



Supporting Information

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The First Organocatalytic Enantioselective

Reduction of Pyridines

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General: Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were technical grade and distilled prior to use. Benzene used was reagent grade and distilled from P₂O₅. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator and visualised by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.040-0.063 mm). Solvent mixtures are understood as volume/volume.

¹H-NMR and ¹³C-NMR were recorded on a Bruker AM 250 or a Bruker AV 400 spectrometer in CDCl₃. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated by s (broadened singlet), s (singlet), d (doublet), t (triplet), m (multiplet)); coupling constants (J) are in Hertz (Hz). Mass spectra (MS-EI, 70 eV) were conducted on GC-MS Shimadzu QP2010 (column: Equity[®]-5, length × I.D. 30 m × 0.25 mm, d_f 0.25 μm, lot # 28089-U, Supelco). IR spectra were recorded on a Jasco FT/IR-420 spectrometer and are reported in terms of frequency of absorption (cm⁻¹). Optical rotations were measured on a Perkin Elmer 241 polarimeter. The enantiomeric excesses were determined by HPLC analysis using a chiral stationary phase column (column: CHIRALCEL OD-H or CHIRALPAK AS-H or CHIRALPAK AD-H; eluent: hexane/2-propanol). The chiral HPLC method were calibrated with the corresponding racemic mixtures. Chemical yields refer to pure isolated substances. The yields and enantiomeric excesses are given in the corresponding tables.

General procedure: Pyridine (0.1 mmol), phosphoric acid **3f** (5 mol%) and Hantzsch dihydropyridine **2** (4 equiv.) were suspended in benzene (2 mL) in a screw-capped vial and flushed with argon. The resulting mixture was allowed to stir at 50°C for 30-36 h. The solvent was removed under reduced pressure and purification of the crude product by column chromatography on silica gel (ethyl acetate/hexane) afforded the pure product.

Physical data:

(2R)-2-propyl-2,3,4,6,7,8-hexahydroquinolin-5(1H)-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =4.50 (br s, 1H), 3.31-3.13 (m, 1H), 2.49 (dt, 1H, $J(\text{H,H})$ =5.0, 16.3 Hz), 2.37-2.09 (m, 5H), 1.99-1.79 (m, 3H), 1.55-1.27 (m, 5H), 0.94 ppm (t, 3H, $J(\text{H,H})$ =6.7 Hz); ^{13}C NMR (100 MHz, CDCl_3 , 25 °C, TMS): δ = 194.38, 158.59, 105.06, 51.33, 37.90, 36.46, 29.50, 27.02, 21.78, 18.86, 18.32, 14.06 ppm; IR (KBr): $\tilde{\nu}$ =3232, 2933, 2860, 1579, 1516, 1404, 1183 cm^{-1} ; MS-EI: m/z (%): 194 (8) $[\text{M}+\text{H}]^+$, 193 (57) $[\text{M}]^{+\bullet}$, 164 (13), 151 (89), 150 (100) $[\text{M}-\text{C}_3\text{H}_7]^+$, 137 (8), 136 (28), 123 (11), 122 (49), 94 (28), 93 (11), 55 (13); $[\alpha]_{\text{D}}^{\text{RT}}$ =+180.6 (c=1.0 in CHCl_3), $[\alpha]_{\text{D}}^{\text{RT}}$ =+31.9 (c=0.2 in EtOH); Lit.¹ $[\alpha]_{\text{D}}^{\text{RT}}$ =-45 (c=0.2 in EtOH) for *S*-enantiomer; HPLC conditions: OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL min^{-1} , major enantiomer: t_{R} =10.5 min; minor enantiomer: t_{R} =15.5 min.

(2R)-2-butyl-2,3,4,6,7,8-hexahydroquinolin-5(1H)-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =4.42 (br s, 1H), 3.28-3.12 (m, 1H), 2.50 (dt, 1H, $J(\text{H,H})$ =5.1, 16.3 Hz), 2.37-2.10 (m, 5H), 1.99-1.80 (m, 3H), 1.55-1.26 (m, 7H), 0.91 ppm (t, 3H, $J(\text{H,H})$ =6.8 Hz); ^{13}C NMR (100 MHz, CDCl_3 , 25 °C, TMS): δ = 194.32, 158.67, 104.99, 51.58, 36.47, 35.44, 29.47, 27.81, 27.00, 22.69, 21.78, 18.31, 14.00 ppm; IR (KBr): $\tilde{\nu}$ =3255, 2932, 2855, 1573, 1512, 1404, 1188 cm^{-1} ; MS-EI: $m/z(\%)$: 208 (10) $[\text{M}+\text{H}]^+$, 207 (49) $[\text{M}]^{+\bullet}$, 164 (22), 151 (80), 150 (100) $[\text{M}-\text{C}_4\text{H}_9]^+$, 136 (22), 122 (31), 94 (23), 93 (10), 55 (13); $[\alpha]_{\text{D}}^{\text{RT}}=+170.0$ ($c=1.0$ in CHCl_3); HPLC conditions: OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL min^{-1} , major enantiomer: $t_{\text{R}}=9.9$ min; minor enantiomer: $t_{\text{R}}=13.6$ min.

(2R)-2-pentyl-2,3,4,6,7,8-hexahydroquinolin-5(1H)-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =4.56 (br s, 1H), 3.27-3.12 (m, 1H), 2.48 (dt, 1H, $J(\text{H,H})$ =5.0, 16.0 Hz), 2.36-2.09 (m, 5H), 1.99-1.79 (m, 3H), 1.53-1.19 (m, 9H), 0.88 ppm (t, 3H, $J(\text{H,H})$ =6.7 Hz); ^{13}C NMR (100 MHz, CDCl_3 , 25 °C, TMS): δ =194.34, 158.49, 105.09, 51.61, 36.50, 35.73, 31.78, 29.51, 27.04, 25.34, 22.57, 21.79, 18.34, 14.02 ppm; IR (KBr): $\tilde{\nu}$ =3281, 2932, 2850, 1573, 1522, 1342, 1189 cm^{-1} ; MS-EI: $m/z(\%)$: 222 (6) $[\text{M}+\text{H}]^+$, 221 (42) $[\text{M}]^{+\bullet}$, 164 (29), 151 (83), 150 (100) $[\text{M}-\text{C}_5\text{H}_{11}]^+$, 136 (21), 122 (25), 94 (20), 55 (12); $[\alpha]_{\text{D}}^{\text{RT}}=+154.8$ ($c=1.0$ in CHCl_3); HPLC conditions: OD-H column, n-hexane/2-propanol = 90/10, flow rate

= 1.0 mL min⁻¹, major enantiomer: t_R=9.4 min; minor enantiomer: t_R=12.8 min.

(2R)-2-(2-phenylethyl)-2,3,4,6,7,8-hexahydroquinolin-5(1H)-one

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.30-7.06 (m, 5H), 4.31 (br s, 1H), 3.29-3.13 (m, 1H), 2.77-2.52 (m, 2H), 2.42 (dt, 1H, J(H,H)=5.4, 16.3 Hz), 2.25 (t, 2H, J(H,H)=6.3 Hz), 2.21-2.07 (m, 3H), 1.91-1.67 (m, 5H), 1.55-1.37 ppm (m, 1H); ¹³C NMR (100 MHz, CDCl₃, 25 °C, TMS): δ=194.43, 158.25, 141.31, 128.67, 128.64, 128.33, 128.31, 126.24, 105.08, 51.21, 37.14, 36.49, 32.24, 29.44, 26.92, 21.75, 18.05 ppm; IR (KBr): $\tilde{\nu}$ =3268, 2938, 2857, 1567, 1511, 1404, 1186 cm⁻¹; MS-EI: m/z(%):256 (8) [M+H]⁺, 255 (44) [M]⁺, 151 (100) [M-C₈H₈]⁺, 150 (51), 136 (20), 123 (21), 122 (25), 94 (15), 91 (21); [α]_D^{RT}=+148.8 (c=1.0 in CHCl₃); HPLC conditions: OD-H column, n-hexane/2-propanol = 80/20, flow rate = 1.0 mL min⁻¹, major enantiomer: t_R=18.4 min; minor enantiomer: t_R=23.8 min.

(2R)-2-(non-3-en-1-yl)-2,3,4,6,7,8-hexahydroquinolin-5(1H)-one

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=5.55-5.25 (m, 2H), 4.55 (br s, 1H), 3.31-3.17 (m, 1H), 2.47 (dt, 1H, J(H,H)=5.3, 16.0 Hz), 2.37-1.78 (m, 12H), 1.54 (q, 2H, J(H,H)=7.3), 1.49-1.18 (m, 7H), 0.87 ppm (t, 3H, J(H,H)=6.7 Hz); ¹³C NMR (100 MHz, CDCl₃, 25 °C, TMS): δ=194.36, 158.42, 131.76, 128.94, 105.04, 51.20, 36.48, 35.34, 32.53, 31.40, 29.50, 29.20, 28.91, 26.97, 22.53, 21.78, 18.18, 14.07 ppm; IR (KBr): $\tilde{\nu}$ =3222, 2923, 2851, 1578,

1513, 1406, 1184 cm^{-1} ; MS-EI: $m/z(\%)$: 276 (13) $[\text{M}+\text{H}]^+$, 275 (62) $[\text{M}]^{+\bullet}$, 274 (23) $[\text{M}-\text{H}]^+$, 218 (17), 176 (11), 164 (12), 162 (18), 152 (10), 151 (100) $[\text{M}-\text{C}_9\text{H}_{16}]^{+\bullet}$, 150 (96) $[\text{M}-\text{C}_9\text{H}_{17}]^+$, 148 (99), 136 (23), 124 (18), 123 (45), 122 (17), 107 (10), 94 (24), 79 (10), 67 (10), 55 (22); $[\alpha]_{\text{D}}^{\text{RT}} = +122.3$ ($c=1.0$ in CHCl_3); HPLC conditions: OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL min^{-1} , major enantiomers: $t_{\text{R(cis+trans)}}=8.8$ min; minor enantiomers: $t_{\text{R(cis)}}=11.7$ min and $t_{\text{R(trans)}}=12.7$ min.

(2R)-2-decyl-2,3,4,6,7,8-hexahydroquinolin-5(1H)-one

^1H NMR (250 MHz, CDCl_3 , 25 $^{\circ}\text{C}$, TMS): $\delta=4.44$ (br s, 1H), 3.28-3.12 (m, 1H), 2.49 (dt, 1H, $J(\text{H,H})=5.3, 16.0$ Hz), 2.37-2.10 (m, 5H), 2.01-1.80 (m, 3H), 1.55-1.16 (m, 19H), 0.87 ppm (t, 3H, $J(\text{H,H})=6.7$ Hz); ^{13}C NMR (100 MHz, CDCl_3 , 25 $^{\circ}\text{C}$, TMS): $\delta=194.39, 158.32, 105.21, 51.63, 36.50, 35.81, 31.91, 29.61, 29.59, 29.56, 29.34, 27.10, 25.68, 22.70, 21.79, 18.36, 14.13$ ppm; IR (KBr): $\tilde{\nu}=3226, 2922, 2851, 1579, 1513, 1406, 1183 \text{ cm}^{-1}$; MS-EI: $m/z(\%)$: 292 (8) $[\text{M}+\text{H}]^+$, 291 (39) $[\text{M}]^{+\bullet}$, 290 (7) $[\text{M}-\text{H}]^+$, 206 (11), 192 (9), 164 (32), 152 (12), 151 (100) $[\text{M}-\text{C}_{10}\text{H}_{20}]^+$, 150 (100) $[\text{M}-\text{C}_{10}\text{H}_{21}]^+$, 136 (17), 124 (13), 122 (14), 94 (14); $[\alpha]_{\text{D}}^{\text{RT}} = +135.5$ ($c=1.0$ in CHCl_3); HPLC conditions: OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL min^{-1} , major enantiomer: $t_{\text{R}}=8.2$ min; minor enantiomer: $t_{\text{R}}=11.2$ min.

(6R)-6-butyl-2-methyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =3.99 (br s, 1H), 3.18-3.00 (m, 1H), 2.25-2.18 (m, 2H), 1.94 (t, 3H, $J(\text{H,H})$ =1.1 Hz), 1.84-1.69 (m, 1H), 1.47-1.14 (m, 7H), 0.85 ppm (t, 3H, $J(\text{H,H})$ =6.8 Hz); ^{13}C NMR (100 MHz, CDCl_3 , 25 °C, TMS): δ =152.41, 123.43, 71.98, 51.26, 35.39, 27.73, 26.62, 22.66, 22.60, 19.95, 13.99 ppm; IR (KBr): $\tilde{\nu}$ =2929, 2858, 2180, 1726, 1616, 1528, 1297 cm^{-1} ; MS-EI: $m/z(\%)$: 178 (18) $[\text{M}]^{+\bullet}$, 122 (18), 121 (100) $[\text{M}-\text{C}_4\text{H}_9]^+$, 119 (7), 95 (5), 94 (9), 82 (5); $[\alpha]_{\text{D}}^{\text{RT}}$ =+112.6 (c =1.0 in CHCl_3); HPLC conditions: AS-H column, n-hexane/2-propanol = 97/3, flow rate = 1.0 mL min^{-1} , major enantiomer: t_{R} =49.5 min; minor enantiomer: t_{R} =55.2 min.

(6R)-2-methyl-6-pentyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =4.07 (br s, 1H), 3.23-3.07 (m, 1H), 2.31-2.17 (m, 2H), 2.00 (t, 3H, $J(\text{H,H})$ =1.1 Hz), 1.90-1.73 (m, 1H), 1.52-1.19 (m, 9H), 0.89 ppm (t, 3H, $J(\text{H,H})$ =6.7 Hz); ^{13}C NMR (100 MHz, CDCl_3 , 25 °C, TMS): δ =152.43, 123.43, 71.96, 51.28, 35.66, 31.74, 26.62, 25.26, 22.61, 22.57, 19.96, 14.01 ppm; IR (KBr): $\tilde{\nu}$ =2928, 2857, 2179, 1615, 1525, 1300 cm^{-1} ; MS-EI: $m/z(\%)$: 192 (16) $[\text{M}]^{+\bullet}$, 135 (8), 122 (20), 121 (100) $[\text{M}-\text{C}_5\text{H}_{11}]^+$, 119 (6), 94 (8); $[\alpha]_{\text{D}}^{\text{RT}}$ =+124.4 (c =1.0 in CHCl_3); HPLC conditions: AS-H column, n-hexane/2-propanol = 97/3, flow rate = 1.0 mL min^{-1} , major enantiomer: t_{R} =48.5 min; minor enantiomer: t_{R} =50.7 min.

(6R)-2-methyl-6-(2-phenylethyl)-1,4,5,6-tetrahydropyridine-3-carbonitrile

^1H NMR (400 MHz, CDCl_3 , 25 °C, TMS): δ =7.28-7.06 (m, 5H), 3.88 (br s, 1H), 3.21-3.10 (m, 1H), 2.72-2.53 (m, 2H), 2.26-2.12 (m, 2H), 1.88 (s, 3H), 1.82-1.67 (m, 2H), 1.54-1.40 ppm (m, 2H); ^{13}C NMR (100 MHz, CDCl_3 , 25 °C, TMS): δ =152.19, 141.13, 128.67, 128.29, 126.29, 123.22, 72.22, 50.84, 37.05, 32.16, 26.49, 22.33, 19.93 ppm; IR (KBr): $\tilde{\nu}$ =2925, 2850, 2179, 1732, 1616, 1523, 1296 cm^{-1} ; MS-EI: m/z (%):227 (8) $[\text{M}+\text{H}]^+$, 226 (46) $[\text{M}]^{+\bullet}$, 135 (7), 123 (6), 122 (73), 121 (100) $[\text{M}-\text{C}_8\text{H}_9]^+$, 119 (11), 105 (7), 94 (11), 91 (34), 82 (20), 65 (10); $[\alpha]_{\text{D}}^{\text{RT}}=+28.6$ ($c=0.5$ in CHCl_3); HPLC conditions: AD-H column, n-hexane/2-propanol = 97/3, flow rate = 1.0 mL min^{-1} , major enantiomer: $t_{\text{R}}=30.5$ min; minor enantiomer: $t_{\text{R}}=34.0$ min.

(6R)-6-decyl-2-methyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

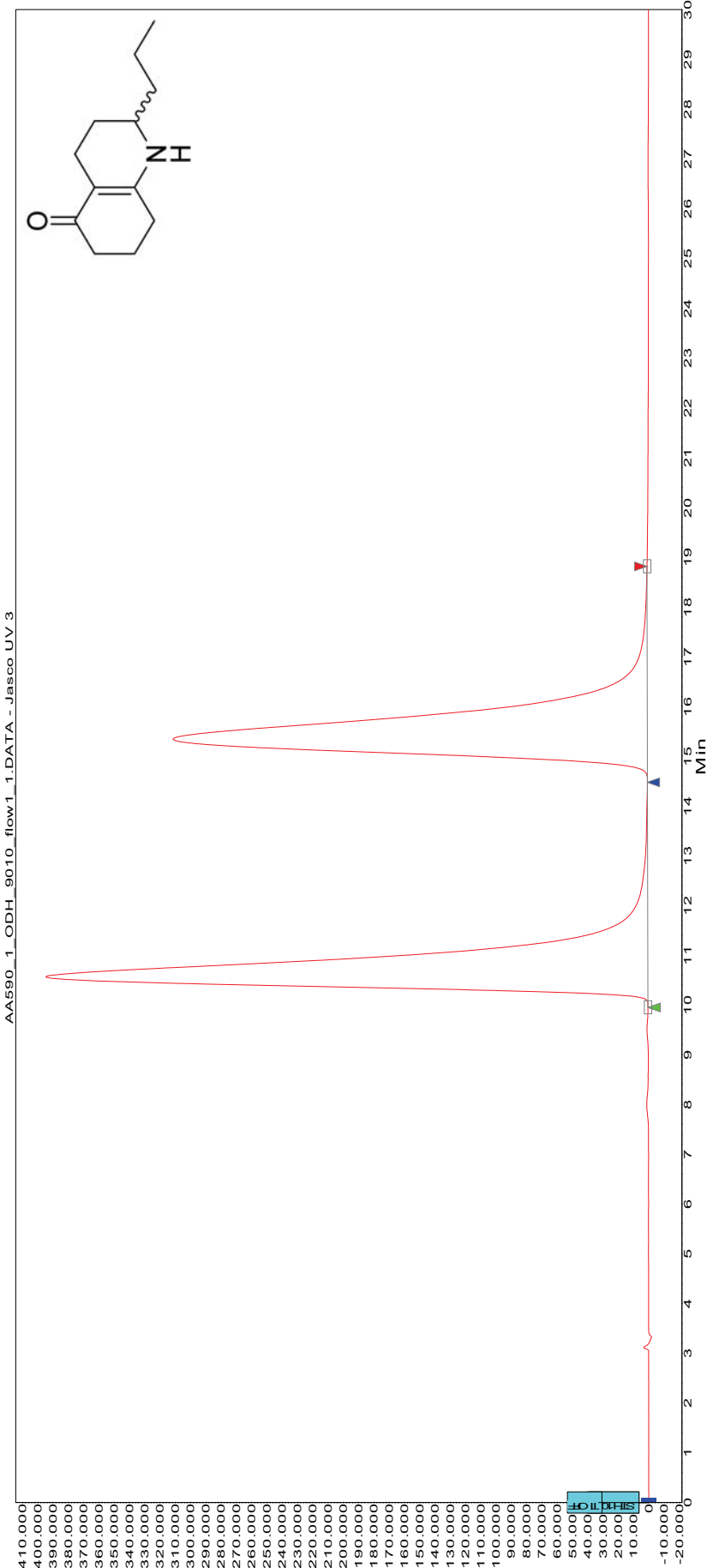
^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =4.02 (br s, 1H), 3.24-3.05 (m, 1H), 2.32-2.18 (m, 2H), 2.00 (s, 3H), 1.90-1.75 (m, 1H), 1.54-1.15 (m, 19H), 0.88 ppm (t, 3H, $J(\text{H},\text{H})=6.7$ Hz); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =152.38, 123.41, 72.04, 51.28, 35.71, 31.92, 29.61, 29.58, 29.55, 29.34, 26.64, 25.59, 22.70, 22.63, 19.97, 14.14 ppm; IR (KBr): $\tilde{\nu}$ =2924, 2853, 2182, 1700, 1644, 1558, 1285 cm^{-1} ; MS-EI: m/z (%):263 (4) $[\text{M}+\text{H}]^+$, 262 (18) $[\text{M}]^{+\bullet}$, 177 (5), 163 (5), 135 (11), 122 (27), 121 (100) $[\text{M}+\text{C}_{10}\text{H}_{21}]^+$, 119 (7), 95 (8), 94 (6), 82 (5); $[\alpha]_{\text{D}}^{\text{RT}}=+73.5$ ($c=1.0$ in CHCl_3); HPLC conditions: AS-H column, n-hexane/2-propanol = 95/5, flow

rate = 1.0 mL min⁻¹, major enantiomer: t_R=15.1 min; minor enantiomer: t_R=16.3 min.

1 (a) H. M. Sklenicka, R. P. Hsung, M. J. McLaughlin, L. L. Wei, A. I. Gerasyuto, W.B. Brennessel, *J. Am. Chem. Soc.* **2002**, 124, 10435. (b) H. M. Sklenicka, R.P. Hsung, L. L. Wei, M. J. McLaughlin, A. I. Gerasyuto, S.J. Degen, *Org. Lett.* **2000**, 2, 1161.

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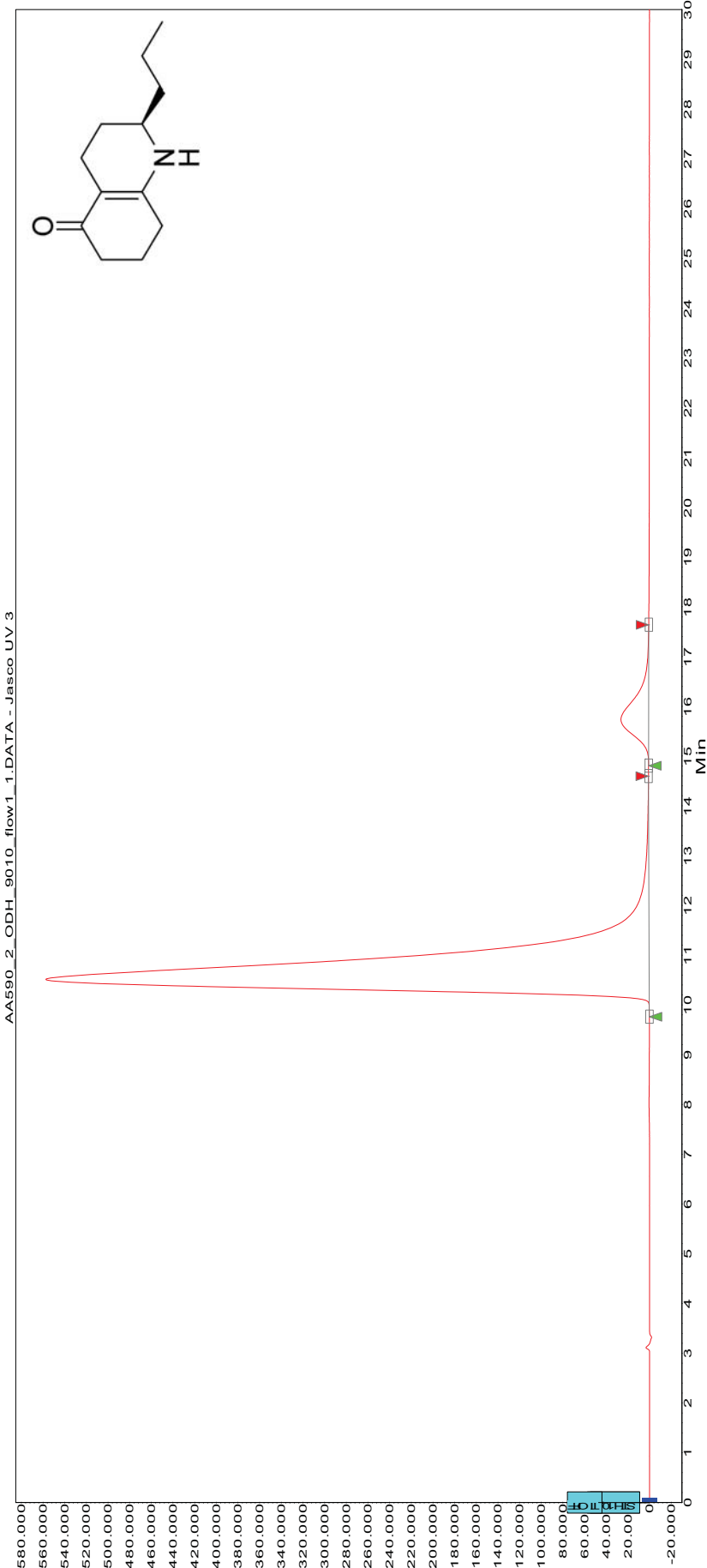
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Date: 19.01.2007 00:41:14



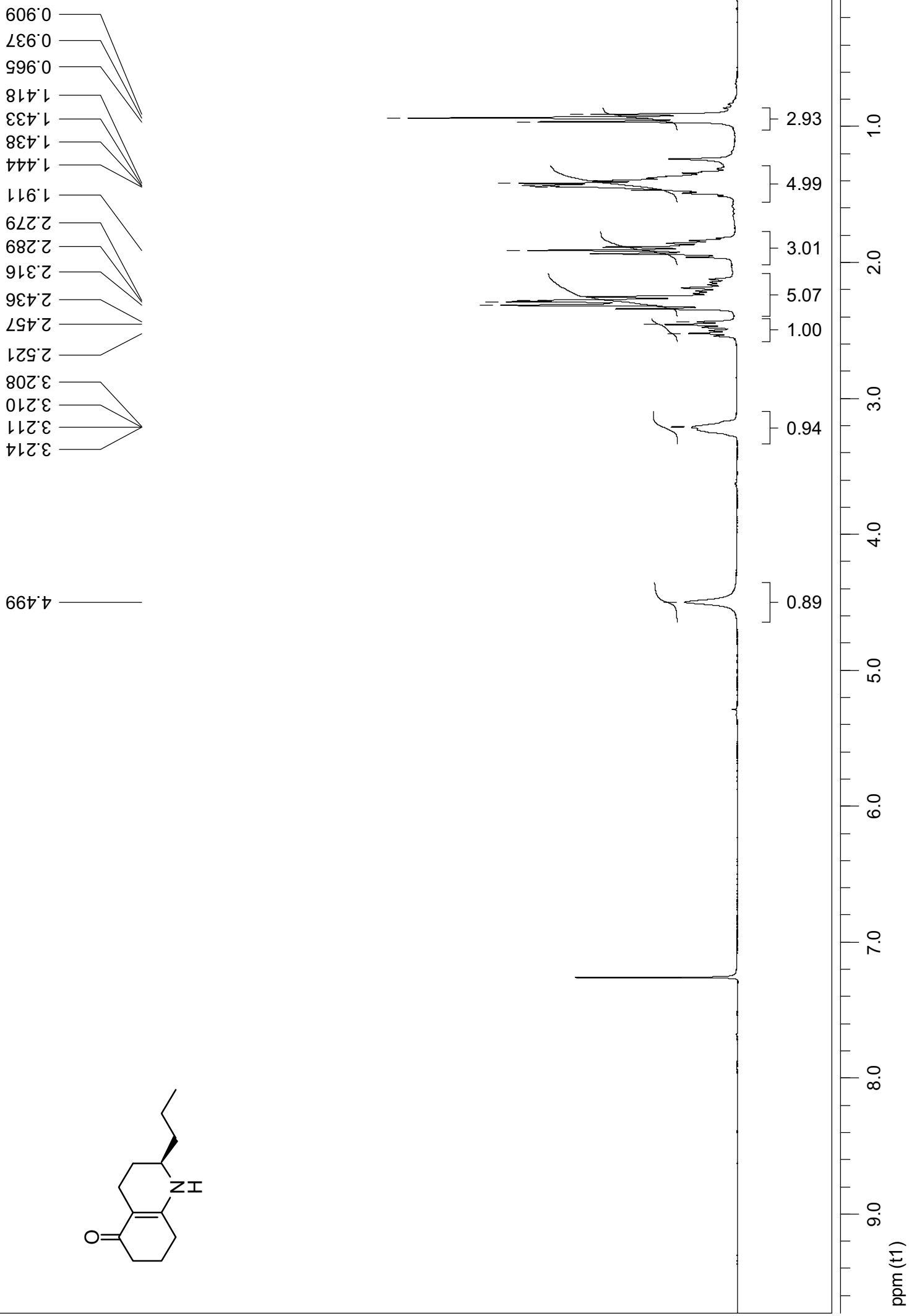
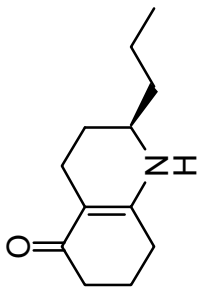
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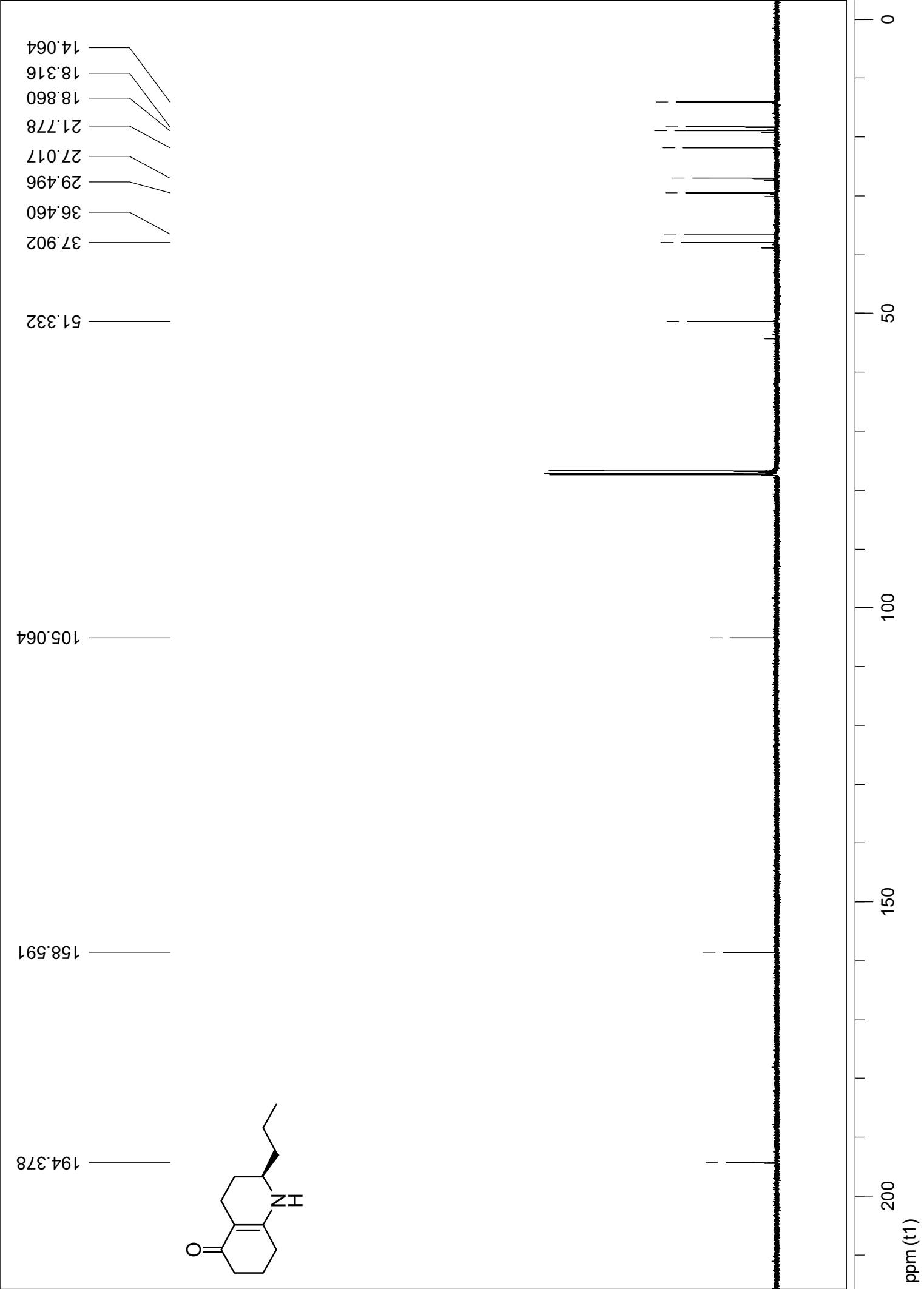
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1	14,806	15,733	17,636	5,470				
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Sample Information

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 Sample Name : AA590-2

Method

===== Analytical Line 1 =====

[GC-2010]

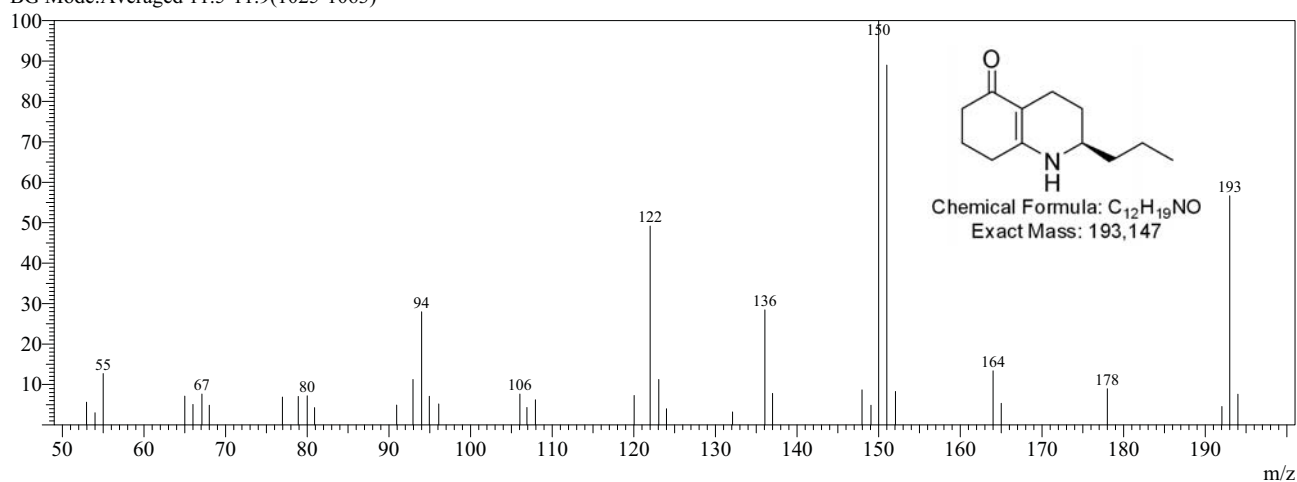
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 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
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20.0	300.0	15.00

Spectrum

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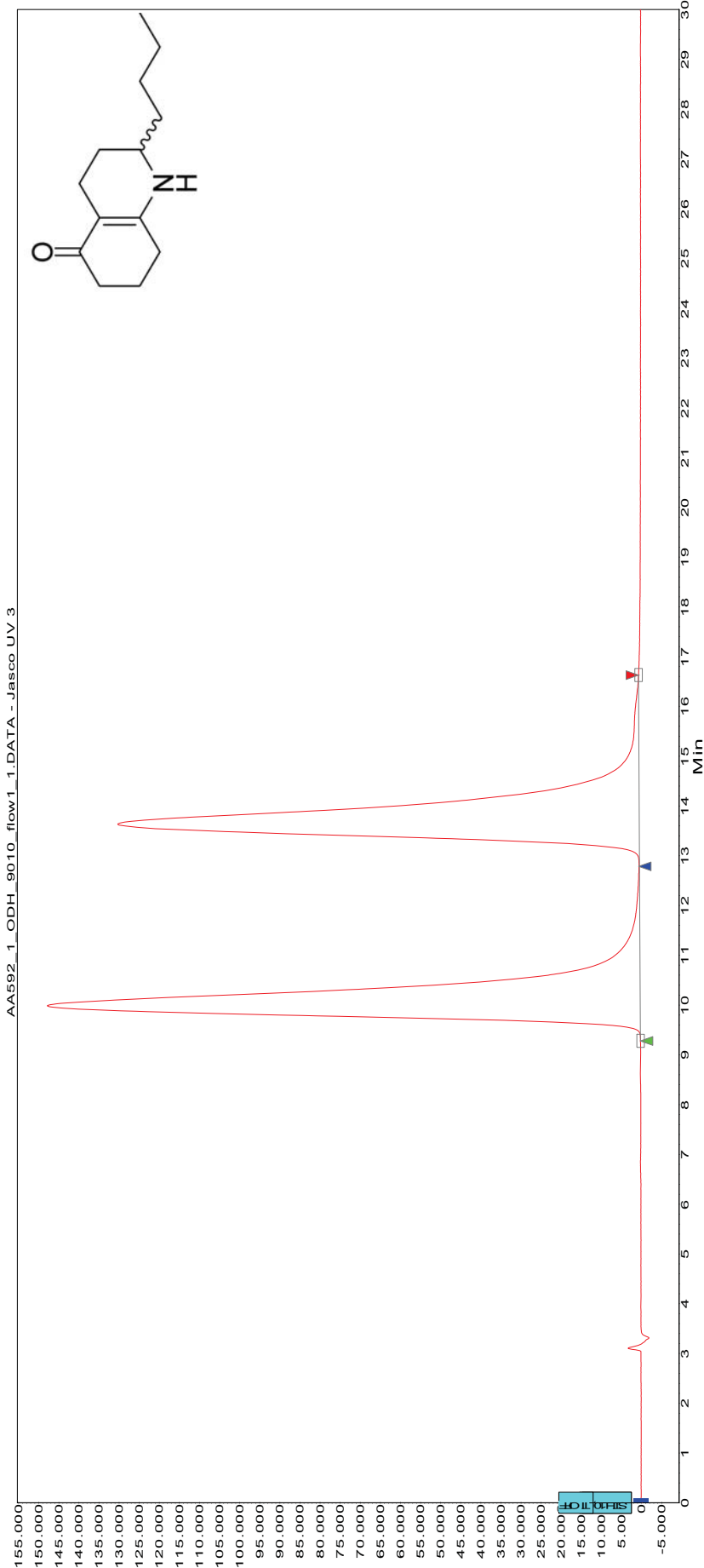
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2	54.00	3.00	12	90.95	4.92	22	123.05	11.23	32	164.00	13.36
3	55.00	12.68	13	92.95	11.22	23	124.00	4.00	33	165.00	5.36
4	65.00	7.11	14	94.00	27.96	24	132.10	3.19	34	178.00	8.94
5	66.00	5.05	15	94.95	7.09	25	136.05	28.44	35	192.05	4.55
6	67.10	7.64	16	96.10	5.17	26	137.00	7.81	36	193.00	56.65
7	68.00	4.87	17	106.05	7.63	27	147.95	8.68	37	194.00	7.58
8	76.95	6.89	18	106.90	4.30	28	149.05	4.88			
9	78.90	7.04	19	107.95	6.20	29	150.00	100.00			
10	80.00	7.18	20	120.05	7.27	30	151.00	88.99			

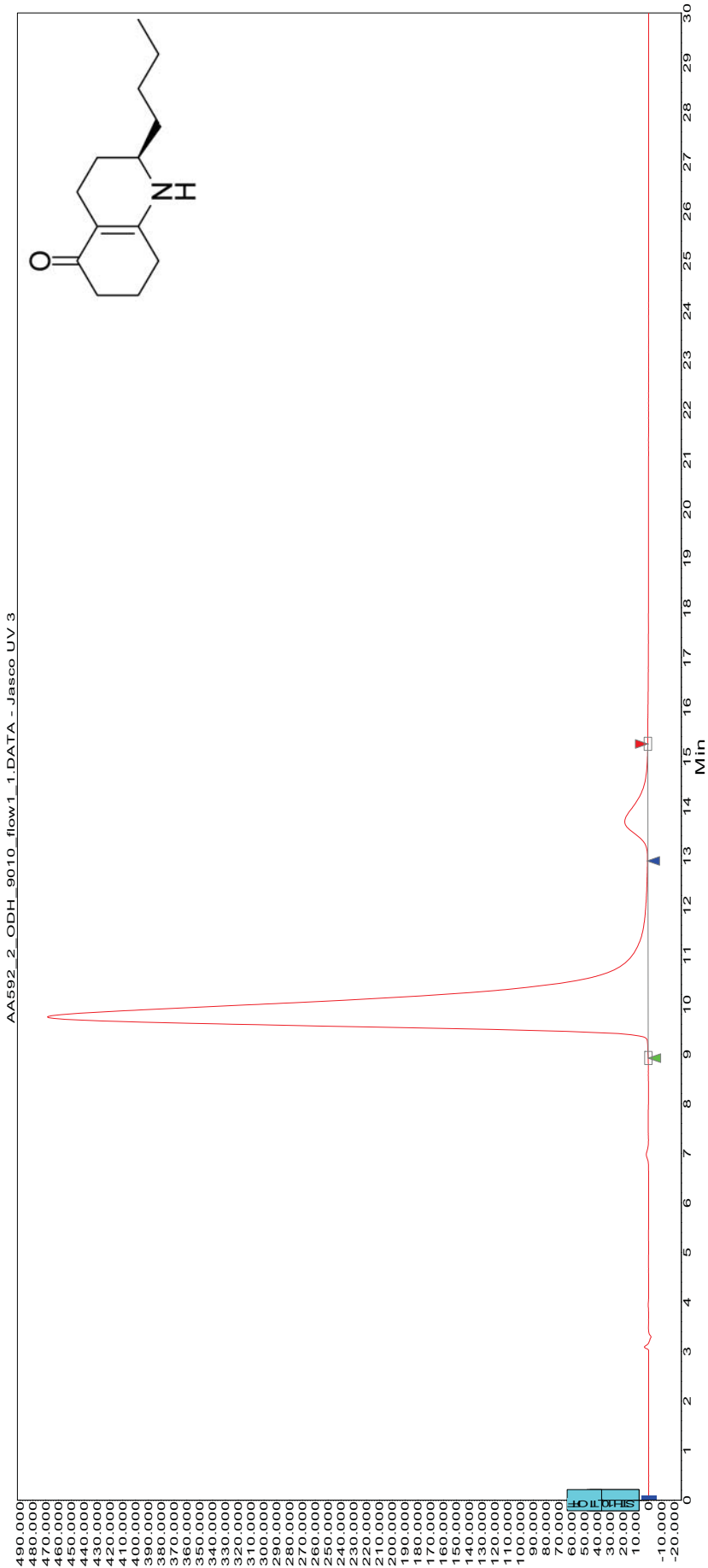
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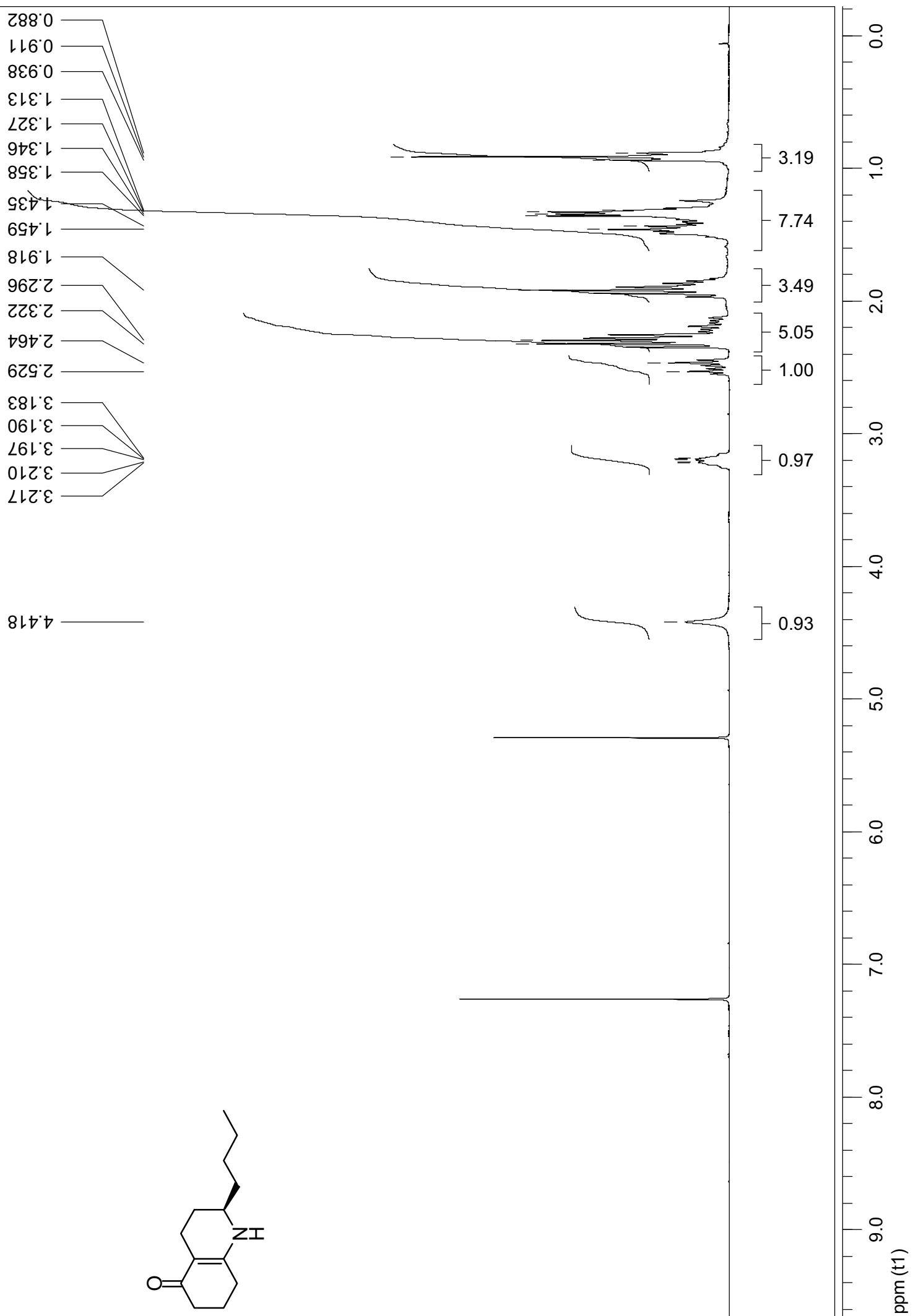
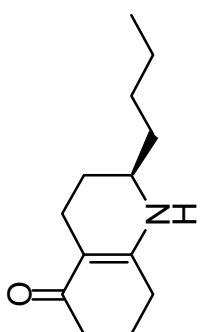
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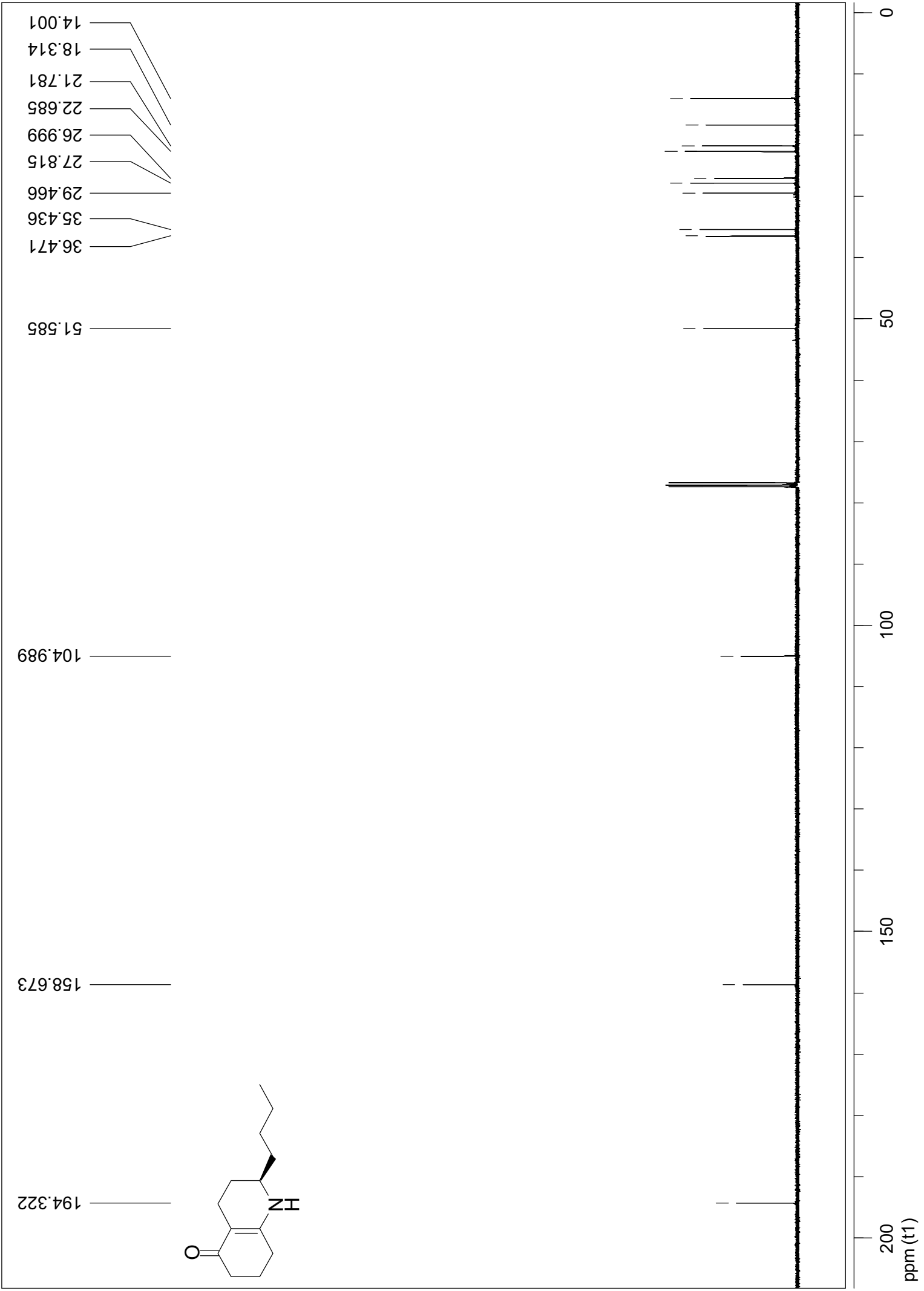


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Sample Information

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Method

===== Analytical Line 1 =====

[GC-2010]

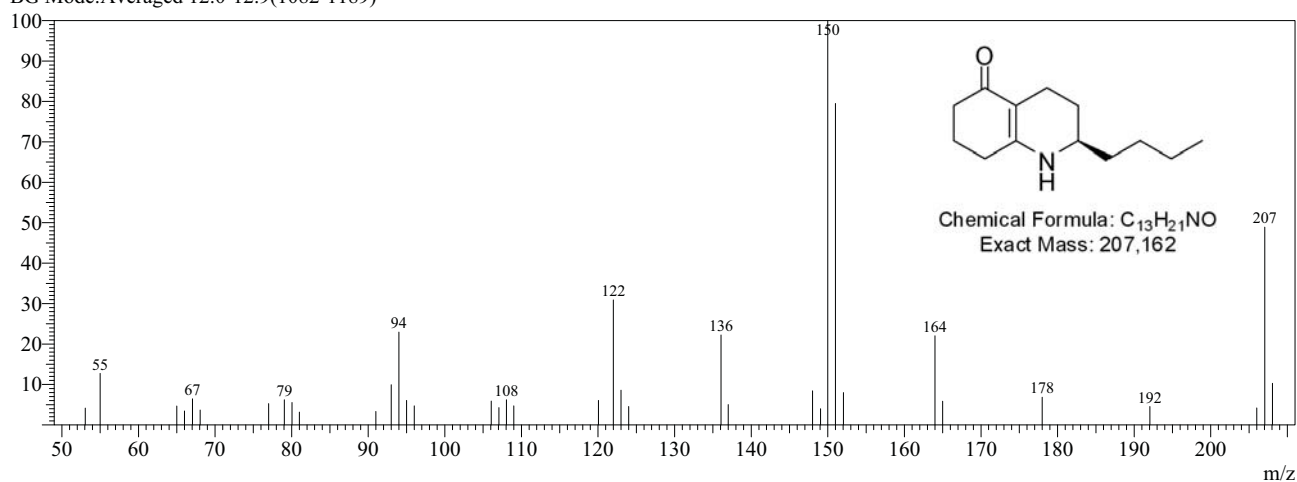
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 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:11.7(Scan#:1043)
 MassPeaks:37 BasePeak:150(671451)
 RawMode:Averaged 11.7-11.7(1042-1044)
 BG Mode:Averaged 12.0-12.9(1082-1189)



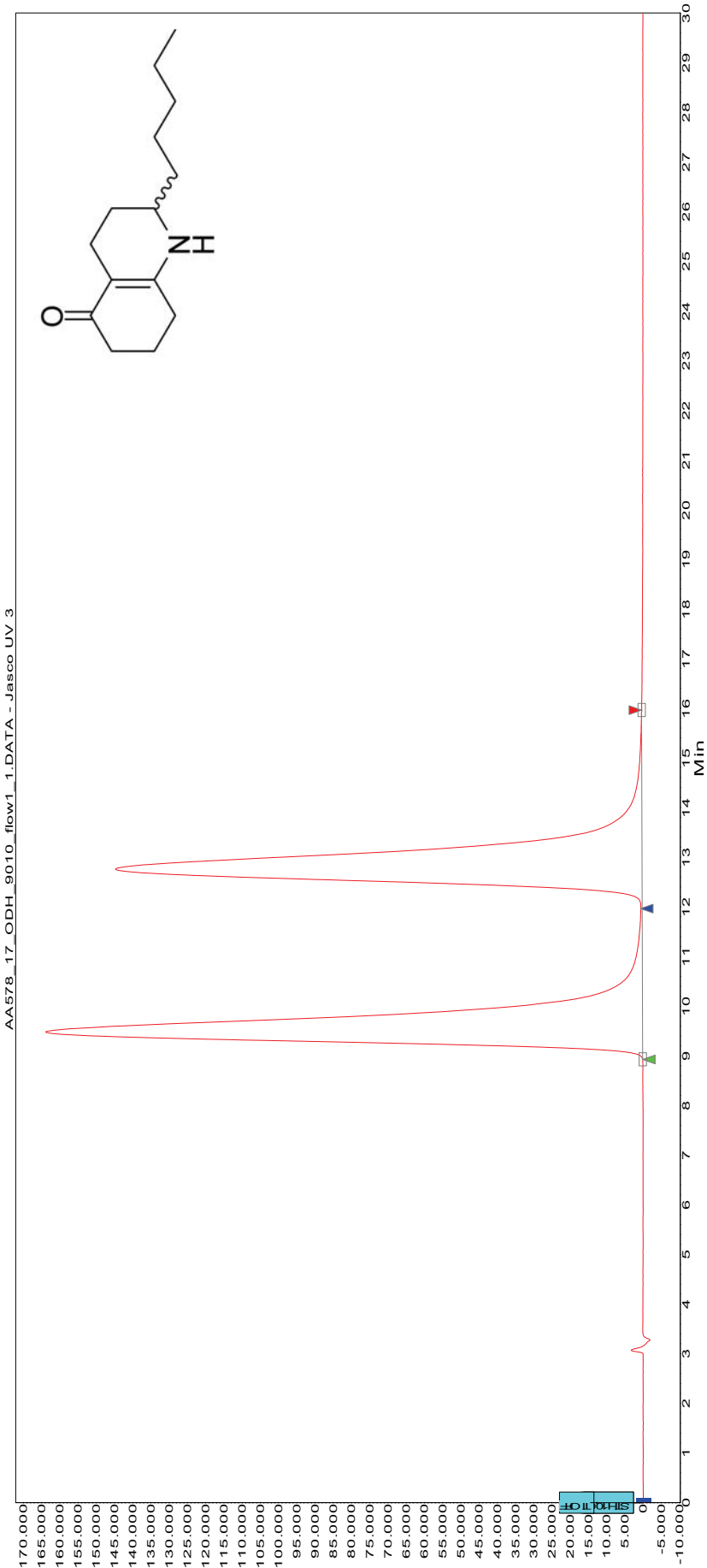
Mass Table

Line#:1 R.Time:11.7(Scan#:1043)
 MassPeaks:37 BasePeak:150(671451)
 RawMode:Averaged 11.7-11.7(1042-1044)
 BG Mode:Averaged 12.0-12.9(1082-1189)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	53.05	4.17	11	91.00	3.34	21	122.00	30.89	31	164.00	22.04
2	55.00	12.73	12	93.00	9.94	22	123.00	8.61	32	165.00	5.87
3	65.00	4.68	13	94.00	22.95	23	124.00	4.53	33	178.00	6.85
4	66.00	3.43	14	95.00	6.05	24	136.05	22.23	34	192.05	4.55
5	67.05	6.43	15	96.00	4.70	25	137.00	5.02	35	206.00	4.19
6	68.05	3.67	16	106.05	5.89	26	148.00	8.43	36	207.05	48.92
7	77.00	5.27	17	107.05	4.26	27	149.05	3.98	37	208.05	10.29
8	79.05	6.23	18	108.05	6.23	28	150.00	100.00			
9	80.05	5.55	19	109.00	4.70	29	151.00	79.50			
10	81.00	3.17	20	120.05	6.06	30	152.05	8.01			

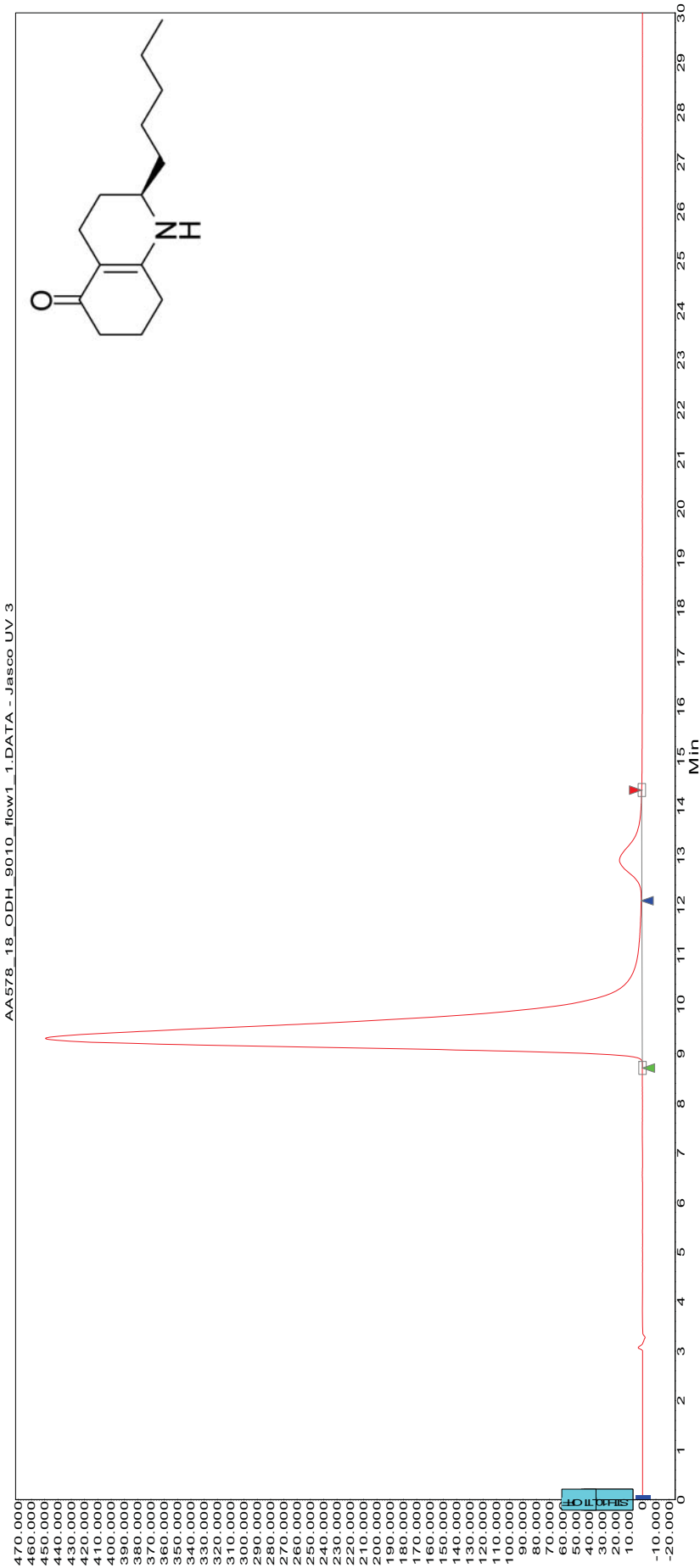
Chromatogram : AA578_17_ODH_9010_flow1_1

Data file: AA578_17_ODH_9010_flow1_1.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 17.01.2007 06:04:36

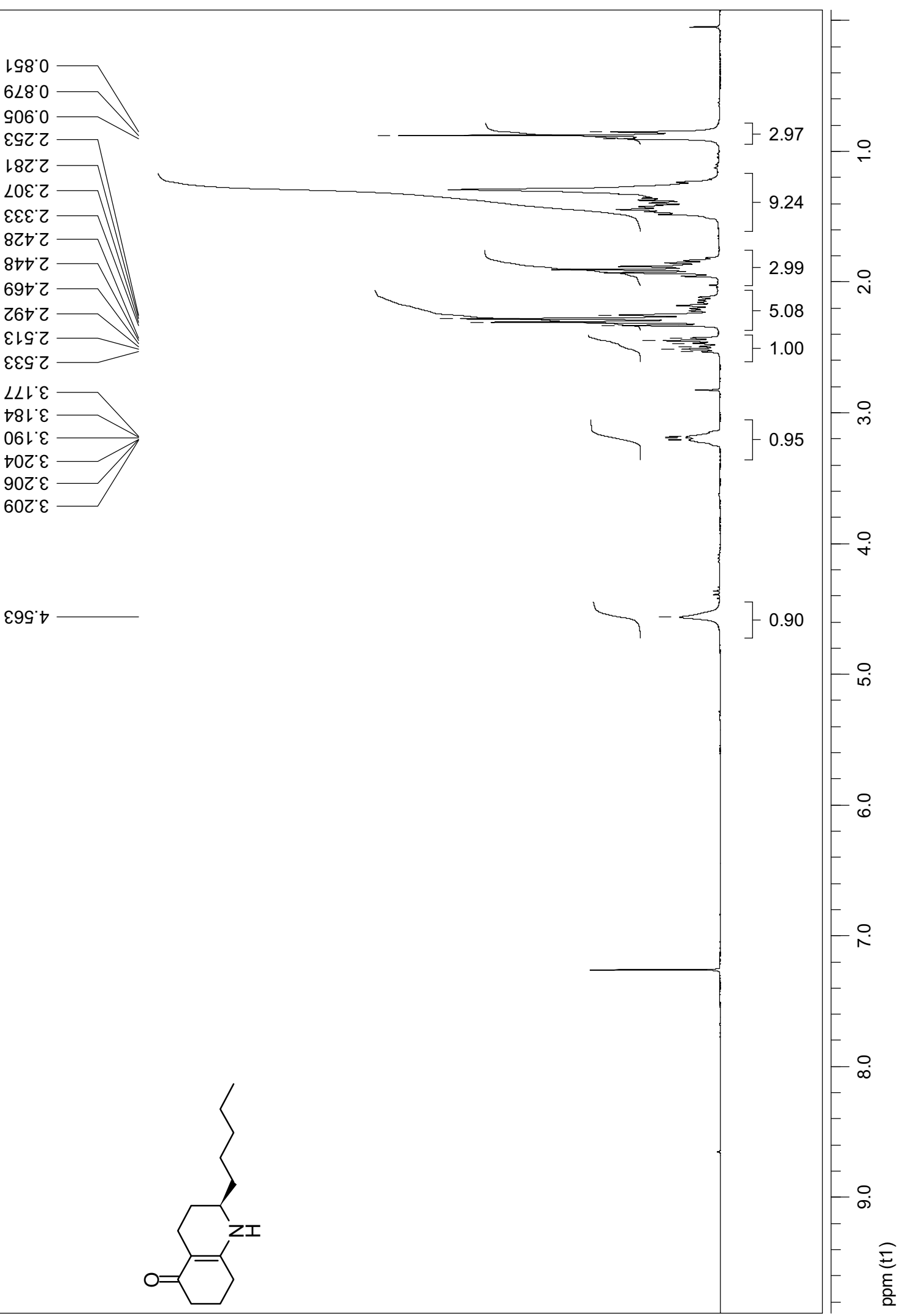
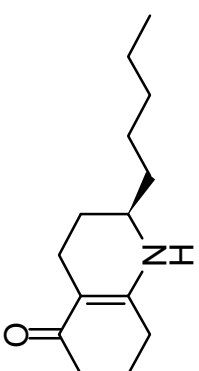


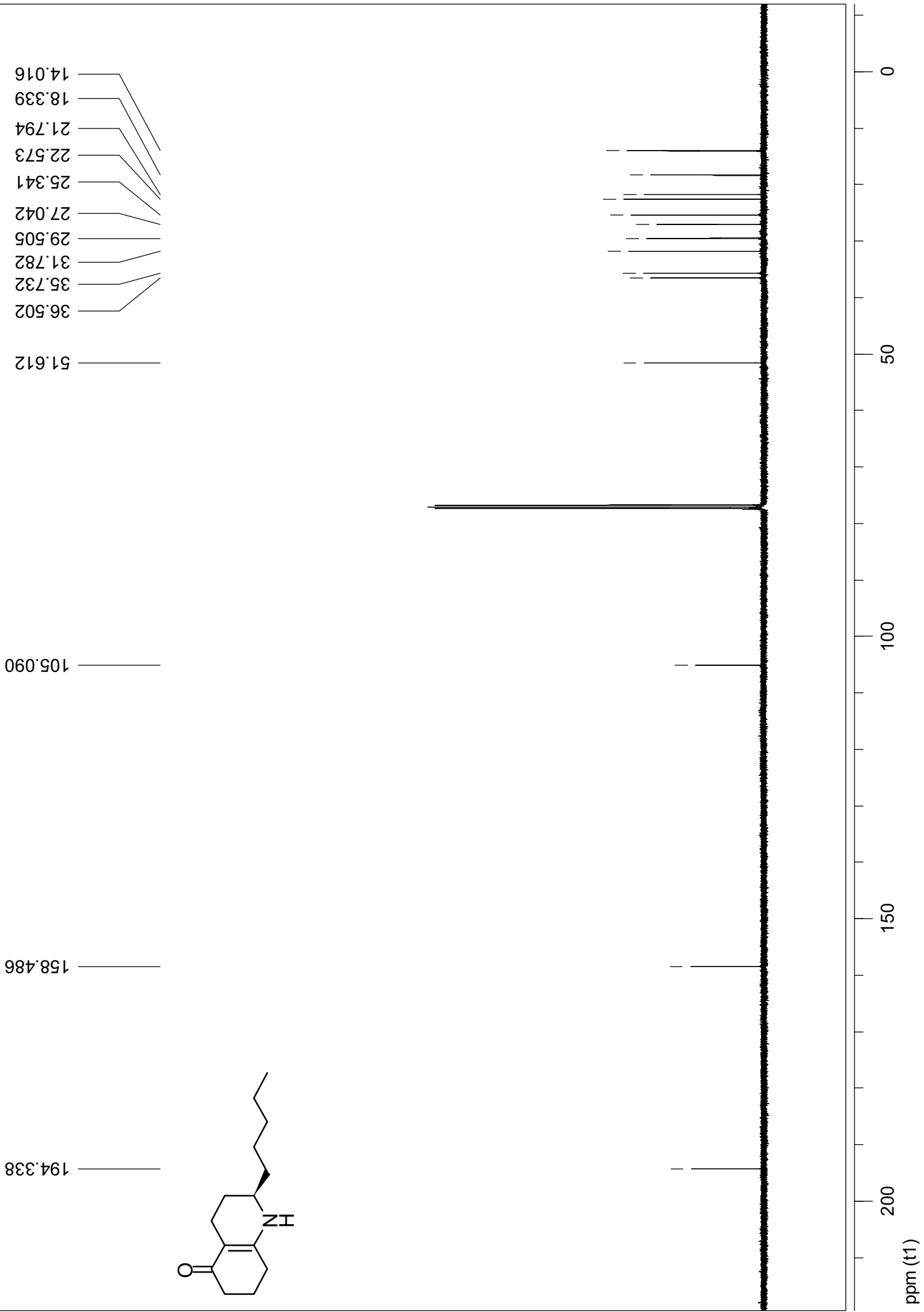
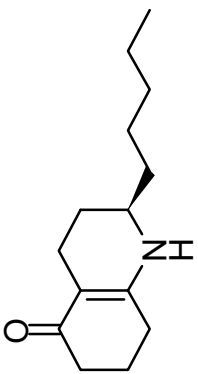
Chromatogram : AA578_18_ODH_9010_flow1_1

Data file: AA578_18_ODH_9010_flow1_1.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 17.01.2007 06:37:20



Index	Start		Time		End		Area %	
	[Min]		[Min]		[Min]		[%]	
1	8,709		9,308		12,087		95,684	
2	12,087		12,900		14,318		4,316	
Total							100,000	





Sample Information

Analyzed : 25.01.2007 20:35:14
 Sample Name : AA578-18

Method

===== Analytical Line 1 =====

[GC-2010]

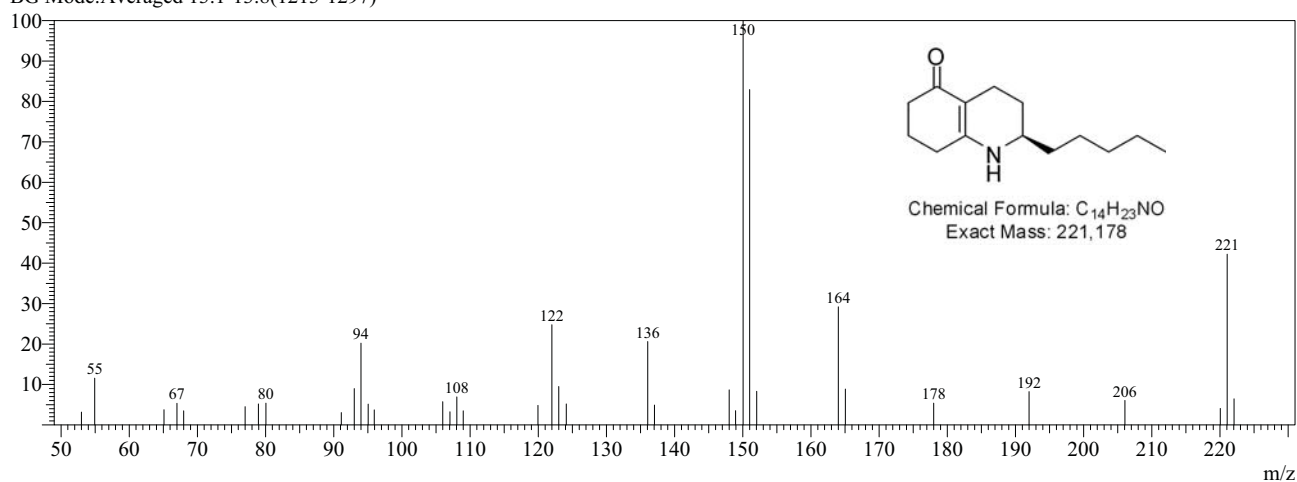
Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:12.2(Scan#:1103)
 MassPeaks:36 BasePeak:150(65878)
 RawMode:Averaged 12.2-12.2(1102-1105)
 BG Mode:Averaged 13.1-13.8(1213-1297)



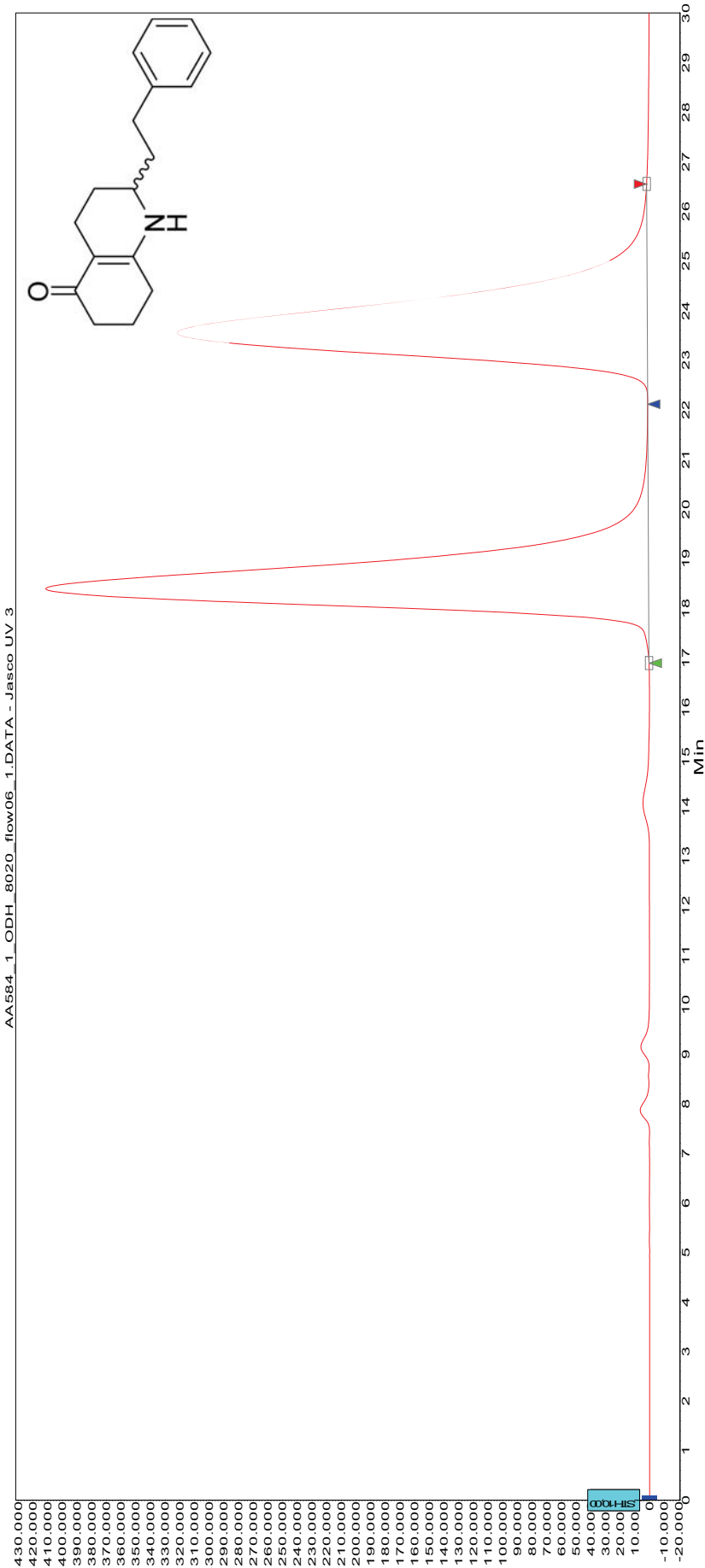
Mass Table

Line#:1 R.Time:12.2(Scan#:1103)
 MassPeaks:36 BasePeak:150(65878)
 RawMode:Averaged 12.2-12.2(1102-1105)
 BG Mode:Averaged 13.1-13.8(1213-1297)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	53.00	3.15	10	93.00	9.00	19	122.00	24.76	28	152.05	8.33
2	54.95	11.56	11	94.00	20.23	20	123.00	9.48	29	164.00	29.20
3	65.10	3.77	12	95.05	5.16	21	124.10	5.17	30	165.05	8.86
4	67.00	5.35	13	95.95	3.71	22	136.05	20.62	31	178.00	5.39
5	68.00	3.48	14	106.00	5.73	23	137.05	4.93	32	192.00	8.21
6	77.00	4.51	15	107.05	3.25	24	148.00	8.66	33	206.05	6.06
7	78.95	5.19	16	108.05	6.91	25	148.95	3.52	34	220.05	4.09
8	80.05	5.38	17	109.00	3.49	26	150.05	100.00	35	221.05	42.22
9	91.10	3.05	18	119.95	4.83	27	151.00	82.93	36	222.05	6.44

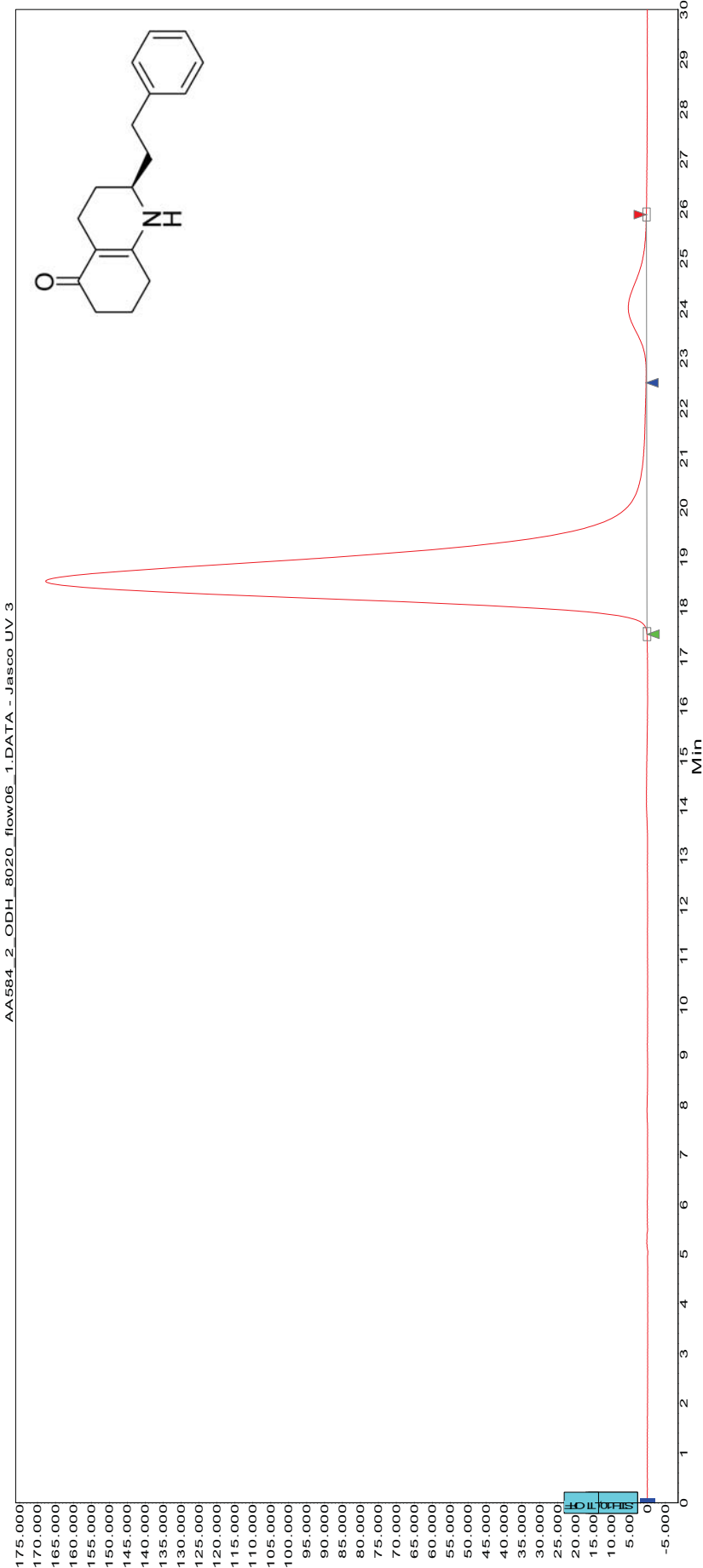
Chromatogram : AA584_1_ODH_8020_flow06_1

Data file: AA584_1_ODH_8020_flow06_1.DATA
Method: HPLC1_ODH_8020_flow06_acq_30
Date: 17.01.2007 18:48:02

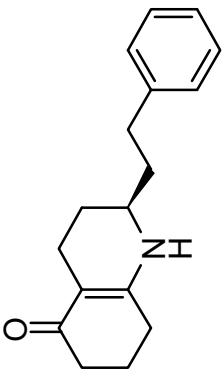


Chromatogram : AA584_2_ODH_8020_flow06_1

Data file: AA584_2_ODH_8020_flow06_1.DATA
Method: HPLC1_ODH_8020_flow06_acq_30
Date: 17.01.2007 19:20:45

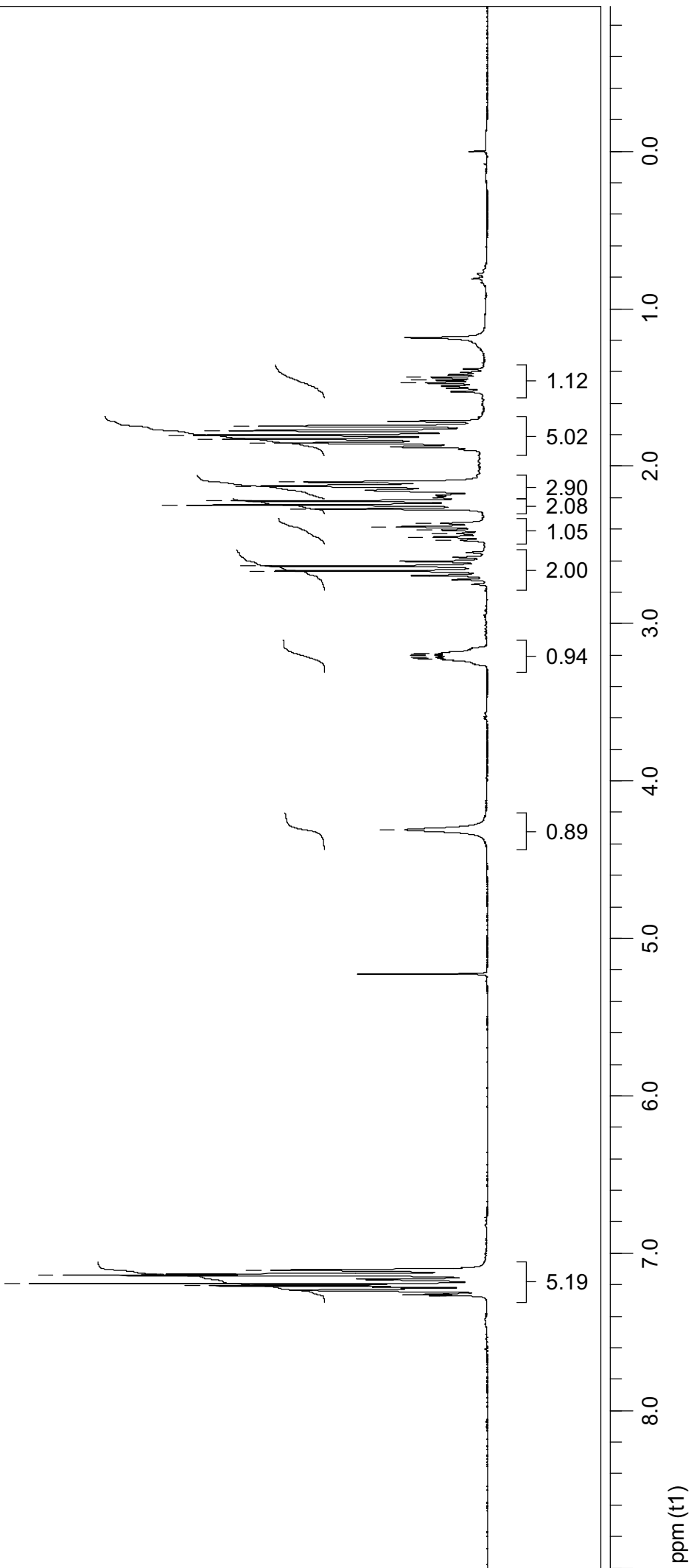


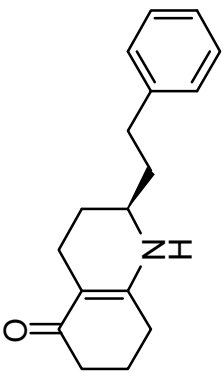
Index	Time		Area %	
	[Min]	[Min]	[Min]	[%]
1	17,448	18,517	22,500	96,216
2	22,500	23,992	25,878	3,784
Total				100,000



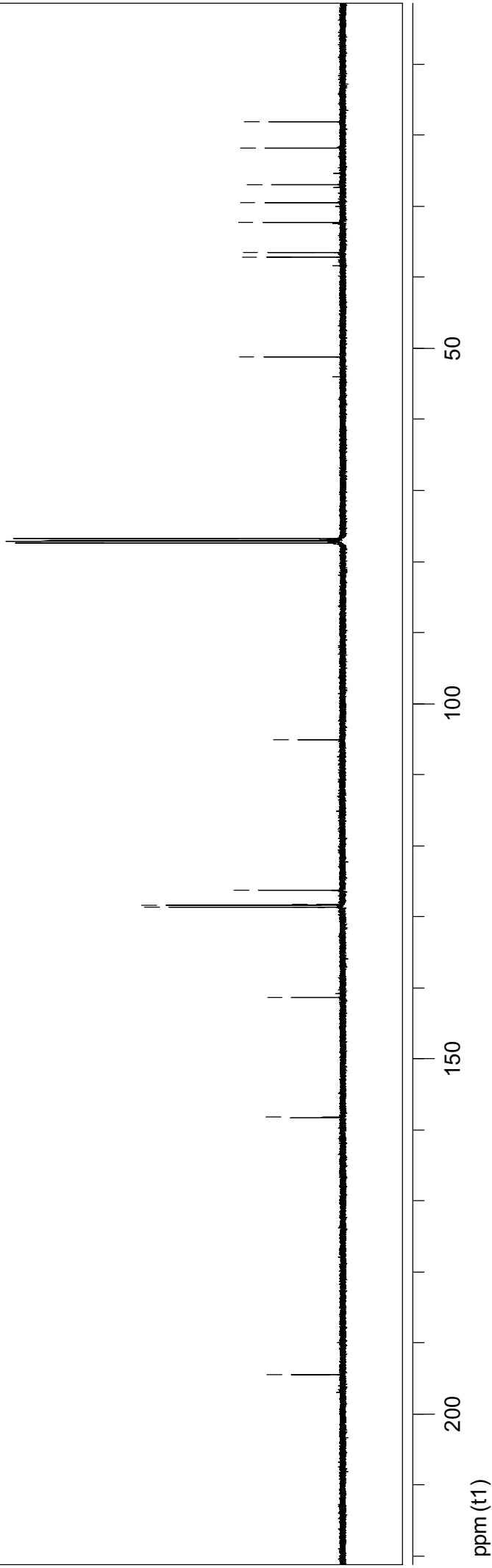
1.436
 1.455
 1.472
 1.746
 1.774
 1.804
 1.827
 1.852
 2.101
 2.127
 2.221
 2.248
 2.272
 2.363
 2.385
 2.407
 2.429
 2.450
 2.472
 2.635
 2.666
 3.188
 3.195
 3.202
 3.214
 3.221
 3.228
 4.310

7.107
 7.132
 7.140
 7.193
 7.207





- | | |
|---------|--|
| 18.055 | |
| 21.751 | |
| 26.921 | |
| 29.436 | |
| 32.244 | |
| 36.493 | |
| 37.141 | |
| 51.206 | |
| 105.084 | |
| 126.236 | |
| 128.306 | |
| 128.326 | |
| 128.645 | |
| 128.666 | |
| 141.309 | |
| 158.246 | |
| 194.432 | |



Sample Information

Analyzed : 26.01.2007 12:18:36
 Sample Name : AA584-2

Method

===== Analytical Line 1 =====

[GC-2010]

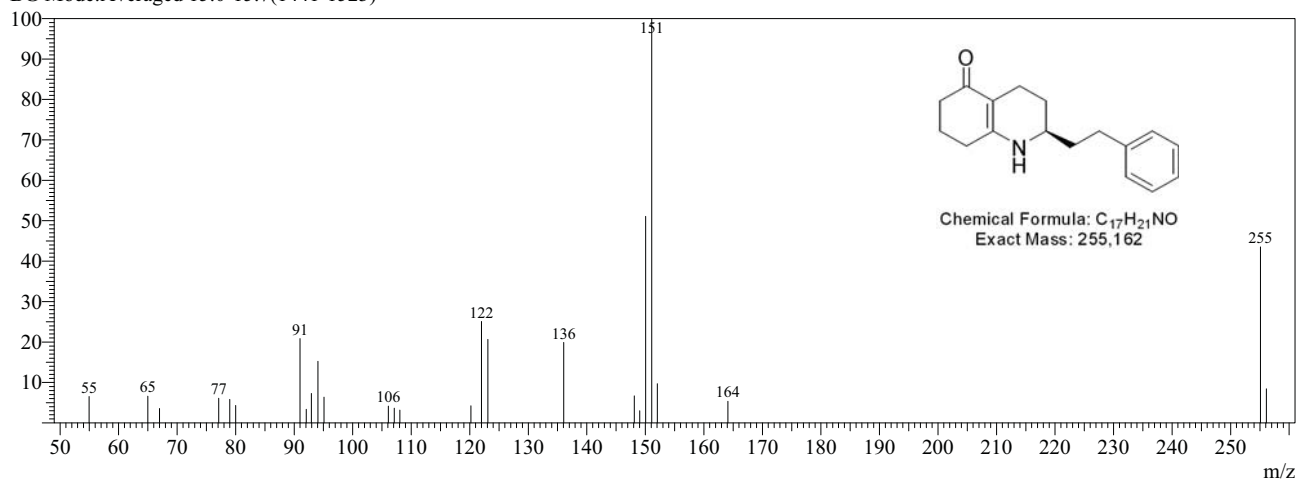
Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:14.3(Scan#:1354)
 MassPeaks:26 BasePeak:151(47214)
 RawMode:Averaged 14.3-14.3(1353-1358)
 BG Mode:Averaged 15.0-15.7(1441-1523)



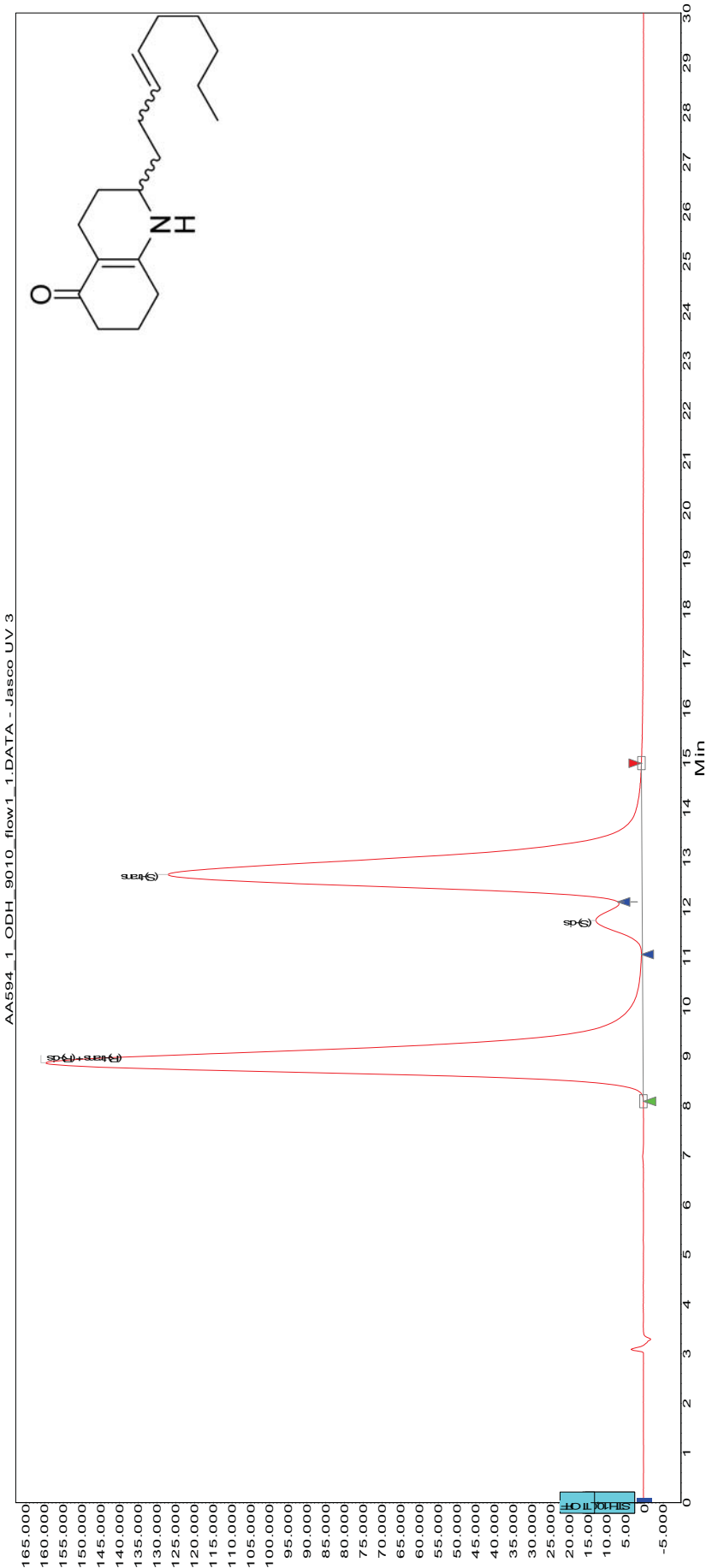
Mass Table

Line#:1 R.Time:14.3(Scan#:1354)
 MassPeaks:26 BasePeak:151(47214)
 RawMode:Averaged 14.3-14.3(1353-1358)
 BG Mode:Averaged 15.0-15.7(1441-1523)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	54.95	6.53	8	92.05	3.35	15	120.20	4.25	22	151.10	100.00
2	65.00	6.59	9	92.95	7.28	16	122.00	25.08	23	152.05	9.70
3	67.00	3.58	10	94.05	15.25	17	123.10	20.66	24	164.10	5.36
4	77.10	6.09	11	95.10	6.39	18	136.05	19.89	25	255.10	43.54
5	79.00	5.80	12	106.10	4.17	19	148.10	6.70	26	256.10	8.43
6	80.00	4.30	13	107.10	3.64	20	149.05	3.03			
7	91.00	20.84	14	108.05	3.18	21	150.05	51.08			

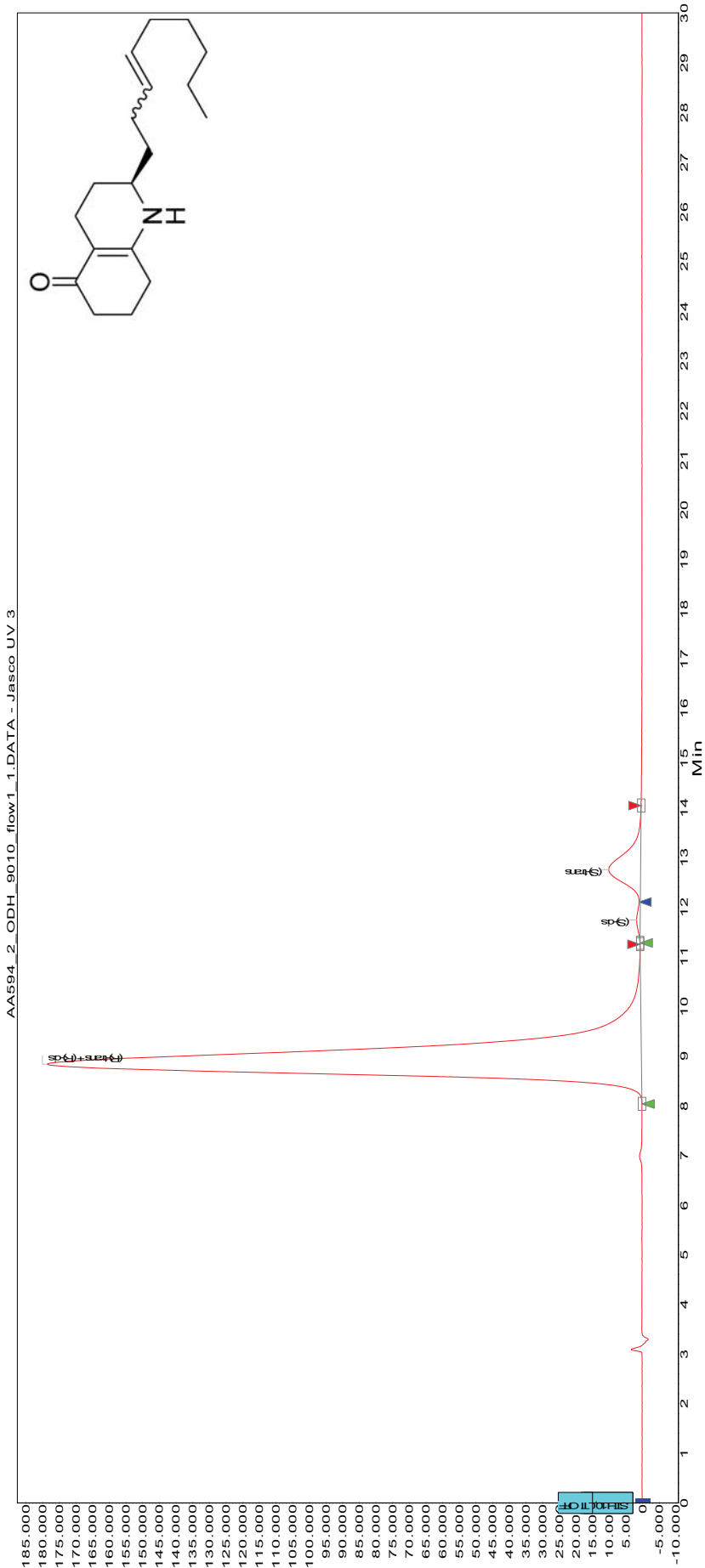
Chromatogram : AA594_1_ODH_9010_flow1_1

Data file: AA594_1_ODH_9010_flow1_1.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 19.01.2007 02:52:01

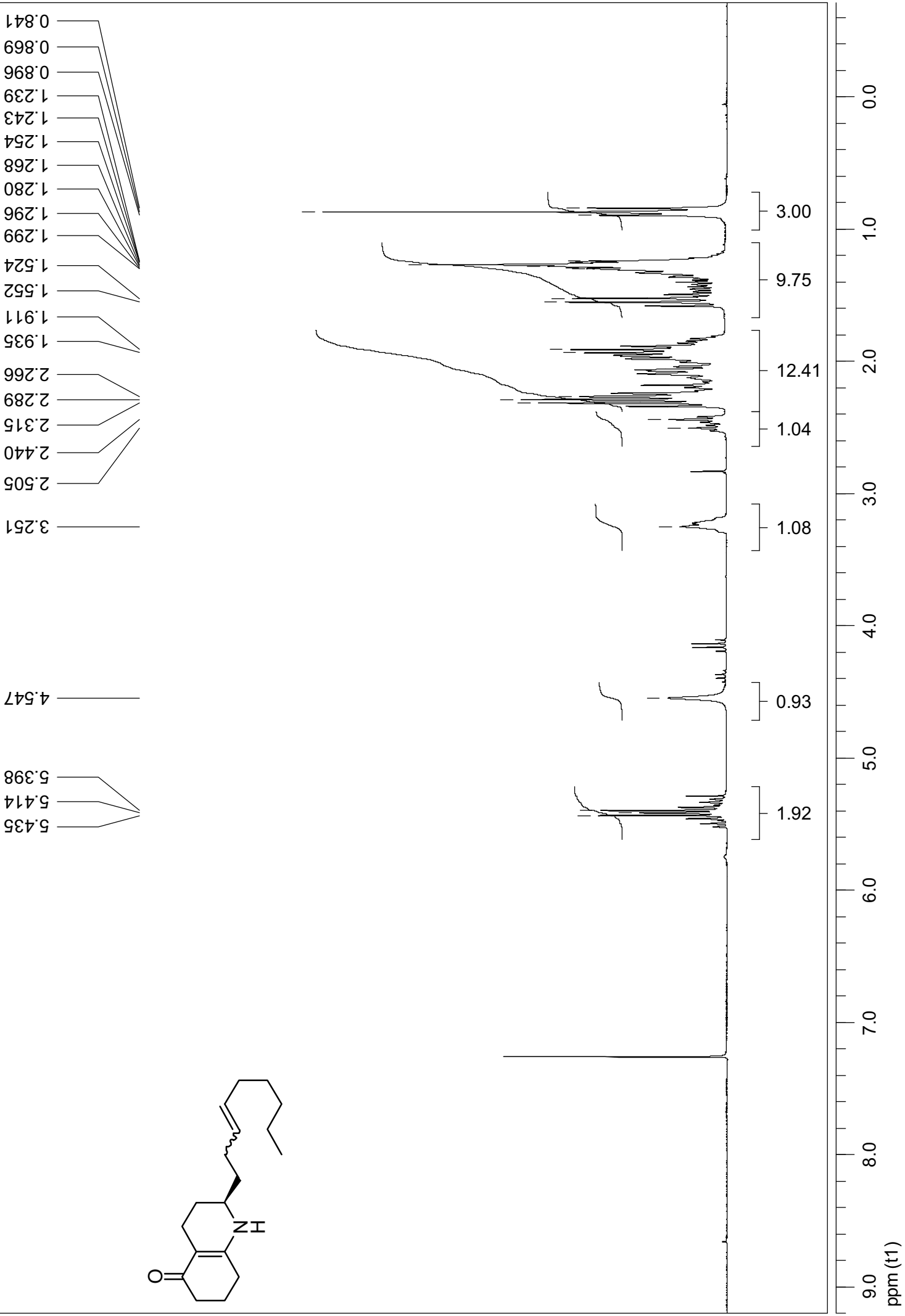
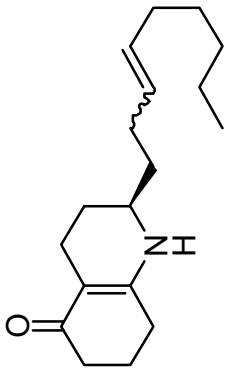


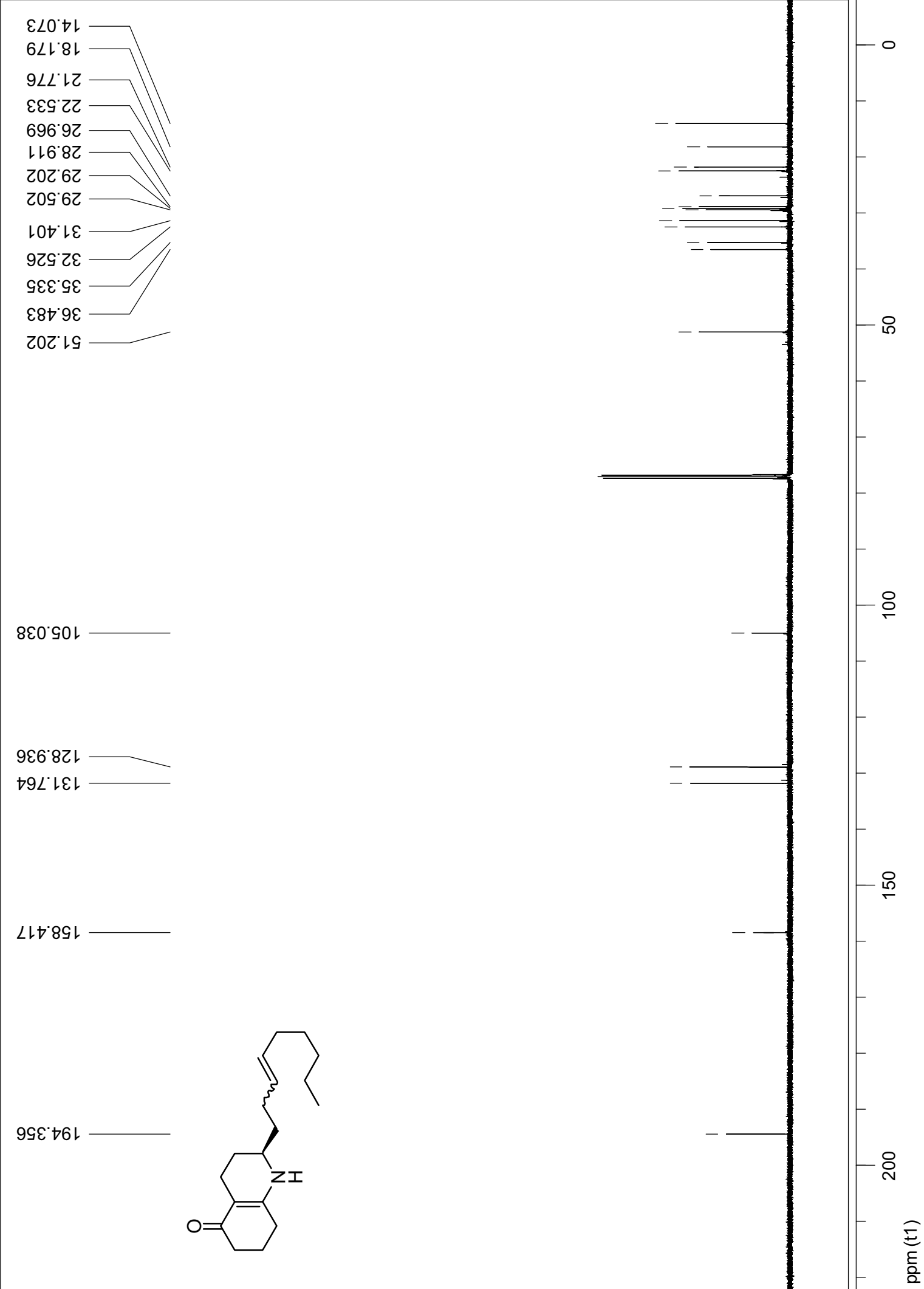
Chromatogram : AA594_2_ODH_9010_flow1_1

Data file: AA594_2_ODH_9010_flow1_1.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 19.01.2007 03:24:41



Index	Name	Start [Min]	Time [Min]	End [Min]	Area %
1	(R)-trans + (R)-cis	8,034	8,833	11,247	93,558
2	(S)-cis	11,274	11,742	12,100	0,495
3	(S)-trans	12,100	12,750	14,041	5,947
Total					100,000





Sample Information

Analyzed : 26.01.2007 03:28:37
 Sample Name : AA594-2

Method

===== Analytical Line 1 =====

[GC-2010]

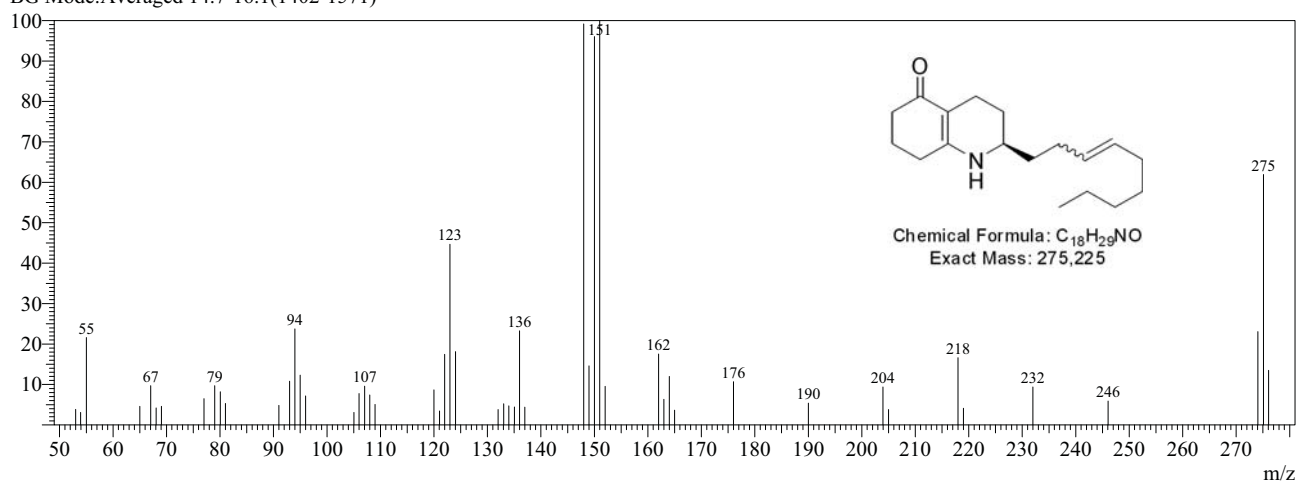
Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:14.1(Scan#:1331)
 MassPeaks:52 BasePeak:151(95491)
 RawMode:Averaged 14.1-14.1(1330-1336)
 BG Mode:Averaged 14.7-16.1(1402-1571)



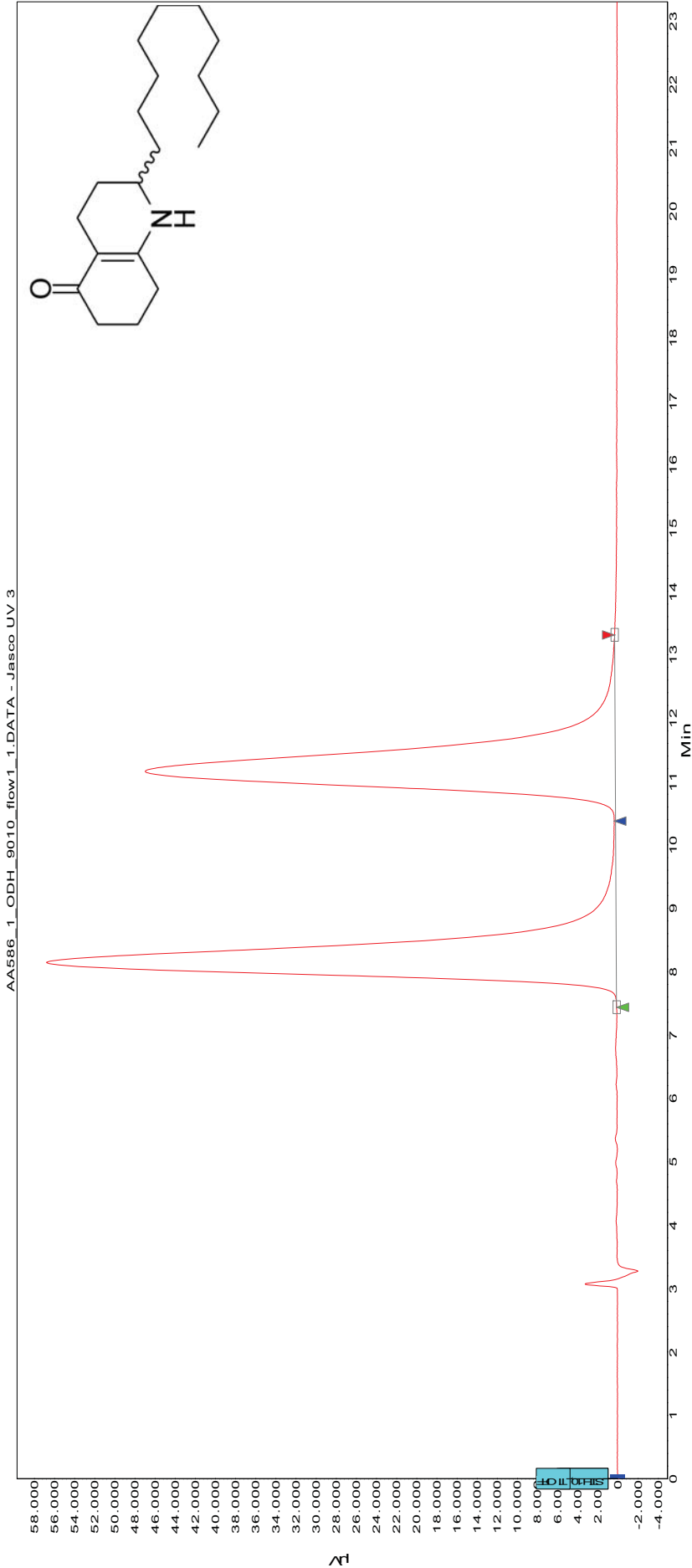
Mass Table

Line#:1 R.Time:14.1(Scan#:1331)
 MassPeaks:52 BasePeak:151(95491)
 RawMode:Averaged 14.1-14.1(1330-1336)
 BG Mode:Averaged 14.7-16.1(1402-1571)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	53.00	3.86	14	94.00	23.73	27	132.00	3.80	40	164.00	12.00
2	53.95	3.07	15	95.00	12.30	28	133.05	5.24	41	165.00	3.64
3	55.00	21.61	16	96.00	7.16	29	134.00	4.72	42	176.00	10.69
4	65.00	4.58	17	105.05	3.08	30	135.05	4.44	43	190.00	5.39
5	67.05	9.68	18	106.00	7.76	31	136.00	23.27	44	203.95	9.37
6	68.05	4.18	19	107.05	9.57	32	137.00	4.41	45	205.00	3.79
7	69.05	4.60	20	108.00	7.41	33	148.00	99.18	46	218.00	16.62
8	77.00	6.49	21	109.00	5.06	34	149.00	14.59	47	219.00	4.11
9	79.00	9.71	22	120.00	8.65	35	150.00	96.02	48	232.00	9.37
10	80.05	8.17	23	121.05	3.45	36	151.00	100.00	49	246.05	5.89
11	81.00	5.30	24	122.00	17.45	37	152.00	9.53	50	274.05	23.08
12	91.00	4.84	25	123.00	44.67	38	162.00	17.54	51	275.10	61.91
13	93.00	10.80	26	124.05	18.13	39	163.00	6.33	52	276.05	13.48

Chromatogram : AA586_1_ODH_9010_flow1_1

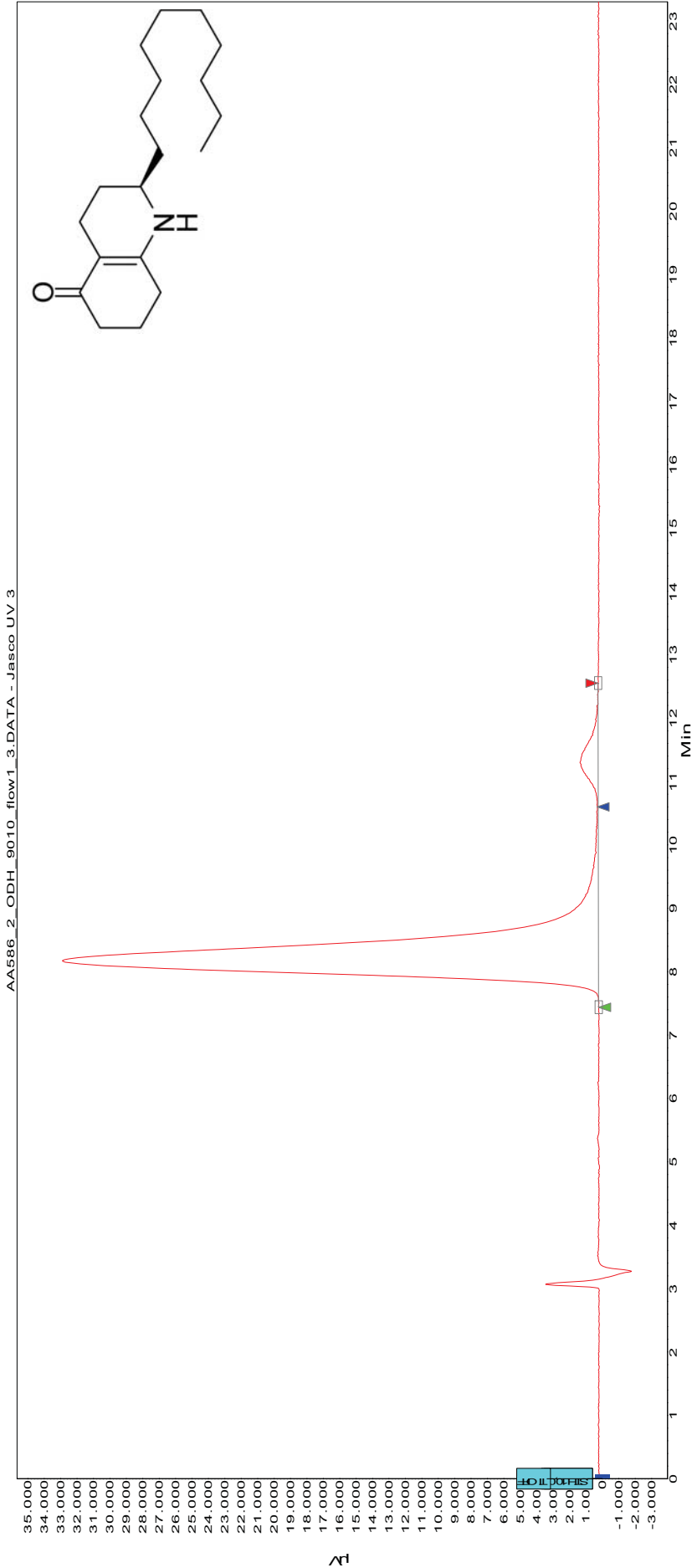
Data file: AA586_1_ODH_9010_flow1_1.DATA
Method: HPLC1_ODH_9010_flow1_acq_60
Date: 17.01.2007 22:56:11



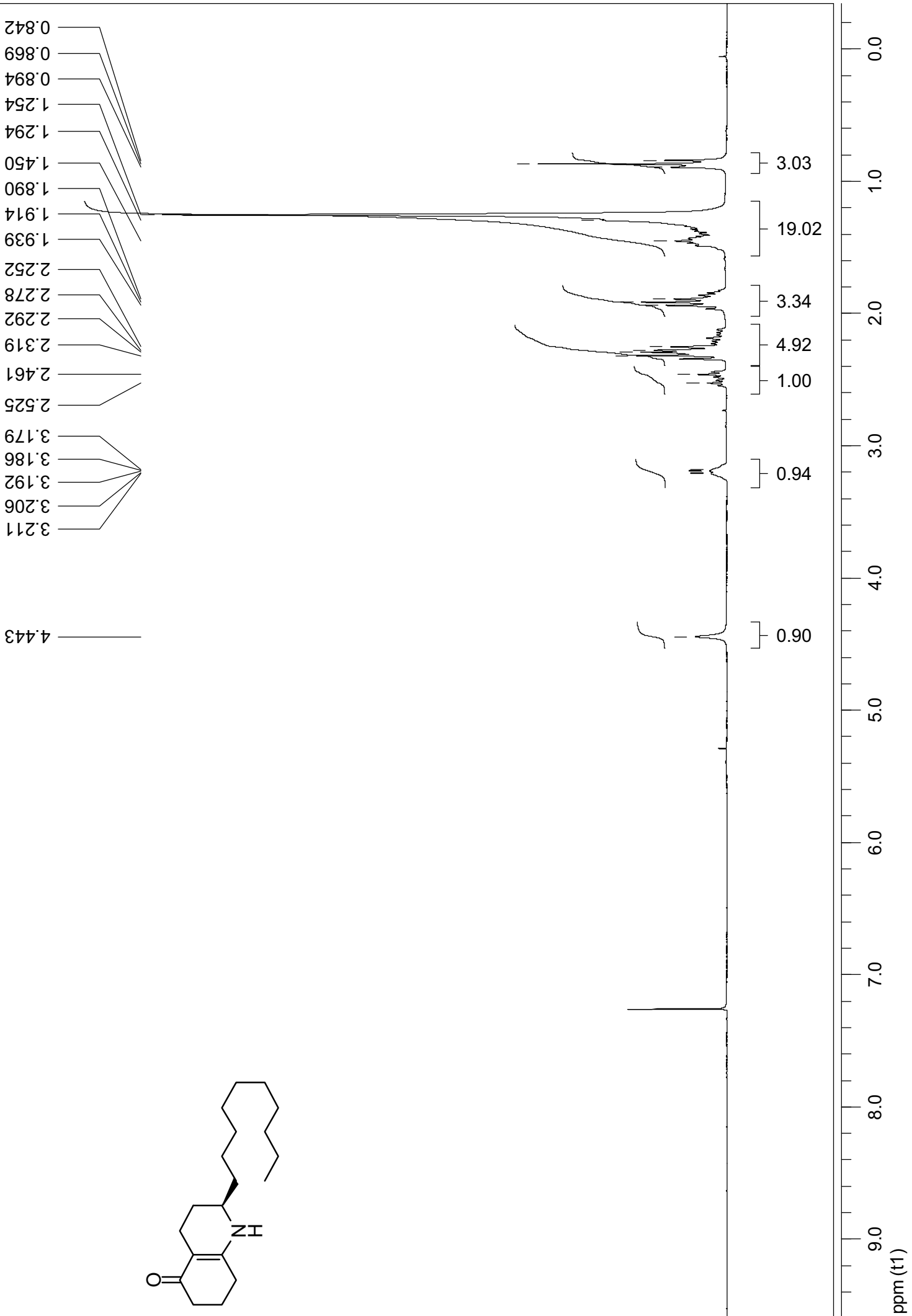
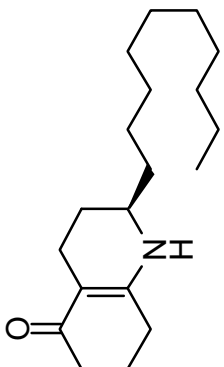
Index	Start		Time		End		Area %	
	[Min]		[Min]		[Min]		[%]	
1	7,438		8,142		10,374		50,061	
2	10,374		11,158		13,311		49,939	
Total							100,000	

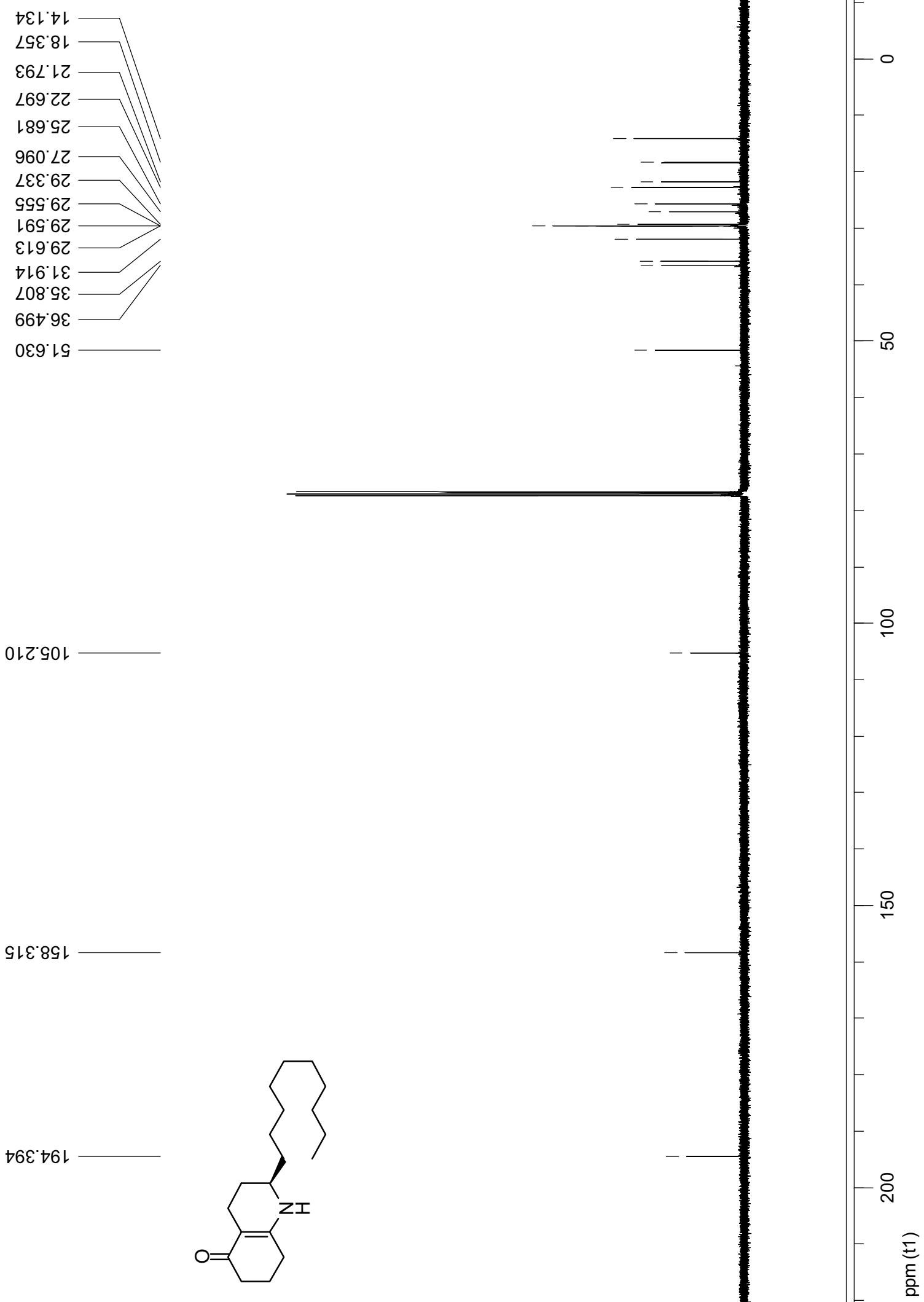
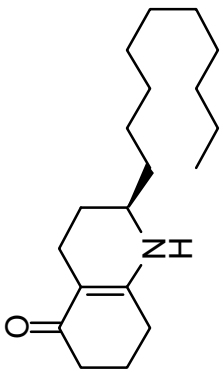
Chromatogram : AA586_2_ODH_9010_flow1_3

Data file: AA586_2_ODH_9010_flow1_3.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 18.01.2007 00:03:03



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7.438	8.175	10.599	96.051
2	10.599	11.308	12.552	3.949
Total				100.000





Sample Information

Analyzed : 26.01.2007 01:32:48
 Sample Name : AA586-2

Method

===== Analytical Line 1 =====

[GC-2010]

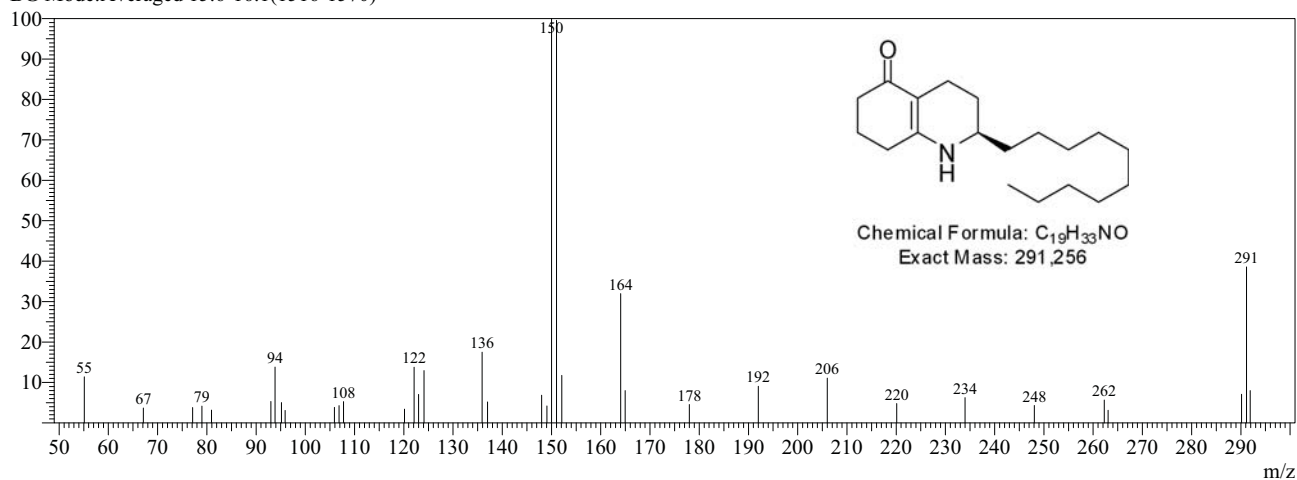
Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:14.8(Scan#:1416)
 MassPeaks:36 BasePeak:150(10270)
 RawMode:Averaged 14.8-14.8(1415-1419)
 BG Mode:Averaged 15.6-16.1(1516-1570)



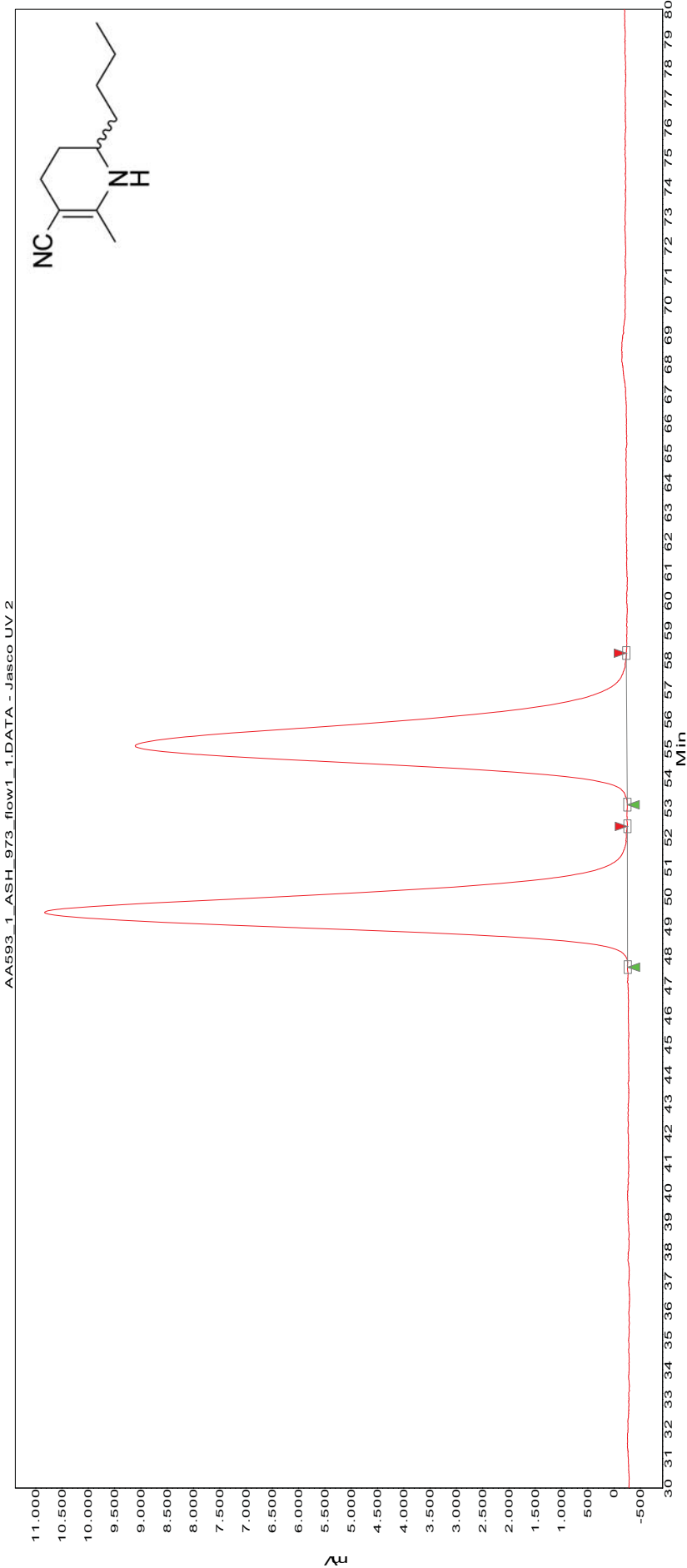
Mass Table

Line#:1 R.Time:14.8(Scan#:1416)
 MassPeaks:36 BasePeak:150(10270)
 RawMode:Averaged 14.8-14.8(1415-1419)
 BG Mode:Averaged 15.6-16.1(1516-1570)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	55.10	11.39	10	105.90	3.83	19	148.00	6.85	28	206.00	11.08
2	67.10	3.68	11	106.85	4.26	20	149.05	4.20	29	220.10	4.80
3	77.10	3.79	12	107.75	5.25	21	150.00	100.00	30	234.00	6.25
4	79.00	4.17	13	120.15	3.41	22	151.00	99.55	31	248.05	4.32
5	80.95	3.17	14	122.10	13.76	23	152.10	11.75	32	262.25	5.68
6	93.00	5.29	15	123.00	7.04	24	164.05	31.97	33	263.05	3.14
7	93.85	13.82	16	124.10	12.92	25	164.95	8.00	34	290.15	7.10
8	95.15	5.02	17	135.90	17.49	26	178.00	4.54	35	291.15	38.59
9	95.90	3.09	18	137.00	5.20	27	192.00	9.06	36	291.90	7.98

Chromatogram : AA593_1_ASH_973_flow1_1

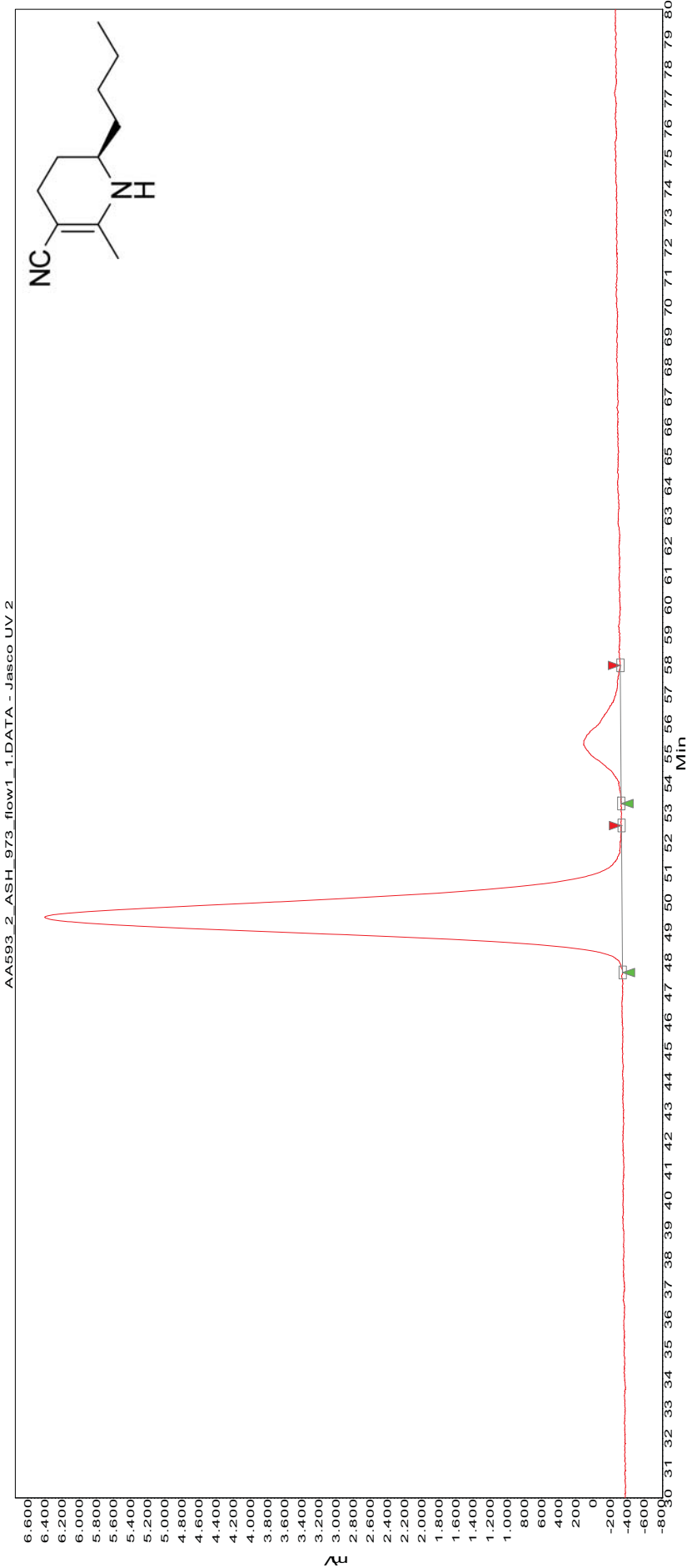
Data file: AA593_1_ASH_973_flow1_1.DATA
Method: HPLC2_ASH_973flow1_acq80
Date: 19.01.2007 05:29:06

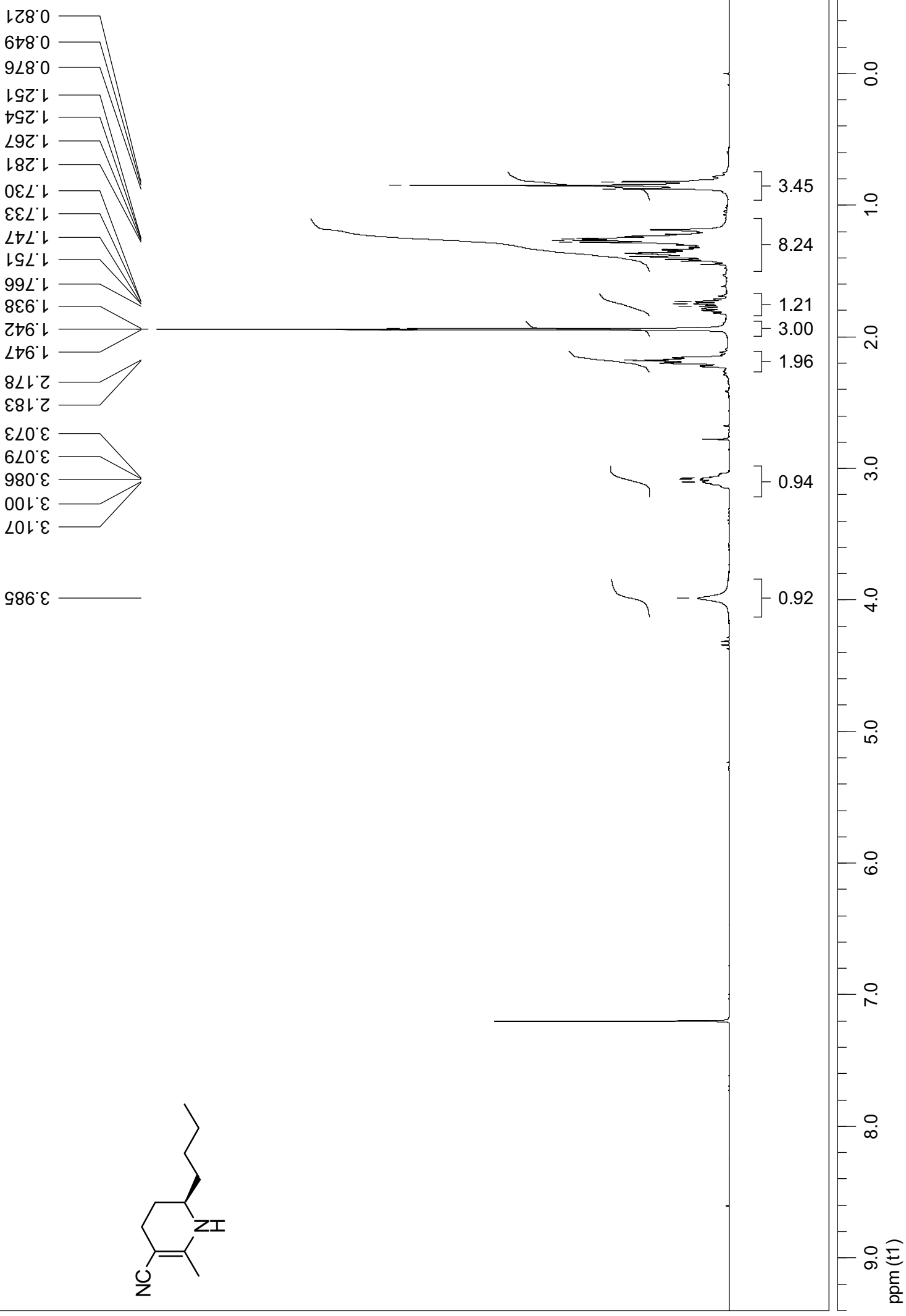
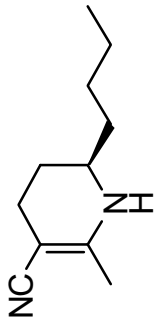


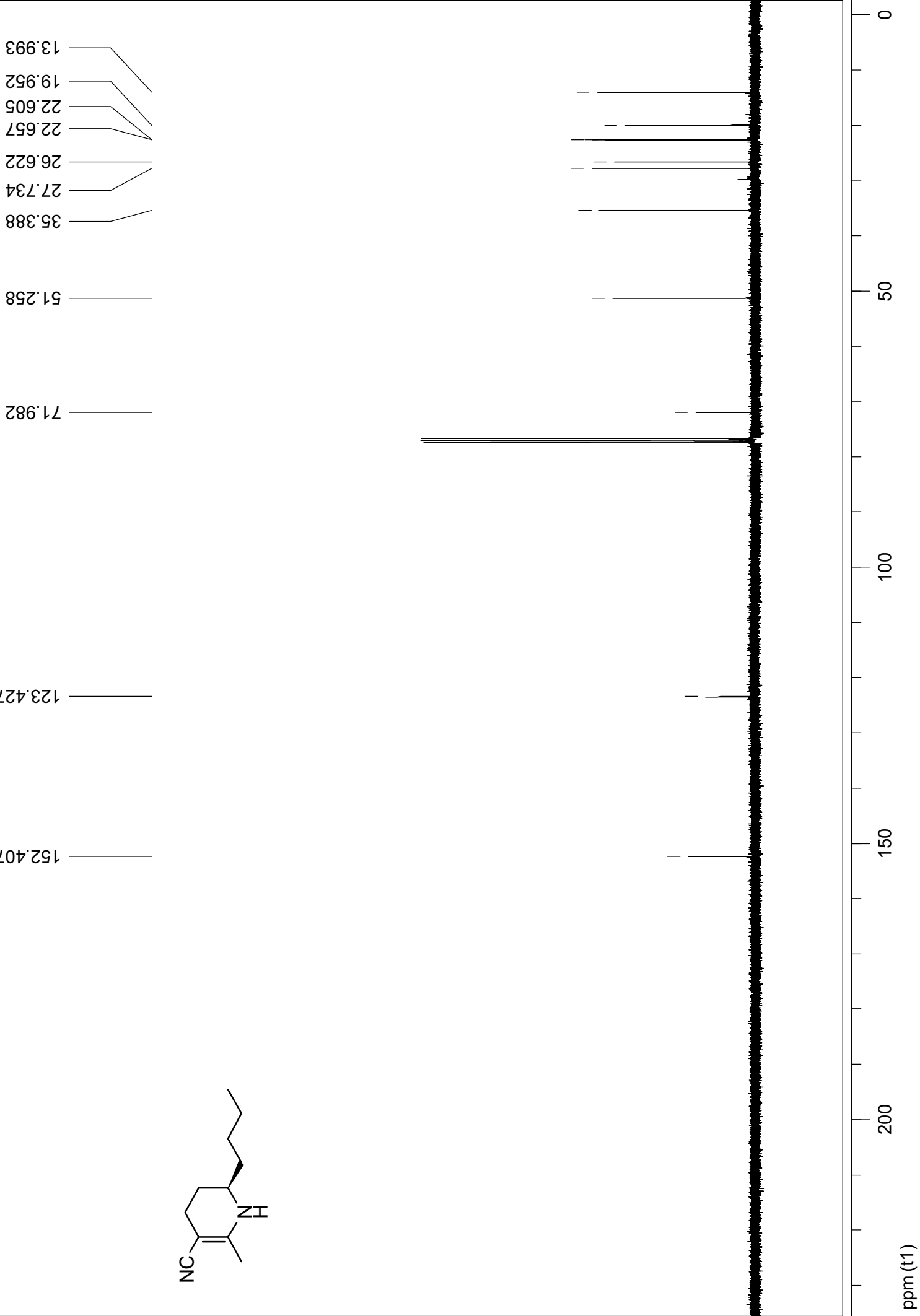
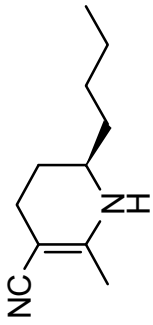
Index	Start		Time		End		Area %	
	[Min]		[Min]		[Min]		[%]	
1	47,611		49,467		52,372		49,843	
2	53,105		55,092		58,233		50,157	
Total							100,000	

Chromatogram : AA593_2_ASH_973_flow1_1

Data file: AA593_2_ASH_973_flow1_1.DATA
Method: HPLC2_ASH_973flow1_acq80
Date: 19.01.2007 06:51:44







Sample Information

Analyzed : 25.01.2007 21:16:16
 Sample Name : AA593-2

Method

===== Analytical Line 1 =====

[GC-2010]

Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

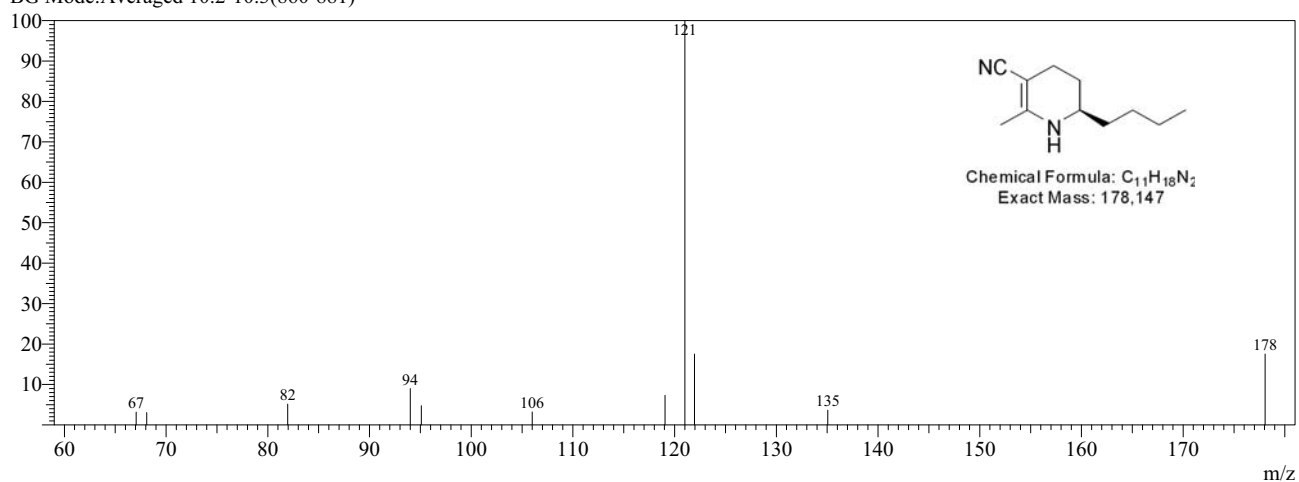
Spectrum

Line#:1 R.Time:9.9(Scan#:827)

MassPeaks:11 BasePeak:121(76527)

RawMode:Averaged 9.9-9.9(826-828)

BG Mode:Averaged 10.2-10.3(860-881)



Mass Table

Line#:1 R.Time:9.9(Scan#:827)

MassPeaks:11 BasePeak:121(76527)

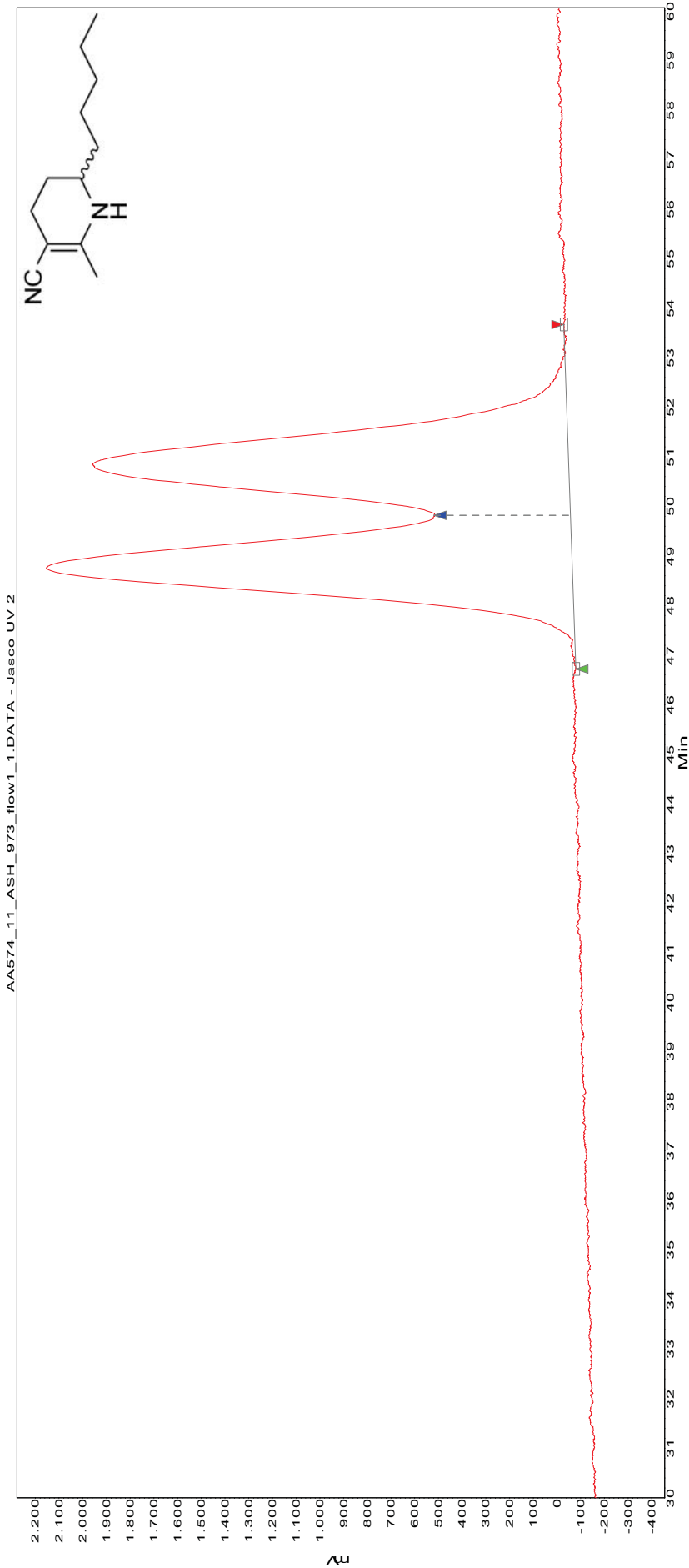
RawMode:Averaged 9.9-9.9(826-828)

BG Mode:Averaged 10.2-10.3(860-881)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	67.05	3.17	4	94.00	8.98	7	119.05	7.34	10	135.05	3.64
2	68.10	3.05	5	95.10	4.74	8	121.00	100.00	11	178.05	17.53
3	81.95	5.15	6	106.00	3.21	9	121.95	17.55			

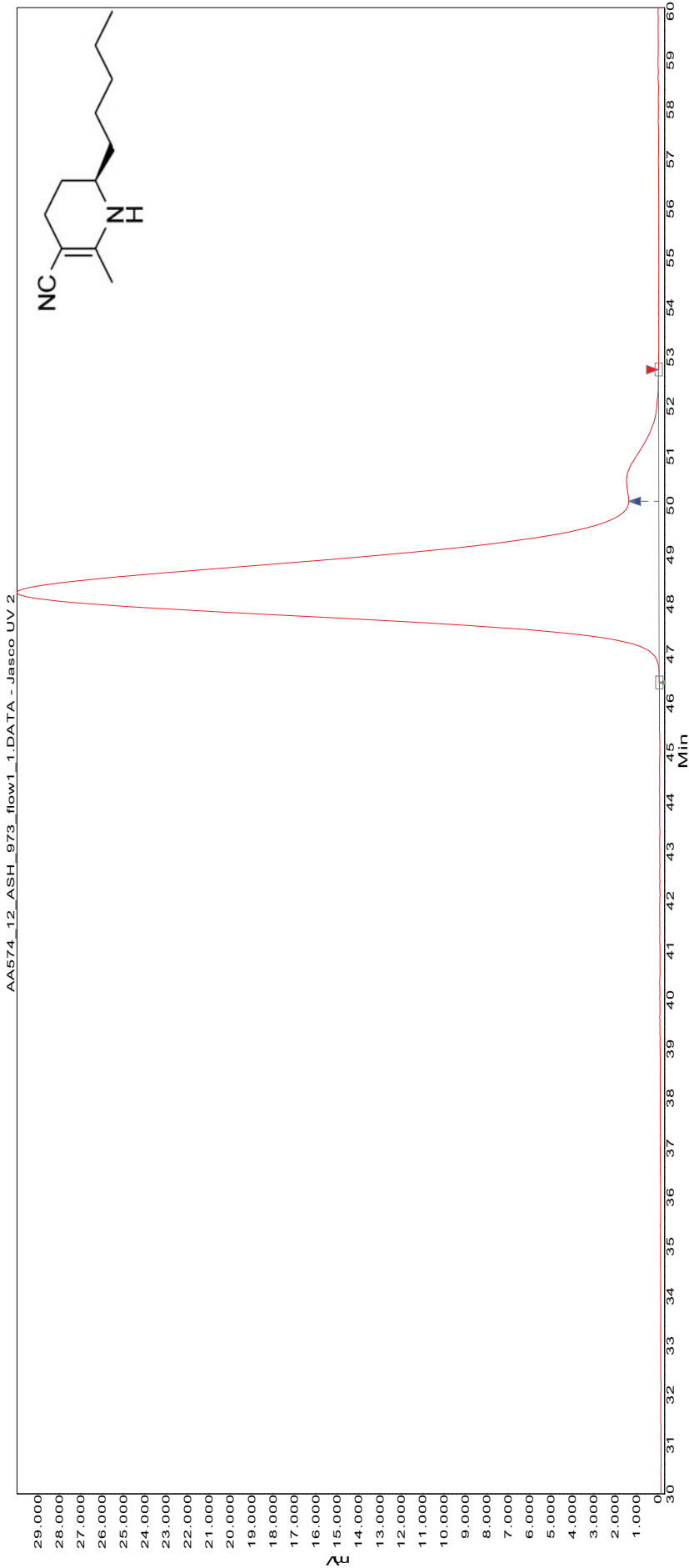
Chromatogram : AA574_11_ASH_973_flow1_1

Data file: AA574_11_ASH_973_flow1_1.DATA
Method: HPLC2_ASH_973flow1_acq80
Date: 18.01.2007 12:19:47

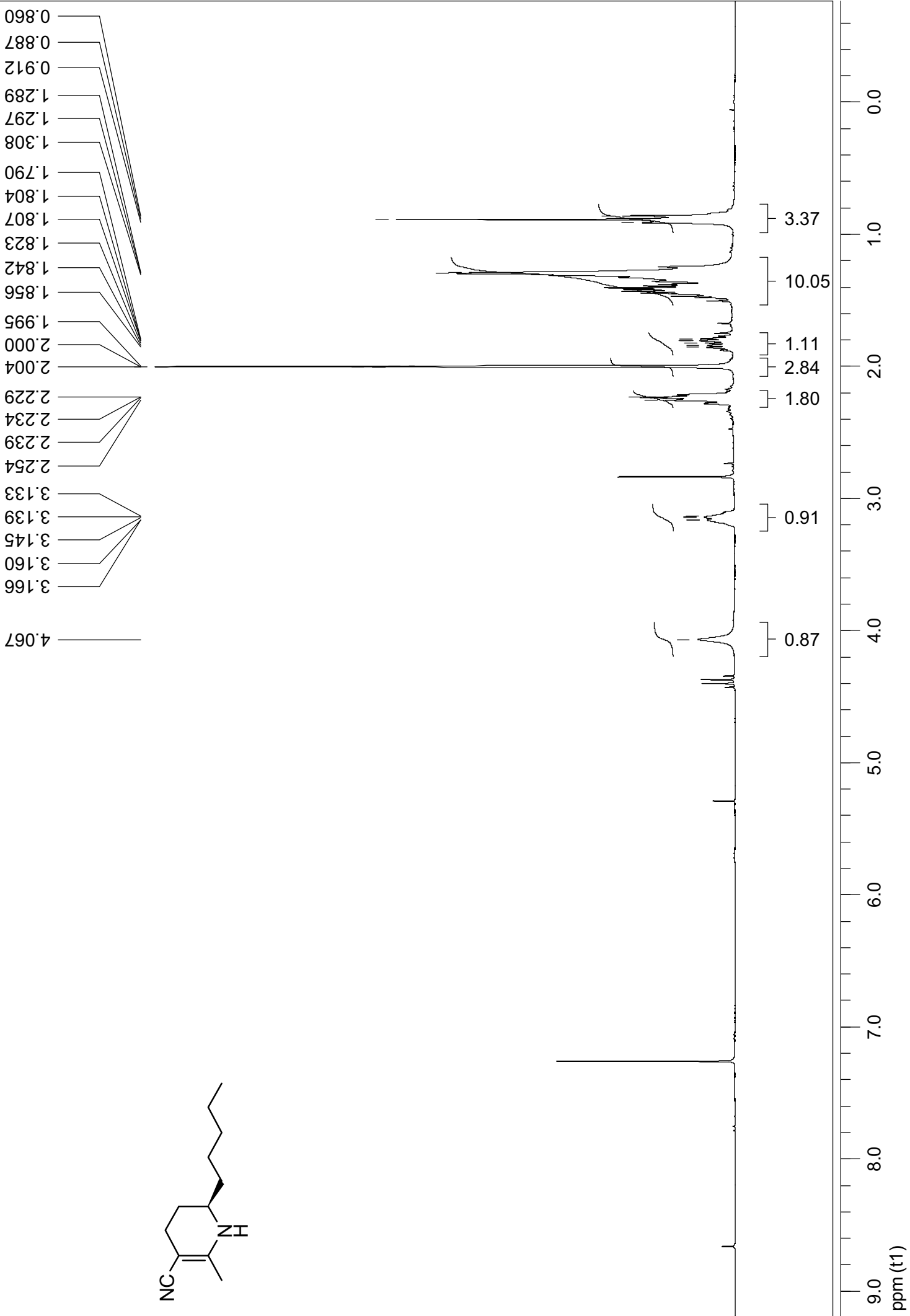
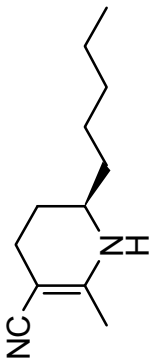


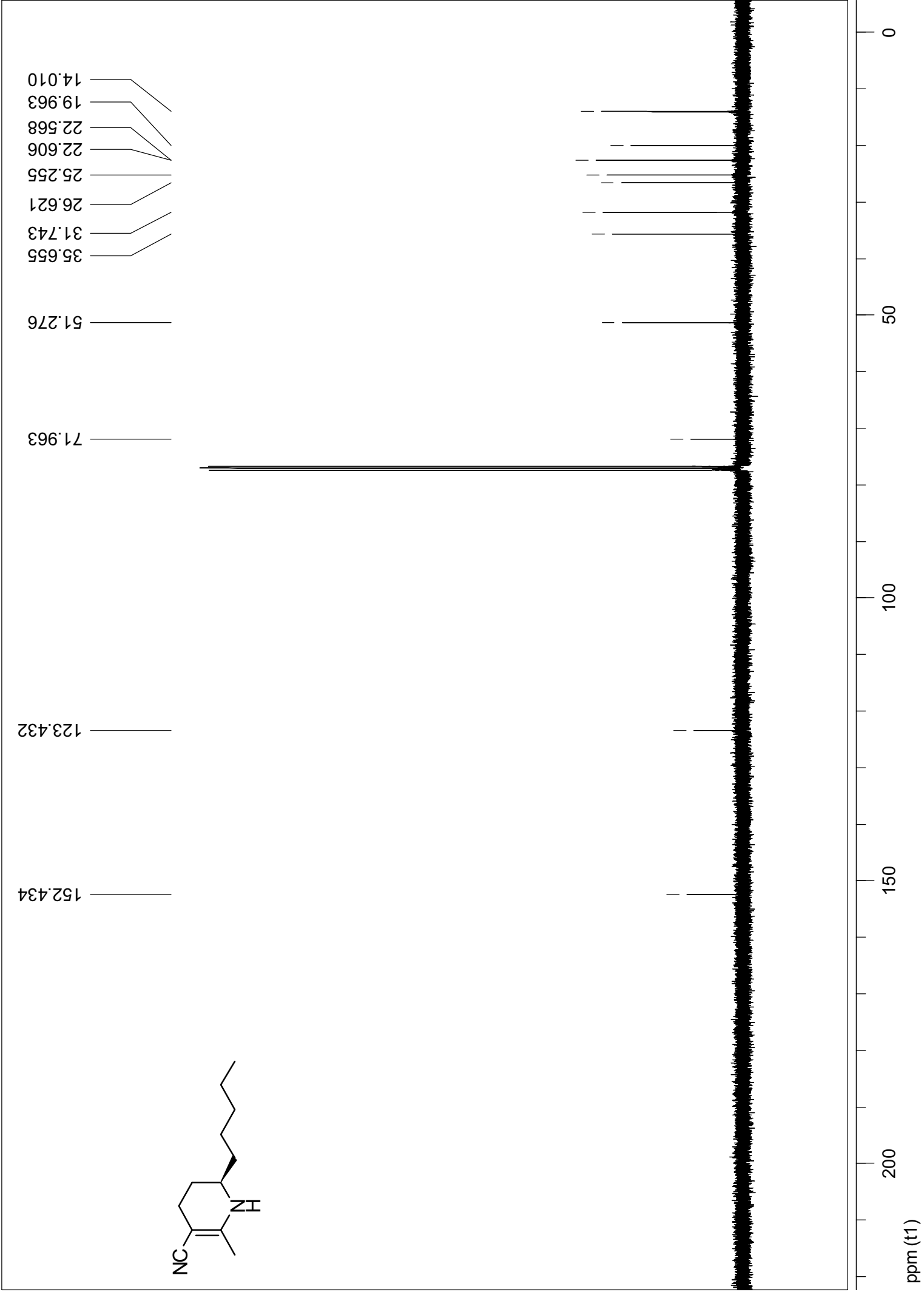
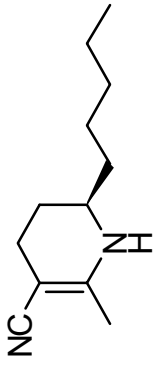
Chromatogram : AA574_12_ASH_973_flow1_1

Data file: AA574_12_ASH_973_flow1_1.DATA
Method: HPLC2_ASH_973flow1_acq80
Date: 18.01.2007 13:22:15



Index	Start		Time		End		Area %	
	[Min]		[Min]		[Min]		[%]	
1	46,409		48,233		50,076		95,688	
2	50,076		50,500		52,737		4,312	
Total							100,000	





Sample Information

Analyzed : 25.01.2007 19:56:26
 Sample Name : AA574-12

Method

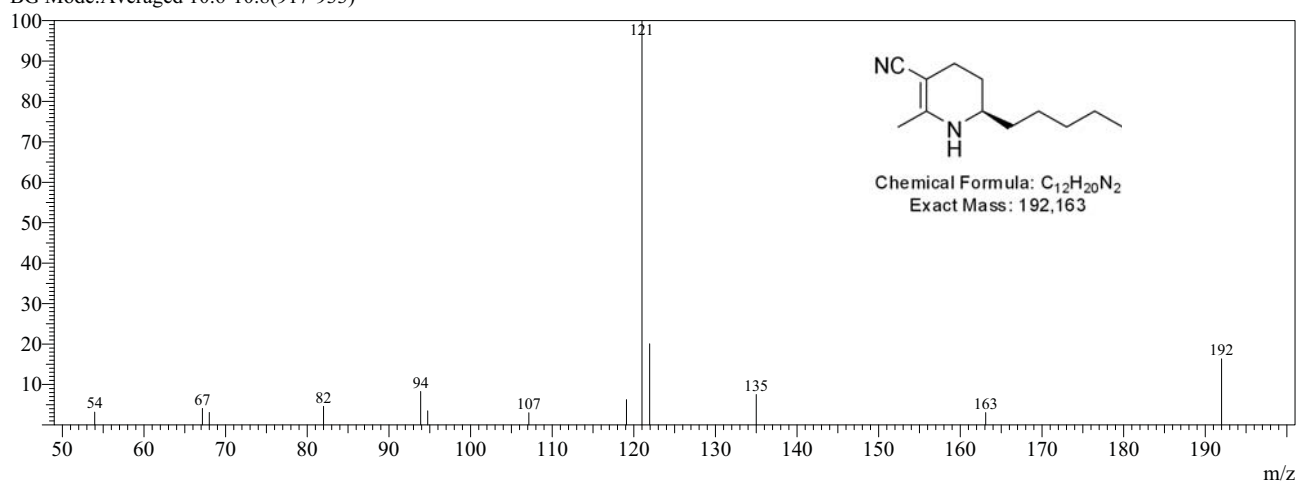
===== Analytical Line 1 =====

[GC-2010]
 Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program		
Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:10.5(Scan#:897)
 MassPeaks:13 BasePeak:121(12409)
 RawMode:Averaged 10.5-10.5(896-898)
 BG Mode:Averaged 10.6-10.8(917-933)



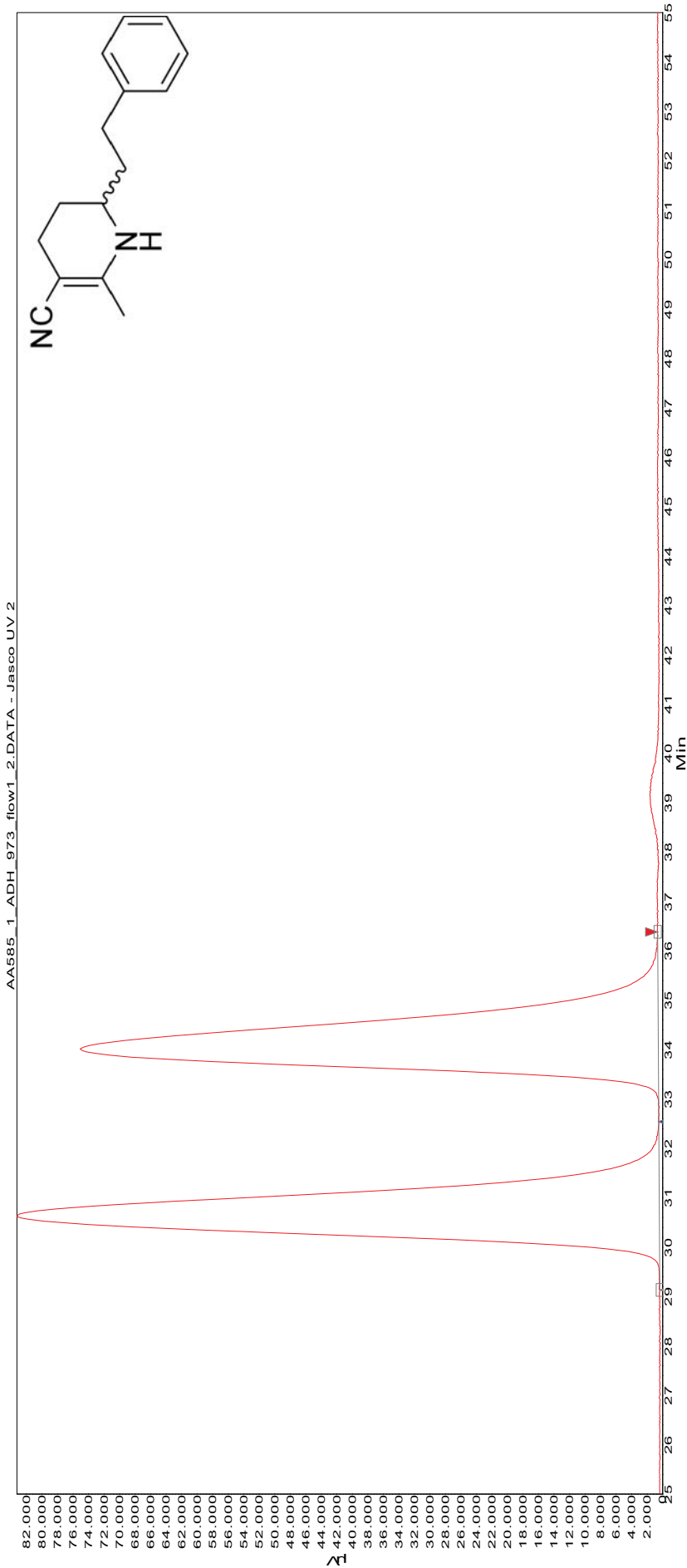
Mass Table

Line#:1 R.Time:10.5(Scan#:897)
 MassPeaks:13 BasePeak:121(12409)
 RawMode:Averaged 10.5-10.5(896-898)
 BG Mode:Averaged 10.6-10.8(917-933)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	53.95	3.18	5	93.90	8.25	9	121.00	100.00	13	192.00	16.34
2	67.15	4.06	6	94.75	3.50	10	121.95	20.07			
3	68.00	3.09	7	107.15	3.01	11	135.00	7.51			
4	82.00	4.59	8	119.10	6.24	12	163.10	3.06			

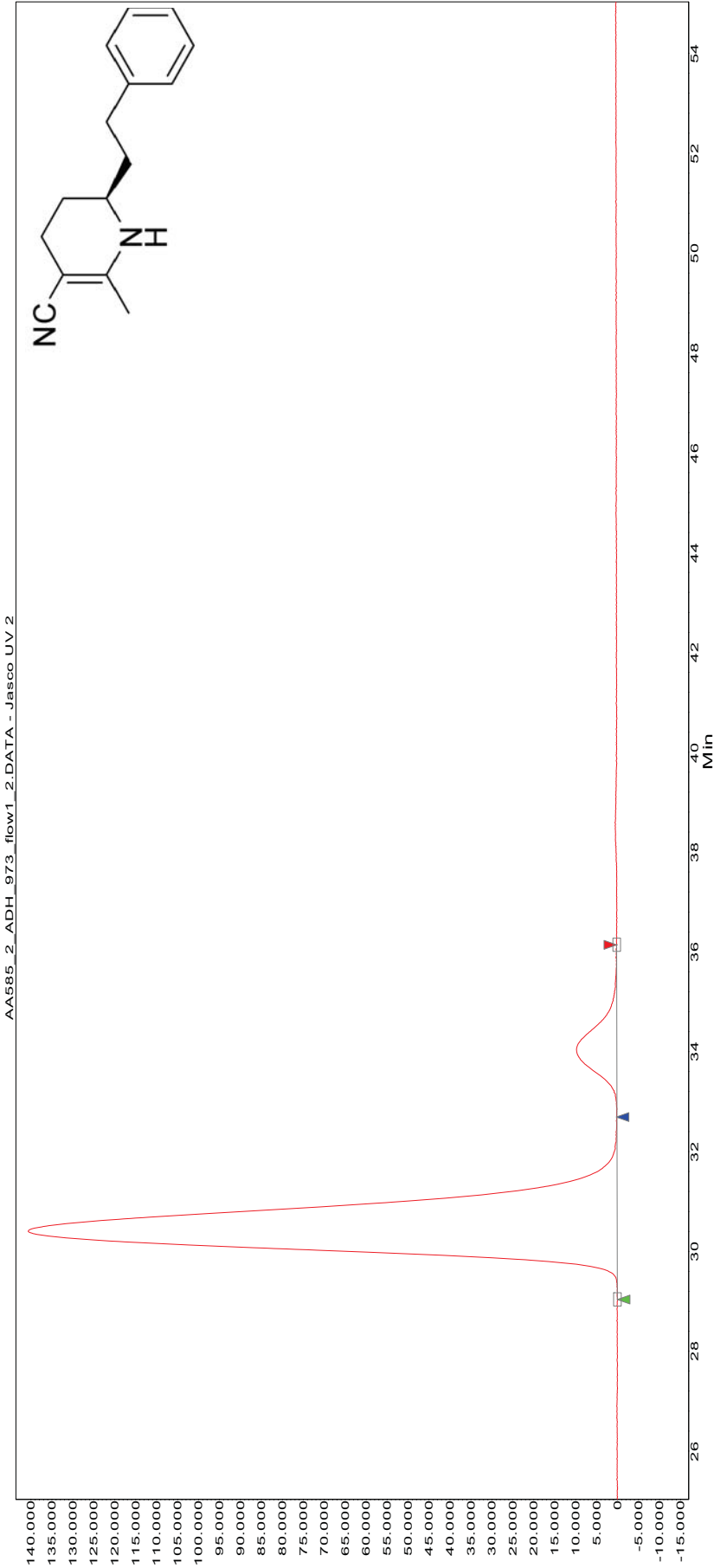
Chromatogram : AA585_1_ADH_973_flow1_2

Data file: AA585_1_ADH_973_flow1_2.DATA
Method: HPLC1_ADH_973_flow1_acq_60
Date: 16.01.2007 15:30:14

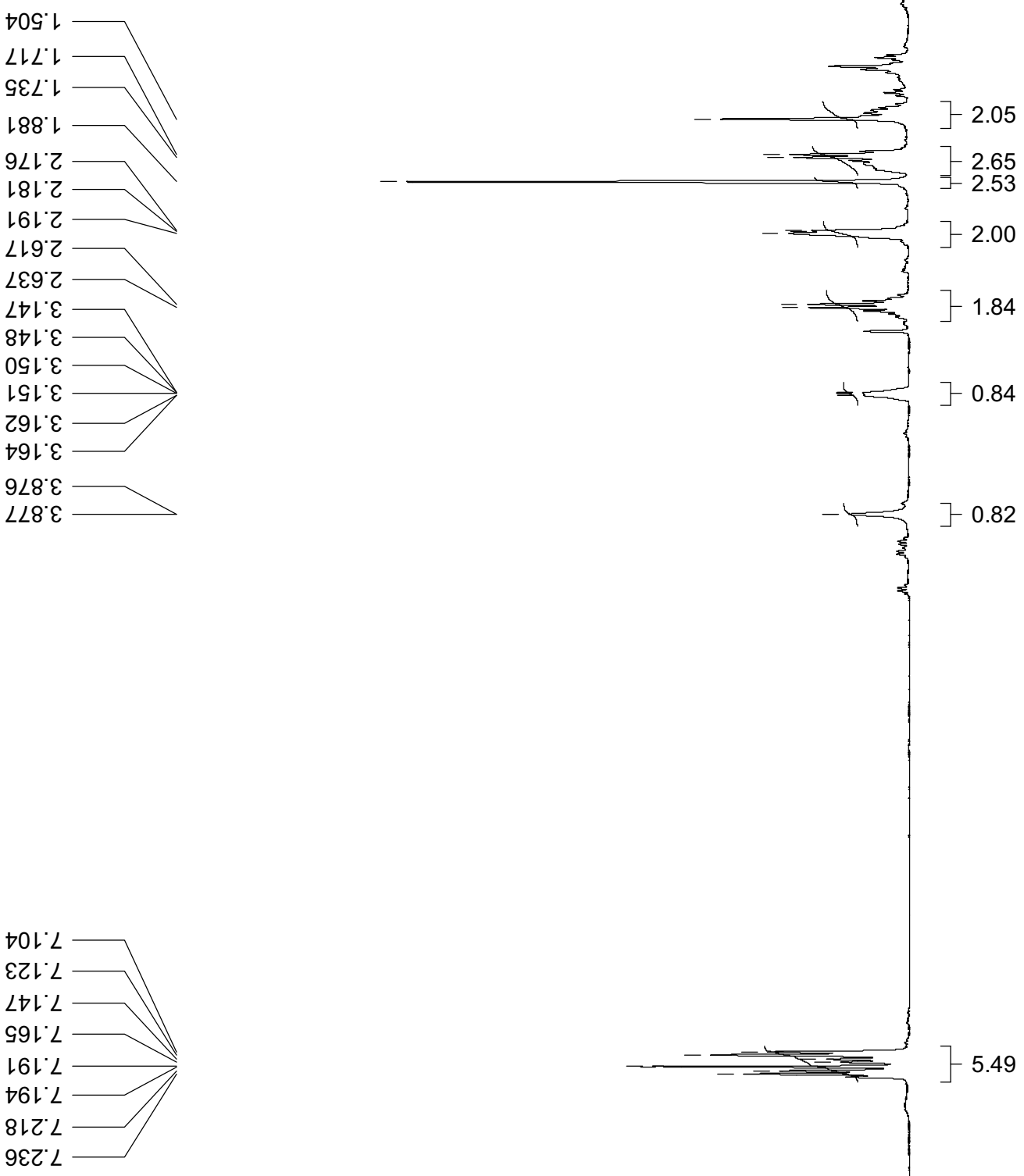
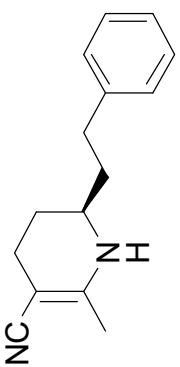


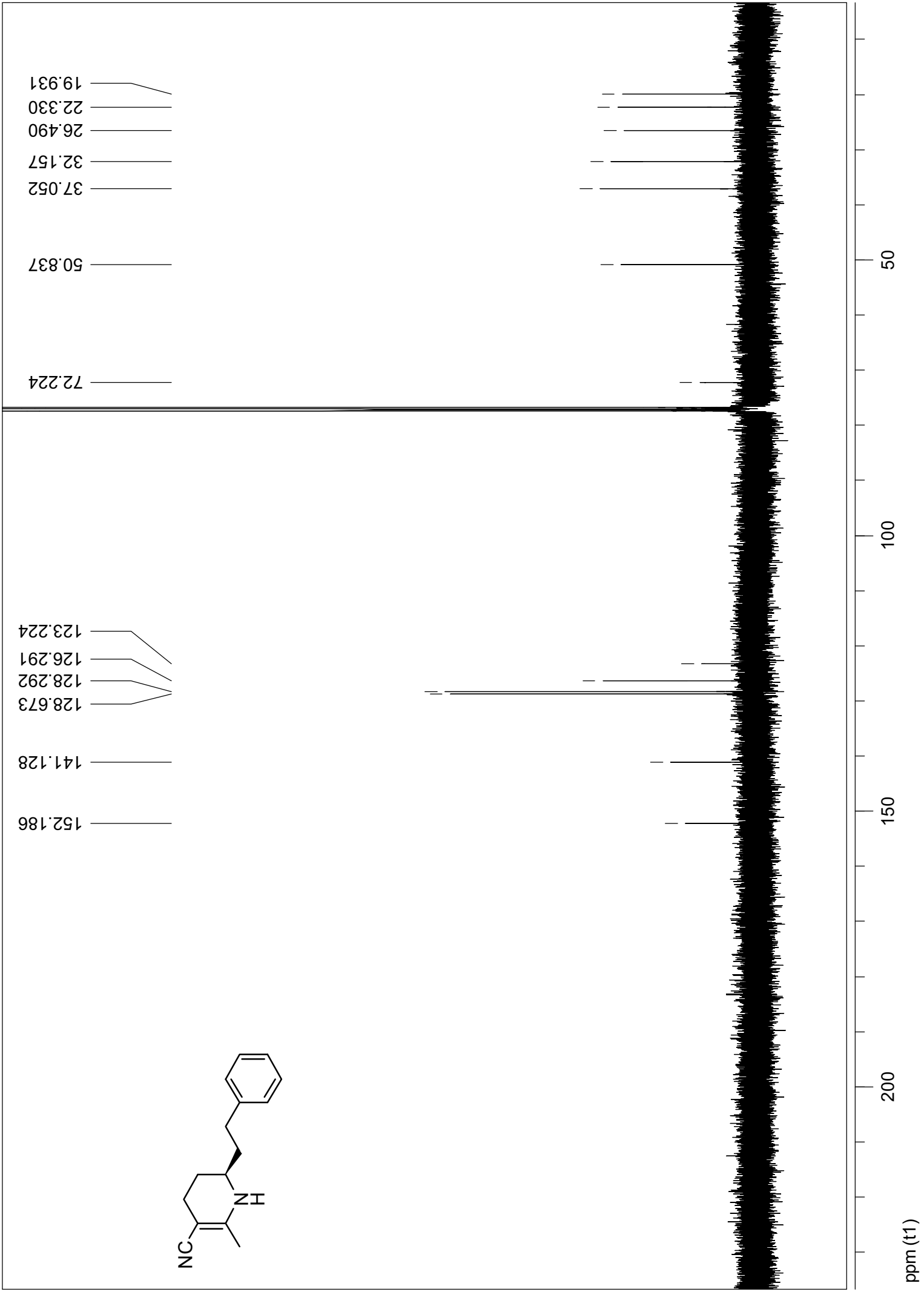
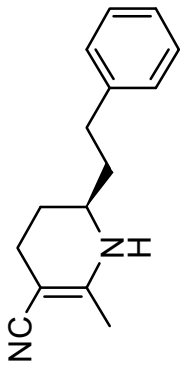
Chromatogram : AA585_2_ADH_973_flow1_2

Data file: AA585_2_ADH_973_flow1_2.DATA
Method: HPLC1_ADH_973_flow1_acq_60
Date: 16.01.2007 16:32:58



Index	Start [Min]	Time [Min]	End [Min]	Area %	
					[%]
1	29,036	30,408	32,691	93,000	
2	32,691	34,033	36,140	7,000	
Total				100,000	





Sample Information

Analyzed : 25.01.2007 23:36:08
 Sample Name : AA585-2

Method

===== Analytical Line 1 =====

[GC-2010]

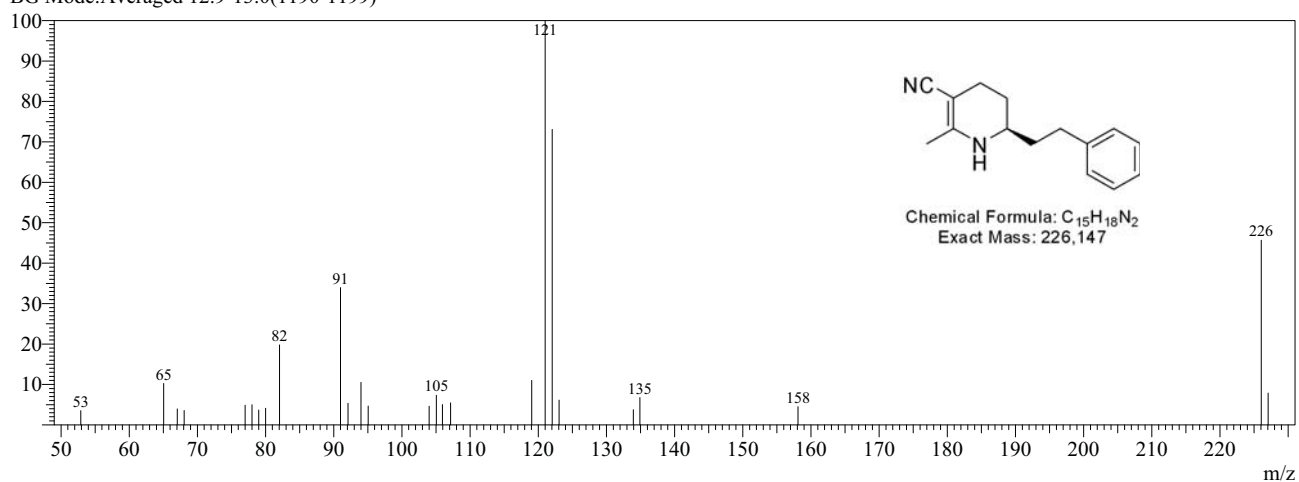
Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:12.6(Scan#:1147)
 MassPeaks:26 BasePeak:121(45699)
 RawMode:Averaged 12.5-12.6(1146-1149)
 BG Mode:Averaged 12.9-13.0(1190-1199)



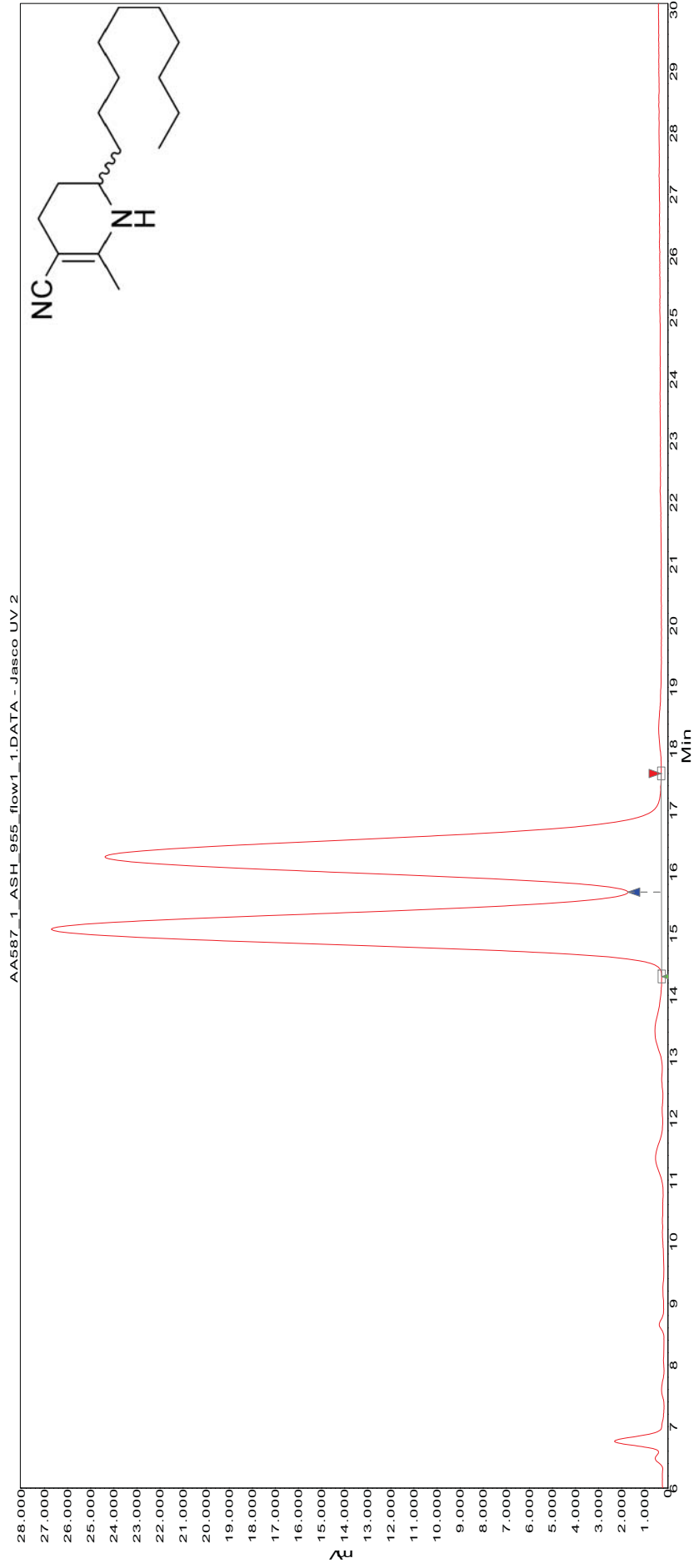
Mass Table

Line#:1 R.Time:12.6(Scan#:1147)
 MassPeaks:26 BasePeak:121(45699)
 RawMode:Averaged 12.5-12.6(1146-1149)
 BG Mode:Averaged 12.9-13.0(1190-1199)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	52.90	3.51	8	80.00	4.11	15	105.05	7.32	22	133.95	3.74
2	65.05	10.25	9	82.05	19.79	16	105.95	5.04	23	134.90	6.75
3	67.05	3.95	10	91.00	33.97	17	107.15	5.46	24	158.10	4.49
4	68.05	3.57	11	92.10	5.32	18	119.05	11.00	25	226.05	45.68
5	77.00	4.86	12	94.00	10.54	19	121.00	100.00	26	227.05	7.89
6	78.00	4.97	13	95.05	4.68	20	122.05	73.09			
7	79.00	3.69	14	104.00	4.64	21	123.05	6.14			

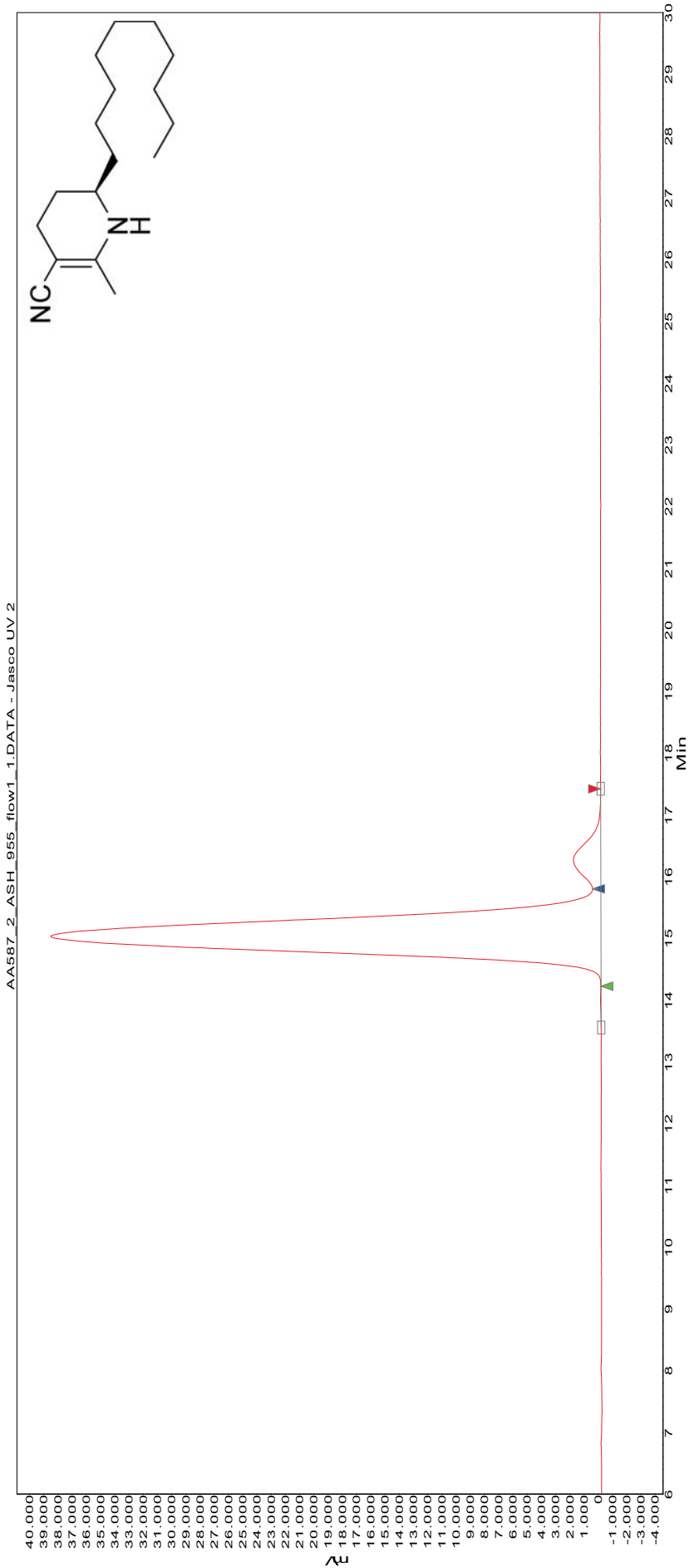
Chromatogram : AA587_1_ASH_955_flow1_1

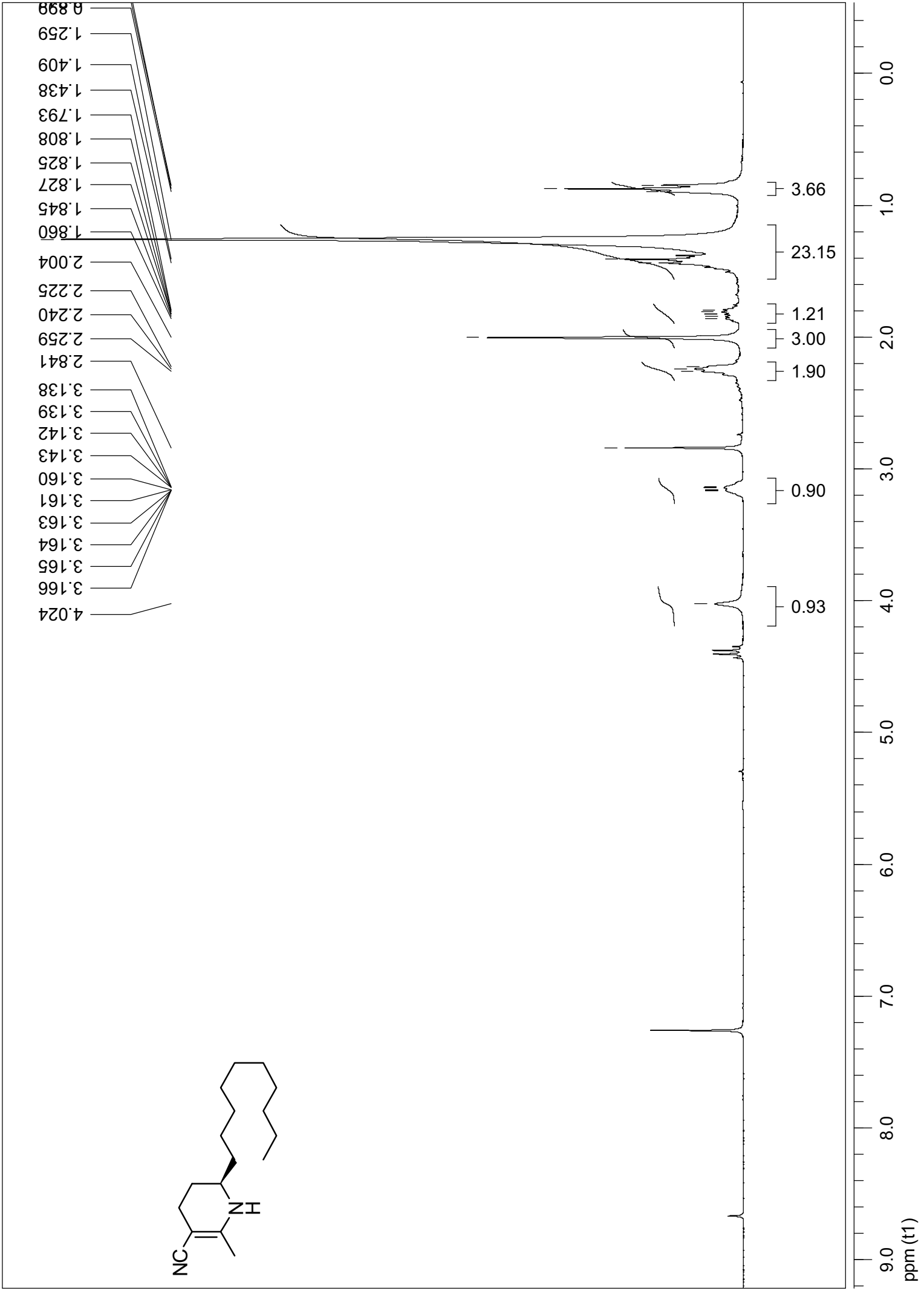
Data file: AA587_1_ASH_955_flow1_1.DATA
Method: HPLC2_ASH_955_flow1_acq60
Date: 18.01.2007 21:04:45

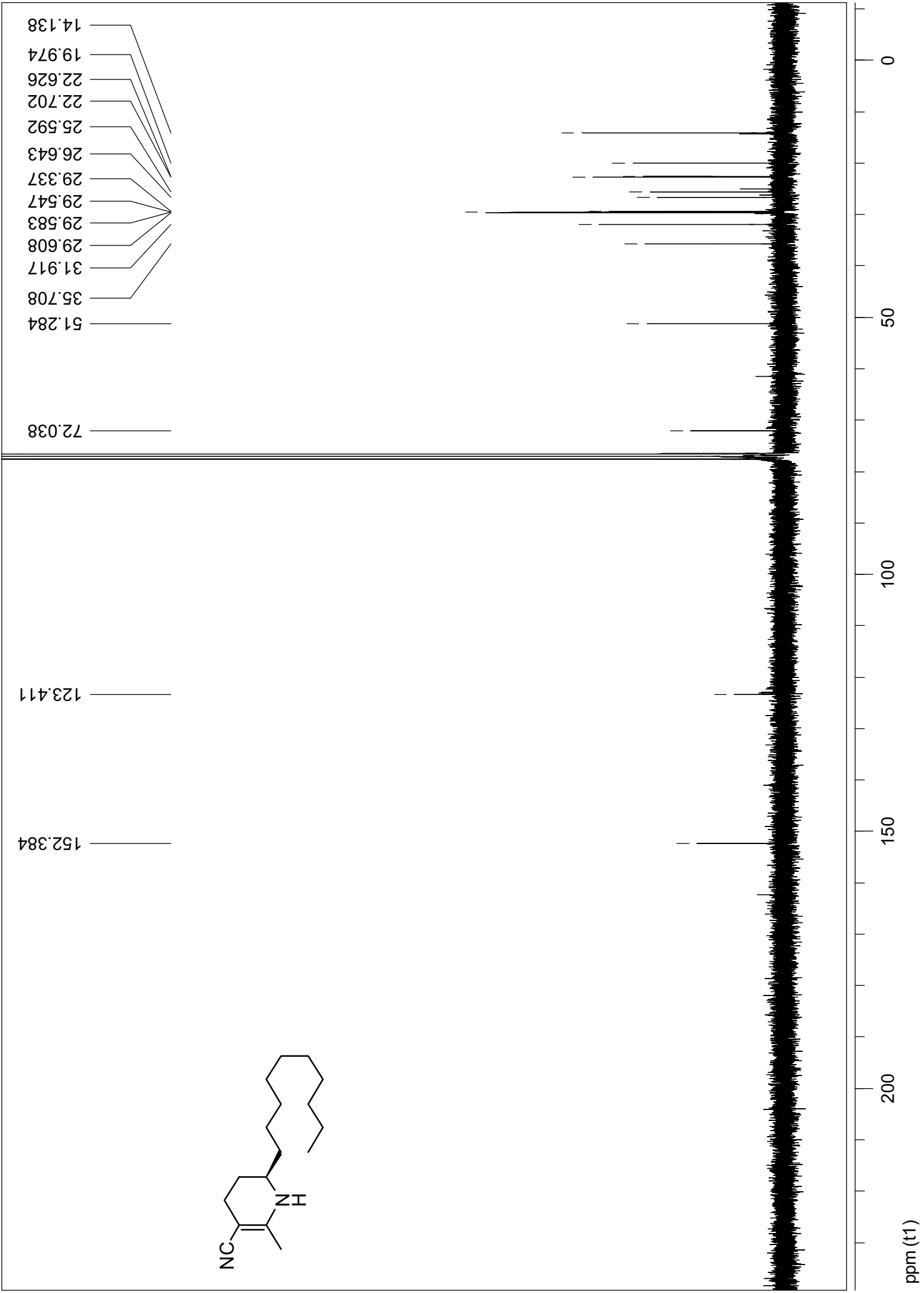
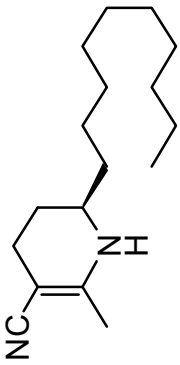


Chromatogram : AA587_2_ASH_955_flow1_1

Data file: AA587_2_ASH_955_flow1_1.DATA
Method: HPLC2_ASH_955_flow1_acq30
Date: 18.01.2007 21:37:18







Sample Information

Analyzed : 25.01.2007 22:18:35
 Sample Name : AA587-2

Method

===== Analytical Line 1 =====

[GC-2010]

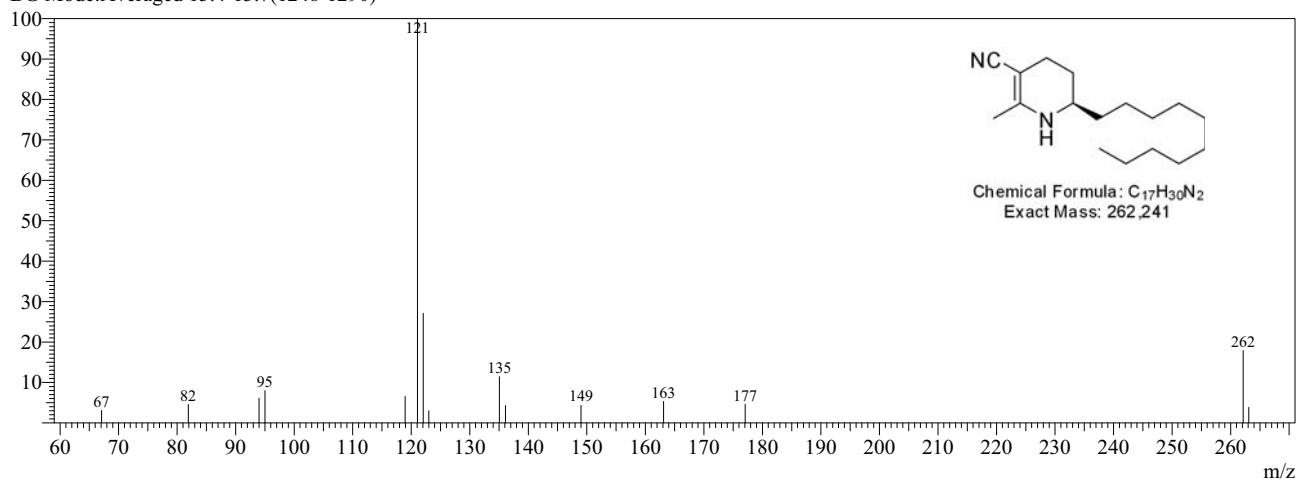
Column Oven Temp. : 40.0 °C
 Injection Temp. : 280.00 °C
 Injection Mode : Split
 Flow Control Mode : Linear Velocity
 Pressure : 24.2 kPa
 Total Flow : 38.3 mL/min
 Column Flow : 0.69 mL/min
 Linear Velocity : 30.0 cm/sec
 Purge Flow : 3.0 mL/min
 Split Ratio : 50.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	40.0	0.00
20.0	300.0	15.00

Spectrum

Line#:1 R.Time:13.0(Scan#:1202)
 MassPeaks:15 BasePeak:121(26474)
 RawMode:Averaged 13.0-13.0(1200-1205)
 BG Mode:Averaged 13.4-13.7(1248-1290)



Mass Table

Line#:1 R.Time:13.0(Scan#:1202)
 MassPeaks:15 BasePeak:121(26474)
 RawMode:Averaged 13.0-13.0(1200-1205)
 BG Mode:Averaged 13.4-13.7(1248-1290)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	67.10	3.10	5	118.95	6.56	9	135.05	11.45	13	177.05	4.58
2	81.90	4.57	6	121.05	100.00	10	136.10	4.33	14	262.15	17.90
3	94.00	6.15	7	122.05	27.12	11	149.00	4.36	15	263.10	3.82
4	95.00	8.01	8	123.00	3.00	12	163.10	5.31			