

## Supplementary Information

### General Information

#### General Information

All starting reagents were commercially available and used without further purification. *L*-cysteine hydrochloride monohydrate and hexanal were purchased from Merck KGaA. 4-methoxybenzaldehyde, 4-hydroxybenzaldehyde, 4-hydroxy-1-naphthalaldehyde, *N,N'*-Dicyclohexylcarbodiimide, pentanal, 2-methylpentanal, trimethylacetaldehyde and propiolic acid were purchased from Sigma-Aldrich. 4-fluorobenzaldehyde, 3-fluorobenzaldehyde, 2-fluorobenzaldehyde and thionyl chloride were purchased from Fluka. 4-cyanobenzaldehyde was purchased from Alfa Aesar GmbH & Co. 2-ethylhexylaldehyde purchased from TCI America. 3,5,5-trimethylhexanal purchased from SAFC. Solvents were purchased from Sigma-Aldrich and dichloromethane was dried over phosphorous pentoxide before use. TLC was performed on Kieselgel 60 F<sub>254</sub> (Merck) silica gel plaques and the compounds were revealed by UV light (254nm), an ethanolic ninhydrine solution (200mg of ninhydrine in 100mL ethanol), an ethanolic phosphomolybdic acid solution (5g of phosphomolybdic acid in 100mL of ethanol/H<sub>2</sub>SO<sub>4</sub> 95/5) or iodine. Infrared (FT-IR) spectra (10T/cm<sup>2</sup> pressure applied to potassium bromide discs) were recorded on a Perkin Elmer FT-IR 1720X spectrometer and the frequencies were expressed in cm<sup>-1</sup>. NMR spectra were recorded with a Bruker AC 400 Hz spectrometer, using tetramethylsilane (TMS) as the internal reference, with chloroform (CDCl<sub>3</sub>) and dimethylsulfoxide-d<sub>6</sub> (DMSO-d<sub>6</sub>) as a solvent. Chemical shifts were reported in parts per million (ppm). LC-MS spectra were recorded with a Waters 2695 Alliance Micromass ZQ LC-MS.

**(2*RS*,4*R*)-2-(4-Methoxy-phenyl)-thiazolidine-4-carboxylic acid (1a)**

Yield: 78%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 3.07 (6t, *J*=9.6Hz, 0.5H); 3.15 (dd, *J*<sub>1</sub>=4.0Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.29 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=10.4Hz, 0.5H); 3.35 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.74 (s, 1.5H); 3.76 (s, 1.5H); 3.87 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=8.4Hz, 0.5H), 4.25 (dd, *J*<sub>1</sub>=4.0Hz, *J*<sub>2</sub>=7.2Hz, 0.5H); 5.46 (s, 0.5H); 5.60 (s, 0.5H); 6.89 (d, *J*=8.4Hz, 1H); 6.92 (d, *J*=8.4Hz, 1H); 7.37 (d, *J*=8.4Hz, 1H); 7.44 (d, *J*=8.4Hz, 1H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>) δ: 37.8, 38.4, 54.9, 55.0, 64.7, 65.2, 70.9, 71.4, 113.5, 113.7, 128.2, 128.4, 130.6, 132.6, 158.7, 159.1, 172.2, 173.0. FT-IR (KBr), cm<sup>-1</sup>: 3451, 2961, 2477, 1583. LC – MS: ELSD 99%, rt = 3.40 min., *m/z* 240 [M + H]<sup>+</sup>, 281 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(4-Fluoro-phenyl)-thiazolidine-4-carboxylic acid (1b)**

Yield: 83%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 3.07 (dd, *J*<sub>1</sub>= 8.8 Hz, *J*<sub>2</sub>= 10.0 Hz, 0.8H); 3.12 (dd, *J*<sub>1</sub>= 4.8 Hz, *J*<sub>2</sub>= 10.0 Hz, 1.2H); 3.87 (dd, *J*<sub>1</sub>= 7.2 Hz, *J*<sub>2</sub>= 8.0 Hz, 0.4H); 4.19 (dd, *J*<sub>1</sub>= 4.8 Hz, *J*<sub>2</sub>= 8.0 Hz, 0.5H), 5.50 (s, 0.4H); 5.65 (s, 0.6H); 7.11-7.58 (m, 4H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>) δ: 37.9, 38.2, 64.1, 65.3, 114.7, 115.0, 125.3, 127.5, 128.9, 159.2, 161.4, 173.1 FT-IR (KBr), cm<sup>-1</sup>: 2978, 2744, 2604, 2449, 1483, 1455, 1432, 1374, 1301, 1275, 1243, 1232, 1205. LC – MS: ELSD 99%, rt = 3.57 min., *m/z* 228 [M + H]<sup>+</sup>, 269 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(3-Fluoro-phenyl)-thiazolidine-4-carboxylic acid (1c)**

Yield: 75%. NMR (DMSO-d<sub>6</sub>) δ: 3.02 (dd, *J*<sub>1</sub>=5.6Hz, *J*<sub>2</sub>=10.0Hz, 1H); 3.27 (dd, *J*<sub>1</sub>=6.4Hz, *J*<sub>2</sub>=10.0Hz, 1H); 4.17 (t, *J*=6.4Hz, 1H); 5.89 (s, 1H); 7.12-7.54 (m, 4H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): 38.2, 38.5, 64.0, 64.1, 65.3, 65.8, 115.5, 115.7, 124.8, 127.5, 129.7, 158.7, 161.2, 172.6, 173.1. FT-IR (KBr), cm<sup>-1</sup>: 2978, 2745, 2605, 2450, 1575, 1483, 1455, 1432, 1374, 1316, 1302, 1275, 1244, 1233, 1206. LC – MS: ELSD 99%, rt = 4.88 min., *m/z* 228 [M + H]<sup>+</sup>, 269 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(2-Fluoro-phenyl)-thiazolidine-4-carboxylic acid (1d)**

Yield: 73%. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>) δ: 3.04 (m, 0.3H); 3.02-3.40 (m, 1.7H); 3.93 (q, *J*<sub>1</sub> = 6.8 Hz, *J*<sub>2</sub> = 9.2 Hz, 0.15H); 4.18 (t, *J* = 6.4 Hz, 0.85H); 5.69 (s, 0.15H); 5.91 (s, 0.85H); 7.14 - 7.42 (m, 3H); 7.55 (t, *J* = 7.2 Hz, 0.85H); 7.71 (t, *J* = 7.2 Hz, 0.15H). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>): 38.3, 38.5; 64.1, 65.6, 65.7, 115.4, 115.7; 124.7, 127.6, 129.5; 157.4, 157.8; 172.7, 173.6 FT-IR (KBr), cm<sup>-1</sup>: 2978, 2744, 2604, 2449, 1483, 1455, 1432, 1374, 1301, 1275, 1243, 1232, 1205. LC – MS: ELSD 99%, rt = 2.93 min., *m/z* 228 [M + H]<sup>+</sup>, 269 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*,4*R*)-2-(4-Hydroxy-phenyl)-thiazolidine-4-carboxylic acid (1e)**

Yield: 70%. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>) δ: 3.05 (dd, *J*<sub>1</sub>=9.2Hz, *J*<sub>2</sub>=10.4Hz, 0.5H); 3.15 (dd, *J*<sub>1</sub>=3.6Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.27 (dd, *J*<sub>1</sub>=7.6Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.35 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.85 (dd, *J*<sub>1</sub>=8.8Hz, *J*<sub>2</sub>=9.2Hz, 0.5H), 4.25 (dd, *J*<sub>1</sub>=4.0Hz, *J*<sub>2</sub>=7.2Hz, 0.5H); 5.40 (s, 0.5H); 5.54 (s, 0.5H); 6.71 (d, *J*=8.8Hz, 1H); 6.74 (d, *J*=8.8Hz, 1H); 7.24 (d, *J*=8.4Hz, 1H); 7.31 (d, *J*=8.8Hz, 1H). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>) δ: 38.2, 38.9; 65.1, 65.6; 71.7, 72.3; 115.4, 115.6; 128.7, 129.0; 129.3, 131.2 157.4, 157.8; 172.7, 173.6. FT-IR (KBr), cm<sup>-1</sup>: 3516, 3319, 3100, 2984, 2696, 1634, 1615, 1597, 1520, 1461, 1430, 1389, 1338, 1309, 1278, 1241, 1200. LC – MS: ELSD 99%, rt = 4.56 min., *m/z* 226 [M + H]<sup>+</sup>.

**(2*RS*,4*R*)-2-(4-Cyano-phenyl)-thiazolidine-4-carboxylic acid (1g)**

Yield: 63%. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>) δ: 3.05-3.08 (m, 0.4H); 3.09 (t, *J* = 9.2Hz, 0.6H); 3.32 (dd, *J*<sub>1</sub>=6.8Hz, *J*<sub>2</sub>=10.0Hz, 0.4H); 3.38 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=7.6Hz, 0.6H); 3.94 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=8.8Hz, 0.6H), 4.12 (t, *J*=6.4Hz, 0.4H); 5.60 (s, 0.6H); 5.82 (s, 0.4H); 7.61 (d, *J*=8.0Hz, 0.8H); 7.73 (d, *J*=8.0Hz, 1.2H); 7.79 (d, *J*=8.4Hz, 0.8H); 7.84 (d, *J*=8.0Hz, 1.2H). FT-IR (KBr), cm<sup>-1</sup>: 3411, 3002, 2229, 1630, 1505, 1476, 1442, 1409, 1381, 1343, 1313, 1293, 1264. LC – MS: ELSD 99%, rt = 3.44 min., *m/z* 235 [M + H]<sup>+</sup>, 276 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-Pentyl-thiazolidine-4-carboxylic acid (1h)**

Yield: 90%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 0.86 (t, *J*=6.8Hz, 3H); 1.25-1.89 (m, 8H); 2.75 (t, *J*=9.6Hz, 0.4H); 2.93 (dd, *J*<sub>1</sub>=5.2Hz, *J*<sub>2</sub>=10.0Hz, 0.6H); 3.08 (dd, *J*<sub>1</sub>=6.8Hz, *J*<sub>2</sub>=10.0Hz, 0.6H); 3.18 (dd, *J*<sub>1</sub>=6.8Hz, *J*<sub>2</sub>=9.6Hz, 0.4H); 3.69 (dd, *J*<sub>1</sub>=6.8Hz, *J*<sub>2</sub>=9.6Hz, 0.4H); 4.06 (dd, *J*<sub>1</sub>=5.2Hz, *J*<sub>2</sub>=6.8Hz, 0.6H); 4.40 (t, *J*=6.4Hz, 0.4H); 4.54 (t, *J*=6.8Hz, 0.6H). FT-IR (KBr), cm<sup>-1</sup>: 3435, 2926, 2857, 2314, 1614, 1366, 1243, 1145. LC – MS: ELSD 99%, rt = 3.01 min., *m/z* 204 [M + H]<sup>+</sup>, 245 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-Butyl-thiazolidine-4-carboxylic acid (1i)**

Yield: 36%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 0.86 (t, *J*=7.0Hz, 1.5H); 0.87 (t, *J*=6.8Hz, 1.5H); 1.26-1.94 (m, 6H); 2.75 (t, *J*=10.0Hz, 0.5H); 2.93 (dd, *J*<sub>1</sub>=5.2Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.08 (dd, *J*<sub>1</sub>=7.0Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.18 (dd, *J*<sub>1</sub>=7.0Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.70 (dd, *J*<sub>1</sub>=7.0Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 4.06 (dd, *J*<sub>1</sub>=5.2Hz, *J*<sub>2</sub>=7.0Hz, 0.5H); 4.40 (t, *J*=6.4Hz, 0.5H); 4.54 (t, *J*=6.8Hz, 0.5H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>) δ: 14.3, 14.3; 22.3, 22.4; 30.0, 30.2; 35.0, 36.8 ; 37.0, 37.4 ; 64.6, 65.6 ; 70.7, 71.5; 172.7, 173.3. FT-IR (KBr), cm<sup>-1</sup>: 2956, 2869, 2750, 2454, 1580, 1379, 1295, 1207, 1140. LC – MS: ELSD 99%, rt = 3.41 min., *m/z* 190 [M + H]<sup>+</sup>, 231 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(1-Ethyl-pentyl)-thiazolidine-4-carboxylic acid (1j)**

Yield: 92%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 0.82-0.89 (m, 6H); 1.24-1.68 (m, 9H); 2.68 (t, *J*=9.6Hz, 0.5H); 2.92 (dd, *J*<sub>1</sub>=5.2Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.01 (dd, *J*<sub>1</sub>=6.8Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.17 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.70 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=9.6Hz, 0.5H); 4.06 (dd, *J*<sub>1</sub>=5.2Hz, *J*<sub>2</sub>=6.8Hz, 0.5H); 4.41 (d, *J*=7.6Hz, 0.5H); 4.52 (d, *J*=7.6Hz, 0.5H). FT-IR (KBr), cm<sup>-1</sup>: 2963, 2932, 2872, 2754, 2617, 2469, 2062, 1604, 1574, 1465, 1438, 1424, 1379, 1309, 1265, 1244, 1207, 1137. LC – MS: ELSD 99%, rt = 3.37 min., *m/z* 232 [M + H]<sup>+</sup>, 295 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(2,4,4-trimethylpentyl)-thiazolidine-4-carboxylic acid (1k)**

Yield: 80%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 0.85-0.94 (m, 12H); 1.03-1.08 (m, 1H); 1.18-1.23 (m, 1H); 1.39-1.84 (m, 3H); 2.75 (td, *J*<sub>1</sub>=4.0Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 2.90-2.97 (m, 0.5H); 3.05-3.12 (m, 0.5H); 3.18 (dd, *J*<sub>1</sub>=6.4Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 3.70 (dd, *J*<sub>1</sub>=7.2Hz, *J*<sub>2</sub>=10.0Hz, 0.5H); 4.04-4.10 (m, 0.5H); 4.45 (t, *J*=6.4Hz, 0.5H); 4.57-4.64 (m, 0.5H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>) δ: 22.7, 22.75, 23.1, 23.2, 28.1, 28.6, 28.6, 29.0, 31.2, 31.25, 31.3, 31.4, 37.0, 37.3, 37.4, 37.6, 44.9, 45.2, 46.4, 47.0, 51.0, 51.6, 51.7, 64.6, 64.63, 65.6, 65.7, 68.9, 69.5, 69.6, 70.3, 172.8, 173.3, 173.4. FT-IR (KBr), cm<sup>-1</sup>: 2957, 2792, 2610, 2358, 1611, 1578, 1467, 1431, 1367, 1314, 1246, 1217, 1134. LC – MS: ELSD 99%, rt = 3.33 min., *m/z* 246 [M + H]<sup>+</sup>, 287 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(1-Methyl-butyl)-thiazolidine-4-carboxylic acid (1l)**

Yield: 62%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 0.82-0.86 (m, 3H); 0.87 (d, *J*= 6.67 Hz, 0.75H); 0.91 (d, *J*= 6.67 Hz, 0.75H); 0.98 (d, *J*=6.67 Hz, 0.75H); 1.08-1.86 (m, 5H); 2.62-2.69 (m, 0.5H); 2.89 (dd, *J*<sub>1</sub>= 5.0 Hz, *J*<sub>2</sub>= 10.2 Hz, 0.5H); 3.00-3.03 (m, 0.5H); 3.12- 3.17 (m, 0.5H); 3.65-3.70 (m, 0.5H); 4.01-4.05 (m, 0.5H); 4.26 (d, *J*= 7.68 Hz, 0.25H); 4.31 (d, *J*= 7.13 Hz, 0.25H); 4.37 (d, *J*= 8.0 Hz, 0.25H); 4.41 (d, *J*= 7.50 Hz, 0.25H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>) δ: 14.5, 14.6, 14.6, 14.7; 17.0, 17.4, 17.6, 17.7; 19.8, 19.9, 19.9, 20.0; 36.8, 36.9, 36.9, 37.1; 37.2, 37.2, 37.3, 37.3; 37.4, 37.4, 37.5, 37.9; 64.8, 64.9, 65.6 ; 76.6, 76.7, 77.4, 77.5; 172.8, 173.3, 173.4. FT-IR (KBr), cm<sup>-1</sup>: 2960, 2930, 2872, 2784, 2623, 2391, 1575, 1424, 1374, 1314, 1303, 1257, 1206, 1136. LC – MS: ELSD 99%, rt = 4.95 min., *m/z* 204 [M + H]<sup>+</sup>, 245 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-*tert*-Butyl-thiazolidine-4-carboxylic acid (1m)**

Yield: 59%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ: 0.97 (s, 0.45H); 1.00 (s, 8.55); 2.63 (t, *J*=10.0Hz, 0.95H); 2.91-2.98 (m, 0.1H); 3.19 (dd, *J*<sub>1</sub>=6.4Hz, *J*<sub>2</sub>=10.0Hz, 0.95H); 3.70 (dd, *J*<sub>1</sub>=6.4Hz, *J*<sub>2</sub>=10Hz, 0.95H); 4.07-4.10 (m, 0.05H); 4.38 (s, 0.95H); 4.2 (s, 0.05H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>) δ: 26.6, 26.7, 33.5, 35.0, 36.6, 38.7, 64.91, 64.93, 80.1, 81.2, 172.3, 172.9. FT-IR (KBr), cm<sup>-1</sup>: 3061, 2980, 2963, 2887, 2659, 2606, 2353, 2158, 1642, 1479, 1432, 1401, 1373, 1358, 1301, 1247, 1199, 1128. LC – MS: ELSD 99%, rt = 3.37 min., *m/z* 190 [M + H]<sup>+</sup>, 231 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*,4*R*)-2-(4-Methoxy-phenyl)-3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester**  
**(3a)**

Yield: 89%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ: 1.29 (t, *J*= 7.2 Hz, 1.2H); 1.31 (t, *J*= 7.2Hz, 1.8H); 2.96 (s, 0.6H); 3.08 (s, 0.4H); 3.27 (dd, *J*<sub>1</sub>= 7.2Hz, *J*<sub>2</sub>= 12.4Hz, 0.6H); 3.35 (dd, *J*<sub>1</sub>= 6.8Hz, *J*<sub>2</sub>= 12.0Hz, 0.6H); 3.41-3.43 (m, 0.8H); 3.79 (s, 1.2H); 3.81 (s, 1.8H); 4.21-4.33 (m, 2H); 4.98 (t, *J*= 7.2Hz, 0.6H); 5.21 (t, *J*= 5.6Hz, 0.4H); 6.33 (s, 0.4H); 6.41 (s, 0.6H); 6.85 (d, *J*= 8.8Hz, 0.8H); 6.89 (d, *J*= 8.8Hz, 1.2H); 7.51 (d, *J*= 8.8Hz, 0.8H); 7.58 (d, *J*= 8.8Hz, 1.2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>): 13.9, 14.1; 32.8, 33.3; 55.4, 55.8; 62.0, 62.4; 65.8, 65.3; 66.8, 67.1; 75.0, 75.4; 81.0, 81.2; 115.1, 115.3; 128.6, 128.8, 129.0, 129.2; 135.2, 135.5; 159.0, 159.3; 161.5, 163.5 ; 169.0, 169.4 FT-IR (KBr), cm<sup>-1</sup>: 3441, 3227, 2932, 2108, 1741, 1635, 1512, 1457, 1384, 1334, 1293, 1241, 1204, 1176, 1024, 905. LC – MS: ELSD 98%, rt = 5.16 min., *m/z* 320 [M + H]<sup>+</sup>, 383 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>.

**(2*RS*, 4*R*)-2-(4-Fluoro-phenyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester**  
**(3b)**

Yield: 37%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ: 1.25-1.36 (m, 3H); 2.96 (s, 0.6H); 3.10 (s, 0.4H); 3.25 (dd, *J*<sub>1</sub>= 7.6 Hz, *J*<sub>2</sub>= 12.0 Hz, 0.6H); 3.37 (dd, *J*<sub>1</sub>= 6.8 Hz, *J*<sub>2</sub>= 12.0 Hz, 0.6H); 3.44 (d, *J*=6.0Hz, 0.8H) 4.22-4.32 (m, 2H); 4.99 (t, *J*= 7.2 Hz, 0.6H); 5.22 (t, *J*= 6.0 Hz, 0.4H); 6.33 (s, 0.5H);

6.42 (s, 0.5H); 7.00 (d,  $J= 8.4\text{Hz}$ , 0.4H); 7.00 (d,  $J= 8.8\text{Hz}$ , 0.4H); 7.05 (d,  $J= 8.8\text{Hz}$ , 0.6H); 7.05 (d,  $J= 8.4\text{Hz}$ , 0.6H); 7.57 (d,  $J= 8.4\text{Hz}$ , 1.2H); 7.58 (d,  $J= 8.8\text{Hz}$ , 0.8H); 7.64 (d,  $J= 8.4\text{Hz}$ , 1.2H); 7.66 (d,  $J= 8.0\text{Hz}$ , 0.8H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 14.0, 14.1; 32.5, 33.5; 62.3, 62.5; 65.8, 65.3; 63.7, 67.1; 75.0, 75.3; 81.2, 81.2; 115.1, 115.2, 115.3, 115.4; 128.6, 128.7, 129.0, 129.1; 133.7, 135.5; 152.3, 155.0; 161.5, 163.5 ; 168.9, 169.4. FT-IR (KBr),  $\text{cm}^{-1}$ : 3243, 2995, 2933, 2584, 2110, 1742, 1634, 1510, 1479, 1384, 1331, 1228, 1207, 1168, 1096, 1024, 965. LC – MS: ELSD 97%,  $\text{rt} = 5.49$  min.,  $m/z$  308  $[\text{M} + \text{H}]^+$ , 371  $[\text{M} + \text{CH}_3\text{CN} + \text{Na}]^+$ .

**(2*RS*, 4*R*)-2-(3-Fluoro-phenyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3c)**

Yield: 58%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 1.35 (t,  $J=7.2\text{Hz}$ , 2.1H); 1.37 (t,  $J=7.2\text{Hz}$ , 0.9H); 2.93 (s, 0.3H); 2.98 (s, 0.7H); 3.28 (dd,  $J_1= 9.0$  Hz,  $J_2= 12.0$  Hz, 0.7H); 3.38 (dd,  $J_1= 6.6$  Hz,  $J_2= 12.0$  Hz, 0.7H); 3.40 (dd,  $J_1= 6.6$  Hz,  $J_2= 12.0$  Hz, 0.3H); 3.46 (dd,  $J_1= 6.8$  Hz,  $J_2= 12.0$  Hz, 0.3H); 4.24 – 4.39 (m, 2H); 4.90 (dd,  $J_1= 6.4$  Hz,  $J_2= 8.4$  Hz, 0.3H); 5.18 (t,  $J= 6.4$  Hz, 0.7H); 6.52 (s, 0.3H); 6.63 (s, 0.7H); 7.00 – 7.33 (m, 3H); 7.82 (td,  $J_1= 1.6$  Hz,  $J_2= 8.0$  Hz, 0.3H); 8.05 (td,  $J_1= 1.6$  Hz,  $J_2= 8.0$  Hz, 0.7H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 14.5, 14.7; 32.2, 33.4, 62.1, 75.2, 76.8, 77.3, 78.8, 80.1, 127.4, 128.5, 128.6, 129.6, 129.7, 152.0, 158.5, 160.5, 169.1. FT-IR (KBr),  $\text{cm}^{-1}$ : 3966, 3851, 3722, 3647, 3466, 3338, 3234, 3068, 2931, 2813, 2691, 2439, 2042, 1912, 1704, 1206. LC – MS: ELSD 99%,  $\text{rt} = 5.48$  min.,  $m/z$  308  $[\text{M} + \text{H}]^+$ , 371  $[\text{M} + \text{CH}_3\text{CN} + \text{Na}]^+$ .

**(2*RS*, 4*R*)-2-(2-Fluoro-phenyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3d)**

Yield: 30%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 1.34-1.38 (m, 3H); 2.93 (s, 0.7H); 2.98 (s, 0.3H); 3.29 (dd,  $J_1 = 8.0$  Hz,  $J_2 = 12.0$  Hz, 0.7H); 3.37 (dd,  $J_1= 6.8$  Hz,  $J_2=12.0$  Hz, 0.7H); 3.39-3.48 (m, 0.6H); 4.12-4.40 (m, 2H); 4.90 (dd,  $J_1= 6.8\text{Hz}$ ,  $J_2= 9.2\text{Hz}$  0.7H); 5.18 (t,  $J= 6.4$  Hz, 0.3H);

6.52 (s, 0.3H); 6.63 (s, 0.7H), 7.00-7.34 (m, 3H); 7.82 (td,  $J_1 = 1.6$  Hz,  $J_2 = 8.0$  Hz, 0.3H); 8.06 (td,  $J_1 = 1.6$  Hz,  $J_2 = 8.0$  Hz, 0.7H). LC – MS: ELSD 99%, rt = 5.45 min.,  $m/z$  308 [M + H]<sup>+</sup>, 371 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>.

**(2*RS*,4*R*)-2-(4-Cyano-phenyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3g)**

Yield: 24%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ: 1.32 (t,  $J = 7.2$  Hz, 3H); 3.10 (s, 0.5H); 3.13 (s, 0.5H); 3.14 (dd,  $J_1 = 1.6$  Hz,  $J_2 = 10.4$  Hz, 0.5H); 3.38 (dd,  $J_1 = 7.2$  Hz,  $J_2 = 10.4$  Hz, 1H); 3.49 (dd,  $J_1 = 7.2$  Hz,  $J_2 = 10.4$  Hz, 0.5H); 4.04 (t,  $J = 6.8$  Hz, 1H); 4.24-4.31 (m, 2H); 5.58 (s, 0.5H); 5.87 (s, 0.5H); 7.58-7.69 (m, 4H). LC – MS: ELSD 97%, rt = 4.53 min.,  $m/z$  315 [M + H]<sup>+</sup>, 378 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>.

**(2*RS*, 4*R*)-2-Pentyl-3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3h)**

Yield: 55%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ: 0.86-0.92 (m, 3H); 1.25-1.34 (m, 9H); 1.77-1.81 (m, 1H); 2.10-2.12 (m, 1H); 3.06 (s, 0.4H); 3.17 (s, 0.6H); 3.30-3.39 (m, 2H); 4.25 (m, 2H); 4.96 (t,  $J = 8.0$  Hz, 0.6H); 5.15 (dd,  $J_1 = 4.8$  Hz,  $J_2 = 6.8$  Hz, 0.4H); 5.31-5.36 (m, 1H). FT-IR (KBr), cm<sup>-1</sup>: 3251, 2932, 2857, 2583, 2112, 1739, 1634, 1393, 1344, 1190, 1025, 892. LC – MS: ELSD 98%, rt = 5.36 min.,  $m/z$  284 [M + H]<sup>+</sup>, 347 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>.

**(2*RS*, 4*R*)-2-Butyl-3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3i)**

Yield: 87%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ: 0.88-0.95 (m, 3H); 1.25-1.62 (m, 7H); 1.78-1.83 (m, 1H); 2.10-2.21 (m, 1H); 3.07 (s, 0.4H); 3.17 (s, 0.6H); 3.29-3.40 (m, 2H); 4.18-4.30 (m, 2H); 4.96 (t,  $J = 8.0$  Hz, 0.6H); 5.15 (dd,  $J_1 = 6.0$  Hz,  $J_2 = 7.2$  Hz, 0.4H); 5.33 (td,  $J_1 = 4.4$  Hz,  $J_2 = 9.6$  Hz, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ: 13.9, 13.9; 14.0, 14.1; 22.0, 22.2; 29.0, 29.0; 32.1, 32.7; 35.6, 37.5; 61.5, 61.9; 62.2, 64.2; 64.3, 66.5; 75.7, 76.0; 78.4, 79.1; 150.7, 151.1; 169.5, 169.8. FT-IR



(KBr),  $\text{cm}^{-1}$ : 3242, 2958, 2107, 1746, 1634, 1385, 1024, 859, 738. LC – MS: ELSD 98%,  $\text{rt} = 4.37$  min.,  $m/z$  270  $[\text{M} + \text{H}]^+$ , 311  $[\text{M} + \text{CH}_3\text{CN}]^+$ , 333  $[\text{M} + \text{CH}_3\text{CN} + \text{Na}]^+$ .

**(2*RS*, 4*R*)-2-(1-Ethyl-pentyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3j)**

Yield: 30%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.85-0.97 (m, 6H); 1.26-1.82 (m, 12H); 3.10 (s, 0.4H); 3.20 (s, 0.6H); 3.33 (m, 2H); 4.19-4.29 (m, 2H); 4.97 (q,  $J = 8.0$  Hz, 0.65H); 5.14 (td,  $J_1 = 2.4$  Hz;  $J_2 = 4.0$  Hz, 0.4H); 5.39 (dd,  $J_1 = 4.0$  Hz;  $J_2 = 8.4$  Hz, 0.6H); 5.45 (dd,  $J_1 = 2.4$  Hz;  $J_2 = 8.4$  Hz, 0.4H). LC – MS: ELSD 99%,  $\text{rt} = 5.37$  min.,  $m/z$  312  $[\text{M} + \text{H}]^+$ , 375  $[\text{M} + \text{CH}_3\text{CN} + \text{Na}]^+$ , 353  $[\text{M} + \text{CH}_3\text{CN}]^+$ .

**(2*RS*, 4*R*)-2-(2,4,4-trimethyl-pentyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3k)**

Yield: 55%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.90 (s, 9H); 0.98 (d,  $J = 6.8$  Hz, 1.8H); 1.02 (d,  $J = 6.4$  Hz, 1.2H); 1.25-1.31 (m, 3H); 1.39-1.94 (m, 5H); 2.88 (s, 0.4H); 3.05 (s, 0.6H); 3.17 (d,  $J = 5.6$  Hz, 0.6H); 3.24-3.40 (m, 1.4H); 4.18-4.29 (m, 2H); 4.94 (td,  $J_1 = 2.8$  Hz,  $J_2 = 8.0$  Hz, 0.6H); 5.15 (t,  $J = 8.0$  Hz, 0.4H); 5.34-5.43 (m, 1H). FT-IR (KBr),  $\text{cm}^{-1}$ : 3939, 3243, 2953, 2868, 2588, 2109, 1743, 1634, 1469, 1393, 1188, 1139, 1094, 1027, 951, 892. LC – MS: ELSD 97%,  $\text{rt} = 5.74$  min.,  $m/z$  326  $[\text{M} + \text{H}]^+$ , 389  $[\text{M} + \text{CH}_3\text{CN} + \text{Na}]^+$ .

**(2*RS*, 4*R*)-2-(1-Methyl-butyl)- 3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3l)**

Yield: 63%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.88-0.97 (m, 3H); 1.04 (d,  $J = 6.4$  Hz, 1.2H); 1.15 (d,  $J = 6.4$  Hz, 1.8H); 1.22-1.33 (m, 5H); 1.42-1.80 (m, 3H); 3.11 (d,  $J = 4.0$  Hz, 0.4H); 3.18 (d,  $J = 4.0$  Hz, 0.6H); 3.25-3.39 (m, 2H); 4.18-4.30 (m, 2H); 4.92 (t,  $J = 8.0$  Hz, 0.4H); 5.00 (t,  $J = 8.0$  Hz, 0.6H); 5.10-5.30 (m, 1H). FT-IR (KBr),  $\text{cm}^{-1}$ : 3456, 3243, 2960, 2107, 1634, 1372, 1028, 940.

LC – MS: ELSD 98%, rt = 6.32 min.,  $m/z$  284 [M + H]<sup>+</sup>, 347 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>, 325 [M + CH<sub>3</sub>CN]<sup>+</sup>.

**(2*RS*, 4*R*)-2-*tert*-Butyl-3-propynoyl-thiazolidine-4-carboxylic acid ethyl ester (3m)**

Yield: 52%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ: 1.03 (s, 4.5H); 1.10 (s, 4.5H); 1.29 (t,  $J$ = 6.8 Hz, 1.5H); 1.32 (t,  $J$ = 6.8 Hz, 1.5H); 3.12 (s, 0.5H); 3.23 (s, 0.5H); 3.39-3.47 (m, 1H); 4.21-4.29 (m, 1H); 5.02 (t,  $J$ = 9.2 Hz, 0.5H); 5.13 (t,  $J$ = 8.8 Hz, 0.5H); 5.33 (s, 0.5H); 5.39 (s, 0.5H). FT-IR (KBr), cm<sup>-1</sup>: 3495, 3233, 2961, 2873, 2108, 1740, 1641, 1626, 1486, 1465, 1397, 1384, 1349, 1231, 1184, 1024. LC – MS: ELSD 99%, rt = 5.99 min.,  $m/z$  270 [M + H]<sup>+</sup>, 333 [M + CH<sub>3</sub>CN + Na]<sup>+</sup>.

Sample Name:

KB-01

Data Collected on:

msrcoury400-mrcoury400

Archive directory:

/home/vnmr1/vnmrsys/data

Sample directory:

KB-01\_20131013\_01

FID/F1A1: PROTON 01

Pulse Sequence: PROTON (zgpg3)

Solvent: dmsd

Data collected on: Oct 13 2013

Operator: vnmr1

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Pulse 45.0 degrees

Acq. time 2.561 sec

Width 6398.0 Hz

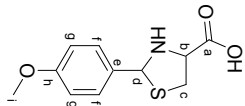
8 repetitions

OBSERVE M1, 400.1759681 MHz

DATA PROCESSING

FT size 32768

Total time 0 min 31 sec



1a





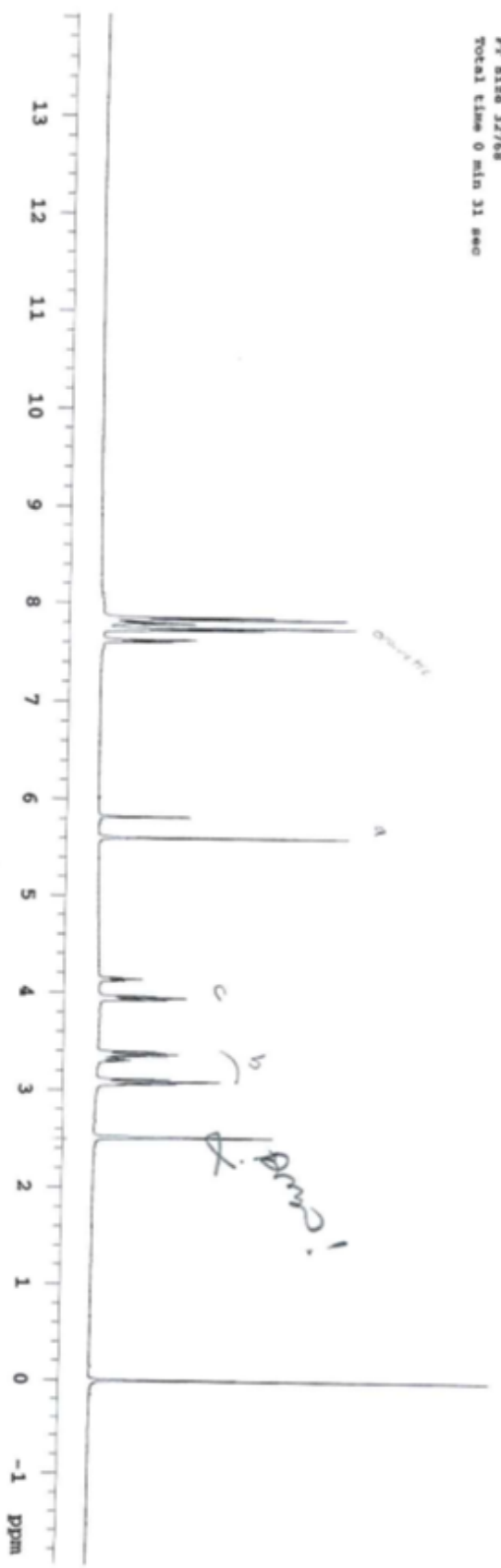
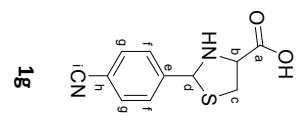


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Data Collected on: mercury400-mercury400  
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Sample directory: KB-04\_20131013\_01  
FID/Flie: PROTON\_01

Pulse Sequence: PROTON (zgpg3)  
Solvent: dmsd  
Data collected on: Oct 13 2013

Operator: vnmr1

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Pulse 45.0 degrees  
Acq. time 2.561 sec  
Width 6398.0 Hz  
8 repetitions  
OBSERVE NI, 400.1759635 MHz  
DATA PROCESSING  
F2 size 33768  
Total time 0 min 31 sec



KB-44

Sample Name:

KB-44

Data Collected on:

mercury400-mercury400

Archive directory:

/home/vnmr1/vnmrns/data

Sample directory:

KB-44\_20131012\_01

Filename: PROTON\_01

Pulse Sequence: PROTON (zgpg3)

Solvent: dms0

Data collected on: Oct 12 2013

Temp: 25.0 C / 298.1 K

Operator: vnmr1

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Pulse 45.0 degrees

Acq. time 2.561 sec

Width 6398.0 Hz

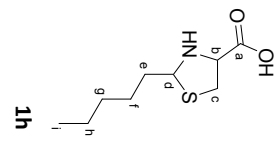
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OBSERVE H1, 400.1759689 MHz

DATA PROCESSING

F2 size 32768

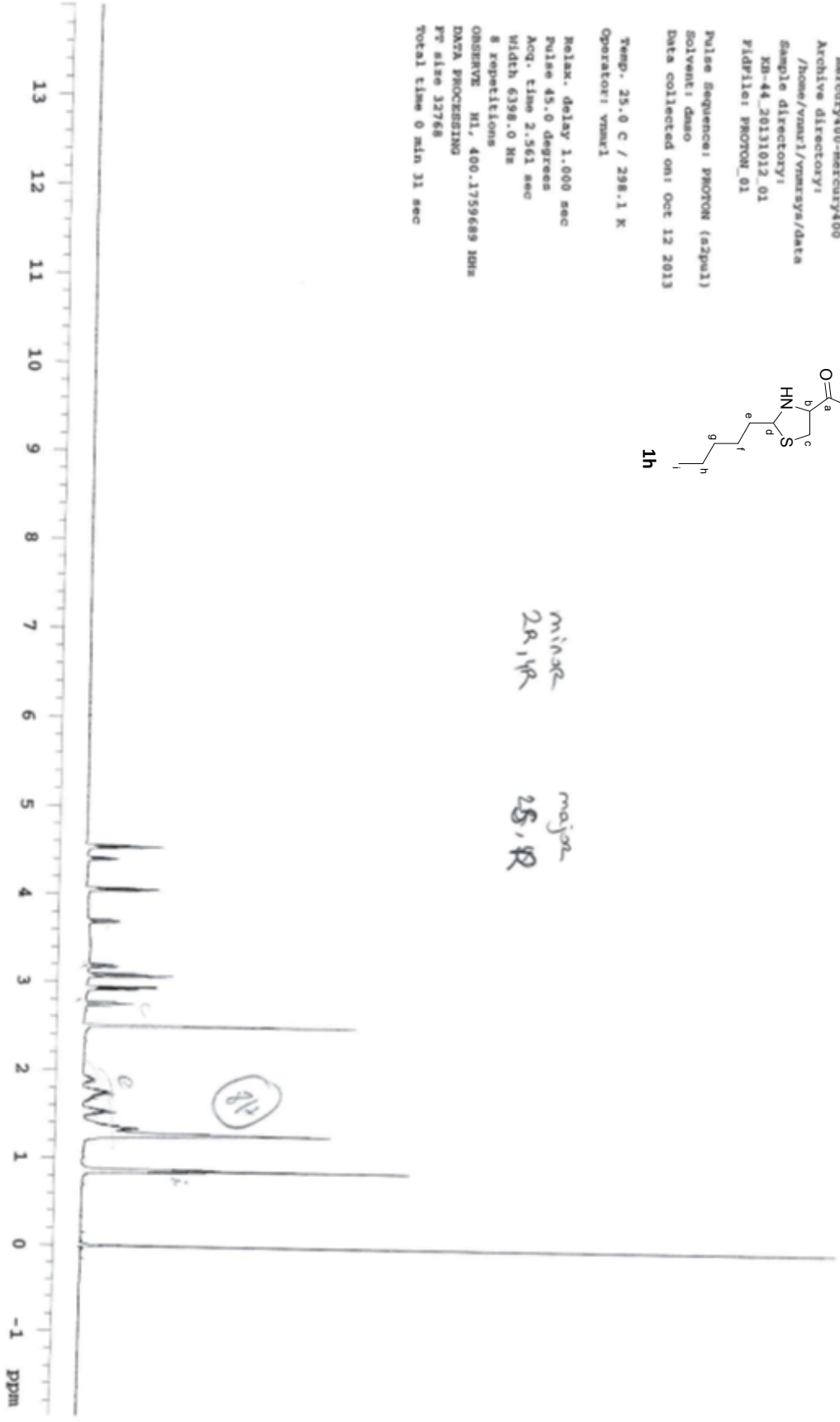
Total time 0 min 31 sec



1h

minor  
2R, 1R

major  
2S, 1R

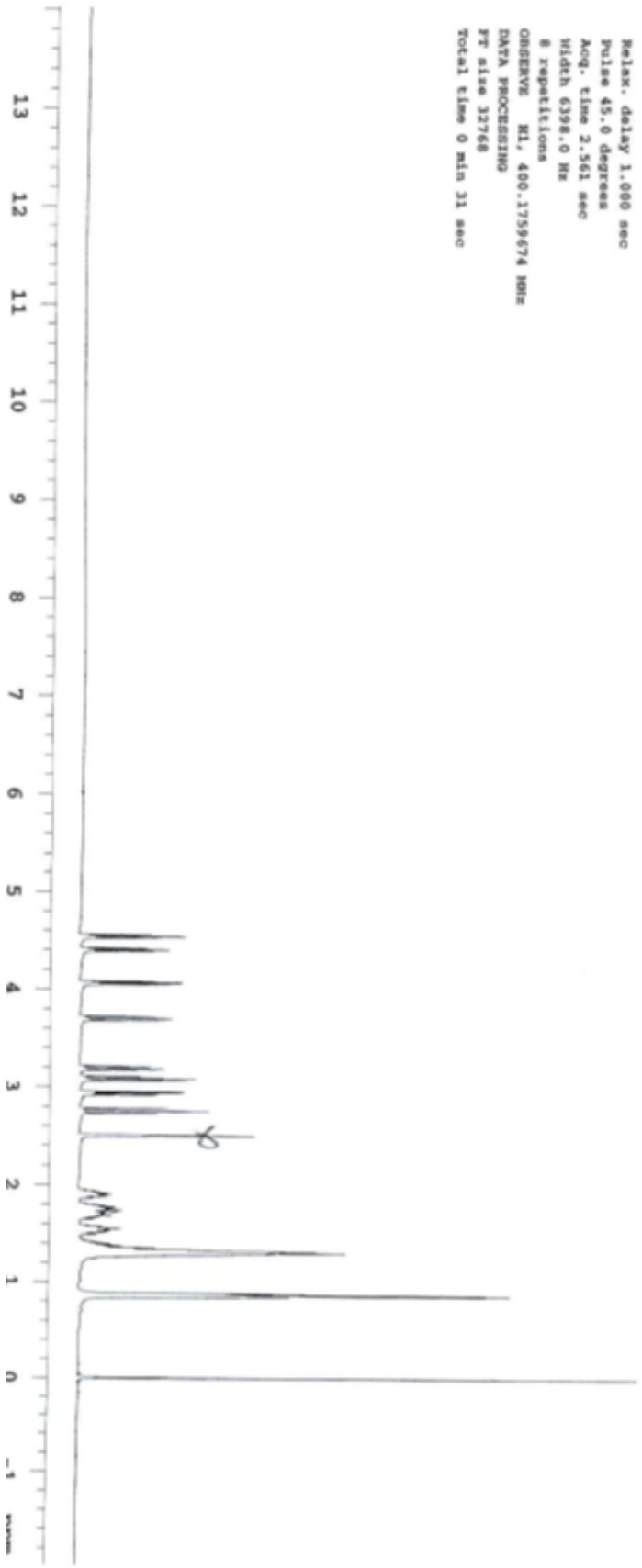
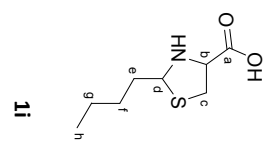


Sample Name: KB-77  
Data Collected on: mercury400-mercury400  
Archive directory: /home/vmax1/vmaxsys/data  
Sample directory: KB-77\_20131013\_01  
FID#1: PROTON\_01

Pulse Sequence: PROTON (s2pu1)  
Solvent: dmsd  
Data collected on: Oct 13 2013

Operator: vmax1

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 2.561 sec  
Width 6398.0 Hz  
8 repetitions  
OBSERVE K1, 400.1799674 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min 31 sec





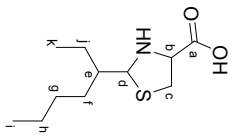
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Sample Name:  
KR-45  
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Archive directory:  
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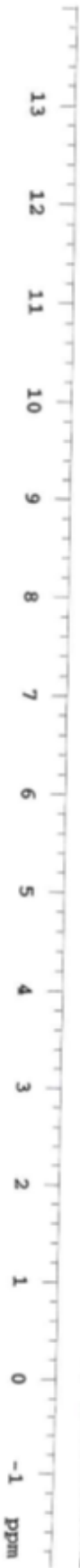
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Solvent: dmsc  
Data collected on: Oct 14 2013

Operator: vmr1

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Acq. time 2.561 sec  
Width 6398.0 Hz  
8 repetitions  
OBSERVE H1, 400.1759681 MHz  
DATA PROCESSING  
PT size 32768  
Total time 0 min 31 sec



1j



KB-51

Sample Name:

KB-51

Data Collected on:

mercury400-mercury400

Archive directory:

/home/vmar1/vmar1/sys/data

Sample directory:

KB-51\_20111012\_01

FIDFile: PROTON\_01

Pulse Sequence: PROTON (zgpg1)

Solvent: dmsc

Data collected on: Oct 12 2013

Temp: 25.0 C / 298.1 K

Operator: vmar1

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Pulse 45.0 degrees

Acq. time 2.561 sec

Width 6398.0 Hz

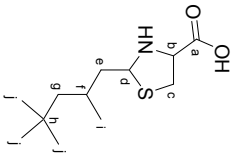
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DATA PROCESSING

FT size 32768

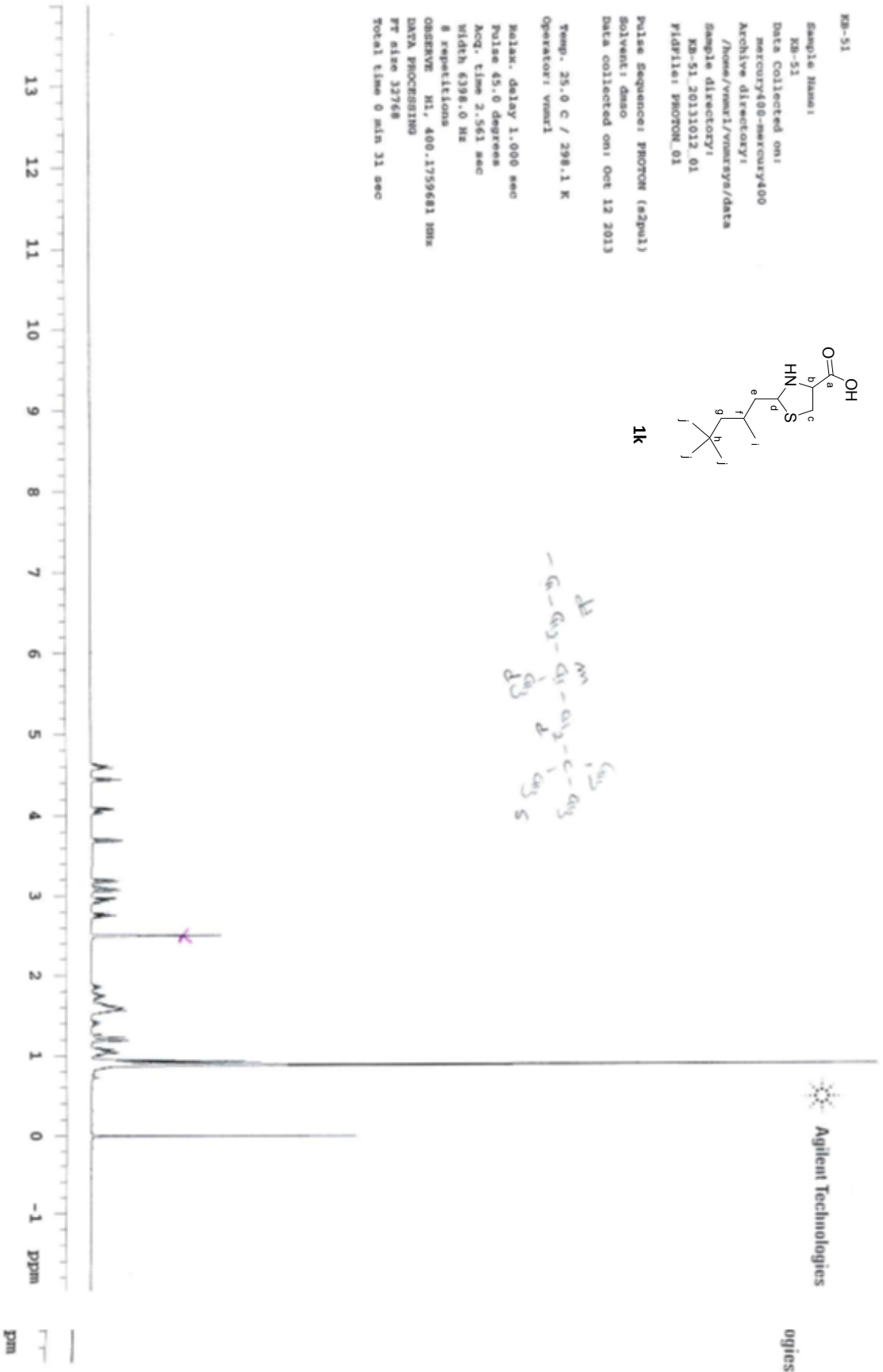
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1k

Handwritten chemical shift assignments in ppm:

- 11.5 (a)
- 7.5 (b)
- 4.5 (c)
- 3.5 (d)
- 2.5 (e)
- 1.0 (f)
- 0.5 (g)
- 0.5 (h)
- 0.5 (i)
- 0.5 (j)



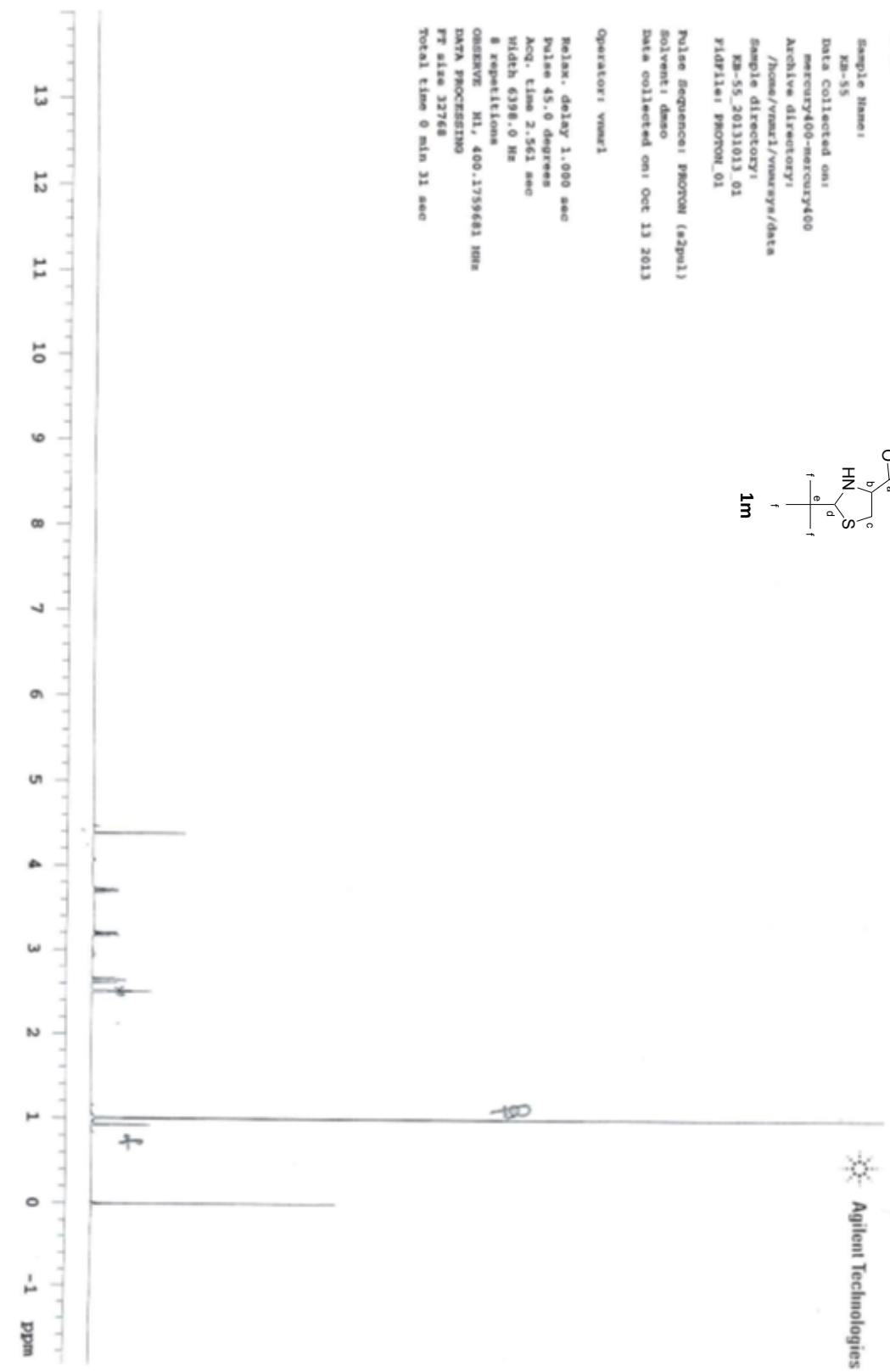
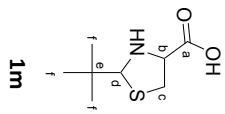
KR-55

Sample Name:  
KR-55  
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Archive directory:  
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Sample directory:  
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FID11a1: PROTON\_01

Pulse Sequence: PROTON (zgpg1)  
Solvent: dms  
Data collected on: Oct 13 2013

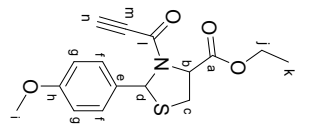
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DATA PROCESSING  
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Total time 0 min 31 sec

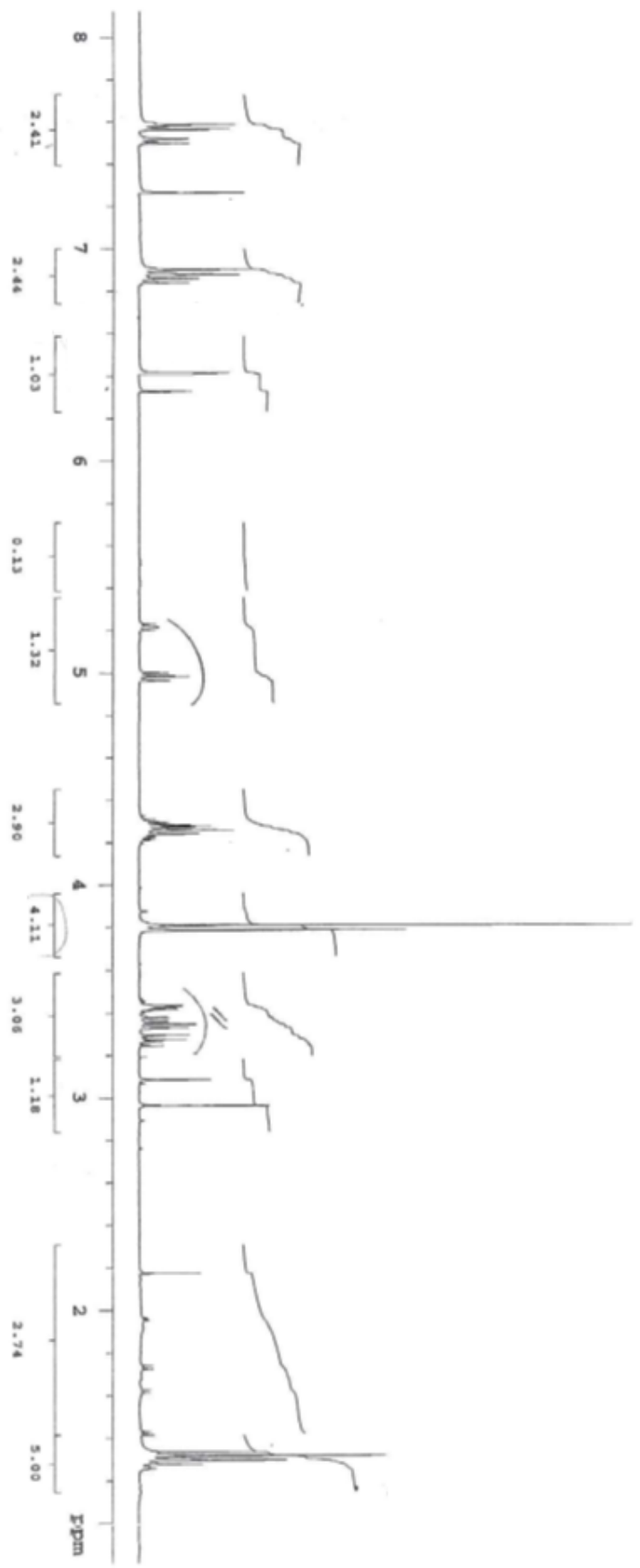


nm





3a



KB8-3.sp0c\_10Apr2012

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File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400MH mercury460°

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.992 sec

Width 6402.0 Hz

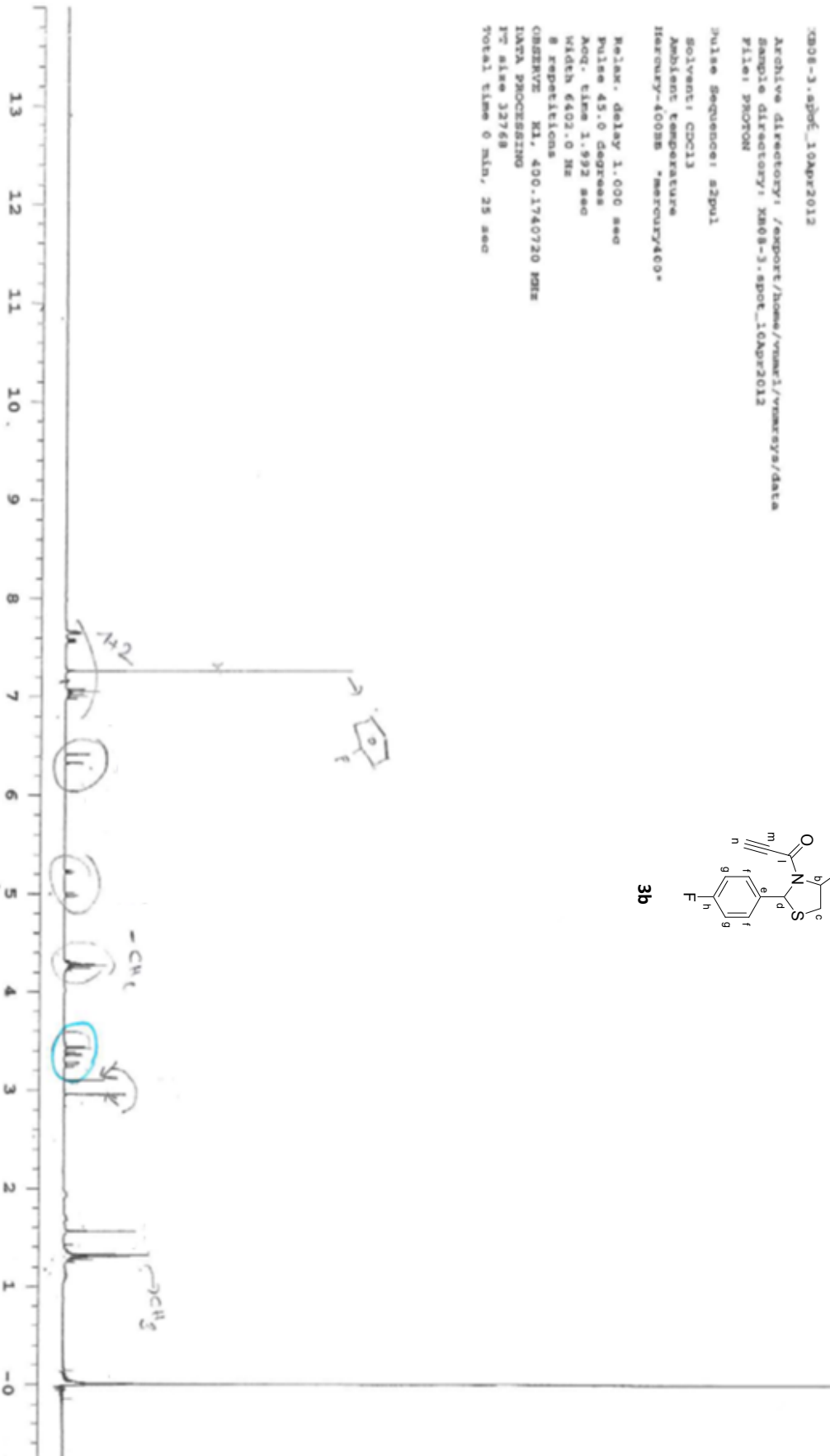
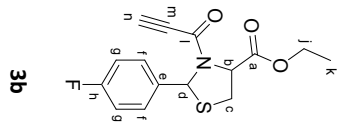
8 repetitions

OBSERVE K1, 400.1740720 MHz

DATA PROCESSING

IT' size 32768

Total time 0 min, 25 sec



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Sample directory: M68\_10oct2012  
File: PROTON

Pulse sequence: zgpg30

Solvent: CDCl3

Ambient temperature

Mercury-400MHz Mercury400-

Relax. delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 1.992 sec

Width: 6462.0 Hz

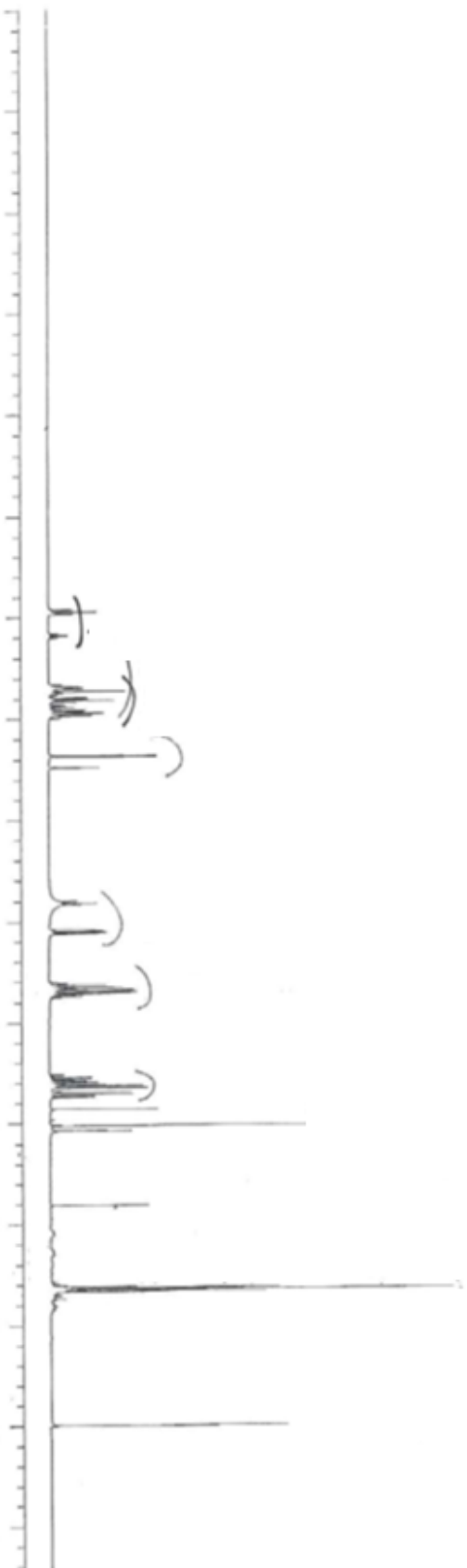
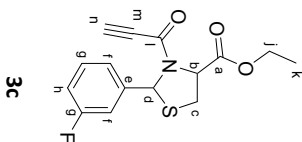
# repetitions

CHERRY HL, 400.1746693 MHz

DATA PROCESSING

FF size 32768

Total time 0 min, 25 sec



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File: PROTON

Pulse Sequence: zgpg30

Solvent: CDCl3  
Ambient temperature  
Mercury-400MB "mercury400"

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Pulse 45.0 degrees

Acq. time 1.992 sec

Width 6402.0 Hz

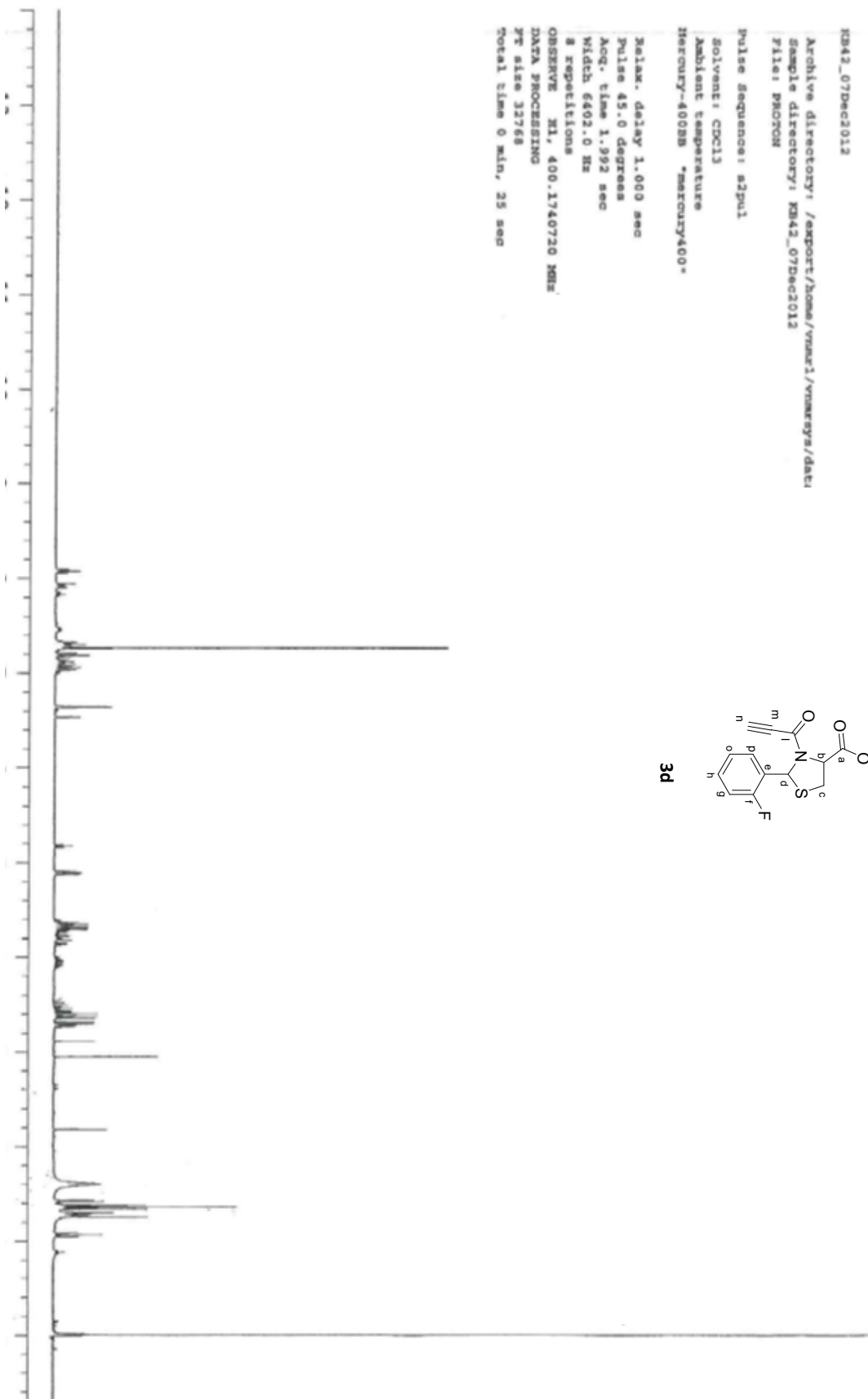
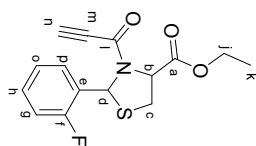
8 repetitions

OBSERVE H1, 400.1740720 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 25 sec





Archive directory: /export/home/wwm1/vnmrsgs/data  
Sample directory: RM31-4\_10Oct2012-15155129  
File: PMOTON

Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

Mercury-400MHz Mercury400+

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.992 sec

Width 6402.0 Hz

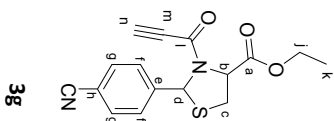
8 repetitions

OBSERVE H1, 400.1740712 MHz

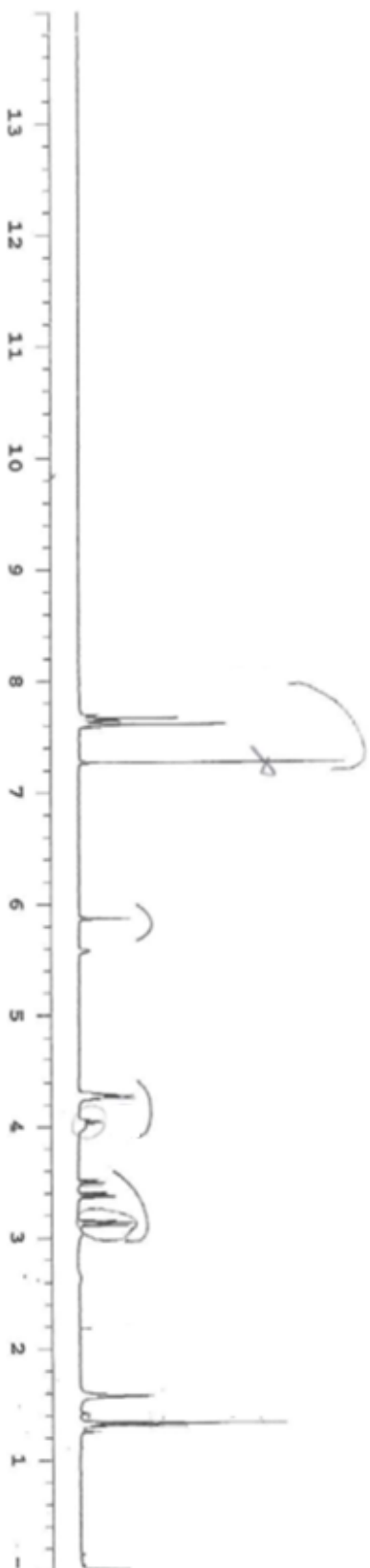
DATA PROCESSING

FF size 32768

Total time 0 min, 25 sec



3b



Archive directory: /export/home/vmmart/vmmartys/data  
Sample directory: KM51-3\_25Aug2012  
File: PROTON

Pulse Sequence: zgpg1

Solvent: CDCl3

Ambient temperature

Mercury-400MHz Mercury400-

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.992 sec

Width 6402.0 Hz

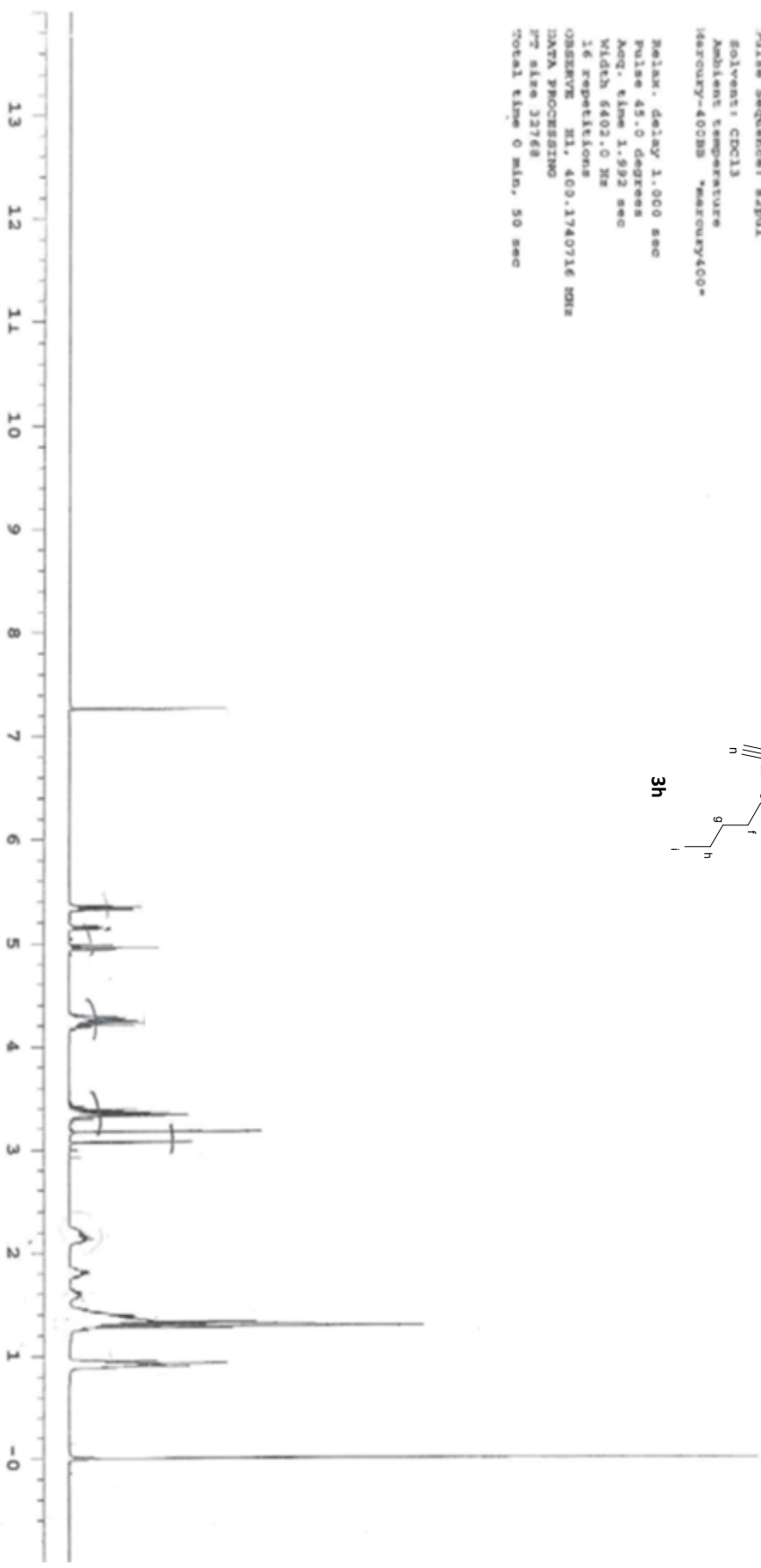
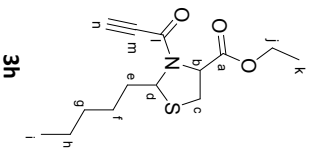
16 repetitions

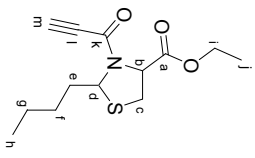
OBSERVE SI, 400.1740716 MHz

DATA PROCESSING

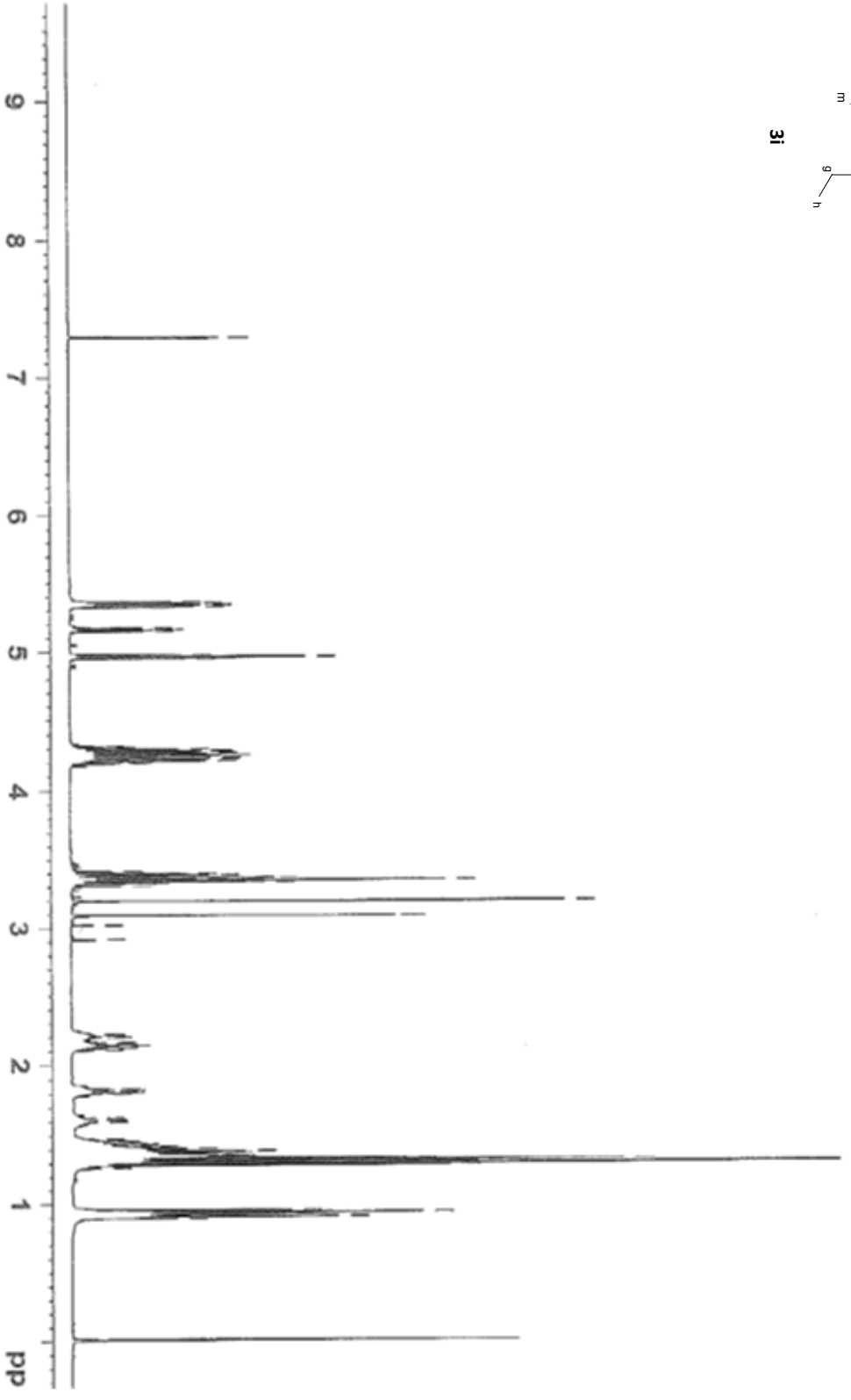
FT size 32768

Total time 0 min, 50 sec





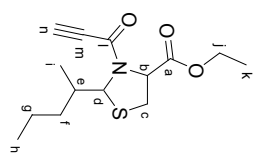
3i



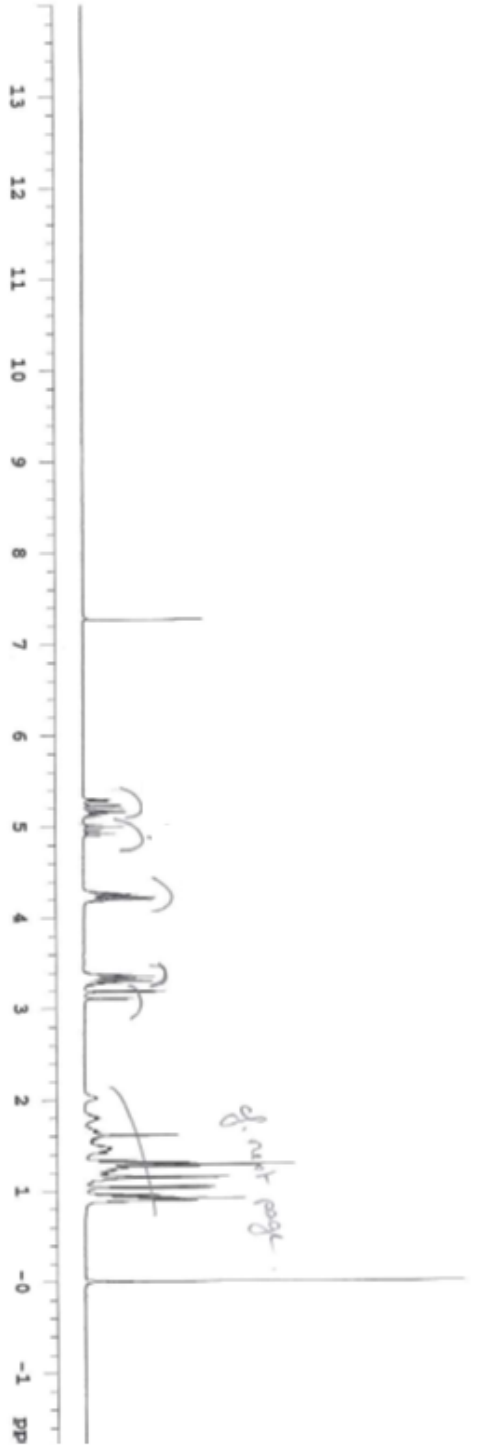
Archive directory: /export/home/vmwt/vmwt/nuvry/data  
Sample directory: MS60-3\_18Oct2012  
File: MS60M

Pulse sequence: zgpg1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400MHz "mercury400"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6402.0 Hz  
8 repetitions  
OBSERVE: H1, 400.1746701 MHz  
DATA PROCESSING  
PC size 32768  
Total time 0 min, 35 sec



31

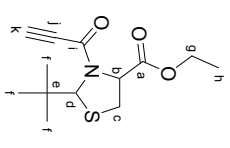


Archive directory: /export/home/vmari/vmariys/data  
Sample directory: X8-61\_25Aug2012  
File: PROGRAM

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Mercury-400MHz mercury400\*

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6402.0 Hz  
16 repetitions  
OBSERVE N1, 400.1740993 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 50 sec



3m

