Brønsted Acid Differentiated Metal Catalysis by Kinetic Discrimination

Magnus Rueping and René M. Koenigs*

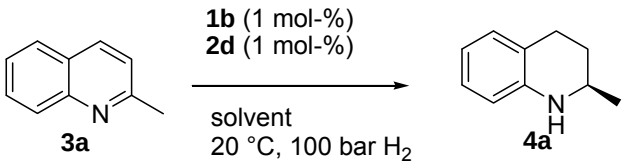
Supporting information

General: Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents used in reactions were p.A. grade and dried only if indicated. Dichloromethane was dried with CaH_2 before use. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminum plates with F-254 indicator, visualized by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.063 – 0.2 mm). Solvent mixtures are understood as volume/volume.

^{1}H-NMR and ^{13}C-NMR were recorded on a Bruker AM 250 respectively a Bruker AV 300 or a Varian Inova 400 spectrometer in $CDCl_3$. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated br (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartett), 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 = 0.25 µm, lot # 28089-U, Supelco). ESI spectra were conducted on a VG-Plattform II (Fisons Instruments). IR spectra were recorded on a Jasco FT/IR-420 and a Perkin Elmer Spectrum 100 spectrometer and are reported in terms of frequency of absorption (cm^{-1}). Melting points were measured on a Büchi 530 apparatus. Optical rotations were measured on a Perkin Elmer 241 polarimeter.

The enantiomeric excesses were determined by capillary GC analysis using a chiral stationary phase column: Shimadzu GC2010 with FID, column: Varian CP-Chirasil-DEX CB (25m x 0.25mm x 0.25µm), carrier gas: H_2 or He. The enantiomeric excess of **4j**-**4n** were determined by HPLC analysis using a chiral stationary phase column (column: Daicel Co. CHIRALCEL OD-H or CHIRALPAK IA; eluent: hexane/2-propanol). The chiral GC and HPLC methods were calibrated with the corresponding racemic mixtures. Chemical yields refer to pure isolated substances. The yields and enantiomeric excesses are given in table.

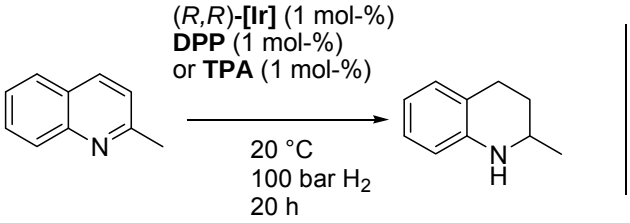
Influence of solvent:

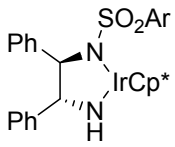


entry	solvent	time	conv[b]	e.r.[b]
1	*o*-xylene	7	55	92 : 8
2	toluene	7	45	92 : 8
3	benzene	7	30	92 : 8
4	trifluorotoluene	7	5	72 : 28
5	1,2-dichloroethane	7	15	57 : 43
6	chloroform	7	Spuren	66 : 34

[a] conditions: 0.15 mmol **5**, 0.8 mL solvent[b] determined via capillary GC

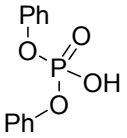
Influence of achiral additives using different enantiopure iridium catalysts



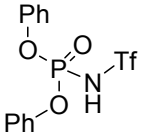


alle R,R

		DPP	TPA
entry	Aryl	%ee	%ee
1	p-Tolyl	*ent* 42	28
2	Mesityl	*ent* 60	*ent* 56
3	2-Naphthyl	*ent* 38	44
4	Pentamethylphenyl	56	54
5	3,5-Xylyl	*ent* 22	62
6	3,5-CF_3-Phenyl	54	62
7	4-Biphenyl	*ent* 28	46
8	Me	*ent* 6	62

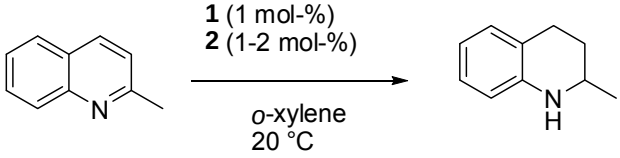


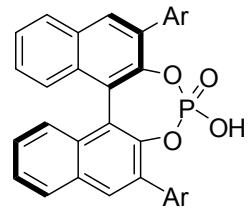
DPP



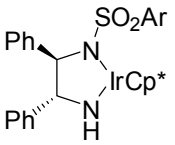
TPA

Influence of hydrogen pressure





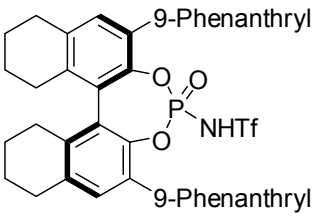
2a: Ar = 9-Phenanthryl



1: Ar = 2-Naphthyl

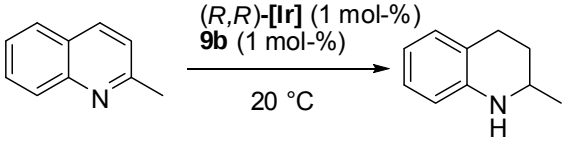
(*R*,*R*)

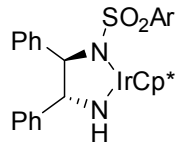
entry	HA	mol%	pressure	temp	time	conv	%ee
2	**2a**	2	30	20	18	>95	84
3	**2a**	2	60	20	22	>95	86
4	**2a**	2	72	20	20	>95	86
5	**2a**	1	100	20	19	>95	90
6	**2a**	2	100	20	19	>95	89.5
7	**9b**	1	100	20	20	>95	88
8	**9b**	1	150	20	20	>95	86

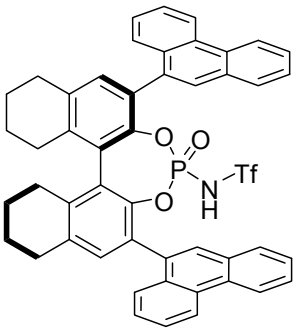


9b

Influence of the sulfonyl moiety







entry	mol%	time	Aryl	pressure	%ee
1*	1	10	p-Tolyl	50	75
2	1	10	Triisopropylphenyl	50->30	28
3	1	10	Mesityl	50->30	*ent* 13
4	1	14	2-Naphthyl	100	88
5	1	14	1-Naphthyl	100	31
6	1	14	9-Phenanthryl	100	37
7	1	14	3,5-Xylyl	100	54
8	1	14	3,5-CF_3-Phenyl	100	52
9	1	24	4-Biphenyl	100	86
10	1	24	Me	100	82

pressure in bar, first number indicates initial pressure, second number is pressure observed at the end of rct

Methods for the determination of enantiomeric excess via capillary GC

Method 1; 100°C, hold temperature for 1 min, then heating 2 °C/min until a temperature of 135 °C is reached, then heating 20 °C/min until a temperature of 200 °C is reached. This temperature is held for 2 min. Carrier gas: H_2

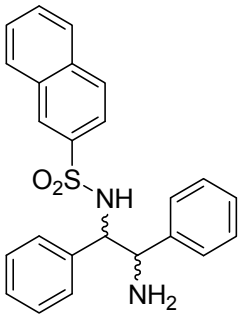
Method 2: 130 °C, then heating 4 °C/min until a temperature of 210 °C is reached. This temperature is held for 1 min. Carrier gas: He

Method 3: 100°C, hold temperature for 1 min, then heating 2 °C/min until a temperature of 140 °C is reached, then heating 40 °C/min until a temperature of 200 °C is reached. This temperature is held for 1 min. Carrier gas: H_2

Method 4: 100°C, hold temperature for 1 min, then heating 2 °C/min until a temperature of 160 °C is reached, then heating 40 °C/min until a temperature of 200 °C is reached. This temperature is held for 1 min. Carrier gas: H_2

Method 5: 100°C, hold temperature for 1 min, then heating 2 °C/min until a temperature of 180 °C is reached, then heating 40 °C/min until a temperature of 200 °C is reached. This temperature is held for 1 min. Carrier gas: H_2

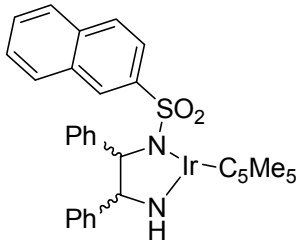
Synthesis of (*rac*)-1,2-Diphenyl-*N*-(2-naphthylsulfonyl)ethylenediamine



The title compound was prepared analogous to the literature method.[1] To an ice-cooled solution of *rac*-1,2-Diphenylethylenediamine (263 mg, 1.2 mmol) in dry Dichloromethane (5 mL) were subsequently added Diisopropylethylamine (0.3 mL, 1.8 mmol, 1.5 eq) and solid 2-Naphthylsulfonylchloride (280 mg, 1.2 mmol, 1 eq). After stirring overnight the crude reaction mixture was purified via column chromatography using hexanes/ethylacetate eluent to yield 342 mg (69 %) of a white fluffy solid.

mp: 183 °C; ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 7.97 (s, 1H), 7.79 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.63 (d, 8.6 Hz, 1H), 7.45-7.6 (m, 2H), 7.42 (dd, *J* = 8.6 Hz, *J* = 1.8 Hz, 1H), 6.9 – 7.2 (m, 10H), 4.47 (d, *J* = 5.1 Hz, 1H), 4.15 (d, *J* = 5.1 Hz, 1H), 1.6 (b, 3H); ^{13}C-NMR (62.9 MHz, $CDCl_3$): δ = 141.079, 139.052, 137.089, 134.501, 131.961, 129.231, 128.855, 128.380, 128.345, 128.229, 128.198, 127.656, 127.528, 127.468, 127.037, 126.980, 126.459, 122.248, 63.335, 60.426; IR (KBr): v = 3378, 3313, 3169, 3056, 3027, 2907, 1590, 1493, 1455, 1394, 1347, 1300, 1201, 1149, 1129, 1075, 958, 937, 905, 871, 819, 796, 767, 750, 698, 641, 564; MS-ESI: *m/z*(%): 403 [M+H]

Synthesis of *rac*-Cp*Ir[**8**] (**1b**)



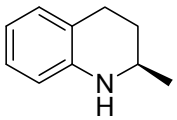
1b was prepared analogous to the literature method.[2] To a solution of $[Cp^*IrCl_2]_2$ (103 mg, 0.13 mmol) and **8** (104 mg, 0.26 mmol, 2 eq) in 2 mL Dichloromethane Triethylamine (190µL, 1.3 mmol, 10 eq) was added. The initially orange solution changed to red. After stirring overnight water was added and the red solution immediately turned dark burgundy red. The aqueous layer was extracted three times with Dichloromethane. The combined organic layers were dried over Na_2SO_4. After removal of solvent and thorough drying in high vacuum the residual

dark-red foamy solid was triturated with 4 mL hexanes overnight yielding a dark-burgundy red microcrystalline solid, which was collected after filtration and washing of the filtrate with hexanes (148 mg, 79 %).

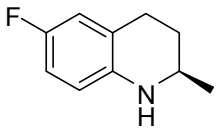
mp: 207 °C; ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 7.4-7.9 (m, 10H), 6.5-7.3 (m, 7H), 5.43 (b, 1H), 4.43 (s, 1H), 4.16 (d, *J* = 3.4 Hz, 1H), 1.92 (s, 15H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 146.673, 146.445, 141.023, 133.814, 131.993, 128.861, 128.674, 127.728, 127.695, 127.664, 127.415, 127.034, 126.892, 126.634, 126.510, 126.343, 126.127, 125.973, 123.392, 85.241, 80.084, 74.077, 10.186; IR (KBr): v = 3331, 2911, 1598, 1491, 1448, 1296, 1126, 1074, 1027, 974, 814, 777, 747, 698, 660, 618; MS-ESI: *m/z*: 729 [M+H]

General procedure for iridium catalyzed hydrogenation

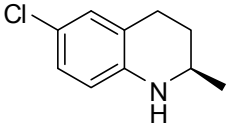
To a vial **1** (0.02mmol) and the appropriate additive and 1.0 mL of solvent were added. After stirring in air for 5 min quinoline (0.2 mmol) was added and the reaction mixture was put in a stainless steel autoclave. The system was connected to a supply of argon/hydrogen and the atmosphere in the autoclave was purged twice with Argon (5 bar). The autoclave was then filled with hydrogen (100 bar). After 16-48 h the pressure was released and the autoclave was purged with twice with Argon (5 bar). The crude reaction mixture was then purified via column chromatography using hexanes/ethylacetate.



2-Methyl-1,2,3,4-tetrahydro-quinoline[3]: $[\alpha]_D^{RT}$ = +80.8 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ =6.9-7.1 (m, 2H), 6.61 (t, *J* = 7.3 Hz, 1), 6.47 (d, *J* = 8.2 Hz, 1H), 3.6 (b, 1H), 3.3-3.5 (m, 1H), 2.6-2.95 (m, 2H), 1.85-2.0 (m, 1H), 1.5-1.7 (m, 1H), 1.2 (d, *J* = 6.3 Hz, 3H); ^{13}C-NMR (62.9 MHz, $CDCl_3$): δ = 144.623, 129.3, 126.7, 121.2, 117.1, 114.1, 47.2, 30.1, 26.6, 22.5; IR (NaCl): v = 3393, 3049, 3014, 2961, 2923, 2843, 1607, 1489, 1451, 1375, 1341, 1308, 1276, 1257, 1153, 1124, 1039, 963, 746; MS-EI: *m/z*(%): 147 (37), 132 (100), 117 (15), 77 (9); Capillary GC: Method 1, minor enatiomer: t_R = 9.28 min, major enantiomer: t_R =9.41 min

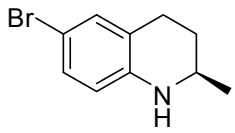


2-Methyl-6-Fluoro-1,2,3,4-tetrahydro-quinoline[3]: $[\alpha]_D^{RT}$ = +66.8 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.6-6.75 (m, 2H), 6.4-6.5 (m, 1H), 4.0 (b, 1H), 3.3-3.4 (m, 1H), 2.6-2.9 (m, 2H), 1.9-2.0 (m, 1H), 1.5-1.7 (m, 1H), 1.21 (d, *J* = 6.3 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 140.5, 122.7 (d, *J* = 6.9 Hz), 115.4 (d, *J* = 21.7 Hz), 115.0 (d, *J* = 7.7 Hz), 113.2 (d, *J* = 22.4 Hz), 47.4, 29.8, 26.6, 22.3; IR (NaCl): 3397,2926, 2848, 1599, 1500, 1376, 1340, 1304, 1254, 1232, 1141, 805; MS-EI: *m/z*(%): 165 (40), 150 (100), 135 (28), 130 (14), 109 (9), 96 (9), 77 (10); Capillary GC: Method 3, minor enatiomer: t_R = 8.37 min, major enantiomer: t_R =9.49 min

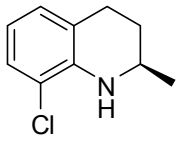


2-Methyl-6-Chloro-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +108.7 (c = 1.0, $CHCl_3$);^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.9-7.0 (m, 2H), 6.40 (d, *J* = 8.2 Hz, 1H), 3.96 (b, 1H), 3.3-3.45 (m, 1H), 2.6-2.9 (m, 2H), 1.9-2.0 (m, 1H), 1.5-1.6 (m, 1H), 1.21 (d, *J* = 6.3 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 143.0, 128.8, 126.5, 122.7, 121.5, 115.1, 47.2, 29.6, 26.4, 22.3; IR (KBr): v = 3405, 2925, 2847, 1604, 1579, 1491, 1376, 1298, 1132, 876, 806; MS-EI:

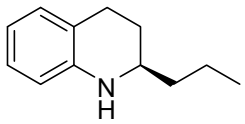
m/z(%): 183 (17), 181 (56), 168 (30), 166 (100), 131 (83), 130 (81), 103 (15), 77 (30); Capillary GC: Method 2, minor enatiomer: t_R = 13.19 min, major enantiomer: t_R =13.36 min



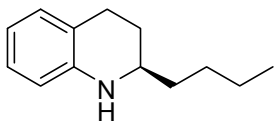
2-Methyl-6-Bromo-1,2,3,4-tetrahydro-quinoline: mp 48-50 °C; $[\alpha]_D^{RT}$ = +64.4 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 7.1-7.2 (m, 2H), 6.36 (d, *J* = 8.3 Hz, 1H), 4.0 (b, 1H), 3.3-3.45 (m, 1H), 2.6-2.9 (m, 2H), 1.85-2.0 (m, 1H), 1.45-1.65 (m, 1H), 1.21 (d, *J* = 6.3 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 143.480, 131.6, 129.3, 123.2, 115.5, 108.5, 47.1, 29.5, 26.3, 22.3; IR (KBr): v = 3399, 2964, 2925, 2843, 1597, 1575, 1488, 1446, 1297, 1133, 877, 805; MS-EI: *m/z*(%): 227 (19), 225 (20), 212 (30), 210 (33), 144 (10), 131 (65), 130 (100), 103 (13), 77 (23); Capillary GC: Method 2, minor enatiomer: t_R = 15.47 min, major enantiomer: t_R =15.63 min



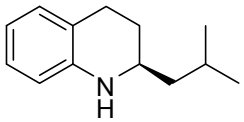
2-Methyl-8-Chloro-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +63.8 (c = 1.0, $CHCl_3$);^{1}H-NMR (250 MHz, $CDCl_3$): δ = 7.06 (d, *J* = 7.9 Hz, 1H), 6.87 (d, *J* = 7.5 Hz, 1H), 6.5 (t, *J* = 7.7 Hz, 1H), 4.4 (b, 1H), 3.4-3.55 (m, 1H), 2.7-2.9 (m, 2H), 1.9-2.0 (m, 1H), 1.5-1.7 (m, 1H), 1.27 (d, *J* = 6.3 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 140.7, 127.4, 126.7, 122.4, 117.8, 116.3, 47.1, 29.6, 26.7, 22.5; IR (NaCl): v = 3413, 3065, 3019, 2924, 1602, 1568, 1493, 1376, 1343, 1330, 1295, 1188, 1139, 1098, 1090, 966, 933, 754, 722; MS-EI: *m/z*(%): 183 (13), 181 (44), 168 (26), 166 (88), 131 (67), 130 (100), 104 (27), 77 (34); Capillary GC: Method 2, major enatiomer: t_R = 9.33 min, minor enantiomer: t_R =9.45 min



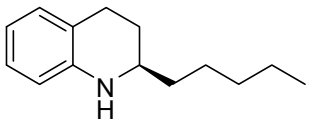
2-Propyl-1,2,3,4-tetrahydro-quinoline[3]: $[\alpha]_D^{RT}$ = +81.6 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.9-7.0 (m, 2H), 6.61 (t, *J* = 7.3 Hz, 1H), 6.51 (d, *J* = 8.2 Hz, 1H), 4.2 (b, 1H), 3.2-3.3 (m, 1H), 2.6-2.9 (m, 2H), 1.9-2.0 (m, 1H), 1.3-1.7 (m, 5H), 0.96 (t, *J* = 6.8 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 144.4, 129.2, 126.7, 121.6, 117.1, 114.2, 51.4, 38.7, 28.0, 26.3, 18.9, 14.1; IR (NaCl): 3404, 3050, 2955, 2926, 2870, 1607, 1585, 1485, 1353, 1309, 1275, 1253, 1125, 745; MS-EI: *m/z*(%): 175 (12), 132 (100), 117 (15), 77 (7); Capillary GC: Method 4, minor enatiomer: t_R = 11.04 min, major enantiomer: t_R =11.17 min



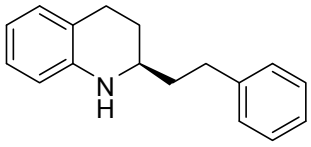
2-Butyl-1,2,3,4-tetrahydro-quinoline[3]: $[\alpha]_D^{RT}$ = +73.0 (c = 1.0, $CHCl_3$);^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.9-7.0 (m, 2H), 6.6-6.75 (m, 1H), 6.52 (d, *J* = 7.9 Hz, 1H), 4.3 (b, 1H), 3.2-3.3 (m, 1H), 2.6-2.9 (m, 2H), 1.9-2.0 (m, 1H), 1.3-1.7 (m, 7H), 0.93 (t, *J* = 7.0 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 129.2, 126.7, 121.6, 117.1, 114.2, 51.6, 36.2, 28.0, 27.9, 26.3, 22.8, 14.076; IR (NaCl): v = 3403, 2926, 2855, 1607, 1585, 1484, 1351, 1309, 1275, 745; MS-EI: *m/z*(%): 189 (9), 132 (100), 117 (13), 77 (6); Capillary GC: Method 4, minor enatiomer: t_R = 13.14 min, major enantiomer: t_R =13.25 min



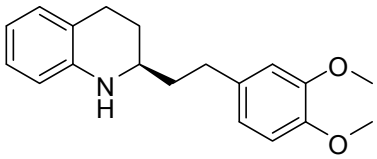
2-*iso*-Butyl-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +79.2 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.9-7.0 (m, 2H), 6.55-6.65 (m, 1H), 6.47 (d, *J* = 8.2 Hz, 1H), 3.7 (b, 1H), 3.3-3.4 (m, 1H), 2.6-2.9 (m, 2H), 1.9-2.0 (m, 1H), 1.7-1.9 (m, 1H), 1.5-1.7 (m, 1H), 1.3-1.5 (m, 2H), 0.95 (d, *J* = 6.6 Hz, 6H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 144.6, 129.2, 126.6, 121.4, 116.9, 114.1, 49.2, 45.9, 28.5, 26.4, 24.4, 23.2, 22.4; IR (NaCl): v = 3407, 3051, 3015, 1606, 1585, 1484, 1384, 1366, 1353, 1308, 1273, 1153, 1001, 927, 744, 716; MS-EI: *m/z*(%): 189 (11), 132 (100), 117 (11), 77 (5); Capillary GC: Method 5, minor enatiomer: t_R = 11.97 min, major enantiomer: t_R =12.12 min



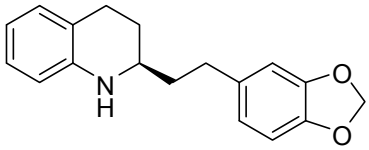
2-Pentyl-1,2,3,4-tetrahydro-quinoline[3]: $[\alpha]_D^{RT}$ = +75.2 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.9-7.0 (m, 2H), 6.60 (dt, *J* = 7.4 Hz, *J* = 1.0 Hz, 1H), 6.48 (d, *J* = 7.4 Hz, 1H), 3.88 (b, 1H), 3.2-3.3 (m, 1H), 2.7-2.9 (m, 2H), 1.9-2.05 (m, 1H), 1.55-1.7 (m, 1H), 1.3-1.55 (m, 8H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C-NMR (75.4 MHz, $CDCl_3$): δ = 144.6, 129.2, 126.7, 121.4, 116.9, 114.1, 51.6, 36.6, 31.9, 28.1, 26.4, 25.4, 22.6, 14.0; IR (NaCl): v = 3407, 2925, 2853, 1607, 1585, 1484, 1352, 1309, 1253, 1125, 745; MS-EI: *m/z*(%): 203 (8), 132 (100), 117 (11), 77 (4); Capillary GC: Method 4, minor enatiomer: t_R = 15.40 min, major enantiomer: t_R =15.49 min



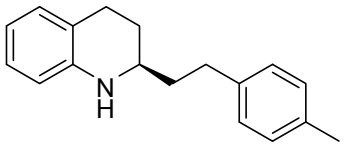
2-Phenethyl-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +73.4 (c = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.27-7.34 (m 3H), 7.17-7.22 (m, 2H), 6.93-6.96 (m, 2H), 6.60 (td, *J* = 7.3 Hz, *J* = 1.1 Hz, 1H), 6.43-6.48 (m, 1H), 3.75 (b, 1H), 3.25-3.35 (m, 1H), 2.7-2.85 (m, 4H), 1.95-2.05 (m, 1H), 1.8-1.88 (m, 2H), 1.62-1.74 (m, 1H); ^{13}C-NMR (100.6 MHz, $CDCl_3$): δ = 144.4, 141.7, 129.2, 128.4, 128.3, 126.6, 125.9, 121.2, 117.0, 114.1, 51.2, 38.3, 32.2, 28.1, 26.3; IR (KBr): v = 3406, 3055, 3023, 2921, 2849, 1605, 1490, 1451, 1354, 1310, 1116, 748, 699; MS-EI: *m/z*(%): 237 (7), 132 (100), 117 (30), 105 (20), 103 (16), 92 (13), 91 (84), 77 (24); HPLC: Daicel Chiralcel OD-H, *n*-hexanes/*i*Propanol 98/2, flow rate 0.6 mL/min, major enantiomer: t_R = 27.9 min, minor enantiomer t_R = 30.5 min



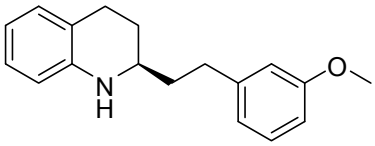
2-[2-(3,4-Dimethoxyphenyl)ethyl]-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +57.9 (c = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 6.94-7.00 (m, 2H), 6.81 (d, *J* = 7.9 Hz, 1H), 6.72-6.78 (m, 2H), 6.58-6.64 (m, 1H), 6.44-6.47 (m, 1H), 3.88 (s, 3H), 3.87 (s, 3H), 3.28-3.35 (m, 1H), 2.66-2.87 (m, 4H), 1.96-2.04 (m, 1H), 1.79-1.86 (m, 2H), 1.63-1.74 (m, 1H); ^{13}C-NMR 100.6 MHz, $CDCl_3$): δ = 148.8, 147.2, 144.4, 134.3, 129.1, 126.6, 121.2, 120.0, 116.9, 114.0, 111.5, 111.2, 56.0, 55.9, 51.2, 38.5, 31.9, 28.1, 26.3; IR (KBr): v = 3395, 3012, 2931, 2845, 1605, 1513, 1310, 1261, 1151, 1029, 752; MS-EI: *m/z*(%): 297 (7), 151 (6), 132 (100), 117 (17), 105 (8), 78 (8); HPLC: Daicel Chiralcel OD-H, *n*-hexanes/*i*Propanol 80/20, flow rate 0.6 mL/min, major enantiomer: t_R = 24.7 min, minor enantiomer t_R = 27.2 min



2-[2-(1,3-Benzodioxol-5-yl)ethyl]-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +63.8 (c = 1.0, $CHCl_3$); ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 6.92-7.00 (m, 2H), 6.70-6.76 (m, 2H), 6.64-6.68 (m, 1H), 6.61 (dd, *J* = 7.3 Hz, *J* = 1.1 Hz, 1H), 6.44-6.48 (m, 1H), 5.93 (s, 2H), 3.76 (b, 1H), 3.25-3.33 (m, 1H), 2.70-2.82 (m, 2H), 2.63-2.70 (m, 2H), 1.95-2.03 (m, 1H), 1.75-1.83 (m, 2H), 1.62-1.72 (m, 1H); ^{13}C-NMR (100.6 MHz, $CDCl_3$): δ = 147.5, 145.6, 144.4, 135.5, 129.1, 126.6, 121.2, 120.9, 117.0, 114.0, 108.7, 108.1, 100.7, 51.0, 38.5, 31.9, 28.0, 26.3; IR (KBr): v = 3408, 3011, 2920, 2850, 1606, 1488, 1442, 1354, 1039, 933, 752; MS-EI: *m/z*(%): 281 (6), 145 (6), 132 (100), 117 (24), 91 (15), 77 (12); HPLC: Daicel Chiralcel OD-H, *n*-hexanes/*i*Propanol 90/10, flow rate 1.0 mL/min, major enantiomer: t_R = 13.9 min, minor enantiomer t_R = 18.4 min



2-(2-[4-Methylphenyl]ethyl)-1,2,3,4-tetrahydro-quinoline: $[\alpha]_D^{RT}$ = +71.3 (c = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.14 (s, 4H), 6.97-7.03 (m, 2H), 6.64 (dd, *J* = 7.4 Hz, *J* = 1.1 Hz, 1H), 6.46-6.50 (m, 1H), 3.8 (b, 1H), 3.29-3.36 (m, 1H), 2.70-2.90 (m, 4H), 2.36 (s, 3H), 1.99-2.06 (m, 1H), 1.81-1.88 (m, 2H), 1.65-1.76 (m, 1H); ^{13}C-NMR (100.6 MHz, $CDCl_3$): δ = 144.5, 138.6, 135.3, 129.2, 129.1, 129.0, 128.9, 128.7, 128.2, 126.6, 121.2, 116.9, 114.1, 51.1, 38.4, 31.8, 28.1, 26.3, 21.1; IR (KBr): v = 3407, 3012, 2920, 2850, 1606, 1585, 1485, 1449, 1354, 1309, 1216, 1117, 807, 750; MS-EI: *m/z*(%): 251 (5), 144 (6), 132 (100), 117 (47), 105 (56), 91 (36), 77 (25), 65 (10); HPLC: CHIRALPAK IA *n*-hexanes/*i*Propanol 98/2, flow rate 1.0 mL/min, major enantiomer: t_R = 7.5 min, minor enantiomer t_R = 6.95 min



2-(2-[3-Methoxyphenyl]ethyl)-1,2,3,4-tetrahydro-quinoline: $[\alpha_D^{20}]$= +65.5 (c = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.14-7.24 (m, 1H), 6.92-6.98 (m, 2H), 6.72-6.82 (m, 3H), 6.61 (dd, *J* = 7.3 Hz, *J* = 1.1 Hz, 1), 6.43-6.47 (m, 1H), 3.80 (s, 3H), 3.75 (b, 1H), 3.26-3.34 (m, 1H), 2.69-2.86 (m, 4H), 1.95-2.05 (m, 1H), 1.80-1.87 (m, 2H), 1.63-1.73 (m, 1H); ^{13}C-NMR (100.6 MHz, $CDCl_3$): δ = 159.6, 144.4, 143.4, 129.4, 129.2, 126.6, 121.2, 120.7, 117.0, 114.1, 114.1, 111.1, 55.2, 51.1, 38.2, 32.3, 28.0, 26.3; IR (KBr): v = 3402, 3010, 2920, 2848, 1604, 1585, 1488, 1435, 1354, 1310, 1258, 1153, 1044, 874, 749, 695; MS-EI: *m/z*(%): 267 (5), 145 (8), 132 (100), 117 (25), 91 (20), 77 (11); HPLC: Daicel Chiralcel OD-H, *n*-hexanes/*i*Propanol 90/10, flow rate 1.0 mL/min, major enantiomer: t_R = 16.2 min, minor enantiomer t_R = 18.3 min

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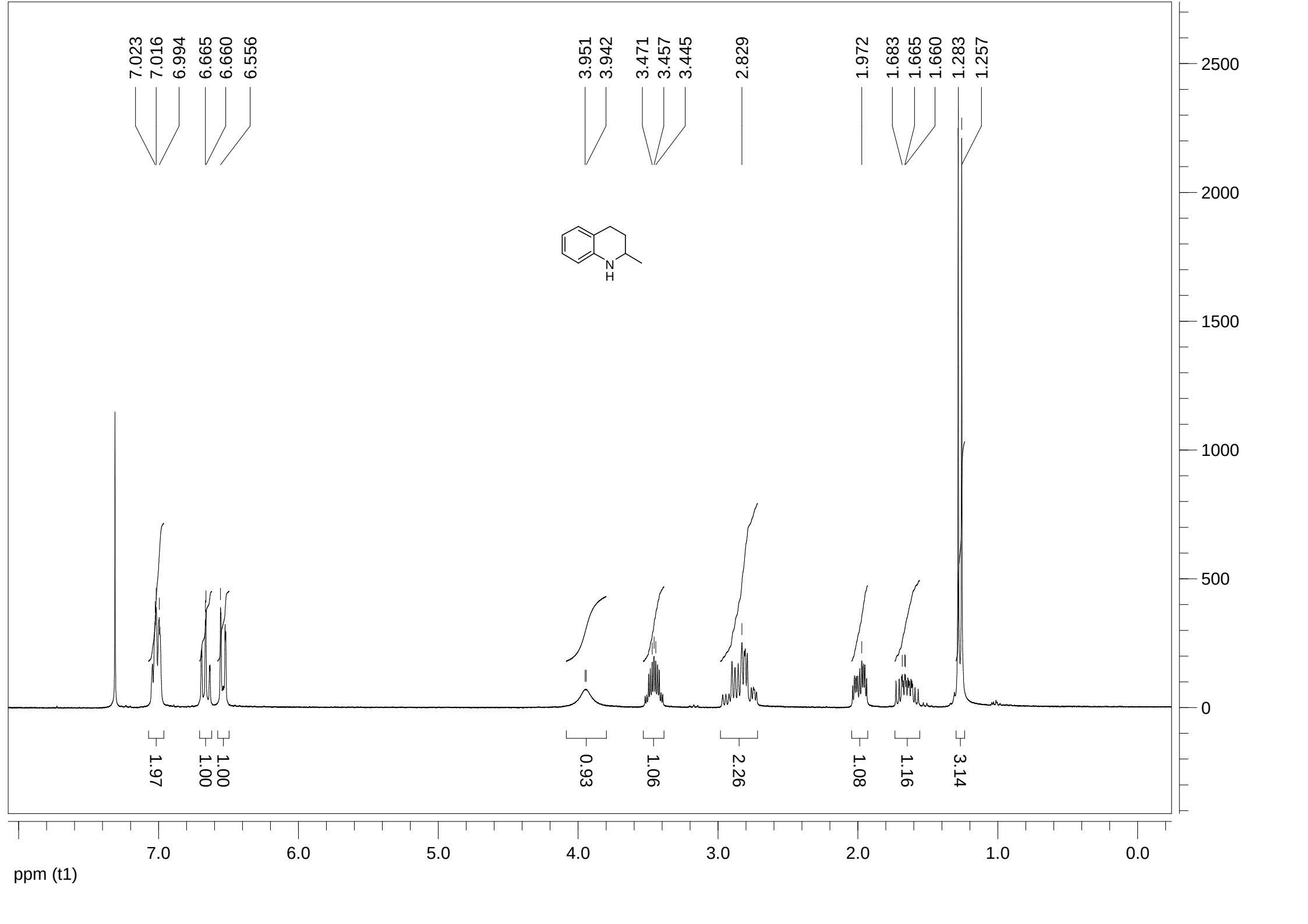
[1] D. Xue, Y.-C. Chen, Xi. Cui, Q.-W. Wang, J. Zhu, J.-G. Deng, *J. Org. Chem.*, 2005, 70, 3584
[2] Z. M. Heiden, T. B. Rauchfuss, *J. Am. Chem. Soc.* **2007**, *129*, 14303
[3] W.-B. Wang, S.-M. Lu, P.-Y. Yang, X.-W. Han, Y.-G. Zhou, *J. Am. Chem. Soc.* **2003**, *125*, 10536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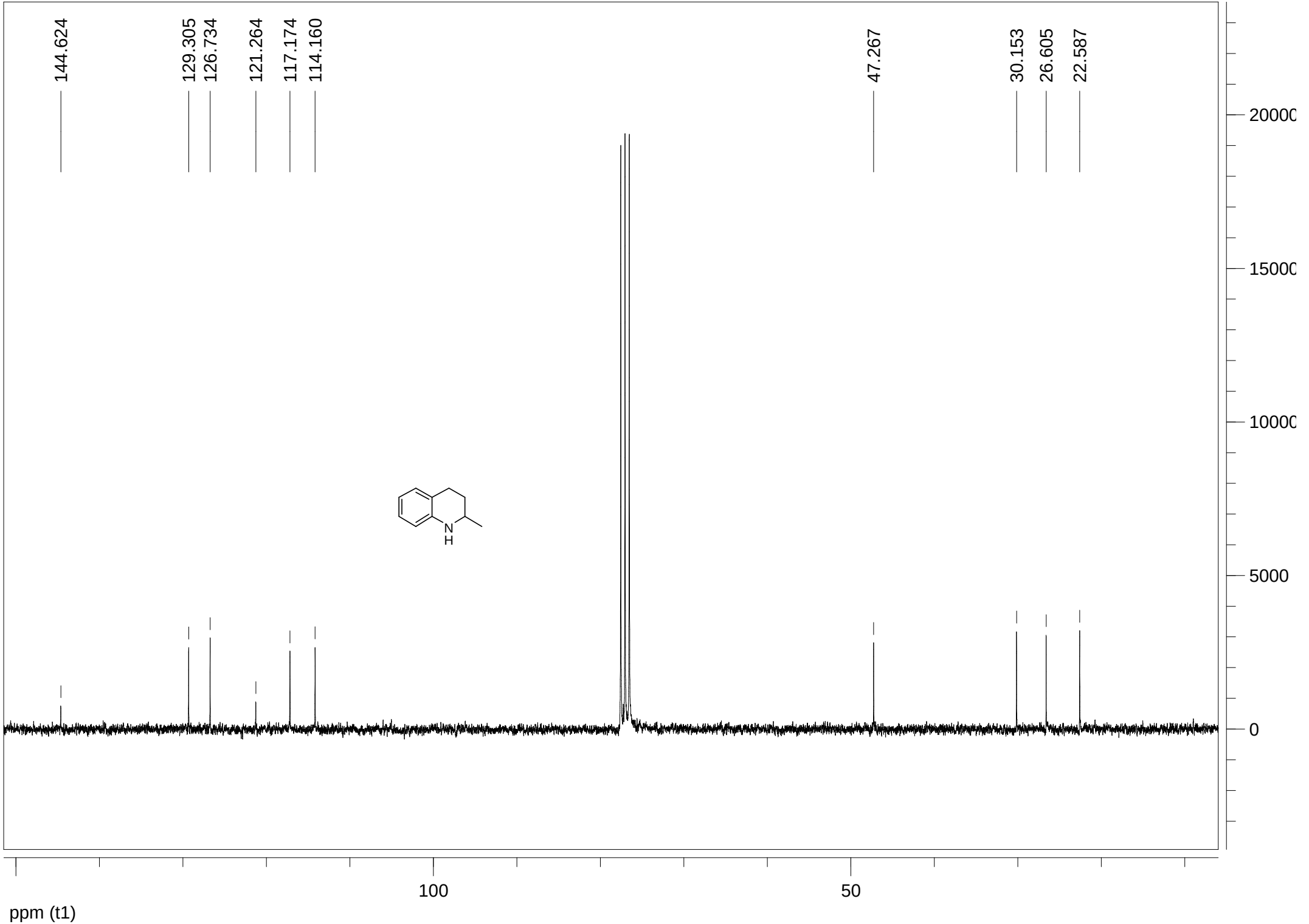
7.016
6.994
6.665
6.660
6.556
3.951
3.942
3.471
3.457
3.445
2.829
1.972
1.683
1.665
1.660
1.283
1.257
N
H
1.97
1.00
1.00
0.93
1.06
2.26
1.08
1.16
3.14
2500
2000
1500
1000
500
0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


144.624
129.305
126.734
121.264
117.174
114.160
47.267
30.153
26.605
22.587
20000
15000
10000
5000
0
N
H
100
50
ppm (t1)

Analysis Date & Time : 12.05.2009 11:01:55
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Sample ID :
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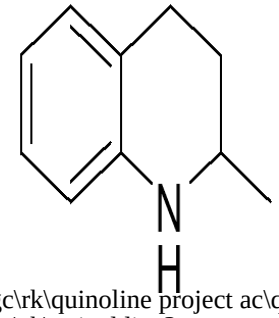
ISTD Amount :



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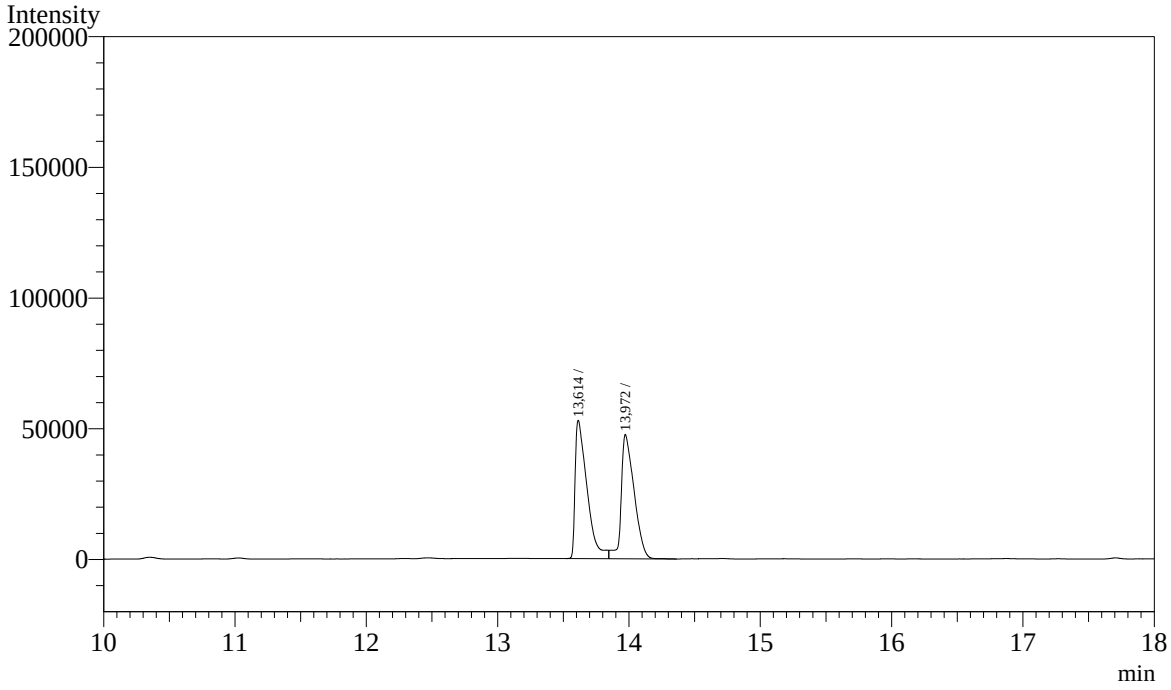
Method Name : F:\data shimadzu chiral gc\rk\quinaldine3.gcm



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Vial# : 4
Sample Name : rk641-51c
Sample ID :
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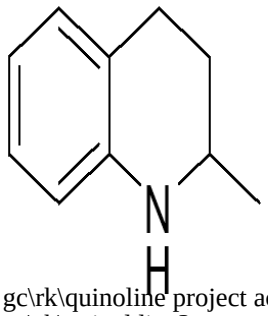
Injection Volume : 1,00
ISTD Amount :



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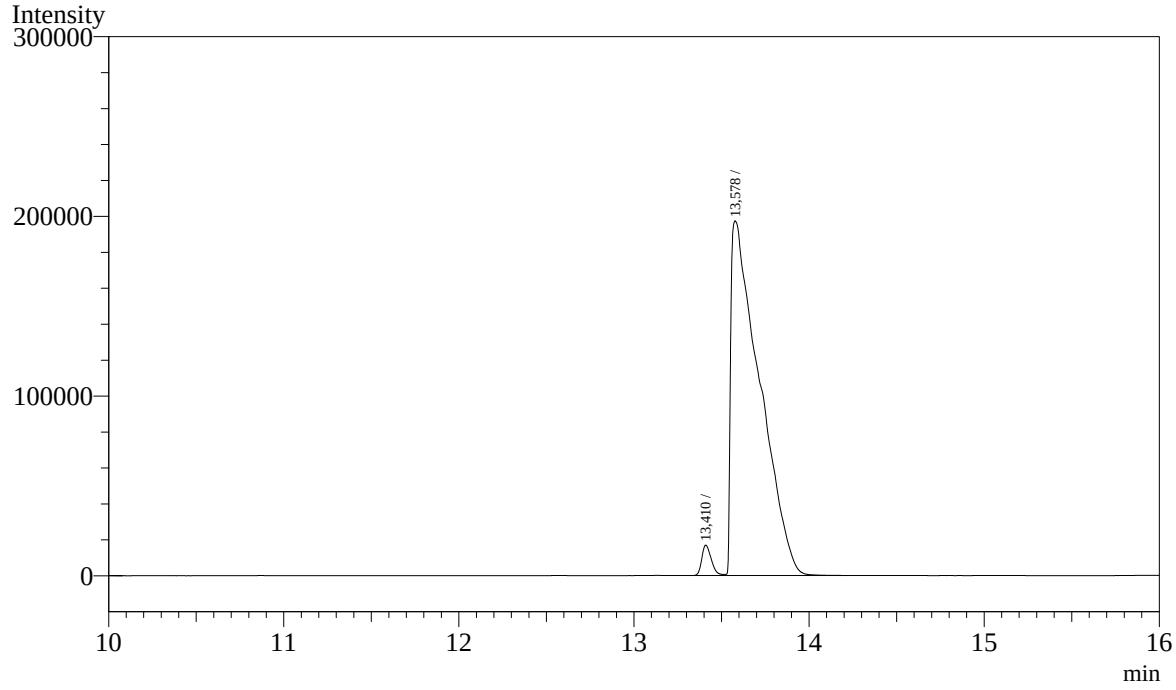

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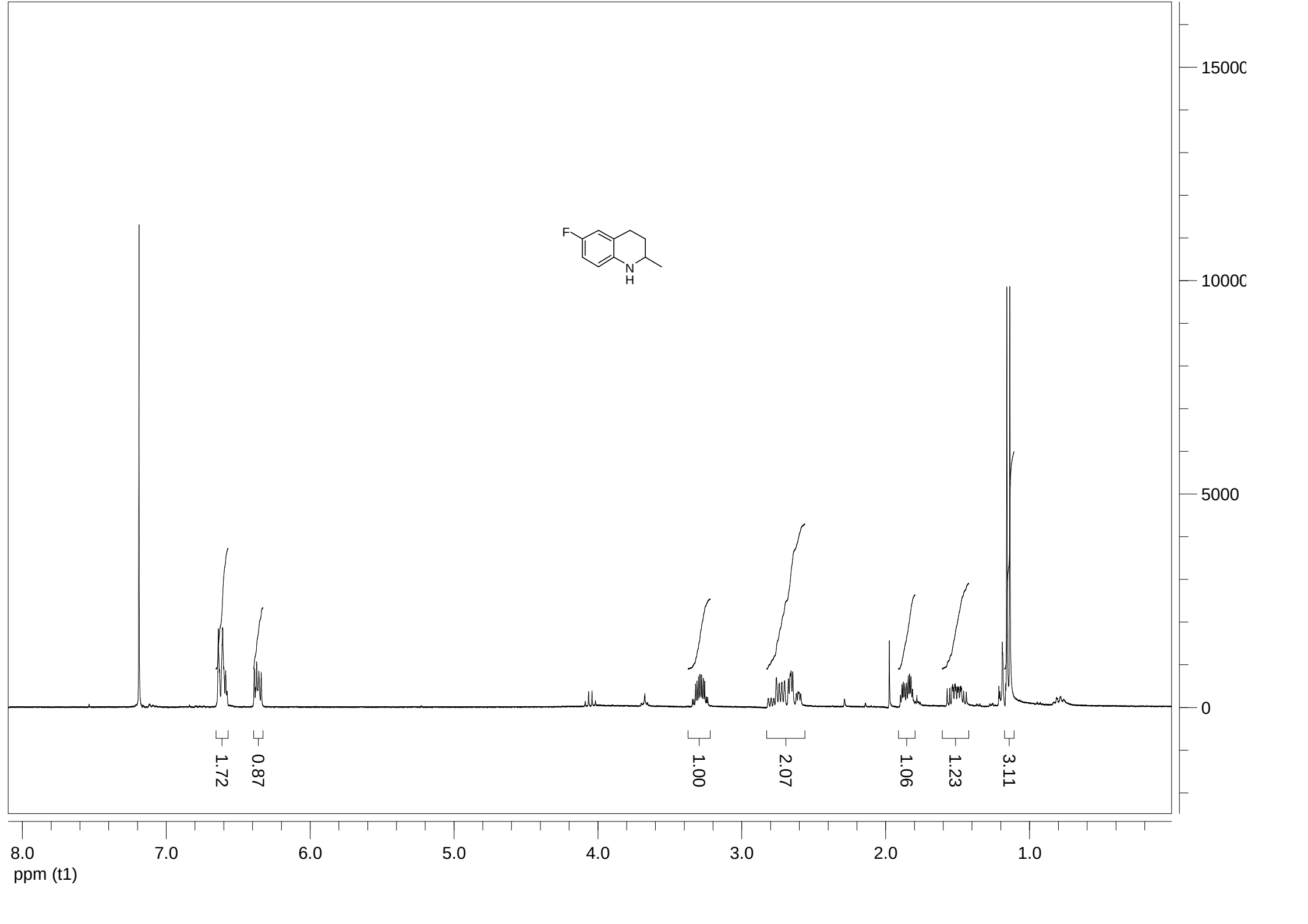
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H
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0
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0.87
1.00
2.07
1.06
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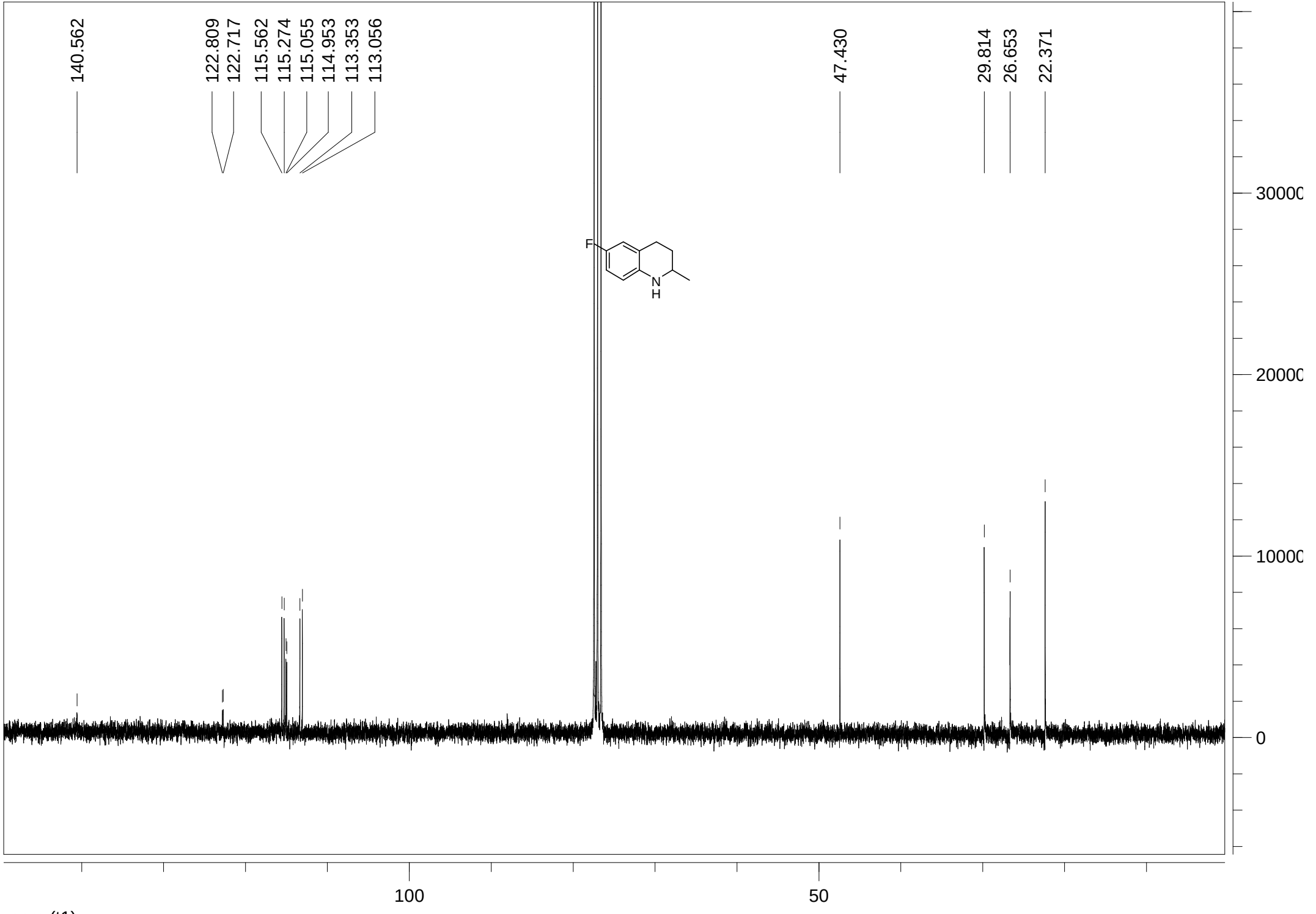
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)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122.809
122.717
115.562
115.274
115.055
114.953
113.353
113.056
47.430
29.814
26.653
22.371
F
N
H
30000
20000
10000
0
100
50
ppm (t1)

Analysis Date & Time : 28.08.2009 11:17:02
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Sample ID :
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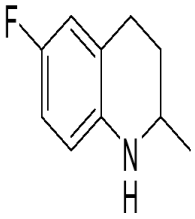
ISTD Amount :



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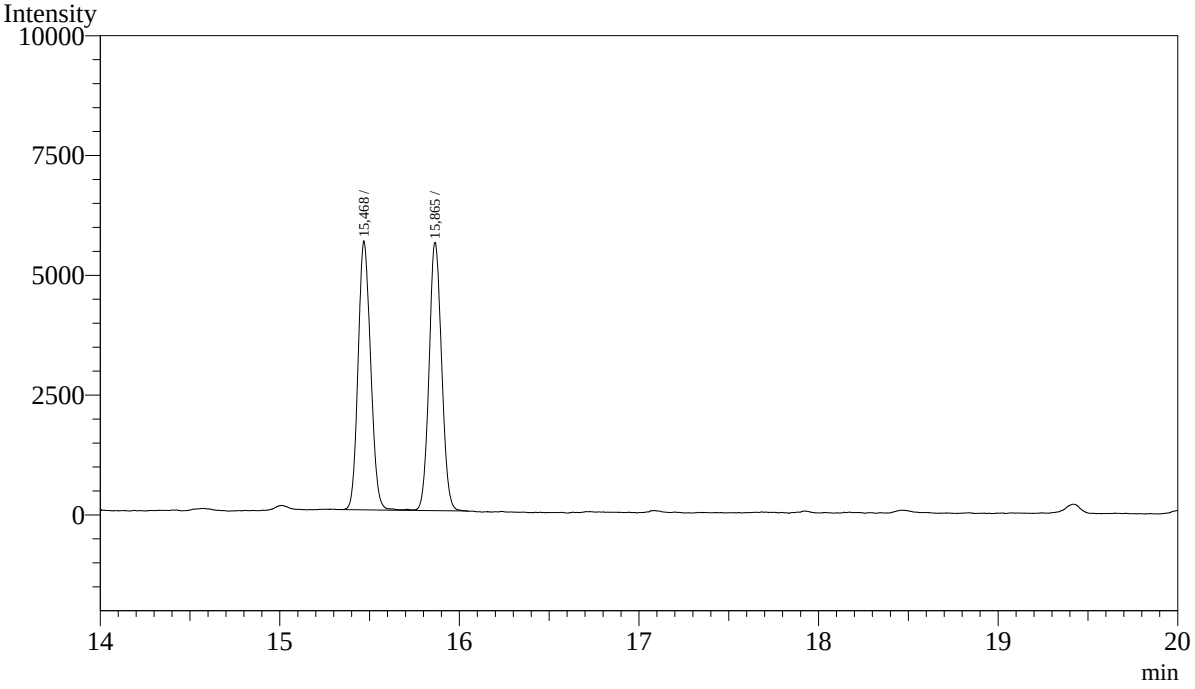

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Analysis Date & Time	: 15.09.2009 08:27:49
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Sample ID	:
Sample Type	: Unknown
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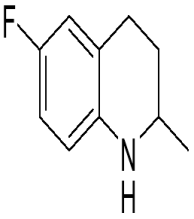
ISTD Amount	:



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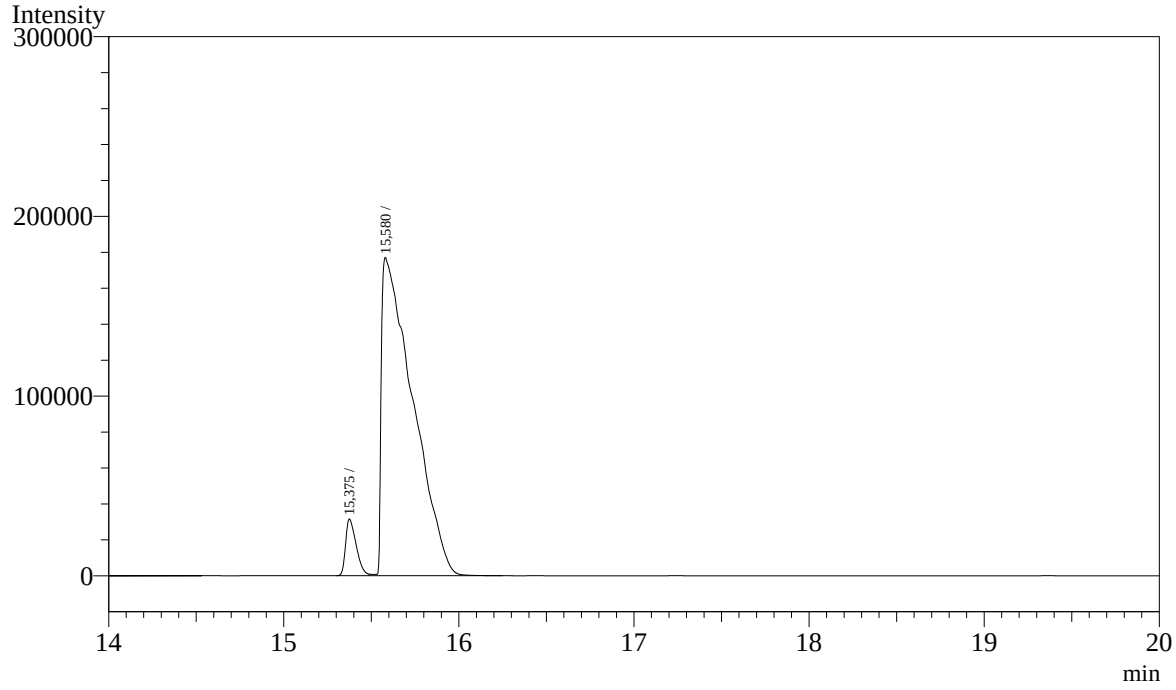

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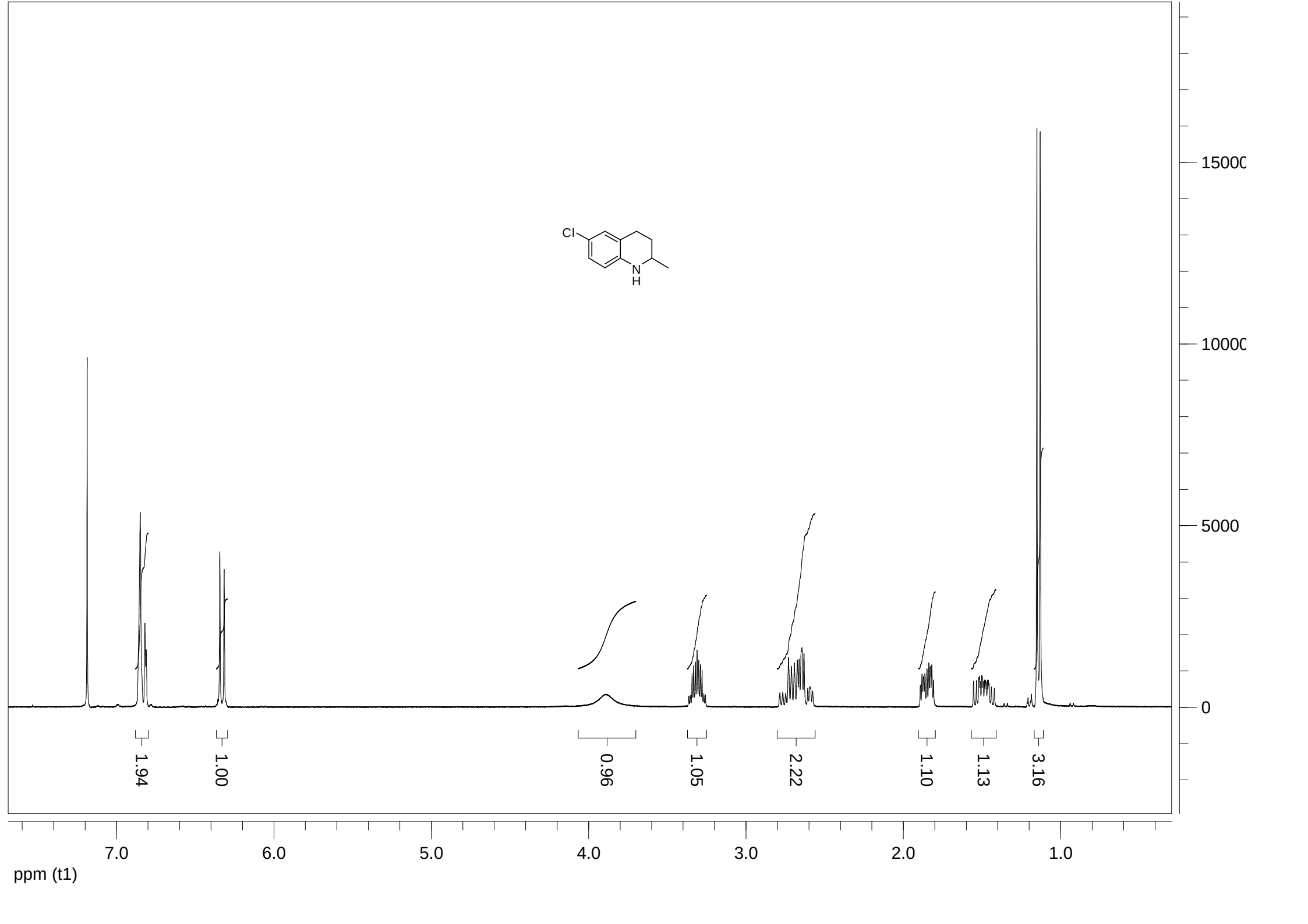
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H
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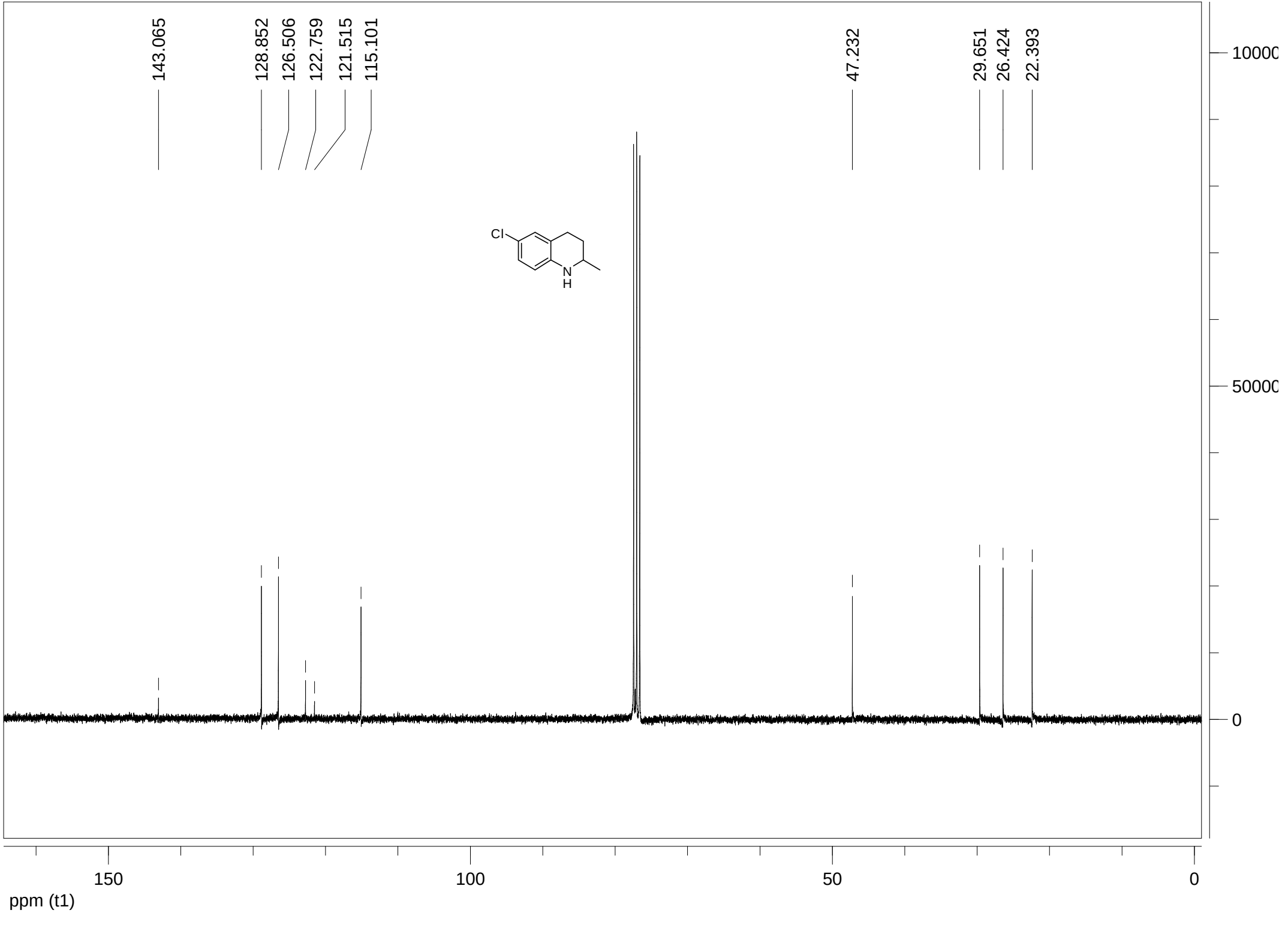
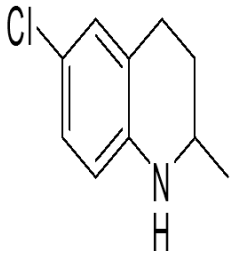
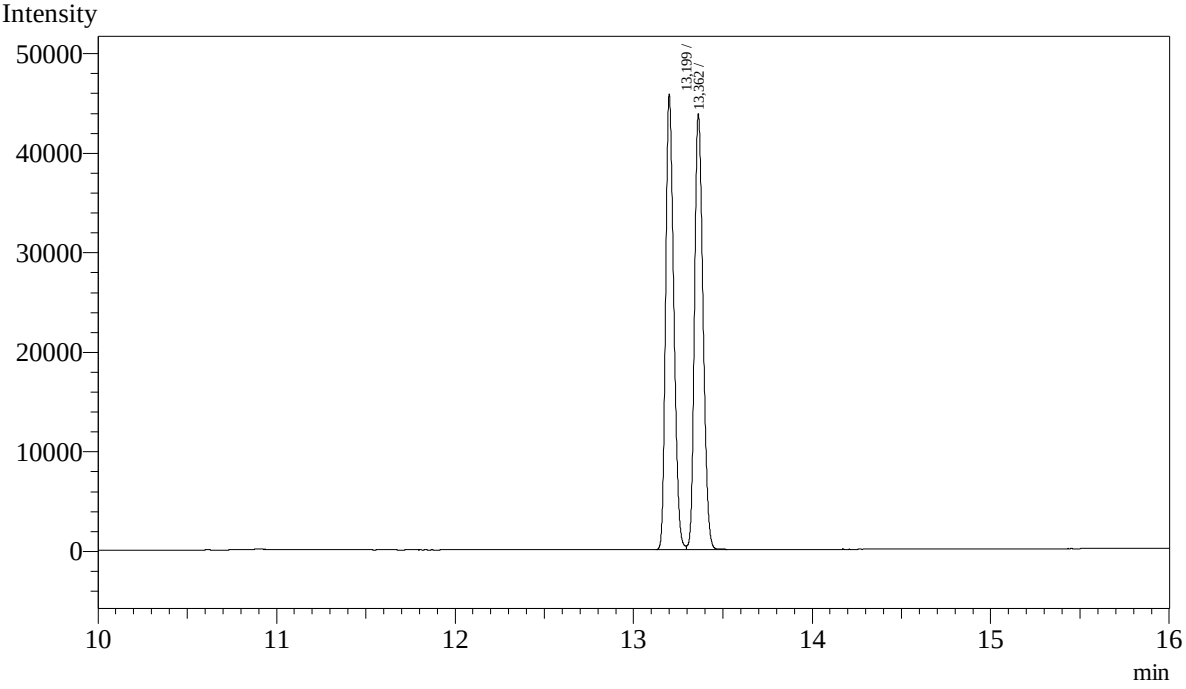
1.94
1.00
0.96
1.05
2.22
1.10
1.13
3.16
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


143.065
128.852
126.506
122.759
121.515
115.101
47.232
29.651
26.424
22.393
Cl
N
H
10000
50000
0
150
100
50
0
ppm (t1)

Analysis Date & Time : 26.01.2009 19:49:46
User Name : Admin
Vial# : 46
Sample Name : rk537-1
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

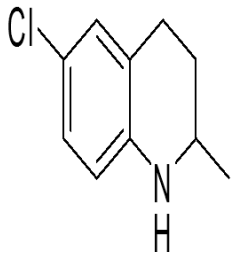


Data Name : D:\DATA_GC\rk\rk537-1.gcd
Method Name : D:\DATA_GC\rk\quinolines\rk-quinaldine-scope 1.gcm

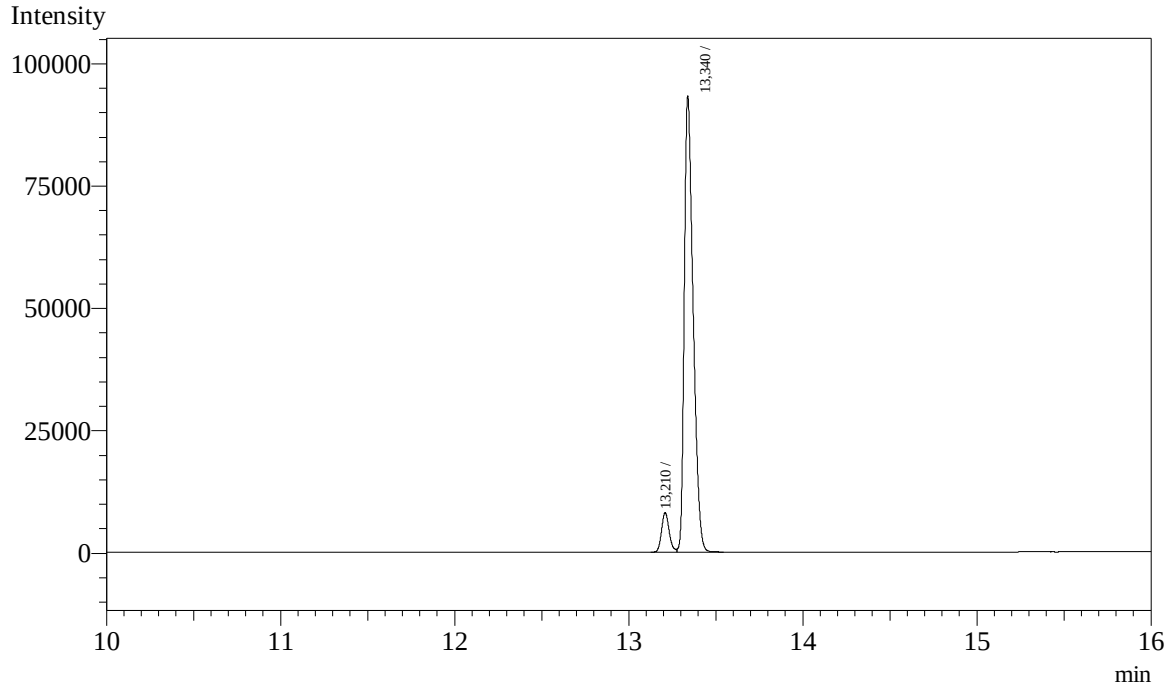


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	13,199	145420	45653	50,016				
2	13,362	145329	43634	49,984		V		
Total		290749	89287					

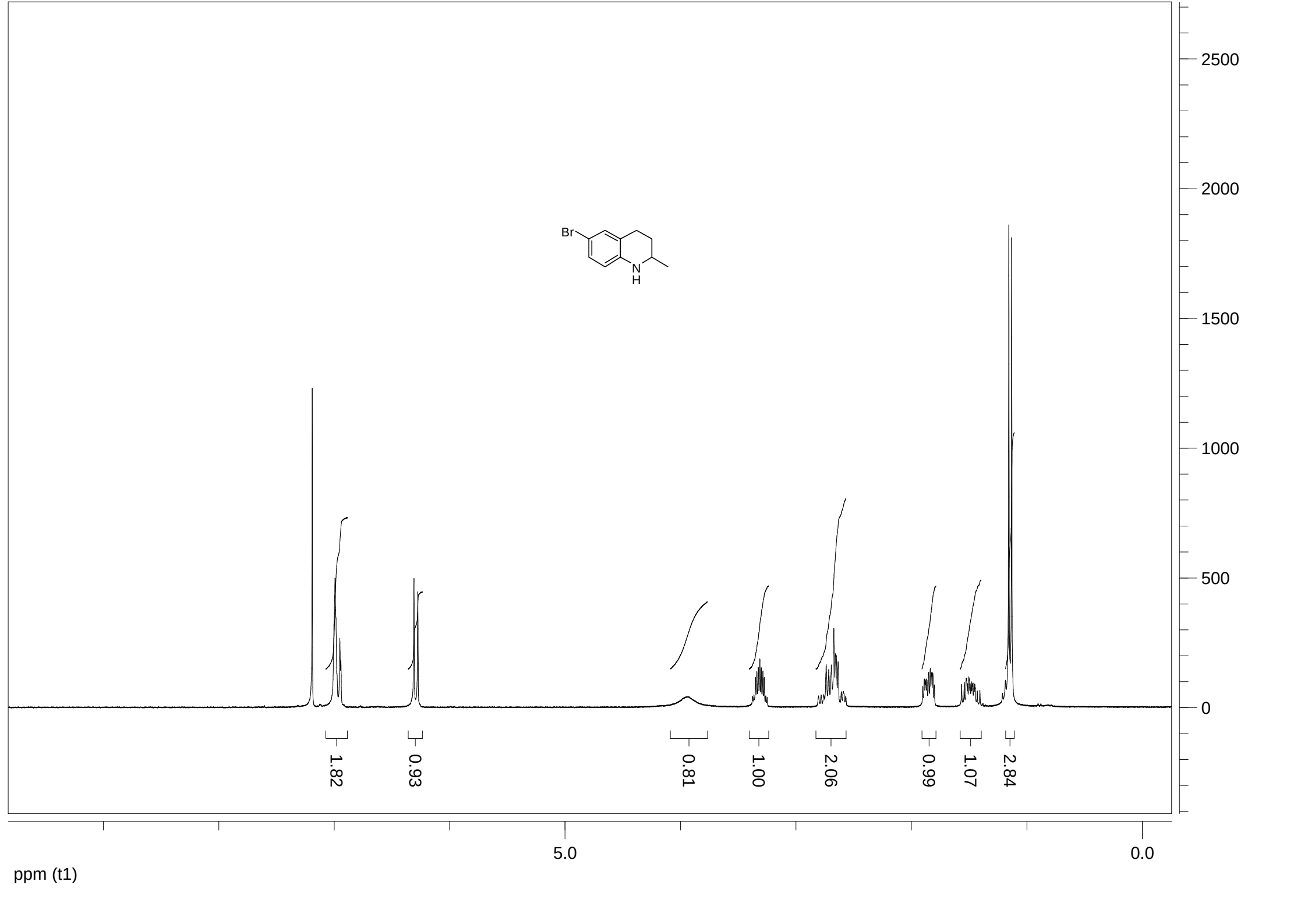
Analysis Date & Time : 10.02.2009 16:16:30
User Name : Admin
Vial# : 41
Sample Name : rk537-5
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

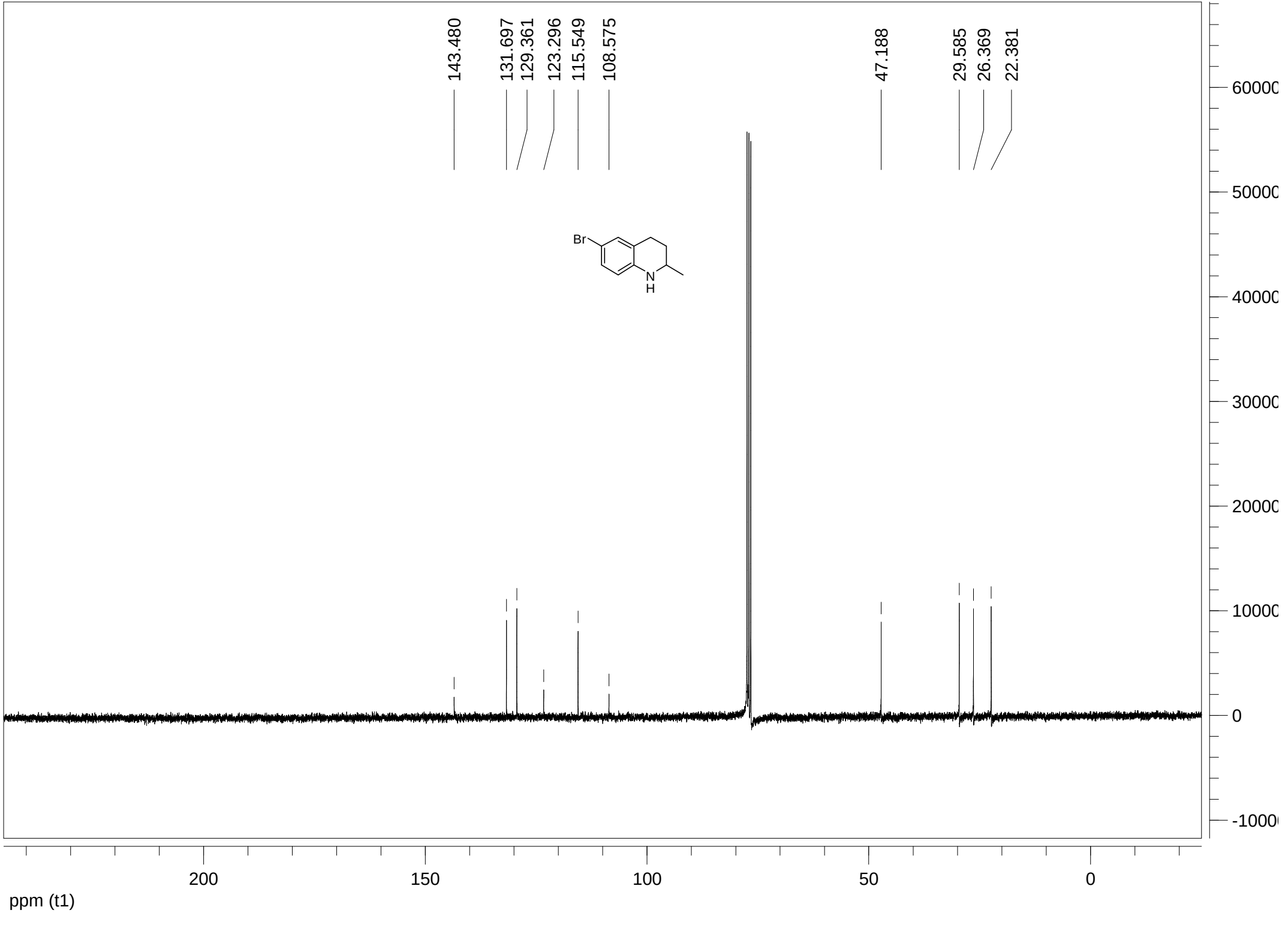


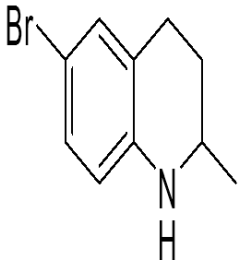
Data Name : D:\DATA_GC\rk\rk537-5.gcd
Method Name : D:\DATA_GC\rk\quinolines\rk-quinaldine-scope 1.gcm



Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	13,210	24470	8039	6,904				
2	13,340	329966	92984	93,096		V		
Total		354436	101023					

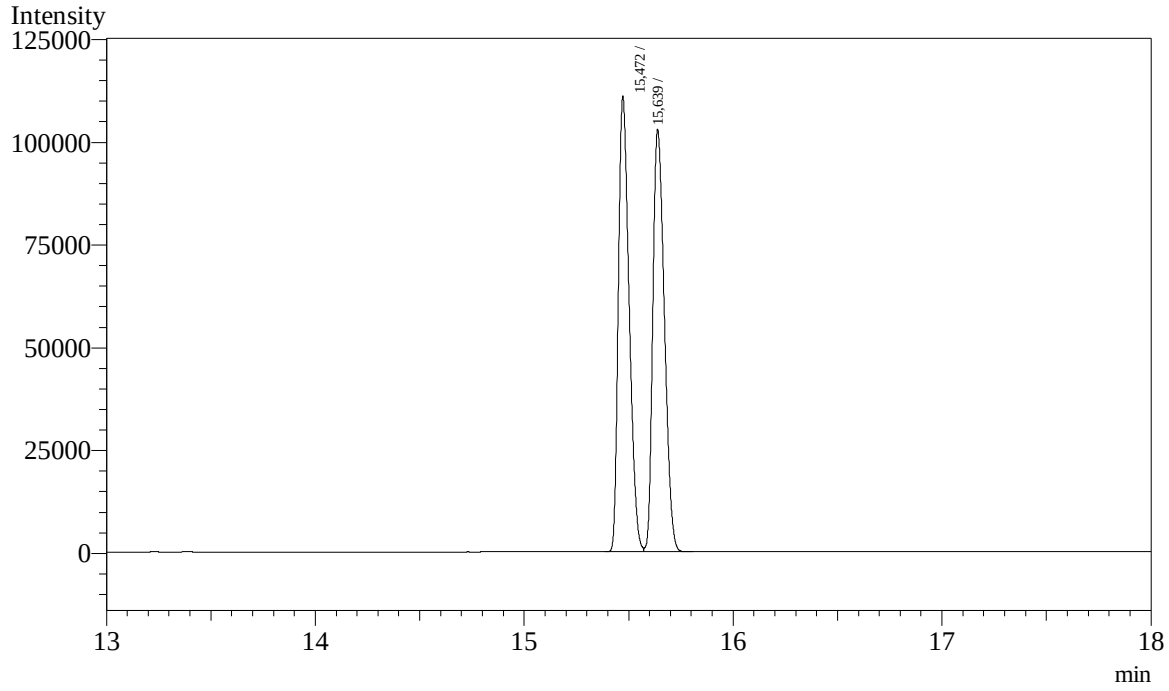

Br
N
H
2500
2000
1500
1000
500
0
1.82
0.93
0.81
1.00
2.06
0.99
1.07
2.84
5.0
0.0
ppm (t1)


143.480
131.697
129.361
123.296
115.549
108.575
47.188
29.585
26.369
22.381
Br
N
H
60000
50000
40000
30000
20000
10000
0
-10000
200
150
100
50
0
ppm (t1)

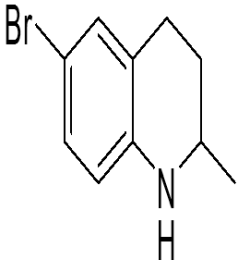


Analysis Date & Time : 26.01.2009 21:15:42
User Name : Admin
Vial# : 47
Sample Name : rk538-1
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

Data Name : D:\DATA_GC\rk\rk538-1.gcd
Method Name : D:\DATA_GC\rk\quinolines\rk-quinaldine-scope 1.gcm

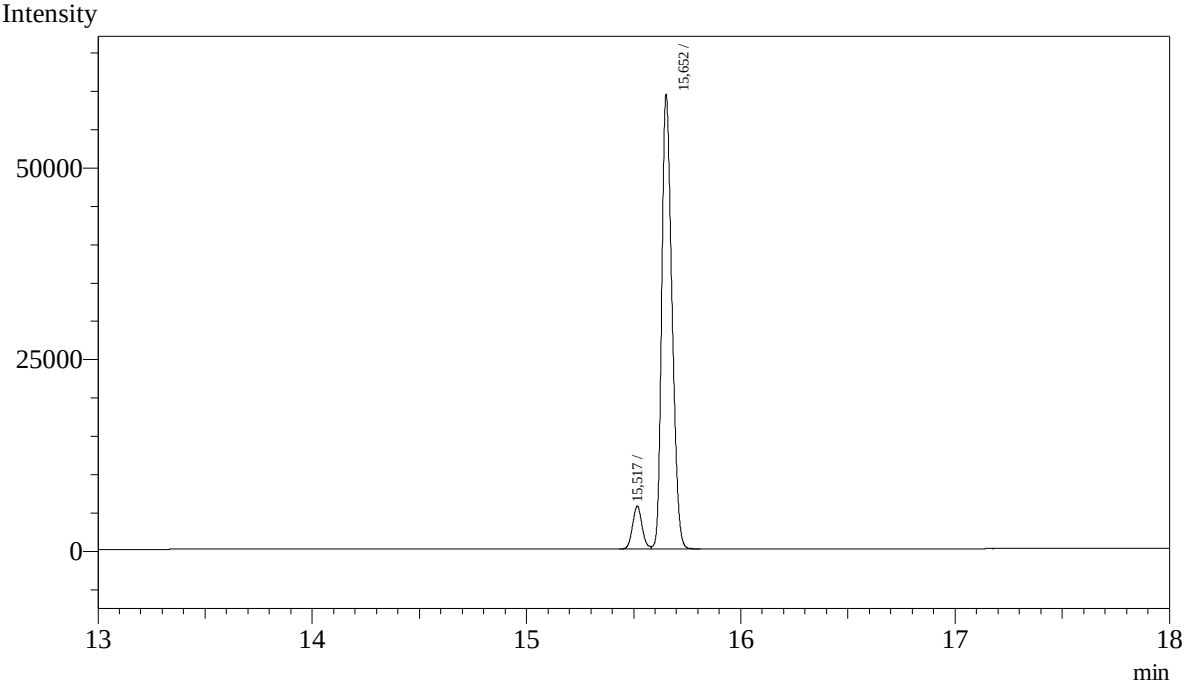


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	15,472	399102	110642	50,182				
2	15,639	396201	102290	49,818		V		
Total		795303	212932					

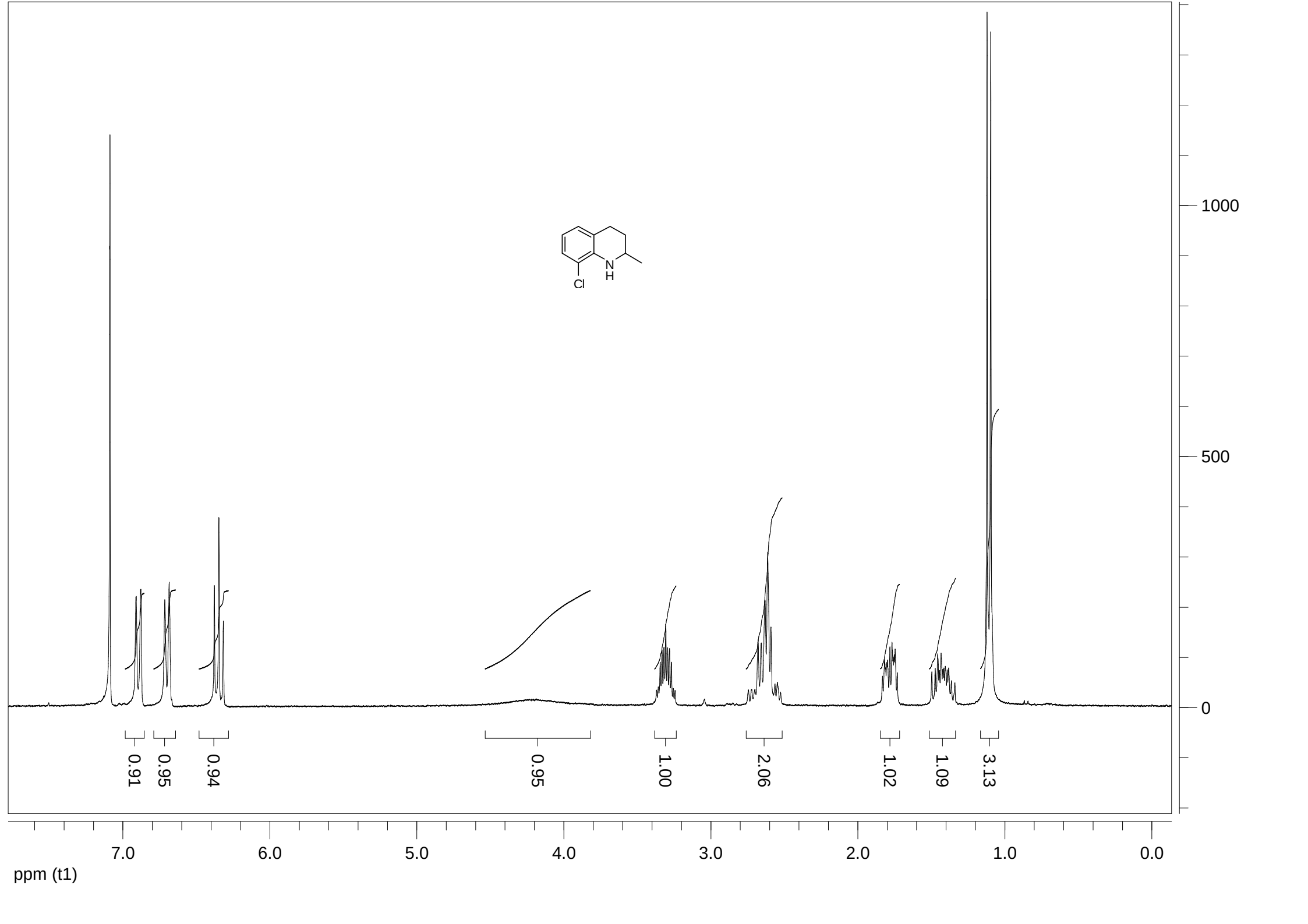


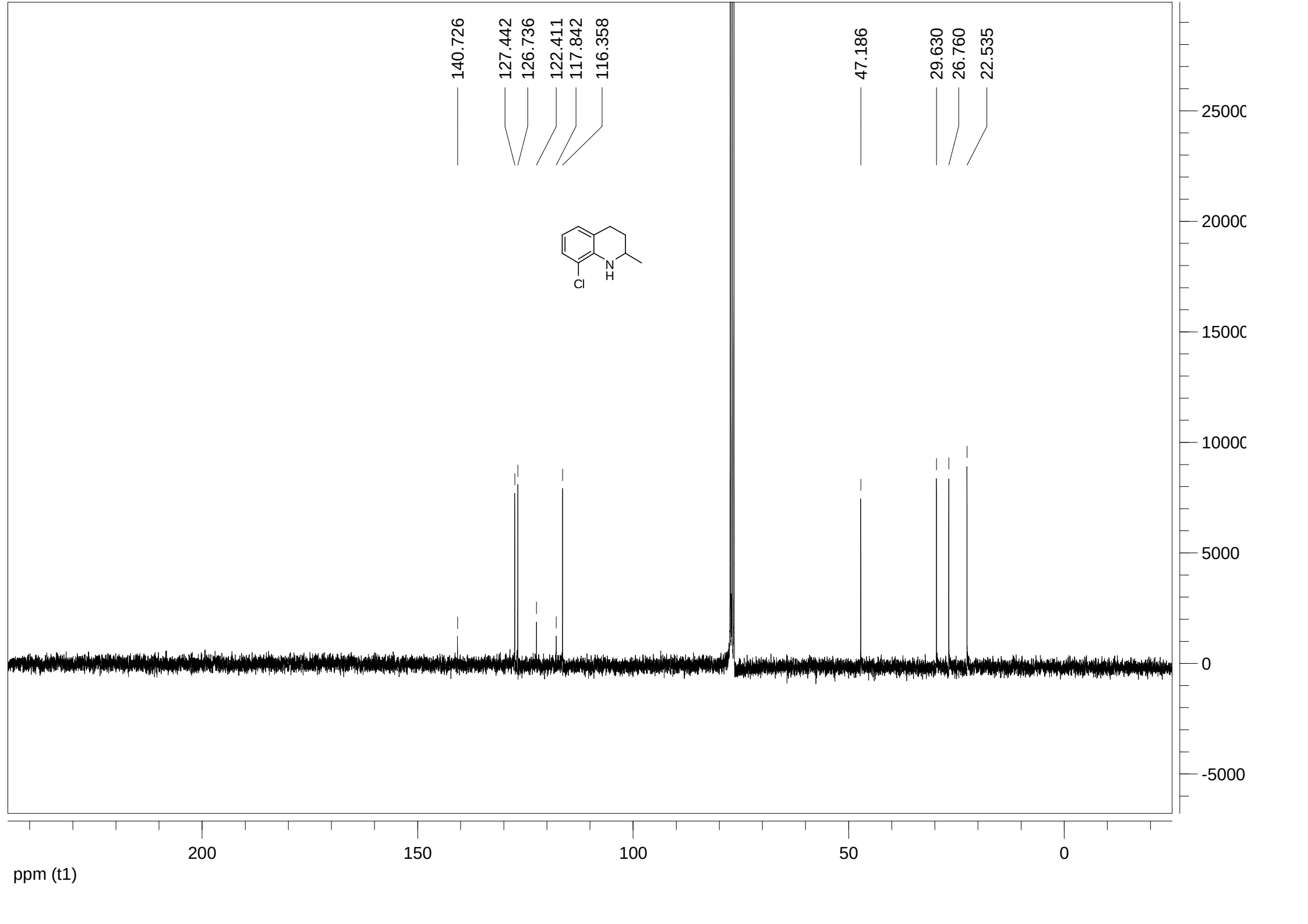
Analysis Date & Time : 10.02.2009 16:42:27
User Name : Admin
Vial# : 42
Sample Name : rk538-5
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

Data Name : D:\DATA_GC\rk\rk538-5.gcd
Method Name : D:\DATA_GC\rk\quinolines\rk-quinaldine-scope 1.gcm

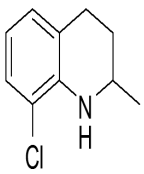


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	15,517	17202	5537	8,018				
2	15,652	197326	59067	91,982		V		
Total		214528	64604					

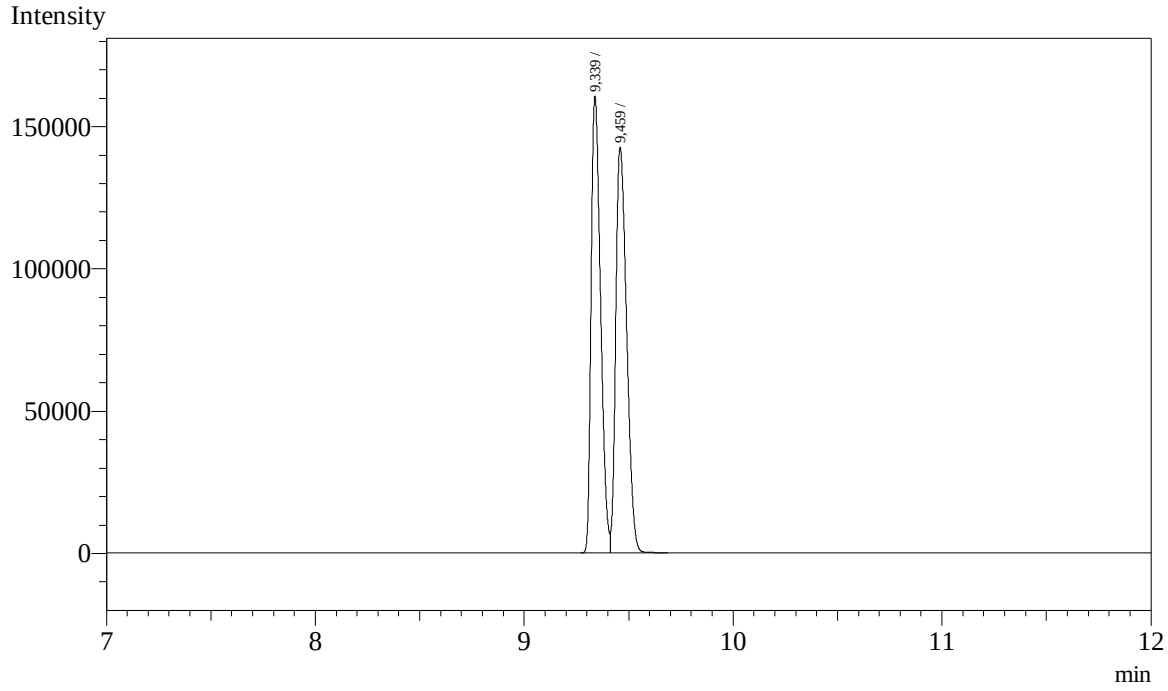

Cl
N
H
1000
500
0
0.91
0.95
0.94
0.95
1.00
2.06
1.02
1.09
3.13
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


140.726
127.442
126.736
122.411
117.842
116.358
47.186
29.630
26.760
22.535
Cl
N
H
25000
20000
15000
10000
5000
0
-5000
200
150
100
50
0
ppm (t1)

Analysis Date & Time : 26.01.2009 20:41:39
User Name : Admin
Vial# : 48
Sample Name : rk540-1
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

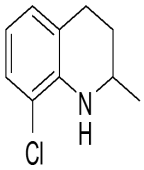


Data Name : D:\DATA_GC\rk\rk540-1.gcd
Method Name : D:\DATA_GC\rk\quinolines\rk-quinaldine-scope 1.gcm

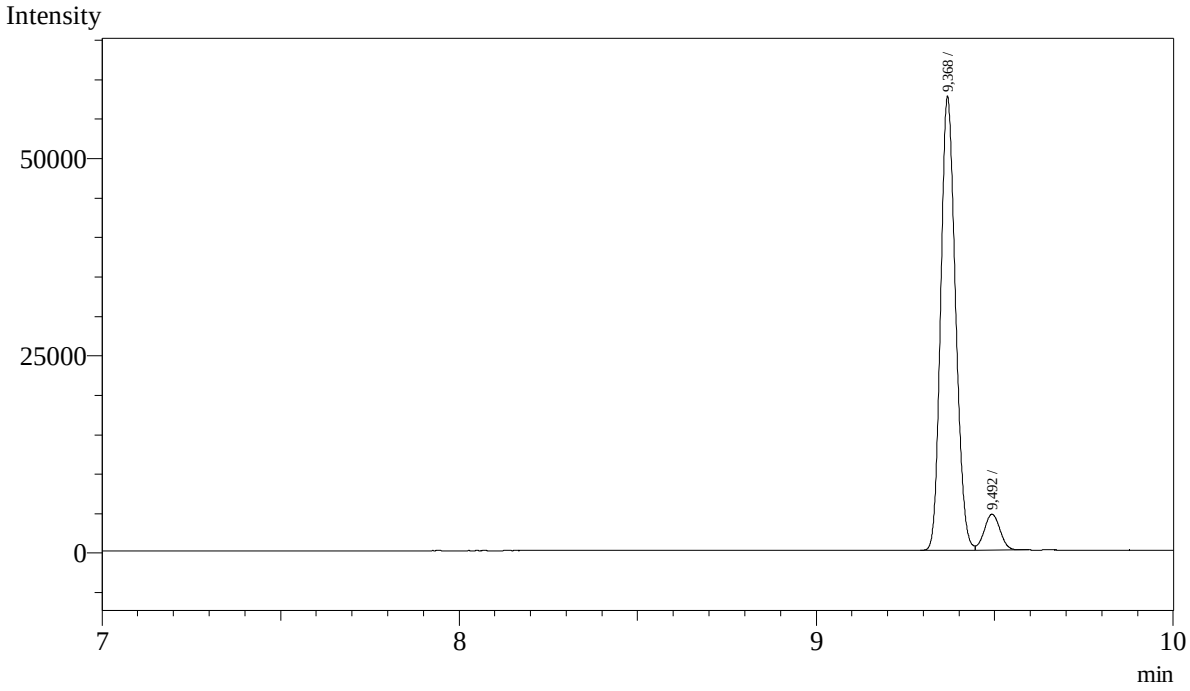


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	9,339	505264	159822	49,548				
2	9,459	514483	141946	50,452		V		
Total		1019747	301768					

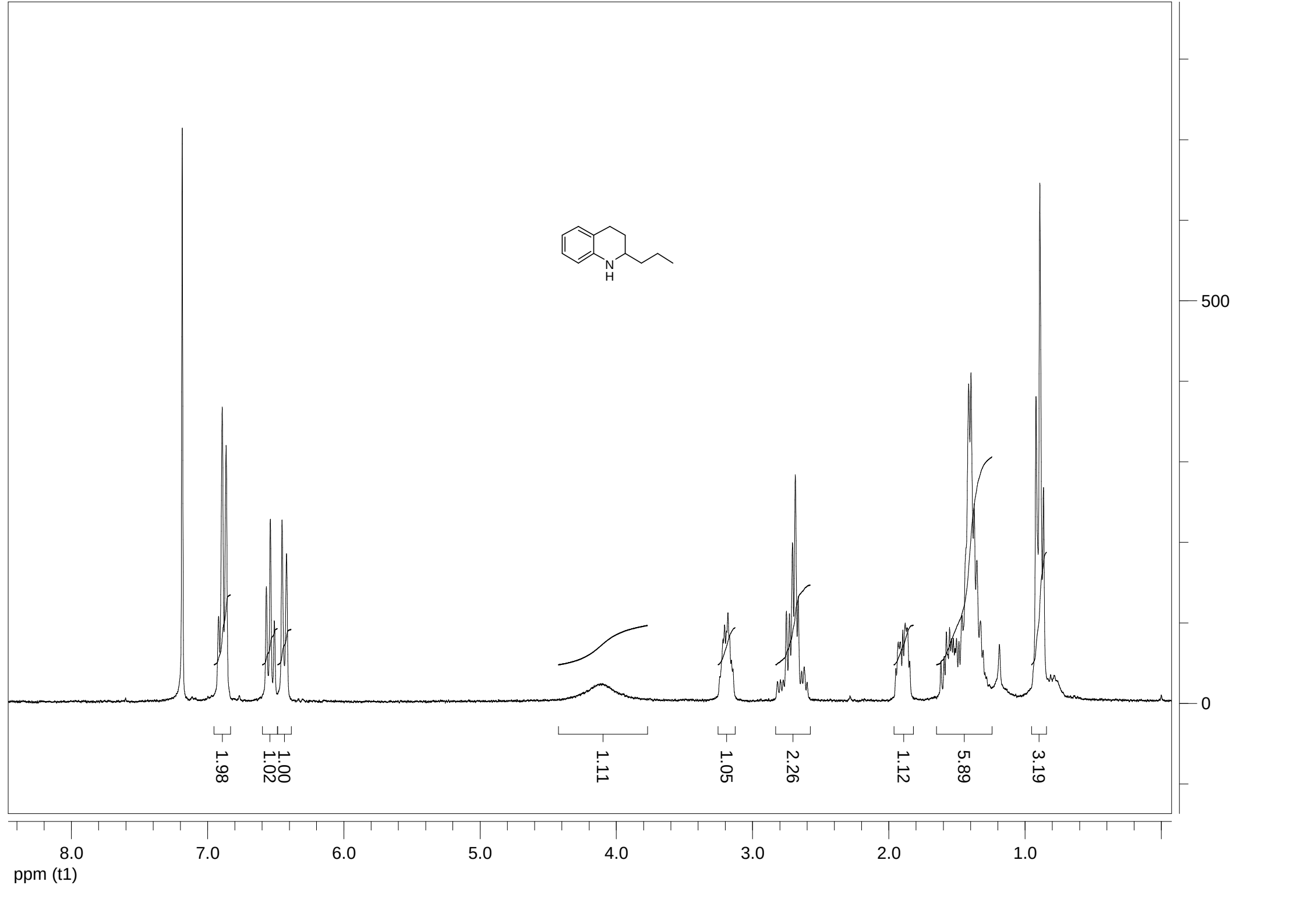
Analysis Date & Time : 10.02.2009 16:08:26
User Name : Admin
Vial# : 43
Sample Name : rk540-4
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

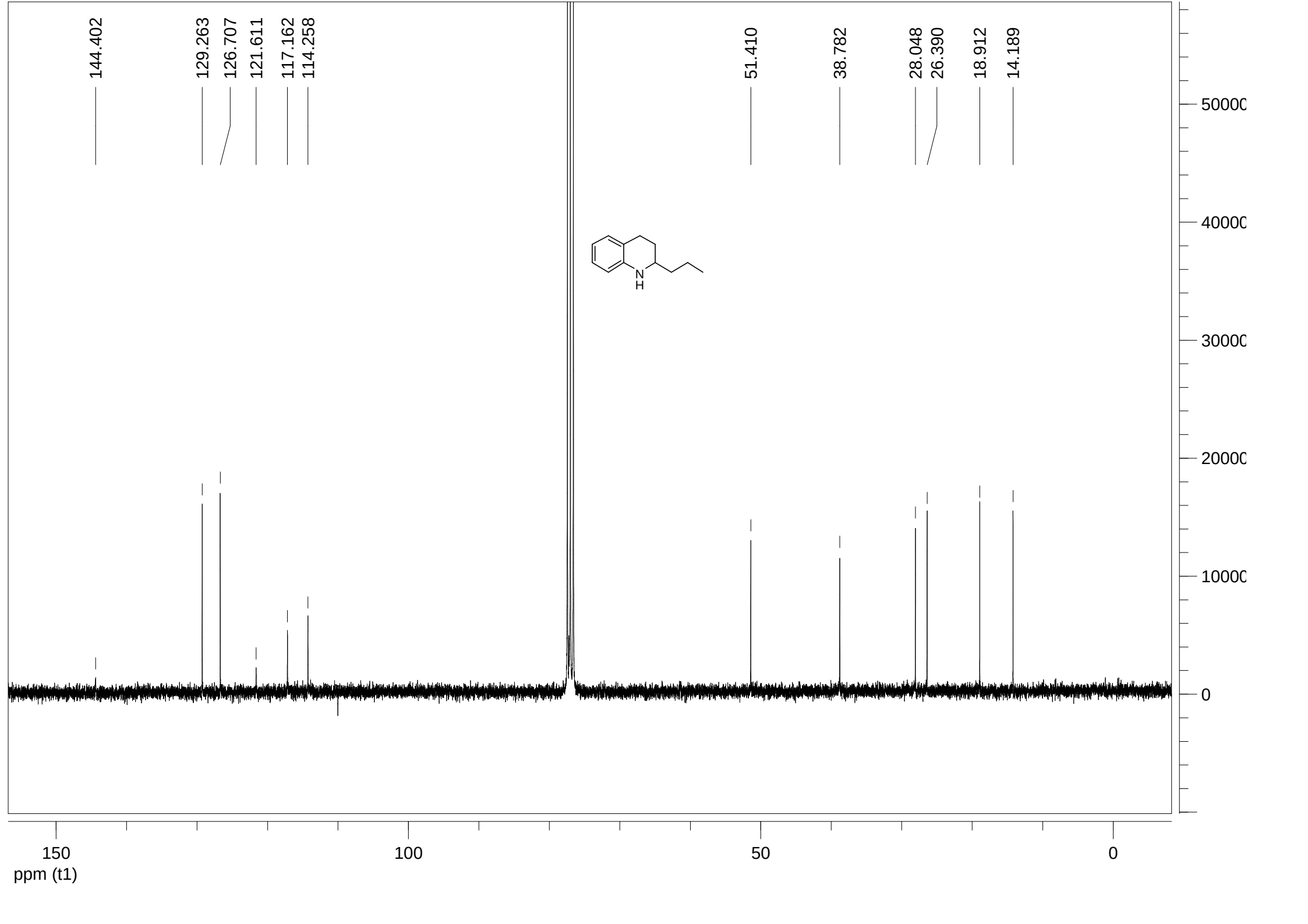


Data Name : D:\DATA_GC\rk\rk540-4.gcd
Method Name : D:\DATA_GC\rk\quinolines\rk-quinaldine-scope 1.gcm

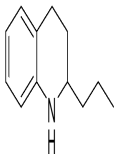


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	9,368	166135	57184	92,222				
2	9,492	14011	4534	7,778		V		
Total		180146	61718					

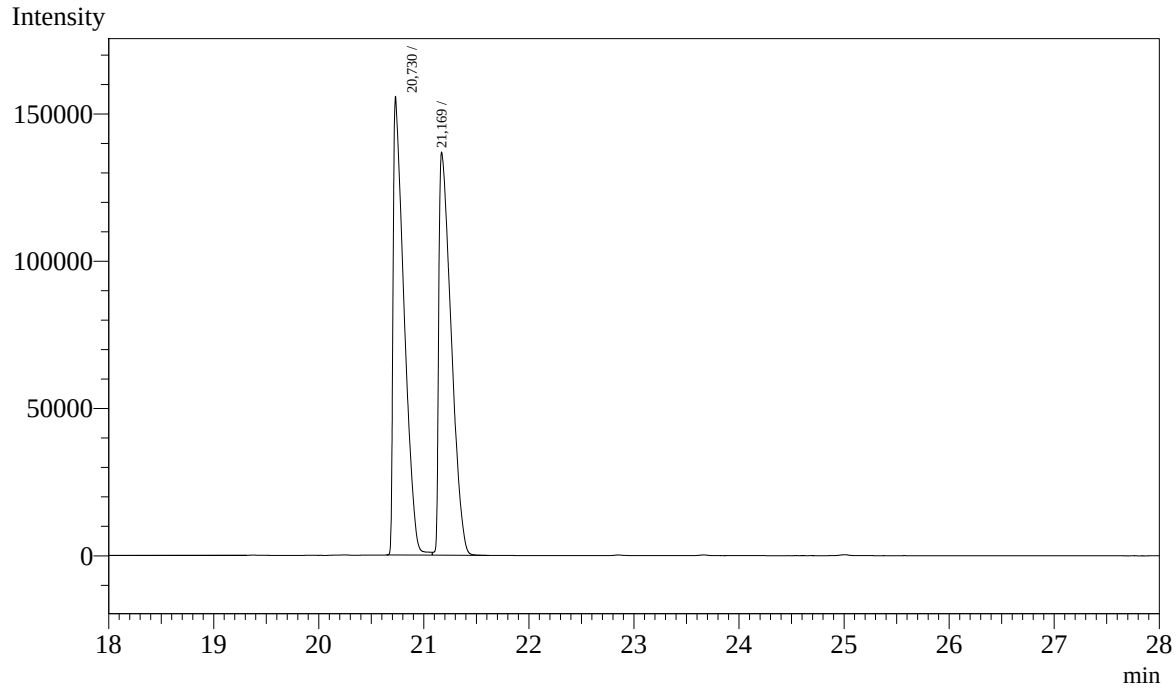

N
H
500
0
1.98
1.02
1.00
1.11
1.05
2.26
1.12
5.89
3.19
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


144.402
129.263
126.707
121.611
117.162
114.258
51.410
38.782
28.048
26.390
18.912
14.189
N
H
50000
40000
30000
20000
10000
0
150
100
50
0
ppm (t1)

Analysis Date & Time : 17.09.2009 11:11:54
User Name : Admin
Vial# : 13
Sample Name : rk_pr_rac
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

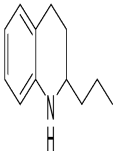


Data Name : F:\data shimadzu chiral gc\rk\quinoline project ac\rk_npr_rac.gcd
Method Name : F:\data shimadzu chiral gc\rk\quinaldine_6cl.gcm

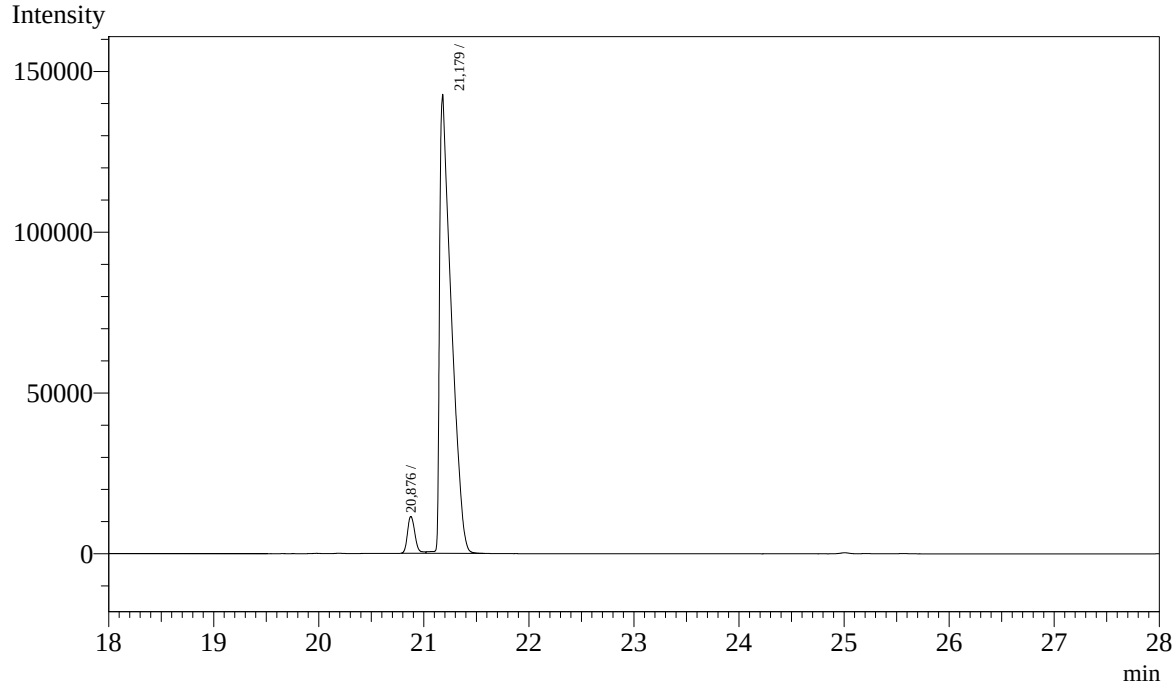


Peak#	Ret.Time	Area	Height	Area%
1	20,730	1138473	155243	50,3049
2	21,169	1124673	136647	49,6951
Total		2263146	291890	100,0000

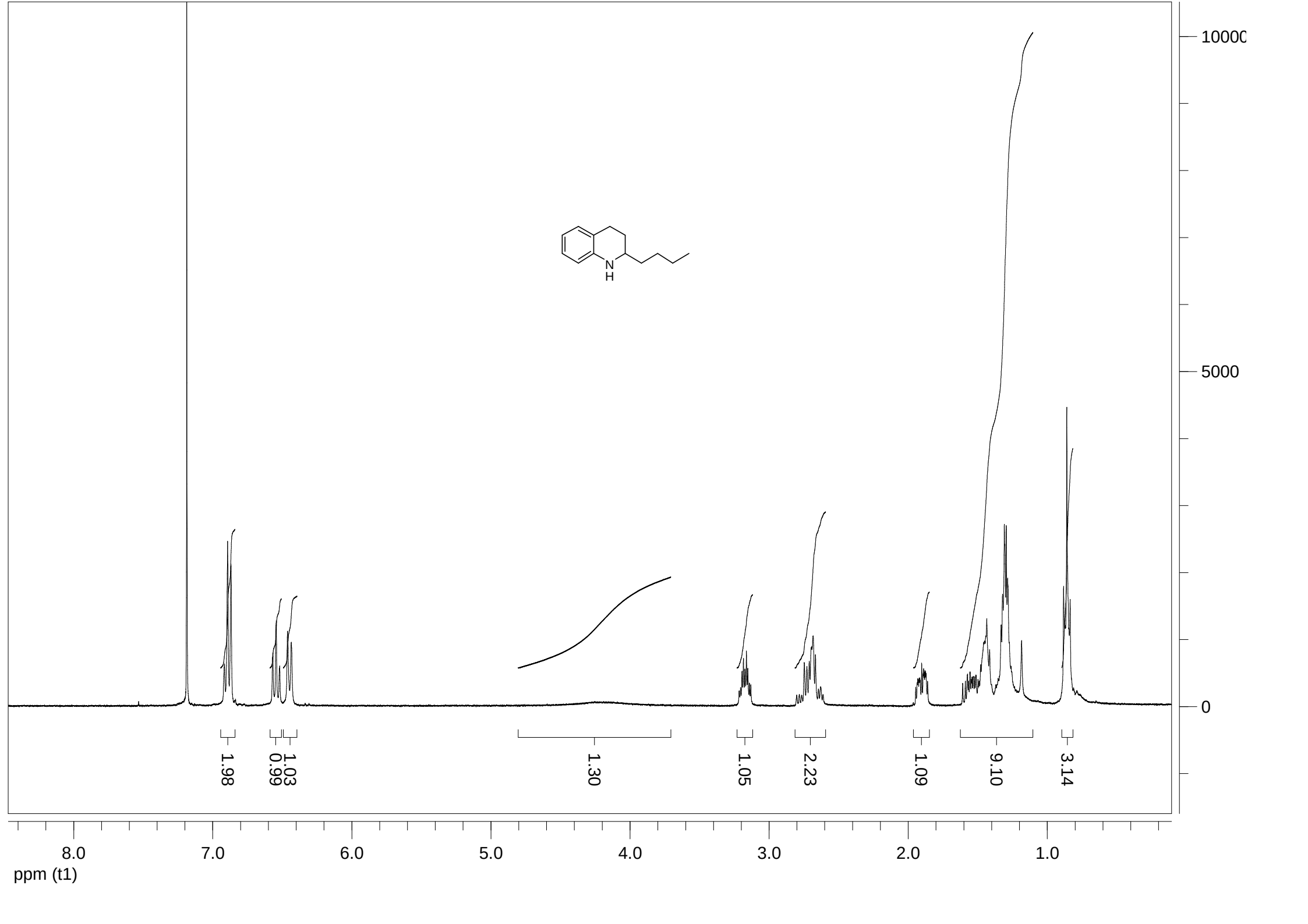
Analysis Date & Time : 17.09.2009 12:40:50
User Name : Admin
Vial# : 15
Sample Name : rk_648_bu_
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

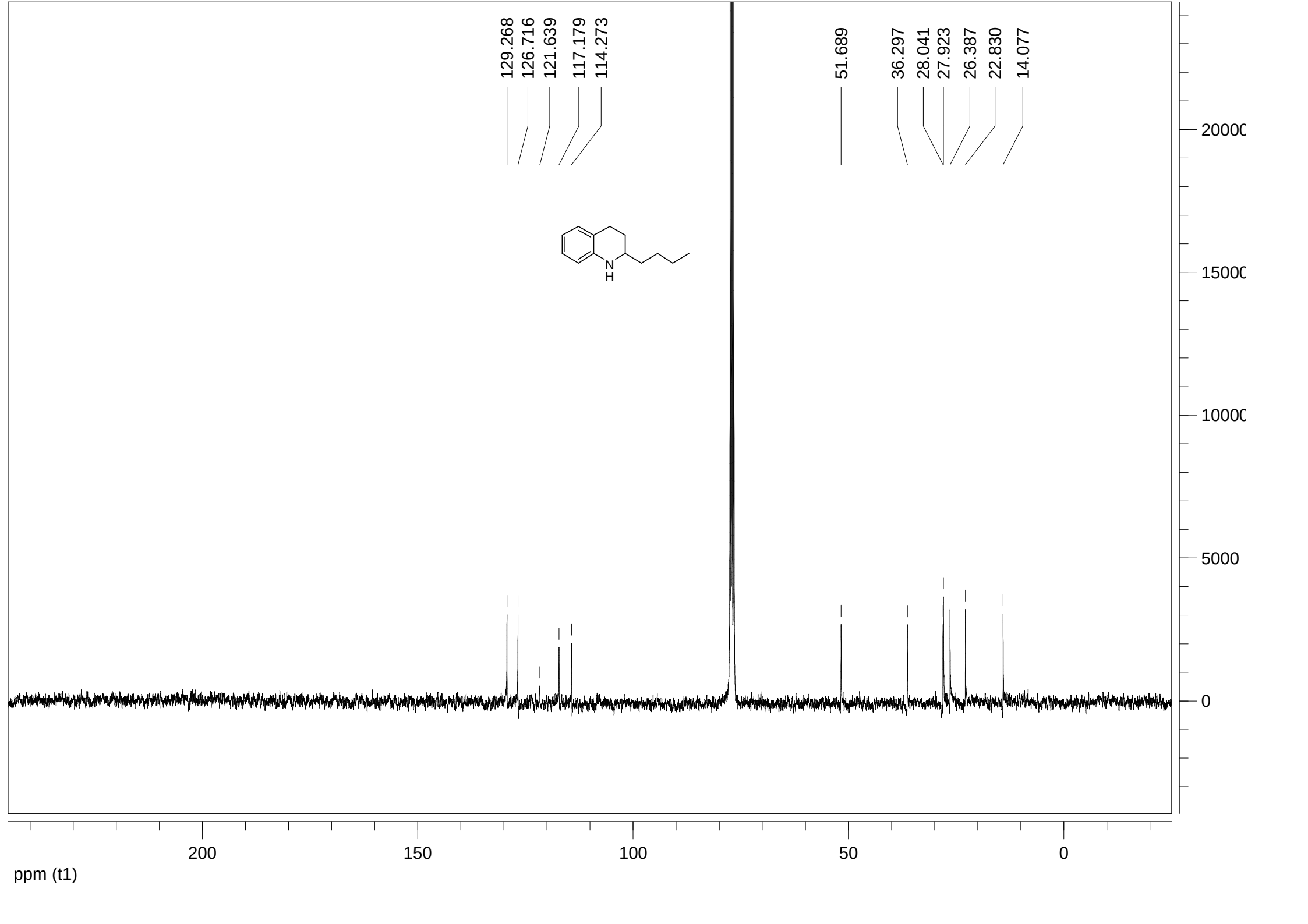


Data Name : F:\data shimadzu chiral gc\rk\quinoline project ac\rk_648_nPr_.gcd
Method Name : F:\data shimadzu chiral gc\rk\quinaldine_6cl.gcm

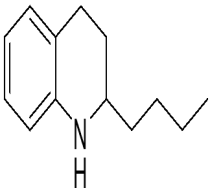


Peak#	Ret.Time	Area	Height	Area%
1	20,876	56286	11456	4,8849
2	21,179	1095968	142648	95,1151
Total		1152254	154104	100,0000

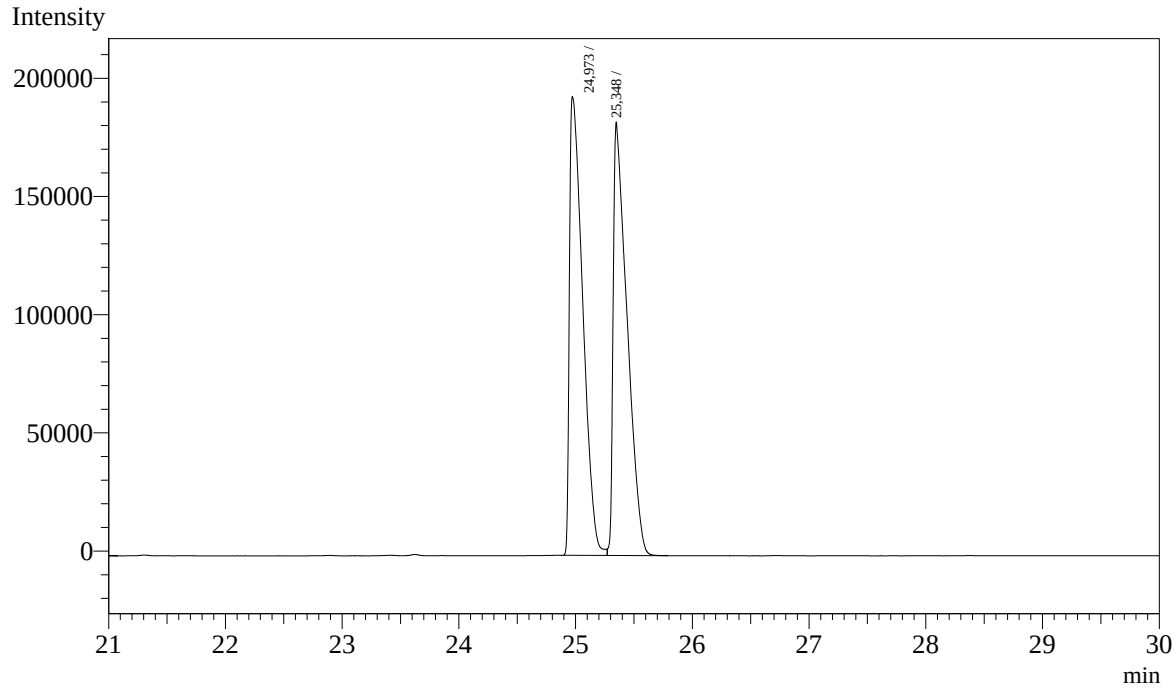

10000
5000
0
1.98
0.99
1.03
1.30
1.05
2.23
1.09
9.10
3.14
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


129.268
126.716
121.639
117.179
114.273
51.689
36.297
28.041
27.923
26.387
22.830
14.077
N
H
20000
15000
10000
5000
0
200
150
100
50
0
ppm (t1)

Analysis Date & Time	: 22.09.2009 11:34:48
User Name	: Admin
Vial#	: 31
Sample Name	: rk_nBu_rac
Sample ID	:
Sample Type	: Unknown
Injection Volume	: 1,00
ISTD Amount	:

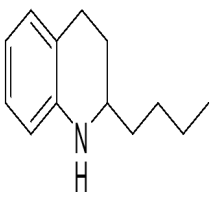


Data Name	: F:\data shimadzu chiral gc\rk\rk_nBu_rac-dil.gcd
Method Name	: F:\data shimadzu chiral gc\rk\quinaldine_6cl.gcm

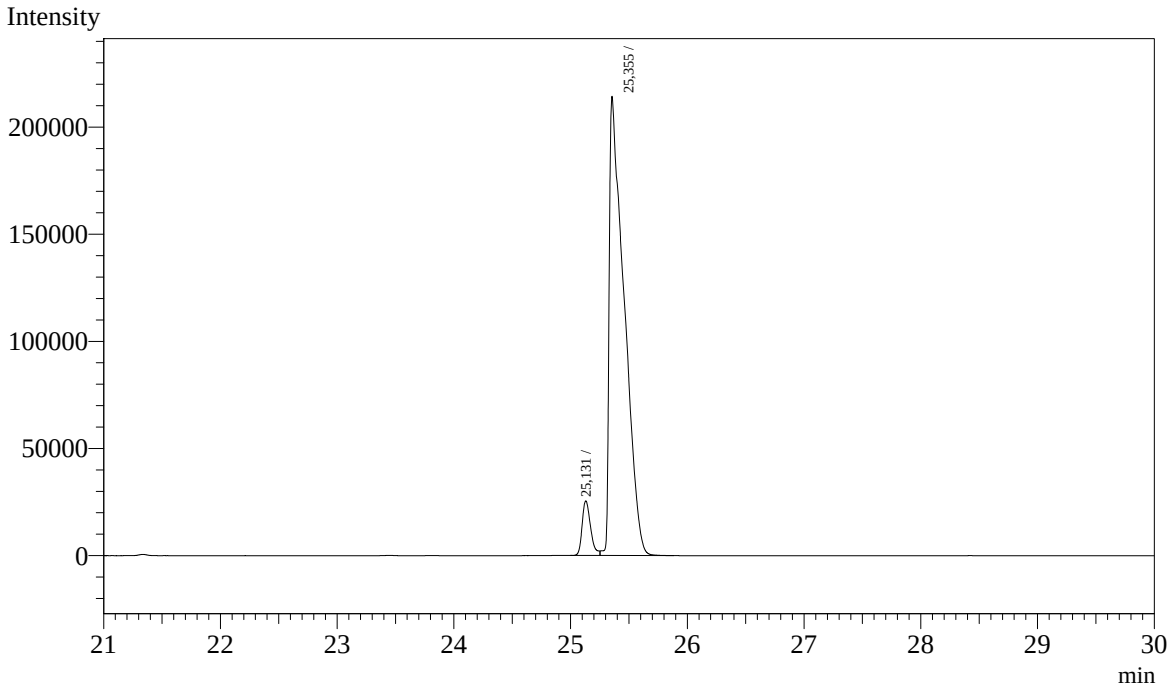


Peak#	Ret.Time	Area	Height	Area%
1	24,973	1540361	193895	50,3401
2	25,348	1519547	181681	49,6599
Total		3059908	375576	100,0000

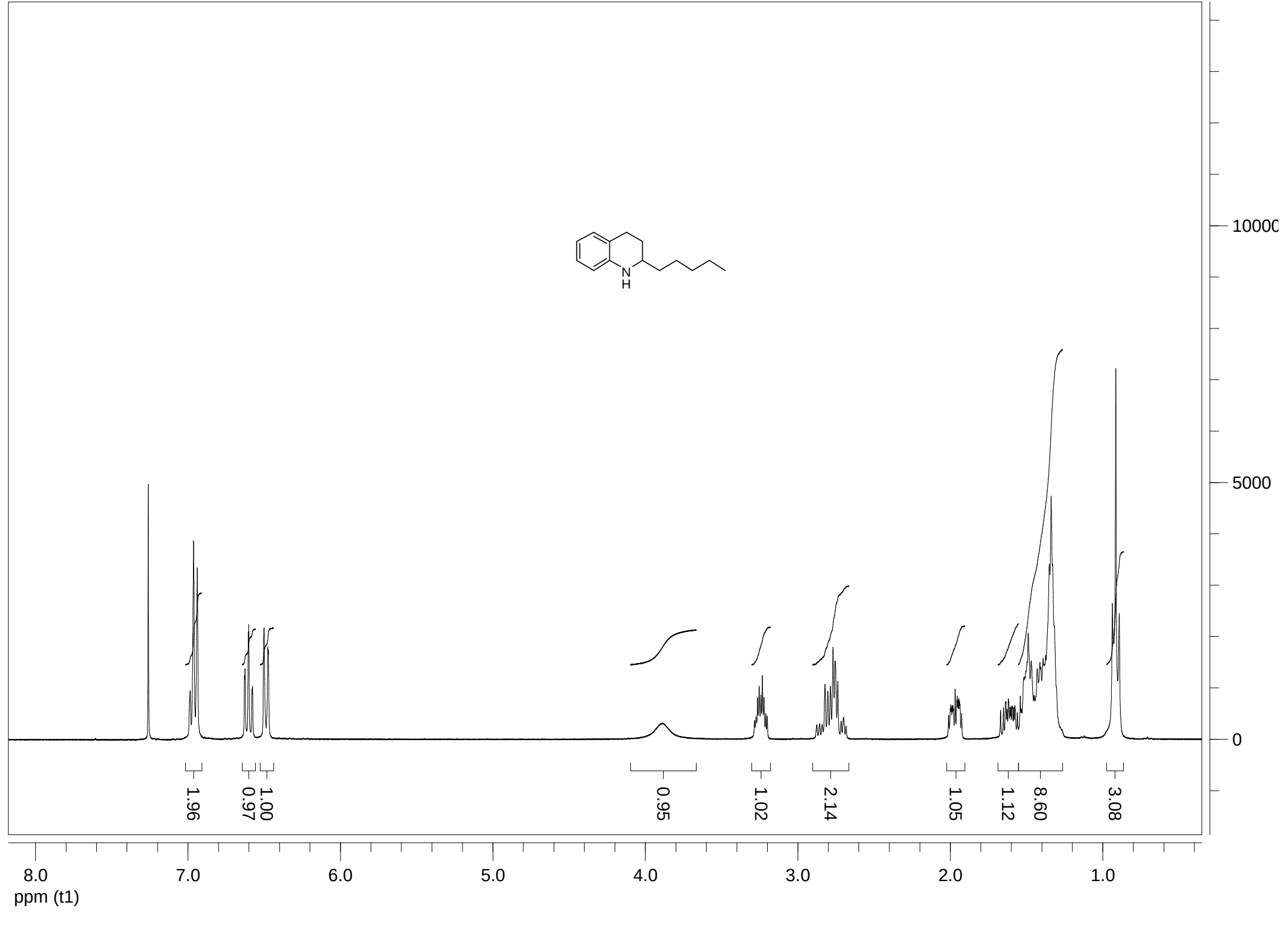
Analysis Date & Time : 17.09.2009 15:17:30
User Name : Admin
Vial# : 16
Sample Name : rk_648_nBu_dil
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

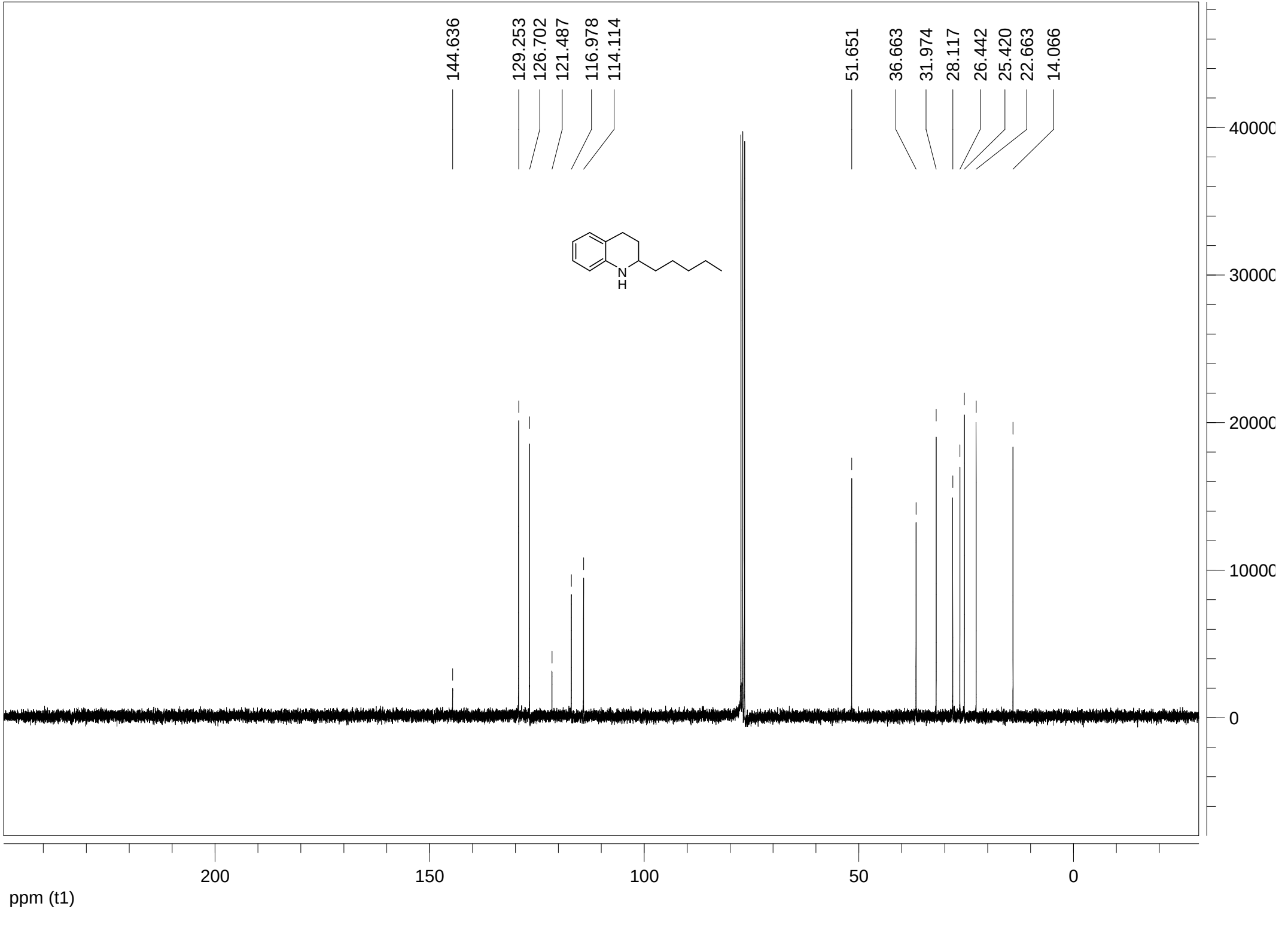


Data Name : F:\data shimadzu chiral gc\rk\quinoline project ac\rk_648_nBu_dil.gcd
Method Name : F:\data shimadzu chiral gc\rk\quinaldine_6cl.gcm

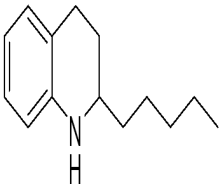


Peak#	Ret.Time	Area	Height	Area%
1	25,131	128615	25400	6,3042
2	25,355	1911542	213967	93,6958
Total		2040157	239367	100,0000


N
H
10000
5000
0
1.96
0.97
1.00
0.95
1.02
2.14
1.05
1.12
8.60
3.08
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

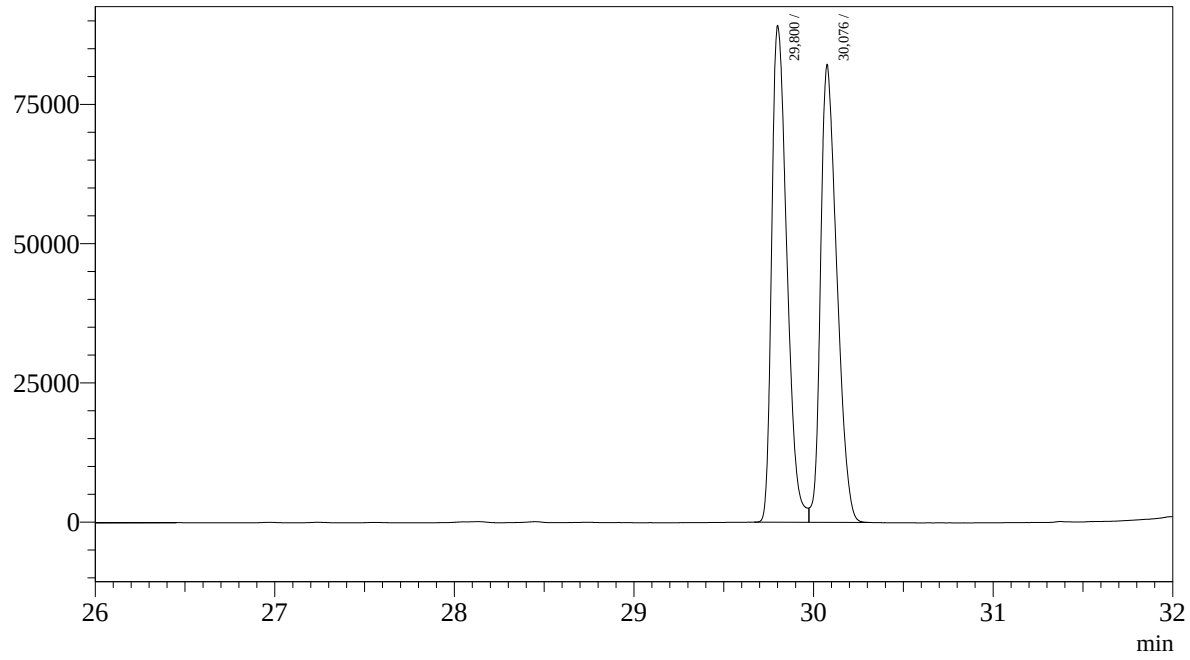

144.636
129.253
126.702
121.487
116.978
114.114
51.651
36.663
31.974
28.117
26.442
25.420
22.663
14.066
N
H
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

Analysis Date & Time	: 17.09.2009 09:42:56
User Name	: Admin
Vial#	: 11
Sample Name	: rk_pent_rac
Sample ID	:
Sample Type	: Unknown
Injection Volume	: 1,00
ISTD Amount	:



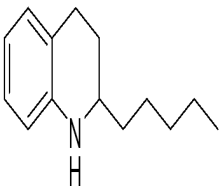
Data Name	: F:\data shimadzu chiral gc\rk\quinoline project ac\rk_npent_rac.gcd
Method Name	: F:\data shimadzu chiral gc\rk\quinaldine_6cl.gcm

Intensity

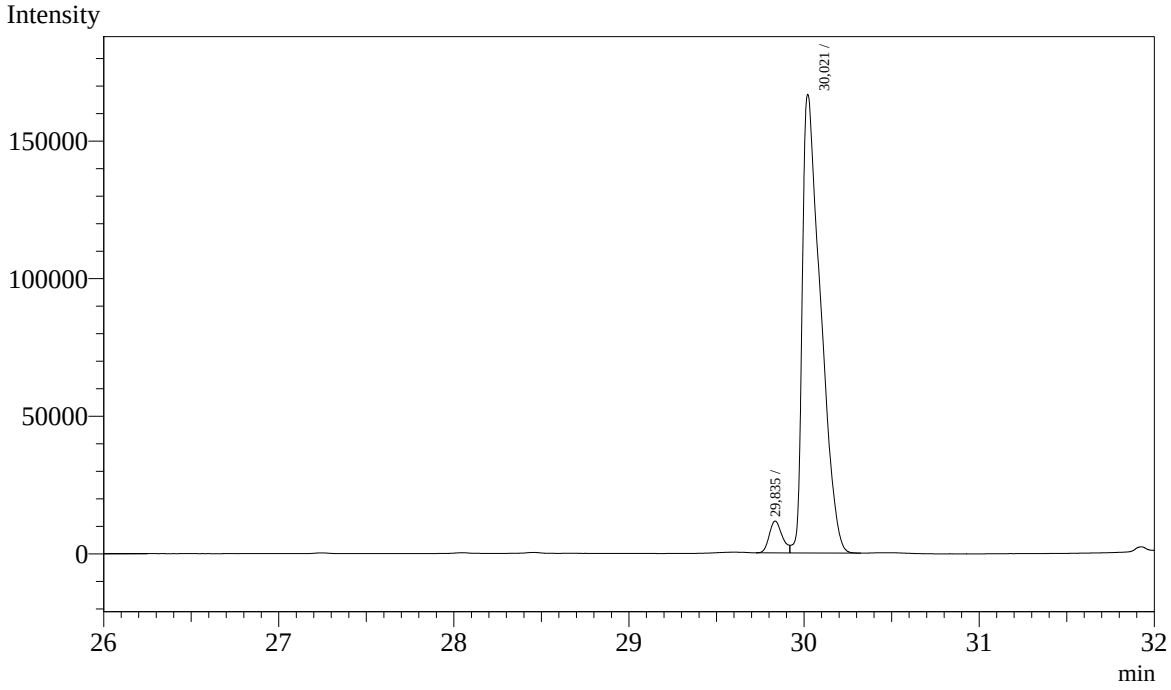


Peak#	Ret.Time	Area	Height	Area%
1	29,800	526644	89181	50,3596
2	30,076	519123	81643	49,6404
Total		1045767	170824	100,0000

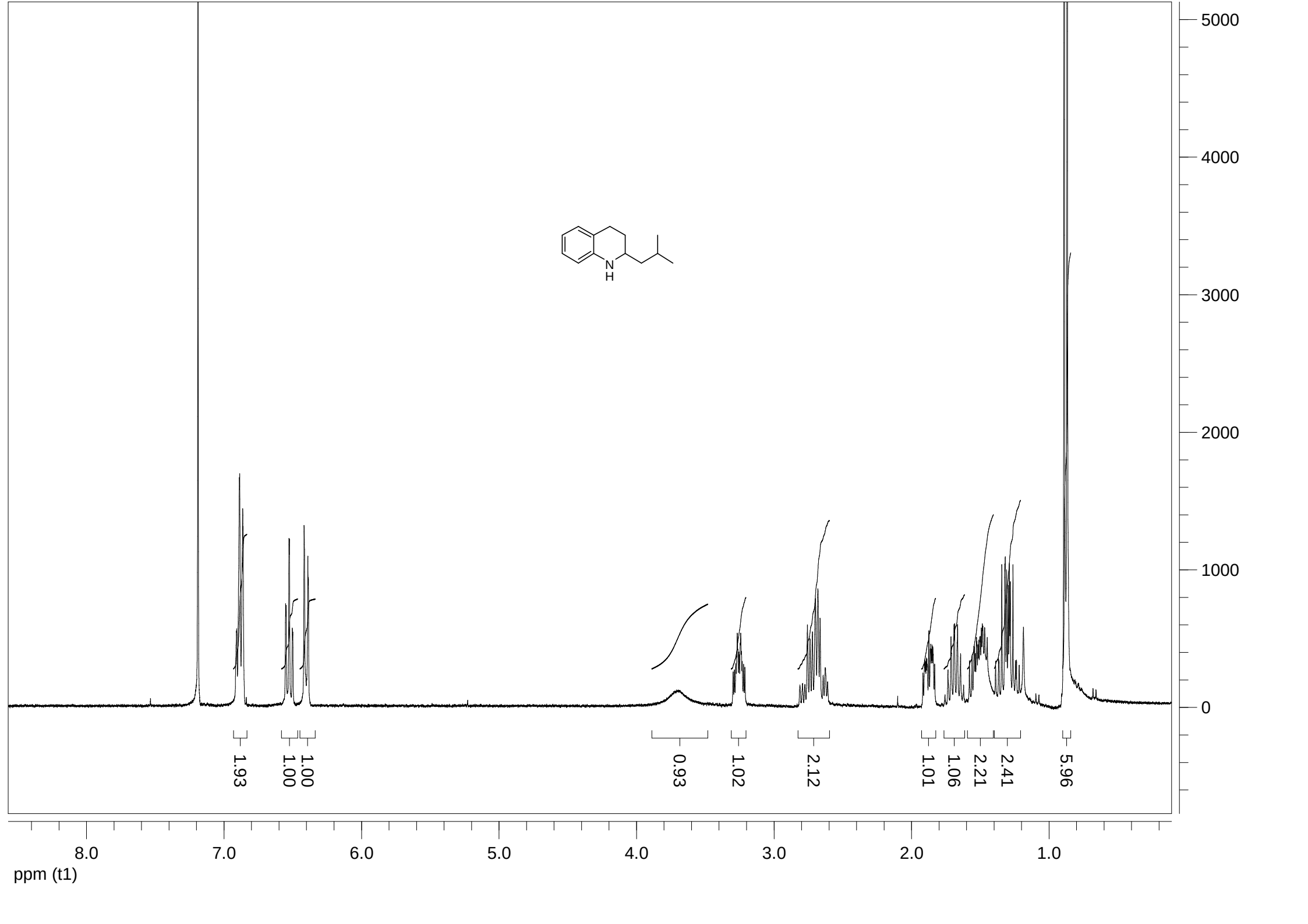
Analysis Date & Time : 17.09.2009 10:27:28
User Name : Admin
Vial# : 12
Sample Name : rk_648_nPent_c
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

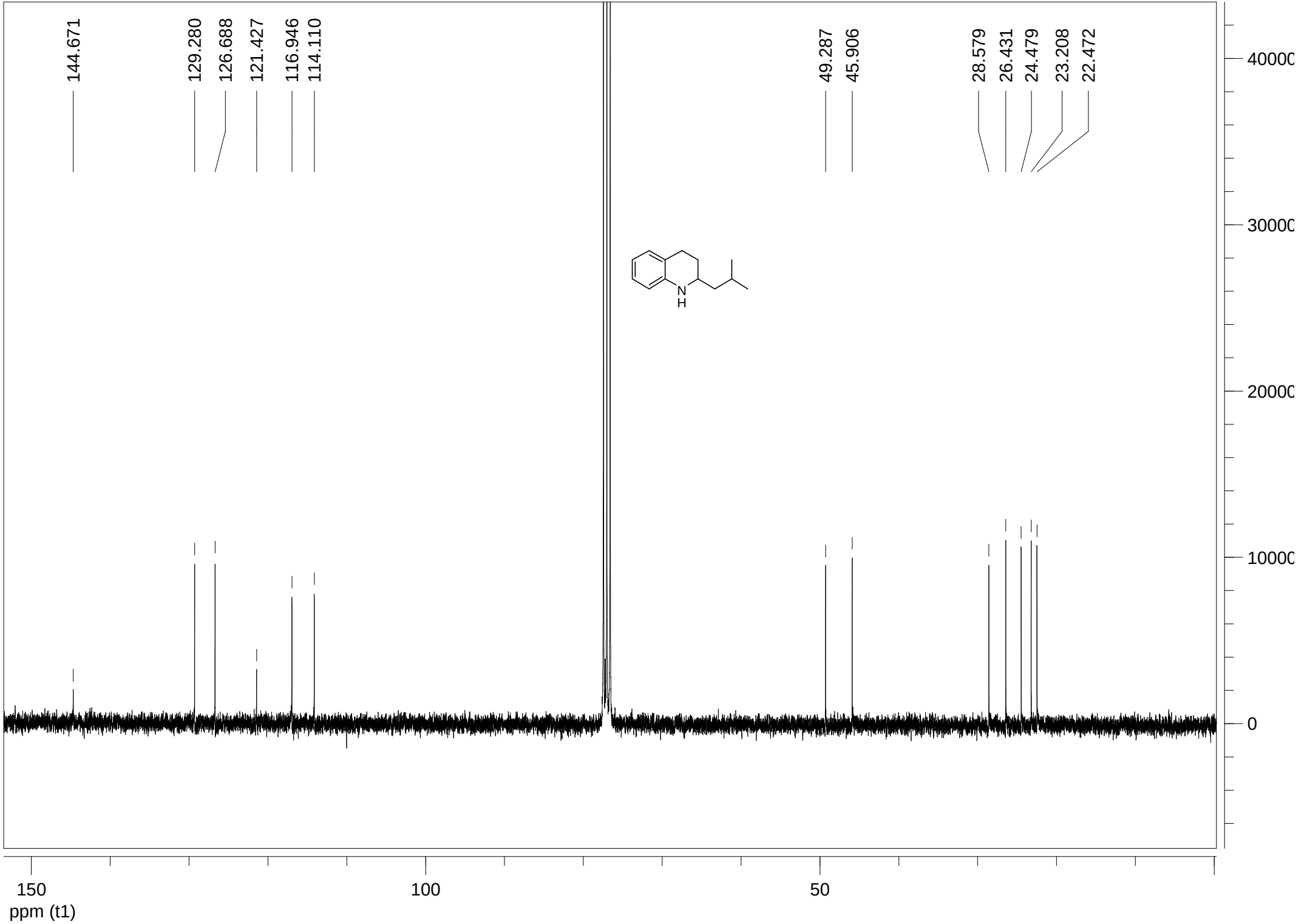


Data Name : F:\data shimadzu chiral gc\rk\quinoline project ac\rk648_nPent_c.gcd
Method Name : F:\data shimadzu chiral gc\rk\quinaldine_6cl.gcm

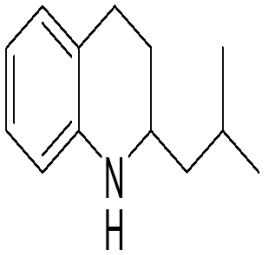


Peak#	Ret.Time	Area	Height	Area%
1	29,835	59575	11542	4,6893
2	30,021	1210859	166443	95,3107
Total		1270434	177985	100,0000

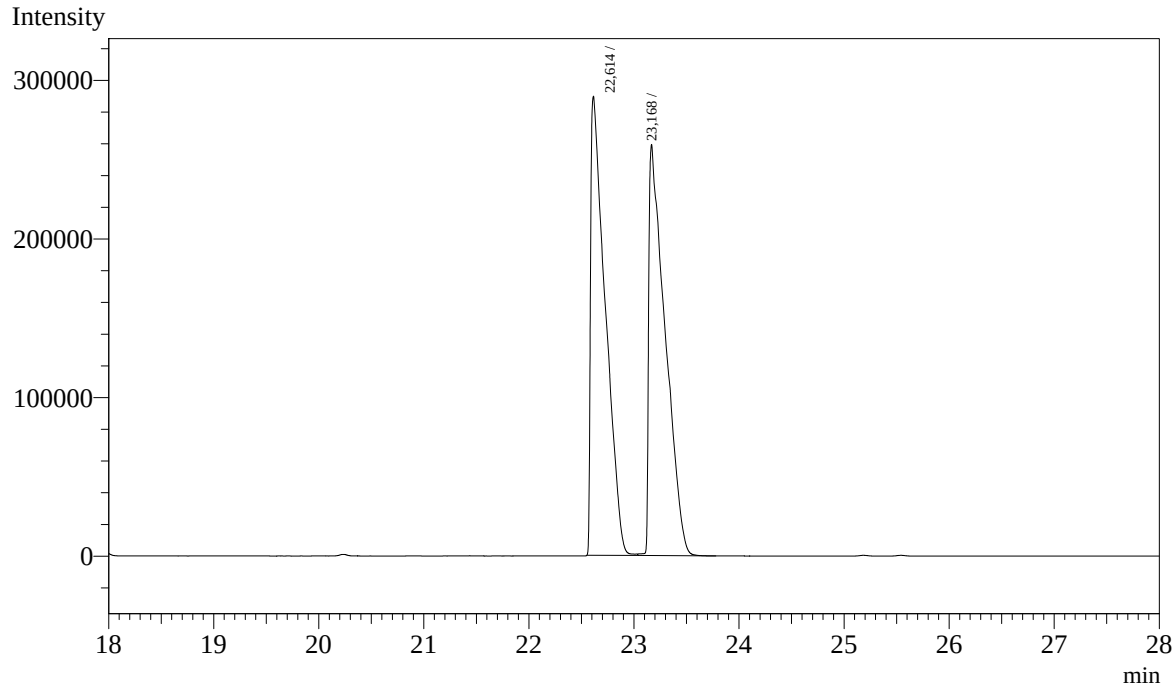

5000
4000
3000
2000
1000
0
1.93
1.00
1.00
0.93
1.02
2.12
1.01
1.06
2.21
2.41
5.96
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


144.671
129.280
126.688
121.427
116.946
114.110
49.287
45.906
28.579
26.431
24.479
23.208
22.472
N
H
40000
30000
20000
10000
0
150
100
50
ppm (t1)

Analysis Date & Time : 20.09.2009 22:38:52
User Name : Admin
Vial# : 28
Sample Name : rk_iBu_rac
Sample ID :
Sample Type : Unknown
Injection Volume : 1,00
ISTD Amount :

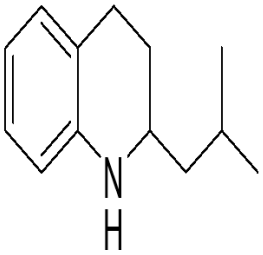


Data Name : F:\data shimadzu chiral gc\rk\quinoline project ac\rk_iBu_rac.gcd
Method Name : F:\data shimadzu chiral gc\rk\quinaldine_6br.gcm

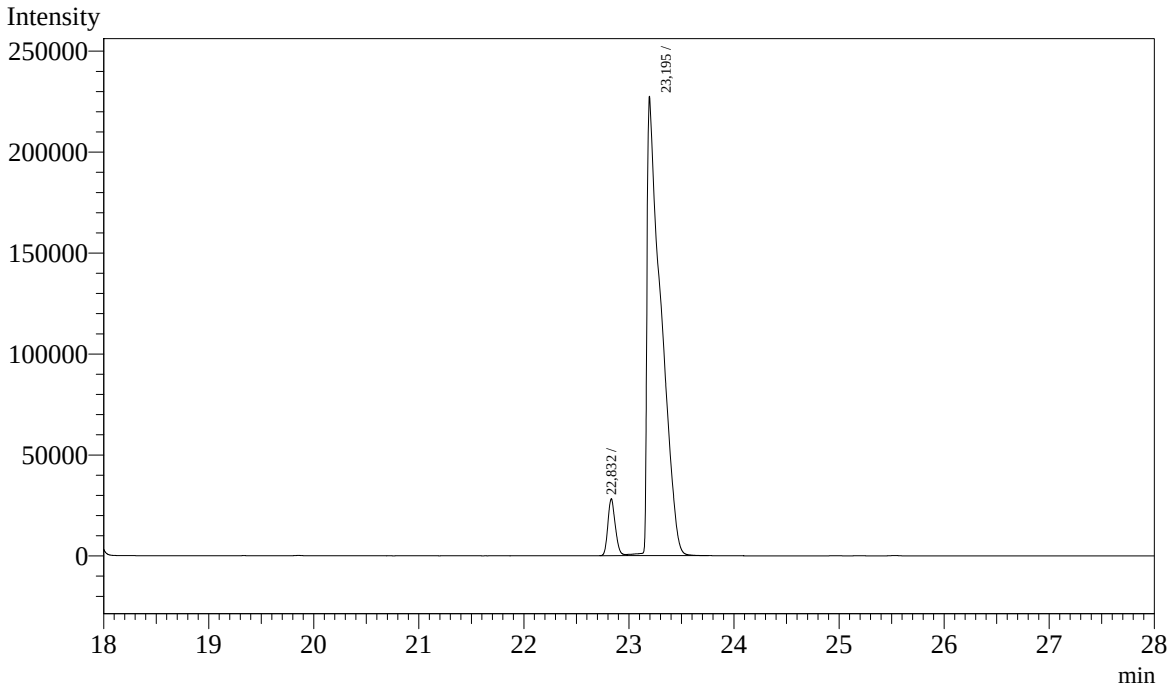


Peak#	Ret.Time	Area	Height	Area%
1	22,614	2821224	288566	50,0481
2	23,168	2815806	259063	49,9519
Total		5637030	547629	100,0000

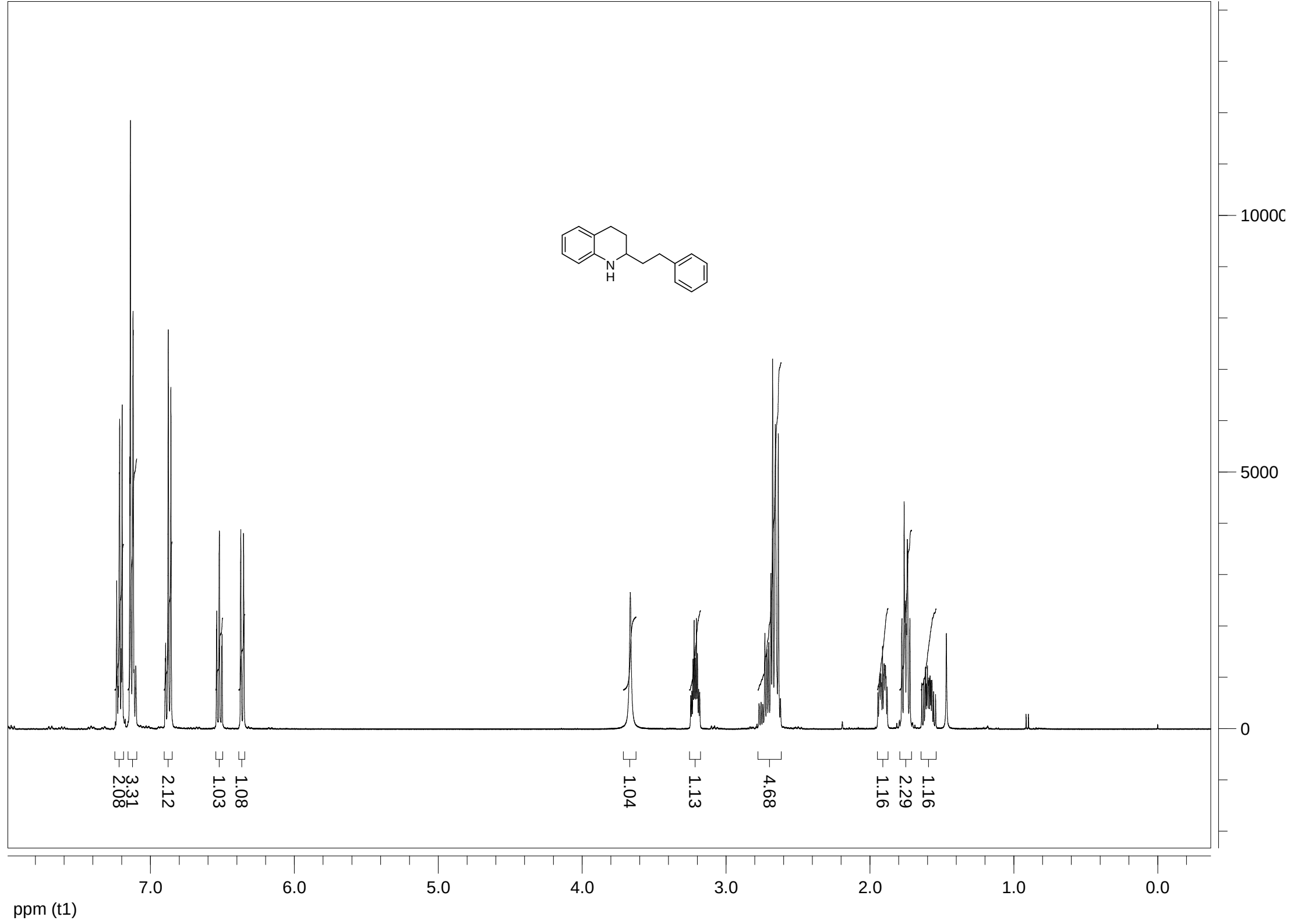
Analysis Date & Time	: 23.09.2009 14:50:54
User Name	: Admin
Vial#	: 30
Sample Name	: rk651_iBu_dil
Sample ID	:
Sample Type	: Unknown
Injection Volume	: 1,00
ISTD Amount	:

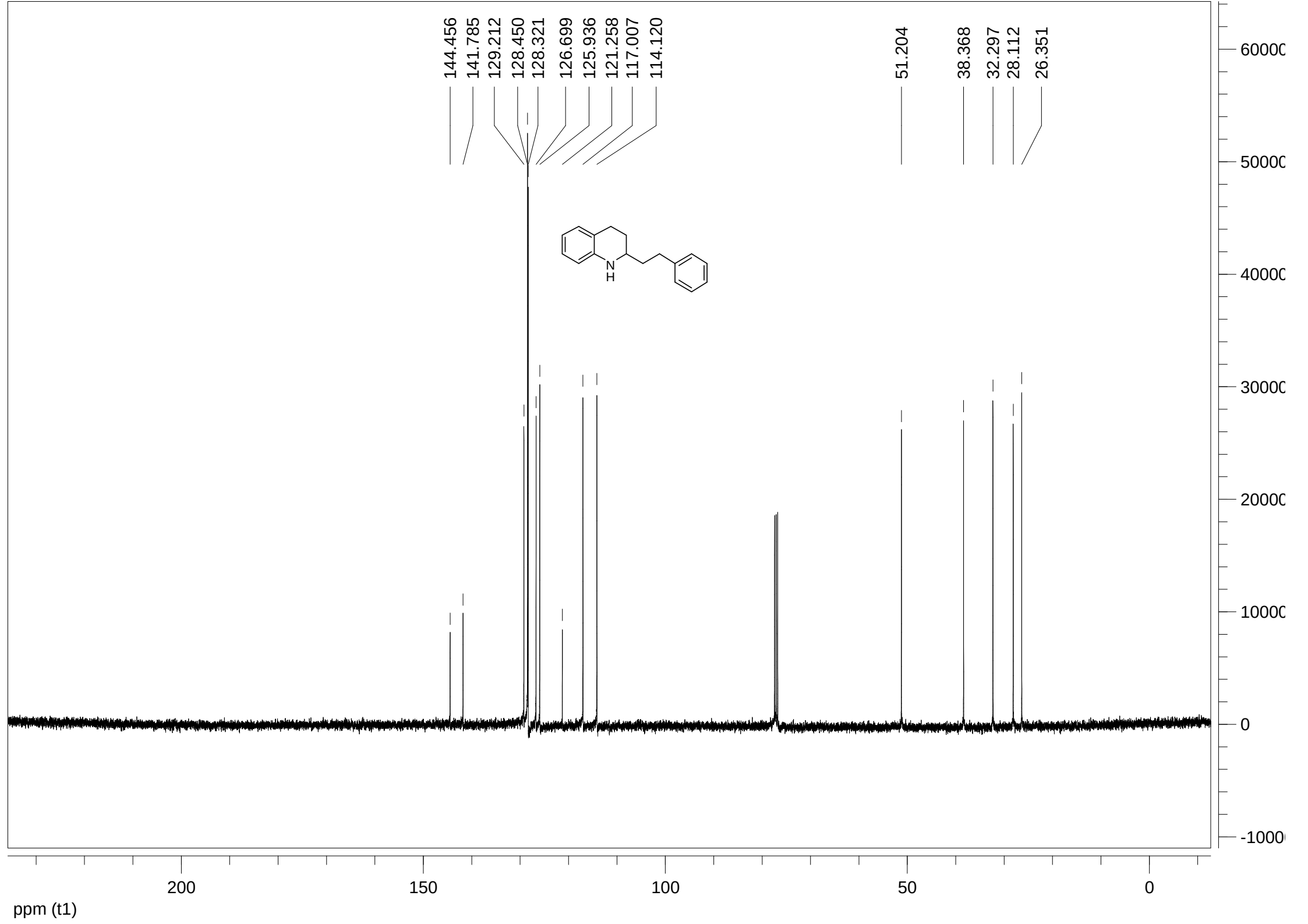


Data Name	: F:\data shimadzu chiral gc\rk\rk651_iBu_dil.gcd
Method Name	: F:\data shimadzu chiral gc\rk\quinaldine_6br.gcm



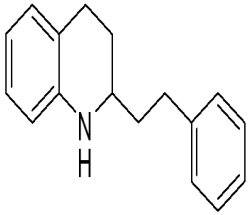
Peak#	Ret.Time	Area	Height	Area%
1	22,832	138433	28222	6,1960
2	23,195	2095801	227093	93,8040
Total		2234234	255315	100,0000

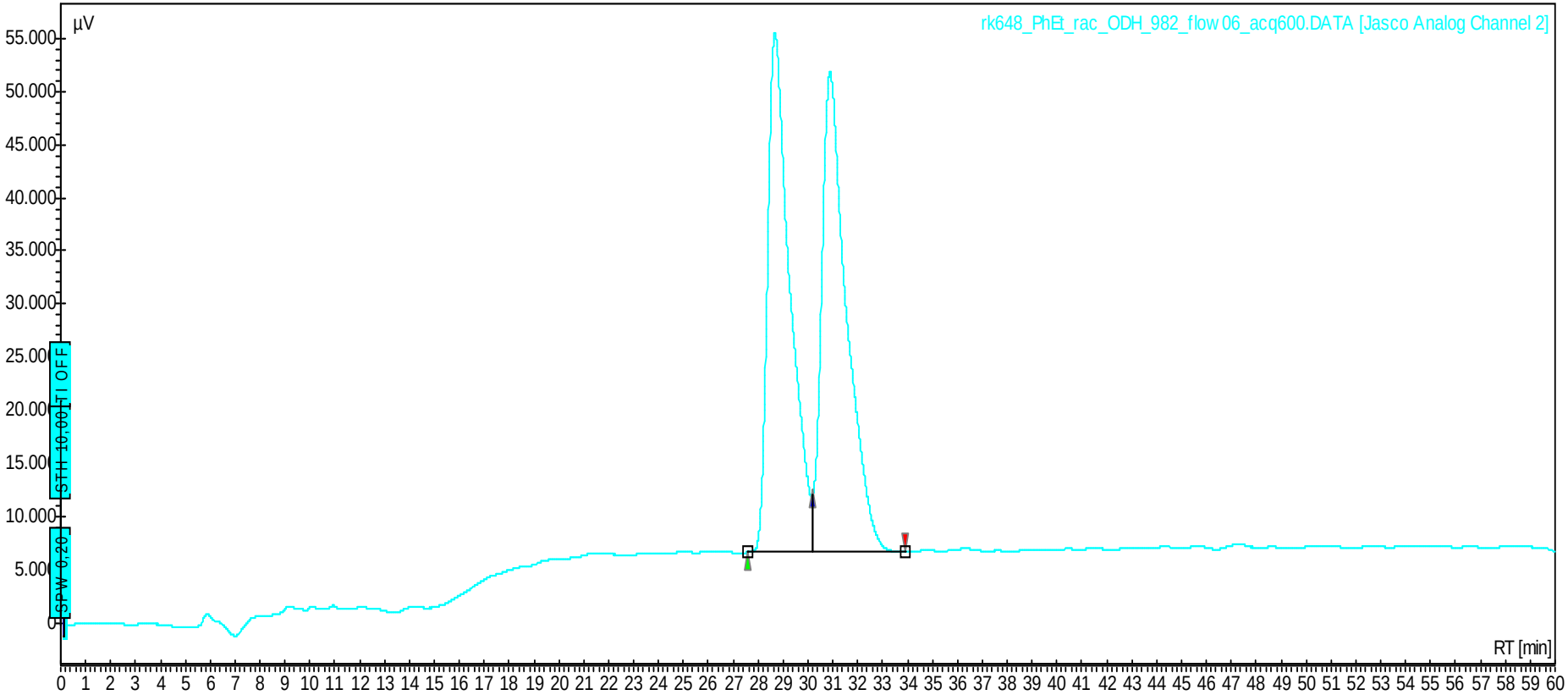

N
H
10000
5000
0
2.08
3.31
2.12
1.03
1.08
1.04
1.13
4.68
1.16
2.29
1.16
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


144.456
141.785
129.212
128.450
128.321
126.699
125.936
121.258
117.007
114.120
51.204
38.368
32.297
28.112
26.351
N
H
60000
50000
40000
30000
20000
10000
0
-10000
200
150
100
50
0
ppm (t1)

Chromatogram : rk648_PhEt_rac_ODH_982_flow06_acq600

Data file: rk648_PhEt_rac_ODH_982_flow06_acq600.DATA
Method: HPLC2_ODH_982_flow06_acq60
Date: 18.09.2009 10:05:12



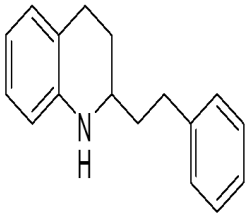


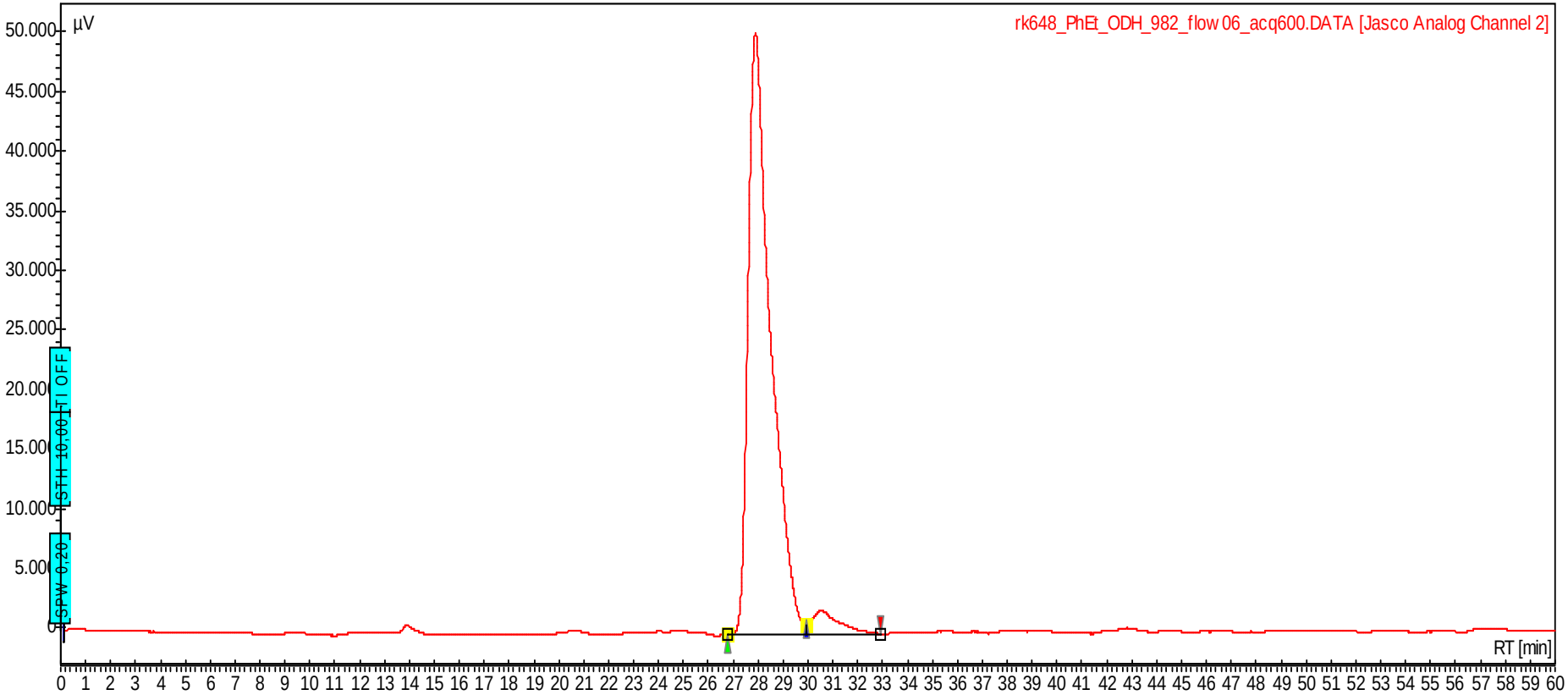
rk648_PhEt_rac_ODH_982_flow06_acq600.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	27,611	28,675	30,204	0,000	49,32	48728,0	50832,8	49,320
2	UNKNOWN	30,204	30,892	33,910	0,000	50,68	45091,2	52235,2	50,680
Total						100,00	93819,2	103068,1	100,000

Chromatogram : rk648_PhEt_ODH_982_flow06_acq600

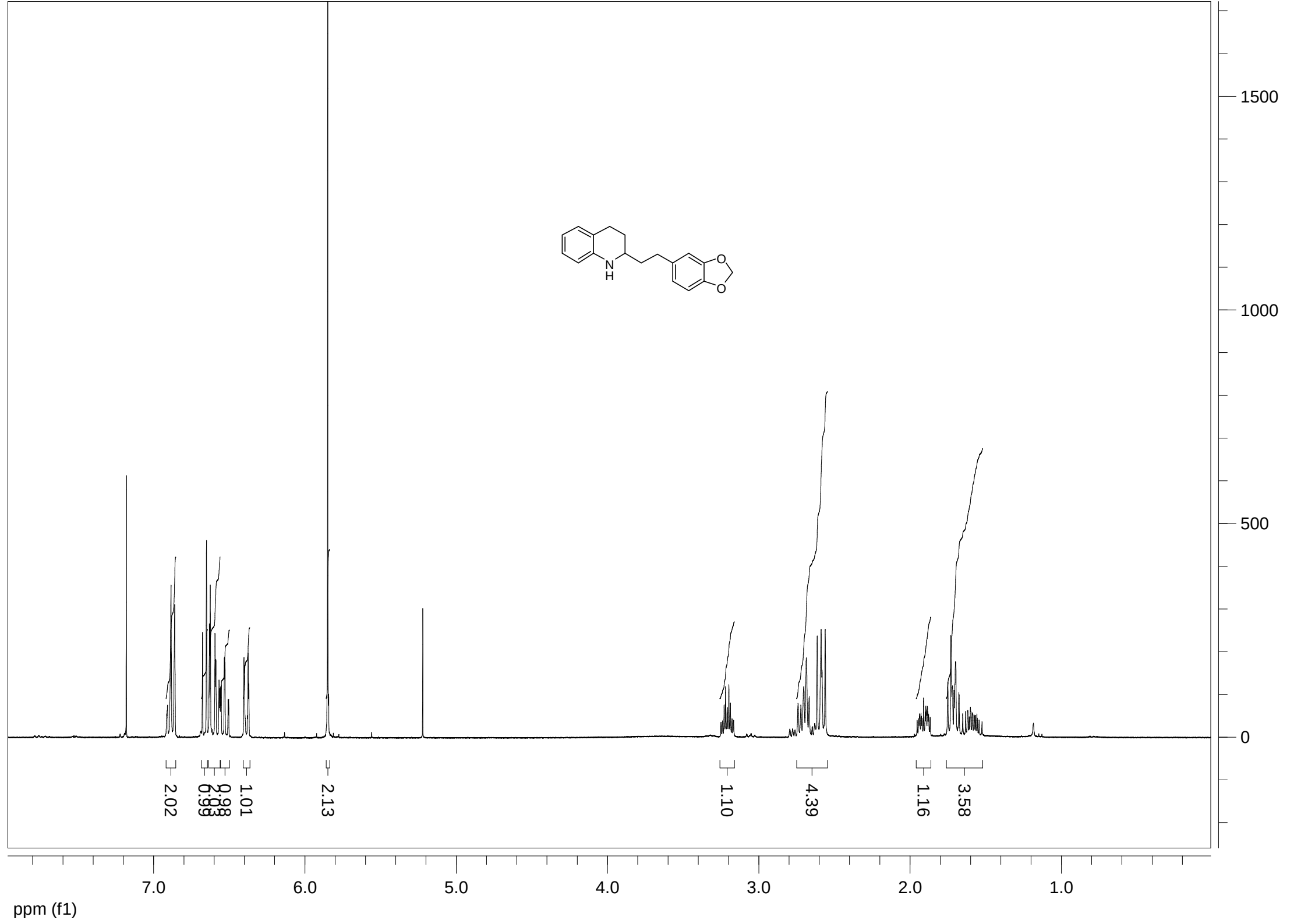
Data file: rk648_PhEt_ODH_982_flow06_acq600.DATA
Method: HPLC2_ODH_982_flow06_acq60
Date: 18.09.2009 11:07:51

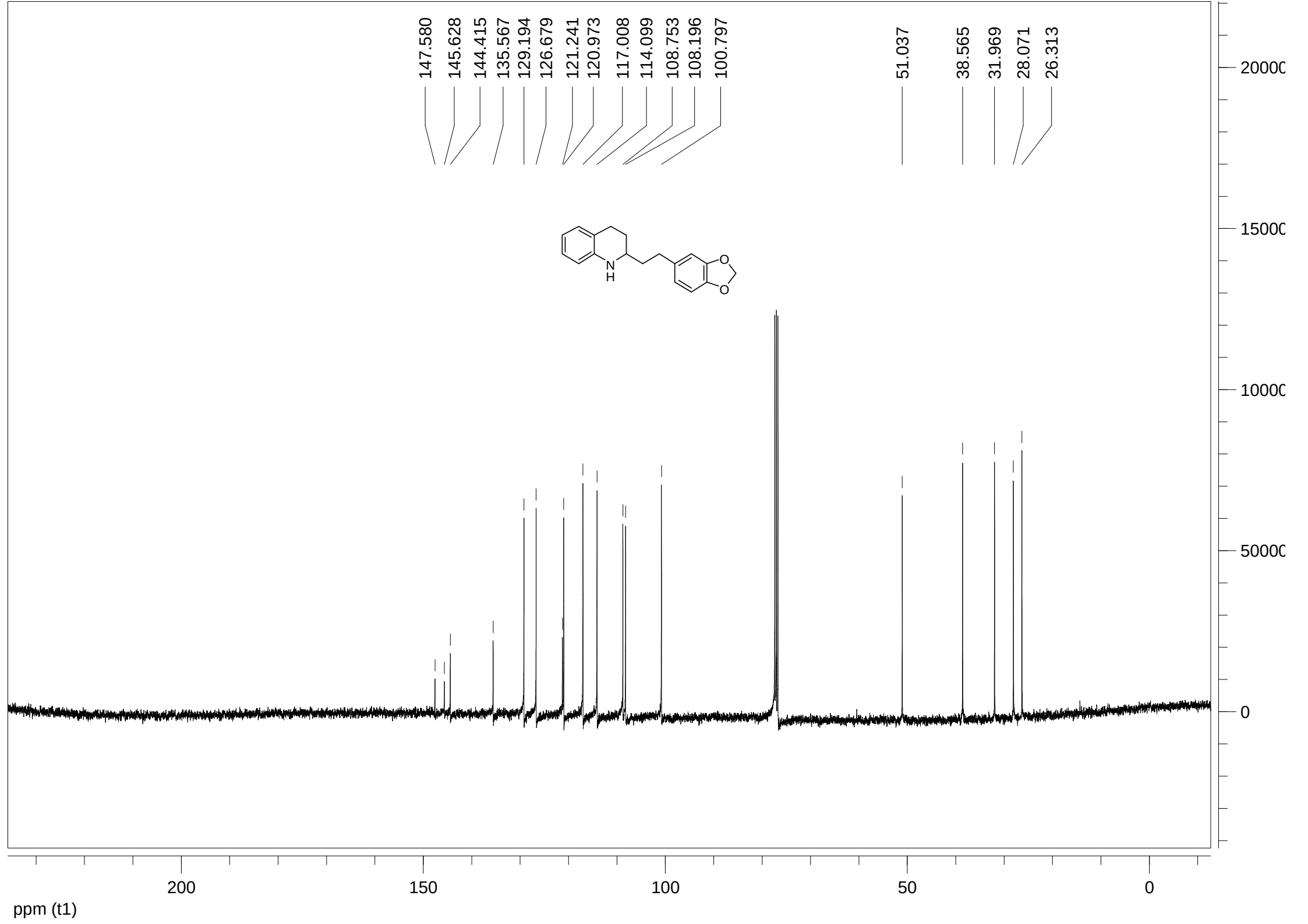




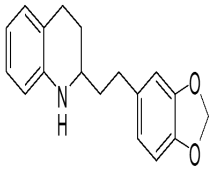
rk648_PhEt_ODH_982_flow06_acq600.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	26,800	27,892	29,952	0,000	95,55	50318,5	52754,0	95,551
2	UNKNOWN	29,952	30,517	32,921	0,000	4,45	1930,5	2456,1	4,449
Total						100,00	52249,0	55210,1	100,000

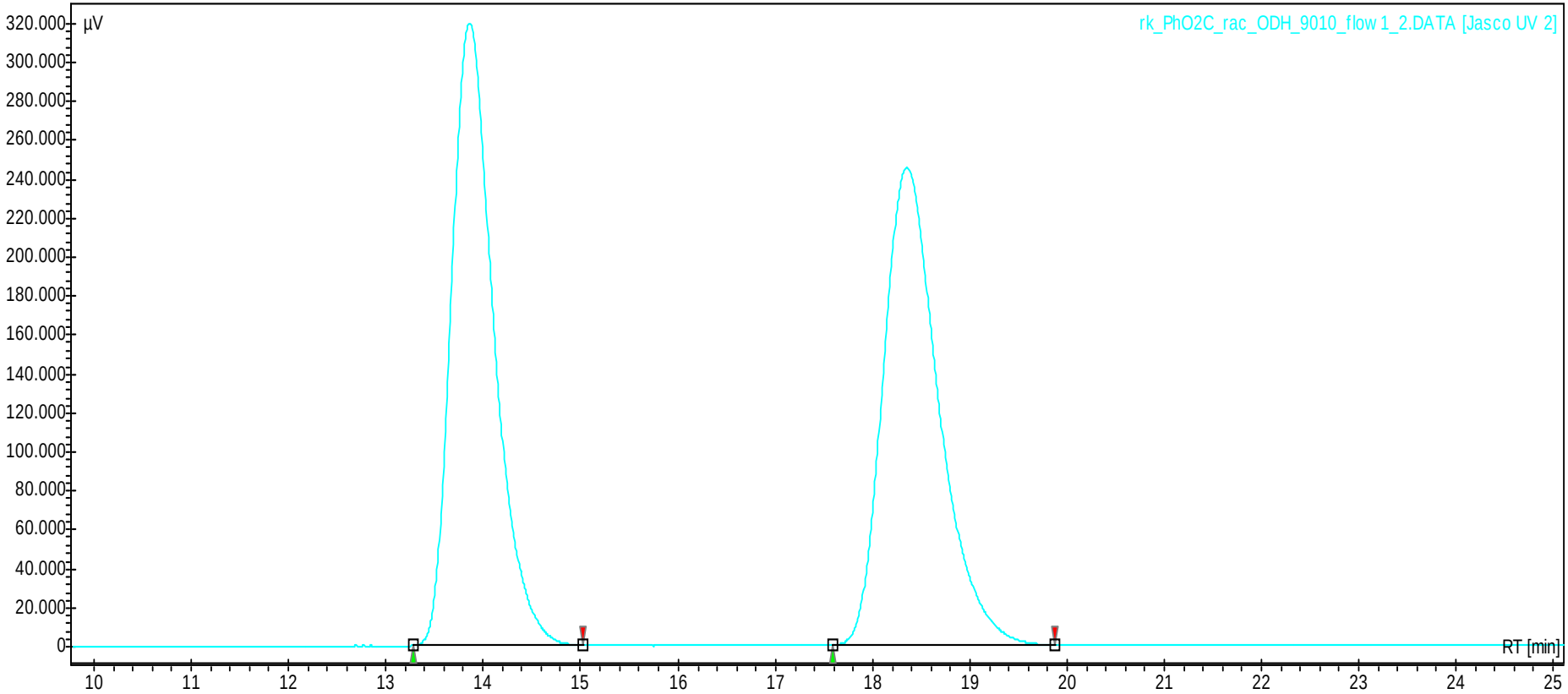

1500
1000
500
0
2.02
0.99
0.98
1.01
2.13
1.10
4.39
1.16
3.58
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


147.580
145.628
144.415
135.567
129.194
126.679
121.241
120.973
117.008
114.099
108.753
108.196
100.797
51.037
38.565
31.969
28.071
26.313
N
H
O
O
20000
15000
10000
50000
0
200
150
100
50
0
ppm (t1)

Chromatogram : rk_PhO2C_rac_ODH_9010_flow1_2



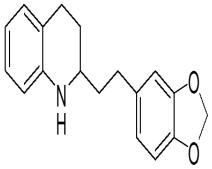
Data file: rk_PhO2C_rac_ODH_9010_flow1_2.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 22.09.2009 09:00:43



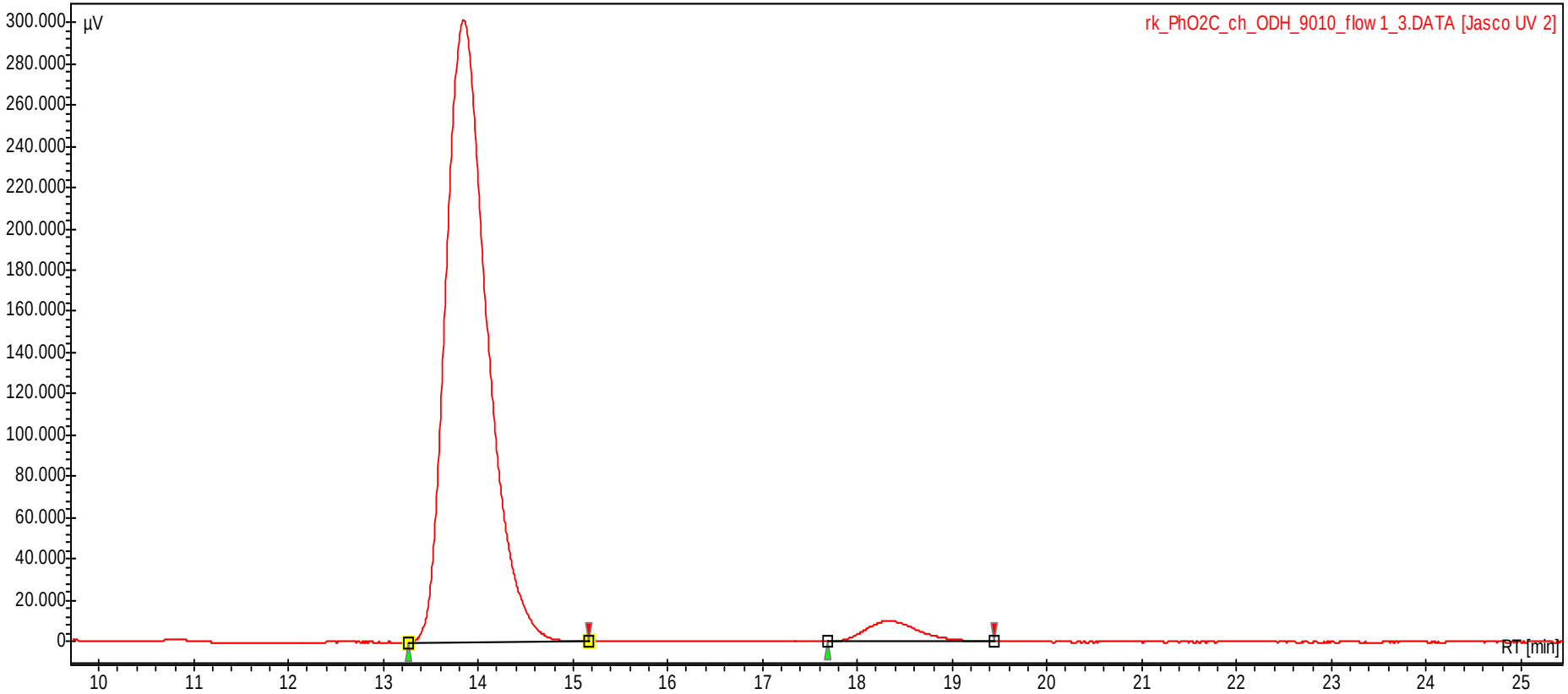
rk_PhO2C_rac_ODH_9010_flow1_2.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,274	13,858	15,018	49,878
2	17,591	18,350	19,866	50,122
Total				100,000

Chromatogram : rk_PhO2C_ch_ODH_9010_flow1_3

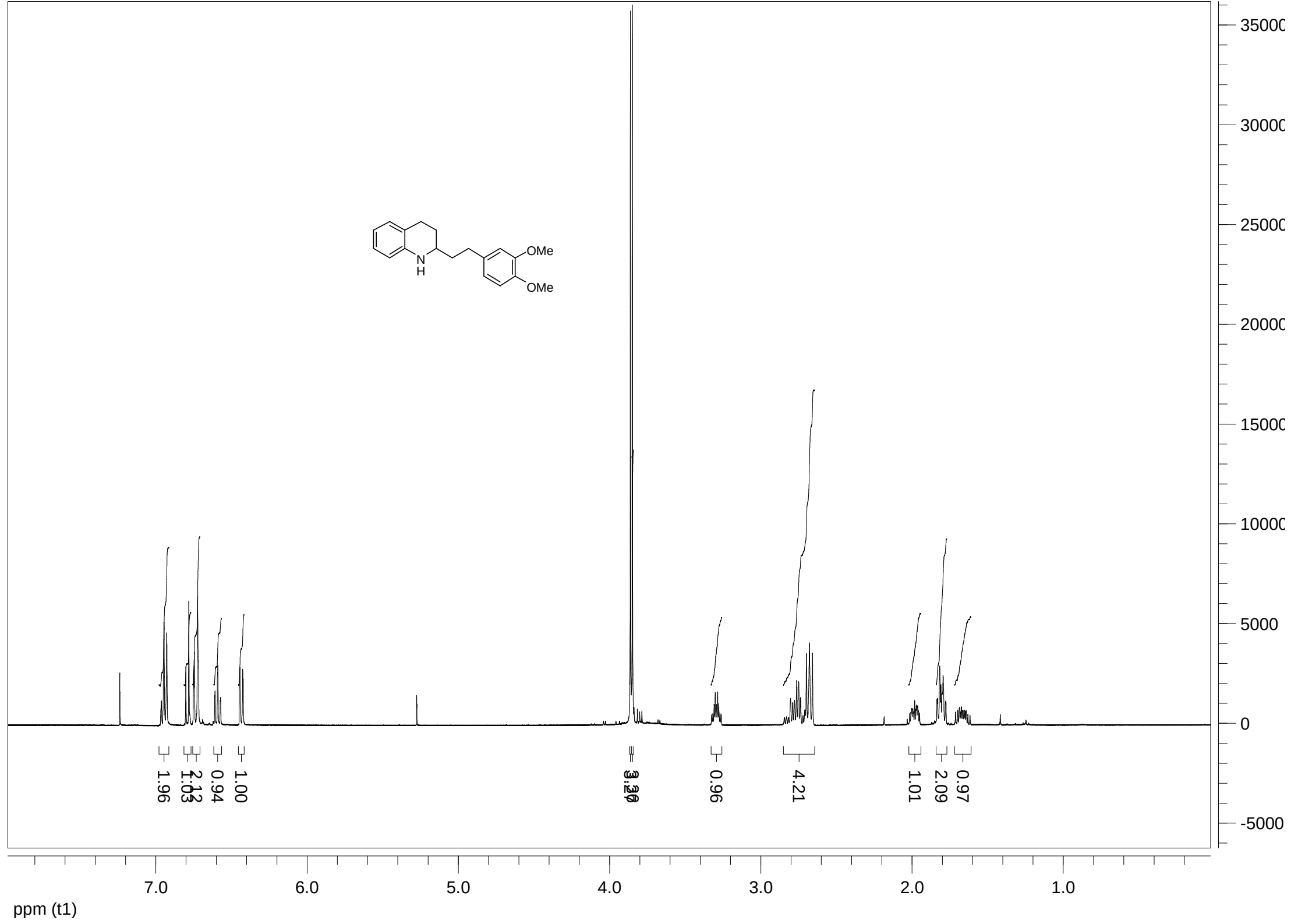


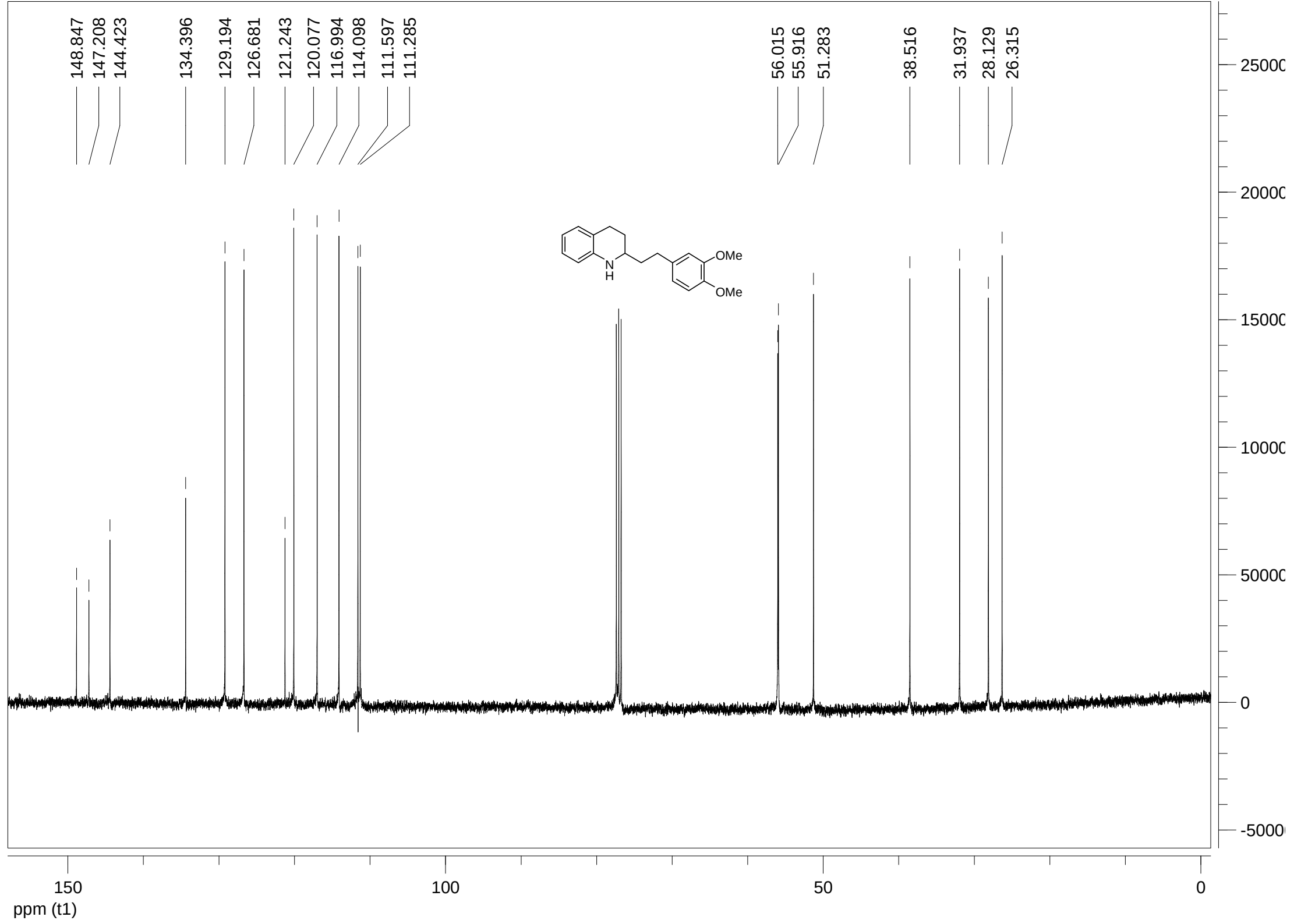
Data file: rk_PhO2C_ch_ODH_9010_flow1_3.DATA
Method: HPLC1_ODH_9010_flow1_acq_30
Date: 22.09.2009 09:33:28



rk_PhO2C_ch_ODH_9010_flow1_3.DATA [Jasco UV 2]

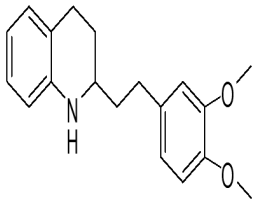
Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,257	13,842	15,157	95,814
2	17,691	18,333	19,440	4,186
Total				100,000

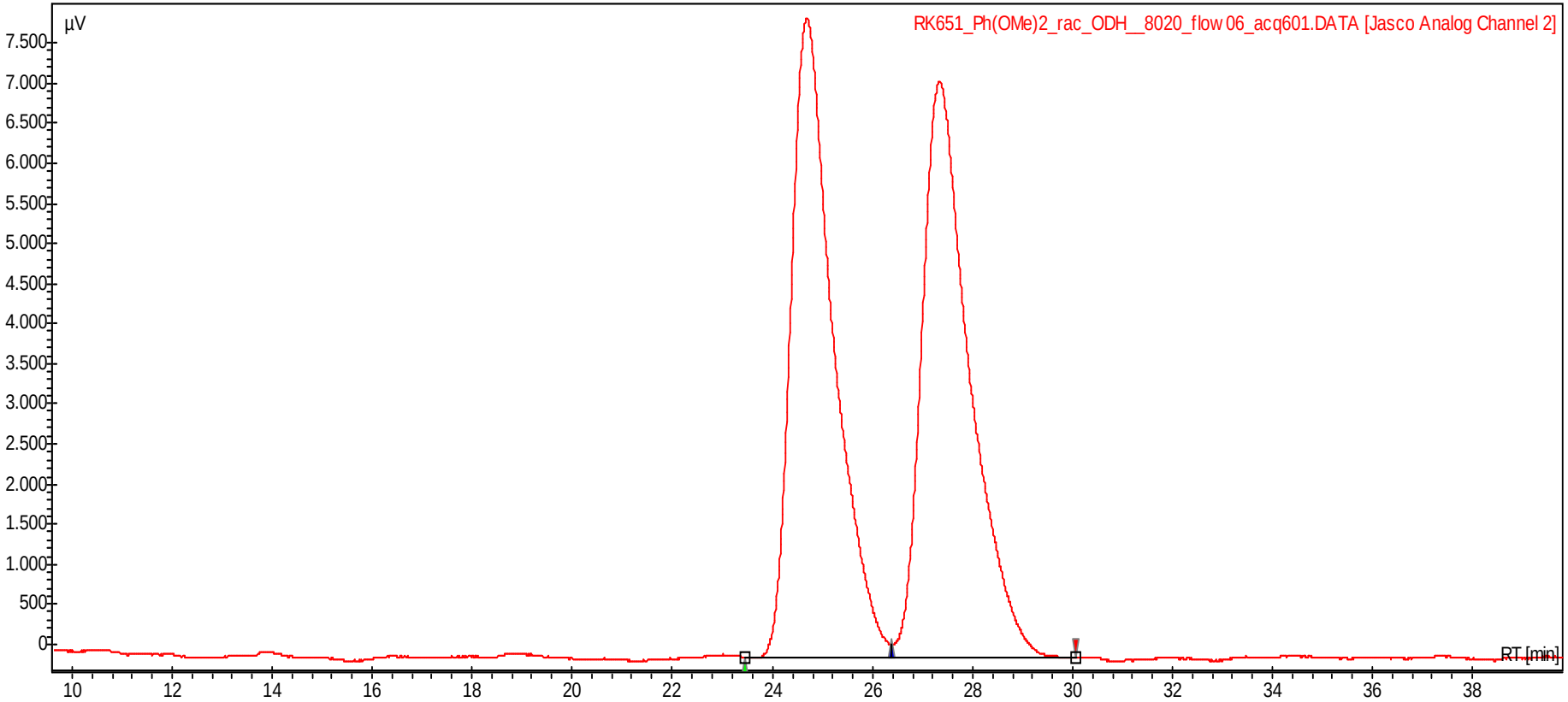

OMe
OMe
N
H
1.96
1.03
2.12
0.94
1.00
3.30
0.96
4.21
1.01
2.09
0.97
35000
30000
25000
20000
15000
10000
5000
0
-5000
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


148.847
147.208
144.423
134.396
129.194
126.681
121.243
120.077
116.994
114.098
111.597
111.285
56.015
55.916
51.283
38.516
31.937
28.129
26.315
OMe
OMe
N
H
25000
20000
15000
10000
50000
0
-50000
150
100
50
0
ppm (t1)

Chromatogram : RK651_Ph(OMe)2_rac_ODH__8020_flow06_acq601

Data file: RK651_Ph(OMe)2_rac_ODH__8020_flow06_acq601.DATA
Method: HPLC2_ODH_8020_flow06_acq60
Date: 19.09.2009 16:06:51

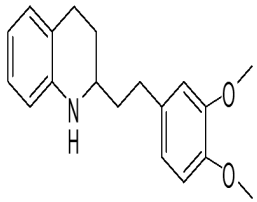




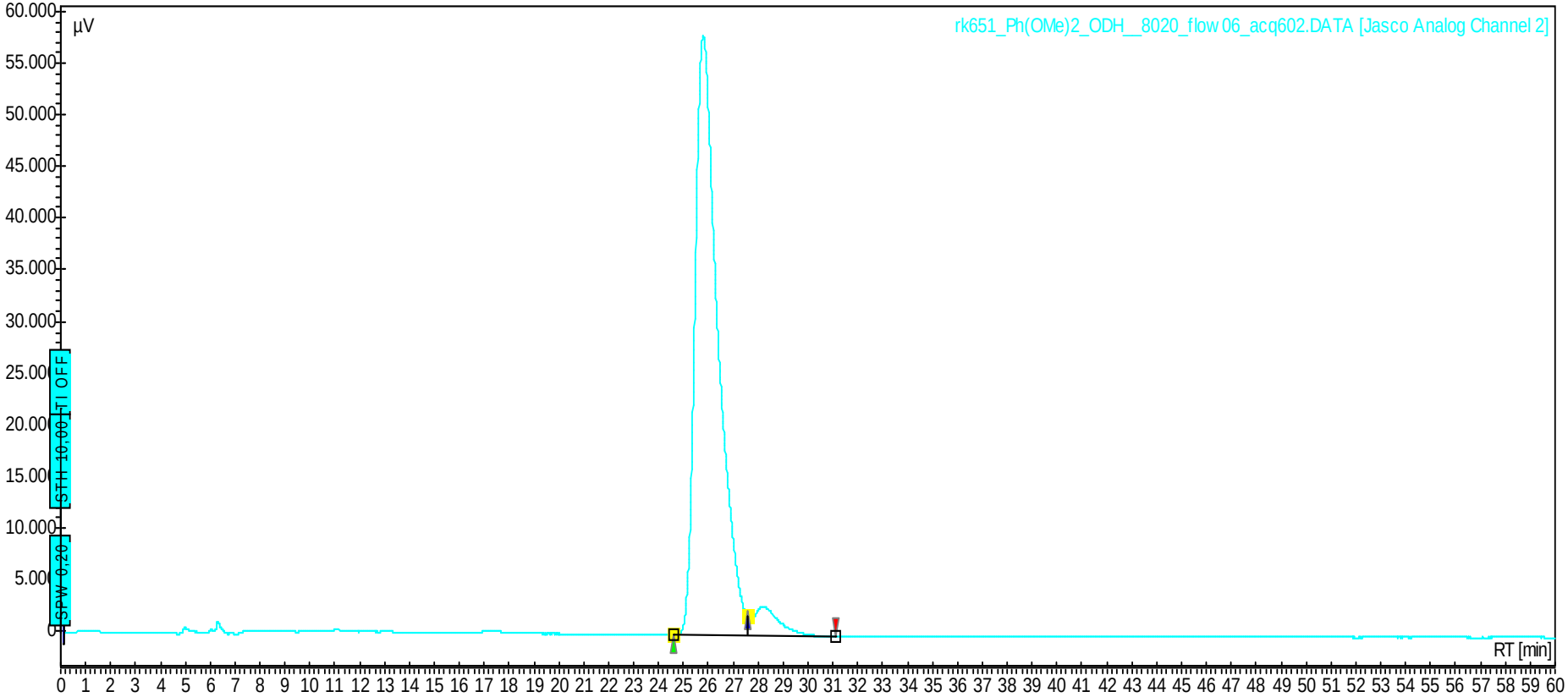
RK651_Ph(OMe)2_rac_ODH__8020_flow06_acq601.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	23,453	24,692	26,384	0,000	49,69	7964,0	7817,8	49,688
2	UNKNOWN	26,384	27,342	30,064	0,000	50,31	7178,1	7915,9	50,312
Total						100,00	15142,1	15733,7	100,000

Chromatogram : rk651_Ph(OMe)2_ODH__8020_flow06_acq602

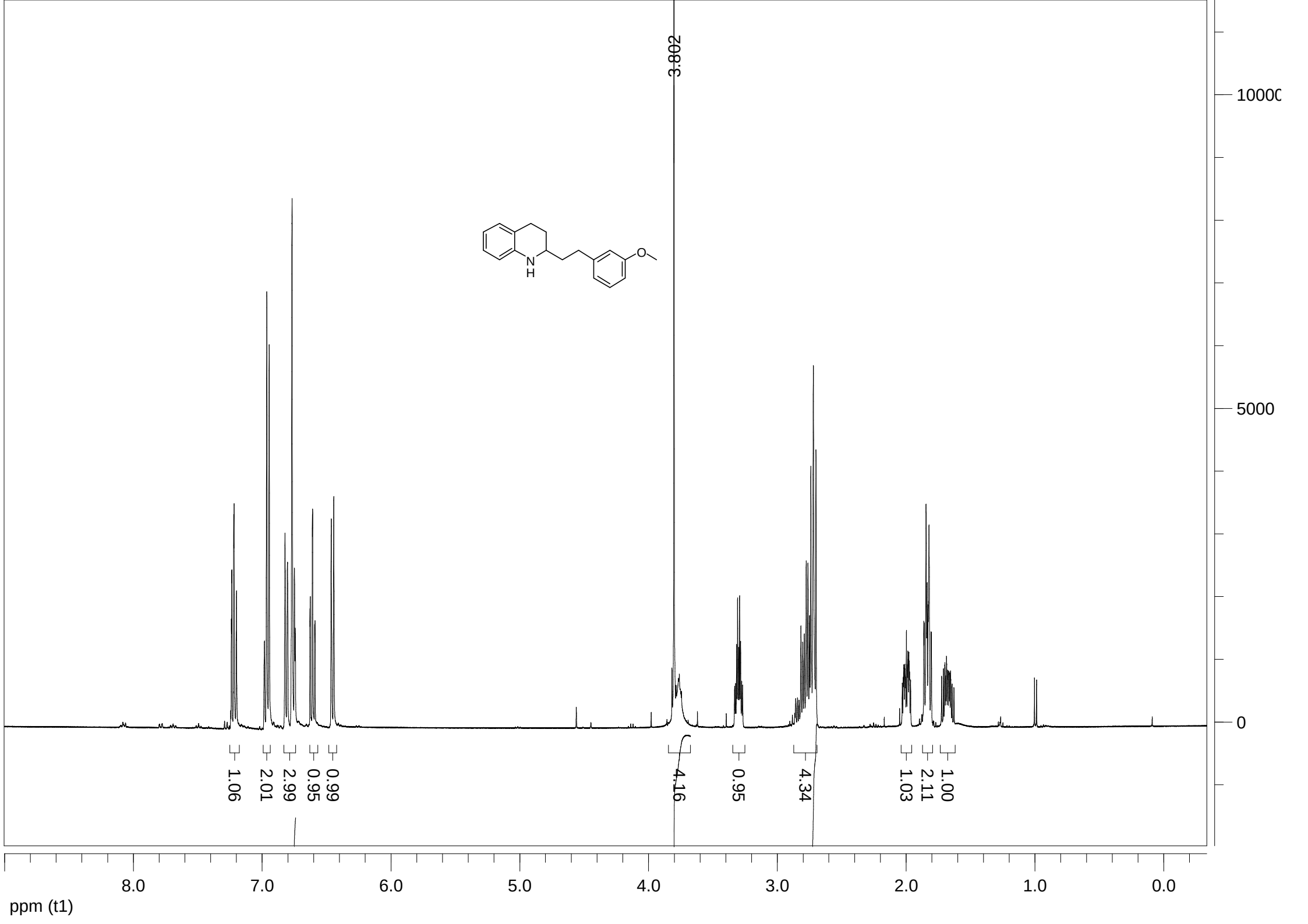


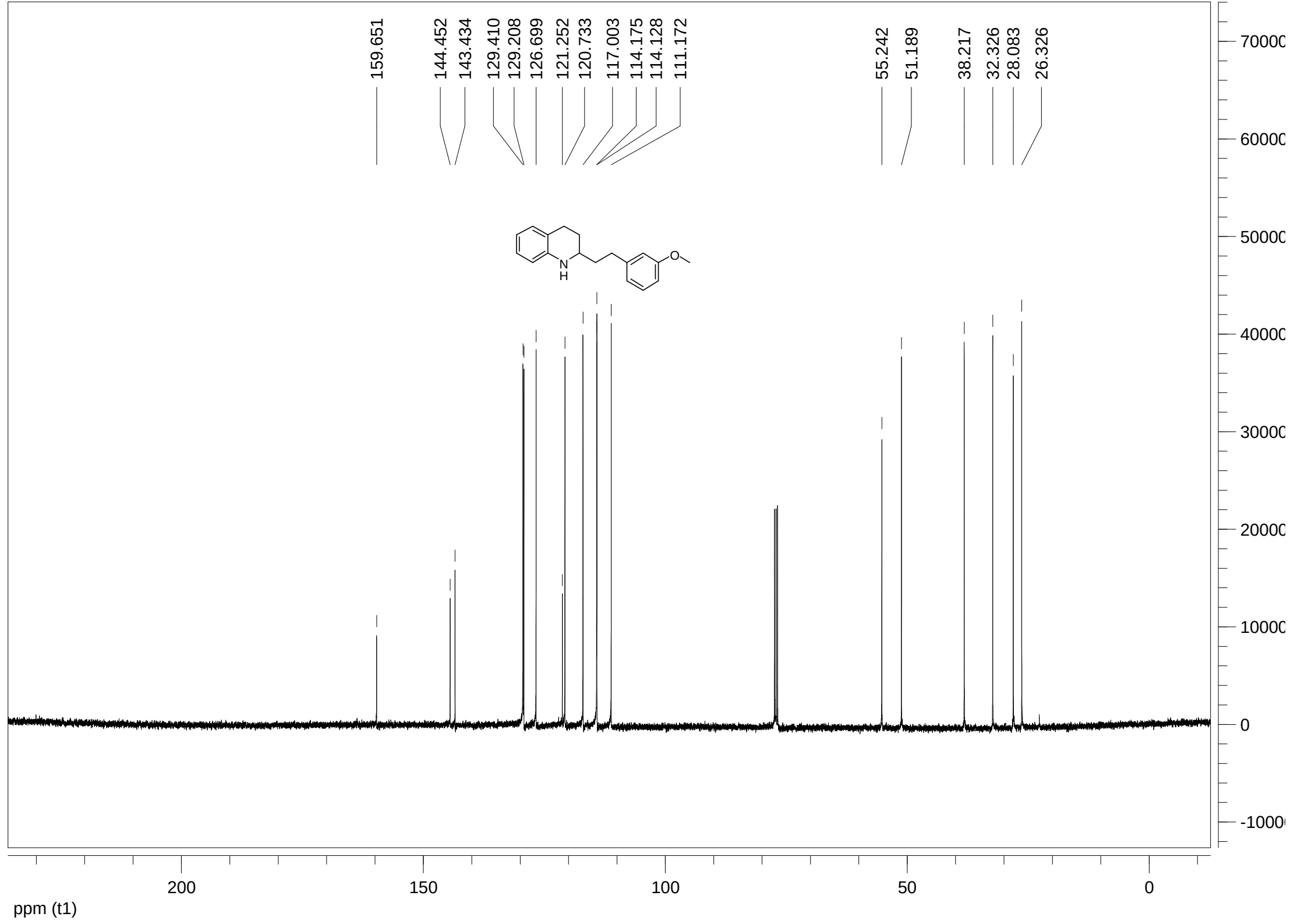
Data file: rk651_Ph(OMe)2_ODH__8020_flow06_acq602.DATA
Method: HPLC2_ODH_8020_flow06_acq60
Date: 20.09.2009 23:31:08



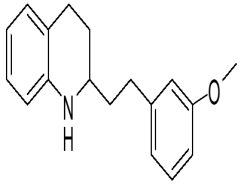
rk651_Ph(OMe)2_ODH__8020_flow06_acq602.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	24,632	25,800	27,559	0,000	94,76	58092,2	61974,0	94,764
2	UNKNOWN	27,559	28,217	31,130	0,000	5,24	2787,8	3424,3	5,236
Total						100,00	60879,9	65398,4	100,000

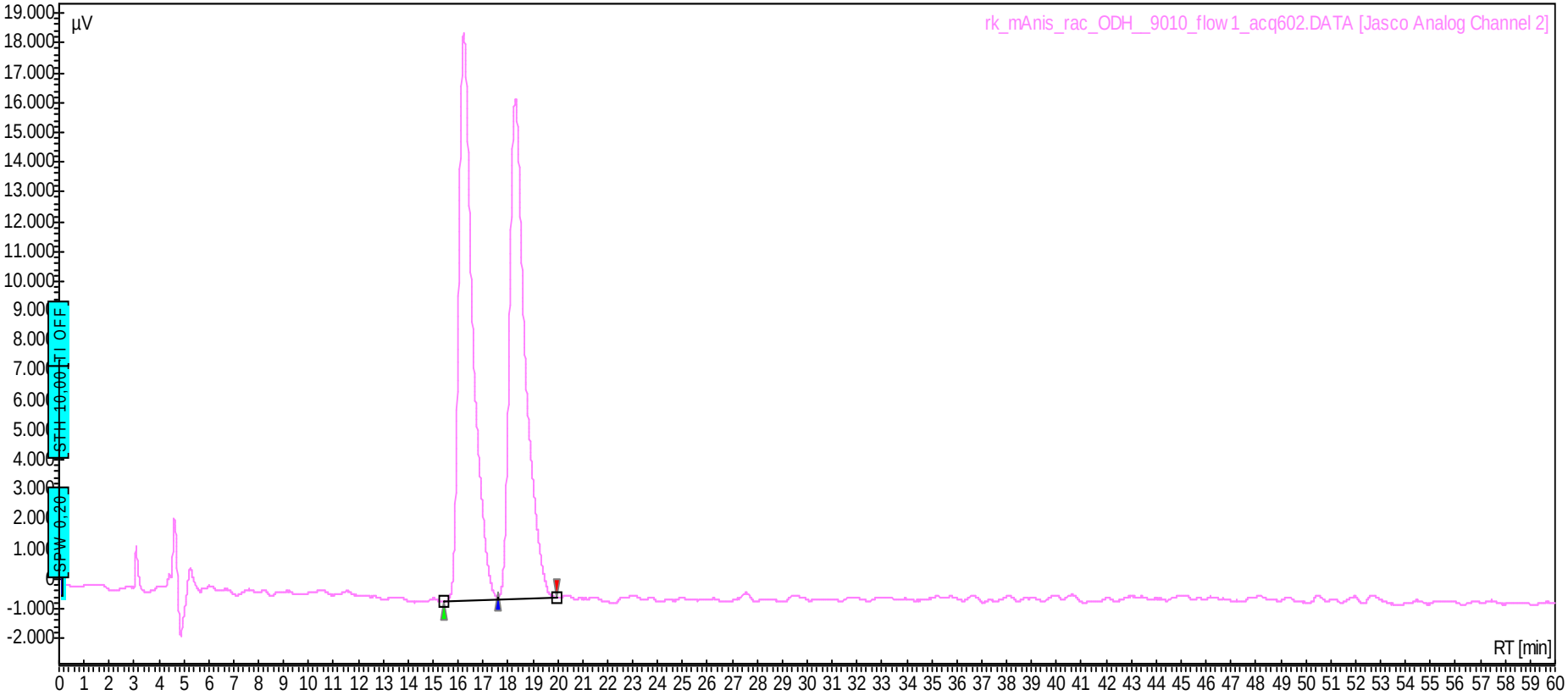

3.802
10000
5000
0
1.06
2.01
2.99
0.95
0.99
4.16
0.95
4.34
1.03
2.11
1.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


159.651
144.452
143.434
129.410
129.208
126.699
121.252
120.733
117.003
114.175
114.128
111.172
55.242
51.189
38.217
32.326
28.083
26.326
N
H
O
70000
60000
50000
40000
30000
20000
10000
0
-10000
200
150
100
50
0
ppm (t1)

Chromatogram : rk_mAnis_rac_ODH__9010_flow1_acq602



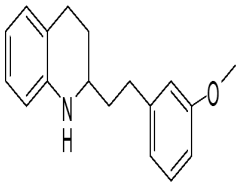
Data file: rk_mAnis_rac_ODH__9010_flow1_acq602.DATA
Method: HPLC2_ODH_9010_flow1_acq60
Date: 24.09.2009 01:28:41



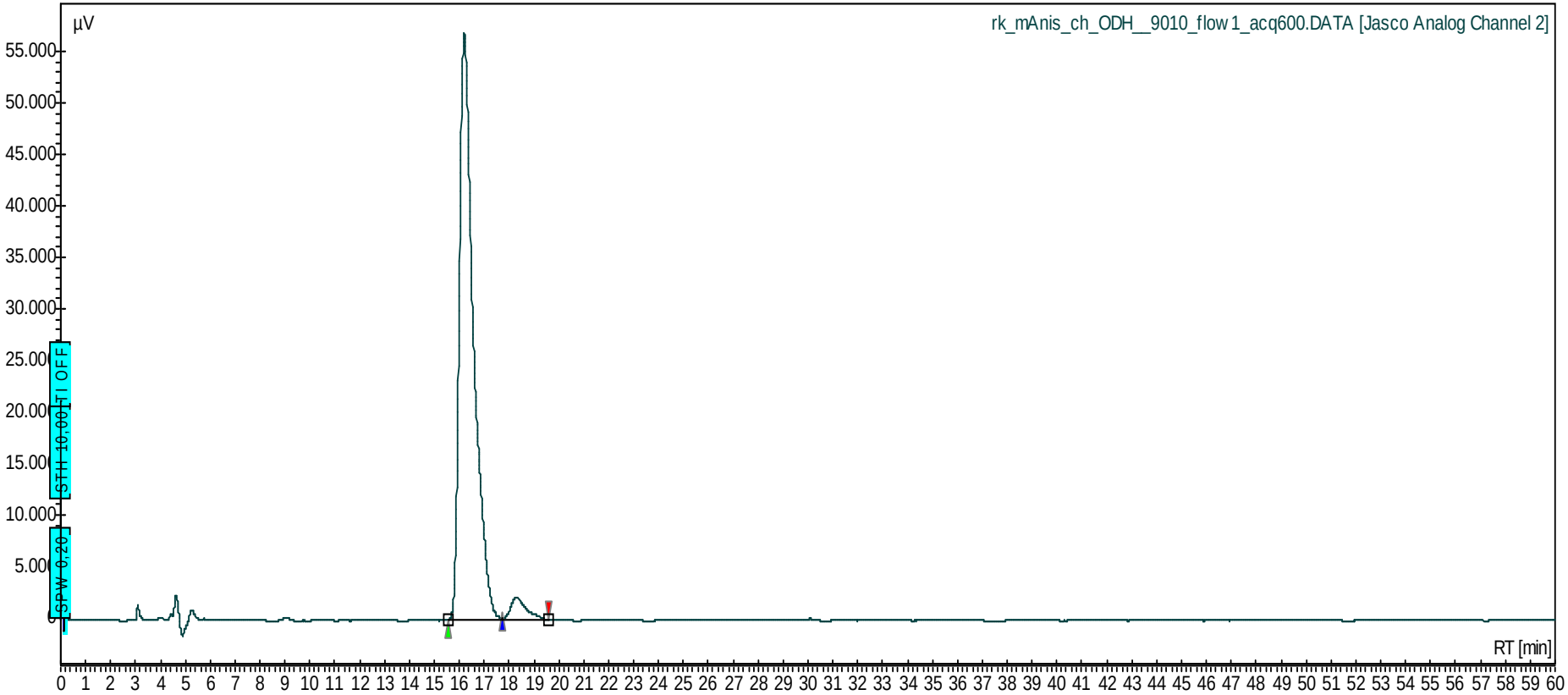
rk_mAnis_rac_ODH__9010_flow1_acq602.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	15,446	16,233	17,607	0,000	50,19	19060,0	12499,7	50,192
2	UNKNOWN	17,607	18,292	19,985	0,000	49,81	16833,5	12404,1	49,808
Total						100,00	35893,5	24903,8	100,000

Chromatogram : rk_mAnis_ch_ODH__9010_flow1_acq600

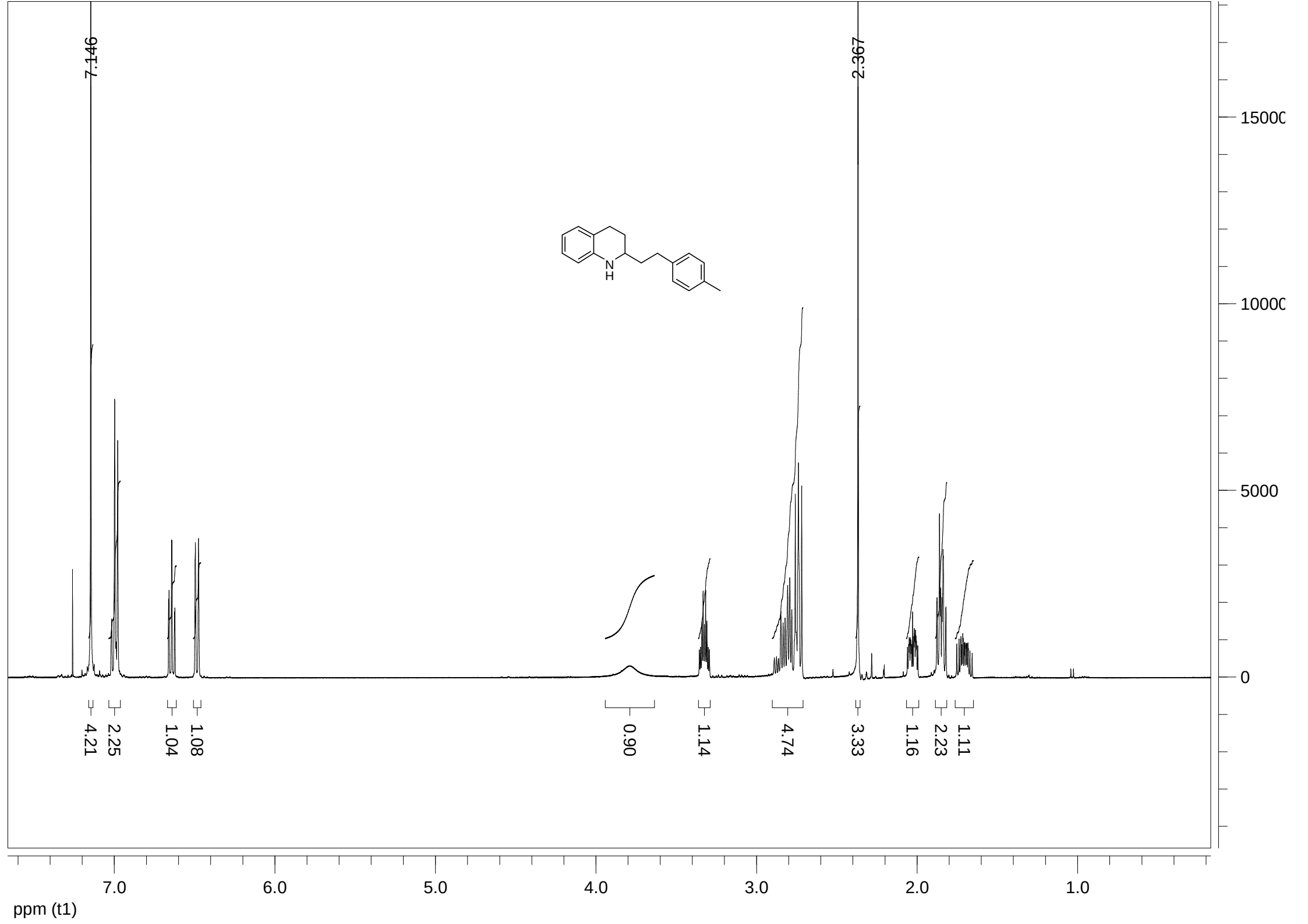


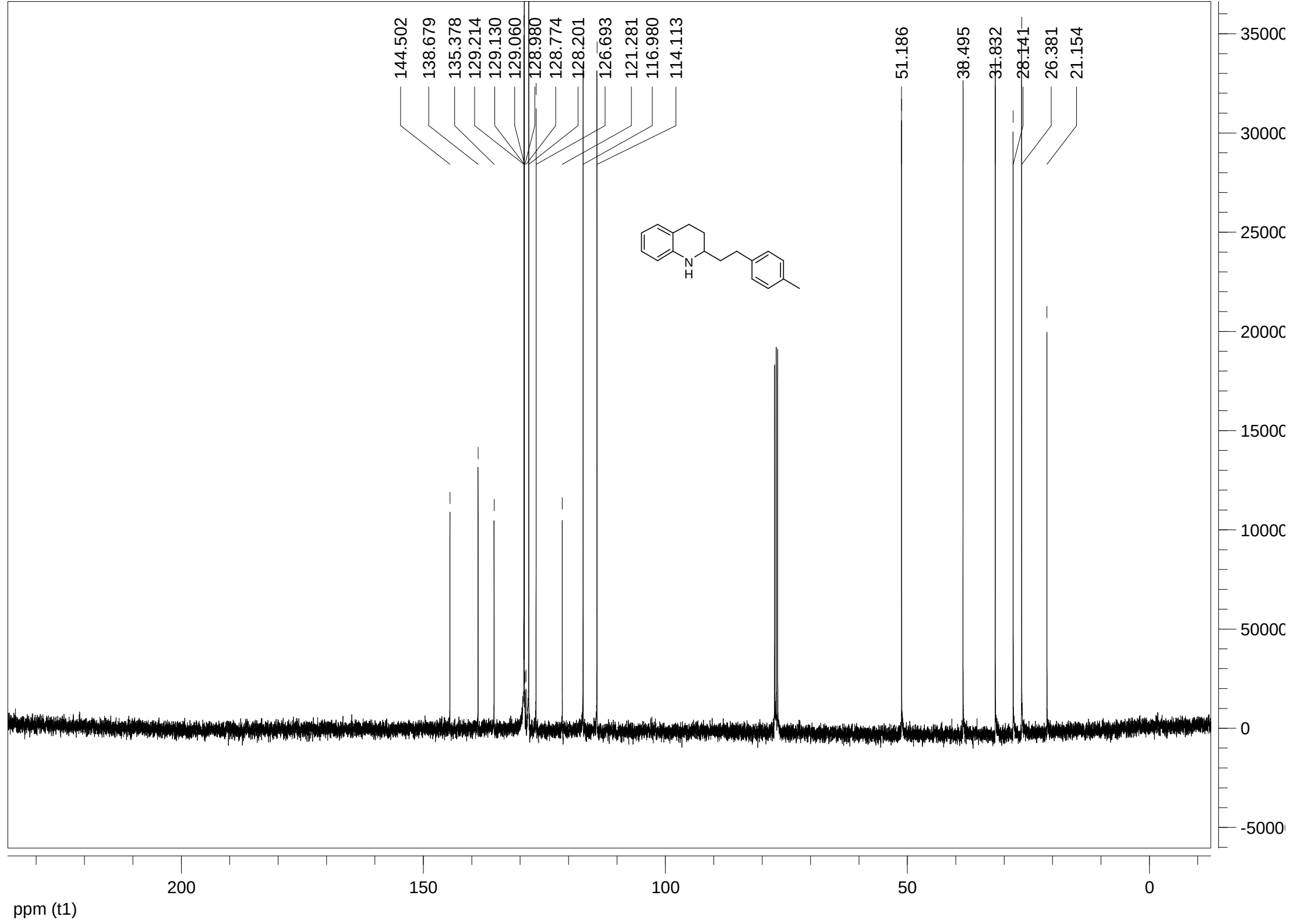
Data file: rk_mAnis_ch_ODH__9010_flow1_acq600.DATA
Method: HPLC2_ODH_9010_flow1_acq60
Date: 24.09.2009 02:31:22



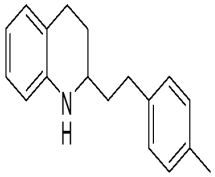
rk_mAnis_ch_ODH__9010_flow1_acq600.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	15,532	16,200	17,716	0,000	95,72	56878,7	37458,7	95,716
2	UNKNOWN	17,716	18,300	19,588	0,000	4,28	2191,1	1676,6	4,284
Total						100,00	59069,8	39135,3	100,000

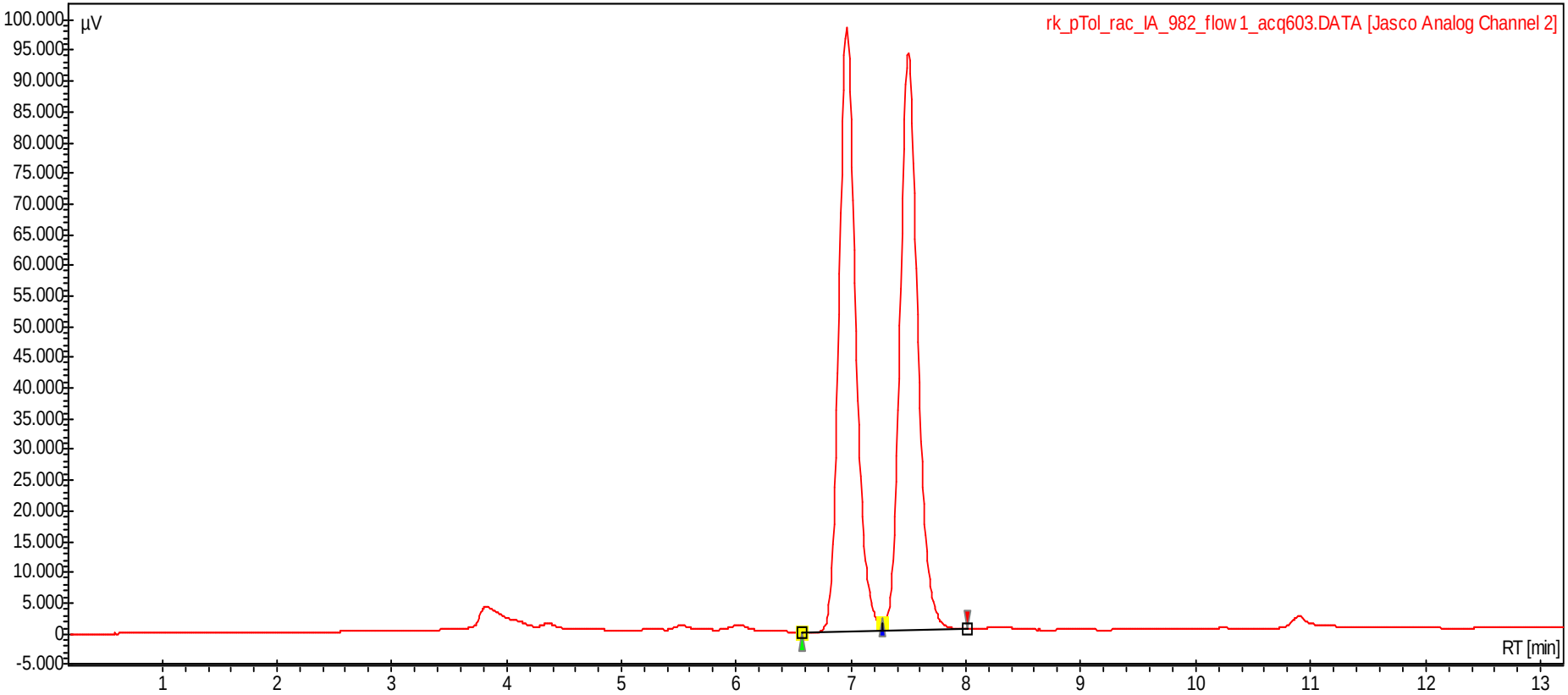

7.146
2.367
N
H
15000
10000
5000
0
4.21
2.25
1.04
1.08
0.90
1.14
4.74
3.33
1.16
2.23
1.11
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


144.502
138.679
135.378
129.214
129.130
129.060
128.980
128.774
128.201
126.693
121.281
116.980
114.113
51.186
38.495
31.832
28.141
26.381
21.154
N
H
35000
30000
25000
20000
15000
10000
50000
0
-5000
200
150
100
50
0
ppm (t1)

Chromatogram : rk_pTol_rac_IA_982_flow1_acq603



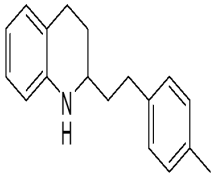
Data file: rk_pTol_rac_IA_982_flow1_acq603.DATA
Method: HPLC2_IA_982_flow1_acq60
Date: 24.09.2009 04:06:39



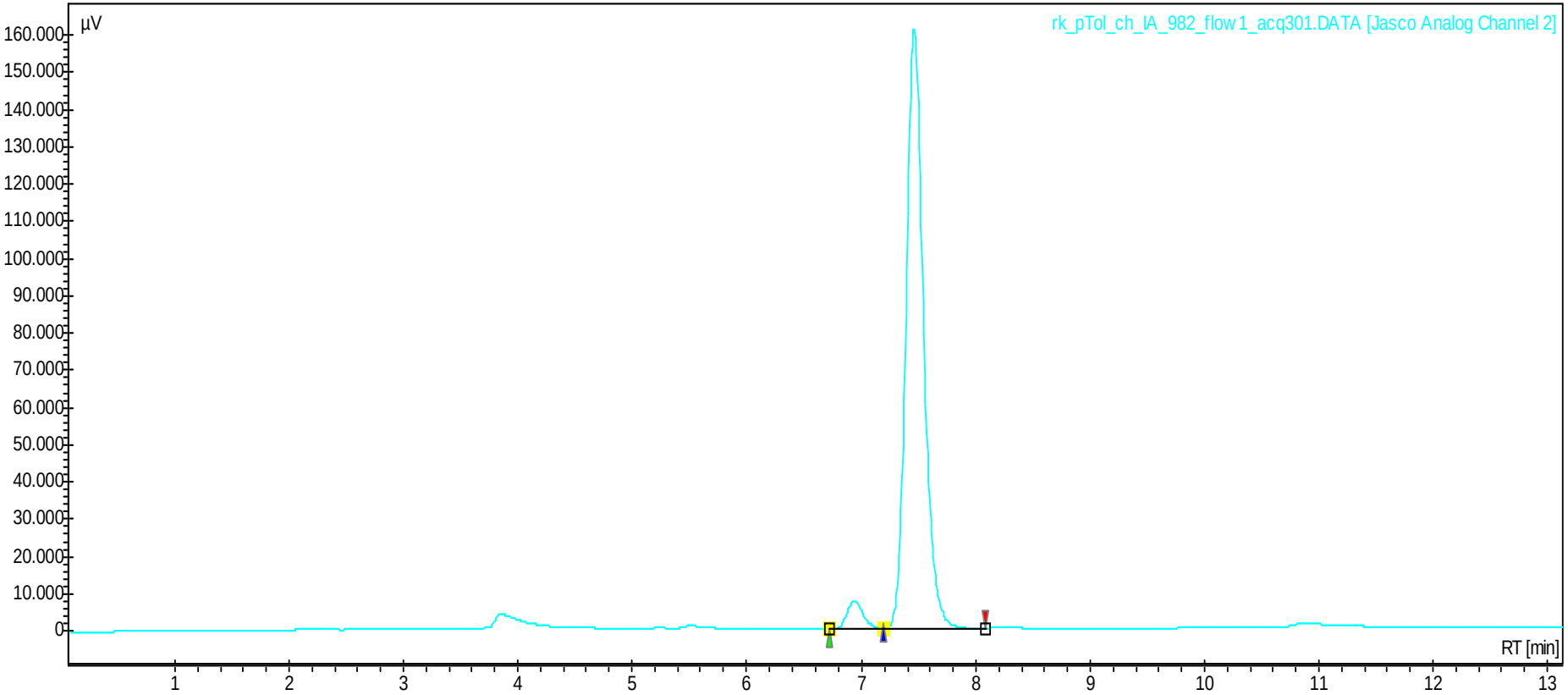
rk_pTol_rac_IA_982_flow1_acq603.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6,572	6,958	7,275	0,000	49,93	98434,4	16686,8	49,926
2	UNKNOWN	7,275	7,500	8,014	0,000	50,07	94128,0	16736,5	50,074
Total						100,00	192562,4	33423,3	100,000

Chromatogram : rk_pTol_ch_IA_982_flow1_acq301



Data file: rk_pTol_ch_IA_982_flow1_acq301.DATA
Method: HPLC2_IA_982_flow1_acq30
Date: 24.09.2009 08:58:11



rk_pTol_ch_IA_982_flow1_acq301.DATA [Jasco Analog Channel 2]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6,727	6,942	7,197	0,000	4,31	7726,0	1288,0	4,314
2	UNKNOWN	7,197	7,467	8,084	0,000	95,69	161258,6	28569,5	95,686
Total						100,00	168984,6	29857,5	100,000