Supporting Information

Palladium-Catalyzed Heteroallylation of Unactivated Alkenes – Synthesis of Citalopram

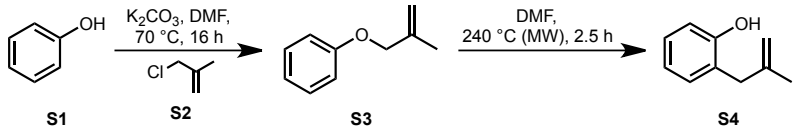
Joanne F. M. Hewitt, Lewis Williams, Pooja Aggarwal, Craig D. Smith, David J. France

WestCHEM, School of Chemistry, University of Glasgow, Glasgow, G12 8QQ, UK.

1. General Methods

Reactions involving air-sensitive agents and dry solvents were performed in glassware that had been dried in an oven (150 °C) or flame-dried prior to use. These reactions were carried out with the exclusion of air using an argon atmosphere. All microwave reactions were carried out using a Biotage Initiator system. NMR spectra were recorded on a Bruker DPX-400 spectrometer (^{1}H NMR at 400 MHz and ^{13}C NMR at 100 MHz) or a Bruker DPX-500 spectrometer (^{1}H NMR at 500 MHz and ^{13}C NMR at 125 MHz). Chemical shifts are reported in ppm. ^{1}H NMR spectra were recorded with $CDCl_3$ as the solvent using residual $CHCl_3$ (δ = 7.26) as internal standard, and for ^{13}C NMR spectra the chemical shifts are reported relative to the central resonance of $CDCl_3$ (δ = 77.16). Signals in NMR spectra are described as singlet (s), doublet (d), triplet (t), quartet (q), quintet (quint), septet (sept), multiplet (m), broad (br) or combination of these, which refers to the spin–spin coupling pattern observed. Spin–spin coupling constants reported are uncorrected. Two-dimensional (COSY, HSQC, HMBC, NOESY) NMR spectroscopy was used where appropriate to assist the assignment of signals in the ^{1}H and ^{13}C NMR spectra. IR spectra were obtained employing a Shimadzu FTIR-8400 instrument with a Golden Gate™ attachment that uses a type IIa diamond as a single reflection element so that the IR spectrum of the compound (solid or liquid) could be detected directly (thin layer). High resolution mass spectra were recorded under FAB, ESI and CI conditions by the analytical services at the University of Glasgow. Flash column chromatography was performed using forced flow of the indicated solvent system on EMD Geduran® Silica Gel 60 as solid support and HPLC graded solvents as eluant. Reactions were monitored by thin layer chromatography (TLC) on Merck silica gel 60 covered aluminum sheets. TLC plates were developed under UV-light and/or with an acidic ethanolic anisaldehyde solution or a $KMnO_4$-solution. Liquid reagents were distilled prior to use where stated. All reagents were purchased from commercial suppliers and used without further purification unless otherwise stated.

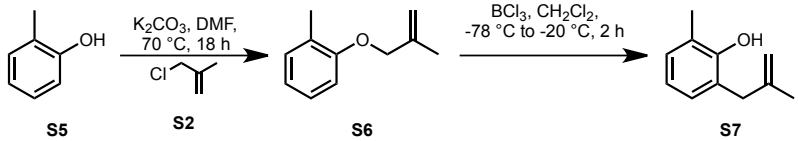
2. Preparation of Substrates



S1 S2 S3 S4

2-(2-methylallyl)phenol S4.[1] To a stirred suspension of K_2CO_3 (8.8 g, 64 mmol) in DMF (160 mL) was added phenol (**S1**) (3.0 g, 32 mmol) followed by 3-chloro-2-methyl-1-propene (**S2)** (3.7 mL, 38 mmol). The resulting mixture was heated at 70 °C for 16 h then cooled to room temperature, quenched with water (250 mL) and extracted with Et_2O (2 x 100 mL). The combined organic extracts were washed with brine (2 x 100 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford 1-(2-methylallyloxy)benzene (**S3**) as a colourless oil (4.7 g, quant.) which was used without any further purification. Analytical data observed were in accordance with literature values.[2]

A solution of 1-(2-methylallyloxy)benzene (**S3**) (1.5 g, 10 mmol) in DMF (8.4 mL) under argon was subjected to microwave irradiation at 240 °C for 2.5 h. The resulting mixture was extracted with Et_2O (3 x 50 mL) and the combined organic extracts washed with water (50 mL) then brine (2 x 50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 19:1) afforded the title compound (**S4**) as a yellow oil (1.2 g, 80%). Analytical data observed were in accordance with literature values.[1]

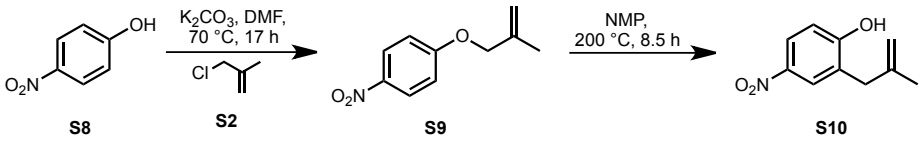


S5 S2 S6 S7

2-methyl-6-(2-methylallyl)phenol S7. To a stirred suspension of K_2CO_3 (5.1 g, 37 mmol) in DMF (93 mL) was added *o*-cresol (**S5**) (2.0 g, 19 mmol) followed by 3-chloro-2-methyl-1-propene (**S2**) (2.2 mL, 22 mmol). The resulting mixture was heated at 70 °C for 18 h then cooled to room temperature, quenched with water (150 mL) and extracted with Et_2O (3 x 75 mL). The combined organic extracts were washed with brine (3 x 50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford 1-(2-methylallyloxy)-2-methylbenzene (**S6**) as a colourless oil (2.9 g, 97%) which was used without any further purification. Analytical data observed were in accordance with

literature values.[3]

To a cooled (−78 °C) solution of 1-(2-methylallyloxy)-2-methylbenzene (**S6**) (1.0 g, 6.2 mmol) in CH_2Cl_2 (31 mL) under argon was added dropwise boron trichloride (1M in hexanes, 6.5 mL, 6.5 mmol). The solution was allowed to warm to −20 °C over 2 h then quenched with H_2O (5 mL) and allowed to warm to room temperature. The resulting mixture was basified with sat. aq. $NaHCO_3$ (25 mL) and extracted with CH_2Cl_2 (3 x 50 mL). The combined organic extracts were dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 39:1) afforded the title compound (**S7**) as a yellow oil (600 mg, 60%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.03 (1H, d, *J* = 7.5 Hz), 6.94 (1H, d, *J* = 7.2 Hz), 6.78 (1H, t, *J* = 7.5 Hz), 5.1 (1H, s, OH), 4.94 (1H, s), 4.89 (1H, s), 3.39 (2H, s), 2.25 (3H, s), 1.75 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.3, 145.0, 129.6, 128.8, 124.8, 124.4, 120.4, 112.5, 40.5, 22.1, 15.9; IR (thin film) 3499, 1470, 1196; HRMS (CI) exact mass calculated for $C_{11}H_{15}O$ $[M+H]^+$ *m/z* 163.1123, found *m/z* 163.1123.

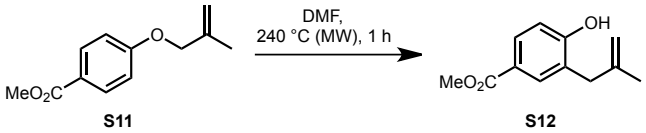


S8 **S2** **S9** **S10**

2-(2-methylallyl)-4-nitrophenol S10.[1] To a stirred suspension of K_2CO_3 (1.0 g, 7.2 mmol) in DMF (18 mL) was added 4-nitrophenol (**S8**) (0.50 g, 3.6 mmol) followed by 3-chloro-2-methyl-1-propene (**S2**) (0.42 mL, 4.3 mmol). The resulting mixture was heated at 70 °C for 17 h then cooled to room temperature, quenched with water (50 mL) and extracted with Et_2O (3 x 50 mL). The combined organic extracts were washed with brine (3 x 50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford 1-(2-methylallyloxy)-4-nitrobenzene (**S9**) as a colourless oil (650 mg, 94%) which was used without any further purification. Analytical data observed were in accordance with literature values.[1,4]

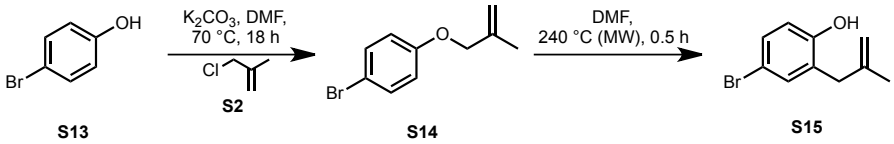
A solution of 1-(2-methylallyloxy)-4-nitrobenzene (**S9**) (650 mg, 3.4 mmol) in *N*-methylpyrrolidinone (1 mL) under argon was heated at 200 °C for 8.5 h. The mixture was diluted with brine (25 mL) and extracted with Et_2O (3 x 25 mL). The combined organic extracts were washed with brine (3 x 25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography

(petroleum ether:EtOAc, 9:1) afforded the title compound (**S10**) as a sticky brown solid (410 mg, 64%). Analytical data observed were in accordance with literature values.[1]



S11 S12

Methyl 4-hydroxy-3-(2-methylallyl)benzoate S12. A solution of methyl 4-(2-methylallyloxy)benzoate (**S11**) (340 mg, 1.6 mmol) in DMF (1.4 mL) under argon was subjected to microwave irradiation at 240 °C for 1 h. The resulting mixture was diluted with water (25 mL), extracted with EtOAc (3 x 25 mL) and the combined organic extracts washed with brine (3 x 25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1 then 4:1) afforded the title compound (**S12**) as a white solid (190 mg, 58%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (1H, dd, *J* = 8.4, 2.2 Hz), 7.82 (1H, d, *J* = 2.1 Hz), 6.85 (1H, d, *J* = 8.4 Hz), 5.79 (1H, s), 4.95 (1H, s), 4.88 (1H, s), 3.88 (3H, s), 3.41 (2H, s), 1.73 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.2, 159.2, 144.3, 133.1, 130.3, 124.8, 122.8, 116.0, 113.1, 52.1, 39.9, 22.1; IR (solid) 3302, 1682, 1601, 1424, 1306, 1287, 1123; HRMS (EI) exact mass calculated for $C_{12}H_{14}O_3$ $[M]^+$ *m/z* 206.0943, found *m/z* 206.0940.

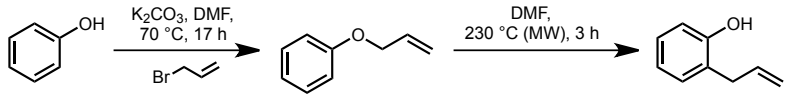


S13 S14 S15

4-bromo-2-(2-methylallyl)phenol S15.[1] To a stirred suspension of K_2CO_3 (0.80 g, 5.8 mmol) in DMF (15 mL) was added 4-bromophenol (**S13**) (0.50 g, 2.9 mmol) followed by 3-chloro-2-methyl-1-propene (**S2**) (0.34 mL, 3.5 mmol). The resulting mixture was heated at 70 °C for 18 h then cooled to room temperature, quenched with water (25 mL) and extracted with Et_2O (2 x 25 mL). The combined organic extracts were washed with brine (2 x 25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford 1-(2-methylallyloxy)-4-bromobenzene (**S14**) as a colourless oil (660 mg, quant.) which was used without any further purification. Analytical data observed were in

accordance with literature values.[5]

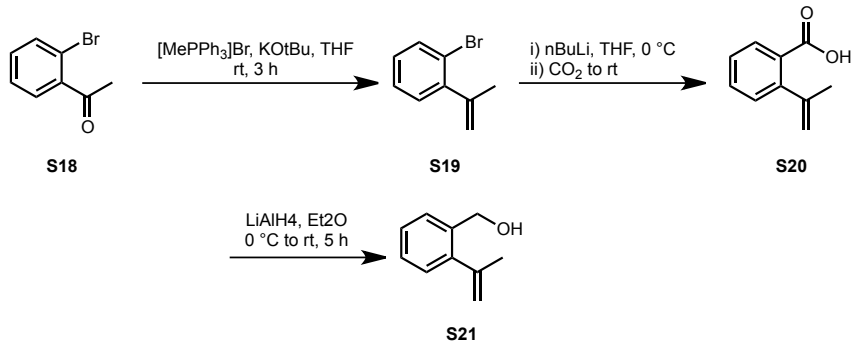
A solution of 1-(2-methylallyloxy)-4-bromobenzene (**S14**) (380 mg, 1.7 mmol) in DMF (1.4 mL) under argon was subjected to microwave irradiation at 240 °C for 30 minutes. The resulting mixture was diluted with water (25 mL), extracted with EtOAc (3 x 25 mL) and the combined organic extracts washed with brine (3 x 25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1) afforded the title compound (**S15**) as a colourless oil (260 mg, 70%). Analytical data observed were in accordance with literature values.[1]



S1 **S16** **S17**

2-allyl phenol S17. To a mixture of phenol (**S1**) (1.0 g, 11 mmol) and potassium carbonate (2.9 g, 21 mmol) in DMF (53 mL) was added allyl bromide (1.1 mL, 13 mmol). The resulting mixture was heated at 70 °C for 17 h then quenched with water (50 mL) and extracted with Et_2O (2 x 50 mL). The combined organic extracts were washed with brine (2 x 50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography afforded 1-(allyloxy)benzene (**S16**) as a colourless oil (1.3 g, 88%). Analytical data observed was in accordance with literature values.[6]

A solution of 1-(allyloxy)benzene (**S16**) (1.3 g, 9.3 mmol) in DMF (7.8 mL) under argon was subjected to microwave irradiation at 230 °C for 3 h. The resulting mixture was extracted with Et_2O (2 x 50 mL) and the combined organic extracts washed brine (2 x 50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 19:1) afforded the title compound (**S17**) as a yellow oil (1.0 g, 81%). Analytical data observed was in accordance with literature values.[7]

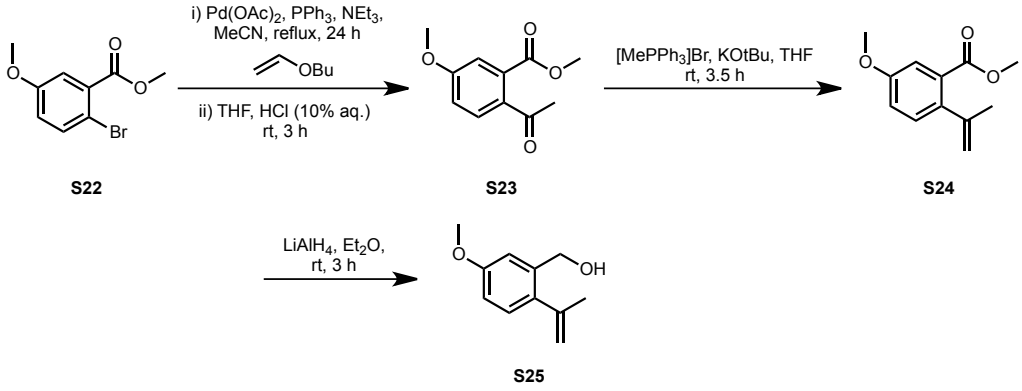


(2-(Prop-1-en-2-yl)phenyl) methanol S21. [8] To a stirred suspension of methyl triphenyl-phosphonium bromide (7.2 g, 20 mmol) in THF (51 mL) was added a solution of potassium *tert*-butoxide (2.3 g, 20 mmol) in THF (21 mL). The resulting bright yellow suspension was stirred at room temperature for 15 minutes then a solution of *o*-bromoacetophenone (**S18**) (3.4 g, 17 mmol) in THF (34 mL) added dropwise. The resulting suspension was stirred at room temperature for 3 h then quenched with sat. aq. NH_4Cl (100 mL). The mixture was extracted with Et_2O (100 mL then 2 x 50 mL) and the combined organic extracts dried ($MgSO_4$), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether) afforded 1-bromo-2-(prop-1-en-2-yl)-benzene (**S19**) as a colourless oil (3.0 g, 91%). Analytical data observed were in accordance with literature values.[9]

To a cooled (0 °C) solution of 1-bromo-2-(prop-1-en-2-yl) benzene (**S19**) (2.1 g, 11 mmol) in Et_2O (21 mL) was added dropwise a solution of *n*-butyllithium (2.1M in hexanes, 5.3 mL, 11 mmol). The resulting yellow suspension was stirred for 15 minutes then added dropwise to a flask charged with carbon dioxide pellets (ca. 50 g). The mixture was allowed to warm to room temperature over 2 h then quenched with sat. aq. $NaHCO_3$ (100 mL). The mixture was extracted with Et_2O (2 x 50 mL) and the aqueous phase adjusted to pH 1 with 1M aq. HCl. The aqueous phase was then extracted with Et_2O (3 x 100 mL) and the combined organic extracts dried ($MgSO_4$), filtered and concentrated *in vacuo* to afford 2-(prop-1-en-2-yl) benzoic acid (**S20**) as a sticky white solid (1.7 g, 99%). Analytical data observed were in accordance with literature values.[1]

To a cooled (0 °C) suspension of lithium aluminum hydride (470 mg, 12 mmol) in Et_2O (34 mL) was added dropwise a solution of 2-(prop-1-en-2-yl) benzoic acid (**S20**) (780 mg, 4.8 mmol) in Et_2O (20 mL). After 5 minutes, the reaction mixture was allowed to warm to room temperature and stirred for

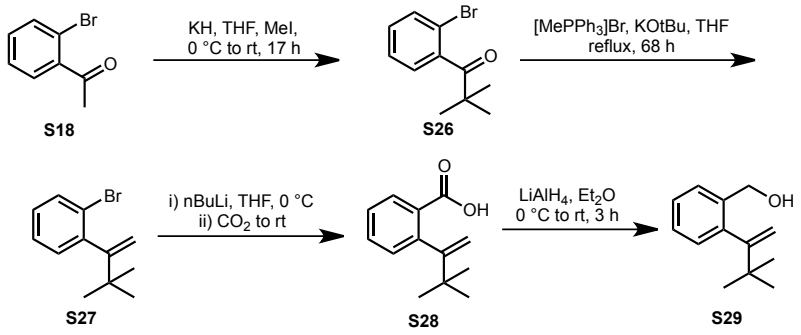
5 h. The mixture was re-cooled to 0 °C and aq. sat. potassium sodium tartrate (25 mL) added slowly. The biphasic mixture was allowed to stir at room temperature for 16 h then extracted with Et_2O (50 mL). The organic extracts were washed with water (50 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1) afforded the title compound (**S21**) as a colourless oil (0.64 g, 90%). Analytical data observed were in accordance with literature values.[8]



(5-methoxy-2-(prop-1-en-2-yl)phenyl)methanol S25. Following a reported procedure,[10] to a suspension of $Pd(OAc)_2$ (0.14 g, 0.62 mmol) and triphenylphosphine (0.32 g, 1.2 mmol) in degassed acetonitrile (16 mL) was added *n*-butyl vinyl ether (5.3 mL, 41 mmol), triethylamine (1.5 mL, 11 mmol) and methyl 2-bromo-5-methoxybenzoate (**S22**) (2.0 g, 8.2 mmol). The resulting mixture was heated at reflux for 24 h then cooled to room temperature, diluted with water (20 mL) and EtOAc (20 mL) and filtered over Celite®, rinsing with EtOAc (25 mL). The filtrate was concentrated *in vacuo* then dissolved in THF (51 mL) and 10% aq. HCl (51 mL) added. The mixture was stirred at room temperature for 3 h then concentrated to low volume, poured into sat. aq. $NaHCO_3$ (100 mL) and extracted with 10:1 EtOAc:DCM (4 x 50 mL). The combined organic extracts were washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 7:3) afforded methyl 2-acetyl-5-methoxybenzoate (**S23**) as a yellow oil (1.5 g, 90%). Analytical data observed were in accordance with literature values.[10]

To a suspension of methyl triphenylphosphonium bromide (2.4 g, 6.8 mmol) in THF (14 mL) was added a solution of potassium *tert*-butoxide (760 mg, 6.8 mmol) in THF (7.0 mL). The resulting bright yellow suspension was stirred at room temperature for 10 minutes then a solution of methyl 2-acetyl-5-methoxybenzoate (**S23**) (1.2 g, 5.7 mmol) in THF (11 mL) added dropwise. The brown suspension was stirred at room temperature for 3.5 h then quenched with water (10 mL) and extracted with Et_2O (2 x 50 mL). The combined organic extracts were washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 19:1 then 9:1) afforded methyl 5-methoxy-2-(prop-1-en-2-yl)benzoate (**S24**) as a yellow oil (610 mg, 52%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (1H, d, *J* = 2.8 Hz), 7.16 (1H, d, *J* = 8.5 Hz), 6.98 (1H, dd, *J* = 8.5 Hz, 2.8 Hz), 5.07–5.06 (1H, m), 4.81–4.80 (1H, m), 3.85 (3H, s), 3.83 (3H, s), 2.06–2.05 (3H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.5, 158.6, 146.2, 137.8, 130.8, 130.6, 117.8, 114.6, 113.7, 55.7, 52.1, 24.3; IR (thin film) 1724, 1285, 1244, 1069, 1034; HRMS (EI) exact mass calculated for $C_{12}H_{14}O_3$ $[M]^+$ *m/z* 206.0943, found *m/z* 206.0941.

To a cooled (0 °C) suspension of lithium aluminum hydride (77 mg, 2.0 mmol) in Et_2O (4.0 mL) was added dropwise a solution of methyl 5-methoxy-2-(prop-1-en-2-yl)benzoate (**S24**) (160 mg, 0.78 mmol) in Et_2O (2.0 mL). The cooling bath was removed and the mixture allowed to warm to room temperature then stirred for 3 h. The mixture was cooled to 0 °C, quenched by dropwise addition of sat. aq. potassium sodium tartrate (10 mL) and stirred for 30 minutes then extracted with Et_2O (2 x 25 mL). The combined organic extracts were washed with water (25 mL), brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford the title compound (**S25**) as a colourless oil (130 mg, 95%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.09 (1H, d, *J* = 8.4 Hz), 7.02 (1H, d, *J* = 2.7 Hz), 6.80 (1H, dd, *J* = 2.7 Hz), 5.21–5.20 (1H, m), 4.87–4.86 (1H, m), 4.69 (2H, d, *J* = 3.9 Hz), 3.82 (3H, s), 2.06–2.05 (3H, m), 1.70–1.69 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.1, 144.7, 139.1, 135.6, 129.4, 115.5, 113.5, 113.3, 63.4, 55.5, 25.2; IR (thin film) 3343, 1607, 1497, 1288, 1233, 1161; HRMS (EI) exact mass calculated for $C_{11}H_{14}O_2$ $[M]^+$ *m/z* 178.0994, found *m/z* 178.0995.



S18 S26 S27 S28 S29

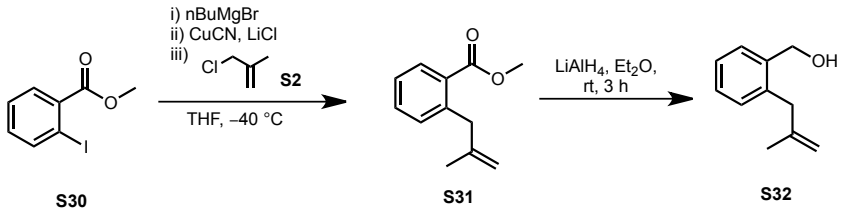
(2-(3,3-dimethylbut-1-en-2-yl)phenyl)methanol S29. To a cooled (0 °C) suspension of potassium hydride (30% suspension in mineral oil, 2.3 g, 18 mmol) in THF (25 mL) was added 2'-bromoacetophenone (**S18**) (1.0 g, 5 mmol). After 15 minutes, iodomethane (1.6 mL, 25 mmol) was added and the mixture stirred at room temperature for 17 h. The mixture was quenched with sat. aq. NH_4Cl (50 mL) and extracted with Et_2O (3 x 50 mL). The combined organic extracts were washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 19:1) afforded 1-(2-bromophenyl)-2,2-dimethylpropan-1-one (**S26**) as a colourless oil (2.4 g) containing residual mineral oil. The material was used directly in the subsequent step.

To a suspension of methyl triphenylphosphonium bromide (4.3 g, 12 mmol) in THF (24 mL) was added a solution of potassium *tert*-butoxide (1.4 g, 12 mmol) in THF (12 mL). The resulting bright yellow suspension was stirred at room temperature for 15 minutes then a solution of 1-(2-bromophenyl)-2,2-dimethylpropan-1-one (**S26**) (2.4 g) in THF (20 mL) added dropwise. The resulting mixture was heated at reflux for 68 h then cooled to room temperature, quenched with sat. aq. NH_4Cl (50 mL) and extracted with Et_2O (2 x 50 mL). The combined organic extracts were washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether) afforded 1-bromo-2-(3,3-dimethylbut-1-en-2-yl)benzene (**S27**) as a colourless oil (2.5 g) containing residual mineral oil.

To a cooled (0 °C) solution of 1-bromo-2-(3,3-dimethylbut-1-en-2-yl)benzene (**S27**) (2.5 g) in Et_2O (5 mL) was added dropwise a solution of *n*-butyllithium (2.0M in hexanes, 5.4 mL, 11 mmol). The resulting mixture was stirred for 15 minutes then CO_2 bubbled through the mixture for 1.5 h. The reaction was quenched with sat. aq. $NaHCO_3$ (50 mL) and extracted with Et_2O (2 x 50 mL). The

aqueous phase was adjusted to pH 1 with conc. aq. HCl then re-extracted with Et_2O (2 x 50 mL). The combined organic extracts were washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford 2-(3,3-dimethylbut-1-en-2-yl)benzoic acid (**S28**) as a pale yellow oil (1.3 g, 62% over three steps). ^{1}H NMR (500 MHz, $CDCl_3$) δ 11.7 (1H, s), 7.99 (1H, dd, *J* = 7.9, 1.2 Hz), 7.47 (1H, td, *J* = 7.5, 1.4 Hz), 7.35 (1H, td, *J* = 7.6, 1.3 Hz), 7.19 (1H, dd, *J* = 7.7, 1.0 Hz), 5.30 (1H, d, *J* = 1.1 Hz), 4.86 (1H, d, *J* = 1.1 Hz), 1.14 (9H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.0, 158.7, 144.9, 131.7, 130.8, 130.7, 129.4, 126.8, 112.3, 36.8, 30.4; IR (thin film) 3377, 2955, 1694, 1260, 1068; HRMS (EI) exact mass calculated for $C_{13}H_{16}O_2$ $[M]^+$ *m/z* 204.1150, found *m/z* 204.1151.

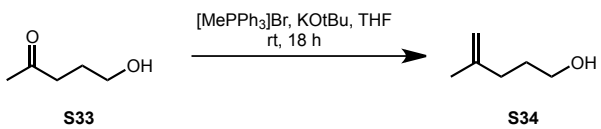
To a cooled (0 °C) suspension of lithium aluminum hydride (480 mg, 13 mmol) in Et_2O (30 mL) was added dropwise a solution of 2-(3,3-dimethylbut-1-en-2-yl)benzoic acid (**S28**) (1.0 g, 4.9 mmol) in Et_2O (5 mL). After 5 minutes, the reaction mixture was allowed to warm to room temperature and stirred for 3 h. The mixture was re-cooled to 0 °C and sat. aq. potassium sodium tartrate (25 mL) added slowly. The biphasic mixture was stirred at room temperature for 16 h then extracted with Et_2O (50 mL). The organic extracts were washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford the title compound (**S29**) as a colourless oil (880 mg, 95%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (1H, d, *J* = 7.0 Hz), 7.30 (1H, td, *J* = 7.5, 1.3 Hz), 7.22 (1H, td, *J* = 7.5, 1.3 Hz), 7.08 (1H, dd, *J* = 7.6, 1.2 Hz), 5.34 (1H, d, *J* = 1.5 Hz), 4.82 (1H, d, *J* = 1.5 Hz), 4.63 (2H, s), 1.56–1.54 (1H, m), 1.12 (9H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.3, 141.5, 138.8, 129.6, 128.1, 127.2, 126.6, 113.3, 63.6, 36.7, 30.0; IR (thin film) 3314, 2955, 1481, 1360, 1190; HRMS (CI) exact mass calculated for $C_{13}H_{17}$ $[M\text{-}OH]^+$ *m/z* 173.1330, found *m/z* 173.1326.



(2-(2-methylallyl)phenyl)methanol S32. Following a modification of a reported procedure,[11] to a mixture of magnesium turnings (0.22 g, 9.2 mmol) and a crystal of iodine in THF (15 mL) was added

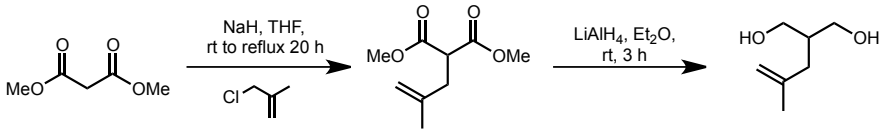
dropwise bromobutane (0.82 mL, 7.6 mmol). The mixture was stirred for 15 minutes the cooled to −40 °C before dropwise addition of a solution of Methyl-2-iodobenzoate (**S30**) (1.0 g, 3.8 mmol) in THF (30 mL). The mixture was stirred at −40 °C for 1.5 h. A freshly prepared solution of LiCl (0.39 g, 9.1 mmol) and CuCN (0.39 g, 9.1 mmol) in THF (20 mL) was added and the mixture was stirred for a further 15 min, followed by the addition of 3-chloro-2-methyl-1-propene (**S2**) (1.5 mL, 15 mmol). The mixture was stirrer at −40 °C for a further 10 min, then warmed to room temperature. The mixture was diluted with EtOAc (20 mL) and filtered over Celite®. The filtrate was washed with 25% aq. NH_4OH (20 mL). The aqueous layer was further extracted with EtOAc (2 × 20 mL), and the combined organic extracts washed with brine (20 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether/EtOAc, 9/1), afforded methyl 2-(2-methylallyl)benzoate (**S31**) as a colourless oil (0.65 g, 89 %). Material was used directly in the next step.

To a cooled suspension of lithium aluminum hydride (260 mg, 6.8 mmol) in Et_2O (15 mL) was added a solution of methyl 2-(2-methylallyl)benzoate (**S31**) (0.50 g, 2.6 mmol) in Et_2O (3.0 mL). The cooling bath was removed and the reaction mixture stirred at room temperature for 3 h then quenched by dropwise addition of sat. aq. potassium sodium tartrate (25 mL) and stirred for 16 h. The mixture was extracted with Et_2O (2 x 25 mL) and the combined organic extracts dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography afforded the title compound (**S32**) as a colourless oil (400 mg, 95%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41–7.39 (1H, m), 7.27–7.25 (2H, m), 7.19–7.18 (1H, m), 4.84 (1H, s), 4.67 (2H, d, *J* = 5.7 Hz), 4.54 (1H, s), 3.41 (2H, s), 1.86 (1H, t, *J* = 5.9 Hz), 1.76 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.8, 139.2, 137.5, 130.7, 128.6, 128.0, 126.9, 111.9, 63.3, 41.1, 22.9; IR (thin film) 3320, 1445, 1038, 1005; HRMS (EI) exact mass calculated for $C_{11}H_{14}O$ $[M]^+$ *m/z* 162.1045, found *m/z* 162.1042.



S33 **S34**

4-methylpent-4-en-1-ol S34.[12] To a stirred suspension of methyl triphenylphosphonium bromide (4.2 g, 12 mmol) in THF (29 mL) was added a solution of potassium *tert*-butoxide (1.3 g, 12 mmol) in THF (12 mL). The suspension turned bright yellow and was stirred at room temperature for 15 minutes before addition of a solution of 1-hydroxy-4-pentanone (**S33**) (1.0 g, 9.8 mmol) in THF (19 mL). The mixture was stirred at room temperature for 18 h then quenched with sat. aq. NH_4Cl (25 mL) and extracted with Et_2O (3 x 25 mL). The combined organic extracts were washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to low volume. Purification by flash chromatography (CH_2Cl_2:Et_2O, 9:1) afforded the title compound (**S34**) as a volatile colourless oil (520 mg, 53%). Analytical data observed were in accordance with literature values.[10]

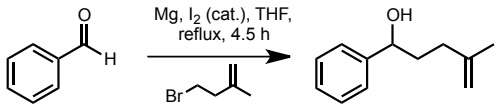


S35 **S2** **S36** **S37**

2-(2-methylallyl)propane-1,3-diol S37.[13] Following a modification of the reported procedure[14], to a cooled (0 °C) suspension of sodium hydride (60% dispersion in mineral oil, 180 mg, 4.5 mmol) in THF (57 mL) was added dropwise dimethyl malonate (**S35**) (0.69 mL, 6.0 mmol). Once effervescence had ceased, 3-chloro-2-methyl-1-propene (**S2**) (0.32 mL, 3.3 mmol) was added then the cooling bath was removed. The mixture was stirred at room temperature for 2 h then heated at reflux for 18 h. After cooling to room temperature, the reaction mixture was quenched by addition of water (25 mL) and extracted with Et_2O (2 x 25 mL). The combined organic extracts were washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1) afforded dimethyl 2-(2-methylallyl)malonate (**S36**) as a colourless oil (340 mg, 60%). Analytical data observed were in accordance with literature values.[15]

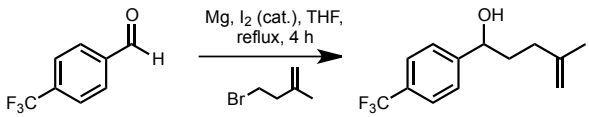
To a cooled (0 °C) suspension of lithium aluminum hydride (180 mg, 4.7 mmol) in Et_2O (10 mL) was added dropwise a solution of dimethyl 2-(2-methylallyl)malonate (**S36**) (330 mg, 1.8 mmol) in Et_2O (3.0 mL). The cooling bath was was removed and the reaction mixture stirred at room temperature for 3 h before quenching by dropwise addition of sat. aq. potassium sodium tartrate (10 mL). The

resulting mixture was stirred at room temperature for 18 h. The organics were separated, washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (CH_2Cl_2:EtOAc, 3:2 then 2:3) afforded the title compound (**S37**) as a colourless oil (130 mg, 58%). Analytical data observed were in accordance with literature values.[13]



S38 **S39** **S40**

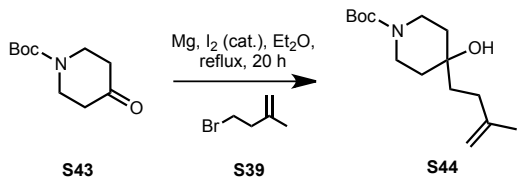
4-methyl-1-phenylpent-4-en-1-ol S40. To a flask charged with magnesium turnings (430 mg, 18 mmol) and a crystal of iodine in Et_2O (17 mL) was added 4-bromo-2-methyl-1-butene[16] (**S39**) (5M in hexanes, 2.0 mL, 10 mmol). The mixture was heated to reflux for 1 h then allowed cooled to room temperature before dropwise addition of benzaldehyde (**S38**) (0.87 mL, 8.6 mmol). The mixture was heated at reflux for 3.5 h then cooled to room temperature, poured onto ice and 1M aq. HCl (25 mL) added dropwise. The mixture was extracted with Et_2O (2 x 50 mL) and the combined organic extracts washed with sat. aq. $NaHCO_3$ (50 mL), brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1) afforded the title compound (**S40**) as a colourless oil (630 mg, 42%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.34 (4H, m), 7.30–7.26 (1H, m), 4.74 (1H, s), 4.71 (1H, s), 4.69 (1H, dd, *J* = 7.6 Hz, 5.5Hz), 2.18–2.12 (1H, m), 2.08–2.02 (1H, m), 1.98–1.82 (3H, m), 1.73 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.6, 144.8, 128.6, 127.7, 126.0, 110.3, 74.4, 37.0, 34.2, 22.7; IR (thin film) 3353, 1451, 1062, 1017; HRMS (CI) exact mass calculated for $C_{12}H_{15}$ $[M\text{-}OH]^+$ *m/z* 159.1174, found *m/z* 159.1169.



S41 **S39** **S42**

1-(4-(trifluoromethyl)phenyl)-4-methylpent-4-en-1-ol S42. To a flask charged with magnesium turnings (430 mg, 18 mmol) and a crystal of iodine in THF (17 mL) was added 4-bromo-2-methyl-1-butene[16] (**S39**) (5M in hexanes, 2.0 mL, 10 mmol). The mixture was heated to reflux for 1 h then

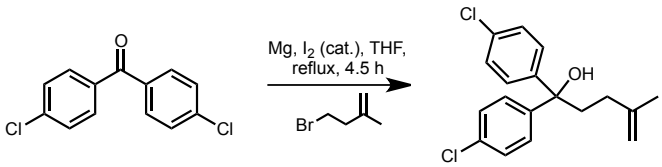
allowed to cool before dropwise addition of 4-(trifluoromethyl)benzaldehyde (**S41**) (1.2 mL, 8.6 mmol). The mixture was heated at reflux for 3 h then cooled to room temperature, poured onto ice and stirred for 30 minutes before addition of 1M aq. HCl (25 mL). The mixture was extracted with Et_2O (2 x 50 mL) and the combined organic extracts washed with sat. aq. $NaHCO_3$ (50 mL), brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1) afforded the title compound (**S42**) as a faintly yellow oil (1.3 g, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (2H, d, *J* = 8.1 Hz), 7.47 (2H, d, *J* 8.5 Hz), 4.79–4.73 (3H, m), 2.27–2.06 (2H, m), 2.04–1.82 (3H, m), 1.75 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.9, 145.3, 130.0 (q, ^2J (C-F) = 32 Hz), 126.3, 125.6 (q, ^3J (C-F) = 3.8 Hz), 124.4 (q, ^1J (C-F) = 270 Hz) 110.7, 73.8, 37.2, 34.0, 22.5; IR (thin film) 3339, 1323, 1122, 1109, 1067; HRMS (EI) exact mass calculated for $C_{13}H_{16}OF_3$ $[M+H]^+$ *m/z* 245.1153, found *m/z* 245.1153.



S43 **S39** **S44**

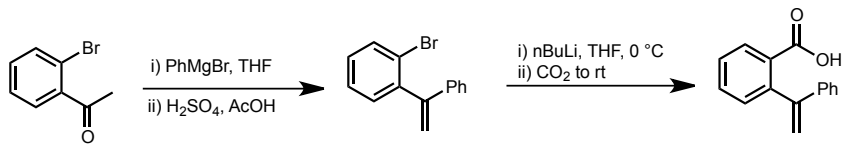
***tert*-butyl-4-hydroxy-4-(3-methylbut-3-en-1-yl)piperidine-1-carboxylate S44.** To a flask charged with magnesium turnings (250 mg, 10.3 mmol) and a crystal of iodine in Et_2O (6 mL) was added 4-bromo-2-methyl-1-butene[16] (**S39**) (860 mg, 5.8 mmol). The mixture was heated to reflux for 30 minutes then cooled to 0 °C before dropwise addition of 1-Boc-4-piperidone (**S43**) (1.0 g, 5.0 mmol) in Et_2O (5 mL). Additional Et_2O (5 mL) was added and the mixture heated at reflux for 20 h then cooled to room temperature and quenched by dropwise addition of sat. aq. NH_4Cl (50 mL). The aqueous phase was further extracted with Et_2O (2 x 50 mL) and the combined organic extracts washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 8:2 to 7:3 then CH_2Cl_2:EtOAc, 93:7) afforded the title compound (**S44**) as a pale yellow oil (174 mg, 13%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.73 (1H, s), 4.72 (1H, s), 3.80 (2H, s), 3.16 (2H, s), 2.12–2.09 (2H, m), 1.75 (3H, s), 1.63–1.60 (2H, m), 1.55–1.53 (4H, m), 1.45 (9H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.0, 146.2, 110.3, 79.5, 70.0, 40.9,

39.9, 36.9, 31.2, 28.6, 22.7; IR (thin film) 3437, 2930, 1694, 1665, 1424, 1366, 1246, 1150; HRMS (CI) exact mass calculated for $C_{15}H_{28}O_3N$ $[M+H]^+$ *m/z* 270.2069, found *m/z* 270.2066.



S45 **S39** **S46**

1,1-bis(4-chlorophenyl)-4-methylpent-4-en-1-ol S46. To a flask charged with magnesium turnings (240 mg, 10 mmol) and a crystal of iodine in THF (17 mL) was added 4-bromo-2-methyl-1-butene[16] (**S39**) (5M in hexanes, 2.0 mL, 10 mmol). The mixture was heated to reflux for 1 h then allowed to cool to room temperature before portionwise addition of 4,4'-dichlorobenzophenone (**S45**) (2.2 g, 8.6 mmol). The mixture was heated at reflux for 3 h then cooled to room temperature and quenched by dropwise addition of 1M aq. HCl (5 mL). The mixture was extracted with Et_2O (3 x 50 mL) and the combined organic extracts washed with sat. aq. $NaHCO_3$ (50 mL), brine (50 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Iterative purification by flash chromatography (petroleum ether:EtOAc, 17:3 then 19:1) followed by removal of a solid impurity by trituration with petroleum ether afforded the title compound (**S46**) as a colourless oil (220 mg, 8%), contaminated with approximately 4% of an unknown impurity. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.32 (4H, m), 7.29–7.27 (4H, m), 4.74 (1H, s), 4.69 (1H, s), 2.40–2.37 (2H, m), 2.27 (1H, s), 2.00–1.97 (2H, m), 1.73 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.9, 145.1, 133.1, 128.6, 127.5, 110.5, 77.9, 39.7, 32.0, 22.8. IR (thin film) 1489, 1093, 1013. HRMS (EI) exact mass calculated for $C_{18}H_{18}OCl_2$ $[M]^+$ *m/z* 320.0735, found *m/z* 320.0738.

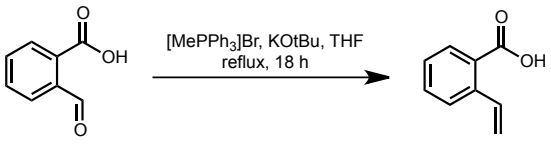


S18 **S47** **S48**

2-(1-phenylvinyl)benzoic acid S48. Following a reported procedure,[1] a solution of bromoacetophenone (**S18**) (2.0 mL, 15 mmol) in THF (2 mL) was added dropwise to a stirred

solution of phenyl magnesium bromide (1 M in THF, 16.6 mL, 16.6 mmol). The resulting mixture was heated to reflux for 2 h, then allowed to cool to room temperature. The reaction was quenched by addition of sat. aq. NH_4Cl (15 mL) and extracted with Et_2O (3 x 20 mL). The combined organic extracts were washed with water (25 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The residue was treated with a solution of H_2SO_4 in acetic acid (4 mL, 20 % v/v) at 50 °C for 5 minutes. The resulting mixture was poured into 1:1 Et_2O/H_2O (100 mL). The aqueous layer was extracted with Et_2O (2 x 100 mL) and the combined organic extracts washed with 1 M aq. $NaHCO_3$ (25 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether/EtOAc; 9:1) afforded 1-bromo-2-(1-phenylvinyl)benzene (**S47**) as a yellow oil (2.75 g, 71%).

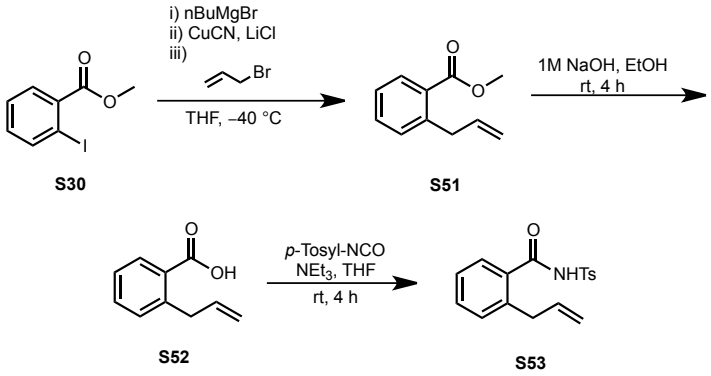
To a cooled (0 °C) solution of 2-(1-phenylvinyl)benzene (**S47**) (1.8 g, 6.7 mmol) in Et_2O (15 mL) was added dropwise *n*-butyllithium (2.3 M in THF, 3.1 mL, 7.1 mmol). The reaction mixture was stirred for 15 minutes. CO_2 was bubbled through the pale yellow mixture for 1 h. The mixture was slowly warmed to room temperature and stirred for an additional 30 minutes. The reaction was quenched with sat. aq. $NaHCO_3$ (50 mL). The aqueous layer was washed with Et_2O (3 x 20 mL), acidified to pH 1 with 1M HCl then extracted with Et_2O (3 x 50 mL). The combined organic extracts were dried ($MgSO_4$), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether/EtOAc; 9:1) afforded the title compound (**S48**) as a colorless solid (579 mg, 38%). Analytical data observed were in accordance with literature values.[1]



S49 **S50**

2-vinylbenzoic acid S50. To a stirred suspension of methyltriphenylphosphonium bromide (19 g, 53 mmol) in THF (80 mL) was added dropwise a solution of potassium tert-butoxide (8.8 g, 79 mmol) in THF (40 mL). The resultant yellow solution was stirred for 1.5 h, before the addition of a solution of 2-carboxybenzaldehyde (**S49**) (5.0 g, 33 mmol) in THF (20 mL). The solution was warmed to 60 °C and stirred overnight, before cooling and the addition of acetic acid (5.0 mL) and the solution was filtered over Celite®. The filtrate was concentrated in vacuo, before being washed

with sat. aq. $NaHCO_3$ (3 x 50 mL). The combined aqueous extracts were acidified to pH1 with 1M aq. HCl, and the aqueous phase was extracted with Et_2O (3 x 50 mL). The organic extracts were dried (Na_2SO_4), filtered and concentrated in vacuo to give 2-vinylbenzoic acid (**S50**) (3.7 g, 74%). No further purification was necessary. Analytical data observed were in accordance with literature values.[17]

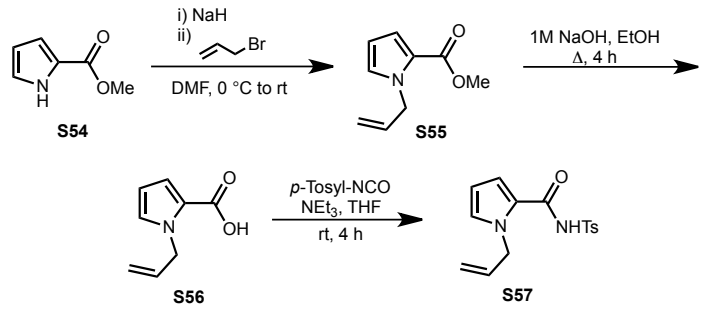


2-allyl-*N*-tosylbenzamide S53. Following a modification of a reported procedure,[9] to a suspension of magnesium turnings (2.0 g, 67 mmol) and a crystal of iodine in THF (30 mL) was added dropwise bromobutane (7.2 mL, 67 mmol). The mixture was stirred for 15 minutes then cooled to −40 °C before dropwise addition of methyl-2-iodobenzoate (**S30**) (5.0 mL, 34 mmol). The mixture was stirred at −40 °C for 1.5 h. A freshly prepared solution of LiCl (3.4 g, 80 mmol) and CuCN (3.4 g, 40 mmol) in THF (60 mL) was added and the mixture was stirred for a further 15 min, followed by the addition of allyl bromide (12 mL, 140 mmol). The mixture was stirrer at −40 °C for a further 10 min, then warmed to room temperature. The mixture was diluted with EtOAc (200 mL) and filtered over Celite®. The filtrate was washed with 25% aq. NH_4OH (200 mL). The aqueous layer was further extracted with EtOAc (2 x 200 mL), and the combined organic extracts washed with brine (200 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (petroleum ether:EtOAc, 9:1), afforded methyl-2-allyl-benzoate (**S51**) as a colourless oil (5.3 g, 90%).

Methyl-2-allyl benzoate (**S51**) (2.7 g, 17 mmol) was dissolved in EtOH (250 mL), and 2 M aq. NaOH (200 mL) added. The mixture was stirred for at room temperature for 4 h then EtOH was removed *in vacuo.* The residue was extracted with Et_2O (2 x 150 mL), acidified to pH 3 with 2M aq. HCl and

extracted with EtOAc (3 x 150 mL). The combined organics were dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford 2-allyl benzoic acid (**S52**) as a colourless solid (2.7 g, quant.), which was used directly without further purification.

To a solution of 2-allyl benzoic acid (**S52**) (2.7 g, 17 mmol) in THF (50 mL) was added *p*-tosyl isocyanate (3.3 g, 17 mmol). The resulting solution was stirred for 10 min then triethylamine (2.3 mL, 17 mmol) added dropwise. Gas evolution was observed on addition. After 1 h, the mixture was diluted with EtOAc (50 mL) and washed with 2 M aq. HCl (50 mL) and brine (50 mL). The organic extracts were dried (Na_2SO_4), filtered and concentrated *in vacuo*. The crude product was purified by column chromatography (CH_2Cl_2) to afford the title compound (**S53**) as a colourless solid (4.6 g, 88%). Analytical data observed were in accordance with literature values.[9]

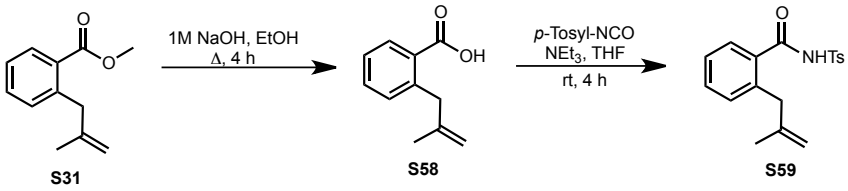


1-allyl-*N*-tosyl-1H-pyrrole-2-carboxamide S57. Following a modification of a reported procedure,[9] to a cooled (0 °C) suspension of NaH (60% dispersion in mineral oil, 480 mg, 12 mmol) in DMF (10 mL) was added a solution of methyl-2-pyrrole-carboxylate (**S54**) (1.0 g, 8.0 mmol) in DMF (2 mL). The resulting mixture was stirred for 20 min at 0 °C, then allyl bromide (1.2 mL, 14 mmol) added dropwise. The mixture was allowed to warm to room temperature, and stirred for 2 h, then quenched by pouring onto ice (30 g). The mixture was extracted with Et_2O (3 x 15 mL) and the combined organic extracts were washed with water (4 x 15 mL), brine (1 x 15 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford methyl-1-allyl-pyrrole-2-carboxylate (**S55**) as a colourless oil (1.3 g, 95%) which was used directly without further purification.

To a solution of methyl-1-allyl-pyrrole-2-carboxylate (**S55**) (1.3 g, 7.6 mmol) in EtOH (18 mL) was added 1 M aq. NaOH (21 mL). The mixture was refluxed for 1.5 h, then EtOH was removed *in vacuo*. The aqueous phase was washed with EtOAc (3 x 25 mL) then acidified to pH 2 with 4 M aq.

HCl and extracted with EtOAc (3 x 25 mL). The combined organic extracts were dried (Na_2SO_4), filtered and concentrated *in vacuo*, to afford 1-allyl-pyrrole-2-carboxylic acid (**S56**) as a colourless solid (1.0 g, 89%) which was used directly without further purification.

To a flask charged with 1-allyl-pyrrole-2-carboxylic acid (**S56**) (0.50 g, 3.3 mmol) in THF (10 mL) was added *p*-tosyl isocyanate (0.77 g, 6.6 mmol). The resulting solution was stirred for 10 min then triethylamine (0.50 mL, 3.6 mmol) added dropwise. Gas evolution was observed on addition. After 1 h, the mixture was diluted with EtOAc (50 mL) and washed with 2 M aq. HCl (50 mL) and brine (50 mL). The organic extracts were dried (Na_2SO_4), filtered and concentrated *in vacuo*. The crude product was purified by column chromatography (CH_2Cl_2) to afford the title compound (**S57**) as a colourless solid (0.71 g, 61%). Analytical data observed were in accordance with literature values.[9]



***N*-tosyl-2-(2-methylallyl)benzamide S59.** Methyl-2-(2-methylallyl)-benzoate (**S31**) (0.65 g, 3.4 mmol) was dissolved in EtOH (7.5 mL), and 2 M aq. NaOH (5.0 mL) added. The mixture was heated to reflux for 4 h, cooled to rt, and EtOH was removed in vacuo. The residue was extracted with Et_2O (2 x 20 mL), acidified to pH 3 with 2M aq. HCl and extracted with EtOAc (3 x 20 mL). The combined organics were dried (Na_2SO_4), filtered and concentrated in vacuo to afford 2-(2-methylallyl)benzoic acid (**S58**) as a colourless solid (0.48 g, 80%), which was used directly without further purification.

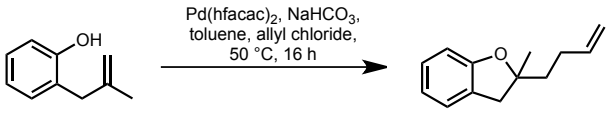
To a solution of 2-(2-methylallyl)-benzoic acid (**S58**) (0.48 g, 2.72 mmol) in THF (30 mL) was added *p*-tosyl isocyanate (0.96 mL, 6.3 mmol). The resulting solution was stirred for 10 min then triethylamine (0.81 mL, 6.3 mmol) added dropwise. Gas evolution was observed on addition. After 4 h, the mixture was diluted with EtOAc (30 mL) and washed with 2 M aq. HCl (30 mL) and brine (30 mL). The organic extracts were dried (Na_2SO_4), filtered and concentrated in vacuo. The crude product was purified by column chromatography (CH_2Cl_2) to afford the title compound (**S59**) as a

colourless solid (0.83 g, 93%). 1H NMR (500 MHz, CDCl3) δ 8.54 (1H, brs), 8.00 (2H, d, J = 8.3 Hz), 7.49–4.48 (1H, m), 7.44–7.41 (1H, m), 7.36 (2H, d, J = 8.3 Hz), 7.30–7.27 (1H, m), 7.20–7.19 (1H, m), 4.80 (1H, s), 4.28 (1H, s), 3.39 (2H, s), 2.46 (3H, s), 1.72 (3H, s); 13C NMR (100 MHz, CDCl3) δ 166.2, 146.5, 145.3, 137.9, 135.6, 133.4, 131.92, 131.90, 129.7, 128.8, 128.6, 127.1, 112.9, 41.4, 23.1, 21.8; IR (thin film) 3217, 2922, 1724, 1595; HRMS (CI/Isobutane) exact mass calculated for $C_{18}H_{20}NO_3S$ $[M+H]^+$ m/z 330.1164; found m/z 330.1158

3. Oxyallylation Reactions

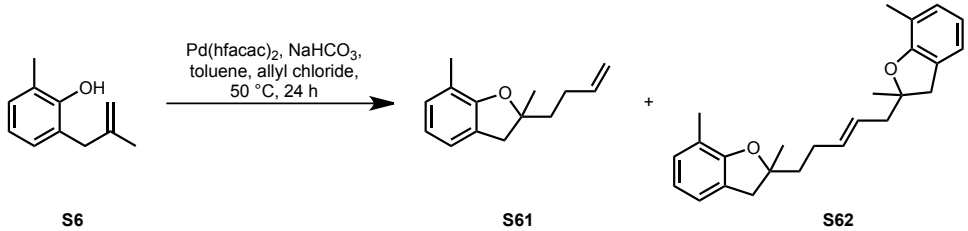
General Procedure for Pd-catalyzed heteroallylation of unactivated alkenes:

A 4 mL screw-top glass vial was charged with the substrate (1 equiv.), toluene (0.25 M), allyl halide (5 equiv.), $NaHCO_3$ (2 equiv.) and $Pd(hfacac)_2$ (5 mol%) and the vial was sealed under ambient atmosphere. The resulting mixture was heated to 50 °C by immersion of the entire vial into a pre-heated aluminum block until the substrate had been consumed, as judged by TLC analysis or ^1H NMR analysis. The reaction mixture was cooled to room temperature then purified directly by flash chromatography on silica gel.



S4 **S60**

2-(but-3-enyl)-2,3-dihydro-2-methylbenzofuran S60. The general procedure was employed for the heterocyclisation of 2-(2-methylallyl)phenol (**S4**) (49 mg, 0.33 mmol) with allyl chloride (0.13 mL, 1.6 mmol) over 16 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 17:3) afforded the title compound (**S60**) as a colourless oil (44 mg, 70%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.14–7.09 (2H, m), 6.81 (1H, t, *J* = 7.4 Hz), 6.73 (1H, d, *J* = 8.0 Hz), 5.83 (1H, m), 5.03 (1H, dd, *J* = 17.1, 1.7 Hz), 4.95 (1H, dd, *J* = 10.2 Hz, 1.4 Hz), 3.10 (1H, d, *J* = 15.6 Hz), 2.94 (1H, d, *J* = 15.5 Hz), 2.20–2.15 (2H, m), 1.85–1.81 (2H, m), 1.44 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.1, 138.5, 128.1, 127.0, 125.2, 120.0, 114.7, 109.5, 88.4, 41.4, 40.5, 28.6, 26.6; IR (thin film) 1481, 1242. HRMS (EI) exact mass calculated for $C_{13}H_{17}O$ $[M+H]^+$ *m/z* 189.1279, found *m/z* 189.1283.

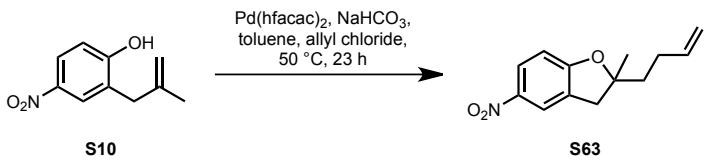


S6 **S61** **S62**

2-(but-3-enyl)-2,3-dihydro-2,7-dimethylbenzofuran S61. The general procedure was employed for the heterocyclisation of 2-methyl-6-(2-methylallyl)phenol (**S6**) (910 mg, 5.6 mmol) with allyl chloride (2.2 mL, 28 mmol) over 24 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 9:1) afforded the title compound (**S61**) as a colourless oil (840 mg, 75%). Side product **S62** was also obtained as a pale yellow oil (159 mg, 16%), contaminated with a small amount of unidentified impurity.

S61: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.97 (1H, d, *J* = 7.3 Hz), 6.93 (1H, d, *J* = 7.5 Hz), 6.73 (1H, t, *J* = 7.4 Hz), 5.90–5.82 (1H, m), 5.04 (1H, dq, *J* = 17.1, 1.7 Hz), 4.96 (1H, ddd, *J* = 10.2, 1.8, 1.3 Hz), 3.10 (1H, d, *J* = 15.4 Hz), 2.96 (1H, d, *J* = 15.4 Hz), 2.23–2.17 (5H, m), 1.89–1.80 (2H, m), 1.46 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.8, 138.7, 129.3, 126.2, 122.5, 119.8, 119.7, 114.5, 87.8, 41.9, 40.6, 28.6, 26.7, 15.4; IR (thin film) 1466, 1261; HRMS (EI) exact mass calculated for $C_{14}H_{18}O$ $[M]^+$ *m/z* 202.1358, found *m/z* 202.1358.

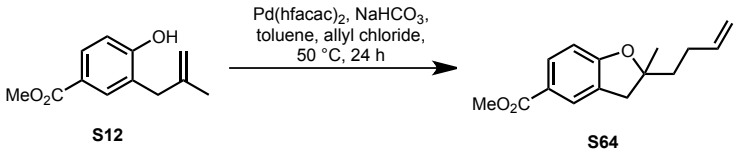
S62: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.00–6.94 (4H, m), 6.78–6.73 (2H, m), 5.61–5.55 (1H, m), 5.52–5.46 (1H, m), 3.12–3.05 (2H, m), 2.98–2.85 (2H, m), 2.51–2.42 (2H, m), 2.27–2.13 (8H, m), 1.83–1.75 (2H, m), 1.47–1.45 (6H, m); ^{13}C NMR (125MHz, $CDCl_3$) δ 157.65, 157.62, 134.1, 129.2, 129.1, 126.3, 126.2, 125.1, 122.5 (corresponds to two carbons), 119.80, 119.76, 119.6, 119.5, 87.8 (corresponds to two carbons), 44.4, 41.6, 41.1, 40.9, 27.5, 26.7, 26.6, 15.50, 15.49; IR (thin film) 1466, 1261; HRMS (EI) exact mass calculated for $C_{25}H_{30}O_2$ $[M]^+$ *m/z* 362.2246, found *m/z* 362.2248.



S10 **S63**

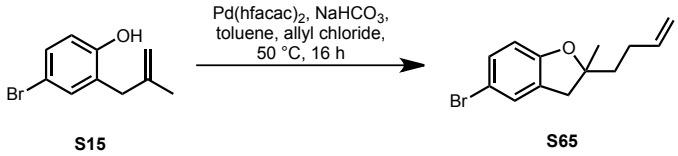
2-(but-3-enyl)-2,3-dihydro-2-methyl-5-nitrobenzofuran S63. The general procedure was employed for the heterocyclisation of 2-(2-methylallyl)-4-nitrophenol (**S10**) (64 mg, 0.33 mmol) with allyl chloride (0.13 mL, 1.6 mmol) over 23 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 3:1) afforded the title compound (**S63**) as a colourless oil (57 mg, 74%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (1H, dd, *J* = 8.8, 2.5 Hz), 8.04–8.03 (1H, m), 6.75

(1H, d, *J* = 8.0 Hz), 5.86–5.78 (1H, m), 5.05 (1H, dq, *J* = 17.1, 3.3 Hz), 4.98 (1H, dq, *J* = 10.2, 1.4 Hz), 3.16 (1H, d, *J* = 16.0 Hz), 3.01 (1H, d, *J* = 16.0 Hz), 2.20–2.14 (2H, m), 1.88 (2H, t, *J* = 8.2 Hz), 1.49 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 142.0, 137.8, 128.4, 126.0, 121.7, 115.2, 109.3, 92.0, 40.6, 40.5, 28.4, 26.6; IR (thin film) 1595, 1508, 1478, 1331, 1271, 1057; HRMS (EI) exact mass calculated for $C_{13}H_{15}O_3N$ $[M]^+$ *m/z* 233.1052, found *m/z* 233.1052.



S12 **S64**

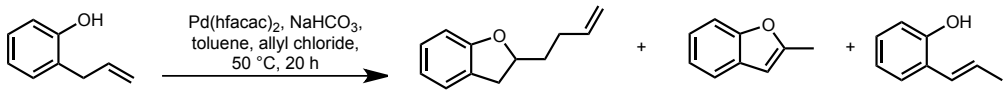
Methyl 2-(but-3-enyl)-2,3-dihydro-2-methylbenzofuran-5-carboxylate S64. The general procedure was employed for the heterocyclisation of methyl 4-hydroxy-3-(2-methylallyl)benzoate (**S12**) (68 mg, 0.33 mmol) with allyl chloride (0.13 mL, 1.6 mmol) over 24 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 1:1) afforded the title compound (**S64**) as a colourless oil (54 mg, 67%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (1H, dd, *J* = 8.4, 1.8 Hz), 7.83 (1H, s), 6.73 (1H, d, *J* = 8.4 Hz), 5.86–5.78 (1H, m), 5.02 (1H, ddd, *J* = 17.1, 1.7, 1.6 Hz), 4.96 (1H, ddd, *J* = 10.2, 1.6, 1.3 Hz), 3.87 (3H, s), 3.11 (1H, d, *J* = 15.7 Hz), 2.96 (1H, d, *J* = 15.7 Hz), 2.18–2.13 (2H, m), 1.86–1.82 (2H, m), 1.46 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.2, 163.2, 138.1, 131.3, 127.4, 127.1, 122.3, 114.9, 109.2, 90.4, 51.9, 40.7, 40.4, 28.4, 26.6; IR (thin film) 1711, 1271; HRMS (CI) exact mass calculated for $C_{15}H_{19}O_3$ $[M+H]^+$ *m/z* 247.1334, found *m/z* 247.1333.



S15 **S65**

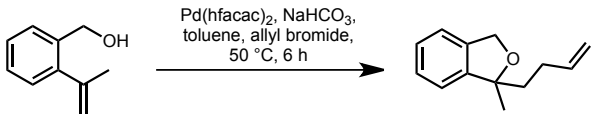
5-bromo-2-(but-3-enyl)-2,3-dihydro-2-methylbenzofuran S65. The general procedure was employed for the heterocyclisation of 4-bromo-2-(2-methylallyl)phenol (**S15**) (75 mg, 0.33 mmol) with allyl chloride (0.13 mL, 1.6 mmol) over 16 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 17:3) afforded the title compound (**S65**) as a colourless

oil (63 mg, 71%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23–7.22 (1H, m), 7.20–7.18 (1H, m), 6.60 (1H, d, *J* = 8.4 Hz), 5.84–5.78 (1H, m), 5.03 (1H, dq, *J* = 17.1, 1.7 Hz), 4.96 (1H, ddd, *J* = 10.2, 1.7, 1.2 Hz), 3.08 (1H, d, *J* = 15.8 Hz), 2.93 (1H, d, *J* = 15.8 Hz), 2.18–2.13 (2H, m), 1.83–1.80 (2H, m), 1.43 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.3, 138.2, 130.9, 129.5, 128.1, 114.8, 111.7, 111.0, 89.4, 41.1, 40.3, 28.4, 26.5; IR (thin film) 1472, 1256, 1234; HRMS (EI) exact mass calculated for $C_{13}H_{15}OBr$ $[M]^+$ *m/z* 266.0306, found *m/z* 266.0307.



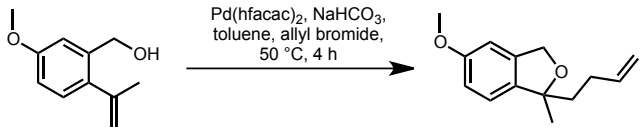
S17 S66 S67 S68

2-(but-3-enyl)-2,3-dihydrobenzofuran S66. To a solution of 2-allylphenol (44 mg, 0.33 mmol) and allyl chloride (0.13 mL, 1.6 mmol) in toluene (1.3 mL) was added $NaHCO_3$ (55 mg, 0.66 mmol) and $Pd(hfacac)_2$ (8.5 mg, 0.017 mmol). The mixture was heated at 50 °C for 20 h then filtered through a short silica plug, rinsing with petroleum ether:EtOAc, 9:1. The ^{1}H NMR spectrum of the crude material showed a mixture of **S66**, **S67**[18] and **S68**[19] in a 1 : 4.9 : 1.3 ratio. An analytical sample of **S66** was obtained by flash chromatography (petroleum ether:CH_2Cl_2, 9:1) for characterization purposes. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15 (1H, d, *J* = 7.3 Hz), 7.10 (1H, t, *J* = 7.7 Hz), 6.82 (1H, t, *J* = 7.6 Hz), 6.76 (1H, d, = 8.0 Hz), 5.91–5.82 (1H, m), 5.08 (1H, ddd, *J* = 17.1, 3.3, 1.6 Hz), 5.01–4.99 (1H, m), 4.81–4.75 (1H, m), 3.28 (1H, dd, *J* = 15.4, 7.8 Hz), 2.87 (1H, dd, *J* = 15.4, 7.8 Hz), 2.32–2.18 (2H, m), 1.98–1.91 (1H, m), 1.80–1.73 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.6, 137.9, 128.1, 127.0, 125.0, 120.3, 115.2, 109.4, 82.7, 35.6, 35.4, 29.8; IR (thin film) 1598, 1479, 1461, 1230 cm^{-1}; HRMS (EI) exact mass calculated for $C_{12}H_{14}O$ $[M]^+$ *m/z* 174.1045, found *m/z* 174.1042.



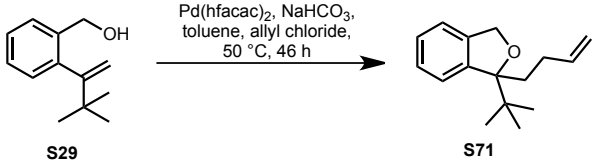
S21 S69

1-(but-3-enyl)-1,3-dihydro-1-methylisobenzofuran S69. The general procedure was employed for the heterocyclisation of (2-(prop-1-en-2-yl)phenyl) methanol (**S21**) (49 mg, 0.33 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 6 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 1:1) afforded the title compound (**S69**) as a colourless oil (48 mg, 77%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28–7.24 (2H, m), 7.19–7.18 (1H, m), 7.09–7.08 (1H, m), 5.81–5.73 (1H, m), 5.10 (1H, d, *J* = 12.3 Hz), 5.06 (1H, d, *J* = 12.3 Hz), 4.93 (1H, ddt, *J* = 17.1, 1.8, 1.7 Hz), 4.87 (1H, ddt, *J* = 10.2, 1.8, 1.3 Hz), 2.15–2.08 (1H, m), 1.94–1.77 (3H, m), 1.48 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.4, 139.2, 138.9, 127.5, 127.4, 121.1, 121.0, 114.2, 88.3, 71.8, 41.1, 28.6, 27.6; IR (thin film) 1451, 1358, 1026. HRMS (CI/Isobutane) exact mass calculated for $C_{13}H_{17}O$ [M+H]+ *m/z* 189.1279, found *m/z* 189.1281.



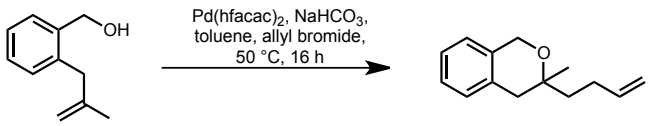
S25 **S70**

1-(but-3-enyl)-1,3-dihydro-5-methoxy-1-methylisobenzofuran S70. The general procedure was employed for the heterocyclisation of (5-methoxy-2-(prop-1-en-2-yl)phenyl)methanol (**S25**) (59 mg, 0.33 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 4 h. Purification of the reaction mixture by flash chromatography (petroleum ether:EtOAc, 19:1) afforded the title compound (**S70**) as a colourless oil (60 mg, 83%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.93 (1H, d, *J*= 8.3 Hz), 6.81 (1H, dd, *J* = 8.3, 2.3 Hz), 6.71 (1H, d, *J* = 2.0 Hz), 5.82–5.74 (1H, m), 5.05 (1H, d, *J* = 12.4 Hz), 5.01 (1H, d, *J* = 12.4 Hz), 4.96–4.92 (1H, m), 4.89–4.86 (1H, m), 3.81 (3H, s), 2.16–2.08 (1H, m), 1.93–1.79 (3H, m), 1.46 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.8, 141.0, 139.1, 137.9, 121.7, 114.1, 113.8, 106.4, 88.0, 77.6, 55.7, 41.3, 28.7, 27.8; IR (thin film) 1493, 1271, 1150, 1028; HRMS (EI) exact mass calculated for $C_{14}H_{18}O_2$ $[M]^+$ *m/z* 218.1307, found *m/z* 218.1310.



S29 **S71**

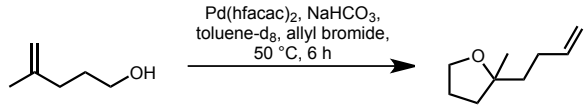
1-(but-3-en-1-yl)-1-(tert-butyl)-1,3-dihydroisobenzofuran (S71). To a solution of (2-(3,3-dimethylbut-1-en-2-yl)phenyl)methanol (**S29**) (63 mg, 0.33 mmol) and allyl bromide (0.14 mL, 1.6 mmol) in toluene (1.3 mL) was added $NaHCO_3$ (55 mg, 0.66 mmol) and $Pd(hfacac)_2$ (8.5 mg, 0.017 mmol). The mixture was heated at 50 °C for 22 h then additional $Pd(hfacac)_2$ (8.5 mg, 0.017 mmol) added. Heating was continued for a further 24 h then the mixture allowed to cool to room temperature and subjected directly to flash chromatography (petroleum ether:CH_2Cl_2, 17:3) to afford the title compound (**S71**) as a colourless oil (45 mg, 60%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28–7.22 (2H, m), 7.17–7.15 (2H, m), 5.79–5.71 (1H, m), 5.11 (1H, d, *J* = 12.2 Hz), 5.06 (1H, d, *J* = 12.2 Hz), 4.89–4.83 (2H, m), 2.08–2.03 (1H, m), 1.98–1.88 (2H, m), 1.45–1.37 (1H, m), 0.96 (9H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.0, 140.9, 139.4, 127.3, 126.8, 123.0, 120.7, 113.9, 96.1, 74.0, 40.2, 34.5, 30.1, 25.8; IR (thin film) 2955, 1040; HRMS (CI) exact mass calculated for $C_{16}H_{23}O$ $[M+H]^+$ *m/z* 231.1749, found *m/z* 231.1751.



S32 **S72**

3-(but-3-enyl)-3,4-dihydro-3-methyl-1H-isochromene S72. The general procedure was employed for the heterocyclisation of (2-(2-methylallyl)phenyl)methanol (**S32**) (54 mg, 0.33 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 16 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 1:1) followed by trituration with petroleum ether to remove a yellow solid afforded the title compound (**S72**) as a colourless oil (48 mg, 71%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18–7.15 (2H, m), 7.10–7.07 (1H, m), 7.03–6.99 (1H, m), 5.90–5.82 (1H, m), 5.05 (1H, ddd, *J* = 17.1, 3.5, 1.7 Hz), 4.96 (1H, ddd, *J* = 10.2, 3.1, 1.3 Hz), 4.80 (1H, d, *J* = 15.5 Hz), 4.75 (1H, d, *J* = 15.5 Hz), 2.79 (1H, d, *J* = 16.1 Hz), 2.66 (1H, d, *J* = 16.1 Hz), 2.25–2.19 (2H, m), 1.75 (1H, ddd, *J* = 13.8, 10.3, 6.1 Hz), 1.61 (1H, ddd, *J* = 13.8, 10.6, 6.4 Hz), 1.25 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.0, 134.4, 133.0, 129.3, 126.5, 126.0, 124.1, 114.4, 72.7, 63.1, 39.2, 38.8, 28.1, 23.4; IR (thin

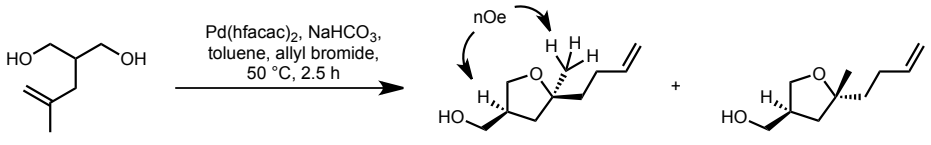
film) 1454, 1373, 1080; HRMS (EI) exact mass calculated for $C_{14}H_{18}O$ $[M]^+$ *m/z* 202.1358, found *m/z* 202.1360.



S34 **S73**

2-(but-3-enyl)-tetrahydro-2-methylfuran S73. A 4 mL glass vial was charged with 4-methylpent-4-en-1-ol (**S34**) (19 mg, 0.19 mmol), toluene-d^8 (0.75 mL), allyl bromide (0.08 mL, 0.95 mmol), $NaHCO_3$ (32 mg, 0.38 mmol) and $Pd(hfacac)_2$ (4.9 mg, 0.01 mmol). The resulting mixture was heated at 50 °C for 6 h then filtered through cotton wool directly into an NMR tube, rinsing with further toluene-d^8 (0.75 mL).

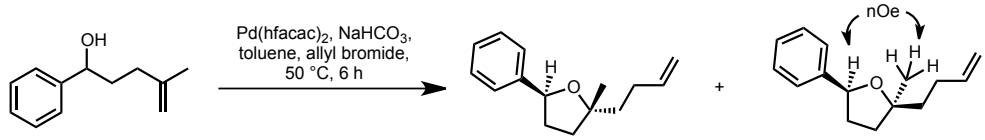
^{1}H NMR analysis indicated a 94% yield of title compound (**S73**) (*cf.* 1,3,5-trimethoxybenzene internal standard). A small amount of the volatile oil was purified by flash chromatography (chloroform) for characterisation. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.88–5.80 (1H, m), 5.02 (1H, dq, *J* = 17.1, 1.7 Hz), 4.94–4.92 (1H, m), 3.86–3.77 (2H, m), 2.17–2.05 (2H, m), 1.98–1.85 (2H, m), 1.77–1.71 (1H, m), 1.67–1.53 (3H, m), 1.18 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.3, 114.2, 82.5, 67.3, 40.4, 36.9, 29.2, 26.2, 25.8; IR (thin film) 2969, 1047; HRMS (CI) exact mass calculated for $C_9H_{17}O$ $[M+H]^+$ *m/z* 141.1279, found *m/z* 141.1275.



S37 **S74** 1.4 : 1 **S75**

(5-(but-3-enyl)-tetrahydro-5-methylfuran-3-yl)methanol S74 and S75. The general procedure was employed for the heterocyclisation of 2-(2-methylallyl)propane-1,3-diol (**S37**) (39 mg, 0.30 mmol) with allyl chloride (0.12 mL, 1.5 mmol) over 2.5 h. Purification of the reaction mixture by flash chromatography (petroleum ether:EtOAc, 19:1) afforded the title compound (**S74** and **S75**) as a colourless oil (32 mg, 63%) and as a 1.4:1 mixture of inseperable diastereomers, determined by nOe as shown above. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.85–5.79 (2H, m, S74), 5.04–5.00 (2H, m, S74, S75), 4.95–4.92 (2H, m, S74, S75), 3.99 (1H, dd, *J* = 8.8, 7.4 Hz, S74), 3.95 (1H, dd *J* = 8.9,

7.3 Hz, S75), 3.66–3.58 (6H, m, S74, S75), 2.63–2.48 (2H, m, S74, S75), 2.14–2.04 (4H, m, S74, S75), 1.97 (1H, dd, *J* 12.7 Hz, S75), 1.84 (1H, dd, *J* = 12.4 Hz, 8.3 Hz, S74), 1.67–1.58 (4H, m, S74, S75), 1.55–1.38 (4H, m, S74, S75), 1.25 (3H, m, S75), 1.18 (3H, s, S74); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.06 (S75), 139.01 (S74), 114.36 (S74), 114.31 (S75), 83.20 (S74), 83.02 (S75), 69.56 (S74), 69.53 (S75), 65.42 (S75), 65.34 (S74), 42.7 (S74), 42.2 (S75), 41.0 (S74), 40.5 (S75), 40.2 (S74), 39.9 (S75), 29.2 (S74), 29.0 (S75), 26.3 (S75), 25.4 (S74); IR (thin film) 3377, 2930, 1049, 1022; HRMS (CI) exact mass calculated for $C_{10}H_{19}O_2$ $[M+H]^+$ *m/z* 171.1385, found *m/z* 171.1388.



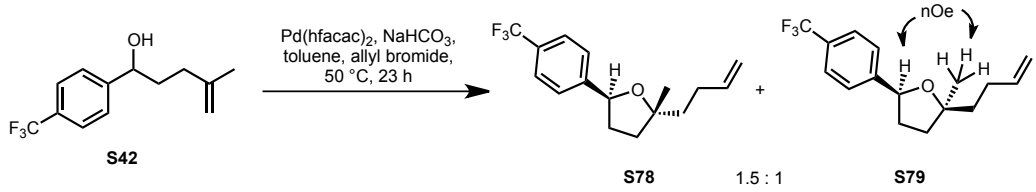
S40 **S76** 1.3 : 1 **S77**

2-(but-3-enyl)-tetrahydro-2-methyl-5-phenylfuran S76 and S77. The general procedure was employed for the heterocyclisation of 4-methyl-1-phenylpent-4-en-1-ol (**S40**) (49 mg, 0.33 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 6 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 3:1) afforded a 1.3:1 mixture of diastereomers (**S76** and **S77**) (55 mg, 77%), determined by nOe as shown above. A sample of each diastereomer was separated by flash chromatography (petroleum ether:CH_2Cl_2, 3:1) for analytical purposes.

S76: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.35 (2H, m), 7.34–7.31 (2H, m), 7.25–7.22 (1H, m), 5.93–5.85 (1H, m), 5.06 (1H, dq, *J* = 17.1, 1.7 Hz), 4.98–4.92 (2H, m), 2.33–2.28 (1H, m), 2.27–2.14 (2H, m), 1.98–1.89 (2H, m), 1.86–1.82 (1H, m), 1.79–1.67 (2H, m), 1.36 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.8, 139.3, 128.4, 127.2, 126.0, 114.2, 83.5, 81.1, 40.9, 37.8, 25.7, 29.2, 27.1; IR (thin film) 1449, 1044,+ 1020; HRMS (EI) exact mass calculated for $C_{15}H_{20}O$ $[M]^+$ *m/z* 216.1514, found *m/z* 216.1519.

S77: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.35 (2H, m), 7.33–7.30 (2H, m), 7.25–7.22 (1H, m), 5.94–5.86 (1H, m), 5.07 (1H, ddd, *J* = 17.1, 1.8, 1.7 Hz), 4.99–4.96 (2H, m), 2.36–2.29 (1H, m), 2.27–2.21 (2H, m), 1.99–1.93 (1H, m), 1.89–1.70 (4H, m), 1.33 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ

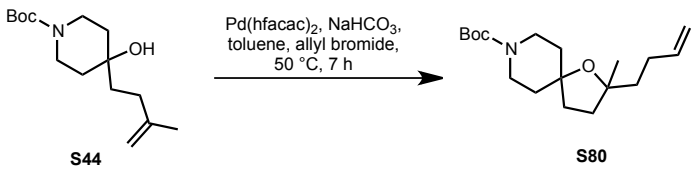
143.6, 139.4, 128.4, 127.3, 126.0, 114.3, 83.3, 80.2, 37.6, 35.6, 29.3, 26.3; IR (thin film) 2967, 1045; HRMS (EI) exact mass calculated for $C_{15}H_{20}O$ $[M]^+$ *m/z* 216.1514, found *m/z* 216.1510.



2-(but-3-enyl)-5-(4-(trifluoromethyl)phenyl)-tetrahydro-2-methylfuran S78 and S79. The general procedure was employed for the heterocyclisation of 1-(4-(trifluoromethyl)phenyl)-4-methylpent-4-en-1-ol (**S42**) (81 mg, 0.33 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 23 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 1:1) afforded a 1.5:1 mixture of diastereomers (**S78** and **S79**) (71 mg, 76%), determined by nOe as shown above.. A sample of each diastereomer was separated by flash chromatography (petroleum ether:CH_2Cl_2, 3:1) for analytical purposes.

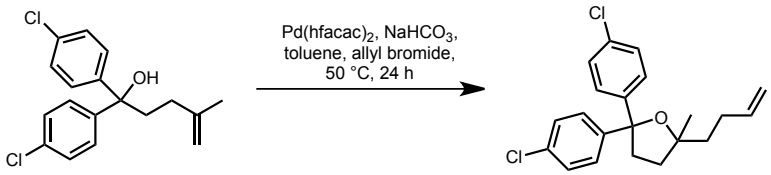
S78: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (2H, d, *J* = 8.2 Hz), 7.46 (2H, d, *J* = 8.4 Hz), 5.92–5.84 (1H, m), 5.05 (1H, ddd, *J* = 17.1, 1.8, 1.7 Hz), 5.00–4.94 (2H, m), 2.39–2.32 (1H, m), 2.25–2.14 (2H, m), 2.00–1.94 (1H, m), 1.90–1.81 (2H, m), 1.79–1.68 (2H, m), 1.35 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.2, 139.1, 129.6 (q, ^2J (C-F) = 32 Hz), 126.1, 125.4 (q, ^3J (C-F) = 3.7 Hz), 124.5 (q, ^1J (C-F) = 272 Hz), 114.3, 84.0, 80.3, 40.8, 37.7, 35.6, 29.1, 27.0; IR (thin film) 1323, 1121, 1065; HRMS (CI) exact mass calculated for $C_{16}H_{20}OF_3$ $[M+H]^+$ *m/z* 285.1466, found *m/z* 285.1460.

S79: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (2H, d, *J* = 8.2 Hz), 7.47 (2H, d, *J* = 8.1 Hz), 5.93–5.85 (1H, m), 5.06 (1H, ddd, *J* = 17.2, 1.8, 1.6 Hz), 5.03 (1H, t, *J* = 7.0 Hz), 4.99–4.96 (1H, m),. 2.42–2.35 (1H, m), 2.29–2.16 (2H, m), 1.98–1.92 (1H, m), 1.86–1.80 (2H, m), 1.78–1.72 (2H, m), 1.33 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.9, 139.1, 129.6 (q, ^2J (C-F) = 33 Hz), 126.1, 125.4 (q, ^3J (C-F) = 3.8 Hz), 124.5 (q, ^1J (C-F) = 272 Hz), 114.4, 83.9, 79.4, 41.4, 37.5, 35.5, 29.3, 26.3; IR (thin film) 1323, 1121, 1065; HRMS (CI) exact mass calculated for $C_{16}H_{20}OF_3$ $[M+H]^+$ *m/z* 285.1466, found *m/z* 285.1467.



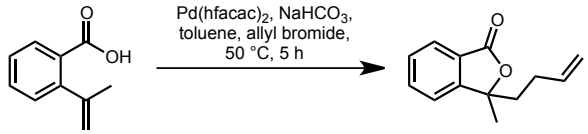
S44 **S80**

***tert*-butyl-2-(but-3-en-1-yl)-2-methyl-1-oxa-8-azaspiro[4.5]decane-8-carboxylate (S80).** The general procedure was employed for the heterocyclisation of *tert*-butyl-4-hydroxy-4-(3-methylbut-3-en-1-yl)piperidine-1-carboxylate (**S44**) (85 mg, 0.31 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 7 h. Purification of the reaction mixture by flash chromatography (CH_2Cl_2 then CH_2Cl_2:EtOAc, 9:1) afforded the title compound (**S80**) as a colourless oil (70 mg, 73%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.88–5.79 (1H, m), 5.01 (1H, ddd, *J* = 17.1, 1.8, 1.6 Hz), 4.94–4.92 (1H, m), 3.46–3.38 (4H, m), 2.15–2.05 (2H, m), 1.89–1.71 (4H, m), 1.60–1.48 (6H, m), 1.45 (9H, s), 1.19 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.1, 139.3, 114.2, 83.0, 80.4, 79.4, 41.8, 41.5, 38.6, 38.1, 36.4, 36.3, 29.3, 28.6, 27.7; IR (thin film) 1692, 1418, 1364, 1244, 1171, 1142. HRMS (EI) exact mass calculated for $C_{18}H_{31}O_3N$ $[M]^+$ *m/z* 309.2304, found *m/z* 309.2308.



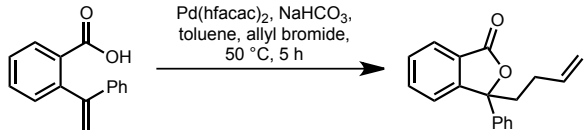
S46 **S81**

2-(but-3-enyl)-5,5-bis(4-chlorophenyl)-tetrahydro-2-methylfuran S81. The general procedure was employed for the heterocyclisation of 1,1-bis(4-chlorophenyl)-4-methylpent-4-en-1-ol (**S46**) (50 mg, 0.16 mmol) with allyl bromide (0.075 mL, 0.80 mmol) over 24 h. Purification of the reaction mixture by flash chromatography (petroleum ether:CH_2Cl_2, 17:3) afforded the title compound (**S81**) as a colourless oil (42 mg, 72%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.34 (4H, m), 7.25–7.23 (4H, m), 5.85 (1H, m), 5.00 (1H, ddd, *J* = 17.1, 1.7, 1.6), 4.94–4.92 (1H, m), 2.65–2.56 (2H, m), 2.24–2.17 (1H, m), 2.13–2.05 (1H, m), 1.88–1.76 (2H, m), 1.69–1.63 (1H, m), 1.61–1.54 (1H, m), 1.25 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.4, 146.1, 139.0, 132.60, 132.57, 128.34, 128.33, 127.30, 127.29, 114.4, 87.2, 84.8, 41.6, 38.8, 37.6, 29.4, 26.6; IR (thin film) 1489, 1090, 1011; HRMS (CI) exact mass calculated for $C_{21}H_{23}OCl_2$ $[M+H]^+$ *m/z* 361.1126, found *m/z* 361.1118.



S20 **S82**

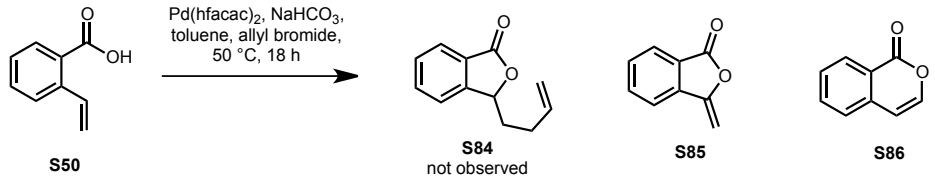
3-(but-3-enyl)-3-methylisobenzofuran-1(3H)-one S82. The general procedure was employed for the heterocyclisation of 2-(prop-1-en-2-yl) benzoic acid (**S20**) (54 mg, 0.33 mmol) with allyl bromide (0.14 mL, 1.6 mmol) over 5 h. Purification of the reaction mixture by flash chromatography (petroleum ether:EtOAc, 9:1) afforded the title compound (**S82**) as a colourless oil (51 mg, 85%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 (1H, dt, *J* = 7.6, 0.9 Hz), 7.66 (1H, td, *J* = 7.5, 1.1 Hz), 7.50 (1H, td, *J* = 7.5, 0.8 Hz), 7.36 (1H, d, *J* = 7.7 Hz), 5.74–5.66 (1H, m), 4.94–4.89 (2H, m), 2.15 (1H, ddd, *J* = 13.6, 11.2, 4.8), 2.10–2.02 (1H, m), 1.96 (1H, ddd, *J* = 13.7, 11.0, 4.6 Hz), 1.80–1.73 (1H, m), 1.65 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 153.9, 137.3, 134.2, 129.1, 126.5, 126.0, 121.0, 115.2, 87.3, 39.3, 28.0, 26.3; IR (thin film) 1751, 1285, 1030; HRMS (EI) exact mass calculated for $C_{13}H_{14}O_2$ $[M]^+$ *m/z* 202.0994, found *m/z* 202.0991.



S48 **S83**

3-(but-3-enyl)-3-phenylisobenzofuran-1(3H)-one S83. A 4 mL screw-top glass vial was charged with 2-(1-phenylvinyl)benzoic acid (**S48**) (90 mg, 0.40 mmol), toluene (1.3 mL), $NaHCO_3$ (67 mg, 0.80 mmol), allyl bromide (0.17 mL, 2.0 mmol) and $Pd(hfacac)_2$ (10 mg, 0.02 mmol). The mixture was heated at 50 °C for 5 h then additional $Pd(hfacac)_2$ (10 mg, 0.02 mmol) added. The reaction was heated 50 °C for a further 19 h. The reaction mixture was cooled to room temperature, and subjected directly to flash chromatography (petroleum ether:EtOAc, 9:1) to afford the title compound (**S83**) as a colourless oil (74 mg, 70 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (1H, d, *J* = 7.6 Hz), 7.63−7.57 (1H, m), 7.50−7.42 (4H, m), 7.33−7.27 (2H, m), 7.26−7.21 (1H, m), 5.65 (1H, ddt, *J* = 17.2, 10.0, 6.3 Hz), 4.90−4.80 (2H, m), 2.55 (1H, ddd, *J* = 14.0, 11.6, 4.8 Hz), 2.26−2.17 (1H, m),

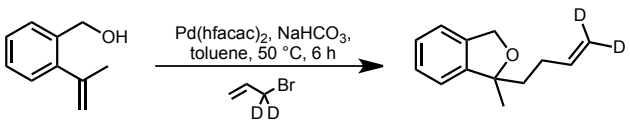
2.01−1.91 (1H, m), 1.88−1.76 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 152.7, 140.2, 136.9, 134.3, 129.1, 128.7, 128.1, 125.9, 124.9, 122.1, 115.1, 89.7, 39.3, 28.0; IR (thin film) 3055, 2918, 1760, 1259; HRMS HRMS (ESI) exact mass calculated for $C_{18}H_{16}O_2$ $[M+Na]^+$ *m/z* 287.1048, found *m/z* 287.1038.



S50 **S84** not observed **S85** **S86**

A 4 mL screw-top glass vial was charged with 2-vinylbenzoic acid (**S50**) (59 mg, 0.40 mmol), toluene (1.3 mL), $NaHCO_3$ (67 mg, 0.80 mmol), allyl bromide (0.17 mL, 2.0 mmol) and $Pd(hfacac)_2$ (10 mg, 0.02 mmol). The mixture was heated at 50 °C for 18 h. The reaction mixture was cooled to room temperature and filtered through a cotton wool plug. Analysis of the crude material revealed no formation of compound **S84** but instead gave unreacted starting material **S50**, *exo*-methylene lactone **S85**[20] and isocoumarin **S86**[21] in a ratio of 4.3: 0.9 : 1.0 respectively.

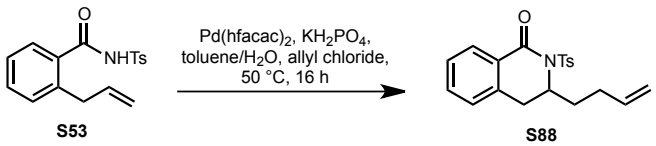
4. Deuterium labeling experiment



S21 **S87**

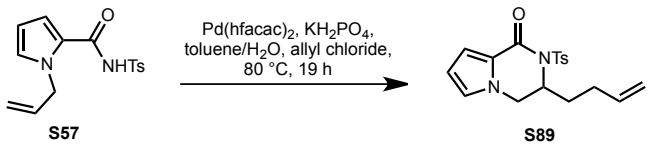
1-(3,3-*d*$_2$-*but*-3-enyl)-1,3-dihydro-1-methylisobenzofuran S87. A 4 mL glass screw-top vial was charged with (2-(prop-1-en-2-yl)phenyl) methanol (**S21**) (51 mg, 0.34 mmol)**,** freshly prepared 3-bromo-3,3-*d*$_2$-prop-1-ene[22] (0.21 mg, 1.7 mmol), toluene (1.3 mL), $NaHCO_3$ (37 mg, 0.68 mmol) and $Pd(hfacac)_2$ (8.5 mg, 0.017 mmol). The mixture was heated at 50 °C for 4 h then additional $Pd(hfacac)_2$ (8.5 mg, 0.017 mmol) added. Heating was continued for a further 2 h, then the mixture was allowed to cool to room temperature and subjected directly to flash chromatography (petroleum ether:EtOAc, 9:1) to afford the title compound (**S87**) as a yellow oil (53 mg, 82%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28–7.24 (2H, m), 7.20–7.18 (1H, m), 7.10–7.08 (1H, m), 5.80–5.73 (1H, m), 5.10 (1H, d, *J* = 12.3 Hz), 5.06 (1H, d, *J* = 12.3 Hz), 2.17–2.08 (1H, m), 1.96–1.87 (3H, m), 1.49 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.3, 139.2, 138.6, 127.5, 127.4, 121.1, 121.0, 113.6 (^{1}*J* (C-D) = 24 Hz), 88.3, 71.8, 41.0, 28.5, 27.6; IR (thin film) 1765, 1451, 1358, 1030; HRMS (CI/Isobutane) exact mass calculated for $C_{13}H_{15}D_2O$ $[M+H]^+$ *m/z* 191.1405, found *m/z* 191.1413.

5. Aminoallylation



S53 **S88**

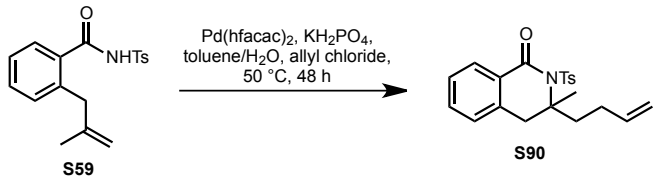
3-(but-3-en-1-yl)-2-tosyl-3,4-dihydroisoquinolin-1(2H)-one S88. A 4 mL screw-top glass vial was charged with 2-allyl-*N*-tosyl-benzamide (**S53**) (50 mg, 0.16 mmol), toluene (0.64 mL), H_2O (0.64 mL), KH_2PO_4 (44 mg, 0.32 mmol), allyl chloride (0.07 mL, 0.79 mmol) and $Pd(hfacac)_2$ (4.0 mg, 0.02 mmol). The mixture was heated at 50 °C for 16 h. The mixture was cooled to room temperature, and subjected directly to flash chromatography (petroleum ether:EtOAc, 9:1) to yield the title compound as a white powdered solid (**S88**) (53 mg, 95%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03−7.95 (3H, m) 7.50−7.46 (1H, m), 7.34−7.19 (4H, m), 5.76 (1H, ddt, *J* = 16.9 Hz, 10.3 Hz, 6.5 Hz), 5.06−4.95 (3H, m), 3.35 (1H, dd, *J* = 16.2 Hz, 5.5 Hz), 2.97 (1H, dd, *J* = 16.3 Hz, 1.8 Hz), 2.41 (3H, s), 2.17−2.10 (2H, m) 1.91−1.80 (1H, m), 1.68−1.49 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0, 144.8, 137.0, 136.9, 136.9, 133.8, 129.5, 129.1, 129.1, 128.3, 128.3, 127.6, 115.8, 55.5, 32.8, 32.4, 30.7, 21.8; IR (thin film) 2924, 1684; HRMS (CI/Isobutane) exact mass calculated for $C_{20}H_{22}NO_3S$ $[M+H]^+$ m/z 356.1320; found m/z 356.1319.



S57 **S89**

3-(but-3-en-1-yl)-2-tosyl-3,4-dihydropyrrolo[1,2-a]pyrazin-1(2H)-one S89. A 4 mL glass screw-top vial was charged with 1-allyl-*N*-tosyl-pyrrole-2-carboxamide (**S57**) (48 mg, 0.16 mmol), toluene (0.64 mL), H_2O (0.64 mL), KH_2PO_4 (44 mg, 0.32 mmol), allyl chloride (0.07 mL, 0.79 mmol) and $Pd(hfacac)_2$ (8.0 mg, 0.01 mmol). The mixture was heated at 80 °C for 19 h. The mixture was cooled to room temperature, and subjected directly to column chromatography (petroleum ether:EtOAc, 90:10) to yield the title compound (**S89**) as a white powdered solid (33 mg, 0.10 mmol, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (2H, d, *J* = 8.4 Hz), 7.31 (2H, d, *J* = 8.1 Hz), 6.97 (1H, dd,

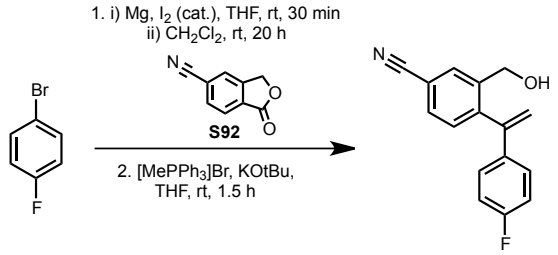
J = 4.0 Hz, 1.5 Hz), 6.76 (1H, dd, 2.2, 1.7 Hz), 6.23 (1H, dd, *J* = 4.0 Hz, 2.5 Hz), 5.80 (1H, ddt, *J* = 10.3, 6.5, 3.5 Hz), 5.09–5.03 (2H, m), 4.98–4.93 (1H, m), 4.29 (1H, dd, *J* = 13.2, 3.9 Hz), 4.15 (1H, dd, 13.2, 1.5 Hz), 2.42 (3H, s), 2.17 (2H, m), 1.84 (1H, m), 1.68 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 144.8, 136.6, 136.5, 129.4, 129.0, 125.1, 122.5, 116.8, 116.2, 111.0, 55.6, 47.0, 31.5, 30.2, 21.7; IR (thin film) 2359, 1670, 1161; HRMS (EI) exact mass calculated for $C_{18}H_{20}N_2O_3S$ $[M]^+$ m/z 344.1197; found m/z 344.1195.



S59 **S90**

3-(but-3-en-1-yl)-3-methyl-2-tosyl-3,4-dihydroisoquinolin-1(2H)-one 90. A 4 mL screw-top glass vial was charged with 2-(2-methyl)-allyl-*N*-tosyl-benzamide (**S59**) (52 mg, 0.16 mmol), toluene (0.64 mL), H_2O (0.64 mL), KH_2PO_4 (44 mg, 0.32 mmol), allyl chloride (0.07 mL, 0.79 mmol) and $Pd(hfacac)_2$ (8.0 mg, 0.01 mmol). The mixture was heated at 50 °C for 48 h. The mixture was cooled to room temperature, and subjected directly to flash chromatography (1:1 petroleum ether/CH_2Cl_2) to yield the title compound as a white powdered solid (**S90**) (17 mg, 33%). Attempts to increase yield by variation of concentration, temperature and time were unsuccessful. ^{1}H NMR (500 MHz, CDCl3) δ 7.95 (2H, d, J = 8.4 Hz), 7.80 (1H, dd, J = 7.8, 0.9 Hz), 7.56–7.36 (2H, m), 7.31–7.13 (3H, m), 5.74 (1H, ddt, J = 17.0, 10.3, 6.4 Hz), 5.00 (1H, dd, J = 17.1, 1.7 Hz), 4.95 (1H, dd, J = 10.1, 1.6 Hz), 3.07 (2H, s), 2.42 (3H, s), 2.23–2.10 (4H, m), 1.93 (3H, s); ^{13}C NMR (100 MHz, CDCl3) δ 157.4, 145.2, 137.1, 136.5, 129.6, 129.2, 128.6, 123.3, 121.4, 116.4, 109.8, 109.4, 56.0, 43.5, 32.1, 30.5, 21.8; IR (thin film) 3394, 1683, 1348; HRMS (CI/Isobutane) exact mass calculated for $C_{21}H_{24}NO_3S$ $[M+H]^+$ m/z; found m/z 370.1475.

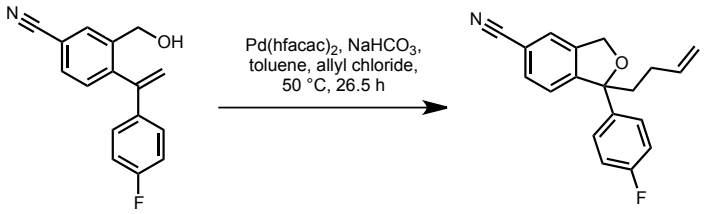
6. Synthesis of Citalopram



S91 **S93**

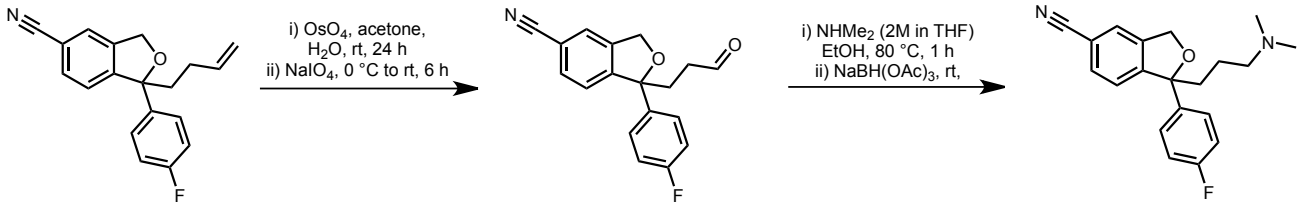
4-(1-(4-fluorophenyl)vinyl)-3-(hydroxymethyl)benzonitrile S93. Following a modification of a reported procedure,[23] to mixture of magnesium turnings (200 mg, 8.2 mmol) and a crystal of iodine in THF (7 mL) was added 1-bromo-4-fluorobenzene (**S91**) (0.76 mL, 6.9 mmol). The mixture slowly warmed and refluxed gently at the neck of the flask. After 30 minutes the mixture had cooled to room temperature and was added by syringe to a cooled (0 °C) suspension of 5-cyanophthalide (**S92**) (1.0 g, 6.3 mmol) in CH_2Cl_2 (10 mL). The cooling bath was removed and the mixture allowed to warm to room temperature then stirred for 20 h. The reaction mixture was quenched with sat. aq. NH_4Cl (25 mL) and extracted with Et_2O (2 x 25 mL). The combined organics were washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. The residue was passed through a silica plug (petroleum ether:EtOAc, 7:3) to afford a yellow oil. The oil was dissolved in THF (11 mL) and added dropwise to a suspension of methyl triphenylphosphonium bromide (4.4 g, 12 mmol) and potassium *tert*-butoxide (1.4 g, 12 mmol) in THF (44 mL). The mixture was stirred at room temperature for 1.5 h then sat. aq. NH_4Cl (25 mL) added and the mixture extracted with Et_2O (50 mL). The organic extracts were washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated in vacuo. Purification by flash chromatography afforded the title compound (**S93**) as a yellow oil (757 mg, 47% over two steps). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (1H, d), 7.61 (1H, dd, *J* = 7.8, 1.6 Hz), 7.33 (1H, d, *J* = 7.8 Hz), 7.20–7.16 (2H, m), 7.02–6.98 (2H, m), 5.79 (1H, s), 5.23 (1H, s), 4.45 (2H, d, *J* = 5.8 Hz), 1.78 (1H, t, *J* = 5.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.9 (d, ^1J (C-F) = 249 Hz), 145.9, 144.6, 140.5, 135.5 (d, ^4J (C-F) = 4 Hz), 131.14, 131.12, 130.8, 128.3 (d, ^3J (C-F) = 8 Hz, 118.9, 116.6, 115.9 (d, ^2J (C-F) = 22 Hz), 112.1, 62.0; IR (thin film) 3439, 2231, 1601,

1507, 1224, 1160; HRMS (EI) exact mass calculated for $C_{16}H_{12}ONF$ $[M]^+$ *m/z* 253.0903, found *m/z* 253.0900.



S93 **S94**

1-(but-3-enyl)-1-(4-fluorophenyl)-1,3-dihydroisobenzofuran-5-carbonitrile S94. A 4 mL glass screw-top vial was charged with of 4-(1-(4-fluorophenyl)vinyl)-3-(hydroxymethyl)benzonitrile (**S93**) (147 mg, 0.58 mmol)**,** allyl chloride (0.22 mL, 2.7 mmol), toluene (2.2 mL), $NaHCO_3$ (92 mg, 1.1 mmol) and $Pd(hfacac)_2$ (14 mg, 0.027 mmol). The mixture was heated at 50 °C for 8.5 h then additional $Pd(hfacac)_2$ (14 mg, 0.027 mmol) added. Heating was continued for a further 18 h then the mixture allowed to cool to room temperature and subjected directly to flash chromatography (petroleum ether:CH_2Cl_2, 1:1) to afford the title compound (**S94**) as a yellow oil (98 mg, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (1H, d, *J* = 7.8 Hz), 7.50 (1H, s), 7.44–7.41 (2H, m), 7.39 (1H, d, *J* = 7.9 Hz), 7.03–7.00 (2H, m), 5.81–5.73 (1H, m), 5.20 (1H, d, *J* = 13.0 Hz), 5.15 (1H, d, *J* = 13.0 Hz), 4.96–4.90 (2H, m), 2.32–2.26 (1H, m), 2.22–2.16 (1H, m), 2.09–2.01 (1H, m), 1.95–1.88 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.2 (d, ^1J (C-F) = 247 Hz), 149.3, 140.5, 139.6 (d, ^4J (C-F) = 3 Hz), 137.9, 132.0, 126.9 (d, ^3J (C-F) = 8 Hz), 125.3, 122.9, 118.7, 115.5 (d, ^2J (C-F) = 21 Hz), 114.8, 111.9, 91.1, 71.4, 40.6, 28.4; IR (thin film) 2231, 1506, 1479, 1225, 1159; HRMS (CI) exact mass calculated for $C_{19}H_{17}ONF$ $[M+H]^+$ *m/z* 294.1294, found *m/z* 294.1288.



S94 **S95** **S96**

Citalopram S96. To a solution of 1-(but-3-enyl)-1-(4-fluorophenyl)-1,3-dihydroisobenzofuran-5-carbonitrile (**S94**) (170 mg, 0.57 mmol) in acetone (19 mL) and water (10 mL) was added NMO (330 mg, 2.8 mmol) then OsO_4 (4% wt. in H_2O, 0.18 mL, 0.03 mmol). The resulting mixture was stirred at room temperature for 23 h then cooled to 0 °C and $NaIO_4$ (290 mg, 1.4 mmol) added. The reaction mixture was allowed to warm to room temperature and stirred for 2 h then quenched with sat. aq. sodium sulfite (25 mL) and extracted with EtOAc (3 x 25 mL). The combined organic extracts were washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo* to afford a yellow oil (**S95**). The oil was dissolved in EtOH (5.7 mL) and dimethylamine (2M in THF, 0.86 mL, 1.7 mmol) added. The resulting mixture was heated to reflux for 1 h then cooled to room temperature. $NaBH(OAc)_3$ (160 mg, 0.74 mmol) was then added in one portion and the mixture stirred at room temperature for 18 h. The reaction mixture was quenched with 1M aq. HCl (5 mL) and partitioned between Et_2O (25 mL) and sat. aq. $NaHCO_3$ (25 mL). The aqueous phase was further extracted with Et_2O (2 x 25 mL) and the combined organic extracts washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (CH_2Cl_2:MeOH:NEt_3, 95:4:1) afforded the title compound (**S96**) as a colourless oil (114 mg, 62% over two steps). Analytical data observed were in accordance with literature values.[24]

6. References

[1] S. Nicolai, S. Erard, D. Fernández González, J. Waser, *Org. Lett.* 2010, **12**, 384–387.

[2] E. Alvarez, T. Cuvigny, M. Julia, *J. Organomet. Chem.* 1988, **339**, 199–212.

[3] M. K. Kyoung, R. K. Hyoung, K. R. Eung, *Heterocycles* 1993, **36**, 497–505.

[4] D. S. Tarbell, *Org. React. (N.Y.)*, 1944, **2**, 22.

[5] F.-N. Li, N.-J. Kim, S.-M. Paek, D.-Y. Kwon, H.-G. Park, Y.-G. Suh, K. H. Min, Y.-D. Jeong, S.-Y. Kim, Y.-H. Park, H.-D. Kim, *Bioorg. Med. Chem.* 2009, **17**, 3557–3567.

[6] M. A. Brimble, C. L. Flowers, M. Trzoss, K. Y. Tsang, *Tetrahedron*, 2006, **62**, 5883–5896.

[7] R. Trivedi, J. A. Tunge, *Org. Lett.* 2009, **11**, 5650–5652.

[8] M. Jiménez-Estrada, A. Navarro, M. V. Flores, R. R. Chilpa, B. Hernández, A. L. Anaya, B. Lotina-Hennsen, *J. Agric. Food. Chem.* 1996, **44**, 2839–2841.

[9] J. Kim, H. B. Li, A. S. Rosenthal, D. Sang, T. A. Shapiro, D. M. Bachi, G. H. Posner, *Tetrahedron* 2006, **62**, 4120–4127.

[10] M. Mercola, M. Dawson, J. Cashman, WO Patent 2010/33643, 2010.

[11] S. Nicolai, C. Piemontesi, J. Waser, *Angew. Chem. Int. Ed.* 2011, **50**, 4680–4683.

[12] A. Fernández-Mateos, P. H. Teijón, L. M. Burón, R. R. Clemente, R. R. González, *J. Org. Chem.* 2007, **72**, 9973–9982.

[13] C.-G. Yang, N. W. Reich, Z. Shi, C. He, *Org. Lett.* 2005, **7**, 4553–4556.

[14] H. Park, Y.-L. Hong, Y. B. Kim, T.-L. Choi, *Org. Lett.* 2010, **12**, 3442–3445.

[15] C. M. Rao Volla, P. Vogel, S. R. Dubbaka, *Tetrahedron* 2009, **65**, 504–511.

[16] W. F. Berkowitz, Y. Wu, *J. Org. Chem.* 1997, **62**, 1536–1539.

[17] S. T. Le Quement, T. E. Nielsen, M. Meldal, *J. Comb. Chem.* 2007, **9**, 1060–1072.

[18] Analytical data was consistent with literature values: Y. Liu, B. Yao, C.-L. Deng, R.-Y. Tang, X.-G. Zhang, J.-H. Li, *Org. Lett.* 2011, **13**, 1126–1129.

[19] Analytical data was consistent with literature values: H. Konishi, T. Ueda, T. Muto, K. Manabe, *Org. Lett.* 2012, **14**, 4722–4725.

[20] Analytical data was consistent with literature values: H. Yamamoto, G. Pandey, Y. Asai, M. Nakano, A. Kinoshita, K. Namba, H. Imagawa, M. Nishizawa, *Org. Lett*. 2007, **9**, 4029–4032.

[21] Analytical data was consistent with literature values: R. C. Larock, S. Varaprath, H. H. Lau, C. A. Fellows, *J. Am. Chem. Soc.* 1984**, 106**, 5274–5284.

[22] I. Piel, M. Steinmetz, K. Hirano, R. Fröhlich, S. Grimme, F. Glorious, *Angew. Chem. Int. Ed.* 2011, **50**, 4983–4987.

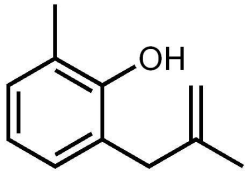
[23] M. H. Rock, H. Petersen, P. Ellegaard, WO Patent 2000/012044, 2000.

[24] B. M. Partridge, S. P. Thomas, V. K. Aggarwal, *Tetrahedron*, 2011, **67**, 10082–10088.

7. NMR Spectra of New Compounds

^{1}H and ^{13}C NMR data for all new compounds follow below.

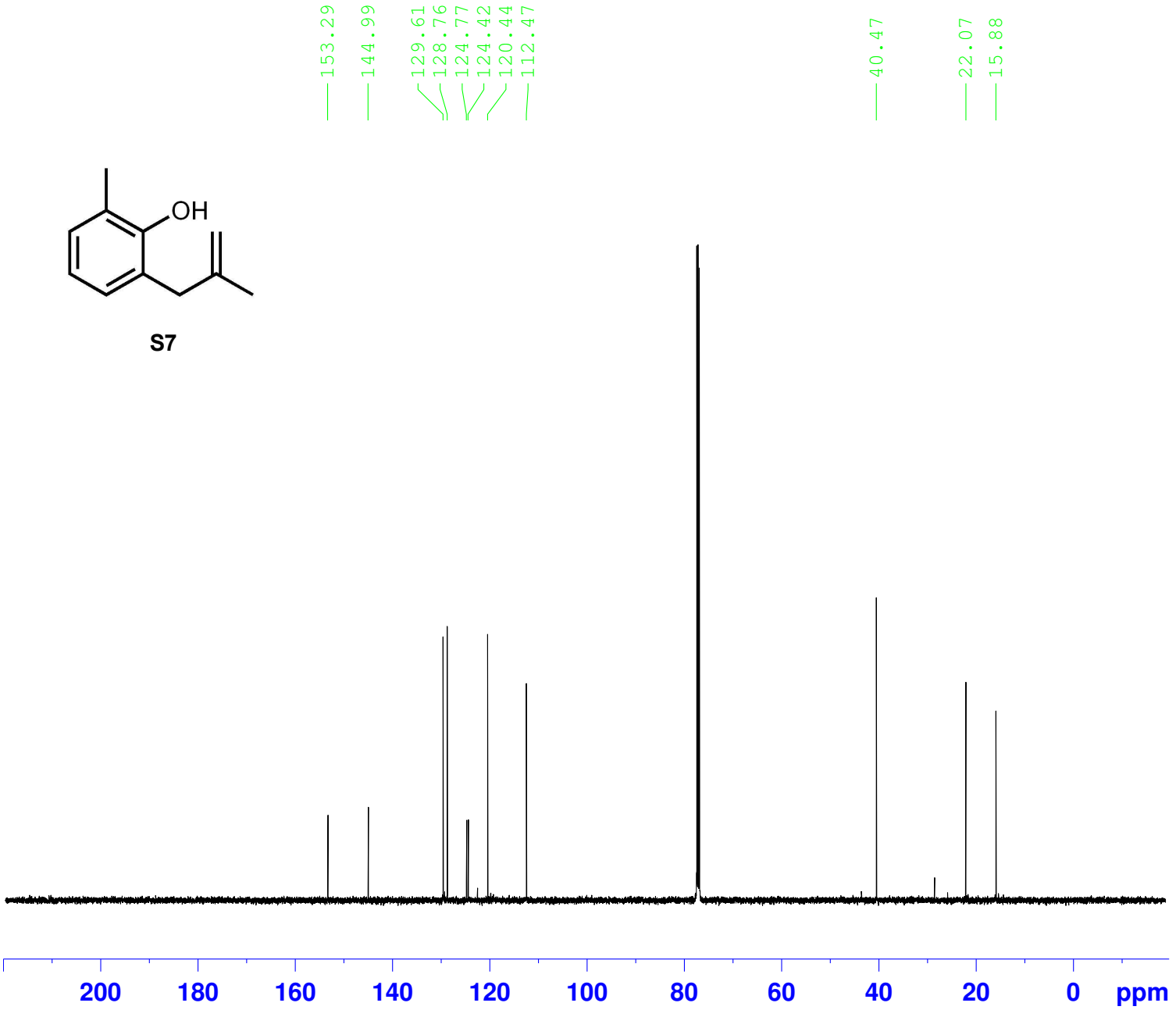
user Joanne Hewitt
JMH-VII-32B
proton.gla CDCl3 /u joahew 32



S7

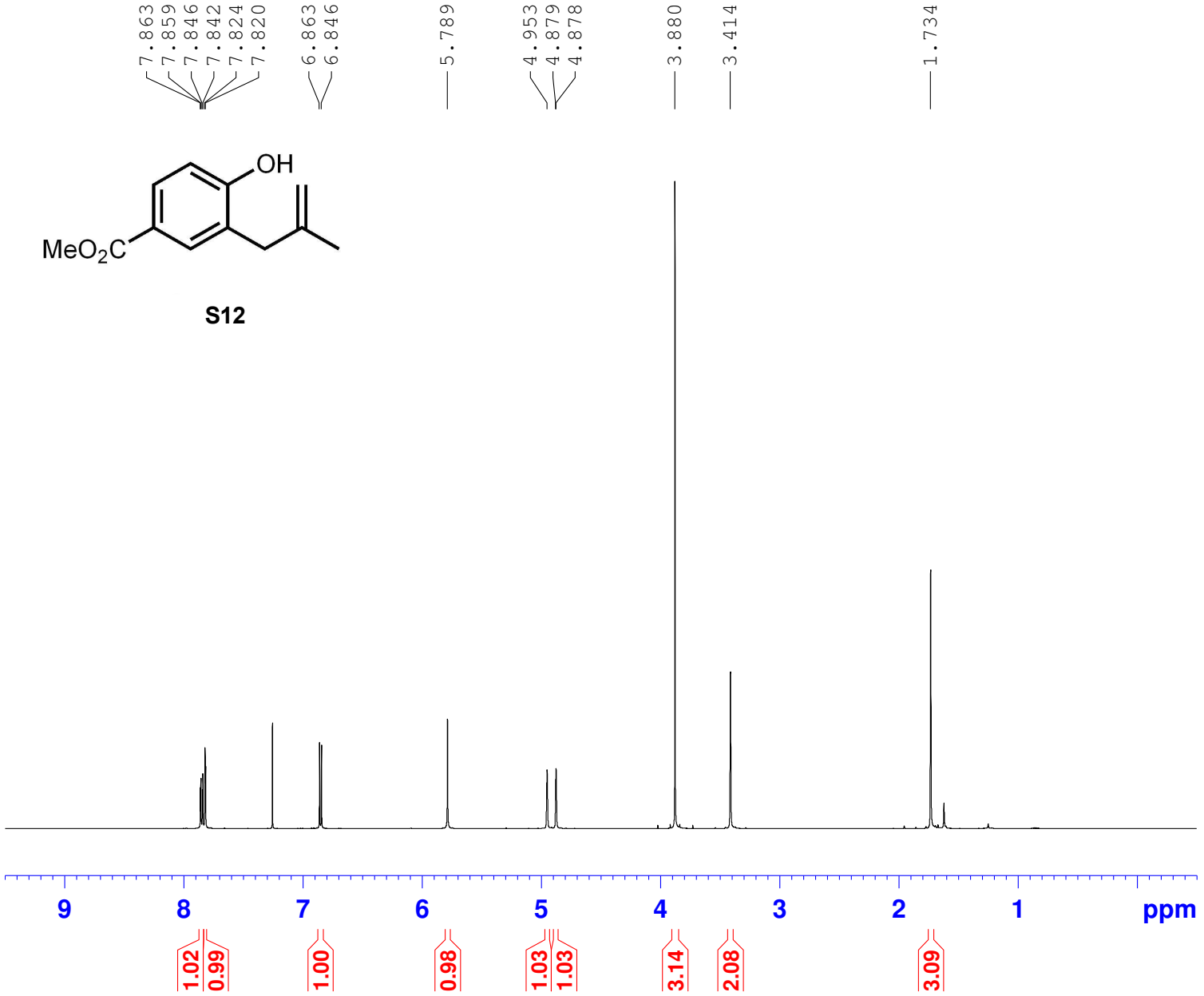
NAME	JMH-VII-32B_2
EXPNO	10
PROCNO	1
Date_	20120920
Time	6.36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	161
DW	48.667 usec
DE	7.02 usec
TE	302.5 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900106 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VII-32B
C13CPD1024.GLA CDCl3 /u joahew 32



NAME	JMH-VII-32B
EXPNO	11
PROCNO	1
Date_	20120920
Time	7.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	303.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728467 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

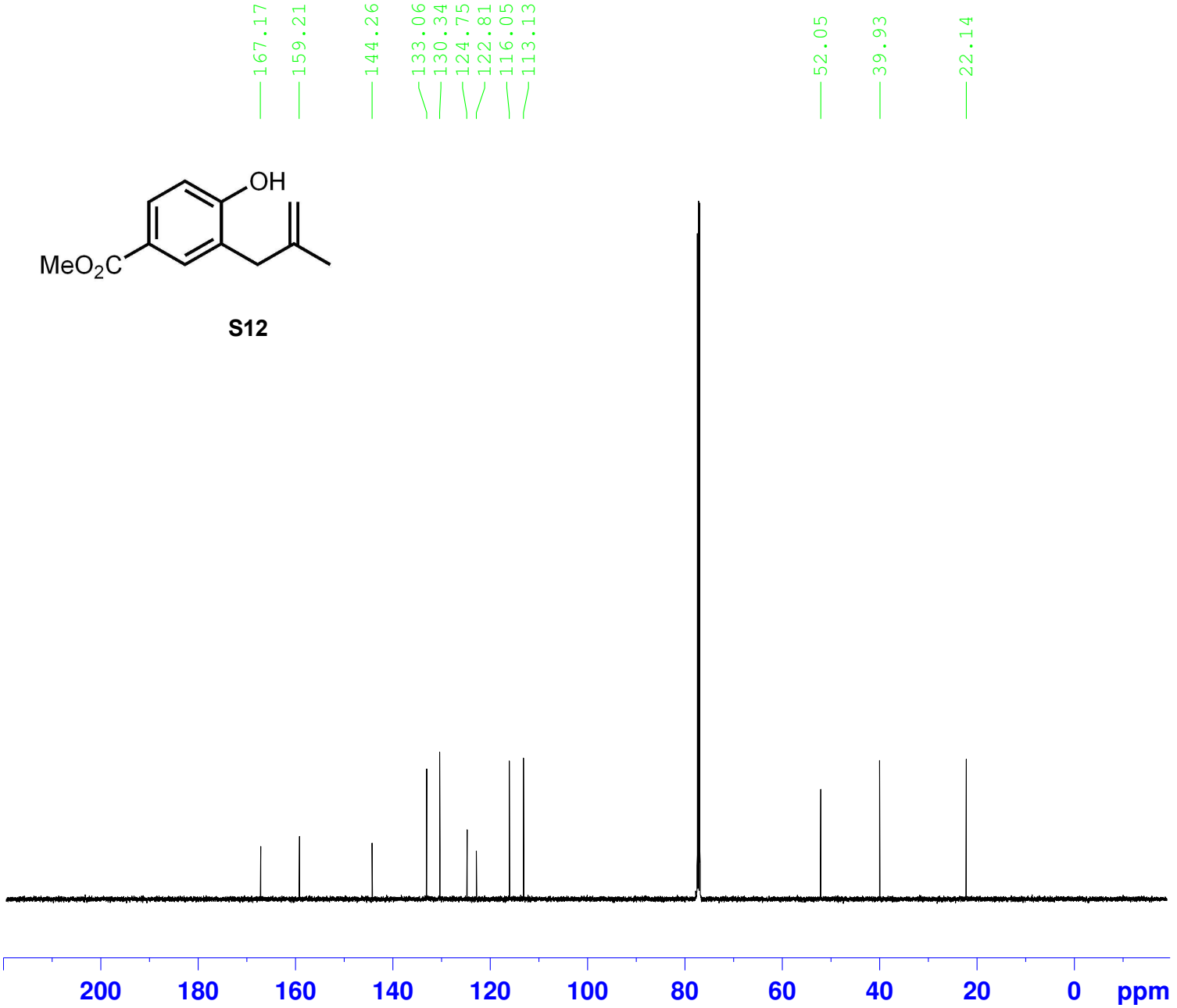
user Joanne Hewitt
JMH-VI-92B
proton.gla CDCl3 /u joahew 5



S12

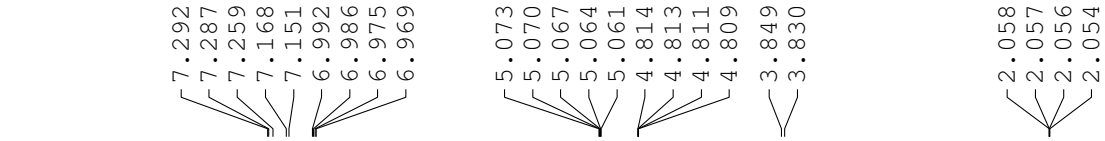
NAME	JMH-VI-92B_3
EXPNO	10
PROCNO	1
Date_	20120828
Time	11.19
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	203
DW	48.667 usec
DE	7.02 usec
TE	271.7 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900124 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

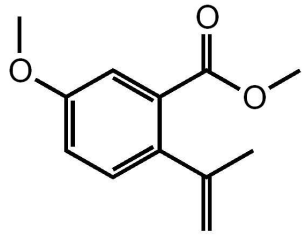
user Joanne Hewitt
JMH-VI-92B
C13CPD1024.GLA CDCl3 /u joahew 5




```
NAME          JMH-VI-92B
EXPNO                 11
PROCNO                 1
Date_           20120828
Time               21.56
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           30000.000 Hz
FIDRES         0.457764 Hz
AQ             1.0923166 sec
RG                  2050
DW                16.667 usec
DE                 8.01 usec
TE                272.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.50 usec
PL1                -2.50 dB
PL1W       147.40557861 W
SFO1        125.7854522 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.85 dB
PL13               20.00 dB
PL2W        18.75546646 W
PL12W        0.38737163 W
PL13W        0.23611732 W
SFO2        500.1920008 MHz
SI                 32768
SF          125.7728588 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

user Joanne Hewitt
JMH-VI-53B
proton.gla CDCl3 /u joahew 25





S24

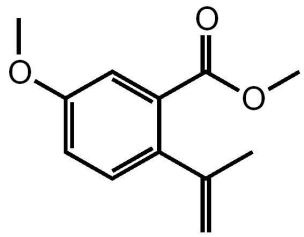
NAME	JMH-VI-53B_2
EXPNO	10
PROCNO	1
Date_	20120719
Time	7.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	203
DW	48.667 usec
DE	7.02 usec
TE	295.1 K
D1	0.50000000 sec
TD0	1

======== CHANNEL f1 ========

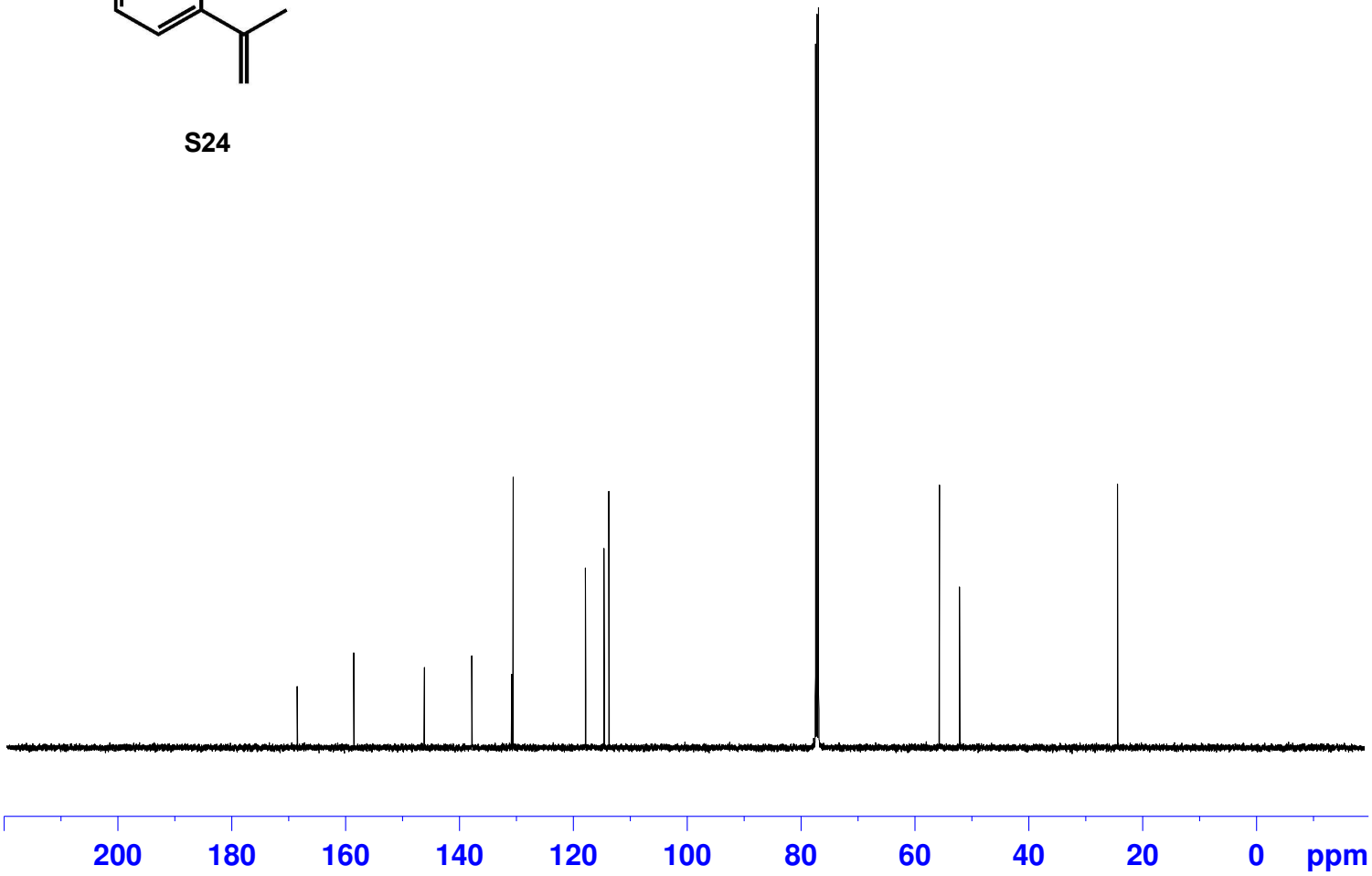
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900112 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VI-53B
C13CPD1024.GLA CDCl3 /u joahew 25

168.51 158.57 146.16 137.82 130.82 130.55 117.83 114.57 113.72 55.67 52.12 24.34

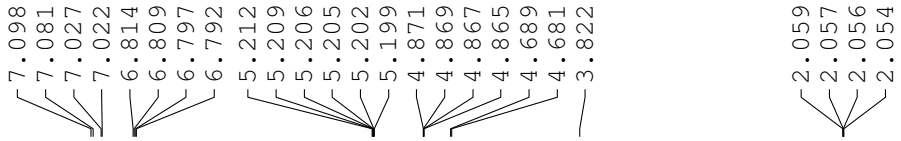


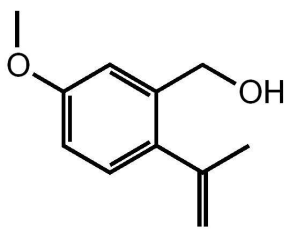
S24



NAME	JMH-VI-53B
EXPNO	11
PROCNO	1
Date_	20120720
Time	7.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728484 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-56A
proton.gla CDCl3 /u joahew 26

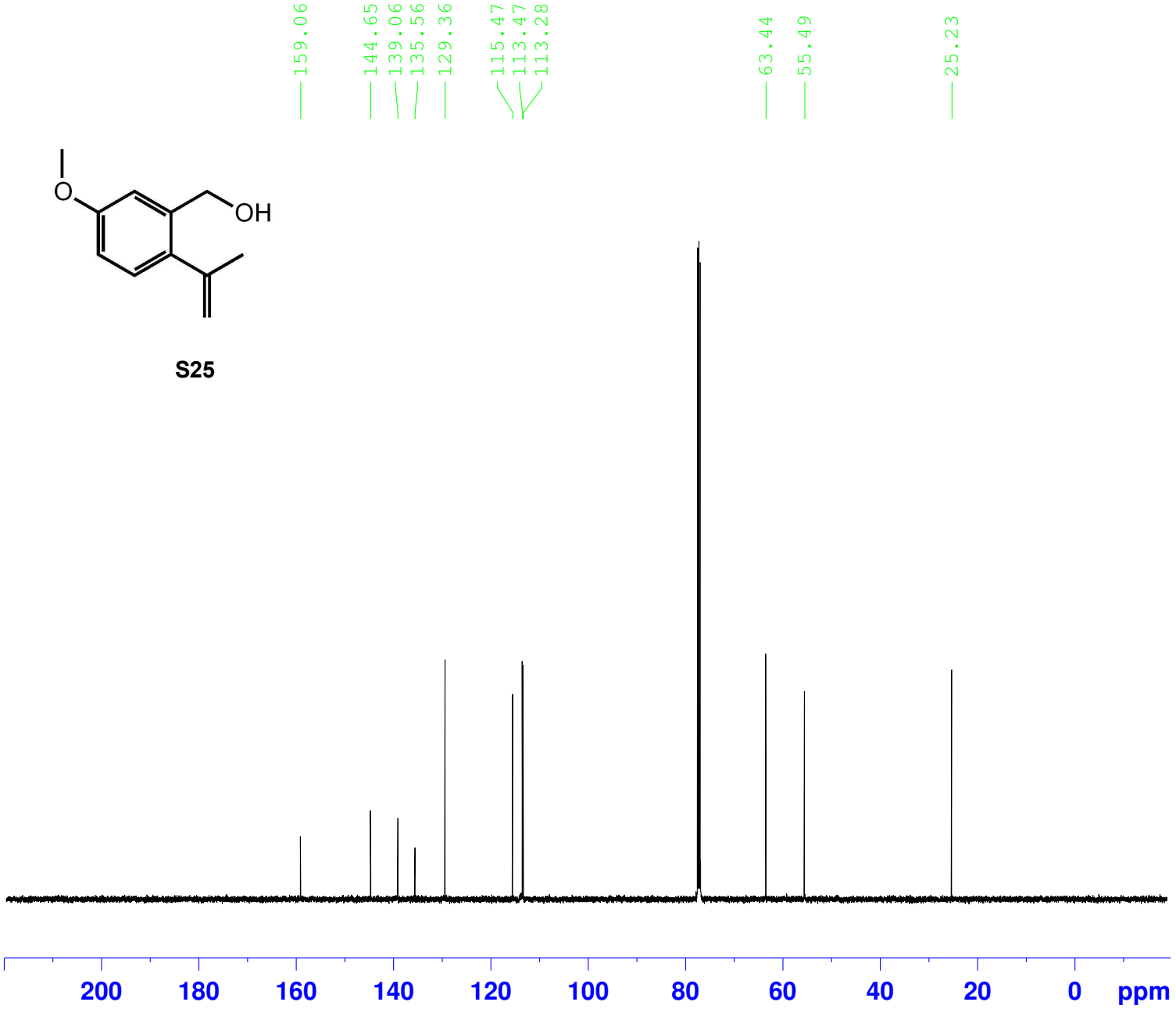




S25

NAME	JMH-VI-56A_2
EXPNO	10
PROCNO	1
Date_	20120721
Time	7.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	181
DW	48.667 usec
DE	7.02 usec
TE	295.3 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900110 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

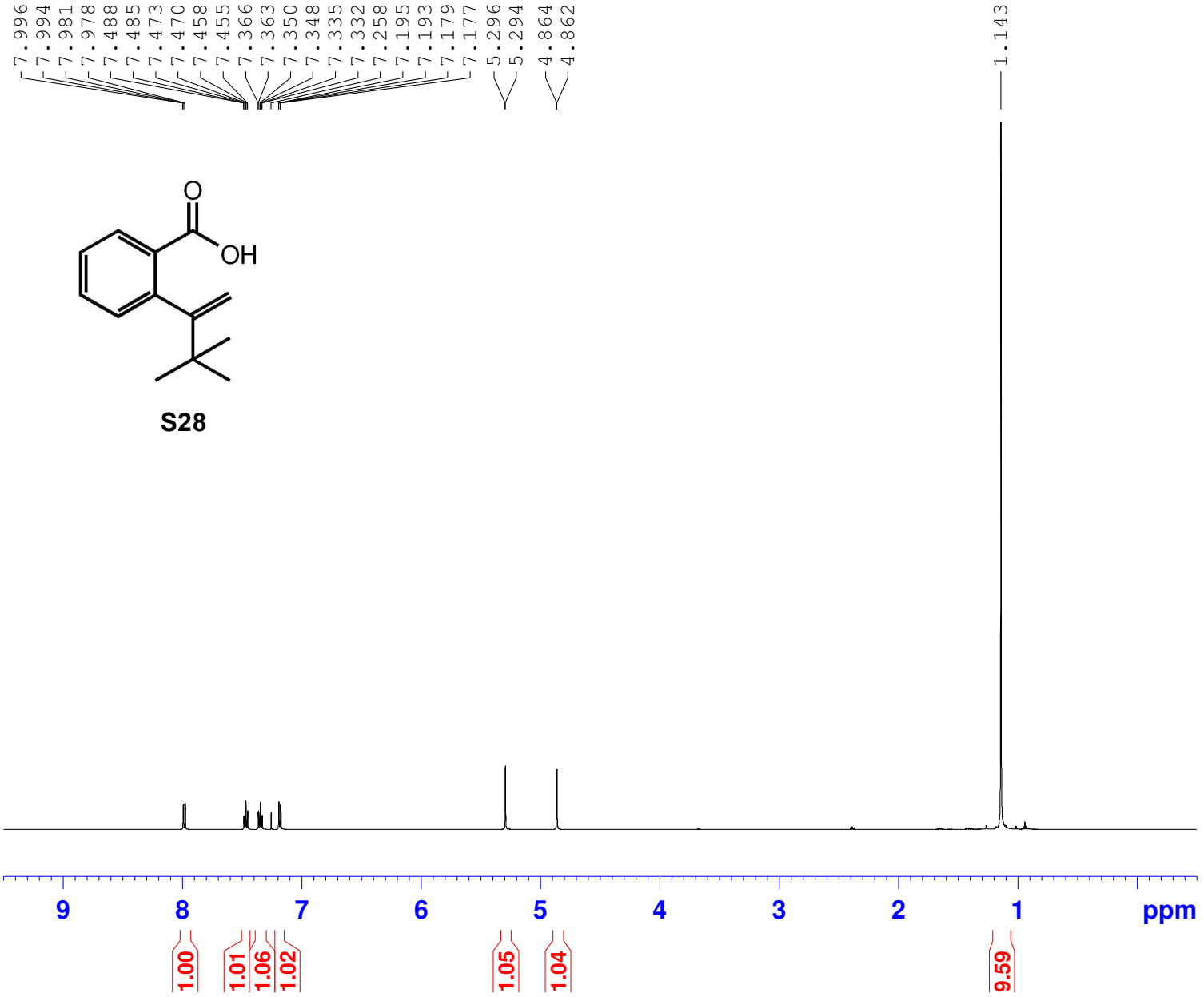
user Joanne Hewitt
JMH-VI-56A
C13CPD1024.GLA CDCl3 /u joahew 26



S25

NAME	JMH-VI-56A
EXPNO	11
PROCNO	1
Date_	20120721
Time	8.02
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728489 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

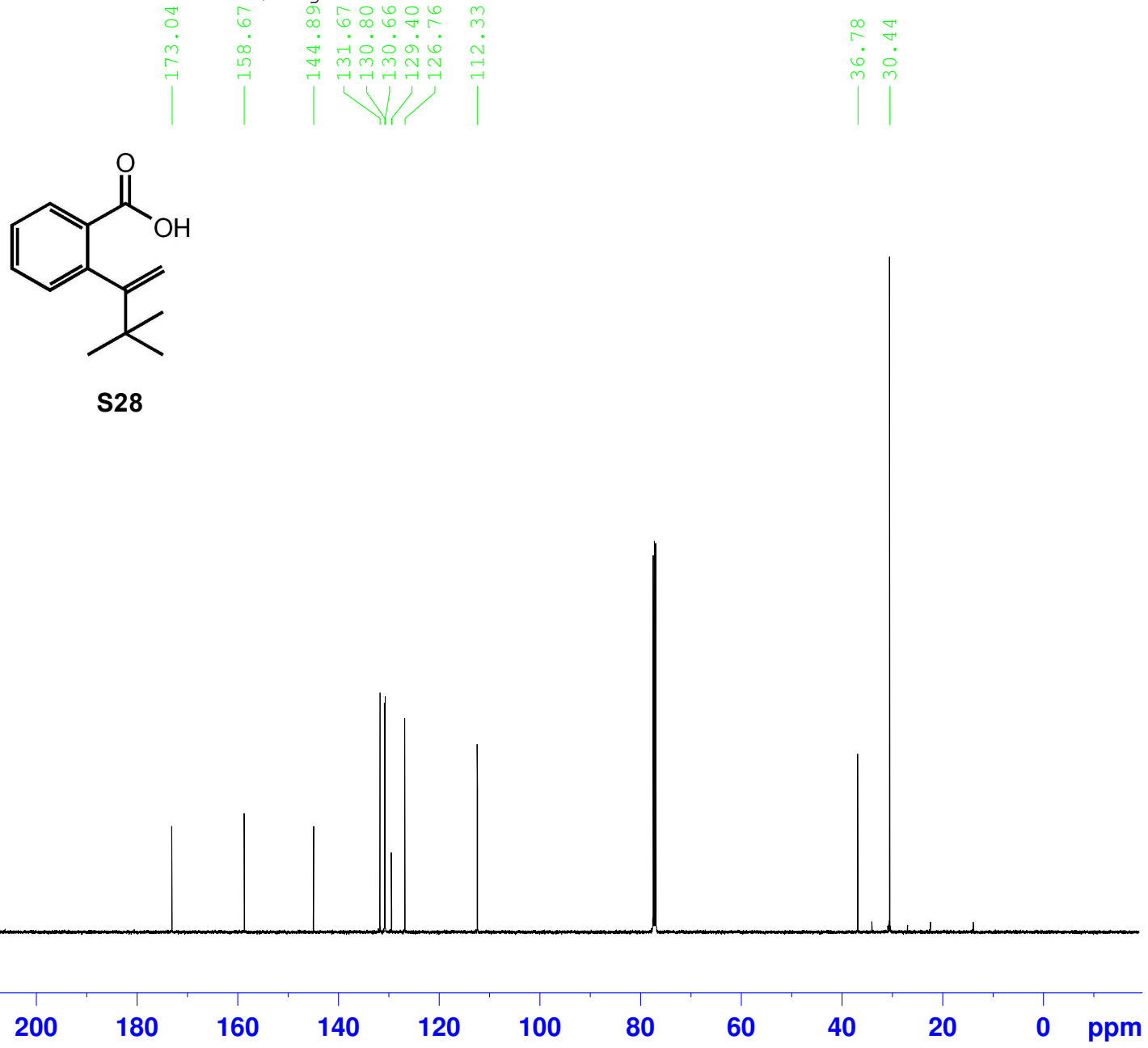
user Joanne Hewitt
JMH-VIII-51A
proton.gla CDCl3 /u joahew 26



S28

NAME	JMH-VIII-51A_2
EXPNO	10
PROCNO	1
Date_	20130116
Time	14.55
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	90.5
DW	48.667 usec
DE	7.02 usec
TE	301.2 K
D1	0.50000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900121 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

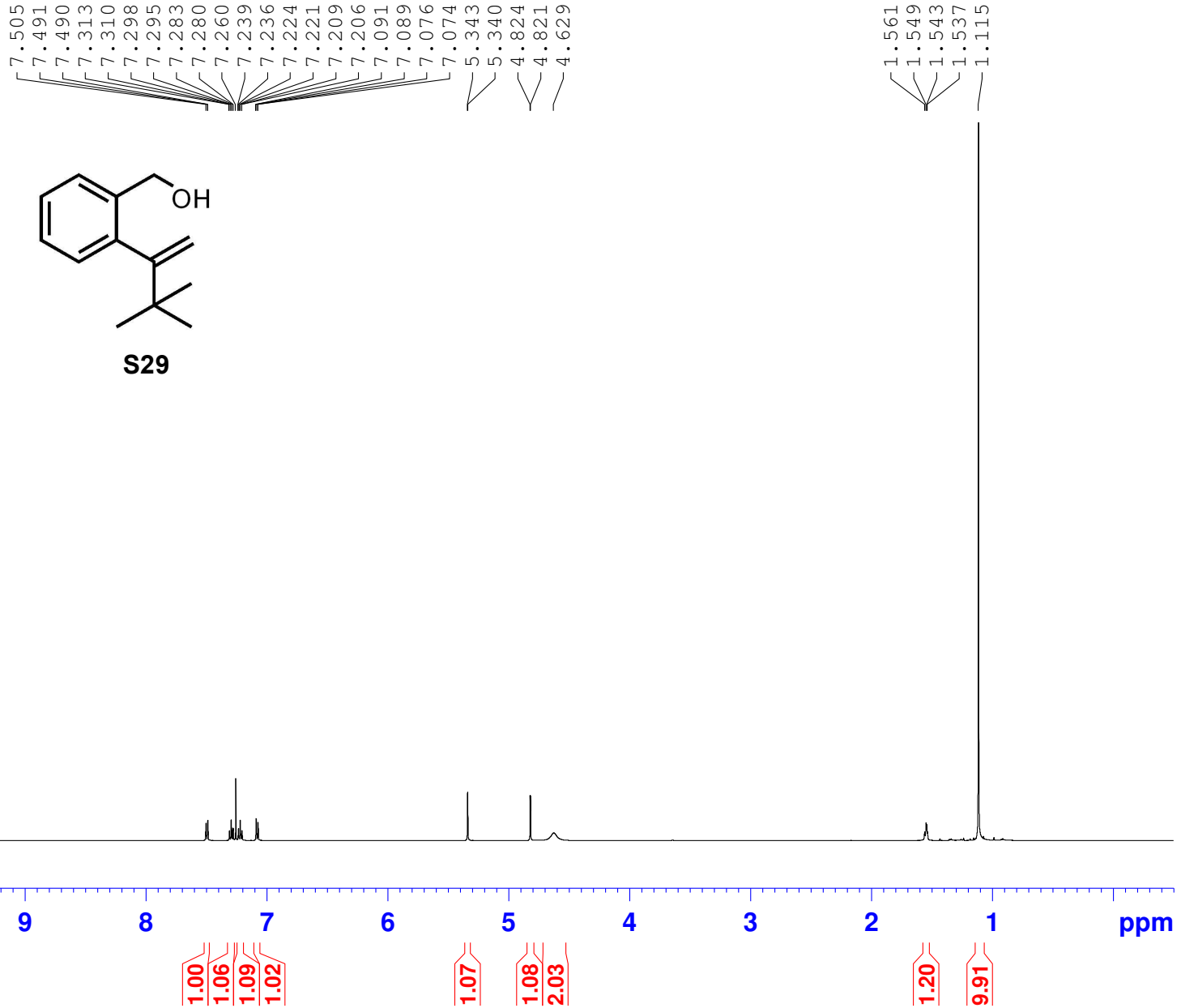
user Joanne Hewitt
JMH-VIII-51A
C13CPD1024.GLA CDCl3 /u joahew 26



S28

NAME	JMH-VIII-51A
EXPNO	20
PROCNO	1
Date_	20130117
Time	8.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	301.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728593 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

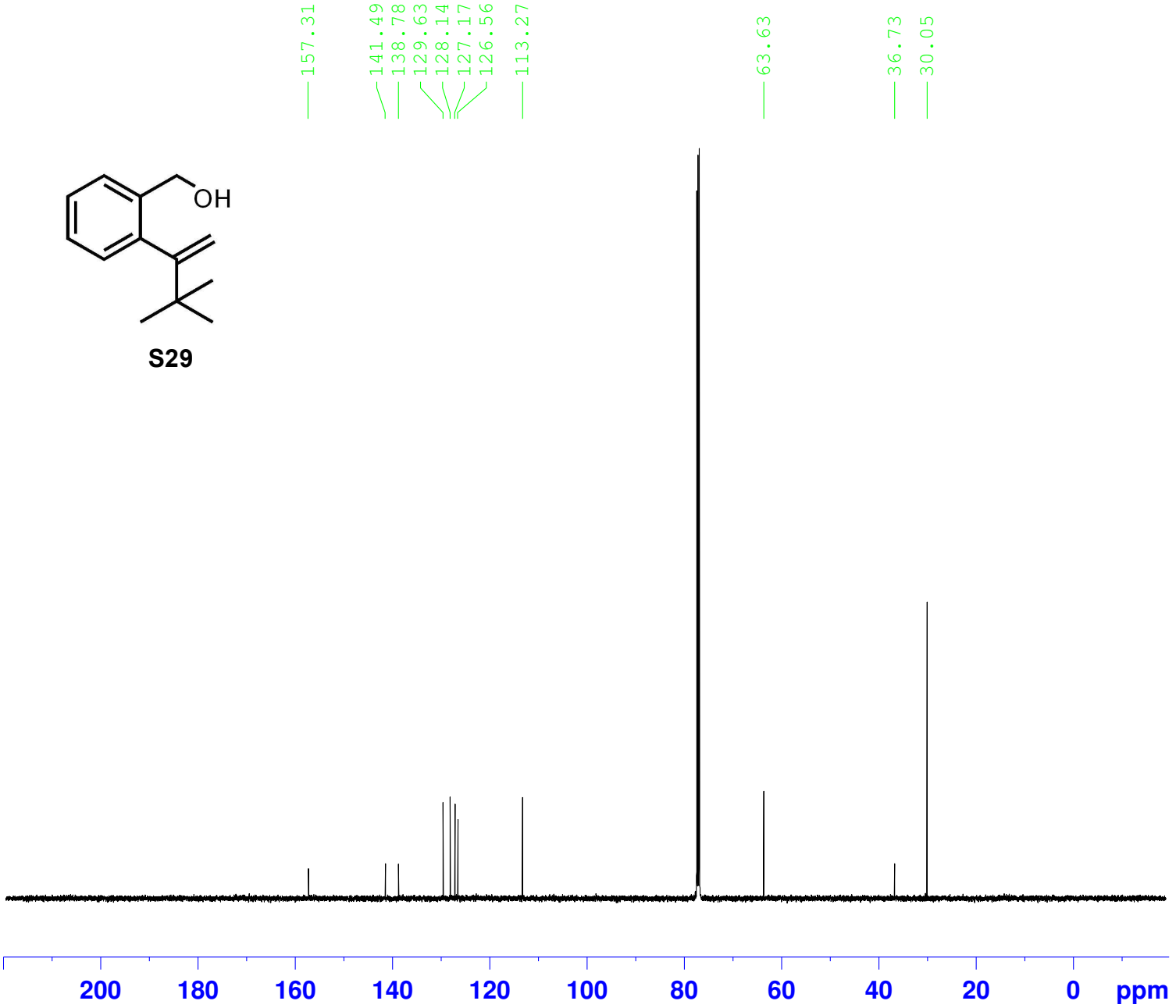
user Joanne Hewitt
JMH-VIII-55A
proton.gla CDCl3 /u joahew 3



S29

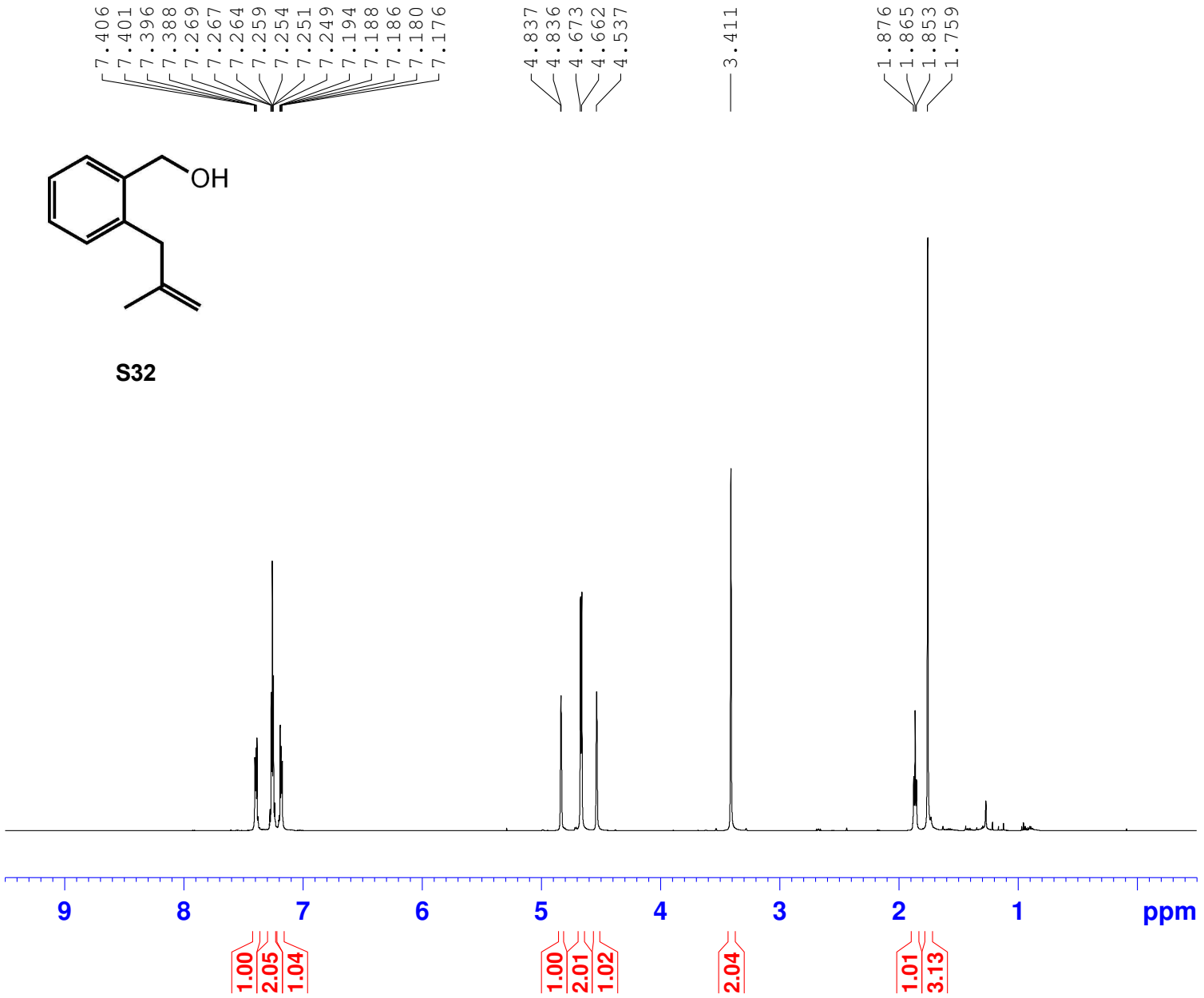
NAME	JMH-VIII-55A_2
EXPNO	10
PROCNO	1
Date_	20130117
Time	11.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	228
DW	48.667 usec
DE	7.02 usec
TE	301.0 K
D1	0.50000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VIII-55A
C13CPD1024.GLA CDCl3 /u joahew 3



NAME	JMH-VIII-55A
EXPNO	14
PROCNO	1
Date_	20130117
Time	13.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	301.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728564 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

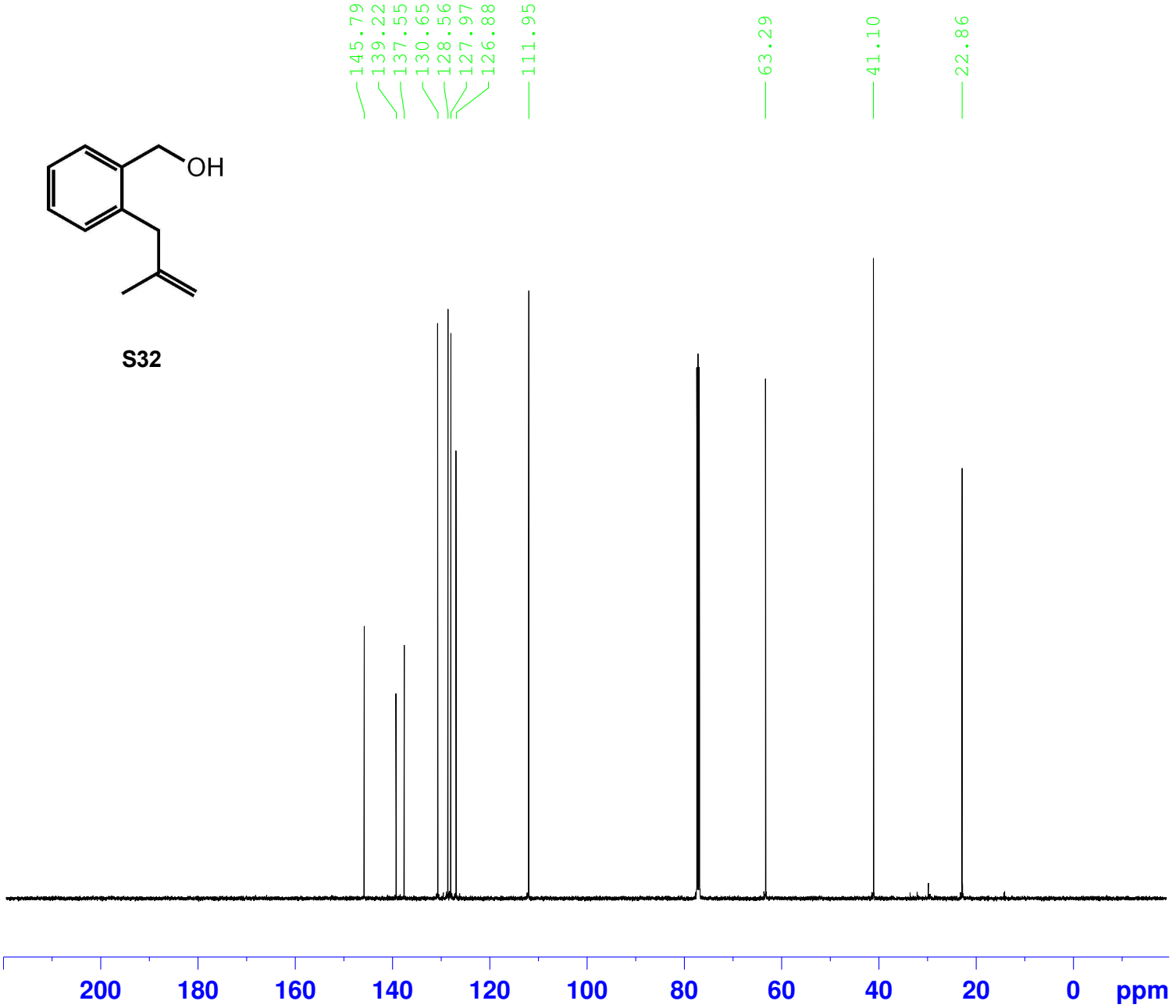
user Joanne Hewitt
JMH-VIII-2B
proton.gla CDCl3 /u joahew 1



S32

NAME	JMH-VIII-2B_2
EXPNO	10
PROCNO	1
Date_	20121116
Time	13.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	64
DW	48.667 usec
DE	7.02 usec
TE	296.5 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900118 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

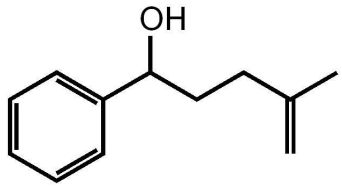
user Joanne Hewitt
JMH-VIII-2B
C13CPD1024.GLA CDCl3 /u joahew 1



NAME	JMH-VIII-2B
EXPNO	11
PROCNO	1
Date_	20121116
Time	13.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	297.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728643 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-13B
proton.gla CDCl3 /u joahew 28

7.359 7.351 7.342 7.299 7.291 7.290 7.282 7.275 7.272 7.259 4.738 4.737 4.736 4.734 4.715 4.714 4.712 4.710 4.699 4.688 4.684 4.673 2.147 2.137 2.128 2.117 2.063 2.057 2.045 1.985 1.974 1.970 1.958 1.955 1.951 1.947 1.943 1.939 1.931 1.928 1.924 1.912 1.890 1.879 1.871 1.867 1.863 1.859 1.852 1.848 1.844 1.833 1.734

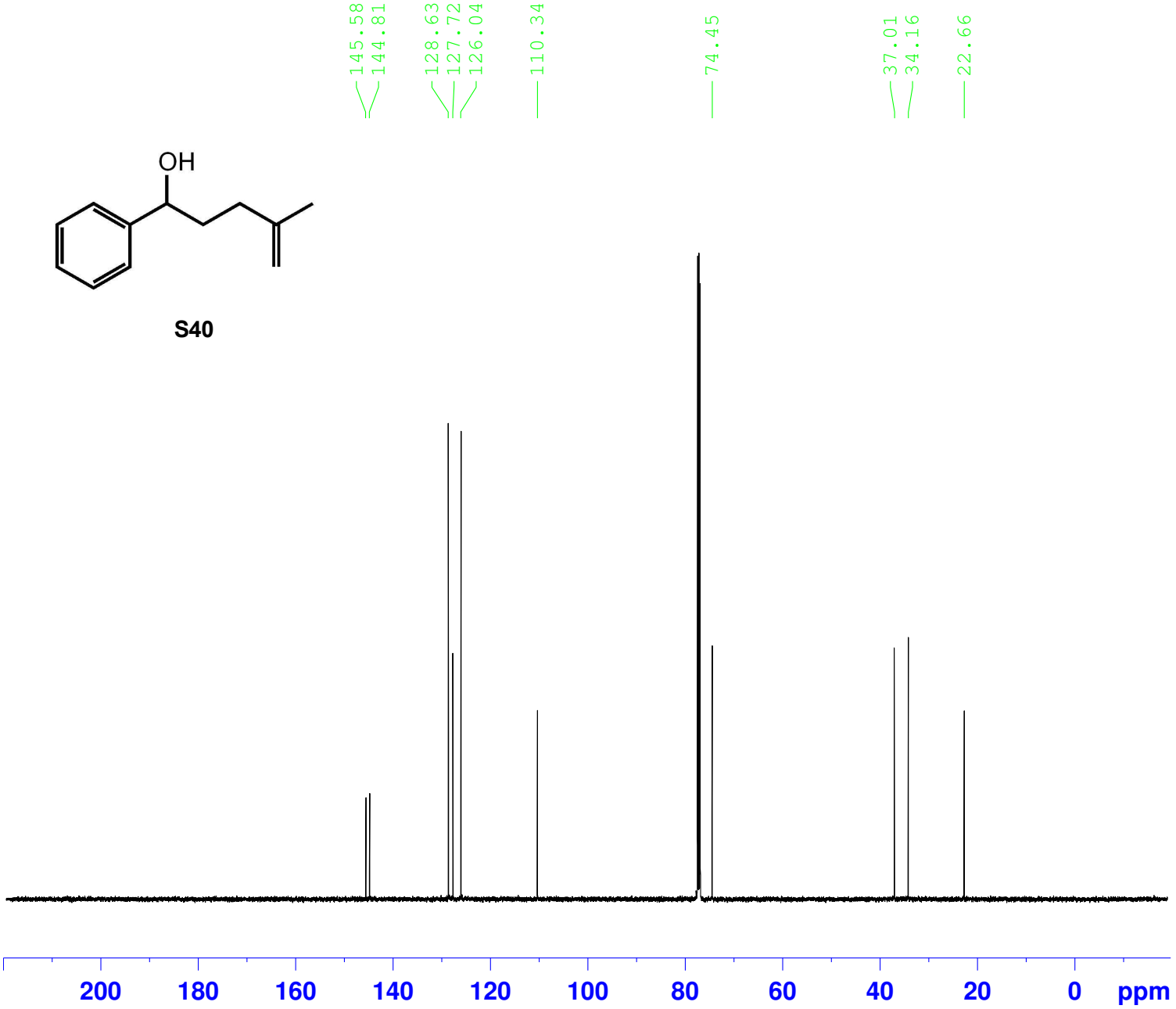


S40

NAME	JMH-VI-13B_2
EXPNO	10
PROCNO	1
Date_	20120618
Time	22.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	128
DW	48.667 usec
DE	7.02 usec
TE	271.5 K
D1	0.50000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900121 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VI-13B
C13CPD1024.GLA CDCl3 /u joahew 28

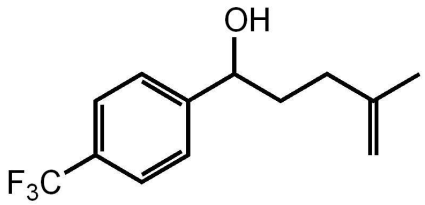


S40

```
NAME          JMH-VI-13B
EXPNO                 11
PROCNO                 1
Date_           20120618
Time               23.47
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH            30000.000 Hz
FIDRES          0.457764 Hz
AQ              1.0923166 sec
RG                   2050
DW                 16.667 usec
DE                  8.01 usec
TE                 272.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.50 usec
PL1                -2.50 dB
PL1W        147.40557861 W
SFO1         125.7854522 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.85 dB
PL13               20.00 dB
PL2W         18.75546646 W
PL12W         0.38737163 W
PL13W         0.23611732 W
SFO2         500.1920008 MHz
SI                 32768
SF           125.7728594 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

user Joanne Hewitt
JMH-VI-63B
proton.gla CDCl3 /u joahew 39

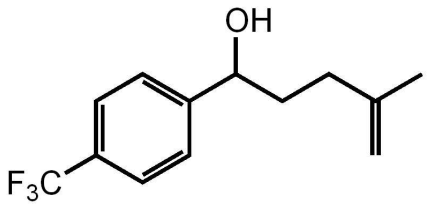
7.620 7.604 7.483 7.466 7.259 4.789 4.780 4.766 4.731 4.729 2.187 2.175 2.168 2.157 2.146 2.139 2.126 2.109 2.094 2.079 2.064 1.972 1.960 1.957 1.955 1.945 1.940 1.933 1.929 1.926 1.918 1.913 1.905 1.900 1.895 1.892 1.886 1.883 1.881 1.876 1.874 1.867 1.863 1.859 1.855 1.849 1.846 1.836 1.746 1.707 1.486 1.221 1.174



S42

NAME	JMH-VI-63B_2
EXPNO	10
PROCNO	1
Date_	20120813
Time	7.57
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	256
DW	48.667 usec
DE	7.02 usec
TE	295.2 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900111 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

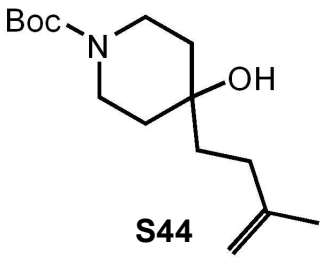
user Joanne Hewitt
JMH-VI-63B
C13CPD1024.GLA CDCl3 /u joahew 39



S42

NAME	JMH-VI-63B
EXPNO	11
PROCNO	1
Date_	20120814
Time	4.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728475 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

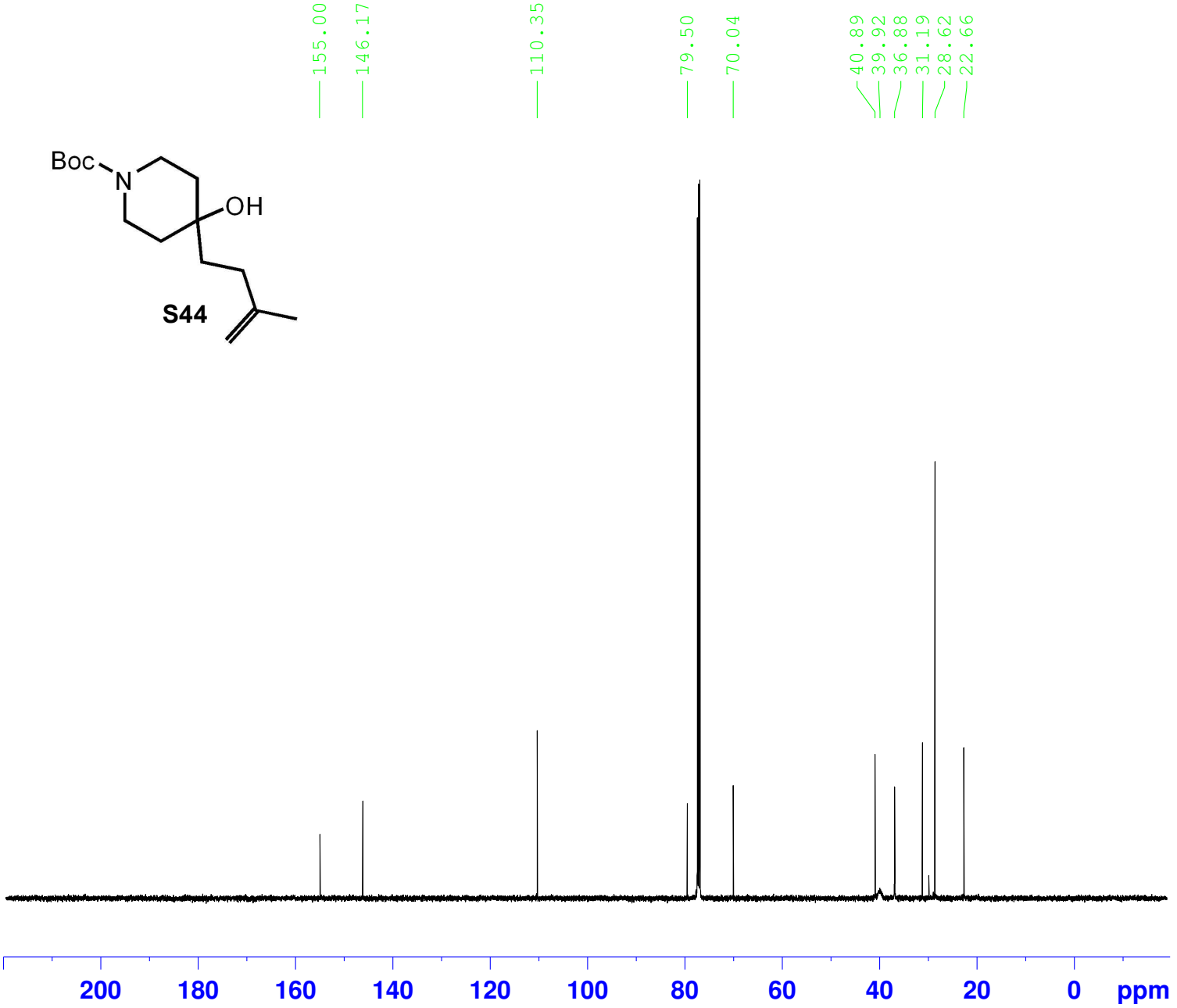
user Joanne Hewitt
JMH-VIII-45F
proton.gla CDCl3 /u joahew 33



S44

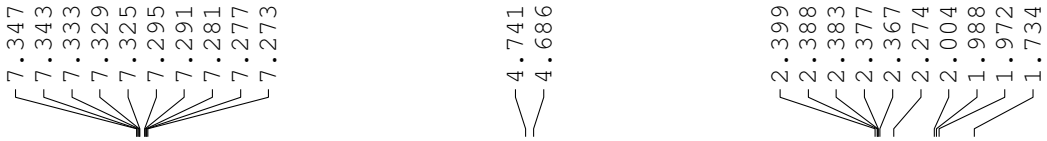
NAME	JMH-VIII-45F_2	
EXPNO	10	
PROCNO	1	
Date_	20130110	
Time	9.04	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	74012	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10273.973	Hz
FIDRES	0.138815	Hz
AQ	3.6019673	sec
RG	128	
DW	48.667	usec
DE	7.02	usec
TE	302.3	K
D1	0.50000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	1H	
P1	11.00	usec
PL1	1.10	dB
PL1W	18.32853889	W
SFO1	500.1930889	MHz
SI	131072	
SF	500.1900117	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

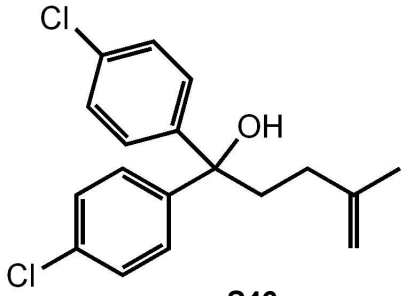
user Joanne Hewitt
JMH-VIII-45F
C13CPD1024.GLA CDCl3 /u joahew 33



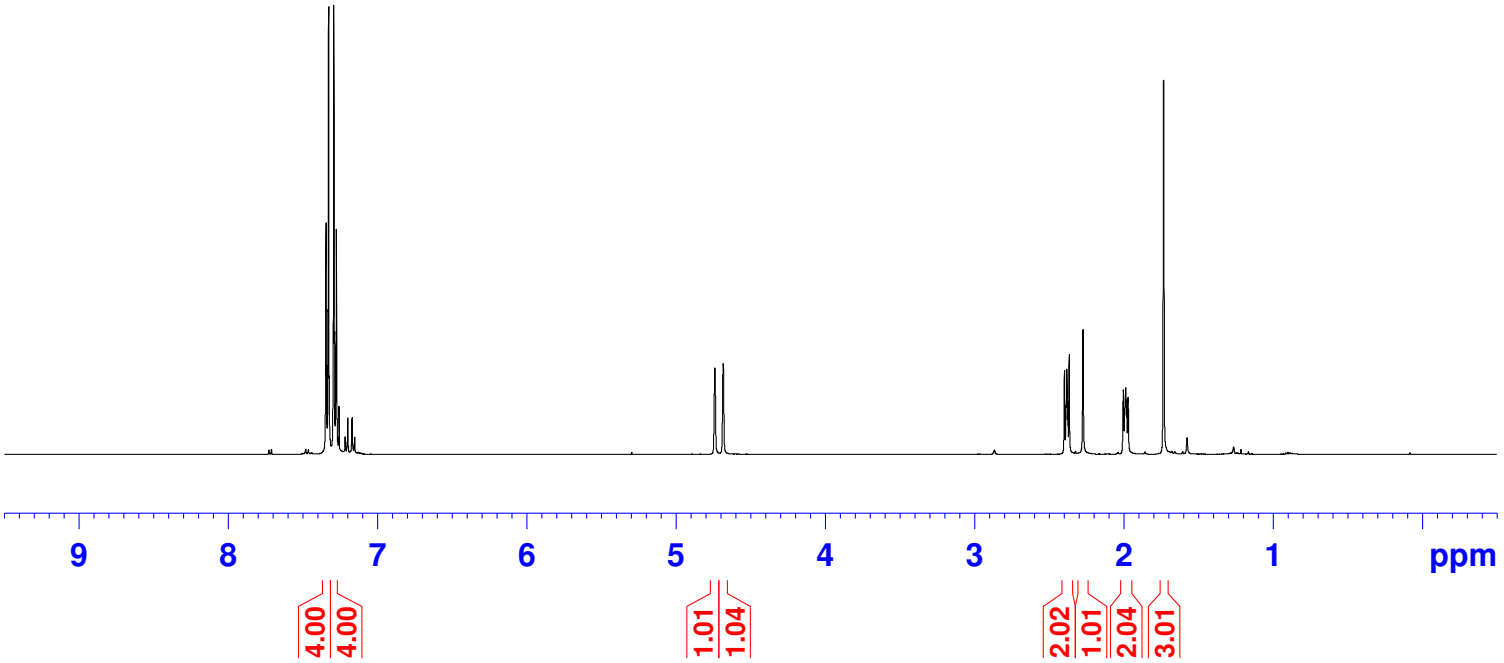
NAME	JMH-VIII-45F
EXPNO	14
PROCNO	1
Date_	20130110
Time	10.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	303.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728558 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VII-4F
proton.gla CDCl3 /u joahew 9





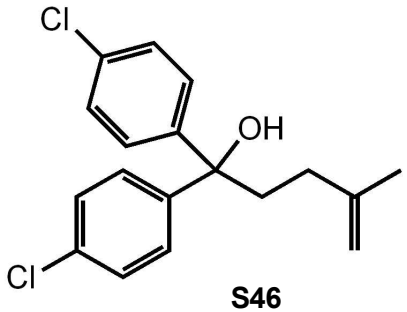
S46



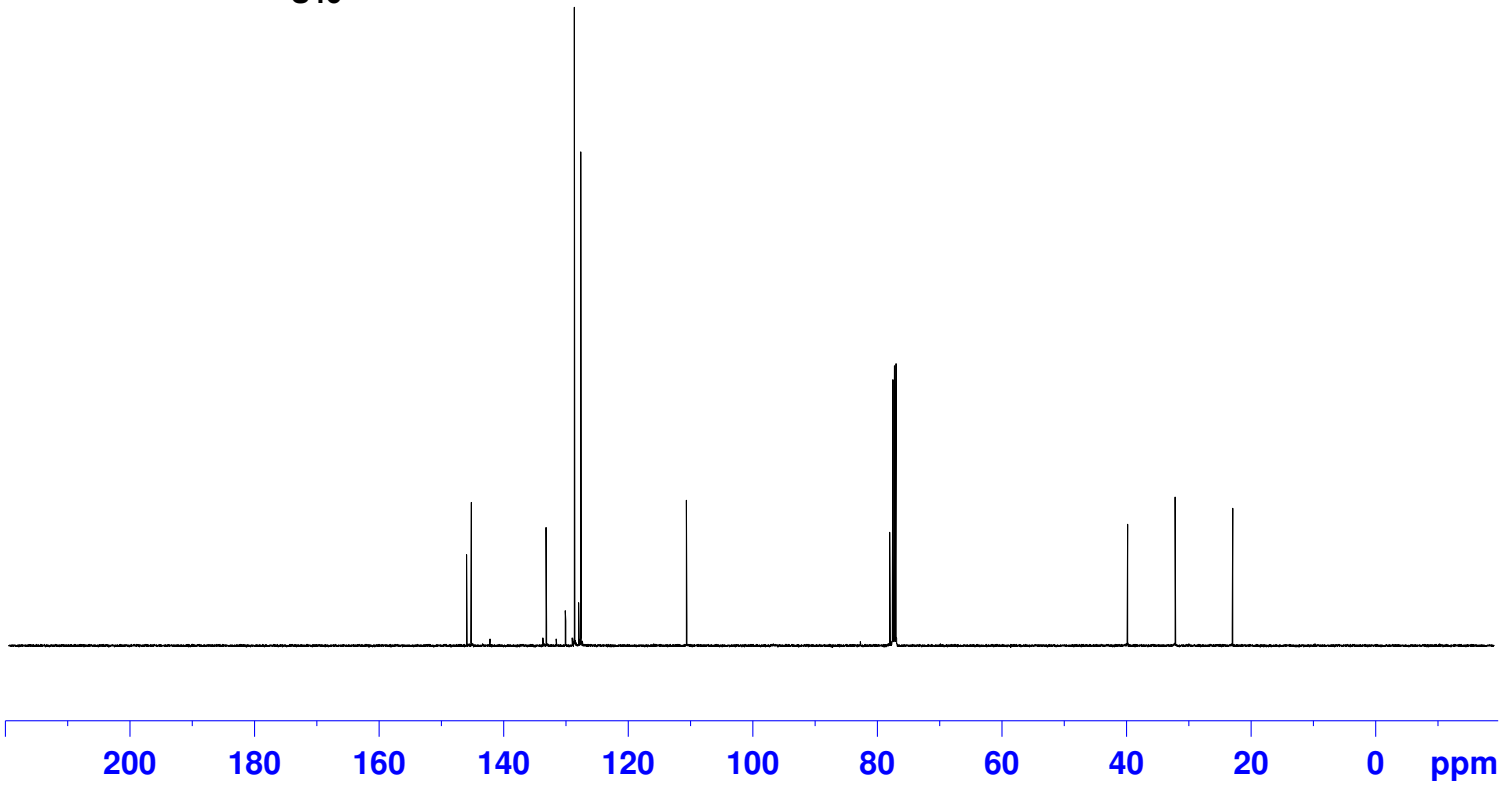
NAME	JMH-VII-4F_2
EXPNO	10
PROCNO	1
Date_	20121110
Time	9.54
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	80.6
DW	48.667 usec
DE	7.02 usec
TE	296.0 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900113 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VII-4F
C13CPD1024.GLA CDCl3 /u joahew 9

145.86
145.12
133.07
128.56
127.52
110.53
77.88
39.68
32.04
22.83

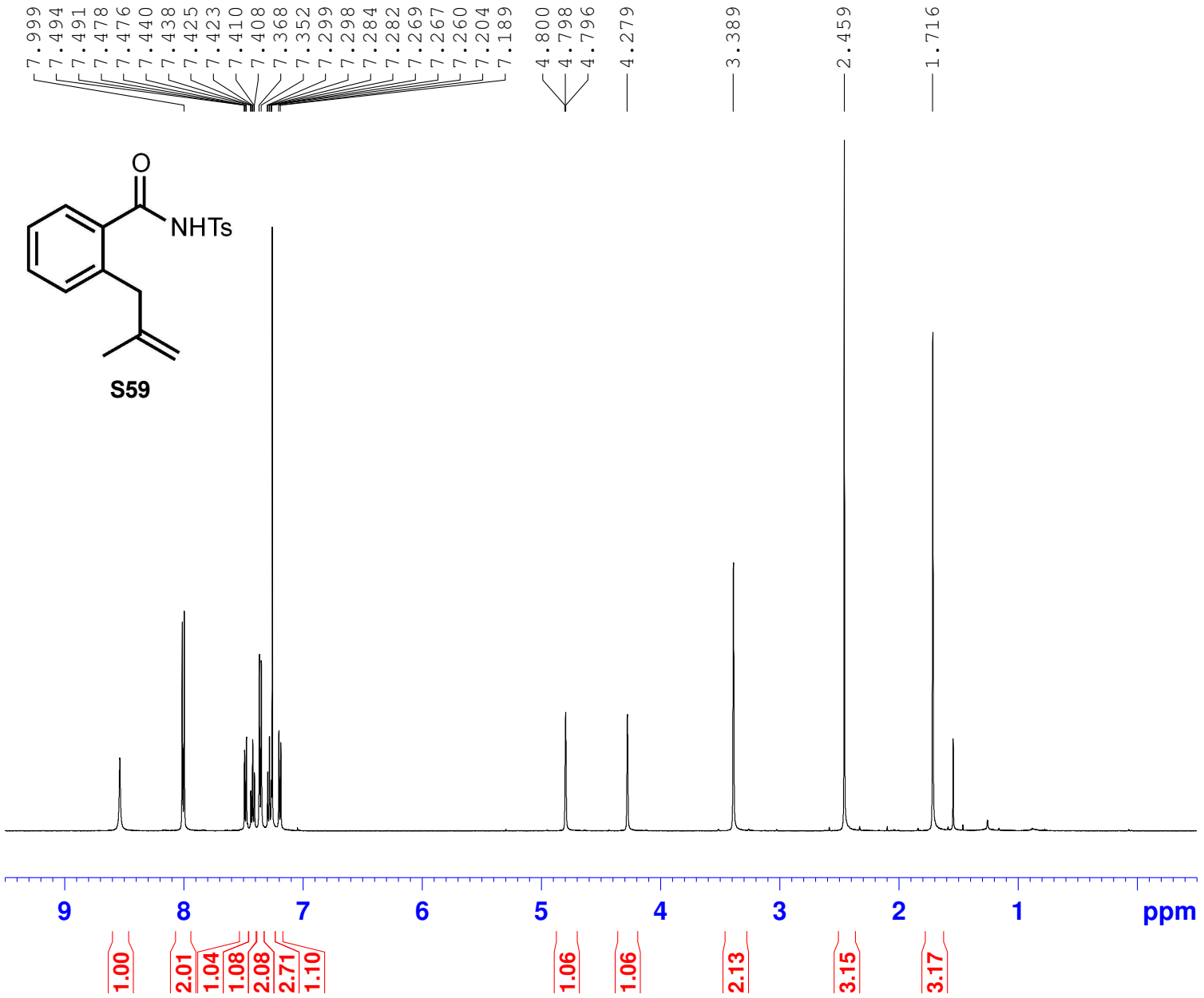


S46



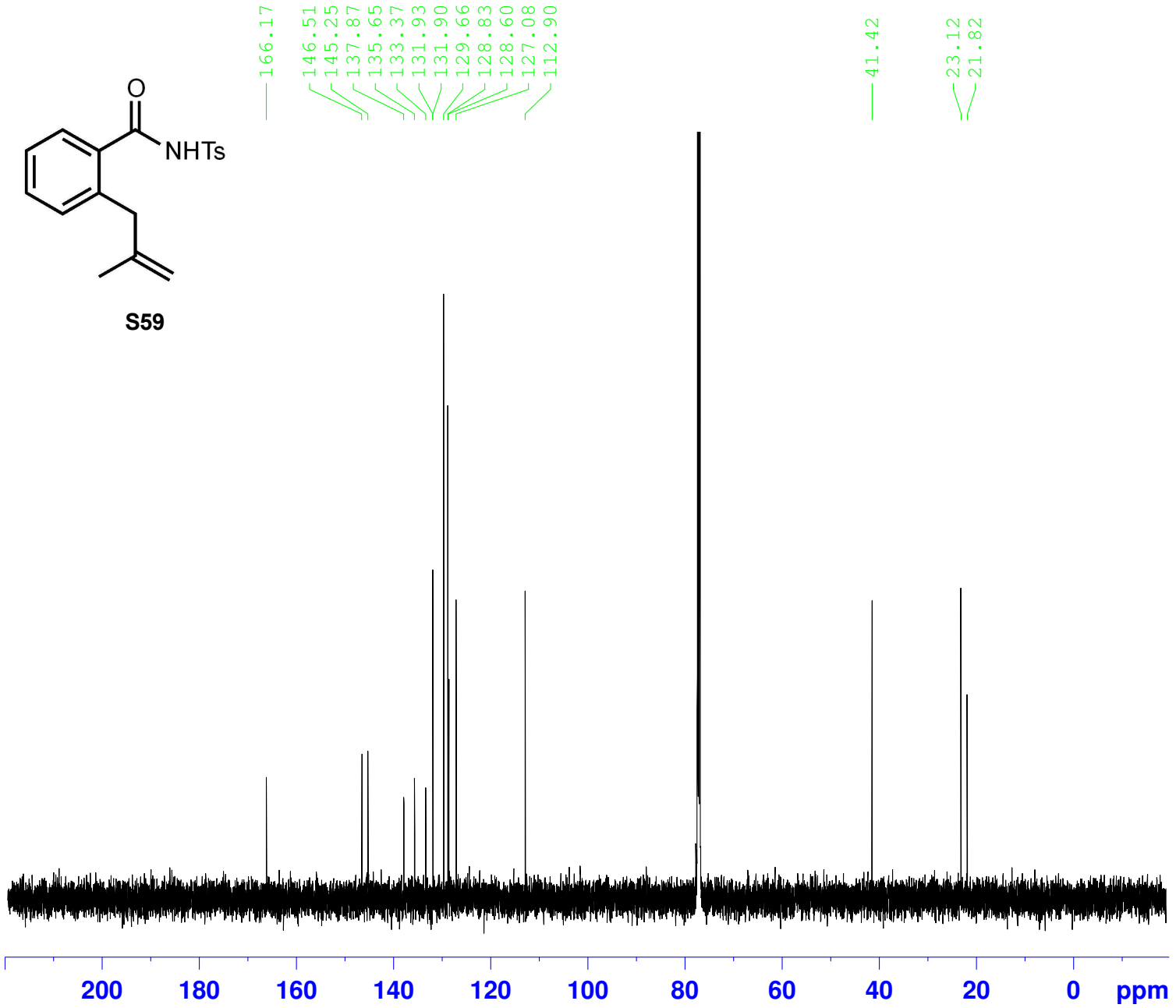
NAME	JMH-VII-4F
EXPNO	11
PROCNO	1
Date_	20121110
Time	10.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728613 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Lewis Williams
proton.gla CDCl3 /u lewiwi 31



NAME	LW3-86_2
EXPNO	10
PROCNO	1
Date_	20130331
Time	11.55
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	322
DW	48.667 usec
DE	7.02 usec
TE	298.6 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900112 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

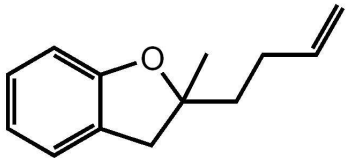
user Lewis Williams
C13CPD1024.GLA CDCl3 /u lewiwi 31



NAME	LW4-86_2
EXPNO	10
PROCNO	1
Date_	20130331
Time	13.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728598 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-IV-14G
proton.gla CDCl3 /u joahew 4

7.136 7.121 7.103 7.087 6.827 6.812 6.798 6.740 6.724 6.875 5.862 5.854 5.849 5.841 5.828 5.820 5.814 5.807 5.794 5.045 5.042 5.011 5.008 4.963 4.960 4.942 4.939 3.113 3.082 2.958 2.927 2.198 2.195 2.185 2.174 2.162 2.152 2.149 1.845 1.833 1.825 1.814 1.811 1.442

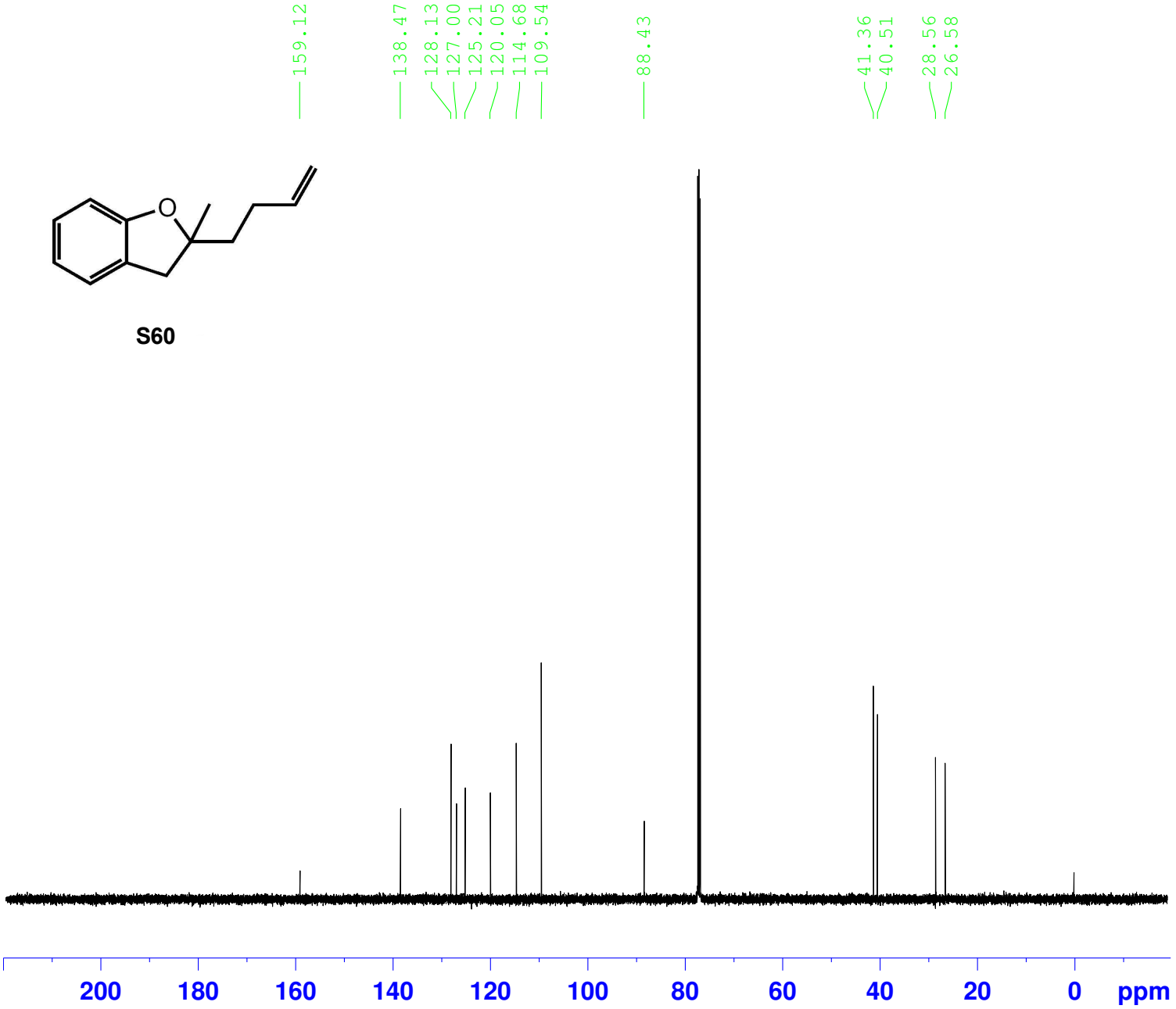


S60

NAME	JMH-IV-14G_2
EXPNO	10
PROCNO	1
Date_	20111128
Time	12.36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	181
DW	48.667 usec
DE	7.02 usec
TE	296.3 K
D1	0.50000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900118 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

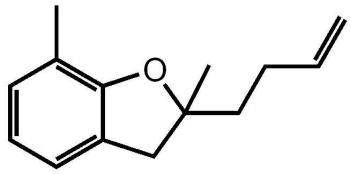
user Joanne Hewitt
JMH-IV-14G
C13CPD1024.GLA CDCl3 /u joahew 4



NAME	JMH-IV-14G
EXPNO	14
PROCNO	1
Date_	20111128
Time	14.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	297.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728563 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-67A
proton.gla CDCl3 /u joahew 49

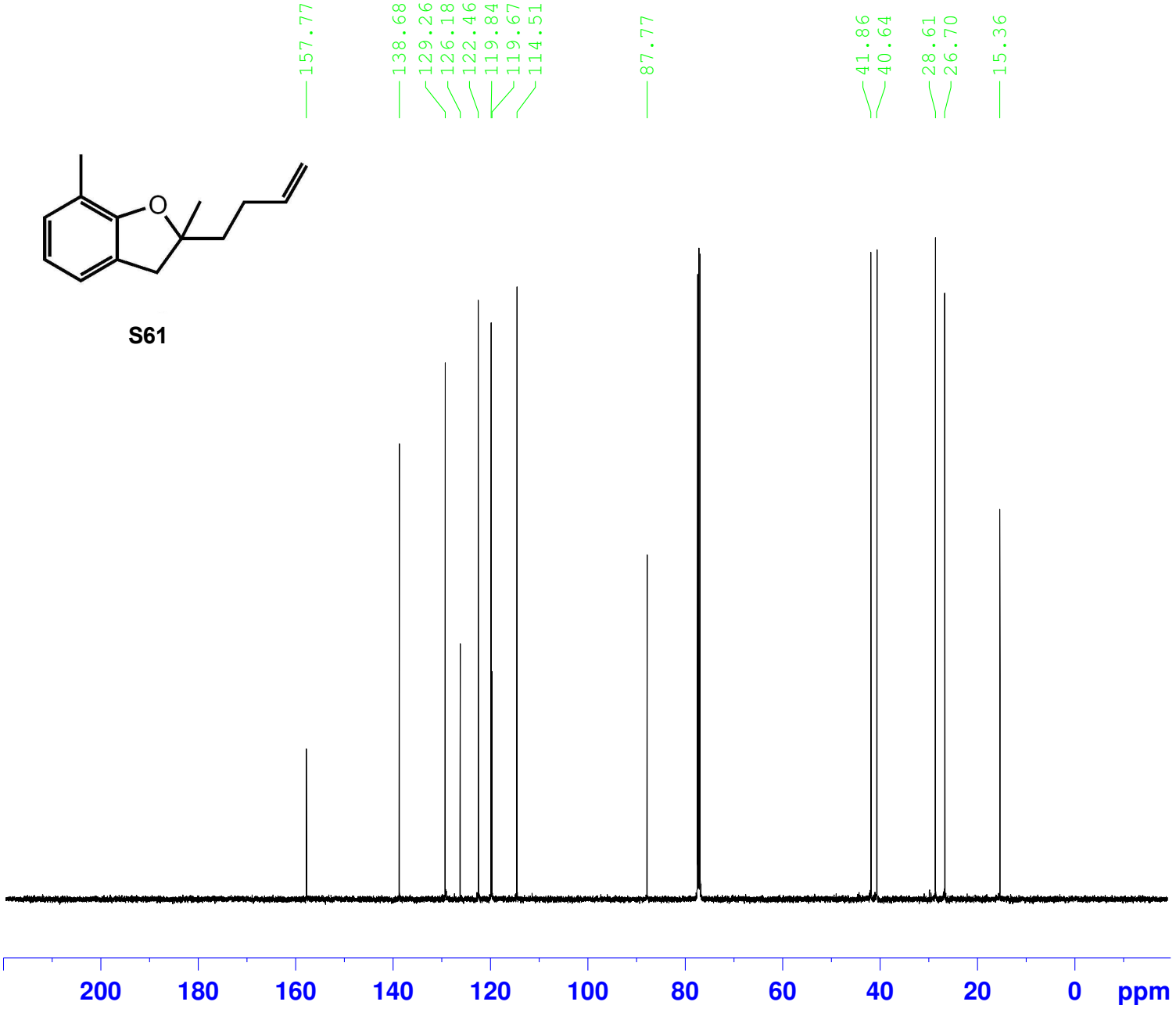
6.972 6.958 6.940 6.939 6.925 6.924 6.745 6.730 6.715 5.891 5.870 5.857 5.836 5.061 5.058 5.054 5.051 5.023 5.020 5.016 4.979 4.976 4.973 4.970 4.958 4.956 4.952 4.950 3.112 3.081 2.970 2.940 2.221 2.217 2.209 2.199 2.196 2.192 2.189 2.186 2.183 2.179 2.174 2.173 1.863 1.859 1.850 1.847 1.843 1.839 1.831 1.826 1.460



S61

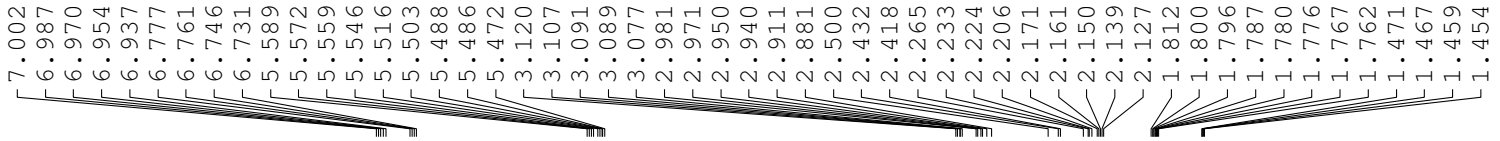
NAME	JMH-VI-67A
EXPNO	10
PROCNO	1
Date_	20120727
Time	7.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	80.6
DW	48.667 usec
DE	7.02 usec
TE	300.0 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900109 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

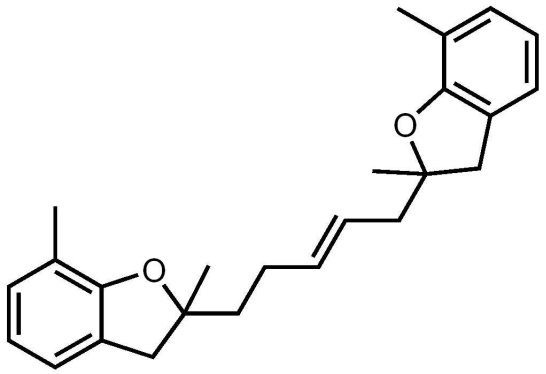
user Joanne Hewitt
JMH-VI-67A
C13CPD1024.GLA CDCl3 /u joahew 49



NAME	JMH-VI-67A
EXPNO	11
PROCNO	1
Date_	20120728
Time	5.46
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728526 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VII-89E
proton.gla CDCl3 /u joahew 1



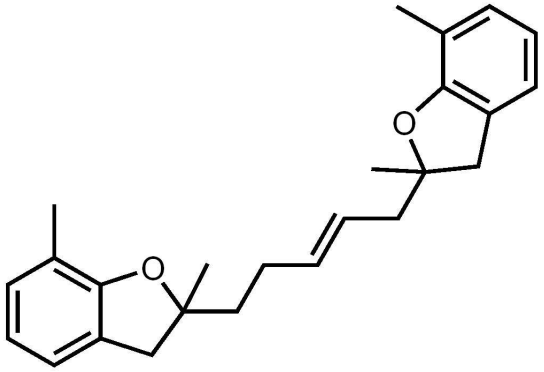


S62

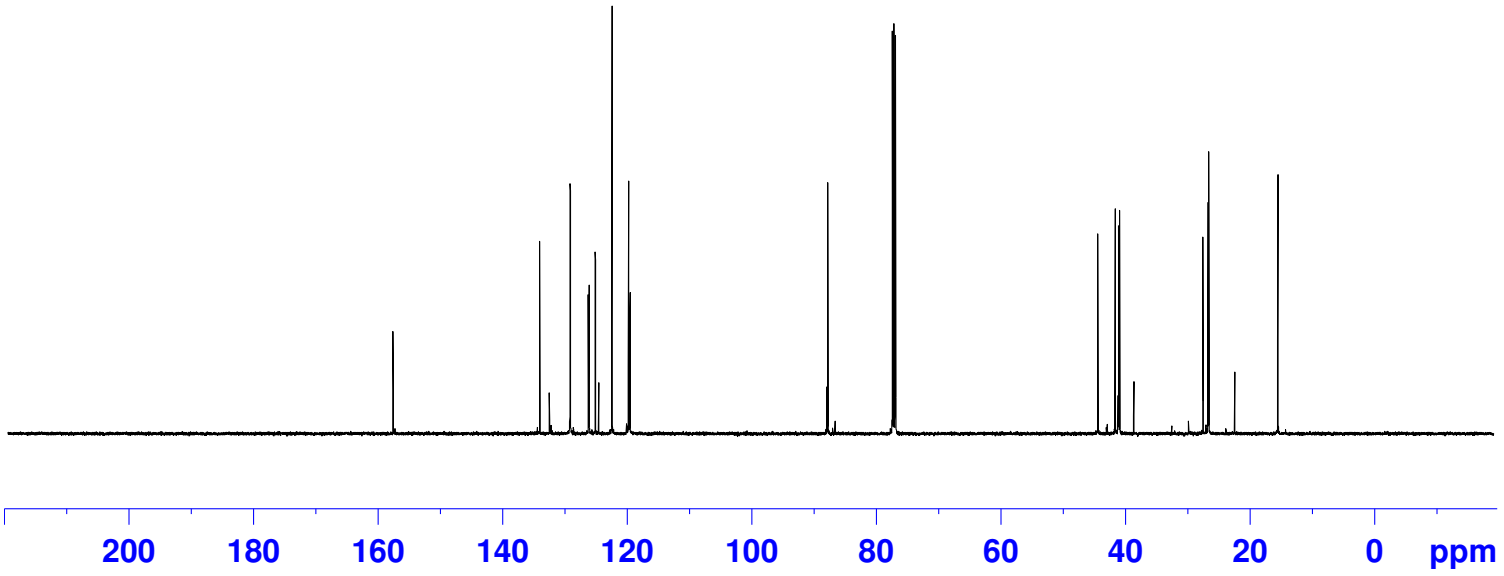
NAME	JMH-VII-89E_2
EXPNO	10
PROCNO	1
Date_	20130617
Time	20.10
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	28.5
DW	48.667 usec
DE	7.02 usec
TE	297.2 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VII-89E
C13CPD1024.GLA CDCl3 /u joahew 1

157.65
157.62
134.06
129.19
129.14
126.26
126.16
125.13
122.45
119.80
119.76
119.56
119.55
87.80
44.41
41.63
41.09
40.91
27.52
26.72
26.58
15.50
15.49



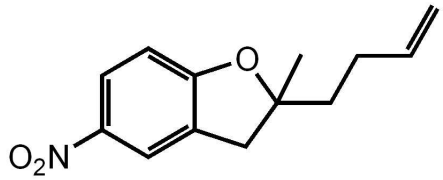
S62



NAME	JMH-VII-89E
EXPNO	11
PROCNO	1
Date_	20130617
Time	21.05
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728669 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-23B
proton.gla CDCl3 /u joahew 29

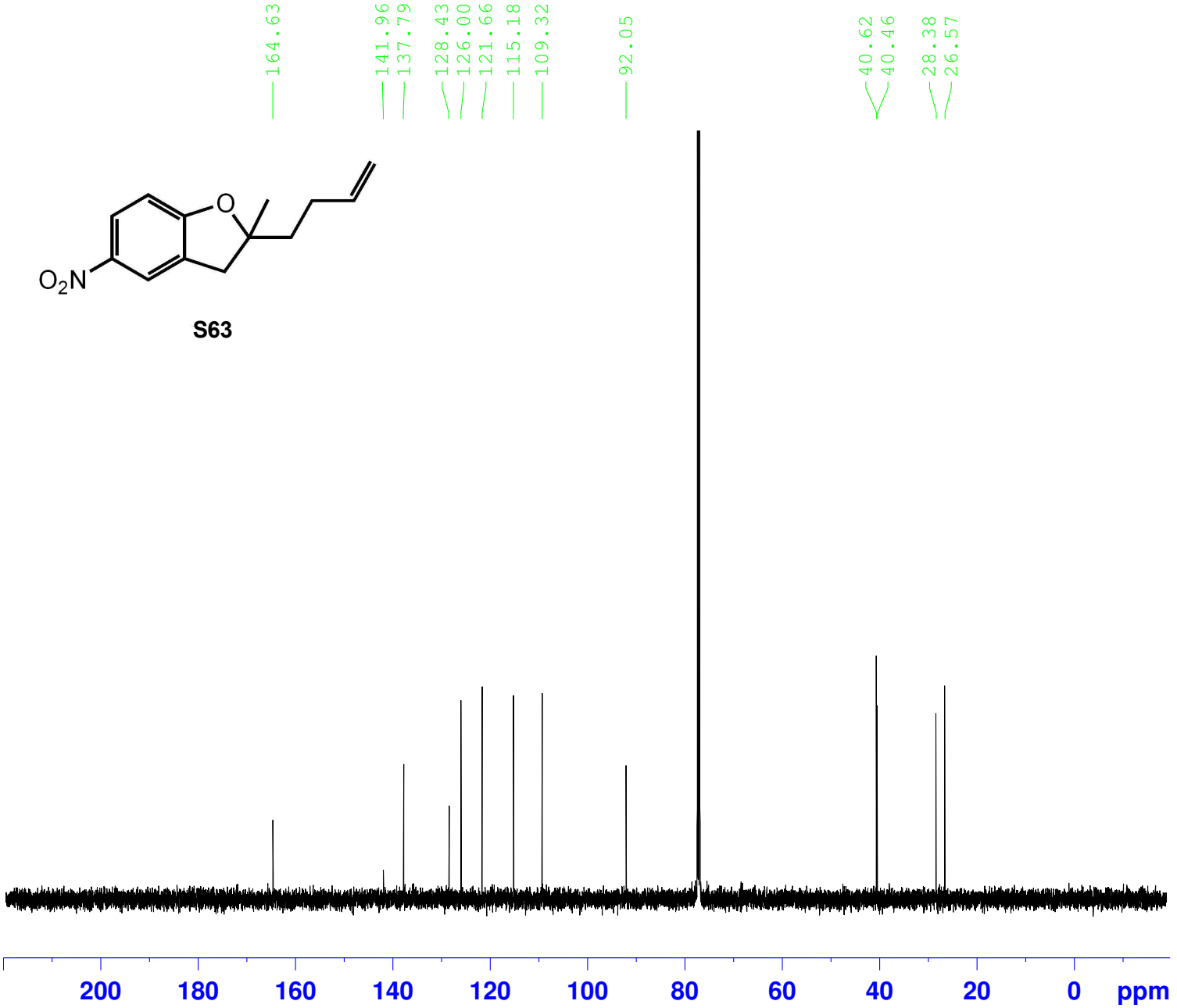
8.101 8.096 8.083 8.078 8.044 8.042 8.040 8.037 8.035 7.260 6.759 6.741 5.850 5.829 5.816 5.795 5.056 5.053 5.049 5.046 5.018 5.015 5.012 4.993 4.990 4.987 4.984 4.972 4.970 4.966 4.964 3.179 3.147 3.023 2.991 2.188 2.185 2.182 2.178 2.175 2.172 2.170 2.167 2.162 2.159 2.157 2.154 2.146 1.894 1.890 1.877 1.861 1.493



S63

NAME	JMH-VI-23B
EXPNO	10
PROCNO	1
Date_	20120720
Time	17.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	228
DW	48.667 usec
DE	7.02 usec
TE	295.3 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900108 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

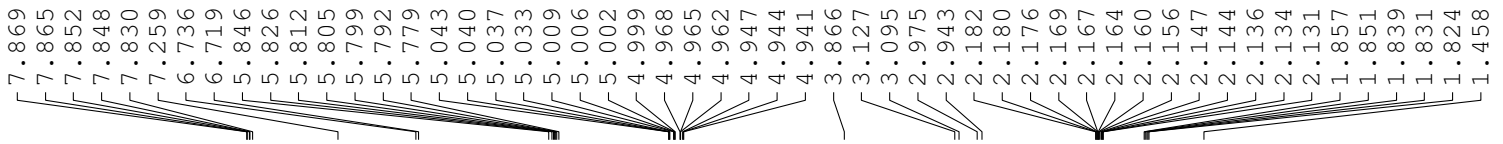
user Joanne Hewitt
JMH-VI-23B
C13CPD256.GLA CDCl3 /u joahew 29

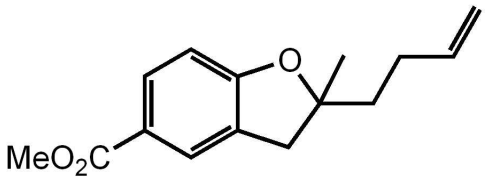


S63

NAME	JMH-VI-23B
EXPNO	11
PROCNO	1
Date_	20120720
Time	17.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728477 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VII-56A
proton.gla CDCl3 /u joahew 17

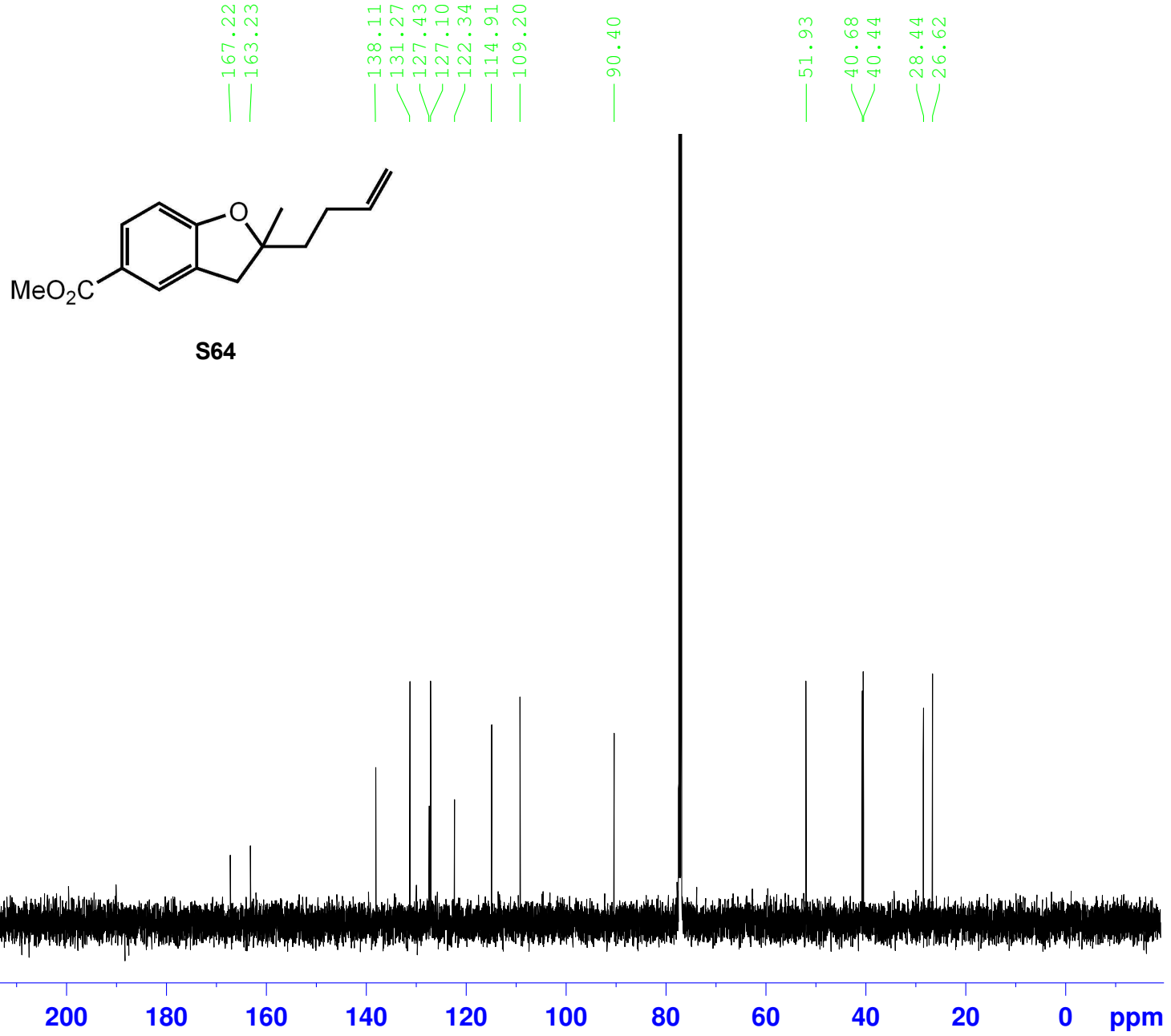




S64

NAME	JMH-VII-56A_2
EXPNO	10
PROCNO	1
Date_	20121004
Time	15.19
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	322
DW	48.667 usec
DE	7.02 usec
TE	298.2 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900119 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

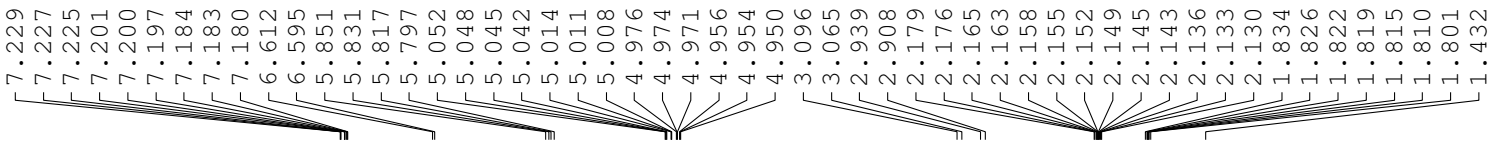
user Joanne Hewitt
JMH-VII-56A
C13CPD256.GLA CDCl3 /u joahew 17

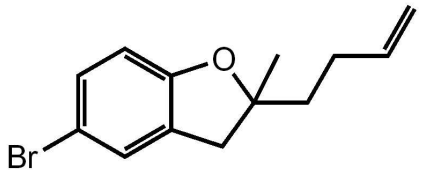


S64

NAME	JMH-VII-56A
EXPNO	11
PROCNO	1
Date_	20121004
Time	15.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728574 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-36A
proton.gla CDCl3 /u joahew 60

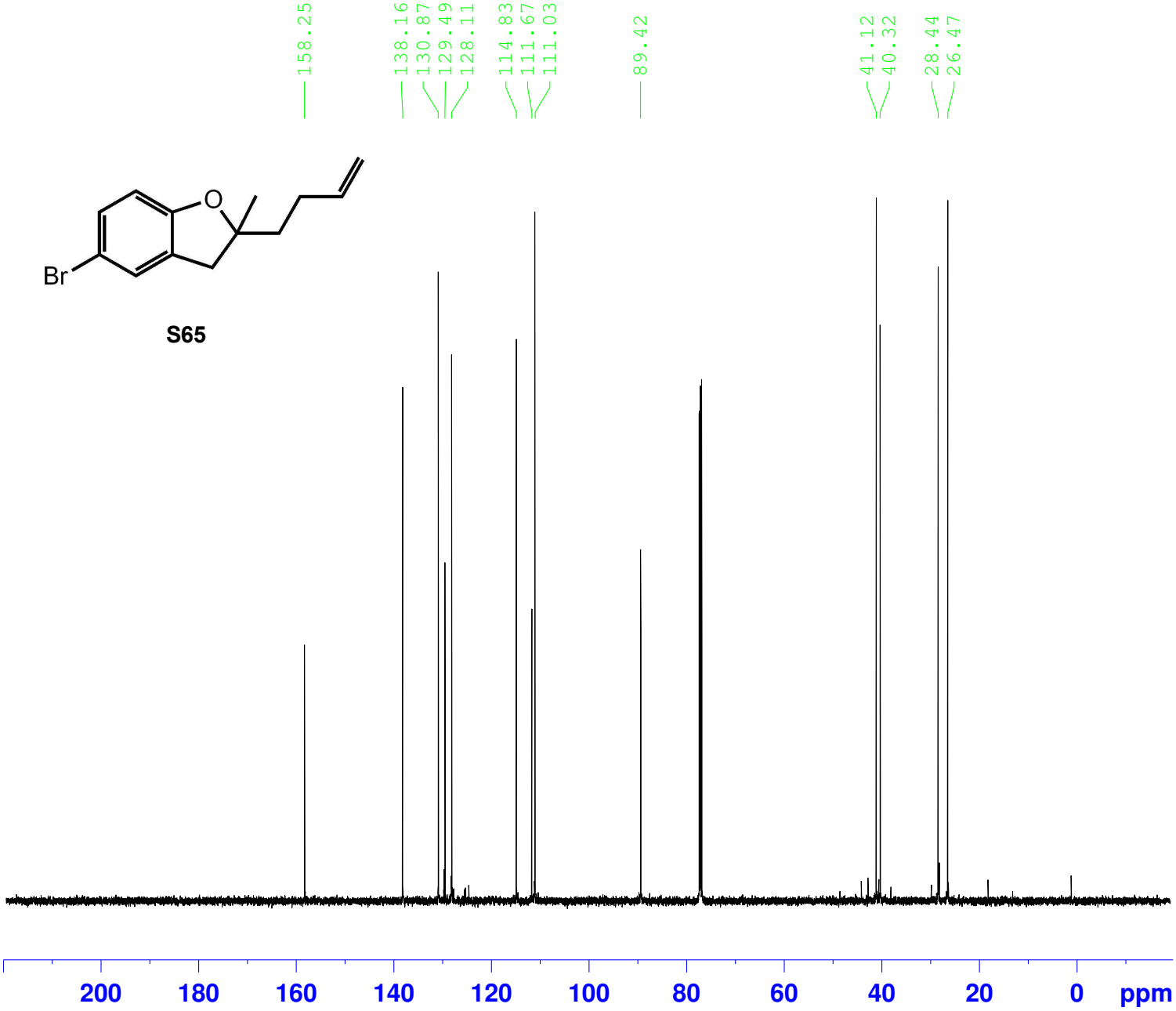




S65

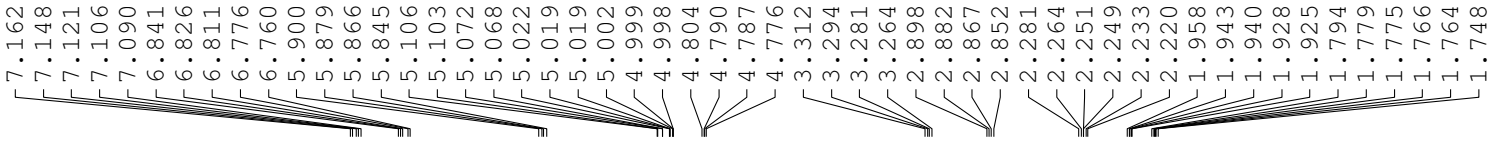
NAME	JMH-VI-36A_2
EXPNO	10
PROCNO	1
Date_	20120627
Time	10.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	36
DW	48.667 usec
DE	7.02 usec
TE	271.5 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900118 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

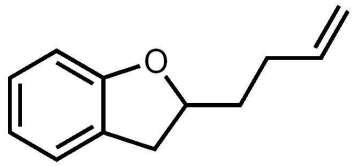
user Joanne Hewitt
JMH-VI-36A
C13CPD256.GLA CDCl3 /u joahew 60



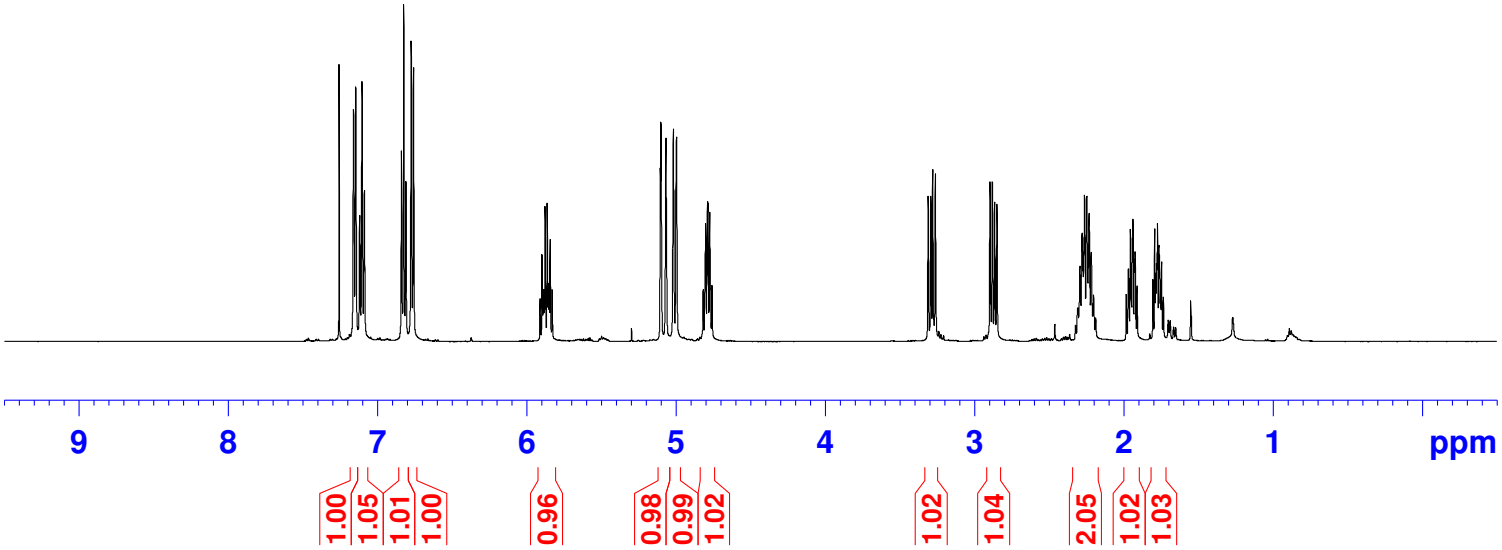
NAME	JMH-VI-36A
EXPNO	11
PROCNO	1
Date_	20120627
Time	11.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	272.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728638 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-IX-21A
proton.gla CDCl3 /u joahew 2



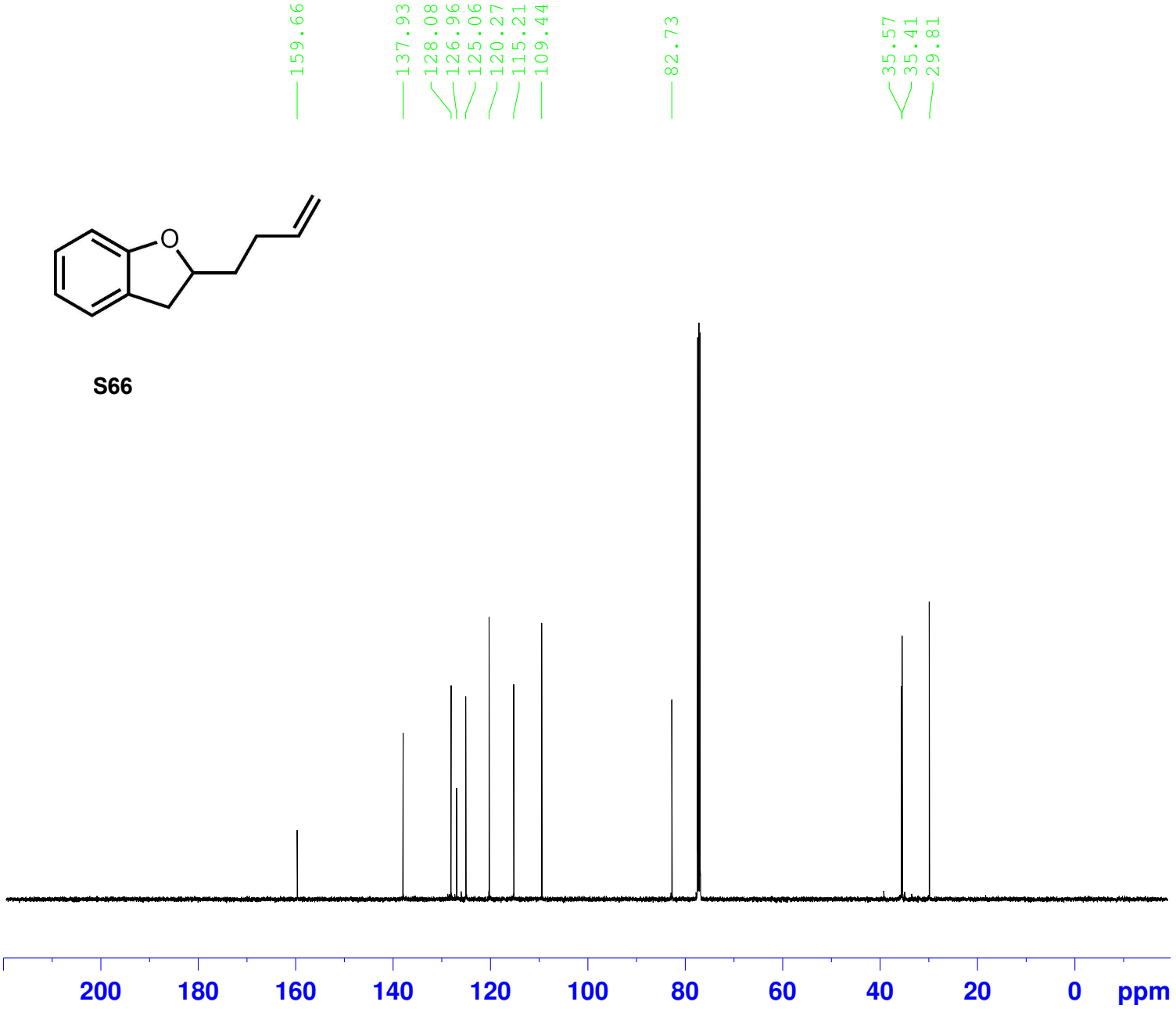


S66



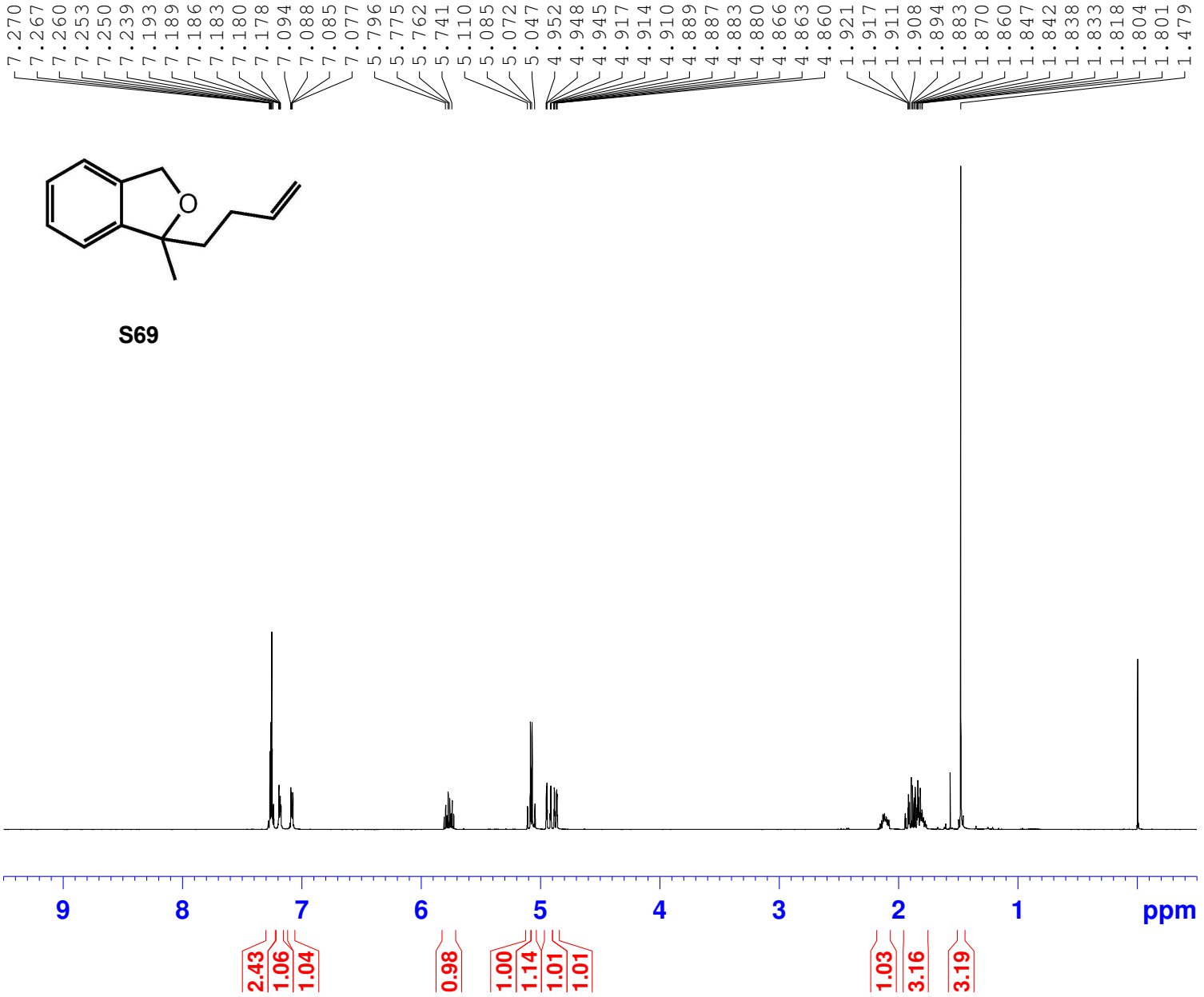
NAME	JMH-IX-21A
EXPNO	10
PROCNO	1
Date_	20130617
Time	21.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	128
DW	48.667 usec
DE	7.02 usec
TE	297.4 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900119 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-IX-21A
C13CPD1024.GLA CDCl3 /u joahew 2



NAME	JMH-IX-21A
EXPNO	11
PROCNO	1
Date_	20130617
Time	22.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	298.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728595 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

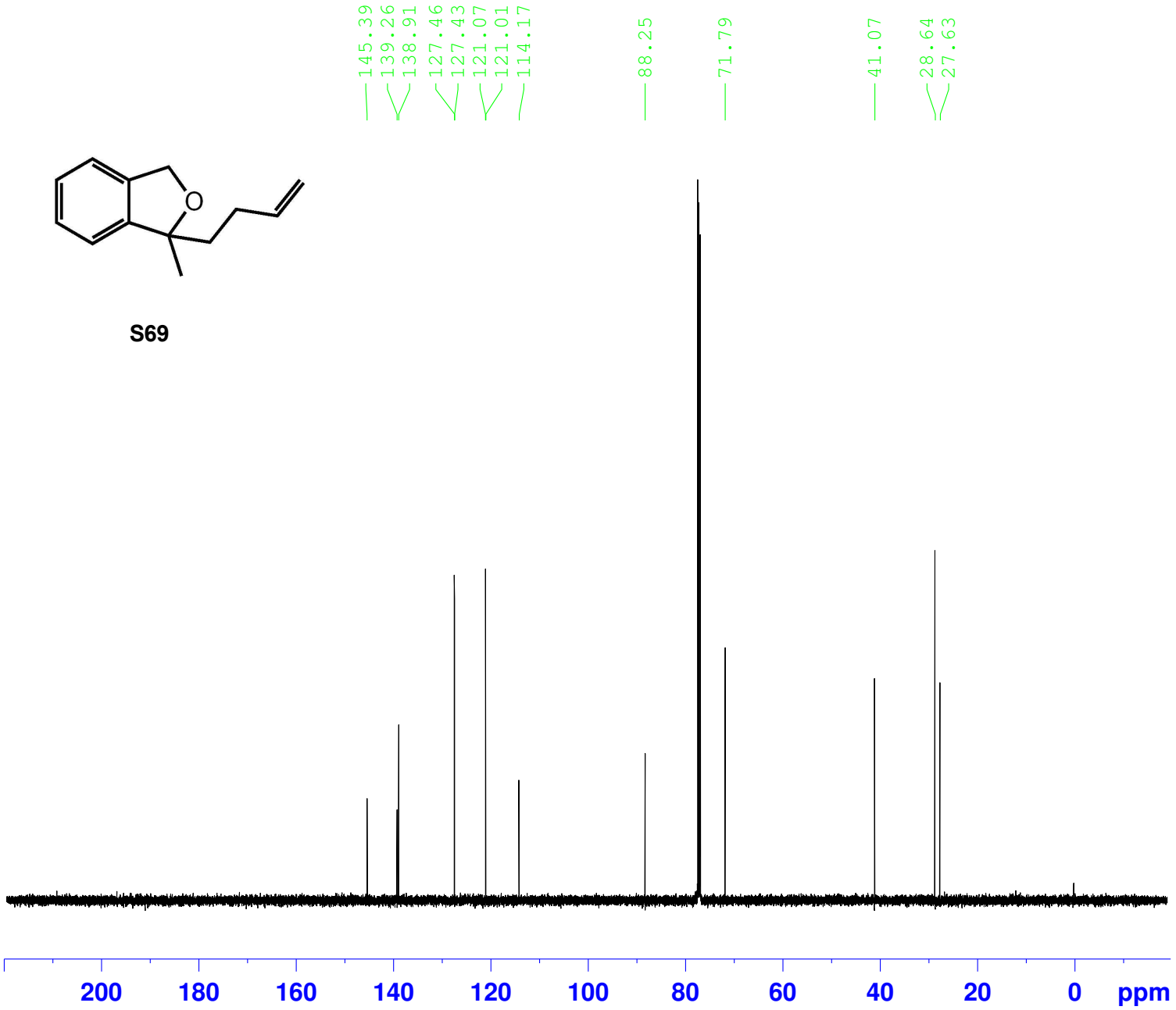
user Joanne Hewitt
JMH-IV-86B
proton.gla CDCl3 /u joahew 10



S69

NAME	JMH-IV-86B_2
EXPNO	10
PROCNO	1
Date_	20120214
Time	22.27
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	161
DW	48.667 usec
DE	7.02 usec
TE	296.8 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900146 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

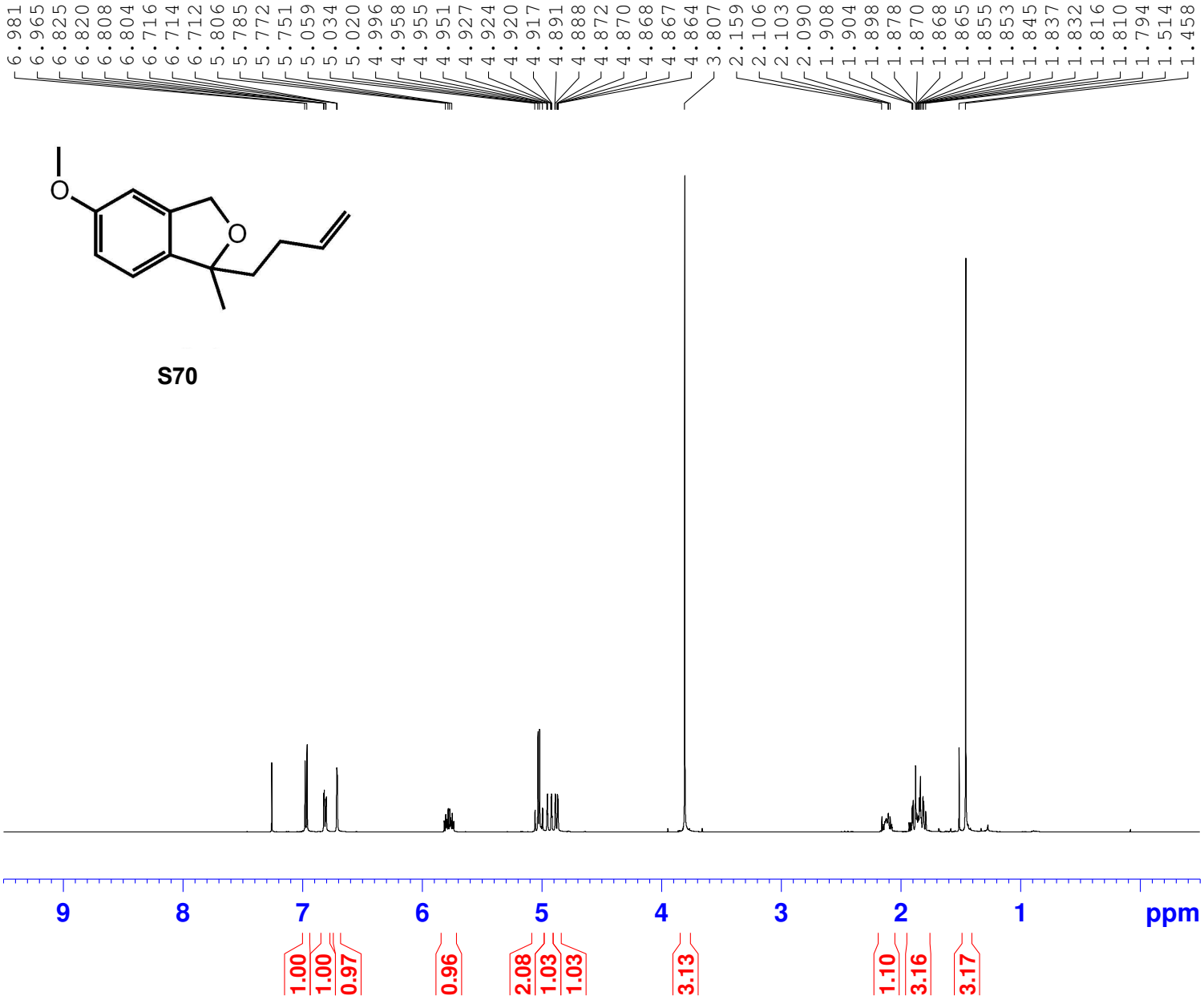
user Joanne Hewitt
JMH-IV-86B
C13CPD1024.GLA CDCl3 /u joahew 10



S69

NAME	JMH-IV-86B
EXPNO	14
PROCNO	1
Date_	20120215
Time	0.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	297.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728579 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-65A
proton.gla CDCl3 /u joahew 48

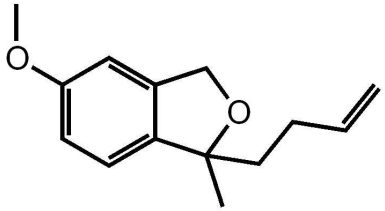


S70

NAME	JMH-VI-65A_2
EXPNO	10
PROCNO	1
Date_	20120727
Time	7.36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	161
DW	48.667 usec
DE	7.02 usec
TE	300.0 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900110 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VI-65A
C13CPD1024.GLA CDCl3 /u joahew 48

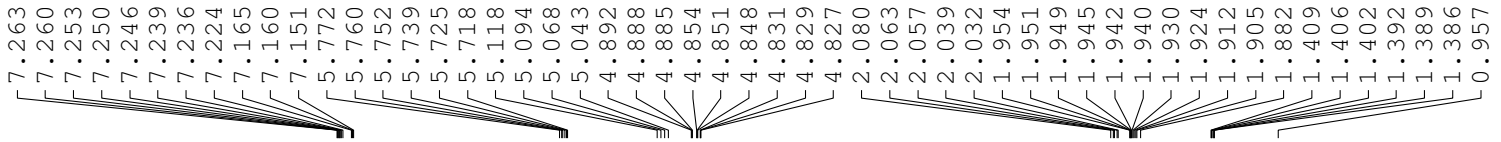
159.82 141.02 139.05 137.86 121.72 114.09 113.81 106.38 88.01 71.62 55.69 41.33 28.73 27.76

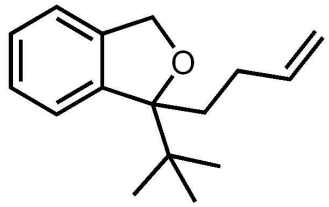


S70

NAME	JMH-VI-65A	
EXPNO	11	
PROCNO	1	
Date_	20120728	
Time	17.35	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	30000.000	Hz
FIDRES	0.457764	Hz
AQ	1.0923166	sec
RG	2050	
DW	16.667	usec
DE	8.01	usec
TE	295.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	7.50	usec
PL1	-2.50	dB
PL1W	147.40557861	W
SFO1	125.7854522	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	1.00	dB
PL12	17.85	dB
PL13	20.00	dB
PL2W	18.75546646	W
PL12W	0.38737163	W
PL13W	0.23611732	W
SFO2	500.1920008	MHz
SI	32768	
SF	125.7728485	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

user Joanne Hewitt
JMH-VIII-72G
proton.gla CDCl3 /u joahew 12

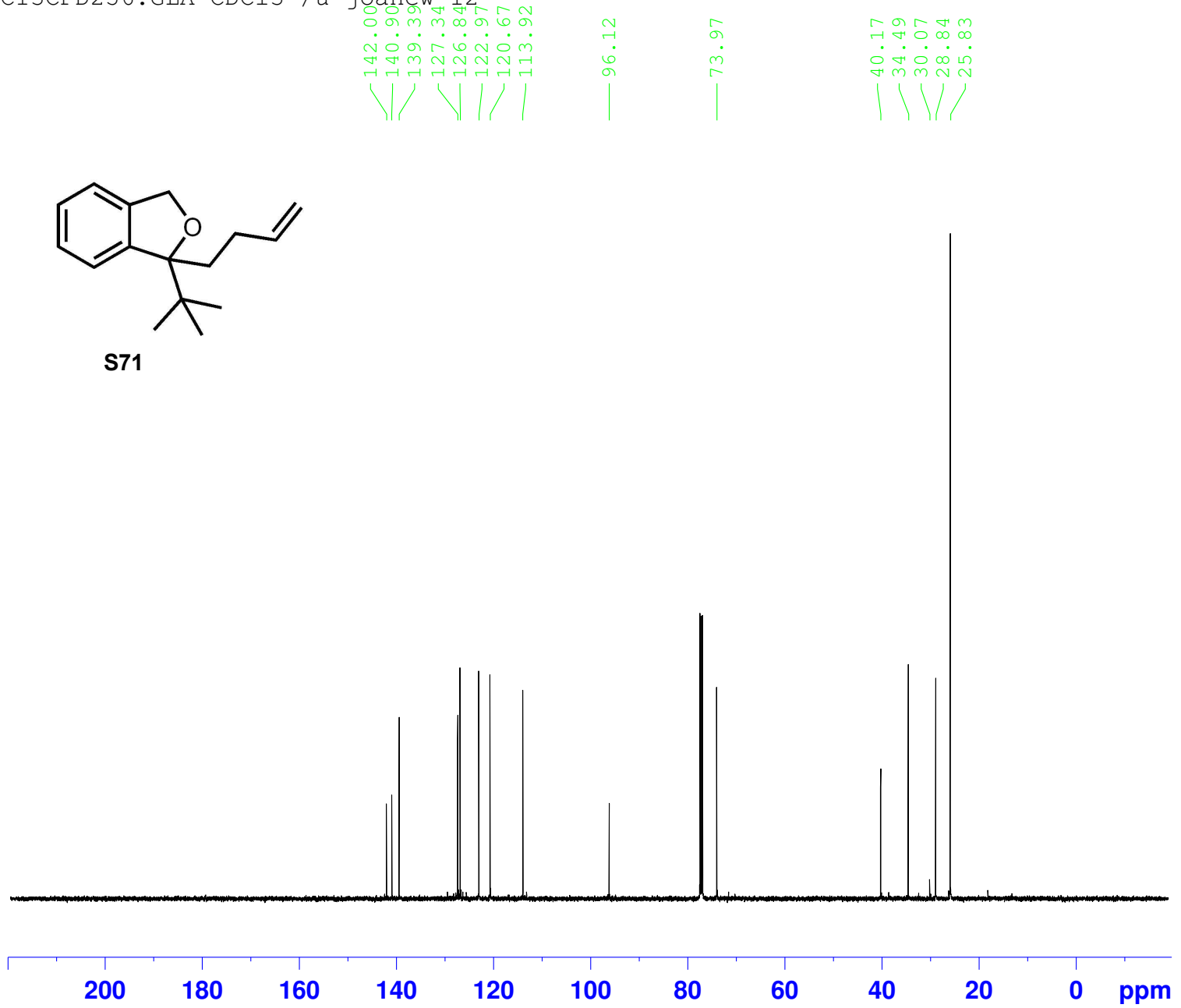




S71

NAME	JMH-VIII-72G_2	
EXPNO	10	
PROCNO	1	
Date_	20130131	
Time	16.44	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	74012	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10273.973	Hz
FIDRES	0.138815	Hz
AQ	3.6019673	sec
RG	36	
DW	48.667	usec
DE	7.02	usec
TE	302.5	K
D1	0.50000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	1H	
P1	11.00	usec
PL1	1.10	dB
PL1W	18.32853889	W
SFO1	500.1930889	MHz
SI	131072	
SF	500.1900121	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

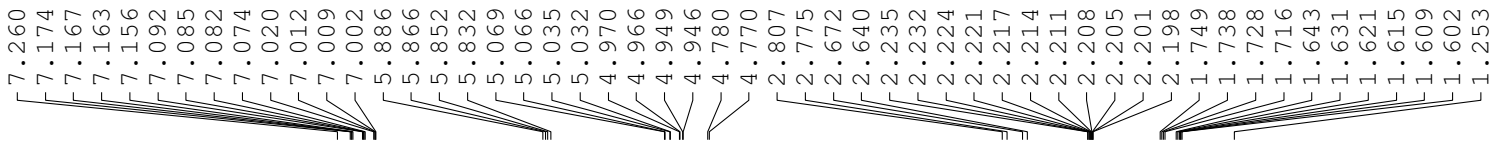
user Joanne Hewitt
JMH-VIII-72G
C13CPD256.GLA CDCl3 /u joahew 12

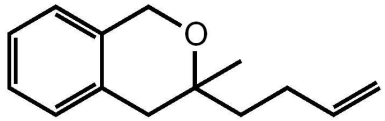


S71

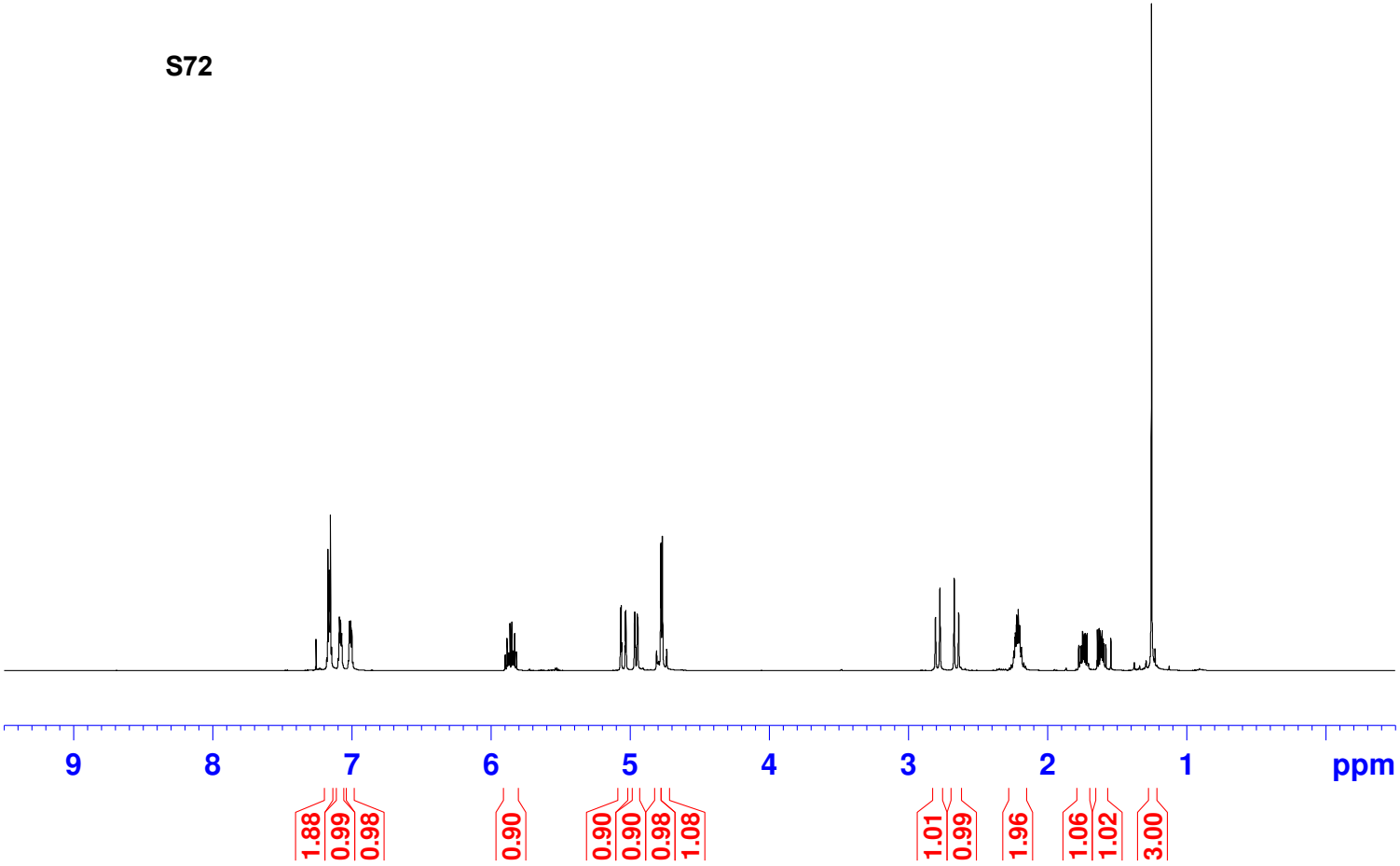
NAME	JMH-VIII-72G
EXPNO	11
PROCNO	1
Date_	20130131
Time	16.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	303.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728589 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-27A
proton.gla CDCl3 /u joahew 10



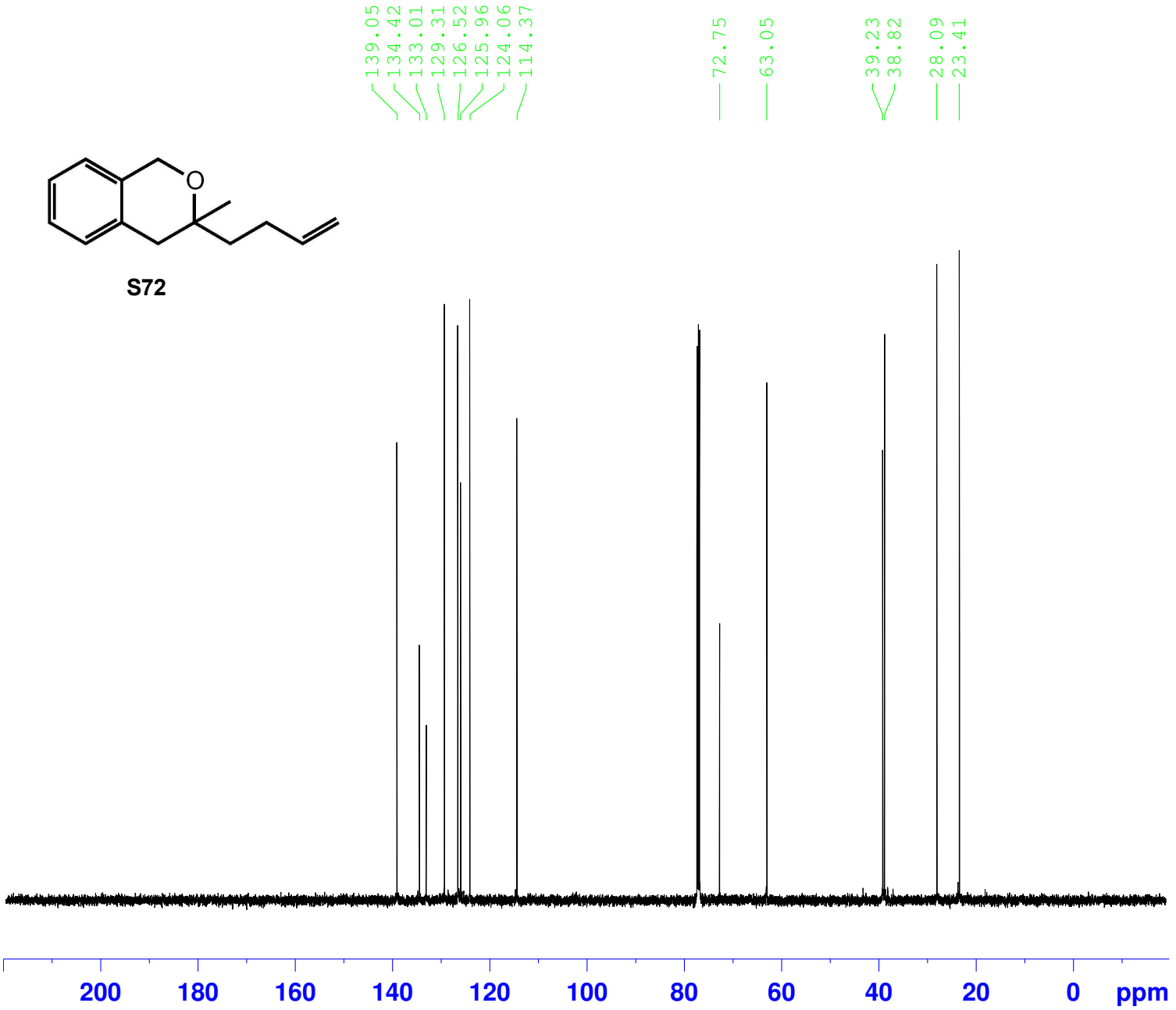


S72



NAME	JMH-VI-27A_2
EXPNO	10
PROCNO	1
Date_	20120628
Time	11.27
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	45.2
DW	48.667 usec
DE	7.02 usec
TE	295.3 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900108 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

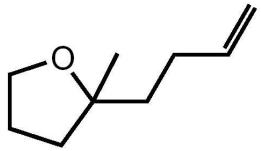
user Joanne Hewitt
JMH-VI-27A
C13CPD256.GLA CDCl3 /u joahew 10



NAME	JMH-VI-27A
EXPNO	11
PROCNO	1
Date_	20120628
Time	11.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728536 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VII-LINEAR
proton.gla CDCl3 /u joahew 44

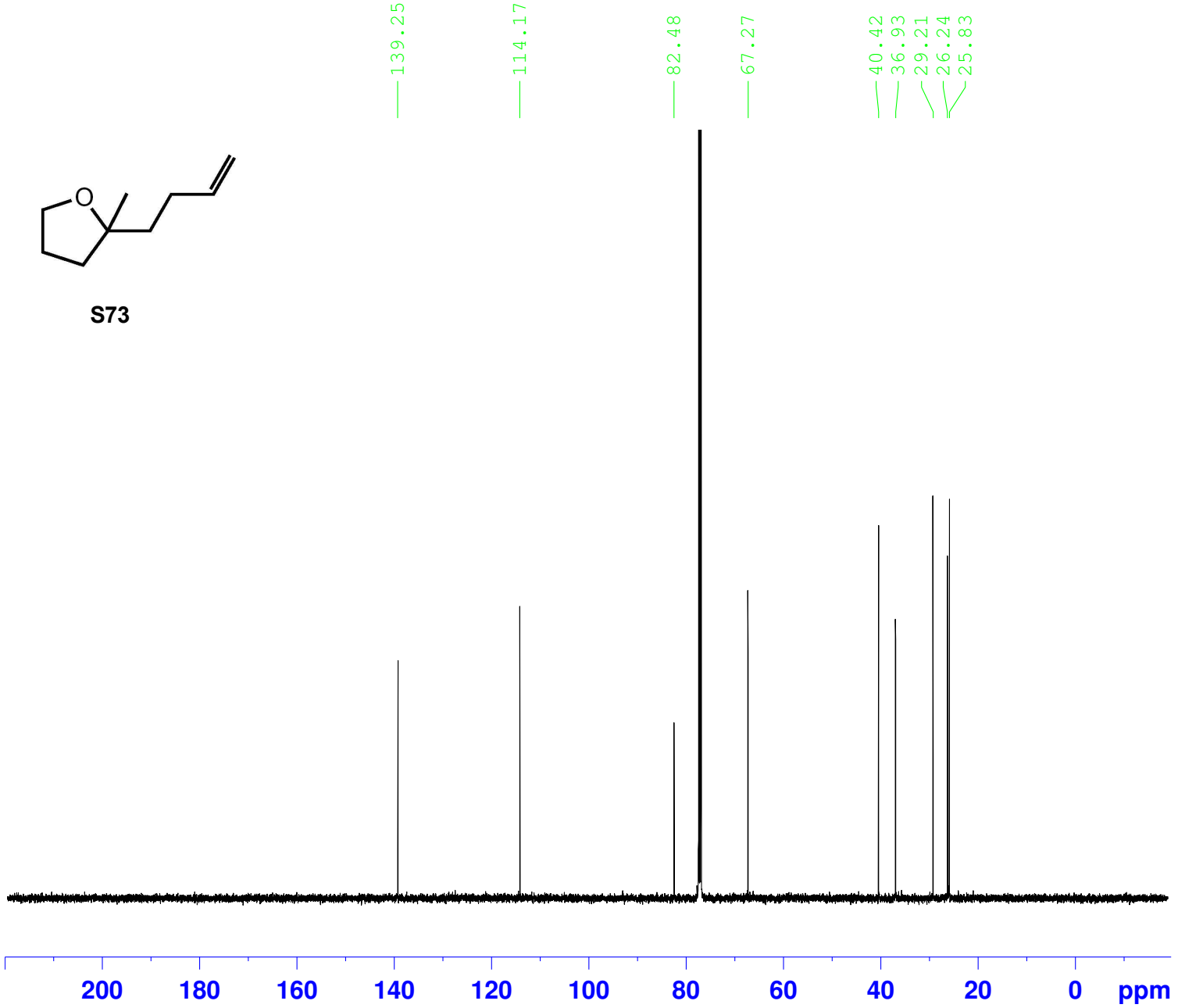
5.847 5.833 5.813 5.038 5.034 5.003 5.000 4.940 4.938 4.920 4.917 4.843 3.831 3.829 3.816 3.813 3.799 3.785 3.782 2.123 2.114 2.111 2.101 2.098 2.089 1.937 1.921 1.916 1.907 1.904 1.901 1.900 1.887 1.885 1.750 1.741 1.725 1.667 1.655 1.650 1.643 1.638 1.630 1.625 1.617 1.605 1.596 1.589 1.583 1.577 1.568 1.556 1.182



S73

NAME	JMH-VII-LINEAR	
EXPNO	10	
PROCNO	1	
Date_	20121002	
Time	12.45	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	74012	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10273.973	Hz
FIDRES	0.138815	Hz
AQ	3.6019673	sec
RG	144	
DW	48.667	usec
DE	7.02	usec
TE	298.2	K
D1	0.50000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	1H	
P1	11.00	usec
PL1	1.10	dB
PL1W	18.32853889	W
SFO1	500.1930889	MHz
SI	131072	
SF	500.1900118	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

user Joanne Hewitt
JMH-VII-LINEAR
C13CPD1024.GLA CDCl3 /u joahew 44

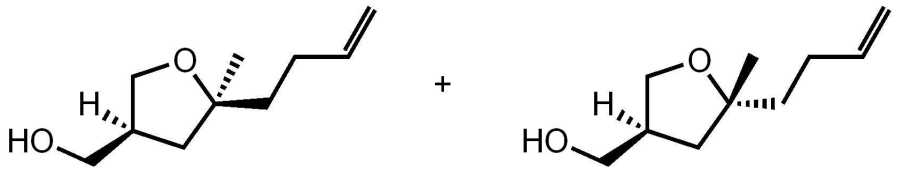


S73

NAME	JMH-VII-LINEAR
EXPNO	11
PROCNO	1
Date_	20121002
Time	13.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728576 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VII-67A
proton.gla CDCl3 /u joahew 15

5.840 5.827 5.040 5.037 5.034 5.006 5.003 5.000 4.949 4.947 4.945 4.943 4.929 4.927 4.925 4.923 4.002 3.987 3.984 3.969 3.964 3.946 3.653 3.642 3.634 3.629 3.621 3.611 3.607 3.596 3.593 2.114 2.105 2.102 2.094 1.857 1.841 1.833 1.816 1.673 1.662 1.652 1.644 1.640 1.632 1.621 1.610 1.480 1.463 1.455 1.438 1.248 1.180



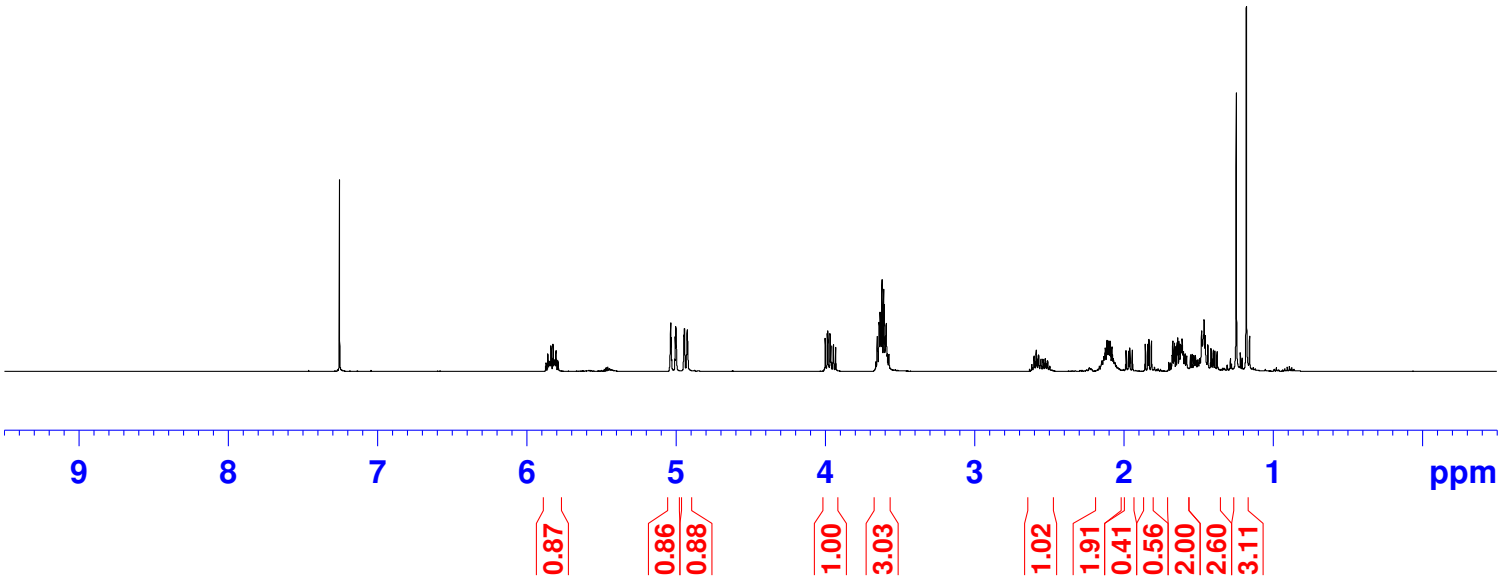
S74

+

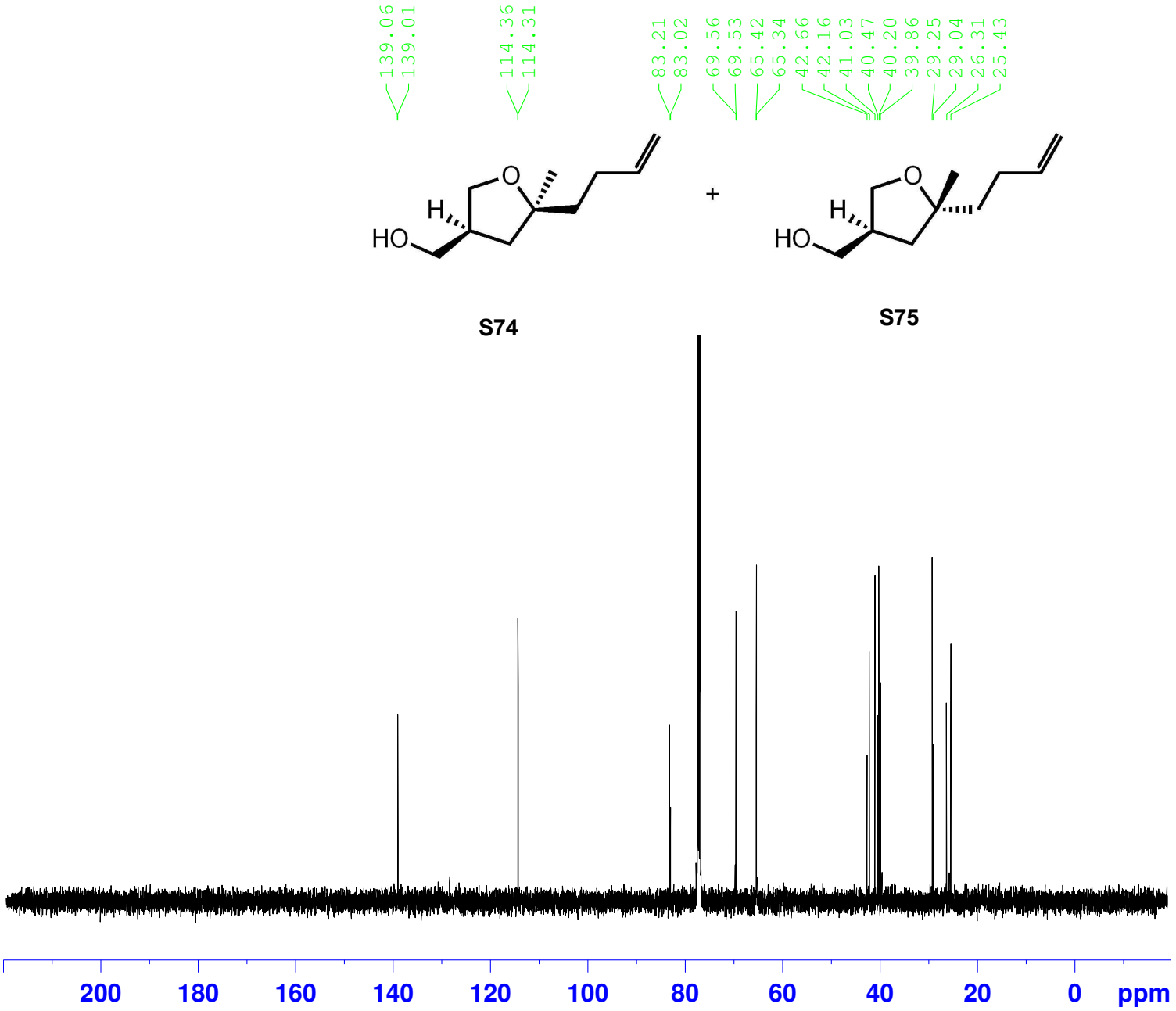
S75

NAME	JMH-VII-67A_2
EXPNO	10
PROCNO	1
Date_	20121009
Time	0.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	203
DW	48.667 usec
DE	7.02 usec
TE	298.3 K
D1	0.50000000 sec
TD0	1

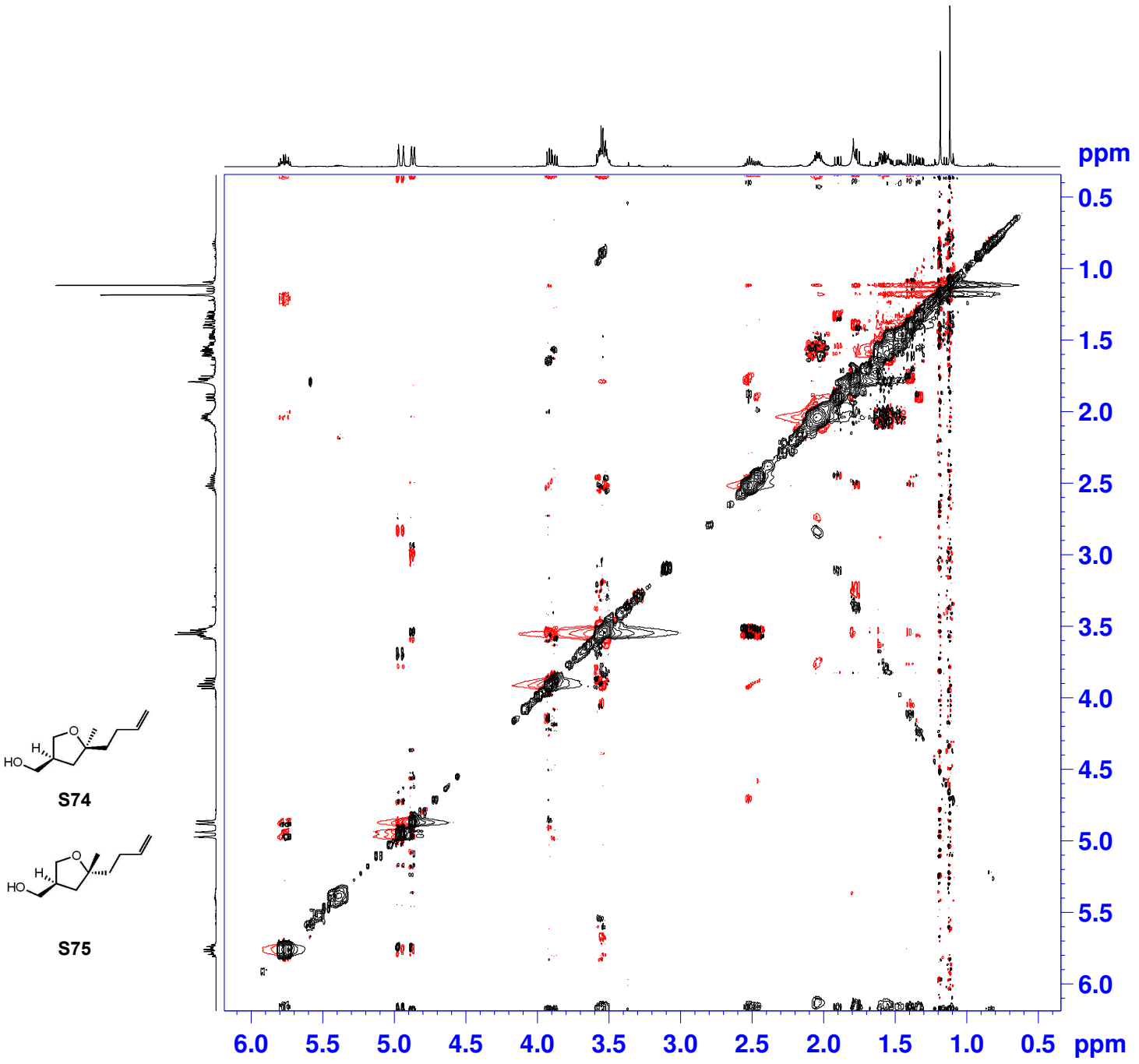
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900131 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



user Joanne Hewitt
JMH-VII-67A
C13CPD1024.GLA CDCl3 /u joahew 15



NAME	JMH-VII-67A	
EXPNO	11	
PROCNO	1	
Date_	20121009	
Time	1.40	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	30000.000	Hz
FIDRES	0.457764	Hz
AQ	1.0923166	sec
RG	2050	
DW	16.667	usec
DE	8.01	usec
TE	298.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	7.50	usec
PL1	-2.50	dB
PL1W	147.40557861	W
SFO1	125.7854522	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	1.00	dB
PL12	17.85	dB
PL13	20.00	dB
PL2W	18.75546646	W
PL12W	0.38737163	W
PL13W	0.23611732	W
SFO2	500.1920008	MHz
SI	32768	
SF	125.7728577	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

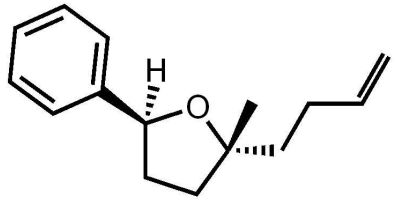


BRUKER

NAME	JMH-VI-58B
EXPNO	21
PROCNO	1
Date_	20121008
Time	14.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	noesygpphzs
TD	2048
SOLVENT	CDCl3
NS	2
DS	4
SWH	2923.977 Hz
FIDRES	1.427723 Hz
AQ	0.3502580 sec
RG	32
DW	171.000 usec
DE	25.34 usec
TE	298.2 K
D0	0.00015699 sec
D1	1.81526899 sec
D8	0.30000001 sec
D16	0.00020000 sec
IN0	0.00034200 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
P32	20000.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1916728 MHz
SP29	24.45 dB
SPNAM29	Crp60,20,20.10
SPOAL29	0.500
SPOFFS29	0.00 Hz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ0	10.00 %
GPZ1	40.00 %
P31	5000.00 usec
ND0	1
TD	256
SFO1	500.1917 MHz
FIDRES	11.421783 Hz
SW	5.846 ppm
FnMODE	States-TPPI
SI	2048
SF	500.1900396 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	500.1900396 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

user Joanne Hewitt
JMH-VI-44C
proton.gla CDCl3 /u joahew 45

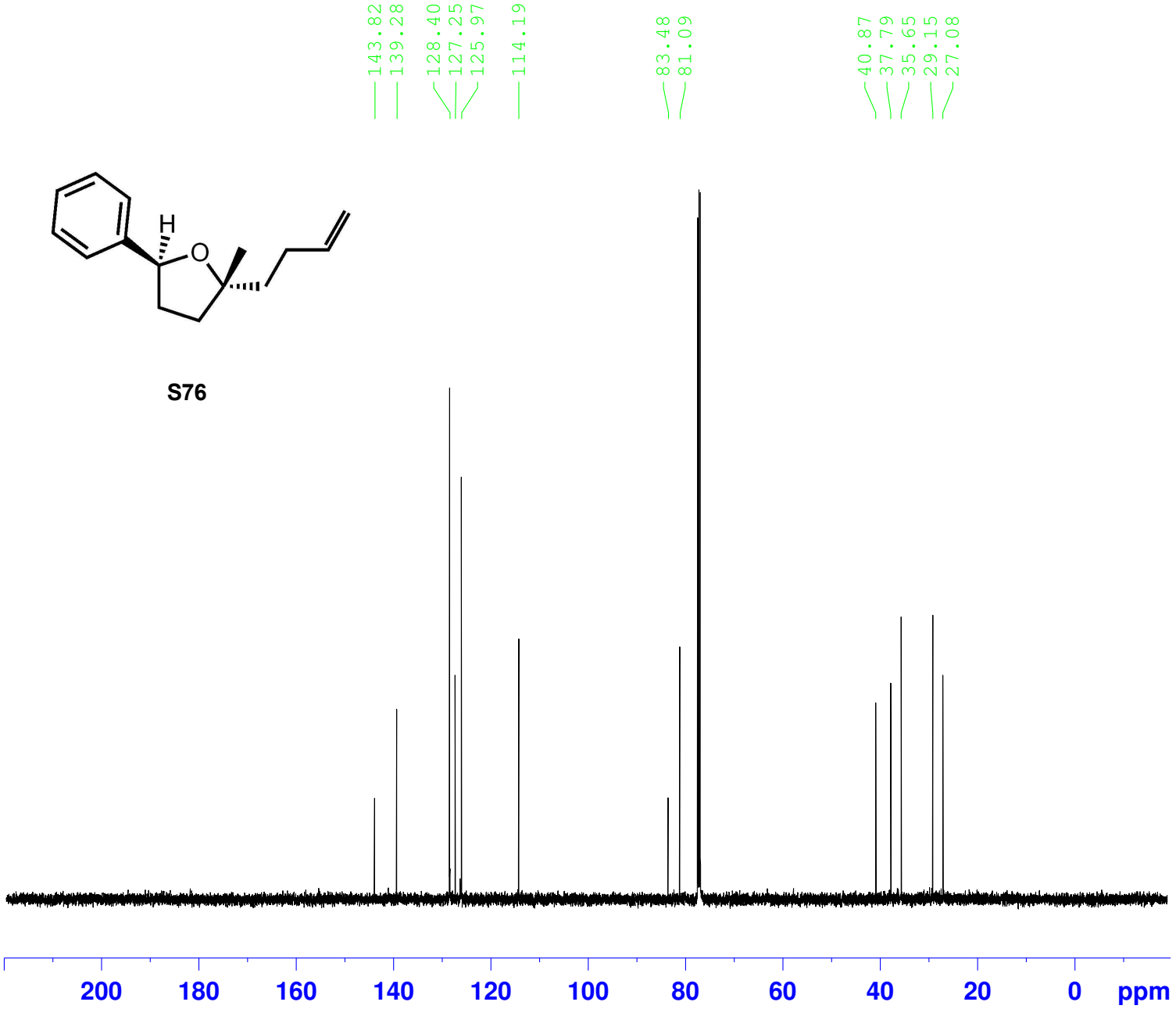
7.373 7.370 7.369 7.356 7.355 7.354 7.337 7.332 7.322 7.319 7.309 7.306 7.261 7.251 7.237 5.898 5.884 5.077 5.074 5.043 5.039 4.975 4.974 4.972 4.971 4.955 4.950 4.937 4.932 2.206 2.195 2.164 1.952 1.945 1.939 1.936 1.933 1.930 1.925 1.913 1.889 1.856 1.841 1.835 1.765 1.754 1.744 1.732 1.729 1.717 1.707 1.696 1.359



S76

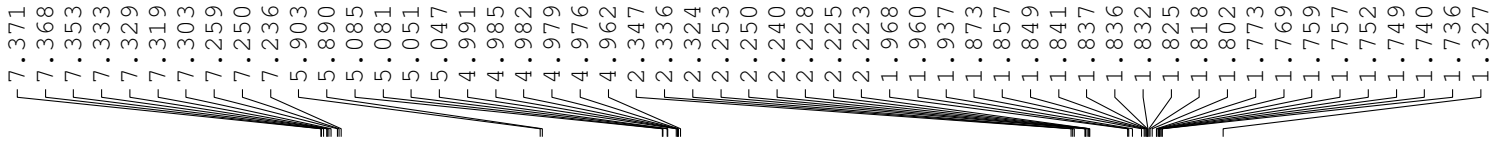
NAME	JMH-VI-44C_2
EXPNO	10
PROCNO	1
Date_	20120711
Time	14.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	128
DW	48.667 usec
DE	7.02 usec
TE	295.2 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900103 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

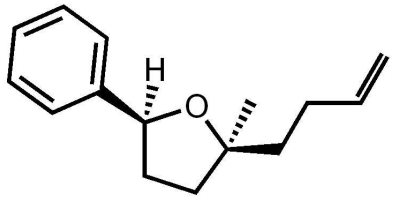
user Joanne Hewitt
JMH-VI-44C
C13CPD256.GLA CDCl3 /u joahew 45



NAME	JMH-VI-44C
EXPNO	11
PROCNO	1
Date_	20120711
Time	14.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728513 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-44D
proton.gla CDCl3 /u joahew 46

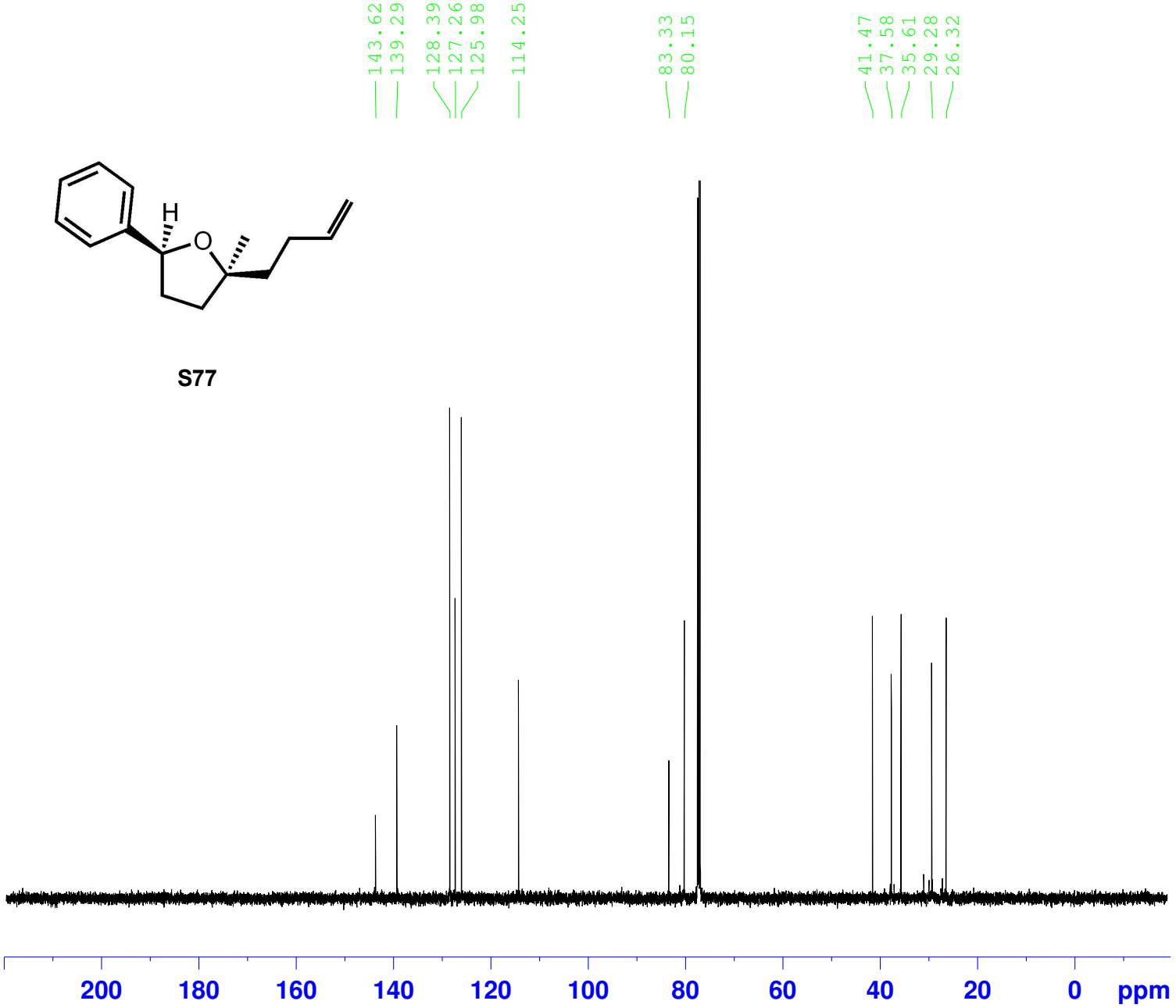




S77

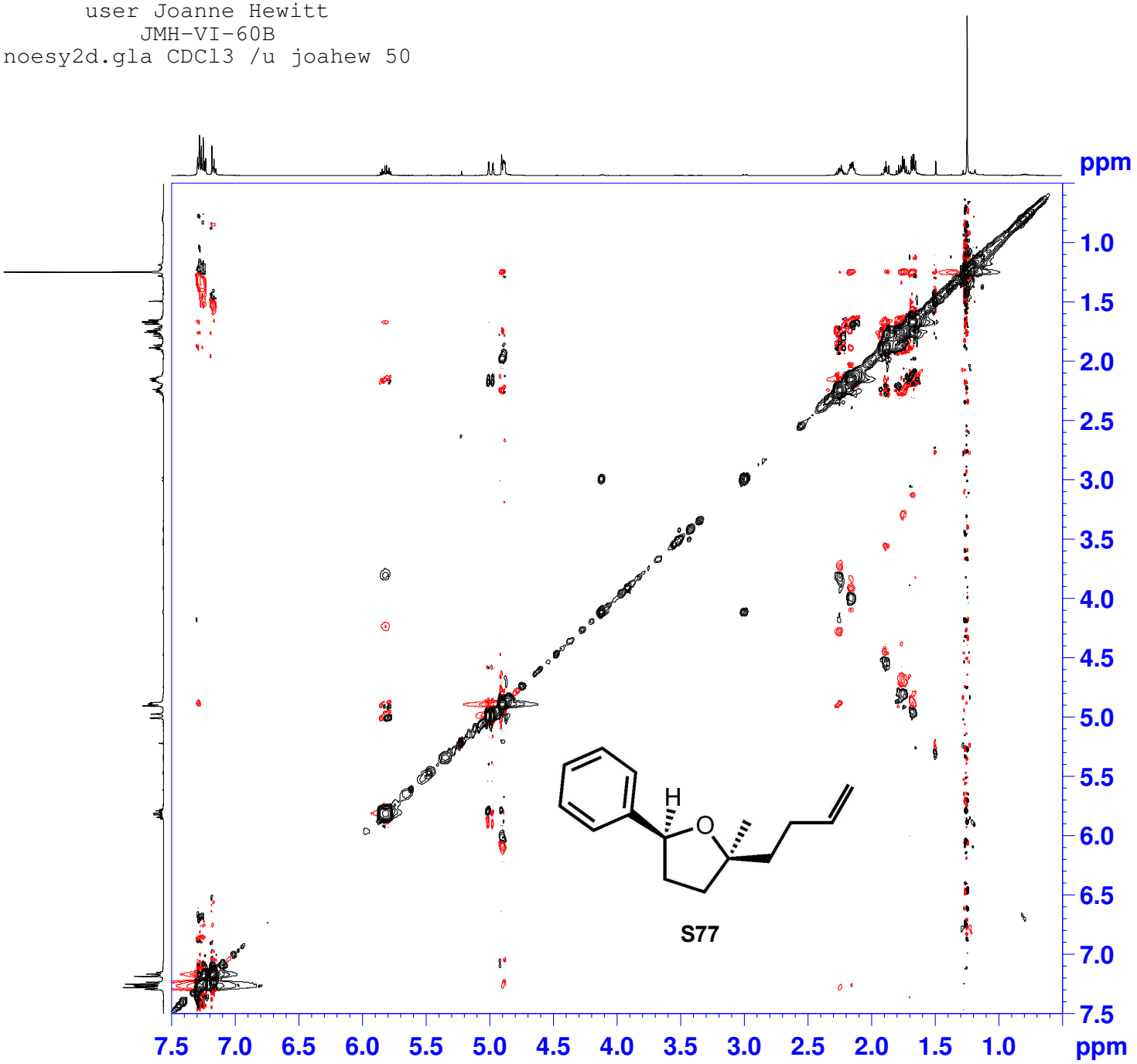
NAME	JMH-VI-44D_2
EXPNO	10
PROCNO	1
Date_	20120712
Time	2.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	114
DW	48.667 usec
DE	7.02 usec
TE	295.3 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900110 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VI-44D
C13CPD256.GLA CDCl3 /u joahew 46



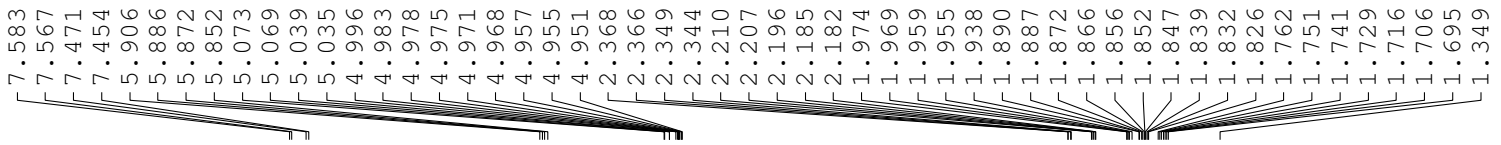
NAME	JMH-VI-44D
EXPNO	11
PROCNO	1
Date_	20120712
Time	2.33
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728501 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

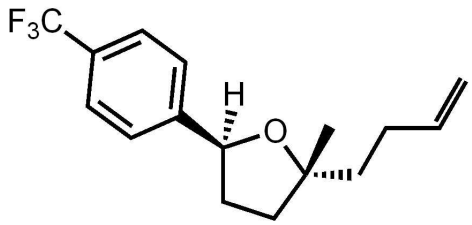
user Joanne Hewitt
JMH-VI-60B
noesy2d.gla CDCl3 /u joahew 50



NAME	JMH-VI-60B
EXPNO	11
PROCNO	1
Date_	20120829
Time	9.17
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	noesygpphzs
TD	2048
SOLVENT	CDCl3
NS	2
DS	4
SWH	3787.879 Hz
FIDRES	1.849550 Hz
AQ	0.2703860 sec
RG	64
DW	132.000 usec
DE	17.93 usec
TE	271.4 K
D0	0.00011799 sec
D1	1.89514101 sec
D8	0.30000001 sec
D16	0.00020000 sec
IN0	0.00026400 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
P32	20000.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1920604 MHz
SP29	24.45 dB
SPNAM29	Crp60,20,20.10
SPOAL29	0.500
SPOFFS29	0.00 Hz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ0	10.00 %
GPZ1	40.00 %
P31	5000.00 usec
ND0	1
TD	256
SFO1	500.1921 MHz
FIDRES	14.796402 Hz
SW	7.573 ppm
FnMODE	States-TPPI
SI	2048
SF	500.1900501 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

user Joanne Hewitt
JMH-VI-81A D1
proton.gla CDCl3 /u joahew 19



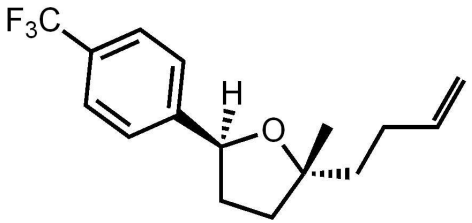


S78

NAME	JMH-VI-81A
EXPNO	10
PROCNO	1
Date_	20120822
Time	11.22
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	203
DW	48.667 usec
DE	7.02 usec
TE	295.2 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900121 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VI-81A D1
C13CPD1024.GLA CDCl3 /u joahew 19

148.16
139.11
129.74
129.49
126.12
125.58
125.44
125.41
125.38
125.35
123.41
114.35
84.02
80.32
40.81
37.73
35.60
29.15
26.98

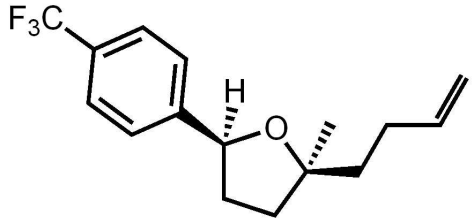


S78

NAME	JMH-VI-81A
EXPNO	11
PROCNO	1
Date_	20120823
Time	5.46
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728470 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-81B D2
proton.gla CDCl3 /u joahew 21

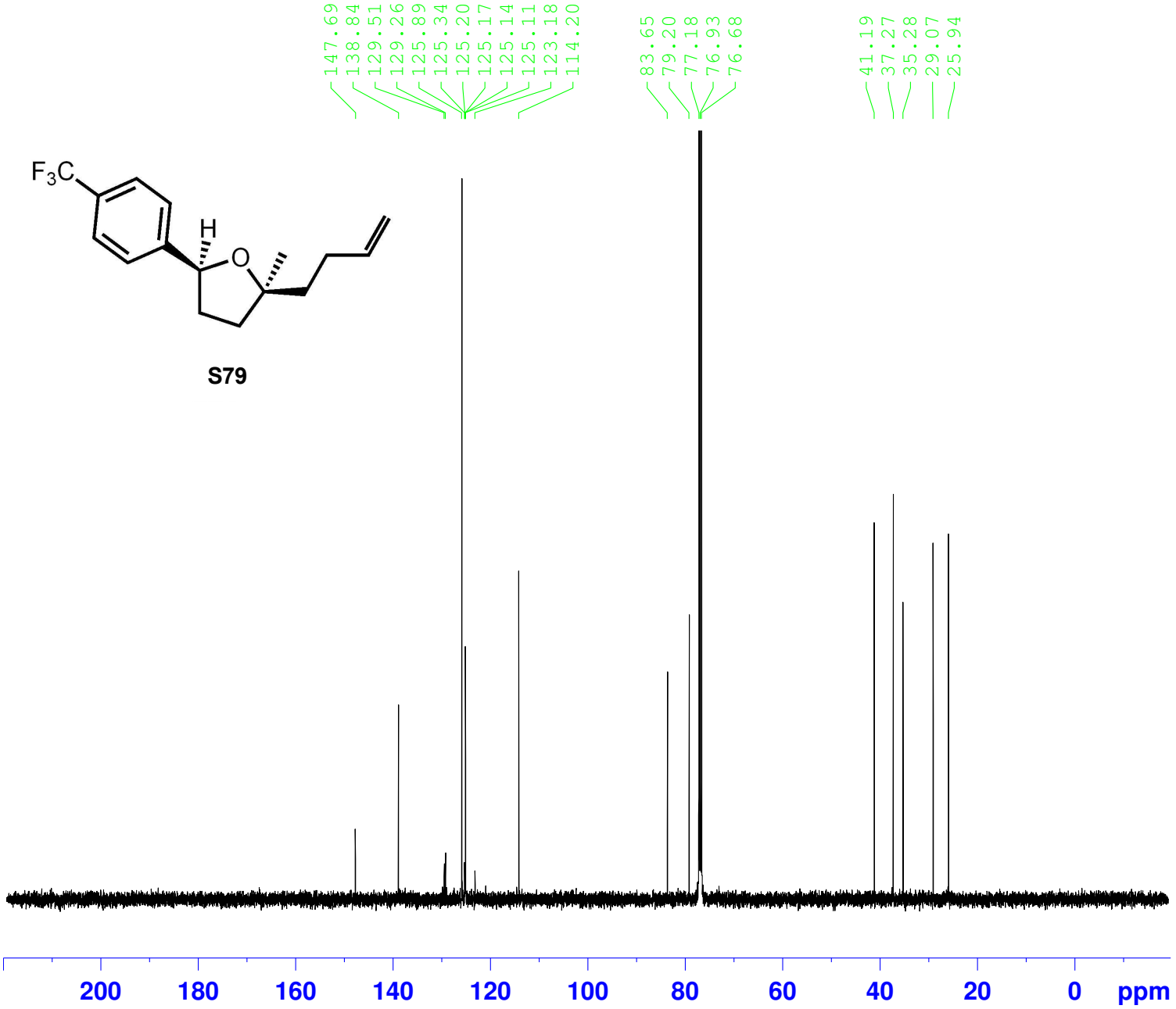
7.587 7.570 7.473 7.457 5.913 5.892 5.879 5.858 5.082 5.078 5.048 5.044 5.041 5.027 5.013 4.991 4.989 4.985 4.971 4.968 4.965 2.390 2.381 2.377 2.244 2.241 2.238 2.224 2.213 2.210 2.208 1.959 1.952 1.944 1.937 1.862 1.845 1.841 1.835 1.832 1.825 1.822 1.818 1.812 1.795 1.770 1.764 1.757 1.751 1.744 1.738 1.731 1.331



S79

NAME	JMH-VI-81B_2
EXPNO	10
PROCNO	1
Date_	20120821
Time	23.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	203
DW	48.667 usec
DE	7.02 usec
TE	295.4 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900108 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Joanne Hewitt
JMH-VI-81B D2
C13CPD1024.GLA CDCl3 /u joahew 21

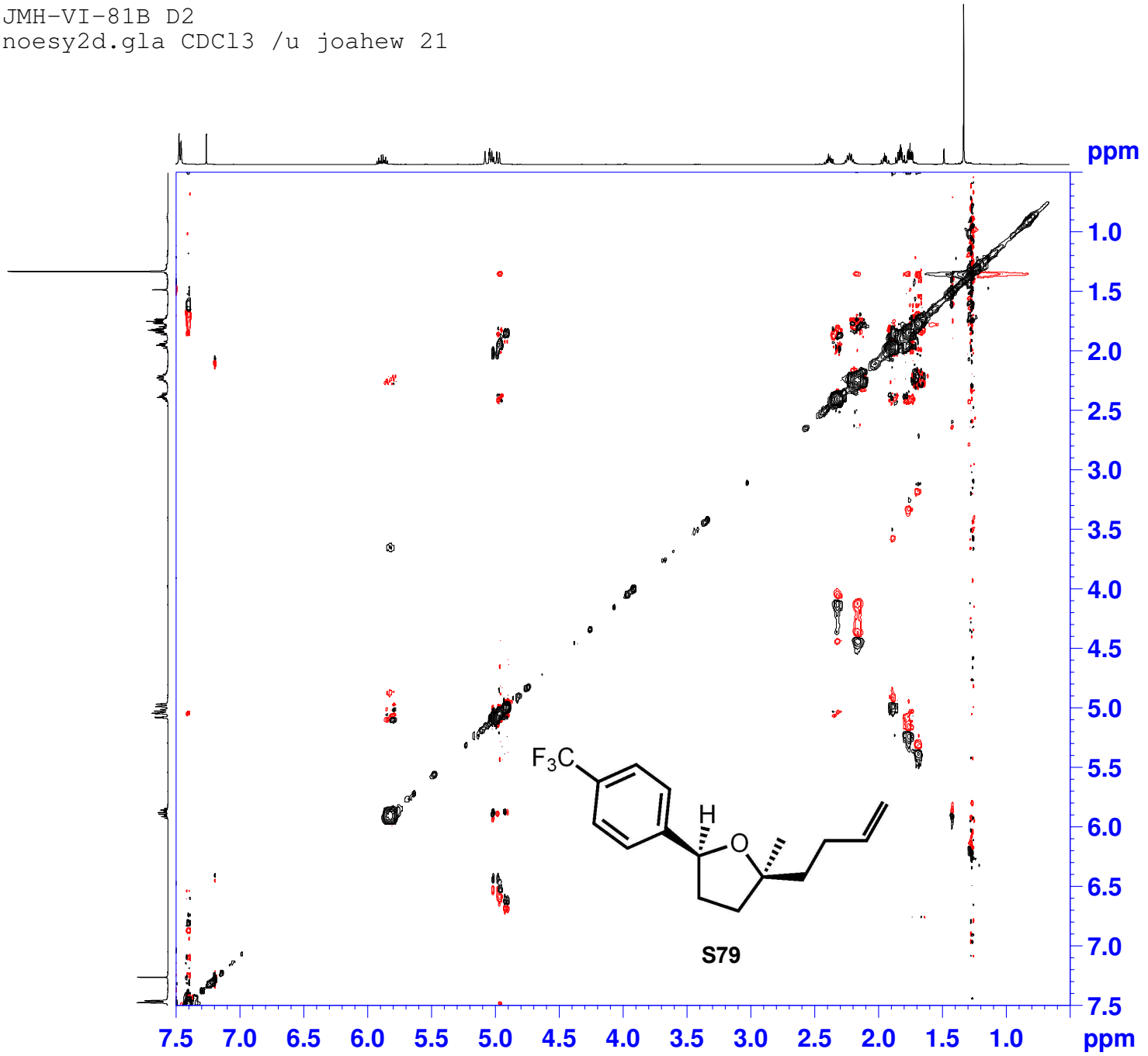


S79

NAME	JMH-VI-81B
EXPNO	11
PROCNO	1
Date_	20120822
Time	0.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728760 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Electronic Supplementary Material (ESI) for Chemical Science

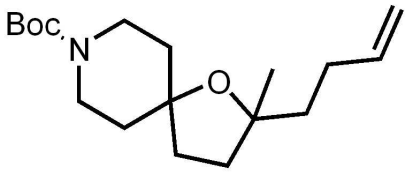
user Joanne Hewitt
JMH-VI-81B D2
noesy2d.gla CDCl3 /u joahew 21



NAME	JMH-VI-81B
EXPNO	14
PROCNO	1
Date_	20120822
Time	1.12
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	noesygpphzs
TD	2048
SOLVENT	CDCl3
NS	2
DS	4
SWH	3875.969 Hz
FIDRES	1.892563 Hz
AQ	0.2642420 sec
RG	114
DW	129.000 usec
DE	17.37 usec
TE	295.1 K
D0	0.00011499 sec
D1	1.90128601 sec
D8	0.30000001 sec
D16	0.00020000 sec
IN0	0.00025800 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
P32	20000.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1921318 MHz
SP29	24.45 dB
SPNAM29	Crp60,20,20.10
SPOAL29	0.500
SPOFFS29	0.00 Hz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ0	10.00 %
GPZ1	40.00 %
P31	5000.00 usec
ND0	1
TD	256
SFO1	500.1921 MHz
FIDRES	15.140504 Hz
SW	7.749 ppm
FnMODE	States-TPPI
SI	2048
SF	500.1900450 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	500.1900000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

user Joanne Hewitt
JMH-VIII-48B
proton.gla CDCl3 /u joahew 25

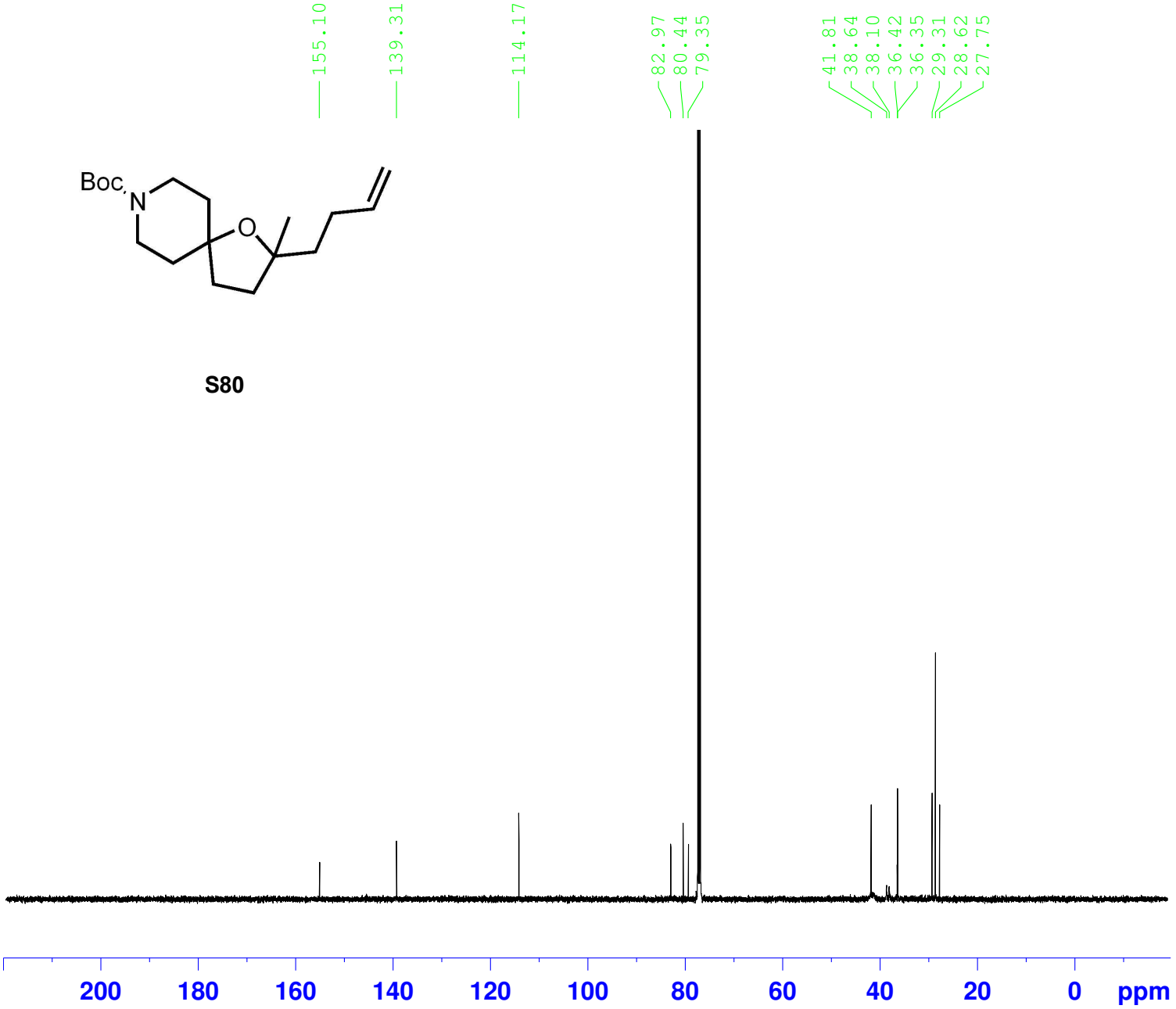
5.842 5.829 5.808 5.028 5.024 4.994 4.990 4.941 4.937 4.920 4.917 3.457 3.449 3.441 3.437 3.432 3.429 3.424 3.419 3.411 2.113 2.110 2.100 2.087 1.880 1.865 1.863 1.853 1.841 1.836 1.823 1.809 1.803 1.794 1.764 1.748 1.744 1.731 1.590 1.578 1.574 1.569 1.563 1.557 1.553 1.548 1.541 1.535 1.527 1.486 1.479 1.446 1.194



S80

NAME	JMH-VIII-48B_2	
EXPNO	10	
PROCNO	1	
Date_	20130116	
Time	8.21	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	74012	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10273.973	Hz
FIDRES	0.138815	Hz
AQ	3.6019673	sec
RG	161	
DW	48.667	usec
DE	7.02	usec
TE	300.8	K
D1	0.50000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	1H	
P1	11.00	usec
PL1	1.10	dB
PL1W	18.32853889	W
SFO1	500.1930889	MHz
SI	131072	
SF	500.1900111	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

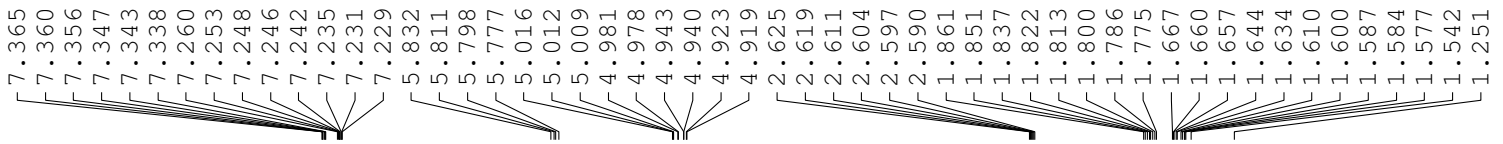
user Joanne Hewitt
JMH-VIII-48B
C13CPD1024.GLA CDCl3 /u joahew 25

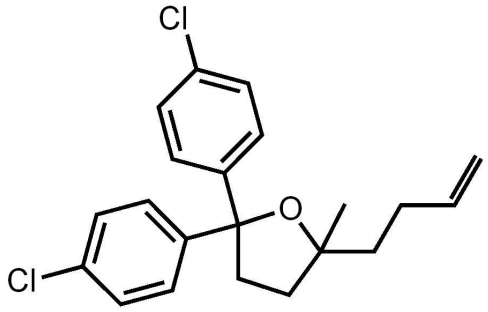


S80

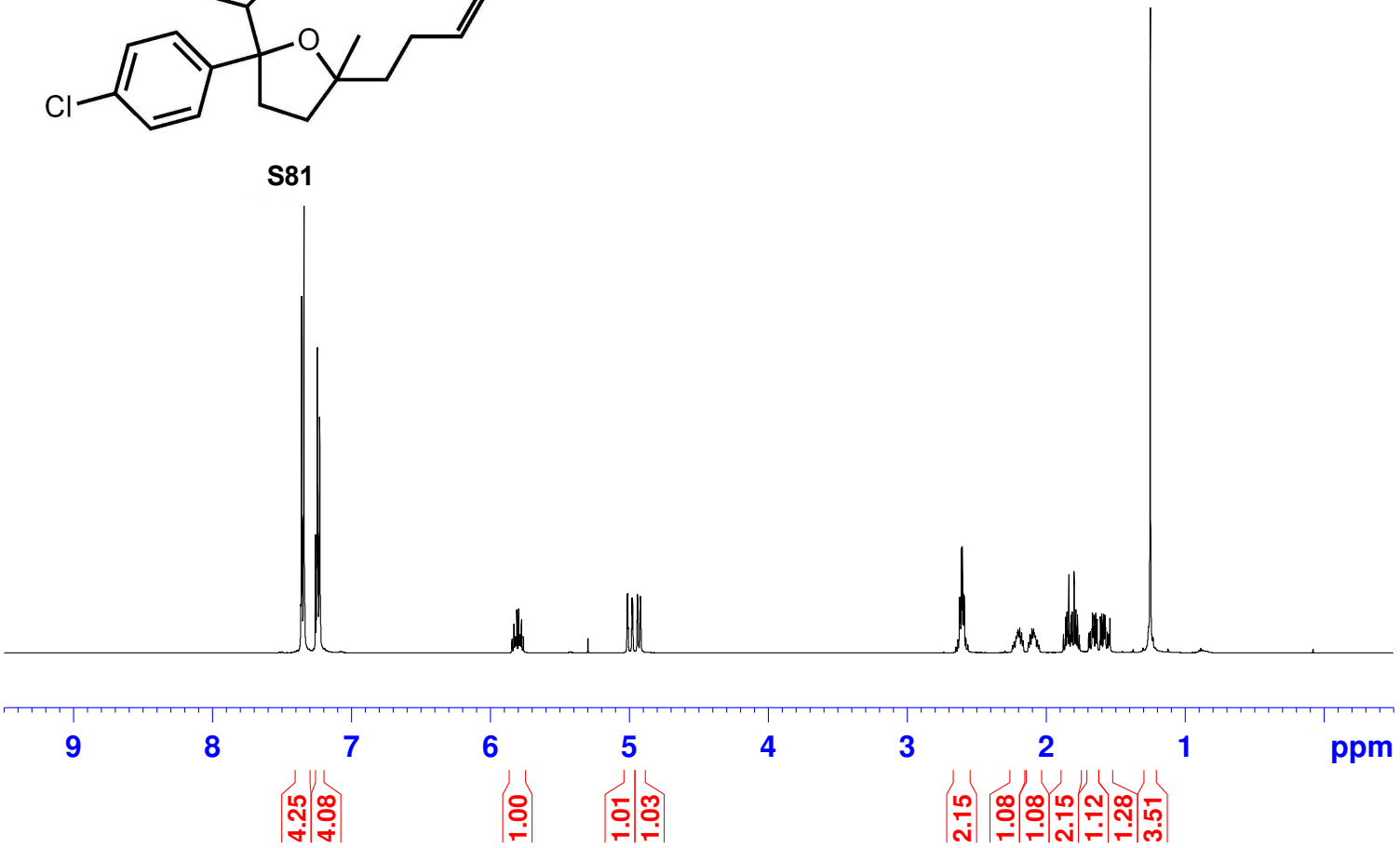
```
NAME          JMH-VIII-48B
EXPNO                   11
PROCNO                   1
Date_             20130116
Time                 10.19
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH             30000.000 Hz
FIDRES          0.457764 Hz
AQ              1.0923166 sec
RG                    2050
DW                  16.667 usec
DE                    8.01 usec
TE                   301.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    7.50 usec
PL1                  -2.50 dB
PL1W         147.40557861 W
SFO1          125.7854522 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 17.85 dB
PL13                 20.00 dB
PL2W          18.75546646 W
PL12W          0.38737163 W
PL13W          0.23611732 W
SFO2          500.1920008 MHz
SI                   32768
SF            125.7728558 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

user Joanne Hewitt
JMH-VII-65B
proton.gla CDCl3 /u joahew 2





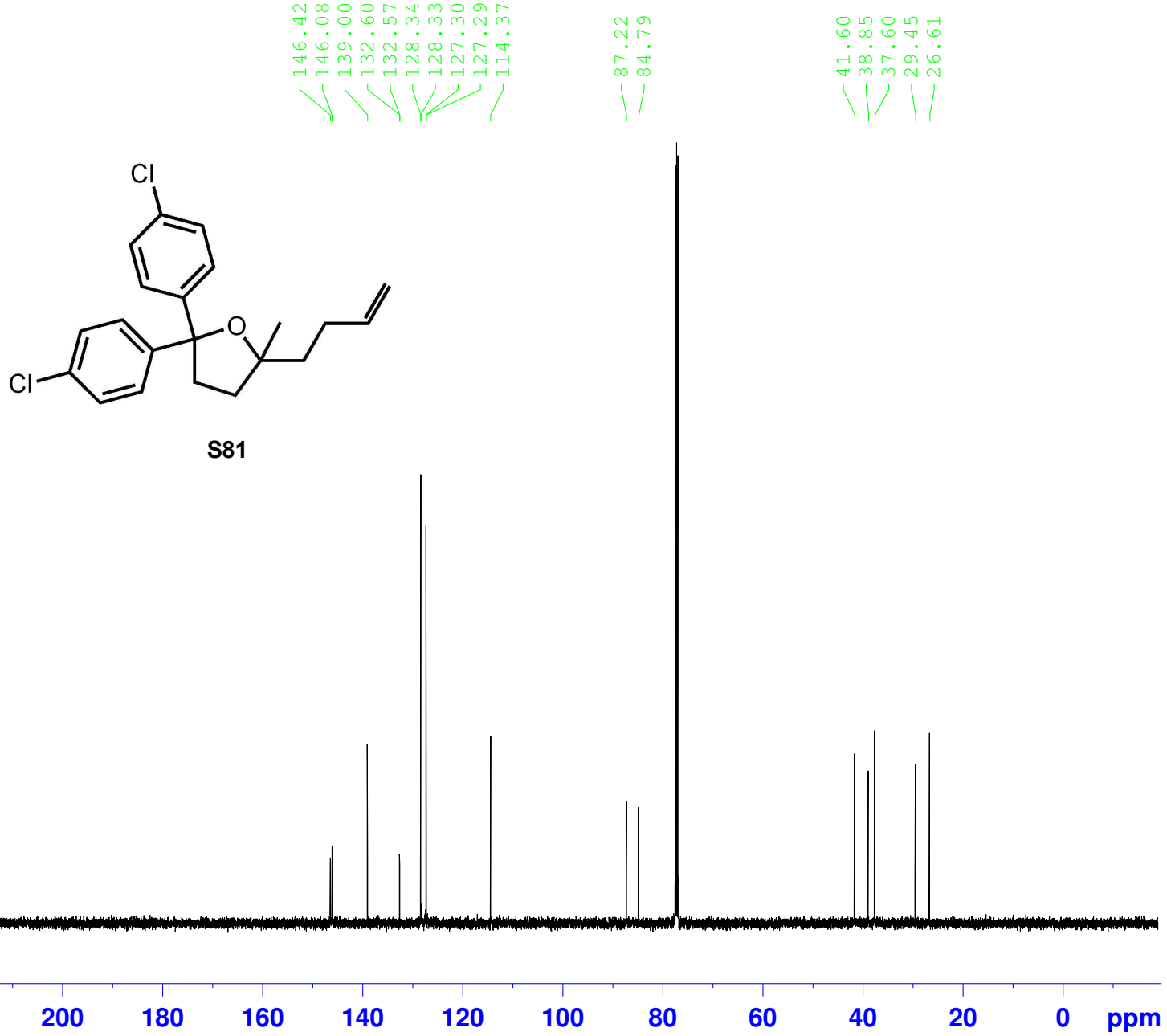
S81



NAME	JMH-VII-65B_2
EXPNO	10
PROCNO	1
Date_	20121005
Time	16.25
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	128
DW	48.667 usec
DE	7.02 usec
TE	298.2 K
D1	0.50000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900112 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

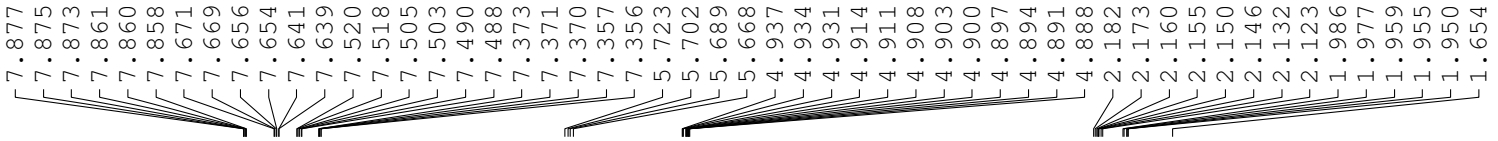
user Joanne Hewitt
JMH-VII-65B
C13CPD256.GLA CDCl3 /u joahew 2

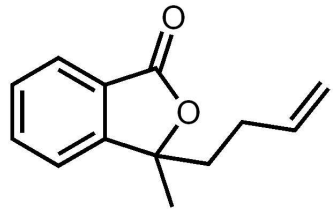


S81

NAME	JMH-VII-65B
EXPNO	11
PROCNO	1
Date_	20121005
Time	16.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728585 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Joanne Hewitt
JMH-VI-82B
proton.gla CDCl3 /u joahew 19

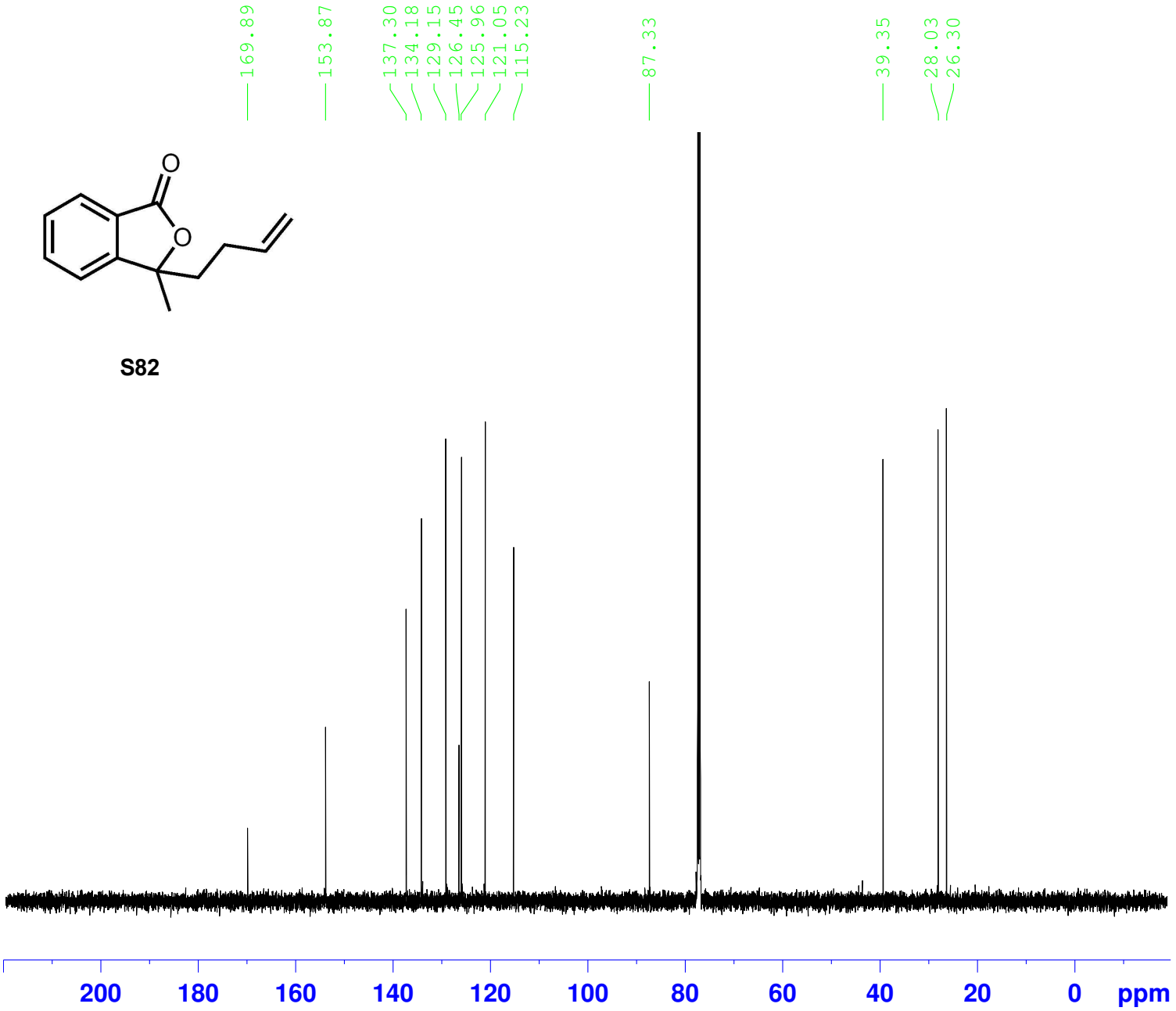




S82

NAME	JMH-VI-82B
EXPNO	20
PROCNO	1
Date_	20120821
Time	22.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	228
DW	48.667 usec
DE	7.02 usec
TE	295.4 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900113 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

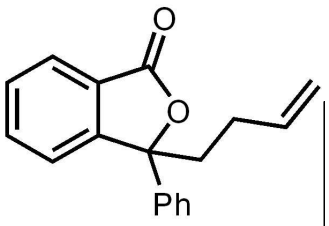
user Joanne Hewitt
JMH-VI-82B
C13CPD1024.GLA CDCl3 /u joahew 19



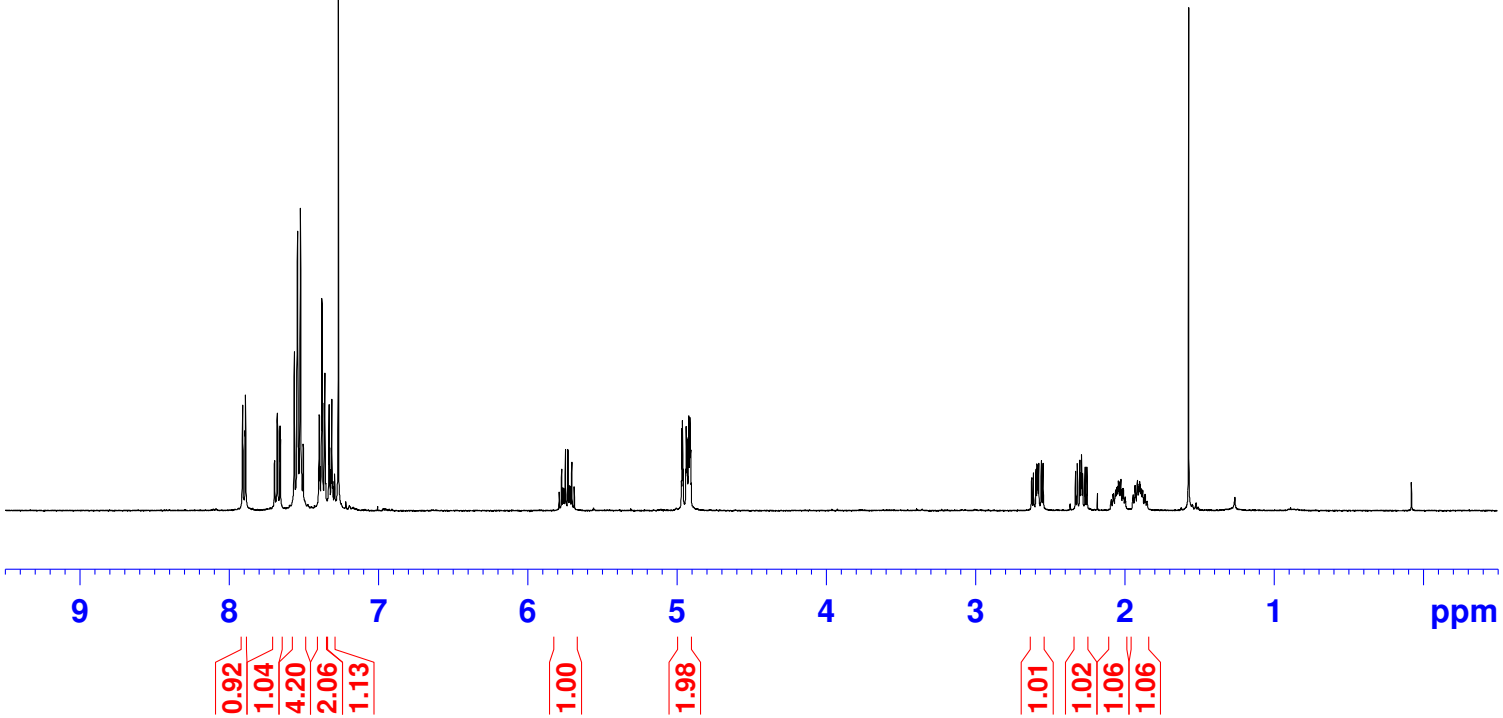
NAME	JMH-VI-82B_2
EXPNO	10
PROCNO	1
Date_	20120821
Time	9.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728478 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

User Craig Smith
CDS-I-74D
PROTON_C_A3.gla CDCl3 u craigsm 1

7.835 7.832 7.830 7.816 7.814 7.620 7.617 7.601 7.598 7.582 7.580 7.486 7.484 7.467 7.464 7.459 7.446 7.445 7.428 7.426 7.319 7.301 7.297 7.285 7.282 7.253 7.235 5.695 5.669 5.652 5.627 4.888 4.884 4.880 4.863 4.859 4.856 4.846 4.841 4.837 4.834 4.830 2.516 2.510 2.504 2.498 2.480 2.469 2.239 2.222 2.210 2.187 2.175

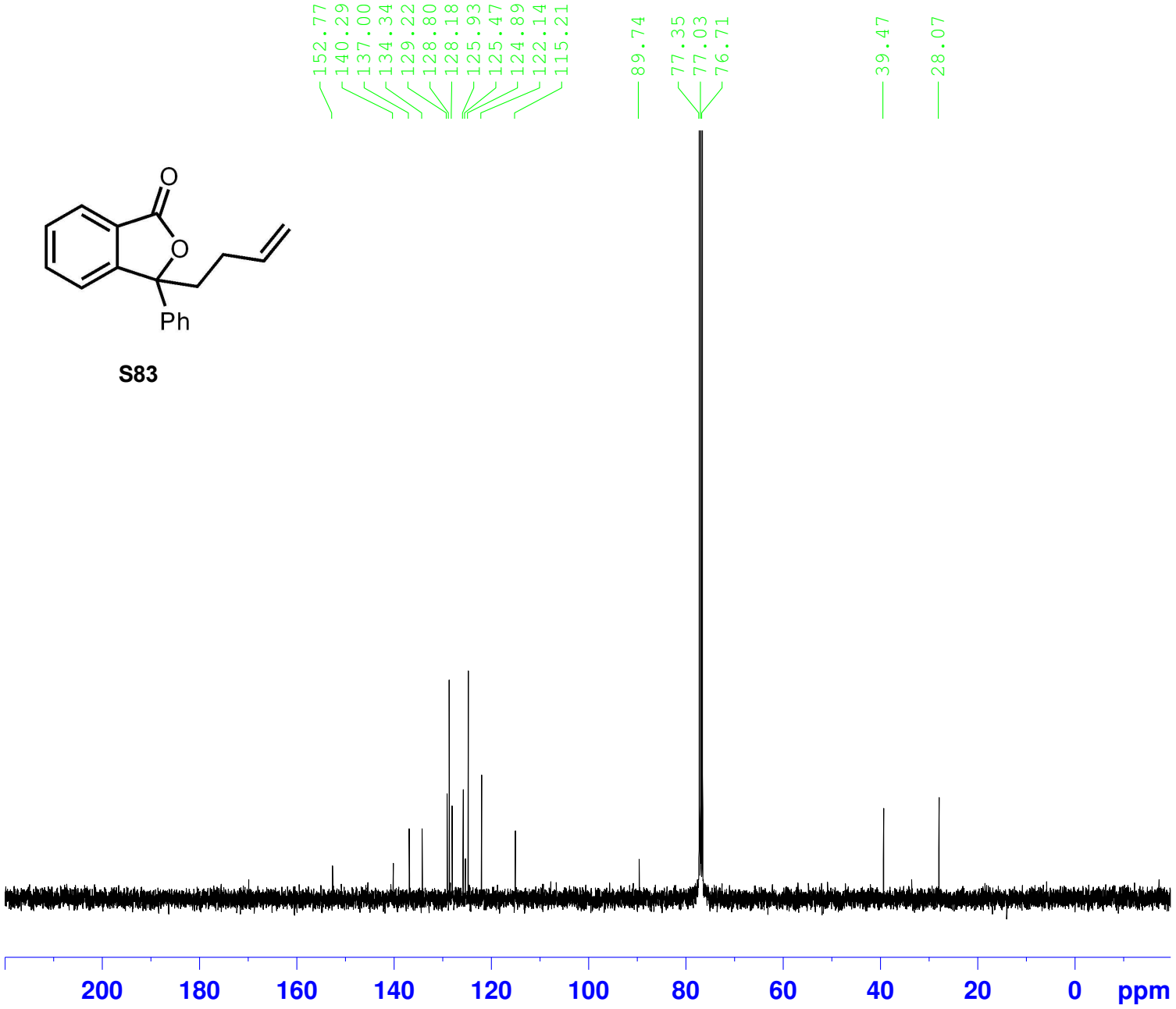


S83



NAME	CDS-I-74D	
EXPNO	10	
PROCNO	1	
Date_	20130213	
Time	22.35	
INSTRUM	AV400	
PROBHD	5 mm QNP 1H/13	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8278.146	Hz
FIDRES	0.126314	Hz
AQ	3.9584243	sec
RG	1149.4	
DW	60.400	usec
DE	6.00	usec
TE	294.0	K
D1	0.01000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.00	usec
PL1	0.00	dB
SFO1	400.1324708	MHz
SI	32768	
SF	400.1300056	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	4.00	

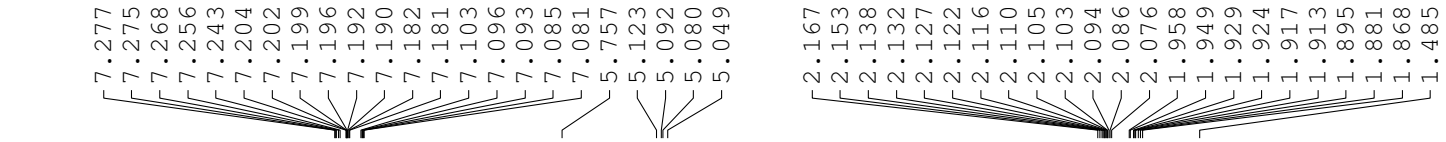
User Craig Smith
CDS-I-74D
C13CPD_320_A3.gla CDCl3 u craigsm 1

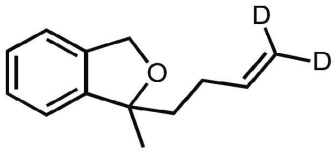


S83

NAME	CDS-I-74D
EXPNO	16
PROCNO	1
Date_	20130213
Time	23.50
INSTRUM	AV400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1000
DS	4
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	4597.6
DW	15.700 usec
DE	6.00 usec
TE	294.0 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.30 usec
PL1	0.00 dB
SFO1	100.6228303 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	94.00 usec
PL2	0.00 dB
PL12	17.00 dB
PL13	22.00 dB
SFO2	400.1316005 MHz
SI	32768
SF	100.6127796 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

User Lewis Williams
PROTON_C_A3.gla CDCl3 u lewiwi 30

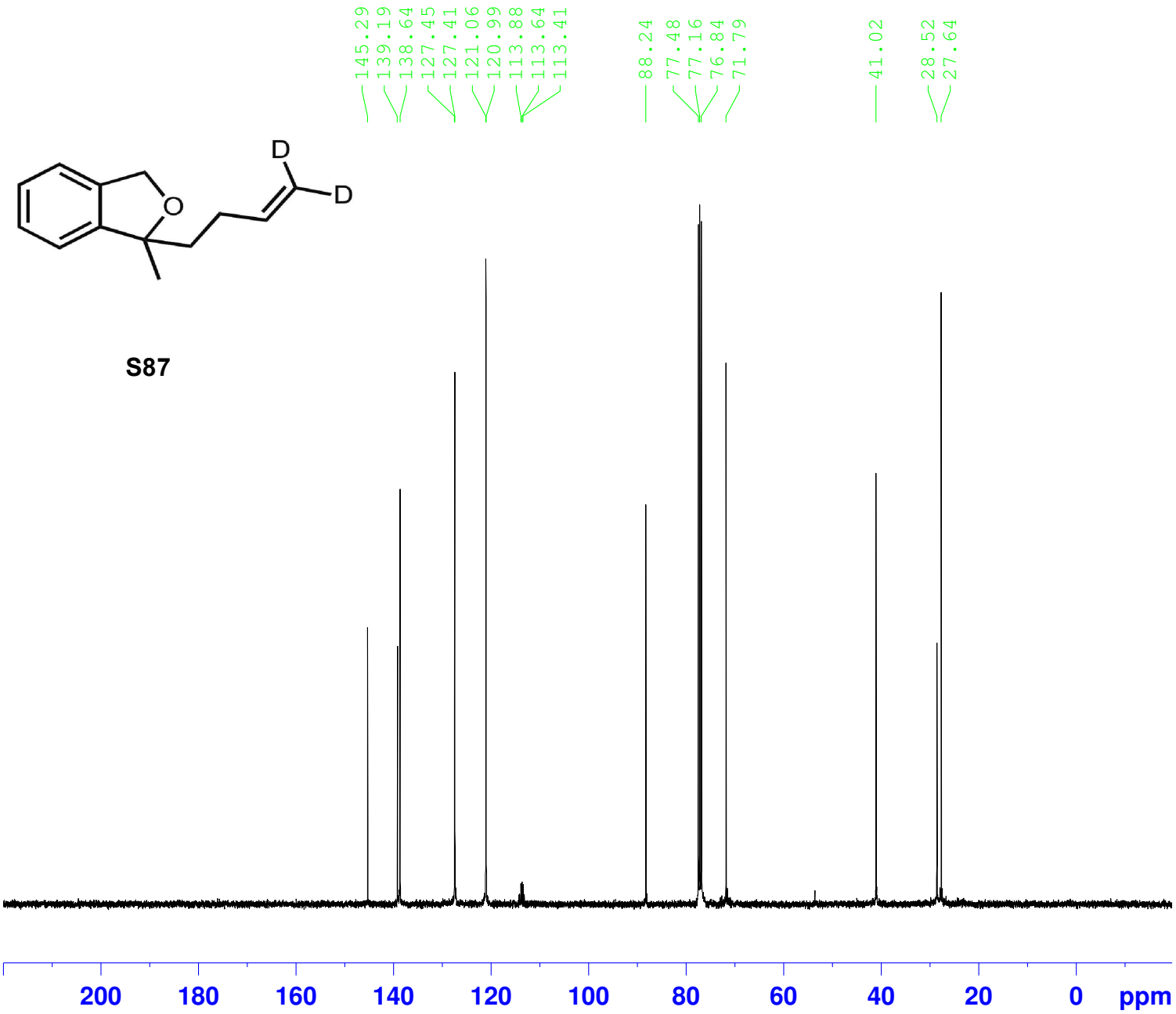




S87

NAME	LW6-46	
EXPNO	70	
PROCNO	1	
Date_	20130228	
Time	17.30	
INSTRUM	AV400	
PROBHD	5 mm QNP 1H/13	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8278.146	Hz
FIDRES	0.126314	Hz
AQ	3.9584243	sec
RG	1149.4	
DW	60.400	usec
DE	6.00	usec
TE	294.0	K
D1	0.01000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.00	usec
PL1	0.00	dB
SFO1	400.1324708	MHz
SI	32768	
SF	400.1300098	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	4.00	

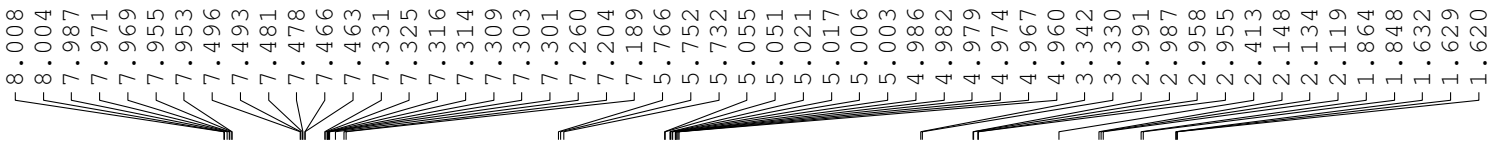
User Lewis Williams
C13CPD_320_A3.gla CDCl3 u lewiwi 5

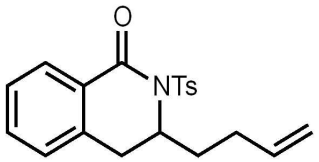


S87

NAME	LW6-46
EXPNO	61
PROCNO	1
Date_	20130228
Time	4.34
INSTRUM	AV400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	6502
DW	15.700 usec
DE	6.00 usec
TE	293.3 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.30 usec
PL1	0.00 dB
SFO1	100.6228303 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	94.00 usec
PL2	0.00 dB
PL12	17.00 dB
PL13	22.00 dB
SFO2	400.1316005 MHz
SI	32768
SF	100.6127592 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Lewis Williams
proton.gla CDCl3 /u lewiwi 13

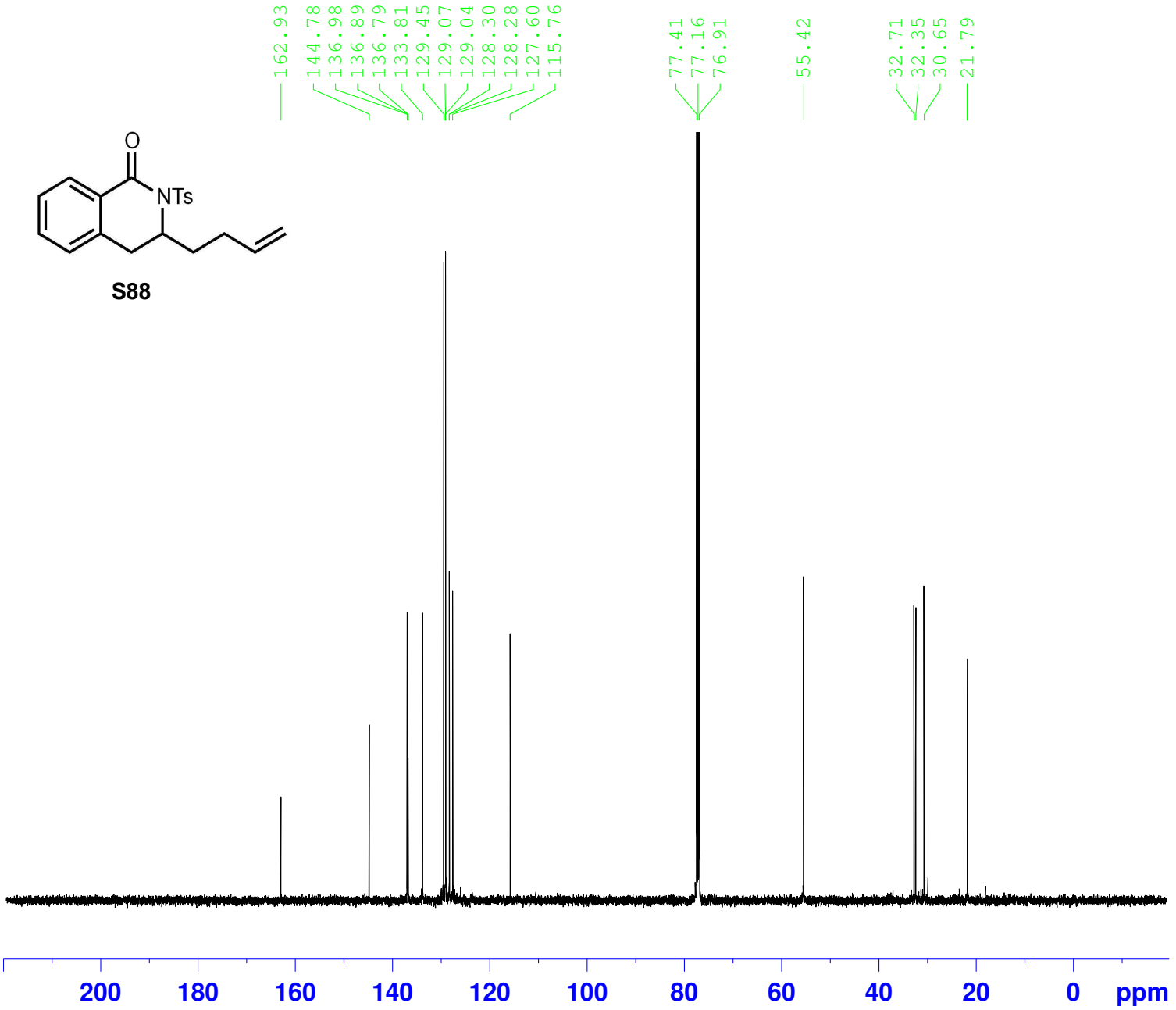




S88

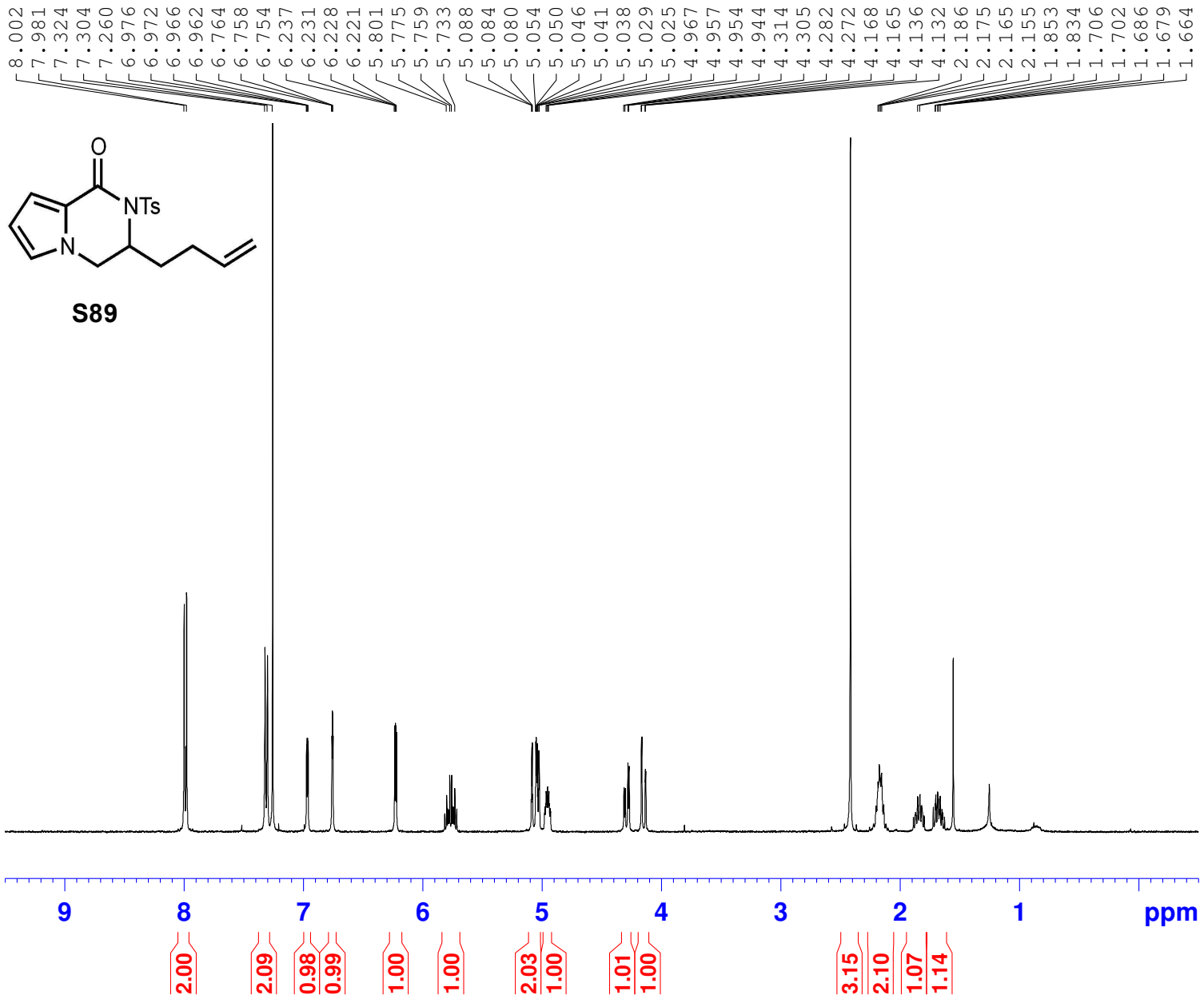
NAME	LW6-06
EXPNO	30
PROCNO	1
Date_	20130220
Time	16.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	228
DW	48.667 usec
DE	7.02 usec
TE	295.6 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Lewis Williams
C13CPD1024.GLA CDCl3 /u lewiwi 53



NAME	LW6-06
EXPNO	21
PROCNO	1
Date_	20130220
Time	9.17
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728614 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

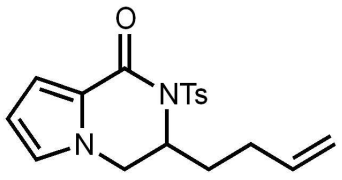
User Lewis Williams
PROTON_C_A3.gla CDCl3 u lewiwi 30



S89

NAME	LW6-21_4
EXPNO	10
PROCNO	1
Date_	20130214
Time	13.57
INSTRUM	AV400
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	1290.2
DW	60.400 usec
DE	6.00 usec
TE	294.2 K
D1	0.01000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
SFO1	400.1324708 MHz
SI	32768
SF	400.1300098 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

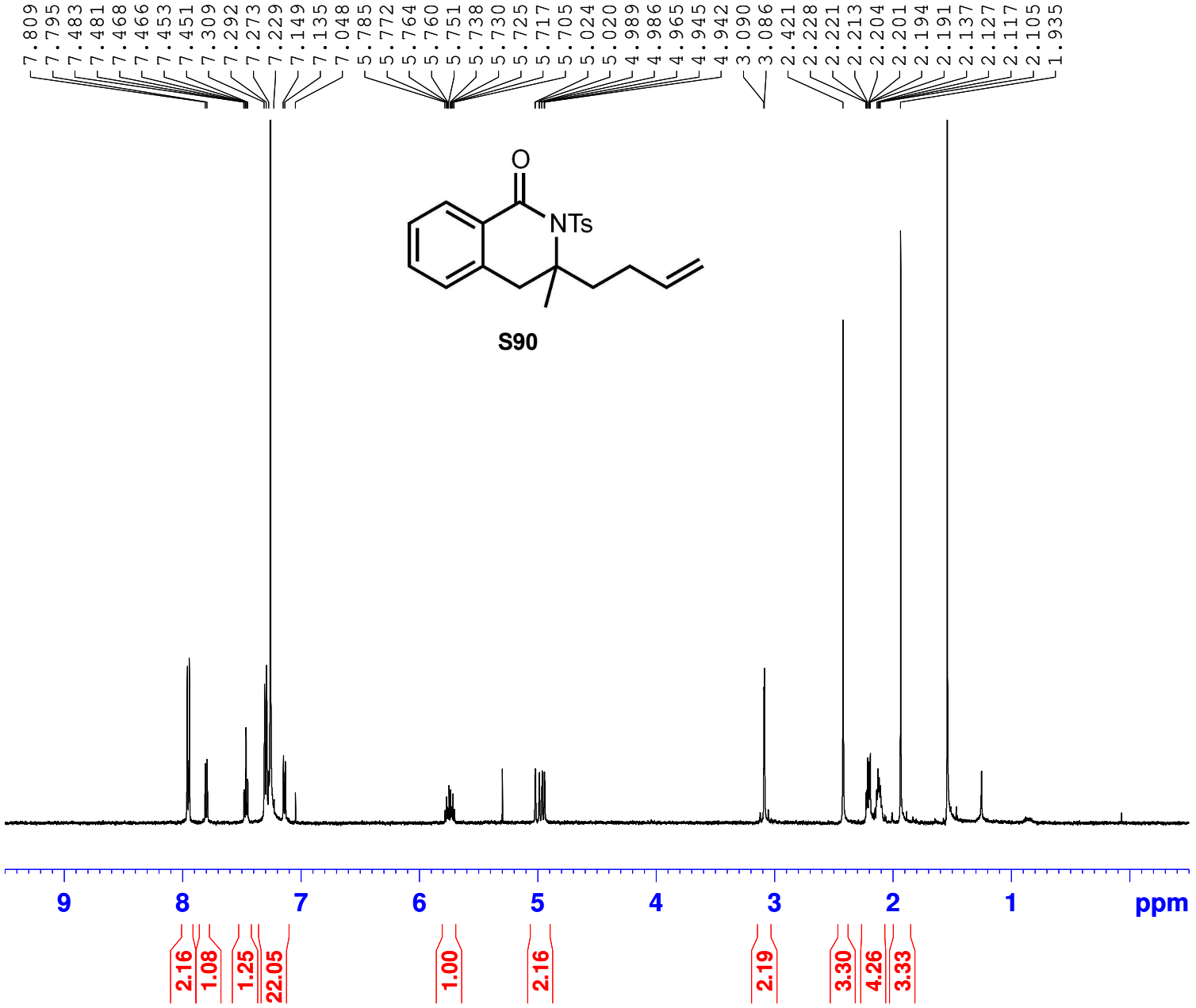
user Lewis Williams
C13CPD1024.GLA CDCl3 /u lewiwi 51



S89

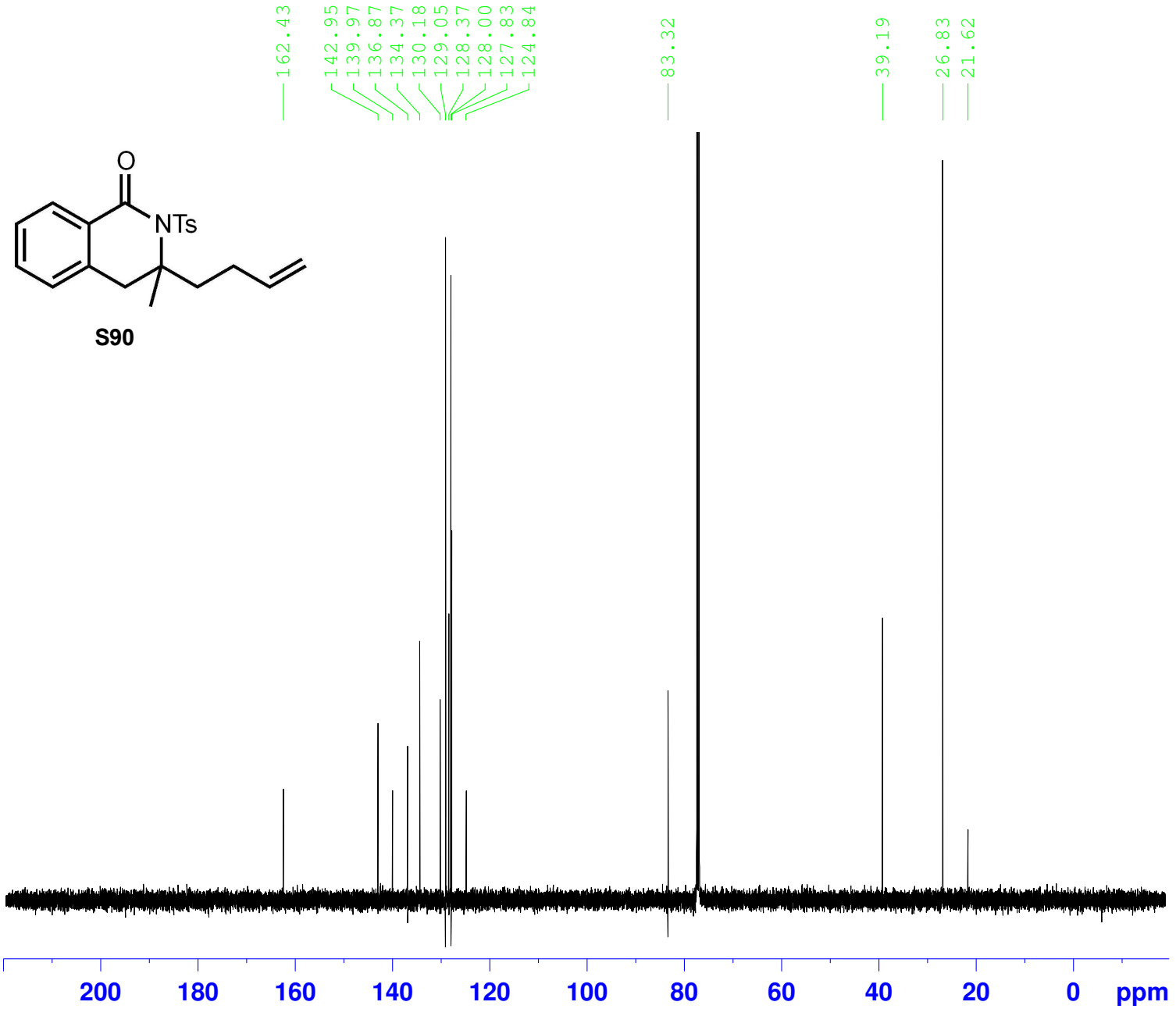
NAME	LW6-21
EXPNO	21
PROCNO	1
Date_	20130211
Time	1.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728606 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

user Lewis Williams
proton.gla CDCl3 /u lewiwi 18



NAME	LW4-24_3
EXPNO	20
PROCNO	1
Date_	20130430
Time	1.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	322
DW	48.667 usec
DE	7.02 usec
TE	295.7 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

user Lewis Williams
PdII red. elim. product
C13CPD1024.GLA CDCl3 /u lewiwi 31

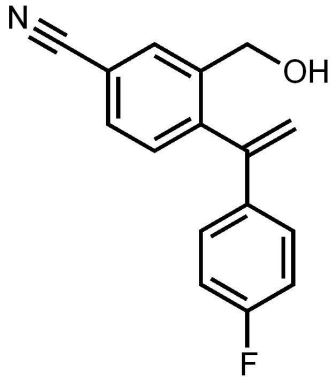


NAME	LW4-24_2
EXPNO	40
PROCNO	1
Date_	20120817
Time	9.39
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728489 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

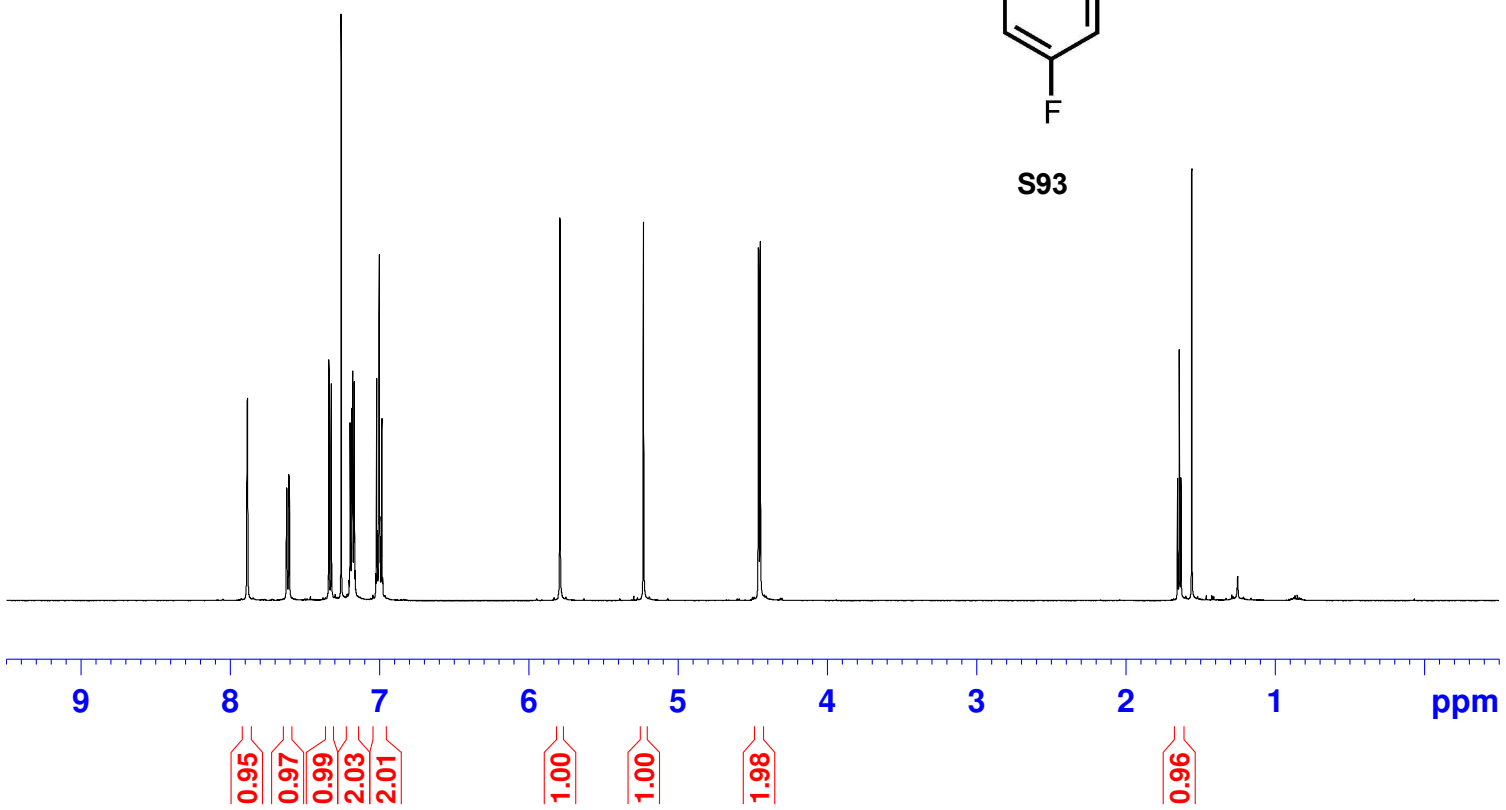
user Joanne Hewitt
JMH-VI-78B
proton.gla CDCl3 /u joahew 4

7.887 7.625 7.622 7.610 7.606 7.341 7.325 7.259 7.199 7.195 7.188 7.186 7.184 7.181 7.175 7.171 7.021 7.017 7.010 7.007 7.004 7.000 6.991 6.986 5.793 5.233 4.463 4.451

1.654 1.643 1.631

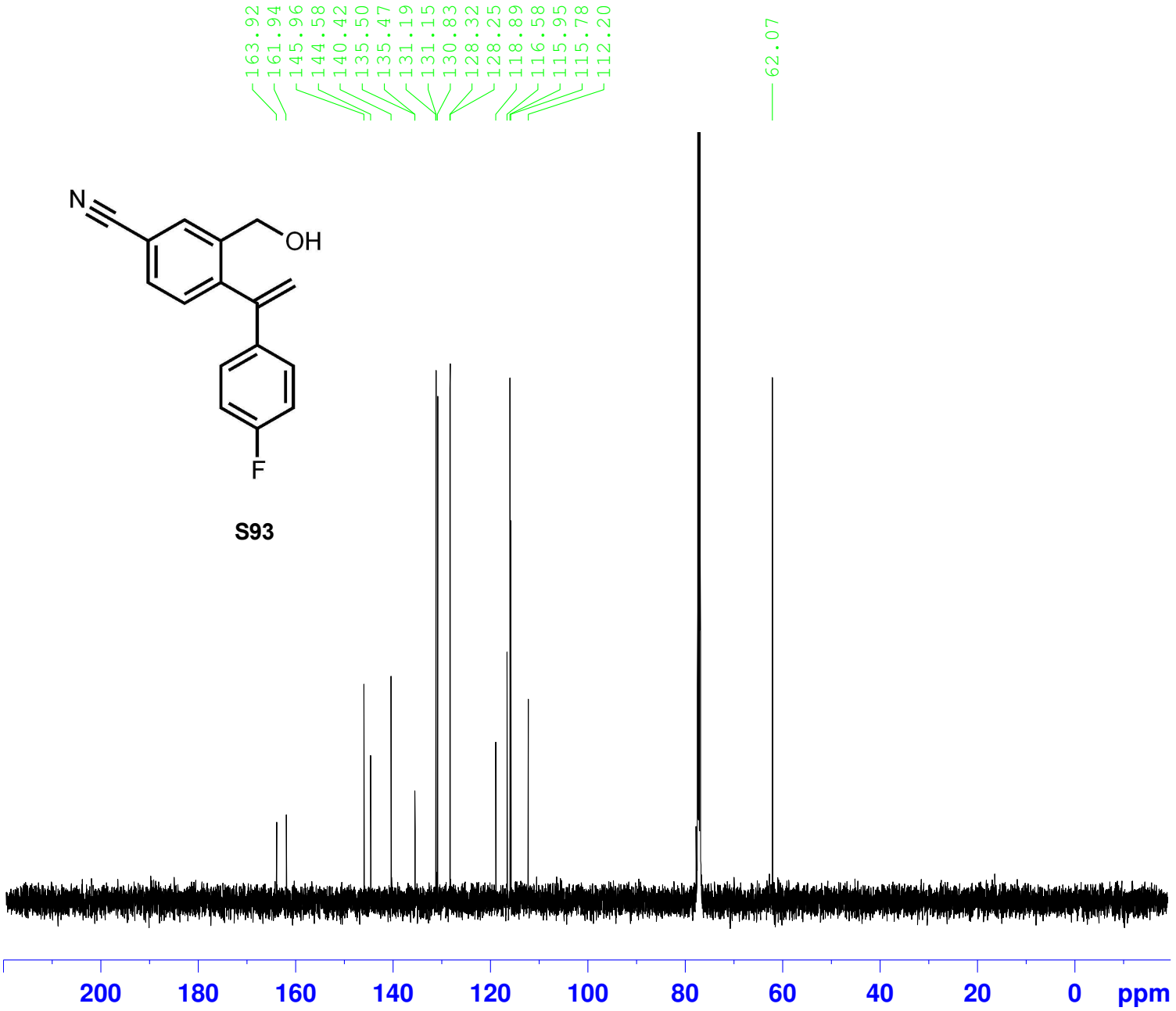


S93



NAME	JMH-VI-78B_3
EXPNO	10
PROCNO	1
Date_	20120828
Time	10.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	74012
SOLVENT	CDCl3
NS	16
DS	2
SWH	10273.973 Hz
FIDRES	0.138815 Hz
AQ	3.6019673 sec
RG	322
DW	48.667 usec
DE	7.02 usec
TE	271.5 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.00 usec
PL1	1.10 dB
PL1W	18.32853889 W
SFO1	500.1930889 MHz
SI	131072
SF	500.1900122 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

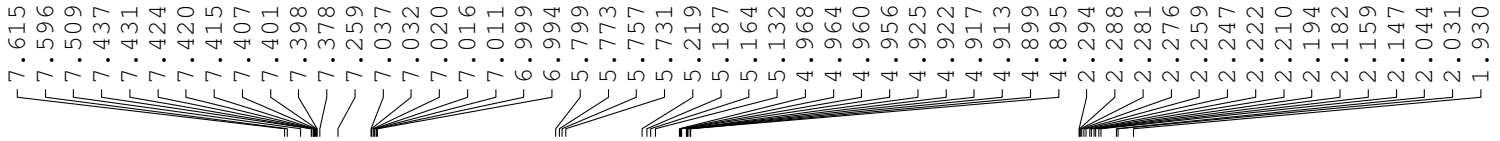
user Joanne Hewitt
JMH-VI-78B
C13CPD1024.GLA CDCl3 /u joahew 4

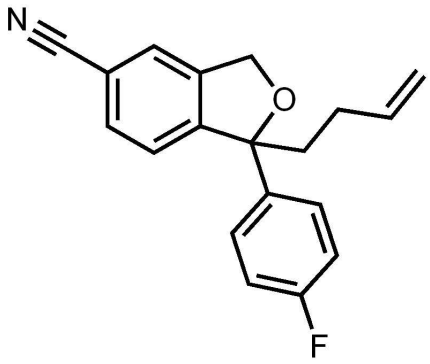


S93

NAME	JMH-VI-78B
EXPNO	14
PROCNO	1
Date_	20120828
Time	20.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	272.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728585 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

User Joanne Hewitt
JMH-VII-50B
PROTON_C_A3.gla CDCl3 u joahew 6

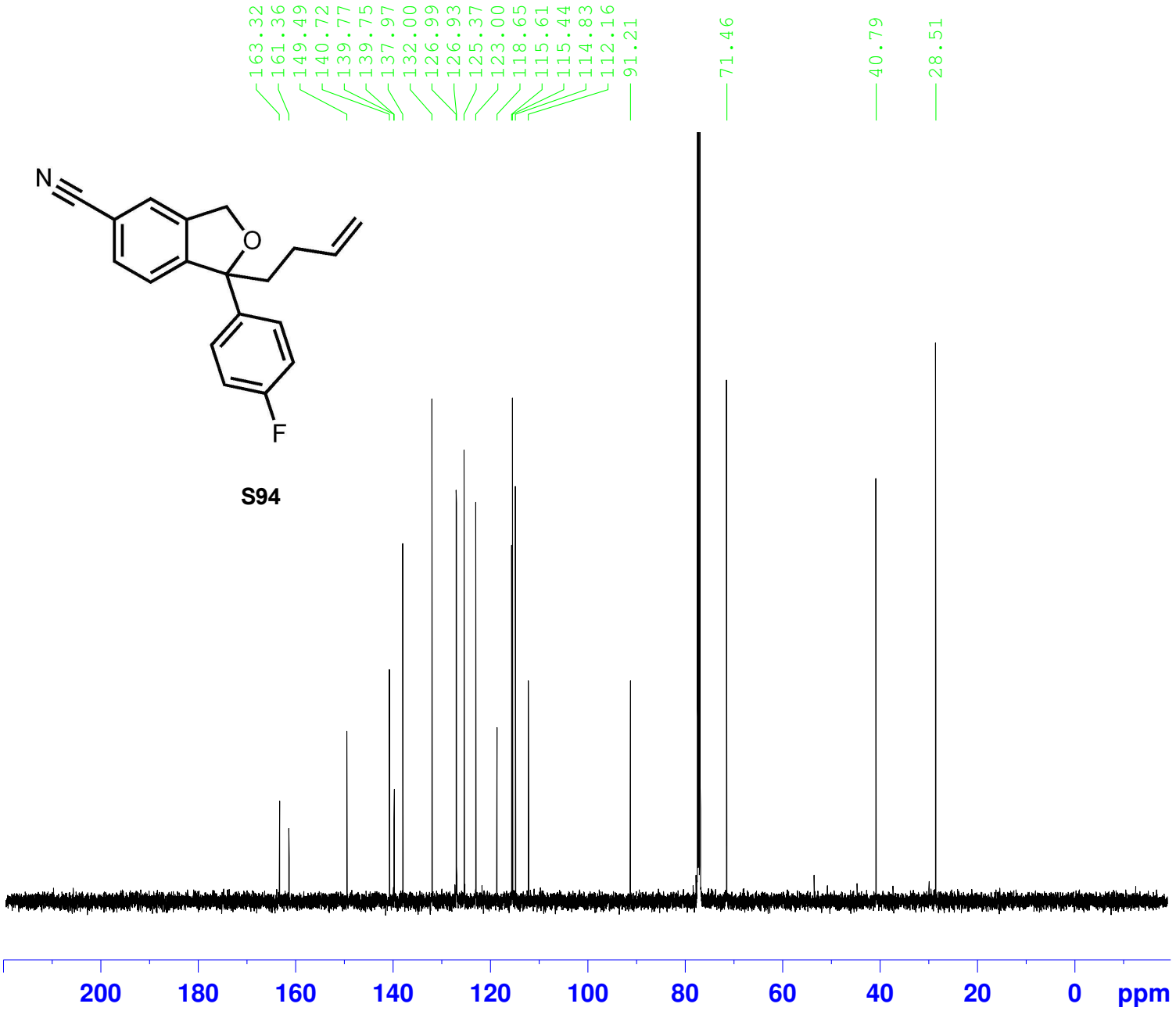




S94

NAME	JMH-VII-50B
EXPNO	10
PROCNO	1
Date_	20120928
Time	16.59
INSTRUM	AV400
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	1024
DW	60.400 usec
DE	6.00 usec
TE	297.7 K
D1	0.01000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
SFO1	400.1324708 MHz
SI	32768
SF	400.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

user Joanne Hewitt
JMH-VI-28A
C13CPD1024.GLA CDCl3 /u joahew 25



NAME	JMH-VI-28A
EXPNO	12
PROCNO	1
Date_	20120724
Time	2.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30000.000 Hz
FIDRES	0.457764 Hz
AQ	1.0923166 sec
RG	2050
DW	16.667 usec
DE	8.01 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.50 dB
PL1W	147.40557861 W
SFO1	125.7854522 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.85 dB
PL13	20.00 dB
PL2W	18.75546646 W
PL12W	0.38737163 W
PL13W	0.23611732 W
SFO2	500.1920008 MHz
SI	32768
SF	125.7728492 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40