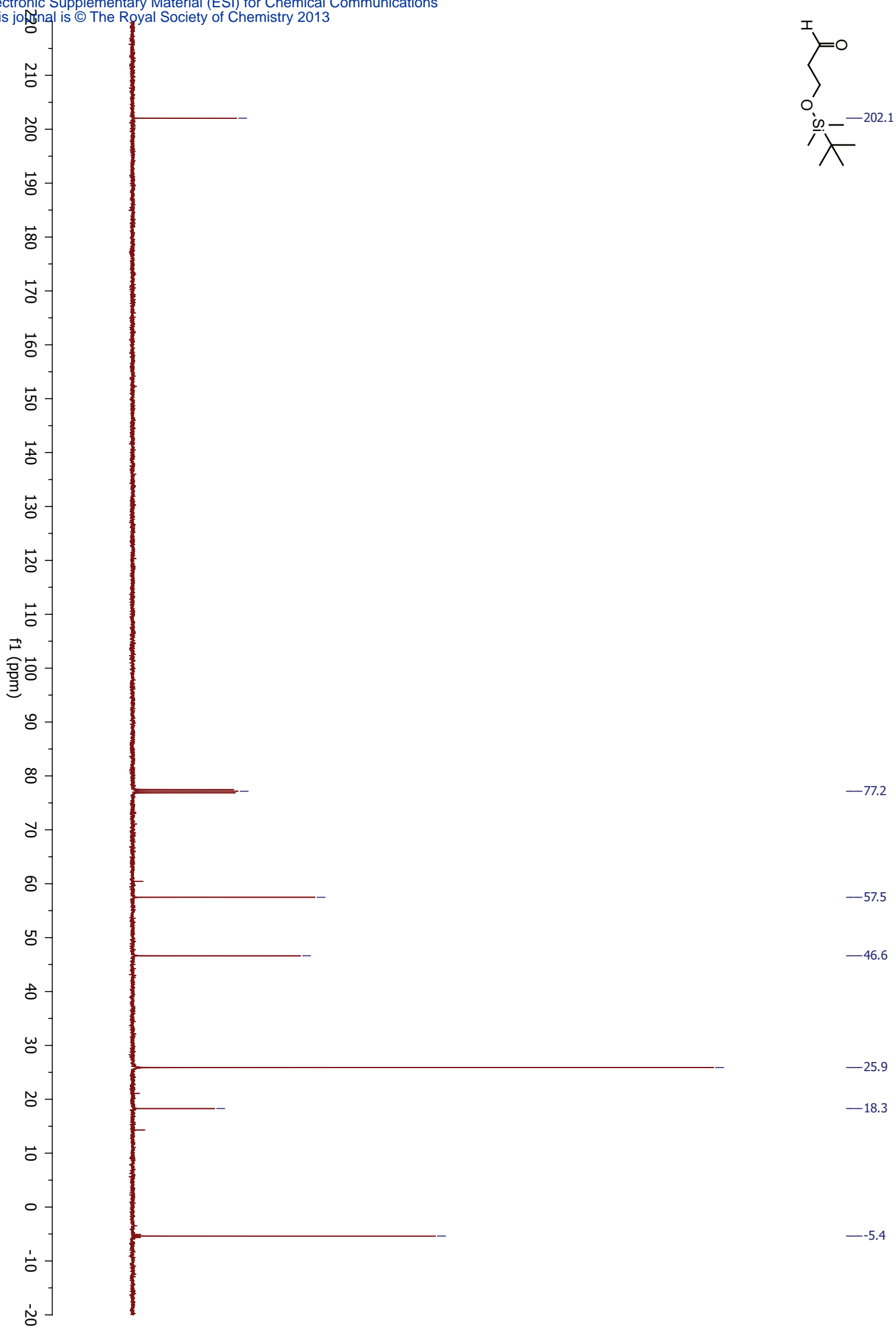


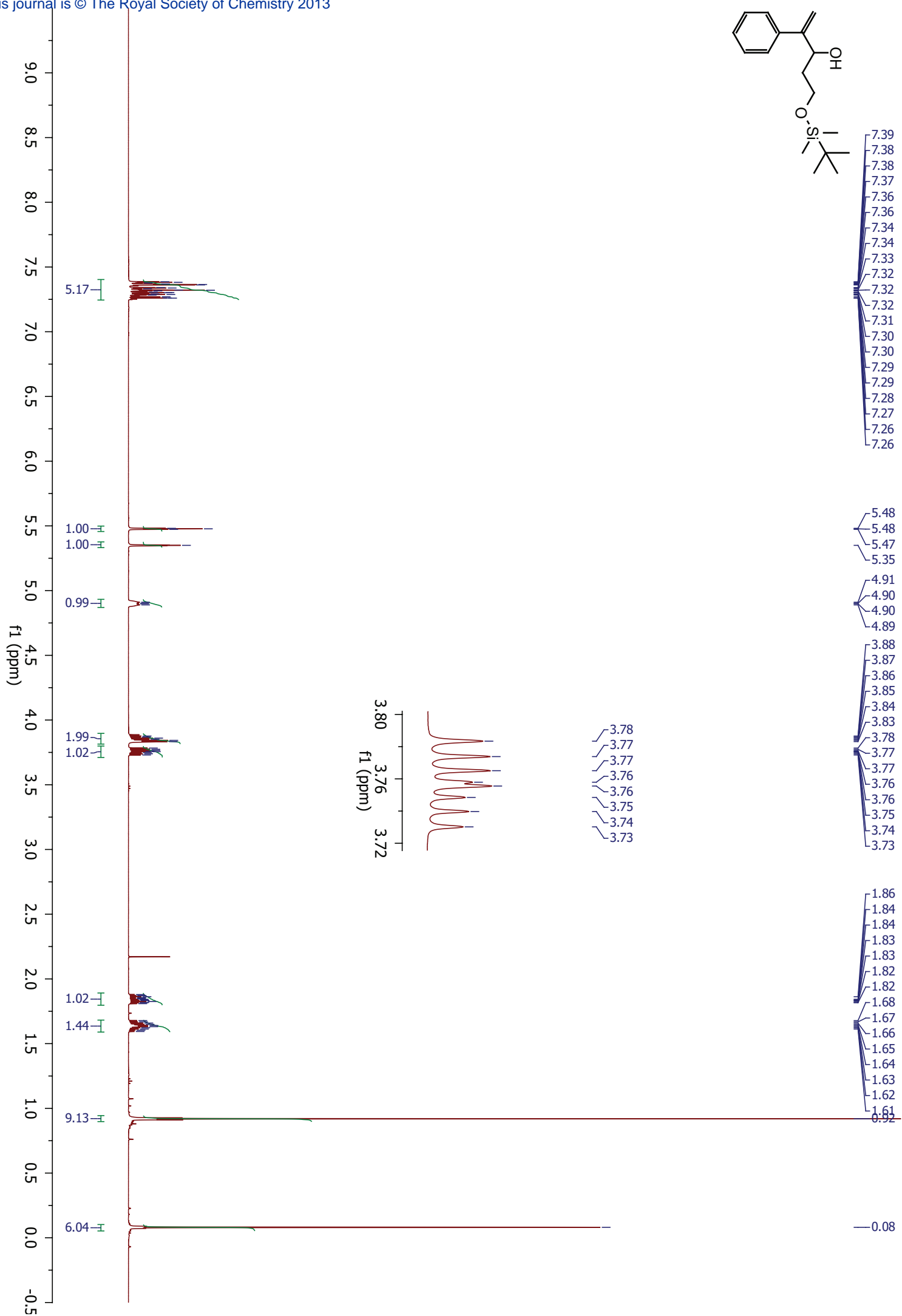


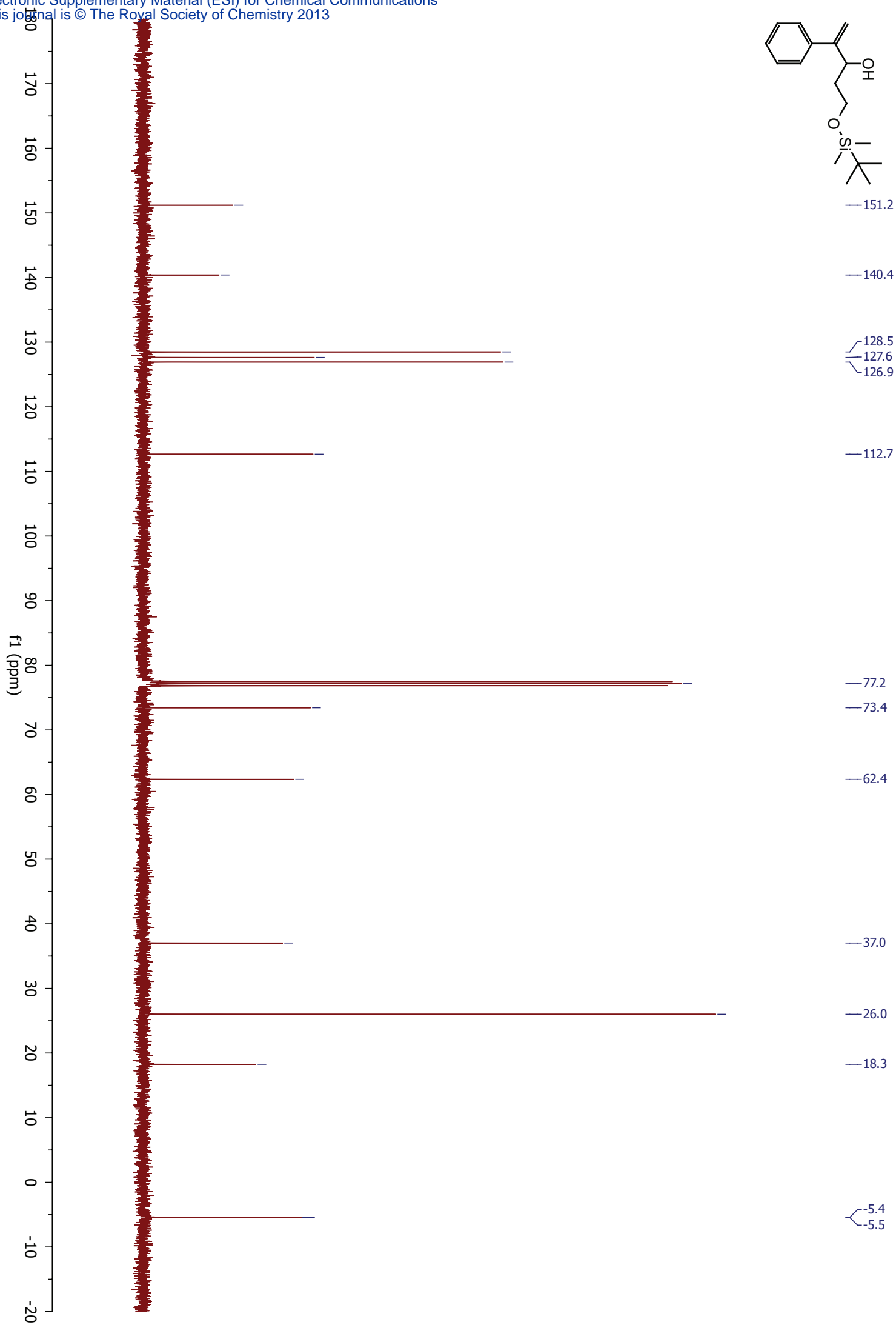


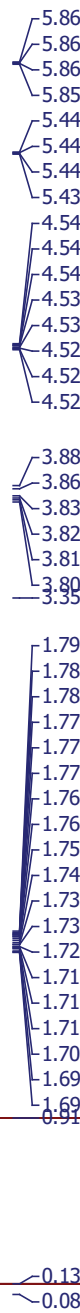


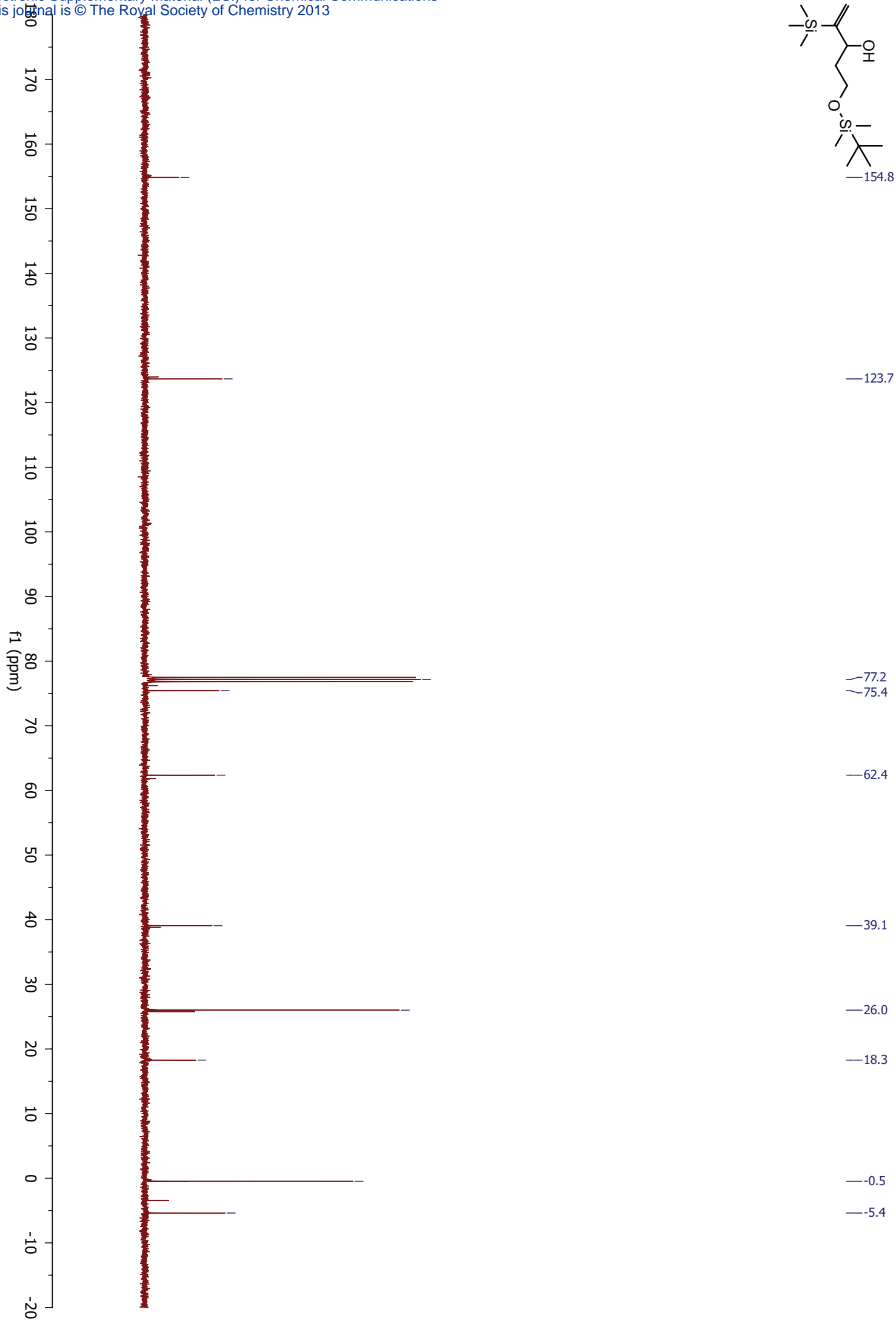
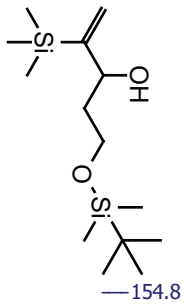


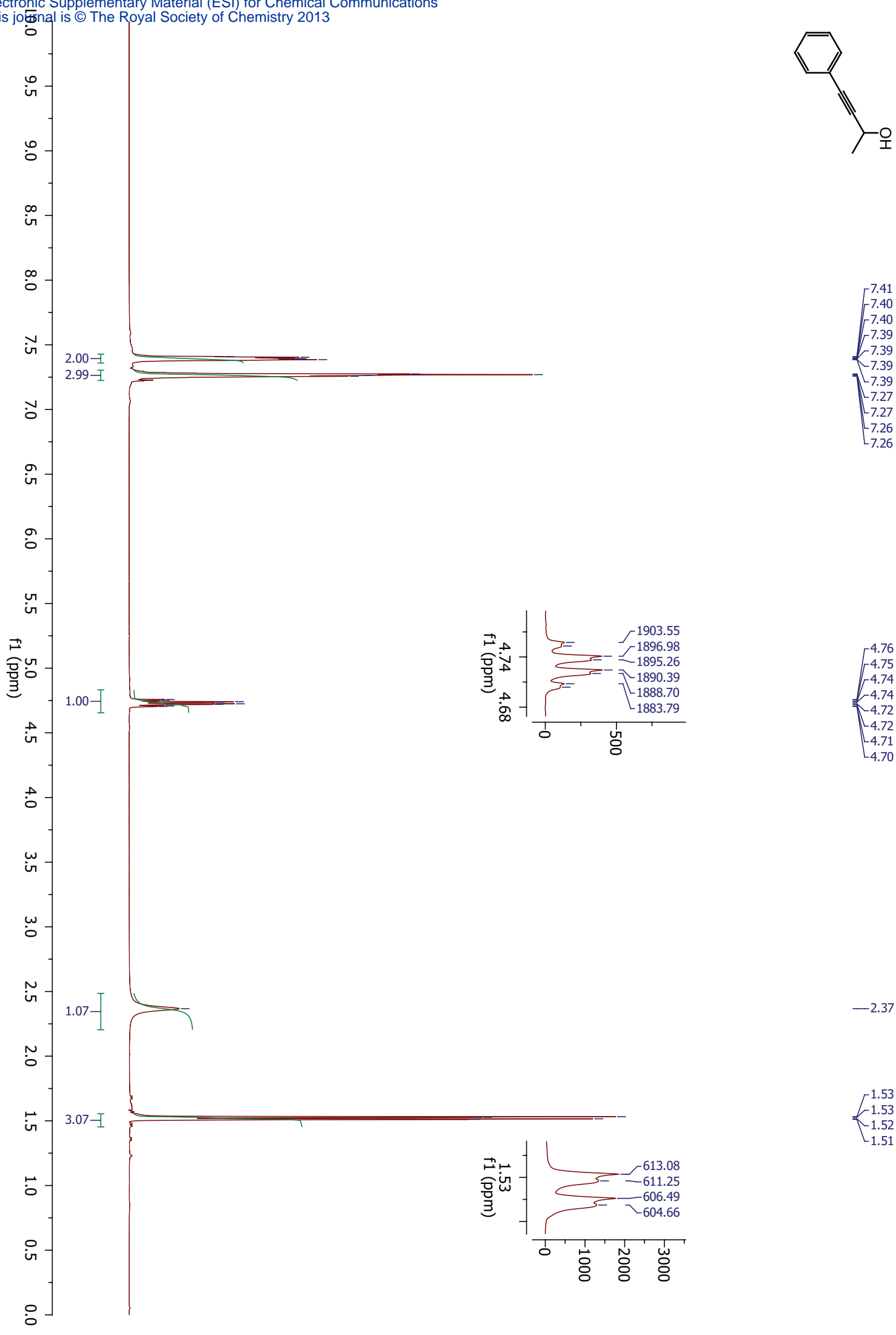
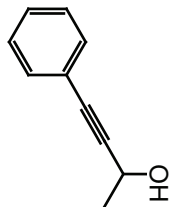


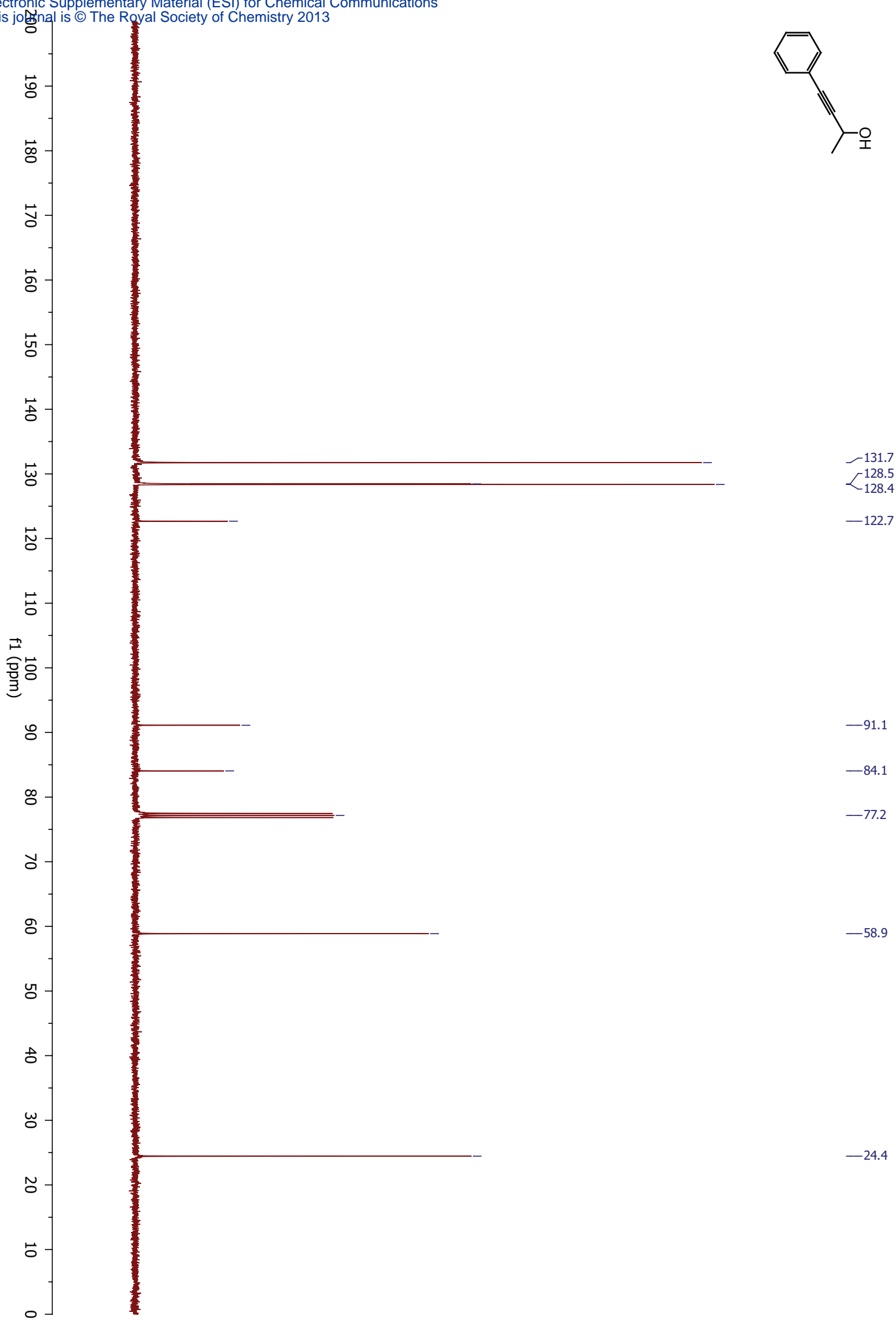
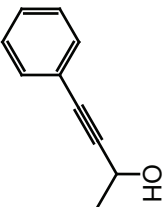


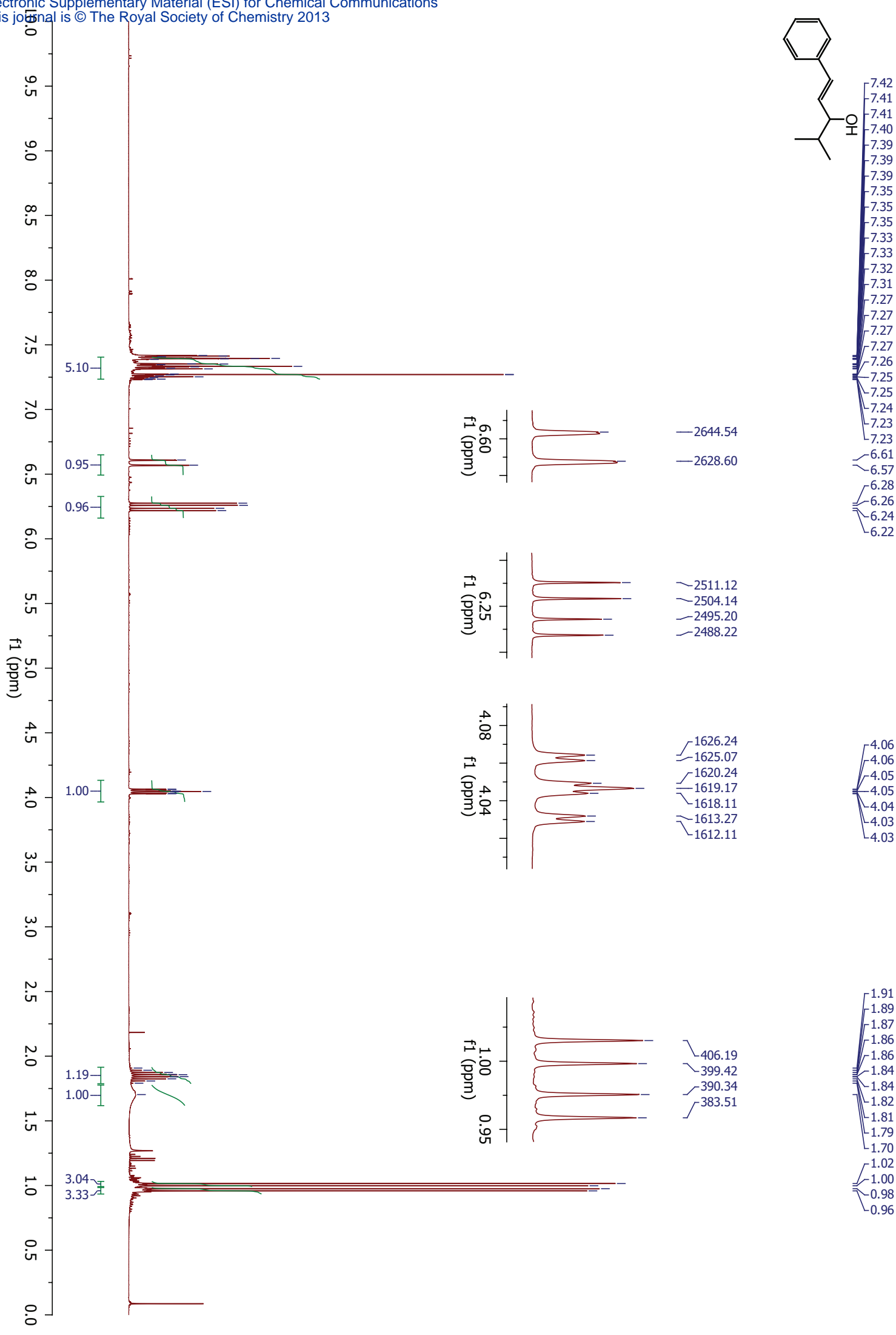


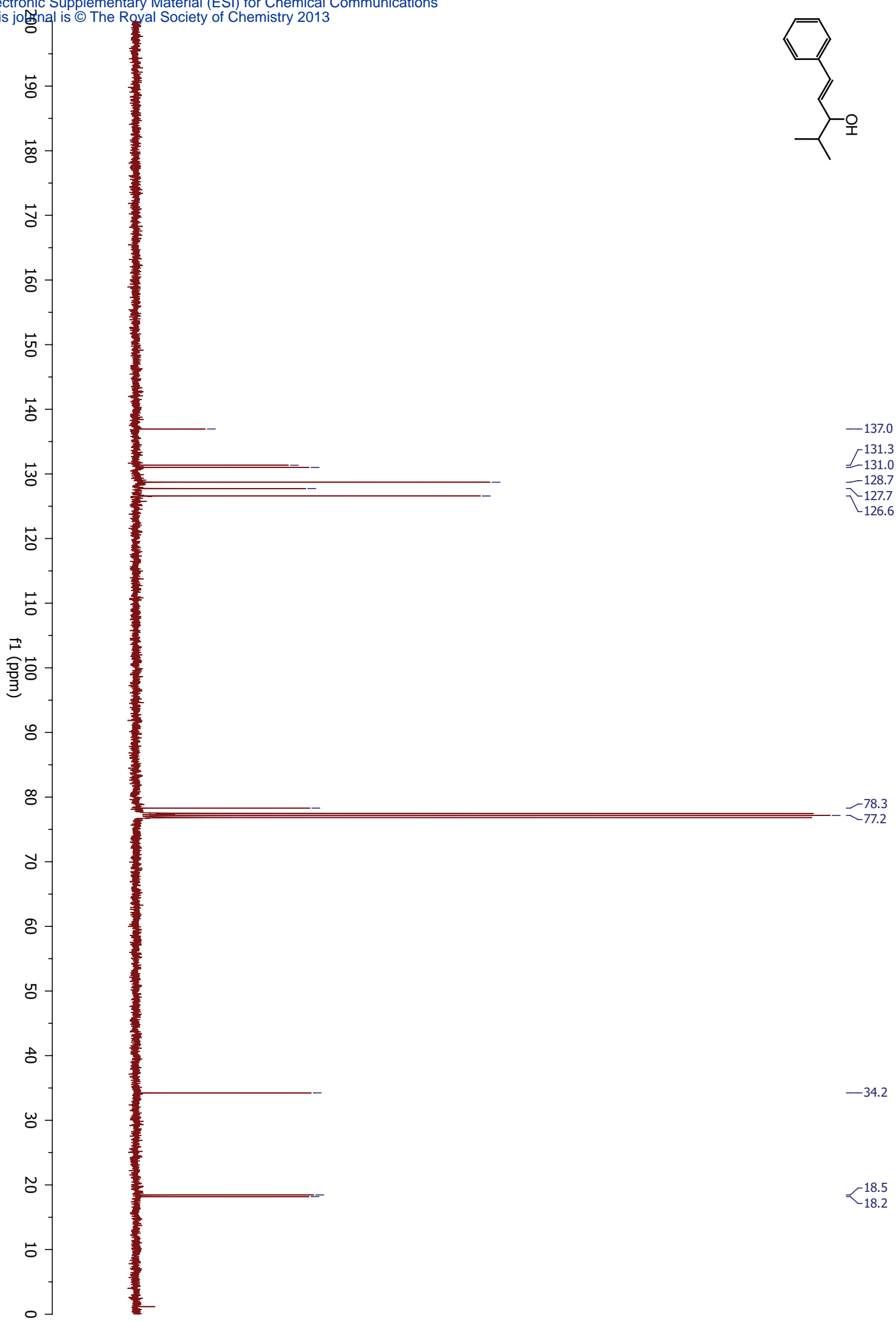
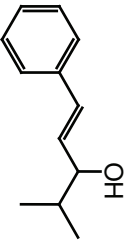


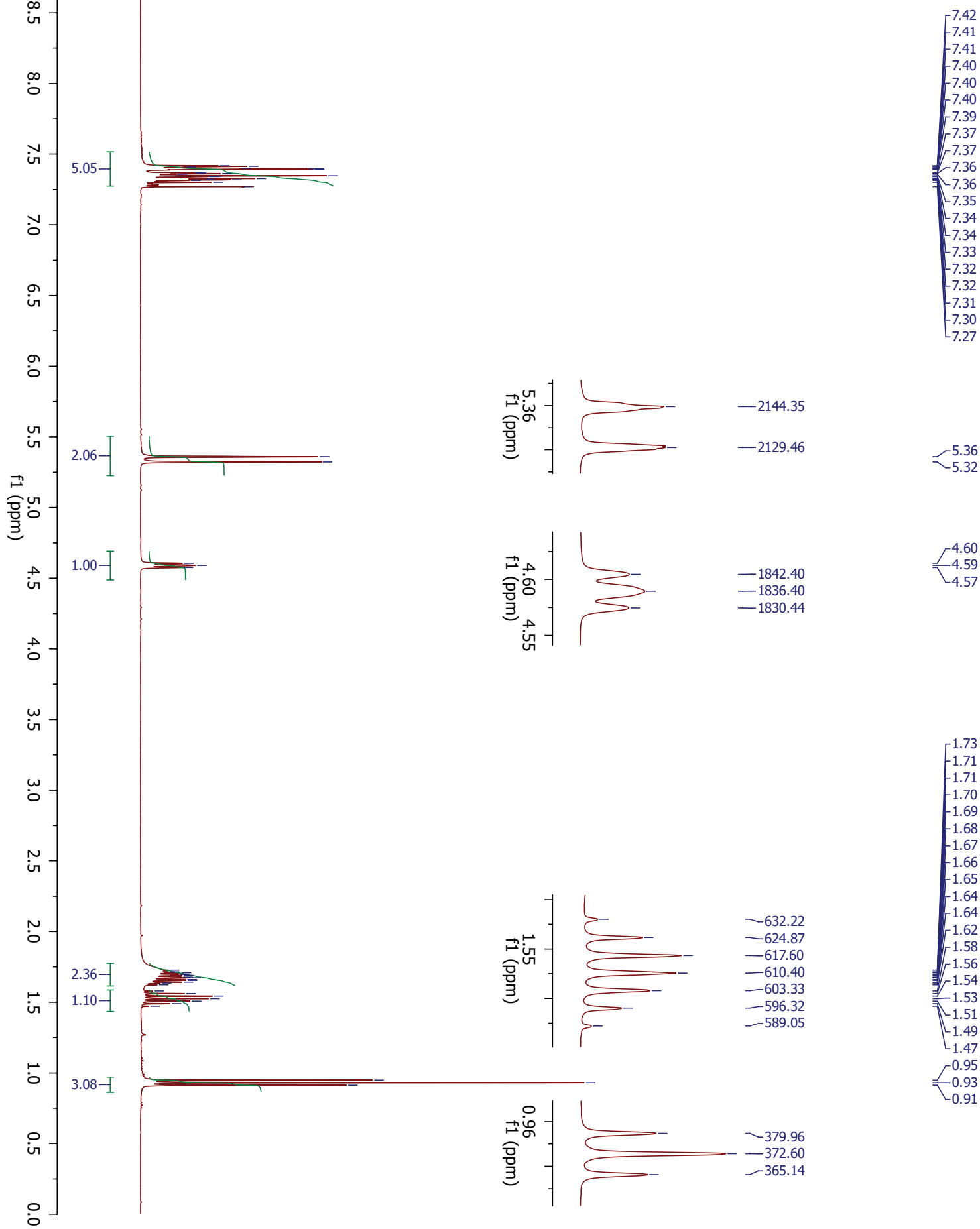
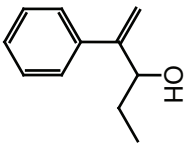


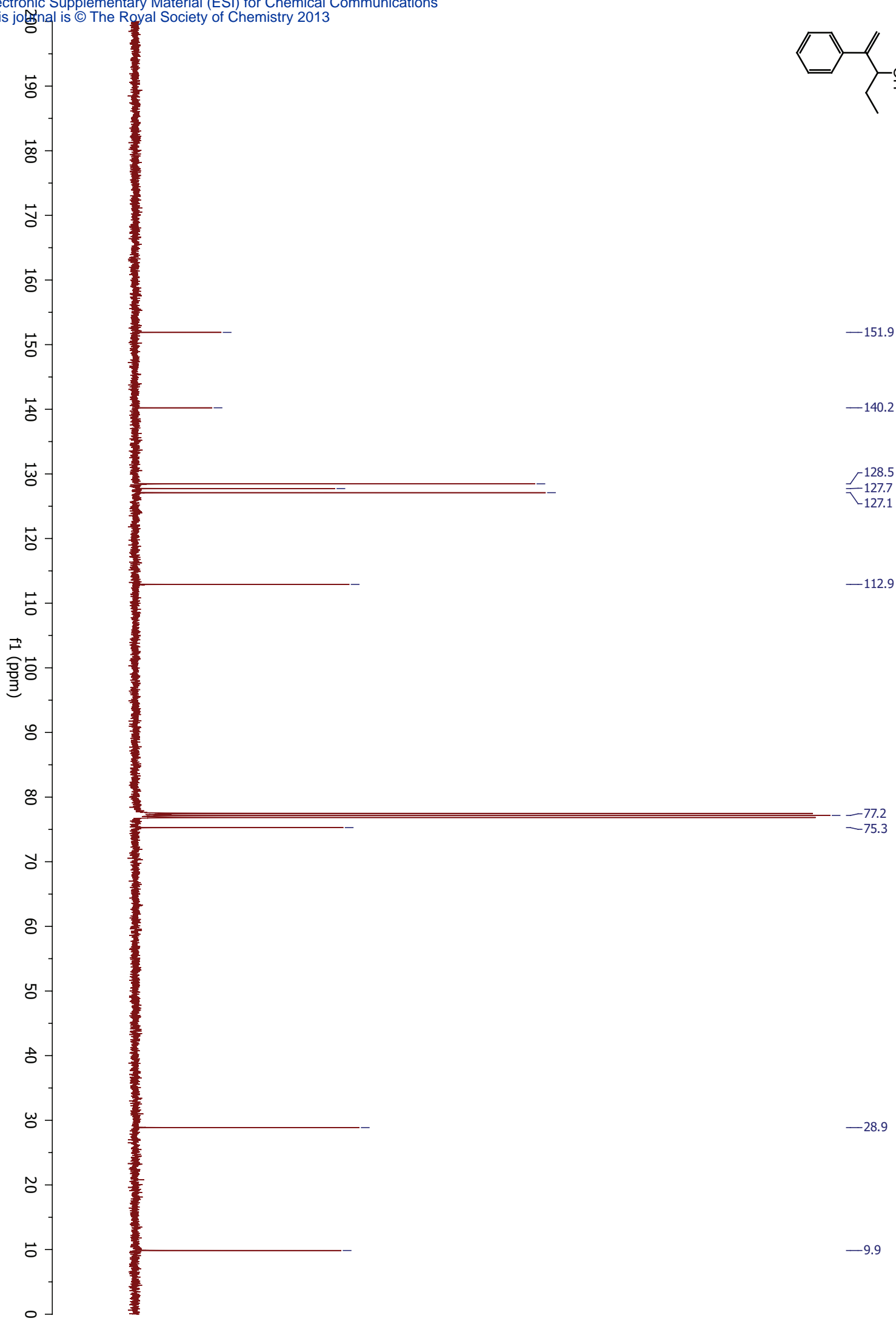
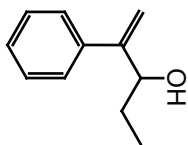


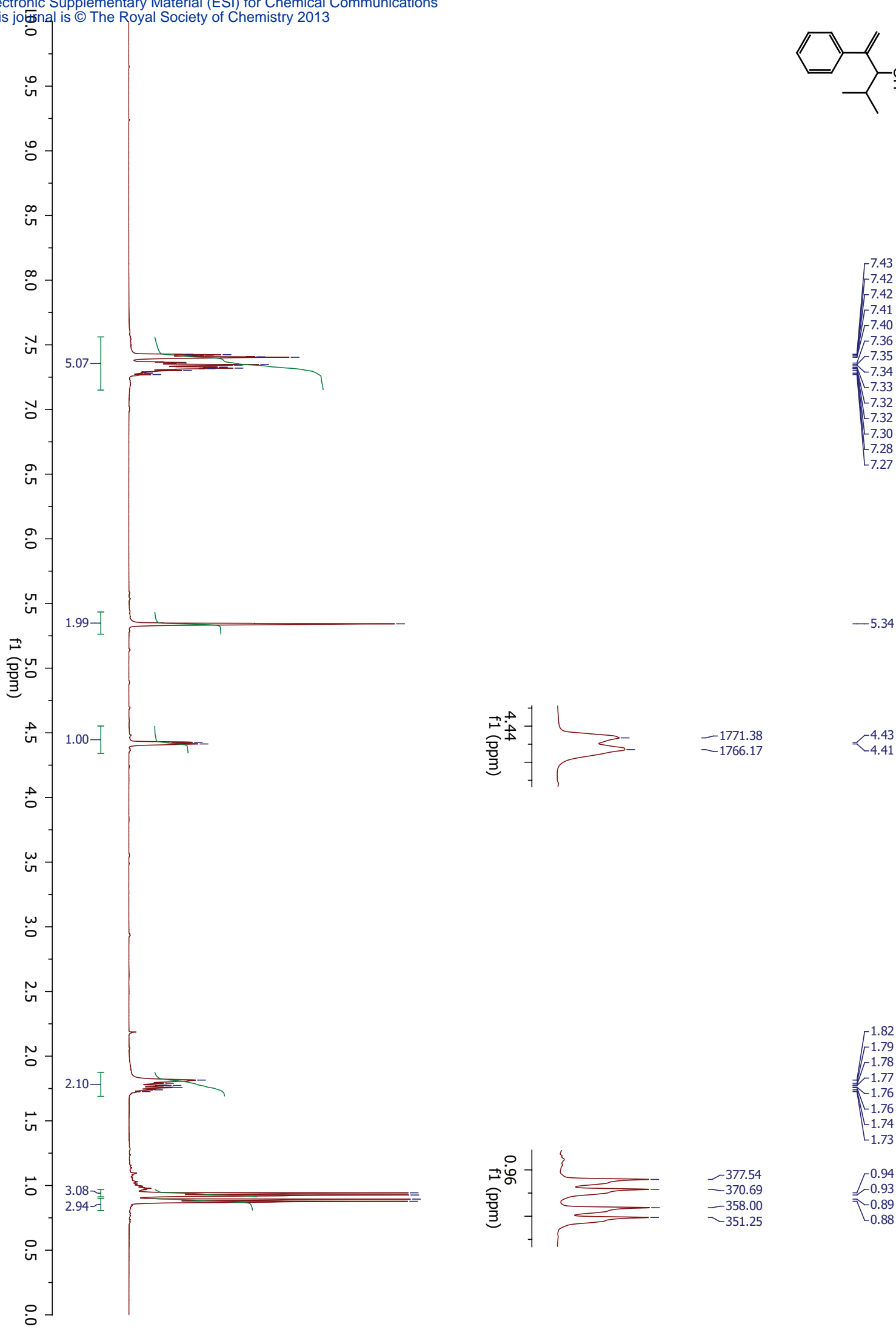
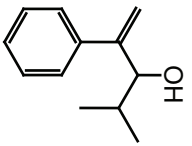


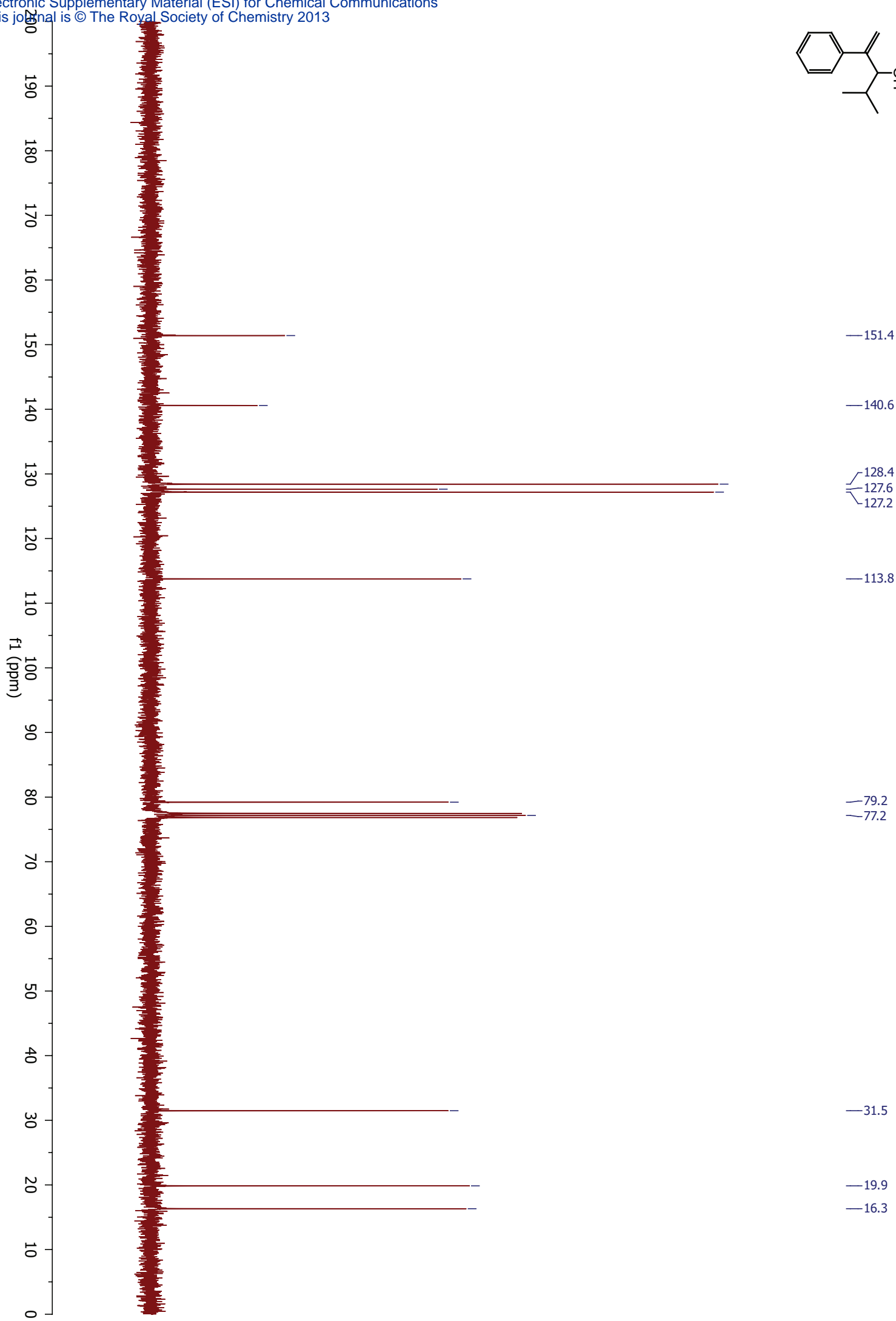
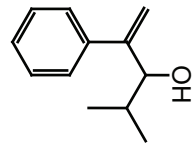


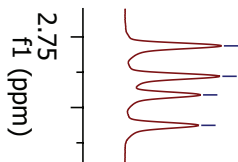
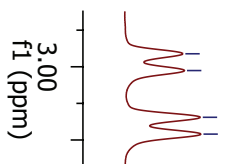
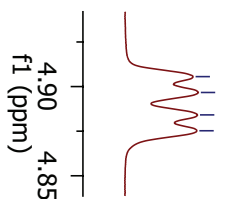
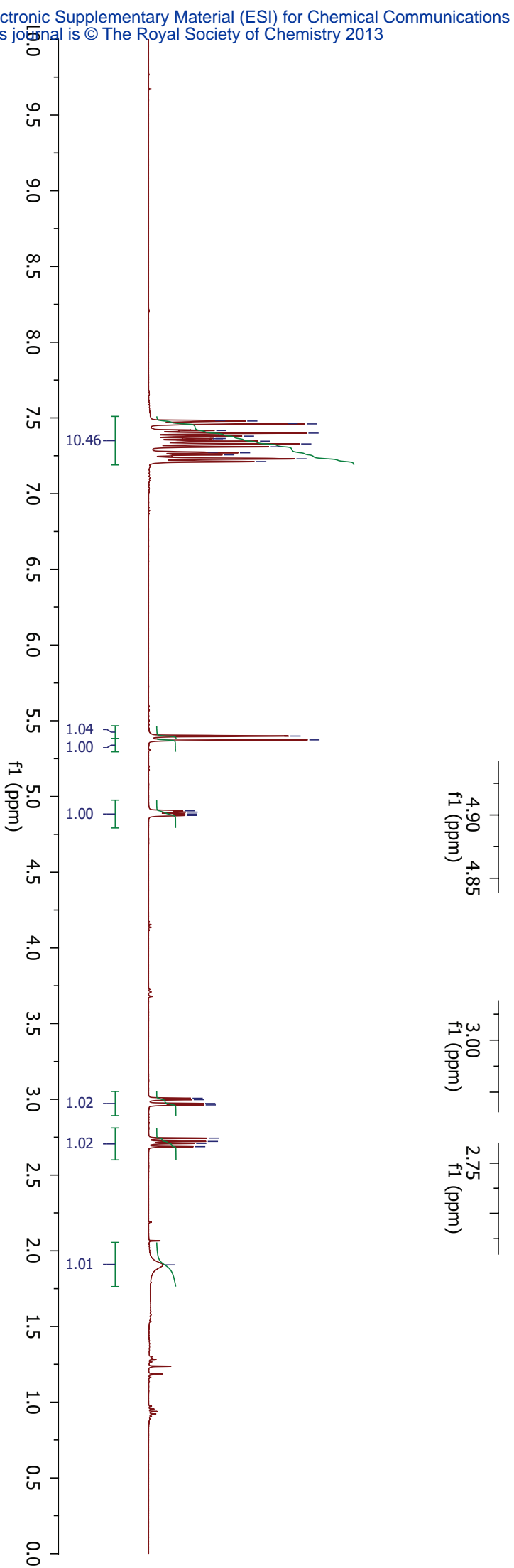
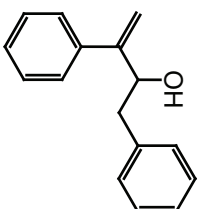












1962.87
1959.31
1954.29
1950.73

1203.23
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1185.67

1097.83
1089.20
1083.93
1075.30

