SUPPORTING INFORMATION

Dual Metal and Lewis Base Catalysis Approach for Asymmetric Synthesis of Dihydroquinolines and the α-Arylation of Aldehydes via N-Acyliminium Ions

Chandra M. R Volla, Eleonora Fava, Iuliana Atodiresei and Magnus Rueping*

Corresponding Author: Prof. Dr. Magnus Rueping

Institute of Organic Chemistry

RWTH Aachen University

Landoltweg 1

D-52074 Aachen (Germany)

Tel: +49.241.8094710

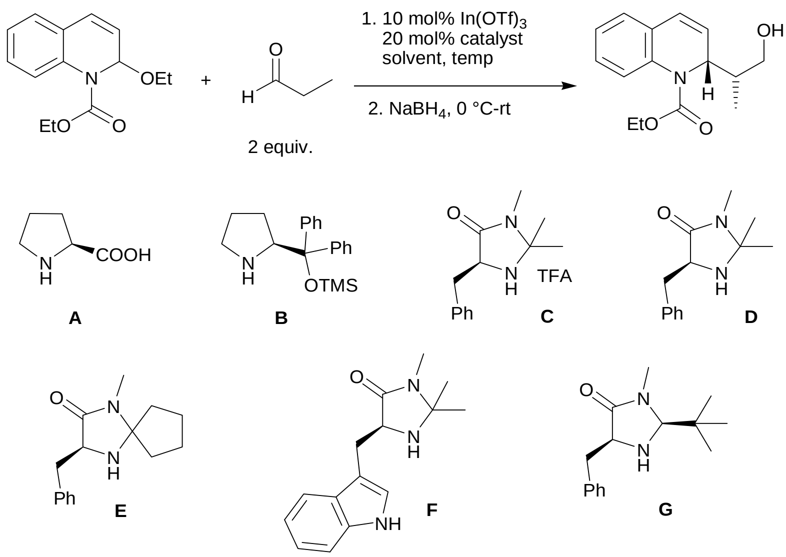
Fax: +49.241.8092665

E-mail: magnus.rueping@rwth-aachen.de

Methods: Unless otherwise stated, reactions were conducted in flame-dried glassware. Solvents after reactions and extraction were evaporated in a rotatory evaporator under vacuum. TLC for reaction monitoring was performed on 60 F_{254} (Merck) with detection by UV light and charring with $KMnO_4$ or Pancaldi reagent. ^{1}H and ^{13}C NMR spectra were recorded by using Mercury 300, Varian Inova 400 and Varian Inova 600 spectrometers for 300, 400 and 600 MHz respectively and are reported relative to Me_4Si (δ 0.0) or to the solvent residual ^{1}H-signal (CH-Cl_3, δ(H) 7.27, CH_2Cl_2 δ(H) 5.3). Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. Data for ^{13}C NMR spectra are reported in terms of chemical shift. IR spectra were recorded on a Perkin-Elmer-100 spectrometer and are reported in frequency of absorption (cm^{-1}). LC-MS mass spectra were measured on a LCQ FLEET instrument. The enantiomeric excesses were determined by HPLC analysis using a chiral stationary phase column (column, Daicel Co. CHIRALCEL OD-H, CHIRALPAK AD-H or CHIRALPAK AS-H; eluent: *n*hexane/ 2-propanol). The chiral HPLC methods were calibrated with the corresponding racemic mixtures. Optical rotations were measured on a Perkin Elmer 241 polarimeter.

Typical Experimental Procedure: In a screw-cap tube were placed 10 mol% of indium triflate, 20 mol% of imidazolidinone catalyst and 1.0 equiv. of the quinoline acetal. The tube was purged with argon and the septum was closed tightly. 1.0 mL of dry toluene was added to the tube and the mixture was stirred for 10 min at the temperature mentioned. Aldehyde (2.0 equiv.) was added slowly and the reaction continued to stir until complete disappearance of the quinoline acetal by TLC. Toluene was evaporated in a rotavap and 1.0 ml of methanol was added to the crude reaction mixture. $NaBH_4$ (2.0 equiv.) were added to the reaction at 0 °C. The reaction was allowed to reach room temperature and continued to stir for 2 h. Methanol was removed in a rotavap. Water was added to the reaction and the resulting mixture was extracted with ethyl acetate (3 times). The combined organic phase was dried over Na_2SO_4, the solvent was removed in a rotavap and the crude product was subjected to column chromatography over silica gel to get the pure product.

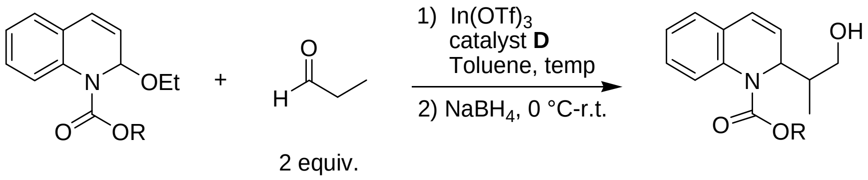
Table 1. Optimization of reaction conditions for the addition of propanal to quinoline acetal



Entry	Catalyst	Solvent	Temp (°C)	Time (h)	Yield[a] (%)	dr[b]	Ee[c] (%)
1	**A**	DCM	RT	6	62	50:50	-11
2	**B**	DCM	RT	6	74	50:50	38
3	**C**	DCM	RT	6	84	51:49	19
4	-	DCM	RT	3	-	-	-
5	**C**[d]	DCM	RT	24	-	-	-
6	**D**	DCM	RT	12	73	52:48	63
7	**E**	DCM	RT	12	72	51:49	57
8	**F**	DCM	RT	12	67	50:50	54
9	**D**	DCM	0	18	72	65:35	87
10	**G**	DCM	0	18	69	50:50	91[e]
11	**D**	$CHCl_3$	0	18	75	67:33	92
12	**D**	Toluene	0	18	78	76:24	94
13	**D**	Acetonitrile	0	18	62	60:40	47
14	**D**	THF	0	18	68	70:30	92
15	**D**	Ethanol	0	18	56	67:33	89

(a) Yield after column chromatography; (b) Diastereomeric ratio was determined by ^1H-NMR; (c) Enantiomeric excess of the major diastereomer was determined by chiral HPLC analysis; (d) Reaction was done in the absence of $In(OTf)_3$; (e) Enantiomeric excess of minor diastereomer.

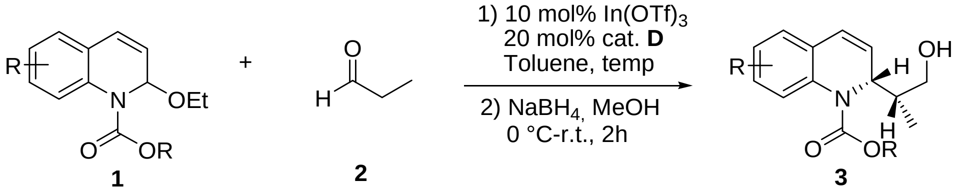
Table 2. Optimization of protecting groups for the addition of propanal to quinoline acetal



Entry	R	$In(OTf)_3$ (mol%)	Catalyst D (mol%)	Temp (°C)	dr[a]	Ee[b] (%)
1	**Et**	10	20	0	76:24	94
2	**Me**	10	20	0	65:35	-
3	***i*-Pr**	10	20	0	79:21	-
4	***i*-Bu**	10	20	0	80:20	93
5	***i*-Bu**	10	20	-10	80:20	96
6	***i*-Bu**	10	20	-20	-	-
7	***i*-Bu**	5	10	0	76:24	95
8	***i*-Bu**	10	10	-10	80:20	97
9	***i*-Bu**	20	20	0	81:19	94
10	***i*-Bu**	30	20	0	79:21	94
11	***i*-Bu**	20	20	-10	81:19	95
12	***i*-Bu**	30	20	-10	79:21	92

(a) Diastereomeric ratio was determined by ^{1}H-NMR; (b) Enantiomeric excess of the major diastereomer was determined by chiral HPLC analysis.

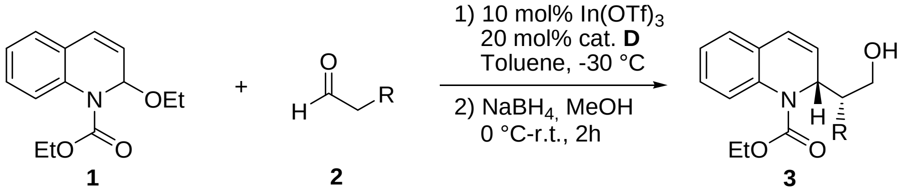
Table 3. Scope of the reaction using different quinoline acetals and propanal



Entry	R	R”	Temp (°C)	Product	Yield[a] (%)	dr[b]	Ee[c] (%) Major diast.	Ee[c] (%) Minor diast.
1	H	Et	0	**3a**	78	76:24	94	86
2	H	*i*-Bu	-10	**3b**	69	80:20	96	82
3[d]	H	*i*-Bu	-10	**3b**	67	80:20	97	82
4	4,7-dichloro	Et	0	**3c**	72	65:35	97	94
5	3-bromo	Et	0	**3d**	74	80:20	97	90
6	3-methyl	*i*-Bu	0	**3e**	81	79:21	94	92
7	6-methyl	Et	0	**3f**	76	65:35	96	82
8	6-methyl	*i*-Bu	-10	**3g**	85	77:23	97	92
9	5-nitro	Et	0	**3h**	78	70:30	90	89
10	6-chloro	Et	0	**3i**	67	80:20	95	84
11	6-bromo	Et	0	**3j**	81	75:25	91	71
12	6-methoxy	Et	0	**3k**	83	75:25	97	87

1.0 equiv. of quinolinium acetal and 2.0 equiv. of propionaldehyde in 1.0 mL of solvent for 18 h. (a) Yield after column chromatography; (b) Diastereomeric ratio was determined by ^{1}H-NMR. (c) Enantiomeric excess of the major diastereomer was determined by chiral HPLC analysis; (d) 10 mol% of $In(OTf)_3$ and 10 mol% of catalyst **D**.

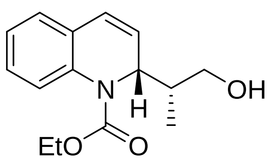
Table 4. Scope of the reaction using different aldehydes with quinoline acetal



Entry	R	Product	Yield[a] (%)	dr[b]	Ee[c] (%) Major diast.	Ee[c] (%) Minor diast.
1[d]	Me	**3a**	78	76:24	94	86
2[d]	Et	**3l**	73	60:40	71	-
2	Et	**3l**	78	65:35	89	66
3	*n*-Pr	**3m**	79	72:28	95	77
4[e]	*n*-Pr	**3n**	71	75:25	90	56
5	*n*-Bu	**3o**	83	75:25	94	68
6	Bn	**3p**	63	60:40	89	72
7	*n*-Hex	**3q**	72	65:35	91	65
8[e]	2-propenyl	**3r**	81	65:35	86	55

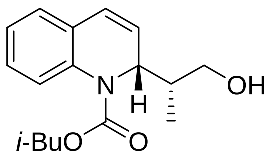
1.0 equiv. of quinolinium acetal and 3.0 equiv. of aldehyde in 1.0 mL of solvent for 72 h. (a) Yield after column chromatography; (b) Diastereomeric ratio was determined by ^{1}H-NMR. (c) Enantiomeric excess was determined by chiral HPLC analysis; (d) Reaction was done at 0 °C for 24 h; (e) The reaction was done using *i*-butyl derivative instead of ethyl.

Ethyl-2-(1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3a)



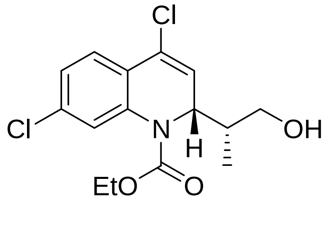
^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.03 (d, *J* = 6.9 Hz, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.45 - 1.74 (m, 1H), 3.32 (d, *J* = 11.4 Hz, 1H), 3.70 (dd, *J* = 11.6, 2.6 Hz, 1H), 4.14 - 4.42 (m, 2H), 4.81 (dd, *J* = 10.6, 6.0 Hz, 1H), 6.15 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.52 (d, *J* = 9.6 Hz, 1H), 7.04 - 7.11 (m, 2H), 7.15 - 7.23 (m, 1H), 7.38 (d, *J* = 7.4 Hz, 1H); **^{13}C NMR (75.0 MHz, $CDCl_3$):** 155.76, 133.87, 129.08, 127.43, 127.32, 126.25, 124.96, 124.52, 124.40, 63.76, 62.72, 53.75, 38.12, 14.38, 13.10; **MS (EI):** ($C_{15}H_{19}NO_3$), 261.2 (11, M^+), 202.2 (99, M^+ - 59), 158.2 (29, M^+ - (59 + 45)), 130.1 (71, M^+ - (59 + 73)); **IR (film):** 3469, 2971, 2928, 1695, 1487, 1458, 1402, 1323, 1276, 1130, 1039, 983, 764 cm^{-1}; $[\alpha]_D$ = +290.4 (c = 6.8, $CHCl_3$, 94% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 7.83 min, minor enantiomer: t_R = 13.28 min.

Isobutyl-2-(1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3b)



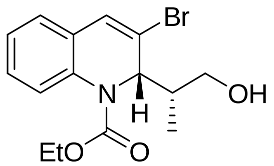
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.91 (d, *J* = 3.2 Hz, 3H), 0.93 (d, *J* = 3.2 Hz, 3H), 1.02 (d, *J* = 6.9 Hz, 3H), 1.54 - 1.67 (m, 1H), 1.96 (pd, *J* = 13.4, 6.9 Hz, 1H), 3.31 (d, *J* = 10.5 Hz, 1H), 3.68 (d, *J* = 9.8 Hz, 1H), 3.93 (dd, *J* = 10.4, 6.3 Hz, 1H), 4.06 (dd, *J* = 10.4, 6.9 Hz, 1H), 4.81 (dd, *J* = 10.6, 6.0 Hz, 1H), 6.14 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.51 (d, *J* = 9.6 Hz, 1H), 7.03 - 7.10 (m, 2H), 7.14 - 7.20 (m, 1H), 7.24 (bs, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 155.91, 133.82, 129.08, 127.40, 127.19, 126.21, 124.96, 124.52, 124.50, 72.91, 63.79, 53.81, 38.14, 27.83, 19.23, 19.11, 13.09; **MS (EI):** ($C_{17}H_{23}NO_3$), 289.3 (4, M^+), 230.3 (57, M^+ - 59), 130.2 (99, M^+ - (59 + 101)); **IR (film):** 3471, 2962, 2880, 1696, 1604, 1487, 1461, 1402, 1324, 1277, 1130, 1032, 981, 944, 764, 709, 577 cm^{-1}; $[\alpha]_D$ = +326.1 (c = 4.0, $CHCl_3$, 97% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 6.91 min, minor enantiomer: t_R = 10.78 min.

Ethyl 4,7-dichloro-2-(1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3c)



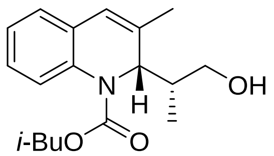
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.01 (d, *J* = 6.9 Hz, 3H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.59 - 1.71 (m, 1H), 3.34 (dd, *J* = 11.9, 2.5 Hz, 1H), 3.62 (dd, *J* = 11.9, 3.2 Hz, 1H), 4.21 - 4.39 (m, 2H), 4.89 (dd, *J* = 10.5, 6.7 Hz, 1H), 6.25 (d, *J* = 6.7 Hz, 1H), 7.13 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.41 (bs, 1H), 7.50 (d, *J* = 8.4 Hz, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 159.03, 135.37, 134.28, 127.36, 126.11, 125.60, 124.75, 124.46, 124.22, 63.52, 63.30, 55.03, 38.23, 14.28, 12.95; **MS (EI):** ($C_{15}H_{17}Cl_2NO_3$), 329.1 (5, M^+), 272.0 (66, (M^+ + 2) - 59), 270.0 (99, (M^+ - 59); **IR (film):** 3468, 2922, 1895, 1703, 1595, 1475, 1410, 1305, 1142, 1097, 1037, 997, 938, 828, 763, 692 cm^{-1}; $[\alpha]_D$ = +236.1 (c = 4.4, $CHCl_3$, 97% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 7.78 min, minor enantiomer: t_R = 18.96 min.

Ethyl 3-bromo-2-(1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3d)



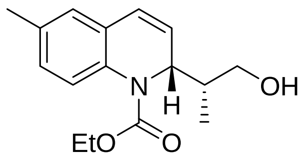
^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.19 (d, *J* = 7.0 Hz, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.70 - 1.83 (m, 1H), 3.33 (t, *J* = 9.5 Hz, 1H), 3.56 (d, *J* = 11.4 Hz, 1H), 4.18 - 4.39 (m, 2H), 5.07 (d, *J* = 9.7 Hz, 1H), 6.89 (s, 1H), 7.05 - 7.12 (m, 2H), 7.19 - 7.24 (m, 1H), 7.40 (bs, 1H); **^{13}C NMR (100.6 MHz, CDCl$_3$):** 154.72, 132.96, 127.83, 127.69, 127.35, 125.79, 124.85, 124.39, 119.37, 64.07, 62.98, 60.28, 37.75, 14.35; **MS (EI):** ($C_{15}H_{18}BrNO_3$), 341.1 (13, M^+ + 2), 339.0 (13, M^+), 282.1 (99, (M^+ + 2) - 59), 279.1 (99, M^+ - 59); **IR (film):** 3468, 2924, 1701, 1570, 1481, 1390, 1252, 1137, 1102, 1036, 939, 882, 823, 762, 697 cm^{-1}; $[\alpha]_D$ = +173.8 (c = 4.4, $CHCl_3$, 97% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 9.03 min, minor enantiomer: t_R = 11.45 min.

Isobutyl 2-(1-hydroxypropan-2-yl)-3-methylquinoline-1(2*H*)-carboxylate (3e)



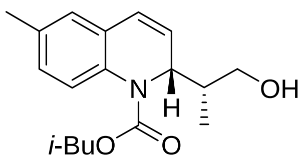
^{1}H NMR (600 MHz, CDCl$_3$): δ = 0.94 (t, *J* = 5.9 Hz, 6H), 1.06 (d, *J* = 7.0 Hz, 3H), 1.67 (bs, 1H), 1.92 - 2.01 (m, 1H), 2.04 (s, 3H), 2.84 (bs, 1H, OH), 3.30 (bs, 1H), 3.61 (d, *J* = 7.8 Hz, 1H), 3.94 (dd, *J* = 9.6, 6.6 Hz, 1H), 4.08 (dd, *J* = 9.9, 7.3 Hz, 1H), 4.69 (d, *J* = 9.9 Hz, 1H), 6.31 (d, *J* = 1.2 Hz, 1H), 7.05 - 7.07 (m, 2H), 7.11 - 7.17 (m, 1H), 7.36 (bs, 1H); **^{13}C NMR (150.9 MHz, CDCl$_3$):** 156.42, 138.06, 133.00, 128.22, 126.37, 125.60, 124.45, 123.94, 121.46, 72.82, 64.41, 57.55, 37.27, 27.85, 19.25, 19.13, 14.44; **MS (EI):** ($C_{18}H_{25}NO_3$), 303.4 (4, M^+), 244.3 (71, M^+ - 59), 144.2 (99, M^+ - (59 + 101)); **IR (film):** 3471, 2963, 1680, 1597, 1576, 1487, 1404, 1322, 1263, 1140, 1075, 1043, 983, 762, 589 cm^{-1}; [α]$_D$ = +196.4 (c = 4.0, $CHCl_3$, 94% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 6.42 min, minor enantiomer: t_R = 9.48 min.

Ethyl 2-(1-hydroxypropan-2-yl)-6-methylquinoline-1(2*H*)-carboxylate (3f)



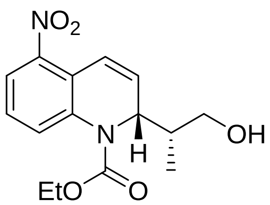
^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.02 (d, *J* = 6.9 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.52 - 1.68 (m, 2H), 2.29 (s, 3H), 3.05 - 3.22 (m, 1H), 3.29 (t, *J* = 10.1 Hz, 1H), 3.68 (d, *J* = 11.1 Hz, 1H), 4.13 - 4.27 (m, 1H), 4.27 - 4.38 (m, 1H), 4.77 (dd, *J* = 10.6, 6.0 Hz, 1H), 6.12 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.46 (d, *J* = 9.6 Hz, 1H), 6.90 (d, *J* = 1.6 Hz, 1H), 6.99 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.23 (bs, 1H); **^{13}C NMR (100.6 MHz, CDCl$_3$):** 155.83, 134.12, 131.22, 129.04, 128.00, 127.21, 126.71, 124.97, 124.16, 63.78, 62.63, 53.76, 38.01, 20.76, 14.38, 13.12; **MS (EI):** ($C_{16}H_{21}NO_3$), 275.2 (6, M^+), 216.2 (84, M^+ - 59), 144.2 (99, M^+ - (59 + 73)); **IR (film):** 3468, 2923, 1693, 1493, 1460, 1396, 1320, 1125, 1038, 984, 820, 766, 710 cm^{-1}; $[\alpha]_D$ = +294.7 (c = 3.6, $CHCl_3$, 96% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 7.64 min, minor enantiomer: t_R = 14.67 min.

Isobutyl 2-(1-hydroxypropan-2-yl)-6-methylquinoline-1(2*H*)-carboxylate (3g)



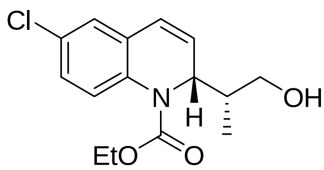
^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.94 (t, *J* =6.1 Hz, 6H), 1.02 (d, *J* = 6.8 Hz, 3H), 1.61 (bs, 1H), 1.90 - 2.01 (m, 1H), 2.30 (s, 3H), 3.19 (bs, 1H, OH), 3.31 (s, 1H), 3.68 (d, *J* = 10.4 Hz, 1H), 3.93 (dd, *J* = 10.1, 6.5 Hz, 1H), 4.07 (dd, *J* = 10.0, 7.2 Hz, 1H), 4.80 (dd, *J* = 10.6, 6.0 Hz, 1H), 6.13 (dd, *J* = 9.2, 6.1 Hz, 1H), 6.47 (d, *J* = 9.6 Hz, 1H), 6.90 (s, 1H), 7.00 (d, *J* = 8.2 Hz, 1H), 7.25 (bs, 1H); **^{13}C NMR (150.9 MHz, $CDCl_3$):** 156.08, 134.10, 131.23, 129.09, 127.90, 127.20, 126.70, 125.02, 124.28, 72.84, 63.81, 53.86, 38.07, 27.86, 20.76, 19.25, 19.13, 13.12; **MS (EI):** ($C_{18}H_{25}NO_3$), 303.2 (21, M^+), 244.2 (22, M^+ - 59), 144.1 (99, M^+ - (59 + 101)); **IR (film):** 3475, 2962, 2880, 1679, 1495, 1464, 1400, 1322, 1279, 1234, 1126, 1033, 981, 891, 819, 766, 710 cm^{-1}; $[\alpha]_D$ = +227.8 (c = 2.6, $CHCl_3$, 97% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 6.79 min, minor enantiomer: t_R = 11.73 min.

Ethyl 2-(1-hydroxypropan-2-yl)-5-nitroquinoline-1(2*H*)-carboxylate (3h)



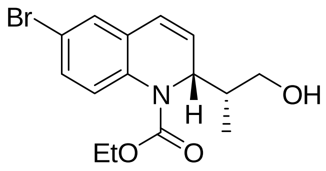
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.03 (d, *J* = 6.9 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.59 - 1.69 (m, 1H), 3.31 - 3.40 (m, 1H), 3.63 (td, *J* = 11.8, 3.7 Hz, 1H), 4.20 - 4.39 (m, 2H), 4.87 (dd, *J* = 10.6, 6.4 Hz, 1H), 6.40 (dd, *J* = 10.0, 6.3 Hz, 1H), 7.08 (d, *J* = 10.1 Hz, 1H), 7.30 (t, *J* = 8.2 Hz, 1H), 7.65 (d, *J* = 7.0 Hz, 1H), 7.70 (dd, *J* = 8.2, 1.1 Hz, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 155.05, 146.18, 135.79, 133.35, 129.50, 126.76, 121.90, 120.55, 119.87, 63.78, 63.24, 53.22, 38.16, 14.29, 12.91; **MS (EI):** ($C_{15}H_{18}N_2O_5$), 306.1 (6, M^+), 247.1 (99, M^+ - 59), 175.1 (65, M^+ - (59 + 73)); **IR (film):** 3443, 2969, 2923, 1704, 1528, 1468, 1385, 1291, 1217, 1136, 1063, 987, 913, 764, 706 cm^{-1}; $[\alpha]_D$ = +331.2 (c = 4.6, $CHCl_3$, 90% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 14.51 min, minor enantiomer: t_R = 20.44 min.

Ethyl 6-chloro-2-(1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3i)



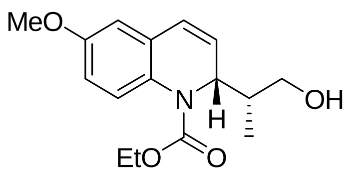
^{1}H NMR (600 MHz, $CDCl_3$): δ = 1.01 (d, *J* = 6.8 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.60 (s, 1H), 3.32 (d, *J* = 11.1 Hz, 1H), 3.64 (d, *J* = 11.0 Hz, 1H), 4.17 - 4.27 (m, 1H), 4.27 - 4.36 (m, 1H), 4.82 (dd, *J* = 10.4, 6.0 Hz, 1H), 6.15 - 6.23 (m, 1H), 6.45 (d, *J* = 9.6 Hz, 1H), 7.07 (s, 1H), 7.13 (d, *J* = 8.5 Hz, 1H), 7.30 (bs, 1H); **^{13}C NMR (150.9 MHz, $CDCl_3$):** 155.46, 132.42, 130.48, 129.64, 128.83, 127.15, 125.87, 125.65, 124.09, 63.70, 62.88, 53.82, 38.27, 14.33, 12.96; **MS (EI):** ($C_{15}H_{18}ClNO_3$), 295.1 (10, M^+), 236.2 (95, M^+ - 59), 164.1 (99, M^+ - (59 + 73)); **IR (film):** 3466, 2926, 1699, 1481, 1396, 1282, 1242, 1135, 1092, 1038, 985, 880, 822, 764, 712, 660 cm^{-1}; $[\alpha]_D$ = +248.5 (c = 7.3, $CHCl_3$, 95% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 8.91 min, minor enantiomer: t_R = 13.02 min.

Ethyl 6-bromo-2-(1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3j)



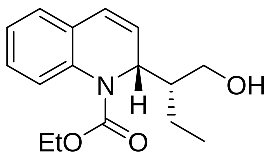
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.99 (d, *J* = 6.9 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.59 (s, 1H), 3.32 (s, 1H), 3.62 (d, *J* = 10.1 Hz, 1H), 4.15 - 4.38 (m, 2H), 4.81 (dd, *J* = 10.5, 6.0 Hz, 1H), 6.17 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.43 (d, *J* = 9.7 Hz, 1H), 7.18 - 7.33 (m, 3H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 155.39, 132.95, 130.45, 130.07, 129.23, 128.82, 126.00, 123.99, 117.33, 63.68, 62.90, 53.79, 38.32, 14.33, 12.96; **MS (EI):** ($C_{15}H_{18}BrNO_3$), 341.1 (10, $M^+ + 2$), 339.0 (10, M^+), 282.1 (99, ($M^+ + 2$) - 59), 279.1 (99, M^+ - 59); **IR (film):** 3469, 2972, 2928, 2881, 1695, 1479, 1391, 1315, 1134, 1080, 1038, 984, 879, 818, 762, 710 cm^{-1}; $[\alpha]_D$ = +204.8 (c = 6.5, $CHCl_3$, 91% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 9.31 min, minor enantiomer: t_R = 14.03 min.

Ethyl 2-(1-hydroxypropan-2-yl)-6-methoxyquinoline-1(2*H*)-carboxylate (3k)



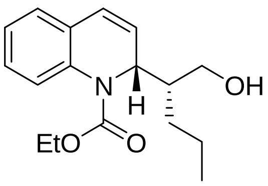
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.02 (d, *J* = 6.9 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.52 - 1.64 (m, 1H + OH), 3.30 (s, 1H), 3.68 (d, *J* = 12.0 Hz, 1H), 3.78 (s, 3H), 4.12 - 4.25 (m, 1H), 4.26 - 4.38 (m, 1H), 4.78 (dd, *J* = 10.7, 6.0 Hz, 1H), 6.16 (dd, *J* = 9.5, 6.1 Hz, 1H), 6.46 (d, *J* = 9.7 Hz, 1H), 6.62 (d, *J* = 2.9 Hz, 1H), 6.74 (dd, *J* = 8.9, 2.9 Hz, 1H), 7.26 (bs, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 156.37, 155.80, 129.87, 128.38, 125.44, 124.96, 114.96, 113.03, 110.83, 63.82, 62.59, 55.42, 53.79, 37.91, 14.40, 13.12; **MS (EI):** ($C_{16}H_{21}NO_4$), 291.1 (16, M^+), 232.1 (99, M^+ - 59), 160.0 (63, M^+ - (59 + 73)); **IR (film):** 3461, 2965, 2933, 1670, 1609, 1495, 1399, 1377, 1301, 1264, 1233, 1162, 1123, 1031, 763, 710 cm^{-1}; $[\alpha]_D$ = +283.7 (c = 7.3, $CHCl_3$, 97% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 10.97 min, minor enantiomer: t_R = 22.95 min.

Ethyl 2-(1-hydroxybutan-2-yl)quinoline-1(2*H*)-carboxylate (3l)



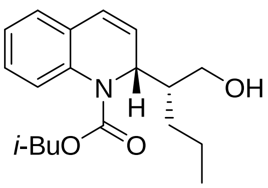
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.8 Hz, 3H), 1.30 (dt, *J* = 7.1, 0.8 Hz, 3H), 1.34 - 1.37 (m, 1H), 1.38 - 1.50 (m, 1H), 1.52 - 1.68 (m, 1H), 3.50 (t, *J* = 10.8 Hz, 1H), 3.61 (d, *J* = 12.0 Hz, 1H), 4.16 - 4.27 (m, 1H), 4.28 - 4.39 (m, 1H), 4.84 (dd, *J* = 10.8, 6.1 Hz, 1H), 6.18 (dd, *J* = 9.6, 6.1 Hz, 1H), 6.51 (d, *J* = 9.6 Hz, 1H), 7.02 - 7.12 (m, 2H), 7.15 - 7.21 (m, 1H), 7.35 (d, *J* = 4.0 Hz, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 155.86, 133.76, 129.26, 127.48, 127.29, 126.24, 124.89, 124.55, 124.45, 62.76, 59.05, 52.92, 44.85, 18.91, 14.35, 11.71; **MS (EI):** ($C_{16}H_{21}NO_3$), 275.2 (5, M^+), 202.2 (99, M^+ - 73), 158.2 (32, M^+ - (73 + 45)), 130.2 (93, M^+ - (73 + 73)); **IR (film):** 3466, 3018, 2925, 1671, 1407, 1328, 1215, 1036, 758, 669 cm^{-1}; $[\alpha]_D$ = +350.0 (c = 3.0, $CHCl_3$, 89% ee) ; **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 7.41 min, minor enantiomer: t_R = 18.76 min.

Ethyl 2-(1-hydroxypentan-2-yl)quinoline-1(2*H*)-carboxylate (3m)



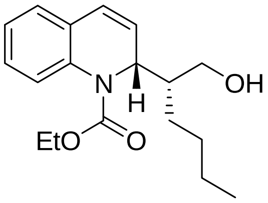
^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.84 (t, *J* = 7.3 Hz, 3H), 1.07 - 1.20 (m, 1H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.23 - 1.28 (m, 1H), 1.35 - 1.51 (m, 2H), 1.53 - 1.66 (m, 1H), 3.45 (t, *J* = 10.5 Hz, 1H), 3.60 (d, *J* = 12.0 Hz, 1H), 4.15 - 4.27 (m, 1H), 4.28 - 4.39 (m, 1H), 4.83 (dd, *J* = 10.8, 6.1 Hz, 1H), 6.18 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.51 (d, *J* = 9.6 Hz, 1H), 7.05 - 7.12 (m, 2H), 7.15 - 7.21 (m, 1H), 7.35 (d, *J* = 3.9 Hz, 1H); **^{13}C NMR (150.9 MHz, $CDCl_3$):** 155.90, 133.74, 129.26, 127.48, 127.28, 126.25, 124.87, 124.55, 124.44, 62.76, 59.58, 52.98, 43.10, 28.32, 20.42, 14.35, 14.30; **MS (EI):** ($C_{17}H_{23}NO_3$), 289.3 (7, M^+), 202.2 (99, M^+ - 87), 158.2 (29, M^+ - (87 + 45)), 130.2 (76, M^+ - (87 + 73)); **IR (film):** 3475, 2958, 2929, 2872, 1675, 1603, 1572, 1489, 1460, 1404, 1325, 1277, 1208, 1131, 1047, 919, 765, 710 cm^{-1}; $[\alpha]_D$ = +250.6 (c = 1.8, $CHCl_3$, 95% ee) ; **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 7.11 min, minor enantiomer: t_R = 21.86 min.

Isobutyl 2-(1-hydroxypentan-2-yl)quinoline-1(2*H*)-carboxylate (3n)



^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85 (t, *J* = 7.3 Hz, 3H), 0.90 - 0.98 (m, 6H), 1.10 - 1.20 (m, 1H), 1.27 - 1.34 (m, 1H), 1.37 - 1.52 (m, 2H), 1.55 - 1.71 (m, 1H), 1.97 (pd, *J* = 13.3, 6.7 Hz, 1H), 3.21 (bs, 1H, OH), 3.47 (d, *J* = 11.7 Hz, 1H), 3.62 (d, *J* = 11.9 Hz, 1H), 3.95 (dd, *J* = 10.3, 6.4 Hz, 1H), 4.08 (dd, *J* = 10.4, 7.0 Hz, 1H), 4.86 (dd, *J* = 10.8, 6.0 Hz, 1H), 6.20 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.53 (d, *J* = 9.6 Hz, 1H), 7.07 - 7.13 (m, 2H), 7.17 - 7.21 (m, 1H), 7.36 (bs, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 156.10, 133.71, 129.28, 127.48, 127.18, 126.24, 124.89, 124.59, 124.57, 72.98, 59.64, 53.07, 43.12, 28.37, 27.83, 20.42, 19.22, 19.10, 14.29; **MS (EI):** ($C_{19}H_{27}NO_3$), 317.1 (3, M^+), 230.0 (96, M^+ - 87), 130.0 (99, M^+ - (87 + 101)); **IR (film):** 3455, 2958, 2929, 2874, 1679, 1488, 1462, 1402, 1319, 1274, 1245, 1127, 1028, 763, 711, 666 cm^{-1}; $[\alpha]_D$ = +242.4 (c = 3.7, $CHCl_3$, 90% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 6.82 min, minor enantiomer: t_R = 14.60 min.

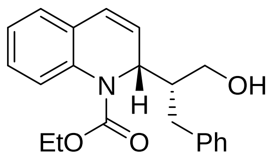
Ethyl 2-(1-hydroxyhexan-2-yl)quinoline-1(2*H*)-carboxylate (3o)



^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.85 (t, *J* = 7.3 Hz, 3H), 1.05 - 1.17 (m, 1H), 1.21 - 1.29 (m, 2H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.29 - 1.35 (m, 1H), 1.35 - 1.45 (m, 2H), 1.57 - 1.67 (m, 1H), 3.28 (bs, 1H, OH), 3.47 (t, *J* = 10.9 Hz, 1H), 3.62 (d, *J* = 12.1 Hz, 1H), 4.18 - 4.29 (m, 1H), 4.30 - 4.38 (m, 1H), 4.84 (dd, *J* = 10.8, 6.0 Hz, 1H), 6.20 (dd, *J* = 9.4, 6.1 Hz, 1H), 6.53 (d, *J* = 9.6 Hz, 1H), 7.07 - 7.14 (m, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.34 (bs, 1H); **^{13}C NMR (150.9 MHz, $CDCl_3$):** 156.02, 133.68, 129.33, 127.48, 127.30, 126.28, 124.87, 124.58, 124.45, 62.81, 59.55, 53.00, 43.24, 29.49, 25.79, 22.96, 14.38, 14.01; **MS (EI):** ($C_{18}H_{25}NO_3$), 303.2 (6, M^+), 202.2 (99, M^+ - 101), 158.2

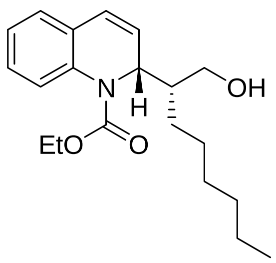
(26, M^+ - (101 + 45)), 130.2 (68, M^+ - (101 + 73)); **IR (film):** 3476, 2929, 2868, 1680, 1484, 1401, 1322, 1128, 1039, 901, 764 cm^{-1}; $[\alpha]_D$ = +178.7 (c = 1.6, $CHCl_3$, 94% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 6.87 min, minor enantiomer: t_R = 18.47 min.

Ethyl 2-(1-hydroxy-3-phenylpropan-2-yl)quinoline-1(2*H*)-carboxylate (3p)



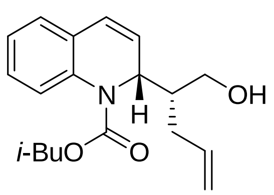
^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.30 (t, *J* = 7.1 Hz, 3H), 1.60 (bs, 1H, OH), 1.67 (t, *J* = 10.9 Hz, 1H), 2.71 - 2.88 (m, 2H), 3.12 - 3.23 (m, 1H), 3.50 (d, *J* = 11.3 Hz, 1H), 4.24 (qd, *J* = 10.7, 7.1 Hz, 1H), 4.35 (qd, *J* = 10.8, 7.1 Hz, 1H), 4.96 (dd, *J* = 10.8, 6.1 Hz, 1H), 6.28 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.60 (d, *J* = 9.6 Hz, 1H), 7.05 - 7.23 (m, 8H), 7.33 (d, *J* = 8.5 Hz, 1H); **^{13}C NMR (100.6 MHz, CDCl$_3$):** 155.81, 140.13, 133.70, 129.29, 128.75, 128.22, 127.44, 127.31, 126.39, 125.89, 125.27, 124.64, 124.40, 62.88, 58.57, 52.76, 45.91, 32.31, 14.34; **MS (EI):** ($C_{21}H_{23}NO_3$), 337.0 (2, M^+), 202.2 (99, M^+ - 135), 158.0 (23, M^+ - (135 + 45)), 130.0 (75, M^+ - (135 + 73)); **IR (film):** 3467, 2930, 1660, 1491, 1403, 1325, 1266, 1210, 1171, 1124, 1028, 943, 896, 833, 765, 698, 659 cm^{-1}; $[\alpha]_D$ = +123.1 (c = 1.6, $CHCl_3$, 89% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 13.29 min, minor enantiomer: t_R = 37.78 min.

Ethyl 2-(1-hydroxyoctan-2-yl)quinoline-1(2*H*)-carboxylate (3q)



^{1}H NMR (400 MHz, CDCl$_3$): δ = 0.83 (t, *J* = 6.8 Hz, 3H), 1.05 - 1.16 (m, 1H), 1.18 - 1.26 (m, 6H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.34 - 1.45 (m, 2H), 1.55 - 1.67 (m, 2H), 3.45 (t, *J* = 10.5 Hz, 1H), 3.60 (d, *J* = 11.9 Hz, 1H), 4.16 - 4.27 (m, 1H), 4.28 - 4.39 (m, 1H), 4.82 (dd, *J* = 10.7, 6.0 Hz, 1H), 6.18 (dd, *J* = 9.6, 6.0 Hz, 1H), 6.51 (d, *J* = 9.6 Hz, 1H), 7.04 - 7.12 (m, 2H), 7.15 - 7.21 (m, 1H), 7.34 (d, *J* = 4.9 Hz, 1H); **^{13}C NMR (100.6 MHz, CDCl$_3$):** 158.66, 133.75, 129.29, 127.48, 127.28, 126.25, 124.86, 124.54, 124.44, 62.76, 59.60, 53.00, 43.30, 31.75, 29.54, 27.27, 26.11, 22.57, 14.35, 14.03; **MS (EI):** ($C_{20}H_{29}NO_3$), 331.3 (4, M^+), 202.2 (99, M^+ - 129), 158.2 (21, M^+ - (129 + 45)), 130.2 (64, M^+ - (129 + 73)); **IR (film):** 3461, 2928, 2860, 1771, 1711, 1609, 1491, 1460, 1396, 1277, 1213, 1107, 1073, 1044, 951, 757, 724, 528 cm^{-1}; $[\alpha]_D$ = +299.3 (c = 4.2, $CHCl_3$, 91% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 6.57 min, minor enantiomer: t_R = 19.42 min.

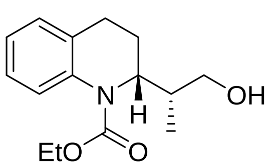
Isobutyl 2-(1-hydroxypent-4-en-2-yl)quinoline-1(2*H*)-carboxylate (3r)



^{1}H NMR (600 MHz, CDCl$_3$): δ = 0.93 (t, *J* = 5.9 Hz, 6H), 1.53 (s, 1H), 1.91 - 2.00 (m, 1H), 2.19 (d, *J* = 13.9 Hz, 1H), 2.32 (dd, *J* = 21.2, 10.4 Hz, 1H), 3.16 (bs, 1H, OH), 3.47 (t, *J* = 10.2 Hz, 1H), 3.62 (d, *J* = 12.0 Hz, 1H), 3.94 (dd, *J*

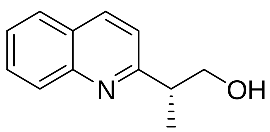
= 10.2, 6.4 Hz, 1H), 4.07 (dd, *J* = 10.2, 7.1 Hz, 1H), 4.90 (dd, *J* = 10.8, 6.0 Hz, 1H), 4.99 (d, *J* = 10.1 Hz, 1H), 5.07 (d, *J* = 17.0 Hz, 1H), 5.73 (dt, *J* = 17.0, 6.4 Hz, 1H), 6.18 (dd, *J* = 9.4, 6.1 Hz, 1H), 6.54 (d, *J* = 9.6 Hz, 1H), 7.06 - 7.13 (m, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.36 (bs, 1H); **^{13}C NMR (150.9 MHz, $CDCl_3$):** 155.97, 136.40, 133.71, 128.78, 127.37, 127.27, 126.29, 125.18, 124.64, 124.59, 116.72, 72.98, 59.86, 52.64, 43.32, 31.10, 27.81, 19.21, 19.09; **MS (EI):** ($C_{19}H_{25}NO_3$), 315.3 (2, M^+), 230.3 (88, M^+ - 85), 130.2 (64, M^+ - (85 + 101)); **IR (film):** 3474, 2962, 2881, 1676, 1490, 1465, 1406, 1325, 1276, 1131, 1023, 914, 765, 733 cm^{-1}; $[\alpha]_D$ = +288.7 (c = 3.5, $CHCl_3$, 84% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 7.56 min, minor enantiomer: t_R = 15.55 min.

Ethyl 2-(1-hydroxypropan-2-yl)-3,4-dihydroquinoline-1(2*H*)-carboxylate (4)



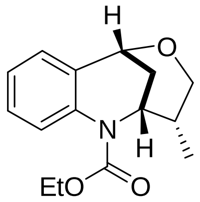
^{1}H NMR (600 MHz, $CDCl_3$): δ = 1.01 (d, *J* = 6.9 Hz, 3H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.44 - 1.54 (m, 1H), 1.76 (dt, *J* = 11.6, 5.5 Hz, 1H), 2.24 (dt, *J* = 13.2, 6.4 Hz, 1H), 2.67 (t, *J* = 6.9 Hz, 2H), 3.30 - 3.39 (m, 1H), 3.71 (d, *J* = 11.4 Hz, 1H), 4.17 (qd, *J* = 14.2, 7.1 Hz, 1H), 4.30 (qd, *J* = 14.2, 7.1 Hz, 1H), 4.39 - 4.46 (m, 1H), 7.04 (t, *J* = 7.4 Hz, 1H), 7.10 (d, *J* = 7.4 Hz, 1H), 7.14 (t, *J* = 7.7 Hz, 1H), 7.32 (d, *J* = 6.9 Hz, 1H); **^{13}C NMR (150.9 MHz, $CDCl_3$):** 156.32, 136.36, 131.90, 127.97, 126.02, 125.34, 124.52, 64.44, 62.36, 54.10, 37.47, 27.08, 24.63, 14.38, 13.63; **MS (EI):** ($C_{15}H_{21}NO_3$), 263.1 (28, M^+), 204.0 (96, M^+ - 59), 160.0 (22, M^+ - (59 + 45)), 132. (71, M^+ - (59 + 73)); **IR (film):** 3472, 3048, 2927, 1695, 1488, 1387, 1244, 1212, 1026, 936, 756 cm^{-1}; $[\alpha]_D$ = +86.2 (c = 2.9, $CHCl_3$, 93% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, major enantiomer: t_R = 8.17 min, minor enantiomer: t_R = 9.24 min.

2-(quinolin-2-yl)propan-1-ol (5)



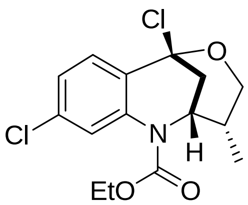
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.41 (d, *J* = 7.2 Hz, 3H), 3.18 - 3.28 (m, 1H), 3.96 (dd, *J* = 11.0, 6.4 Hz, 1H), 4.08 (dd, *J* = 11.0, 3.5 Hz, 1H), 7.32 (d, *J* = 8.5 Hz, 1H), 7.50 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H), 7.68 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.78 (dd, *J* = 8.1, 1.4 Hz, 1H), 8.00 (ddd, *J* = 8.5, 1.8, 0.8 Hz, 1H), 8.11 (dd, *J* = 8.5, 0.6 Hz, 1H); **^{13}C NMR (100.6 MHz, $CDCl_3$):** 165.61, 147.00, 136.80, 129.59, 128.82, 127.47, 126.80, 126.07, 120.70, 66.64, 42.23, 17.39; **MS (EI):** ($C_{12}H_{13}NO$), 187.4 (14, M^+), 172.3 (63, M^+ - 15), 170.3 (99, M^+ - 17), 156.0 (91, M^+ - 31); **IR (film):** 3389, 2925, 1668, 1603, 1566, 1503, 1457, 1429, 1382, 1309, 1218, 1037, 977, 832, 756, 664, 621, 480 cm^{-1}; $[\alpha]_D$ = +2.7 (c = 3.6, $CHCl_3$, 92% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 97/03, flow rate = 1.0 mL/min, minor enantiomer: t_R = 23.26 min, major enantiomer: t_R = 24.20 min.

Ethyl 3,4-benzo-8-methyl-6-oxa-2-aza-bicyclo[3.3.1]nonane-2-carboxylate (6a)



^{1}H NMR (600 MHz, CDCl$_3$): δ = 0.82 (d, *J* = 6.8 Hz, 3H), 1.34 (t, *J* = 7.1 Hz, 3H), 2.00 (d, *J* = 13.2 Hz, 1H), 2.06 - 2.15 (m, 1H), 2.24 (td, *J* = 13.2, 3.0 Hz, 1H), 2.75 (t, *J* = 12.1 Hz, 1H), 3.43 (dd, *J* = 12.0, 6.2 Hz, 1H), 4.21 - 4.30 (m, 2H), 4.74 (s, 1H), 4.83 (d, *J* = 3.1 Hz, 1H), 7.20 - 7.26 (m, 1H), 7.04 (t, *J* = 7.3 Hz, 1H), 7.28 (t, *J* = 7.9 Hz, 1H), 8.23 (d, *J* = 8.6 Hz, 1H); **^{13}C NMR (150.9 MHz, CDCl$_3$):** 155.03, 140.18, 130.38, 128.85, 124.45, 123.10, 120.57, 68.20, 63.66, 62.00, 51.07, 35.06, 29.64, 14.42, 13.68; **MS (EI):** ($C_{15}H_{19}NO_3$), 262.1 (13, M^+ + 1), 261.0 (99, M^+), 202.0 (47, M^+ - 59), 130.0 (64, M^+ - (59 + 73)); **IR (film):** 2954, 1706, 1599, 1480, 1384, 1301, 1213, 1131, 1050, 979, 937, 862, 761, 595, 505 cm^{-1}; $[\alpha]_D$ = +21.4 (c = 6.2, $CHCl_3$, 93% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 97/03, flow rate = 0.5 mL/min, minor enantiomer: t_R = 19.56 min, major enantiomer: t_R = 25.13 min.

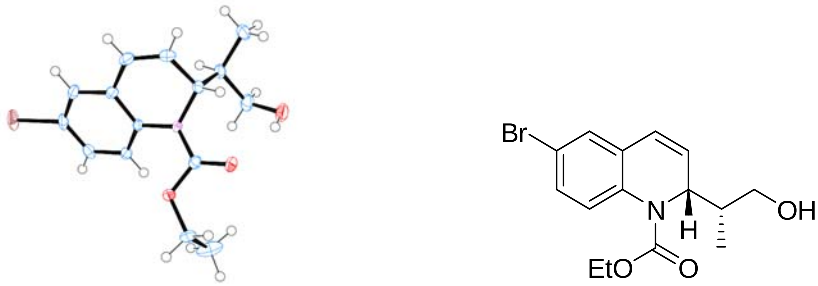
Ethyl 3,4-(3-chloro-benzo)-5-chloro-8-methyl-6-oxa-2-aza-bicyclo[3.3.1]nonane-2-carboxylate (6c)



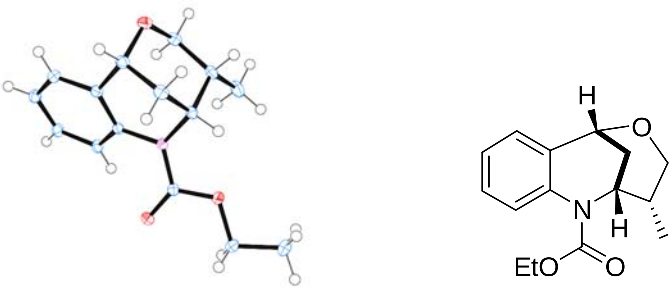
^{1}H NMR (400 MHz, CDCl$_3$): δ = 0.82 (d, *J* = 6.8 Hz, 3H), 1.34 (t, *J* = 7.1 Hz, 3H), 2.07 - 2.21 (m, 1H), 2.59 (d, *J* = 3.5 Hz, 2H), 2.79 (t, *J* = 12.3 Hz, 1H), 3.71 (dd, *J* = 12.3, 6.4 Hz, 1H), 4.24 - 4.33 (m, 2H), 4.88 (q, *J* = 3.6 Hz, 1H), 7.10 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.75 (d, *J* = 8.5 Hz, 1H), 8.22 (d, *J* = 2.1 Hz, 1H); **^{13}C NMR (100.6 MHz, CDCl$_3$):** 154.30, 139.54, 135.60, 129.43, 123.88, 123.72, 120.75, 93.96, 68.54, 62.71, 54.10, 40.25, 33.44, 14.34, 12.75; **MS (EI):** ($C_{15}H_{17}Cl_2NO_3$), 333.0 (4, M^+ + 4), 331.0 (15, M^+ + 2), 330.4 (18, M^+ + 1), 328.8 (42, M^+), 179.8 (99, M^+ - 149); **IR (film):** 2927, 2856, 1714, 1597, 1564, 1477, 1410, 1375, 1306, 1219, 1201, 1137, 1043, 934, 876, 824, 764, 601 cm^{-1}; $[\alpha]_D$ = +40.7 (c = 2.7, $CHCl_3$, 93% ee); **HPLC conditions:** AD-H column, *n*-hexane/2-propanol = 97/03, flow rate = 1.0 mL/min, minor enantiomer: t_R = 13.98 min, major enantiomer: t_R = 15.63 min.

X-Ray crystal structure analysis.#

Major diastereomer – (R)-Ethyl 6-bromo-2-((R)-1-hydroxypropan-2-yl)quinoline-1(2*H*)-carboxylate (3j)



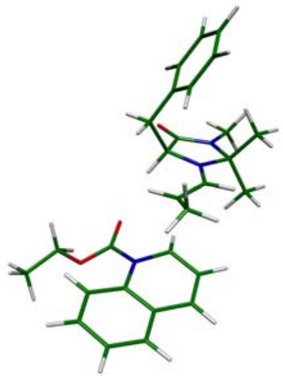
Ethyl 3,4-benzo-8-methyl-6-oxa-2-aza-bicyclo[3.3.1]nonane-2-carboxylate (6a)



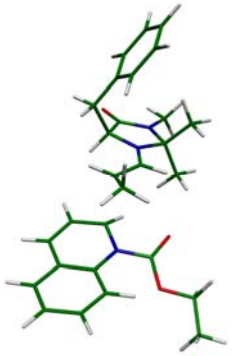
CCDC 1414145-1414146 contain the supplementary crystallographic data for this publication. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

DTF Calculations

The geometry optimization and frequency calculations of the transition states TS1 and TS2 (only the most stable structures are shown) were performed using Gaussian09 program package[1-3] at the B3LYP/6-31G* level.[4,5] The thermal corrections were calculated at 298 K. Solvent effects (toluene) were taken into account at the PCM/B3LYP/6-31G*//B3LYP/6-31G* level.[6] The corrections for dispersion interactions were estimated with the DFT-D3 program developed by Grimme.[7]

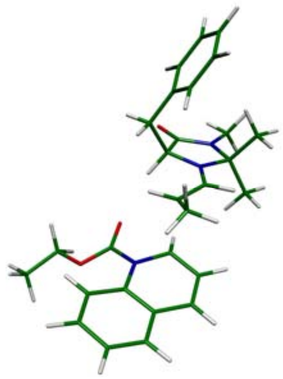


TS1 (0.0 kcal/mol)



TS2 (1.29 kcal/mol)

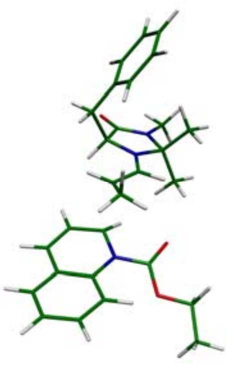
ΔG_{TS1} = -1476.874595 a.u.; ΔG_{TS2} = -1476.872537 a.u.



TS1

N	-1.70671800	-0.30969400	0.71018000
C	-1.79068100	0.94570400	-0.05274500
C	-2.83148300	1.74050800	0.72844100
N	-3.28600100	0.96581600	1.75719400
C	-2.69895000	-0.37165800	1.83720300
C	-2.19691700	0.81209300	-1.54701000
C	-3.46764400	0.03486500	-1.81456600
C	-4.72612300	0.63972800	-1.67003300
C	-5.89417500	-0.08716600	-1.90475200
C	-5.82389700	-1.42726700	-2.29295700
C	-4.57775800	-2.03570100	-2.45165000
C	-3.41047400	-1.30741600	-2.21438800
O	-3.19725300	2.86873800	0.44890600
C	-4.33859100	1.42702200	2.64909600
C	-3.74542200	-1.47217700	1.59868000
C	-1.97402000	-0.56897000	3.17984700
H	-0.83321200	1.47535400	-0.00133200
H	-5.23408200	0.80272900	2.56390300
H	-4.58391700	2.44541200	2.34410200
H	-4.00337800	1.43718600	3.69136600
H	-4.47833000	-1.47389200	2.41106000
H	-4.26420400	-1.31518800	0.65079600
H	-3.27447300	-2.46054100	1.58575800
H	-2.69113800	-0.53140600	4.00492200
H	-1.22792900	0.21686000	3.33352500
H	-1.48473500	-1.54727600	3.22535700

H	-2.29817500	1.84640800	-1.89449800
H	-1.36671300	0.36862200	-2.10531400
H	-2.44222100	-1.78358700	-2.35515600
H	-4.51377100	-3.07283700	-2.76945900
H	-6.73388700	-1.98894900	-2.48391400
H	-6.86028800	0.39805500	-1.79667300
H	-4.78717100	1.68478700	-1.37873100
C	-0.85153700	-1.30508300	0.46636100
H	-1.03404000	-2.20826500	1.04690400
C	0.26300300	-1.29257500	-0.37014400
H	0.40076500	-0.42788700	-1.01234300
C	0.84745800	-2.59540000	-0.86145100
H	0.31948700	-2.95022500	-1.75641300
H	1.90126000	-2.48963400	-1.13500300
H	0.77402700	-3.38502500	-0.10545600
H	1.54187000	-2.06028200	2.57003200
C	2.23797500	-1.66527500	1.83819600
H	3.75938700	-3.11459600	2.18701900
C	3.44775500	-2.24488600	1.61555700
N	2.79452200	0.12934500	0.31859600
C	4.32778200	-1.73613800	0.60281000
C	1.84863100	-0.50720400	1.10649100
C	3.97693200	-0.54953100	-0.09829800
C	5.50761200	-2.42176700	0.25127200
H	1.11032200	0.16970400	1.50715600
H	4.49914600	0.74571200	-1.74918300
C	6.30481600	-1.97504300	-0.78807000
H	5.76894900	-3.32148000	0.80101400
H	7.21018100	-2.51036400	-1.05493600
C	5.91916800	-0.83581900	-1.50708300
H	6.52131400	-0.49421400	-2.34336600
C	4.76900000	-0.12768000	-1.17534500
C	2.49097100	1.51105500	0.05175100
O	1.37892700	1.95373800	0.24795300
O	3.54145600	2.19991400	-0.35411900
C	3.31855200	3.63841100	-0.56990600
H	2.91756300	4.05168600	0.35848200
H	2.56175300	3.74368400	-1.35140600
C	4.64908400	4.24819100	-0.95364300
H	5.03224600	3.82083300	-1.88530700
H	4.51617700	5.32454000	-1.10335100
H	5.39315400	4.10176800	-0.16525500



TS2

N	-1.70767800	0.37766700	-0.28322400
C	-2.30787700	-0.60215800	-1.19814400
C	-3.35450400	0.22696300	-1.93985800
N	-3.35674800	1.48486100	-1.41580600
C	-2.37914900	1.72308800	-0.35238600
C	-2.95843400	-1.85596100	-0.54821100
C	-3.97186300	-1.57217700	0.53984900
C	-5.29519000	-1.23112300	0.21966200
C	-6.22055300	-0.95622300	1.22755300
C	-5.84082700	-1.02417300	2.57011100
C	-4.53084300	-1.37515900	2.90035000
C	-3.60575900	-1.64727500	1.89080700
O	-4.07131500	-0.20074700	-2.82778000
C	-4.29459700	2.49849900	-1.87440800
C	-3.05811200	2.06836100	0.98166500
C	-1.37183900	2.80660400	-0.77297500
H	-1.55957600	-0.94264000	-1.92592900
H	-4.96524300	2.81096400	-1.06741900
H	-4.88387500	2.04412000	-2.67216200
H	-3.77180600	3.37523900	-2.26994800
H	-3.57569800	3.02882100	0.90082900
H	-3.77857300	1.29674300	1.26142000
H	-2.31508300	2.16769700	1.77948300
H	-1.87576100	3.77419800	-0.85564300
H	-0.93379800	2.56199500	-1.74596300
H	-0.56692900	2.91360000	-0.04131700
H	-3.43064300	-2.38191600	-1.38556800
H	-2.17309400	-2.51259300	-0.16215200
H	-2.59116200	-1.93695700	2.15651200
H	-4.23174700	-1.44745600	3.94245300
H	-6.56482100	-0.81867400	3.35342100
H	-7.24321400	-0.70158700	0.96352000
H	-5.60027300	-1.18777100	-0.82243100
C	-0.66376200	0.15231900	0.51460100
H	-0.41309200	0.97729100	1.17561100
C	0.18612600	-0.95641600	0.51917900
H	-0.11322200	-1.82815300	-0.05348200
C	1.02990400	-1.23492800	1.74130200
H	0.44119900	-1.73307500	2.52286300
H	1.86888600	-1.89543000	1.50694900
H	1.42792000	-0.31079800	2.17543800
H	0.91788000	-1.45500300	-2.55718500
C	1.76852200	-1.32165400	-1.89761700
H	2.87846900	-2.97238600	-2.65125900
C	2.84107700	-2.15240800	-1.93989900
N	2.89680600	0.09318700	-0.29179000
C	3.94192900	-1.97489900	-1.03452500
C	1.73632800	-0.22197700	-0.98274000
C	3.95784400	-0.85243100	-0.16303300
C	4.98530400	-2.91899600	-0.96309600
H	1.13616600	0.64256400	-1.21682400
H	4.98123000	0.07488300	1.50134500
C	6.00189900	-2.78531900	-0.03320900
H	4.96526400	-3.76657400	-1.64223600
H	6.79993700	-3.51881800	0.01744300
C	5.97790500	-1.70306900	0.85585700

H	6.75460200	-1.60281100	1.60778000
C	4.96999000	-0.74610900	0.80083200
C	2.91383800	1.43293500	0.22610900
O	1.88703900	2.06873800	0.34583900
O	4.13400300	1.86953200	0.48370100
C	4.23276400	3.25616000	0.95965100
H	3.65721200	3.33337200	1.88572800
H	3.76601700	3.89655600	0.20733300
C	5.70214300	3.56213600	1.15449100
H	5.80915100	4.59931300	1.48828200
H	6.14852500	2.91309400	1.91400300
H	6.25708400	3.44434600	0.21925500

[1] Gaussian 09, Revision A.02, Gaussian, Inc., Wallingford CT, 2009.

[2] The calculations were performed using the computing resources of the computing center of RWTH Aachen.

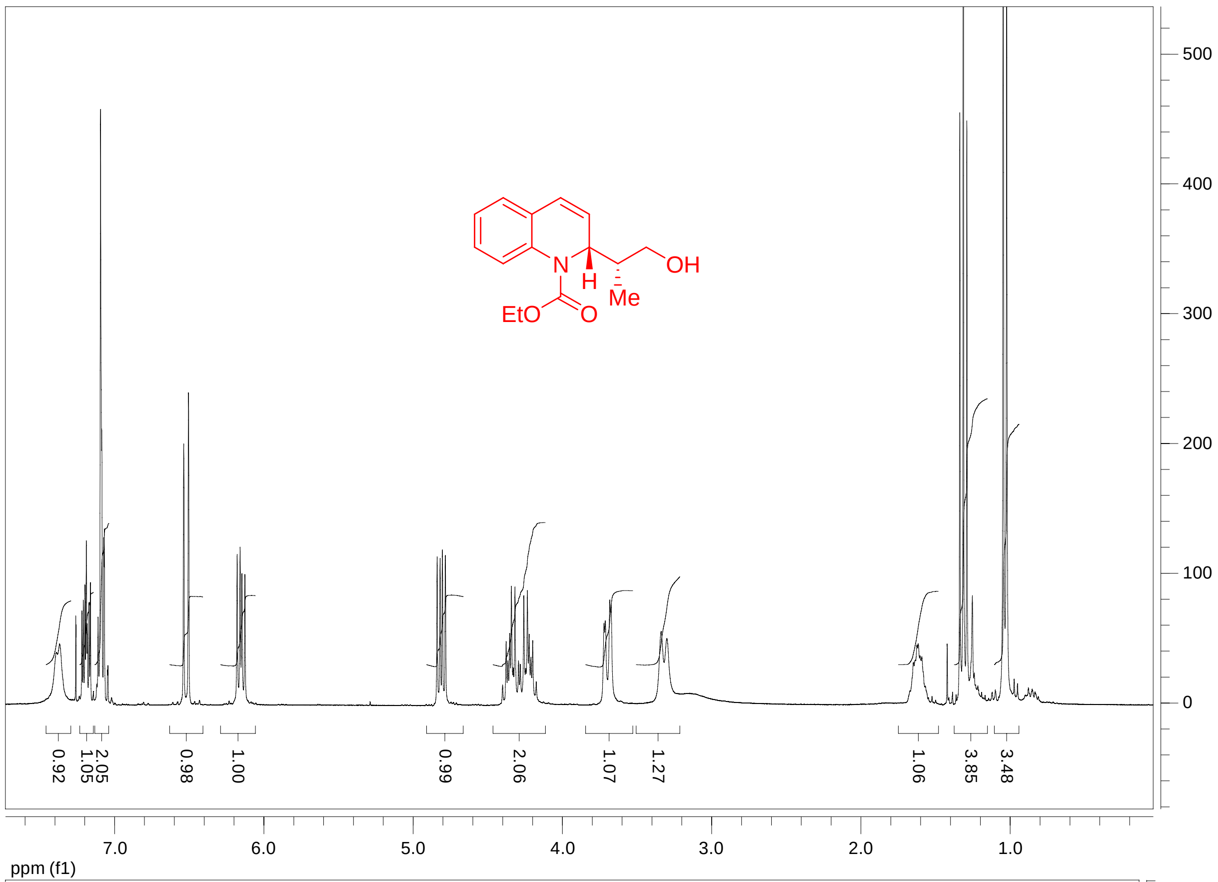
[3] Due to its non-coordinative nature, the counter anion was not taken into consideration for the calculations.

[4] a) C. Lee, W. Yang and R. G. Parr, *Phys. Rev. B,* 1988, **37**, 785-789; b) B. Miehlich, A. Savin, H. Stoll and H. Preuss, *Chem. Phys. Lett.,* 1989, **157**, 200-206; c) A. D. Becke, *J. Chem. Phys.,* 1993, **98**, 5648-5652; d) P. J. Stephens, F. J. Devlin, C.F. Chabalowski and M. J. Frisch, *J. Phys. Chem.,* 1994, **98**, 11623-11627.

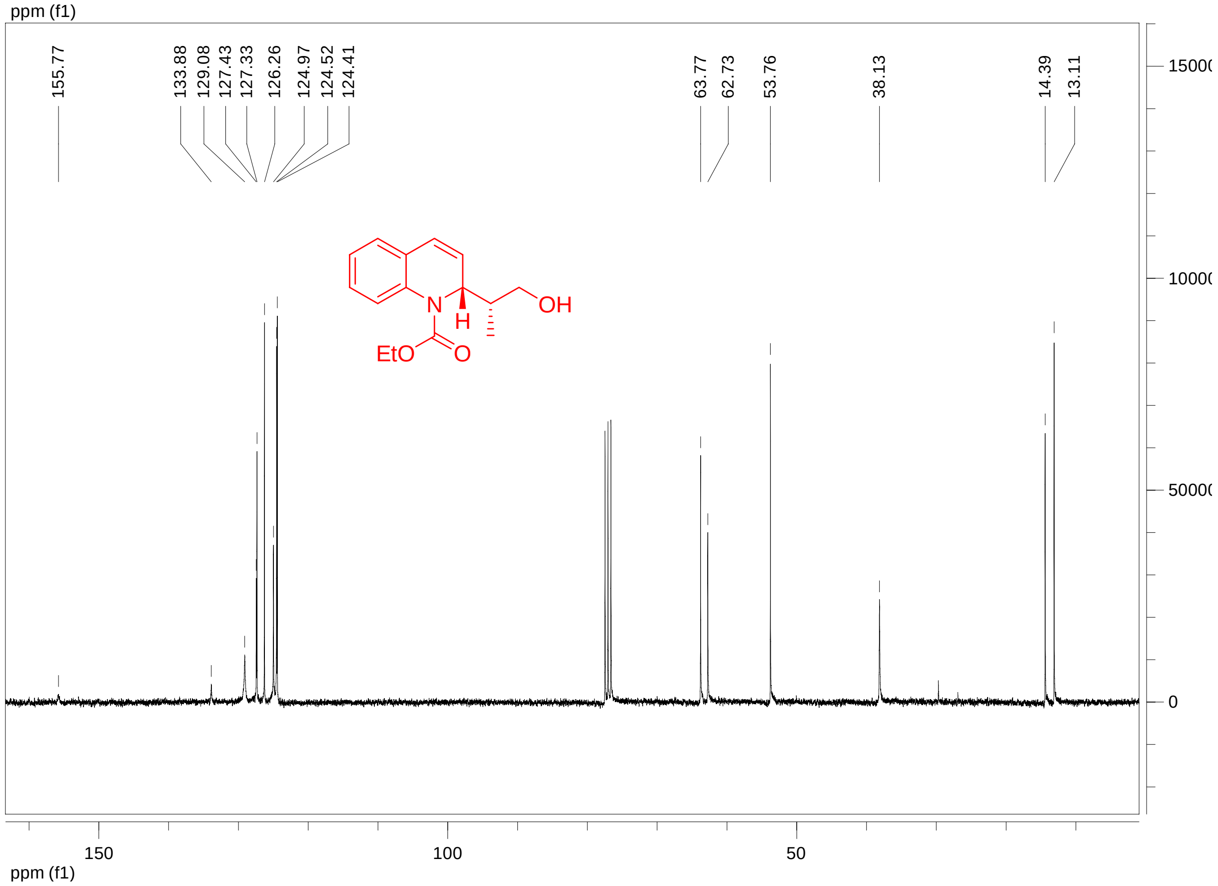
[5] a) V. Barone and M. Cossi, *J. Phys. Chem. A,* 1998, **102**, 1995-2001; b) J. Tomasi, B. Mennucci and R. Cammi, *Chem. Rev.,* 2005, **105**, 2999-3093.

[6] a) W. J. Hehre, R. Ditchfeld and J. A. Pople, *J. Chem. Phys*., 1972, **56**, 2257-2261; b) C. Hariharan and J. A. Pople, *Theor. Chim. Acta,* 1973, **28**, 213-222.

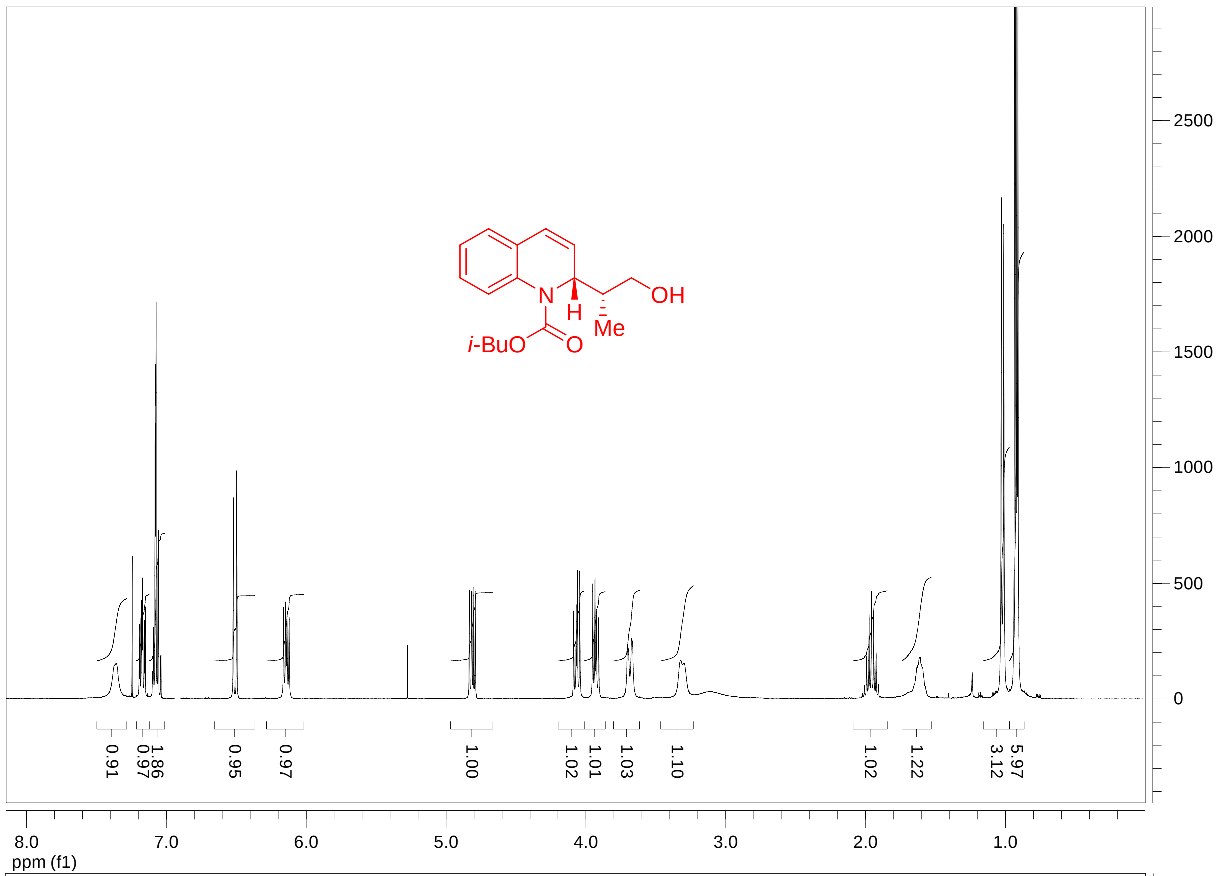
[7] S. Grimme, J. Antony, S. Ehrlich and H. Krieg, *J. Chem. Phys.,* 2010, **132**, 154104-154119.



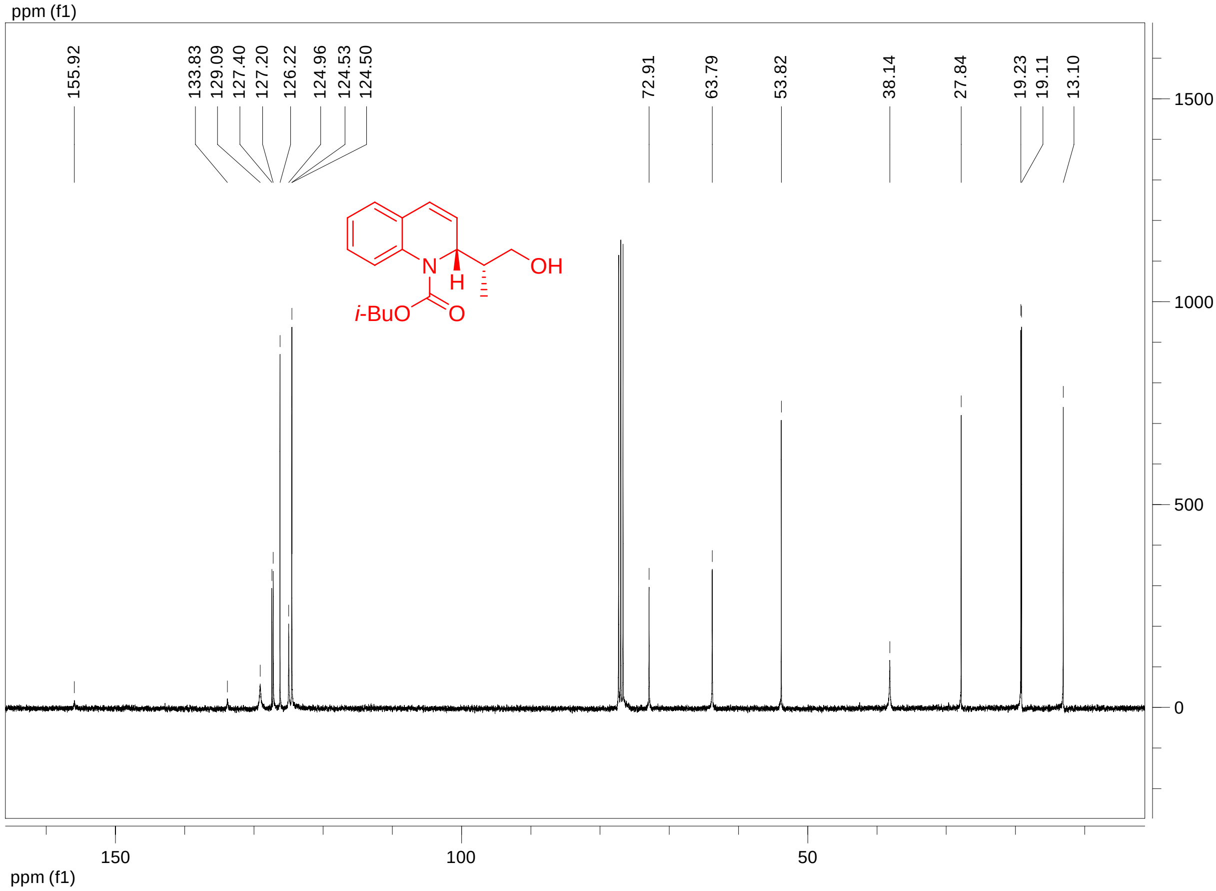
OH
N
H
Me
EtO
O
0.92
1.05
2.05
0.98
1.00
0.99
2.06
1.07
1.27
1.06
3.85
3.48
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)
500
400
300
200
100
0



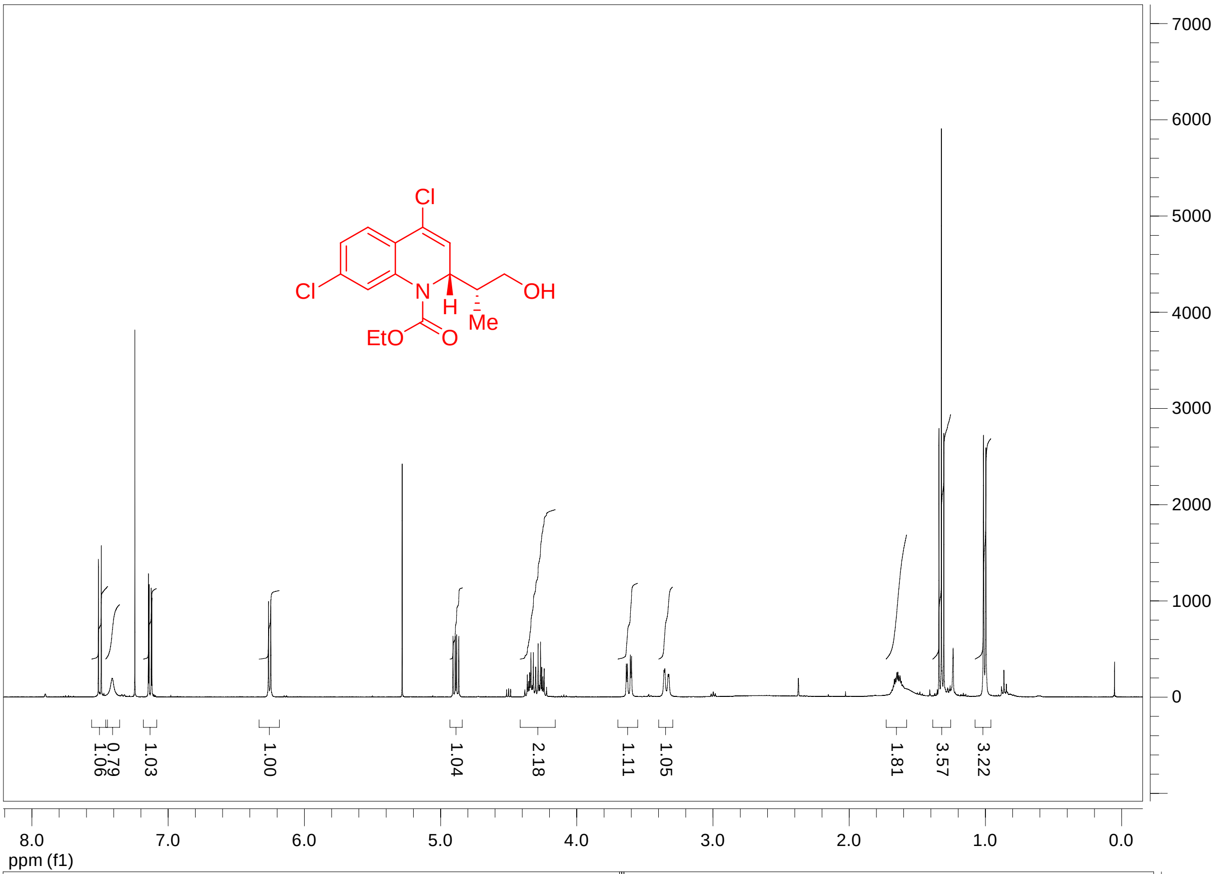
155.77
133.88
129.08
127.43
127.33
126.26
124.97
124.52
124.41
63.77
62.73
53.76
38.13
14.39
13.11
OH
N
H
EtO
O
150
100
50
ppm (f1)
0



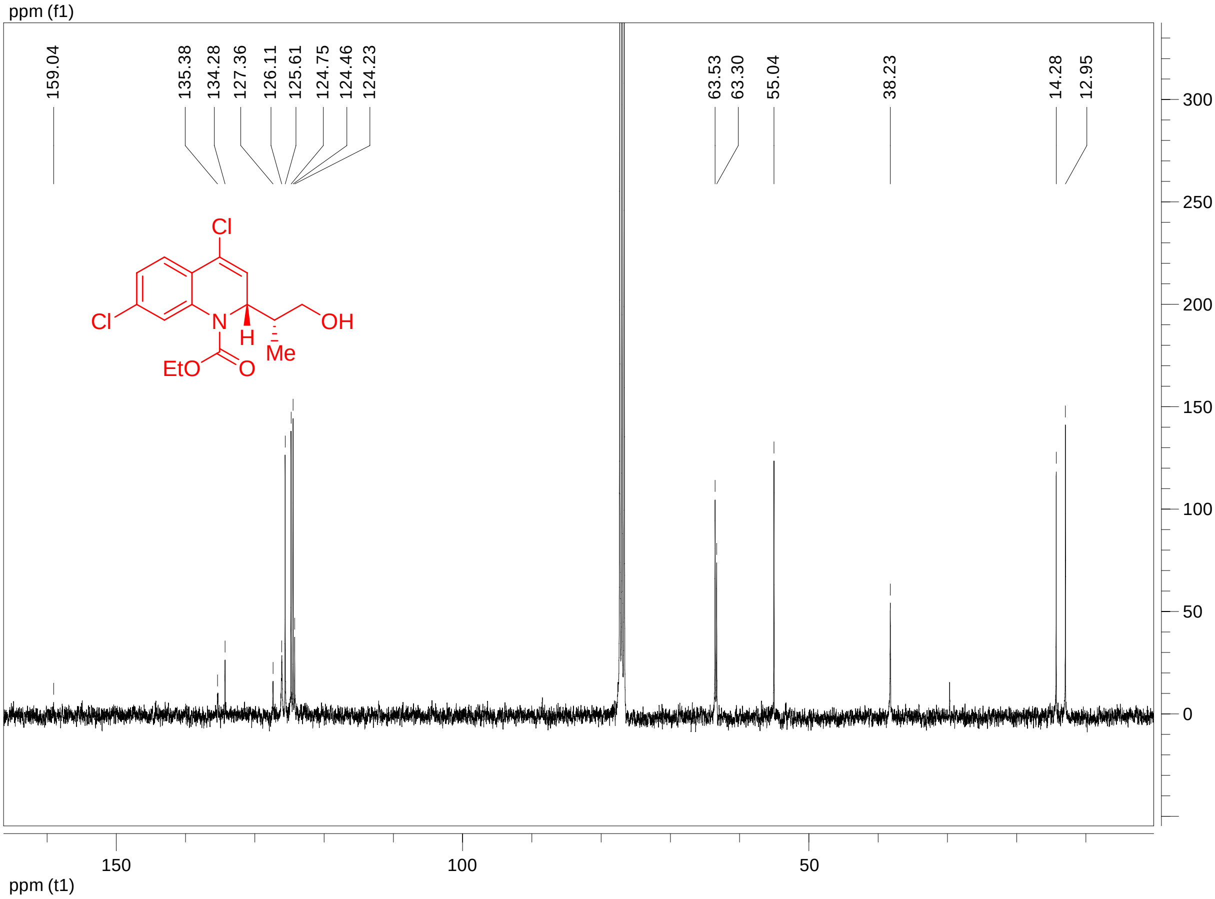
N
H
OH
Me
i-BuO
O
0.91
0.97
1.86
0.95
0.97
1.00
1.02
1.01
1.03
1.10
1.02
1.22
3.12
5.97
2500
2000
1500
1000
500
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



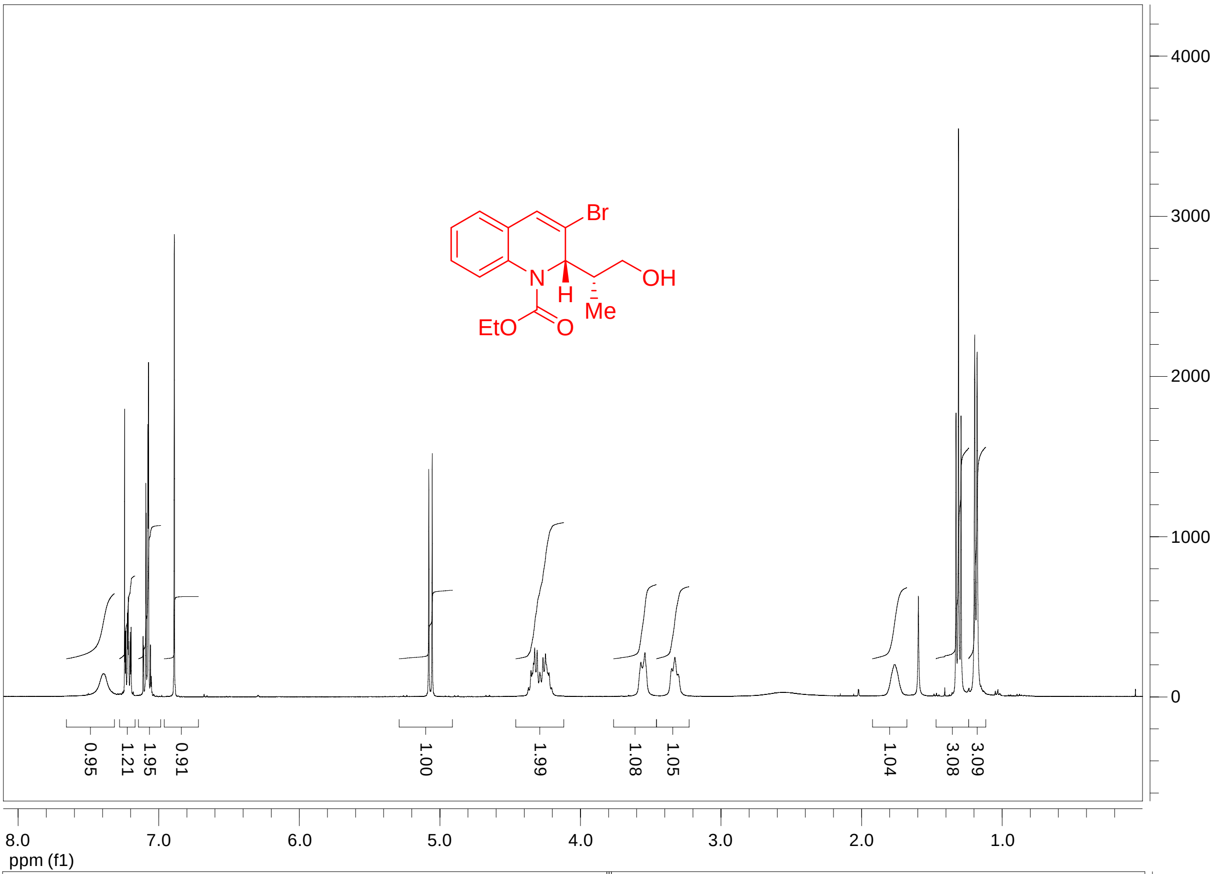
155.92
133.83
129.09
127.40
127.20
126.22
124.96
124.53
124.50
72.91
63.79
53.82
38.14
27.84
19.23
19.11
13.10
N
H
OH
i-BuO
O
1500
1000
500
0
150
100
50
ppm (f1)

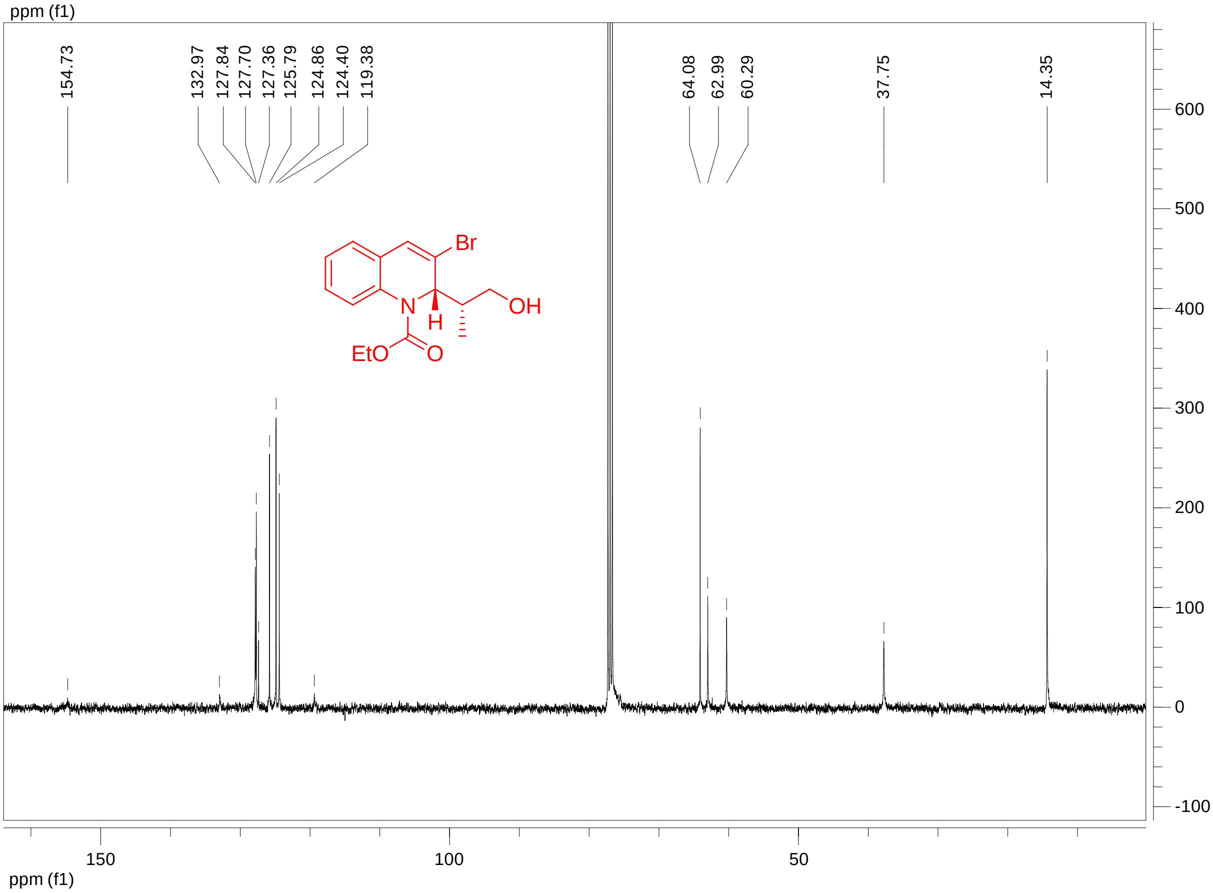


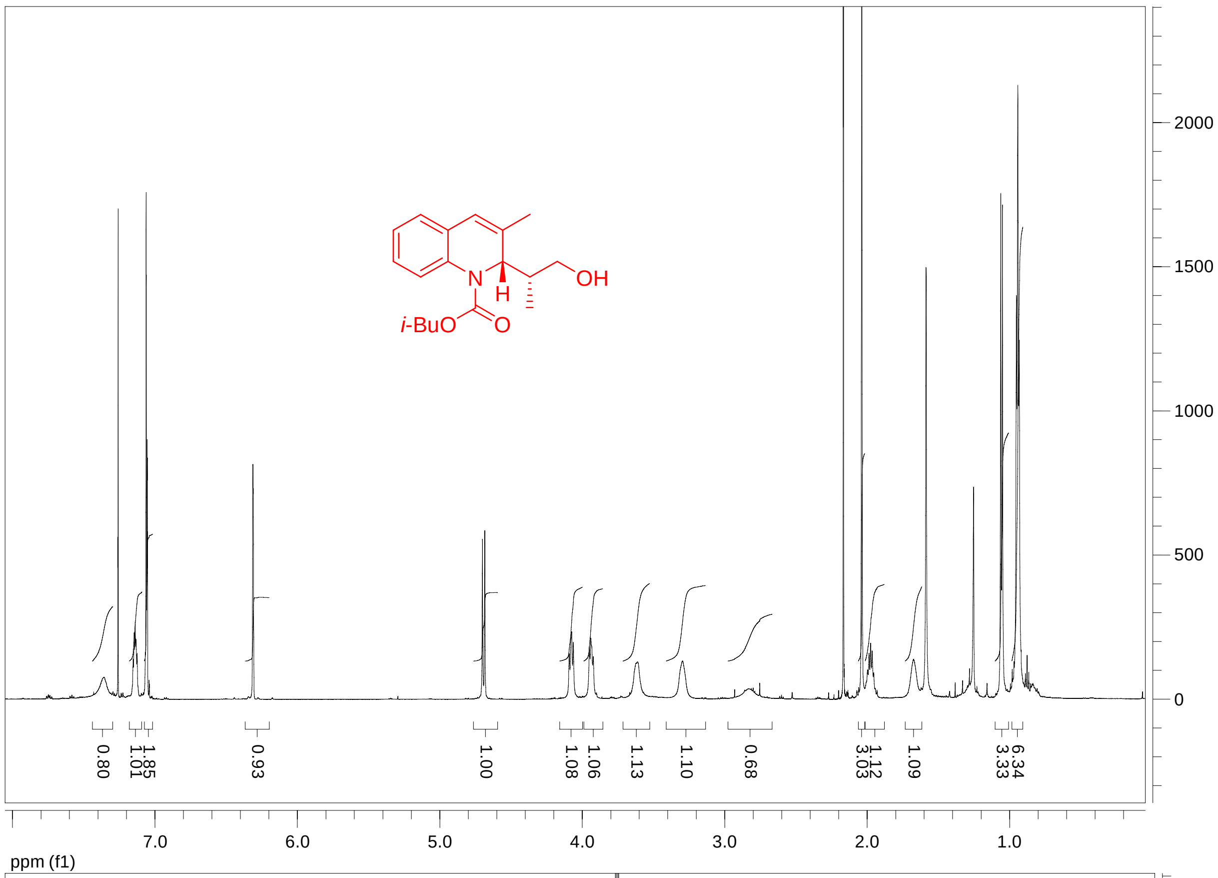
Cl
Cl
N
H
OH
Me
EtO
O
1.06
0.79
1.03
1.00
1.04
2.18
1.11
1.05
1.81
3.57
3.22
ppm (f1)



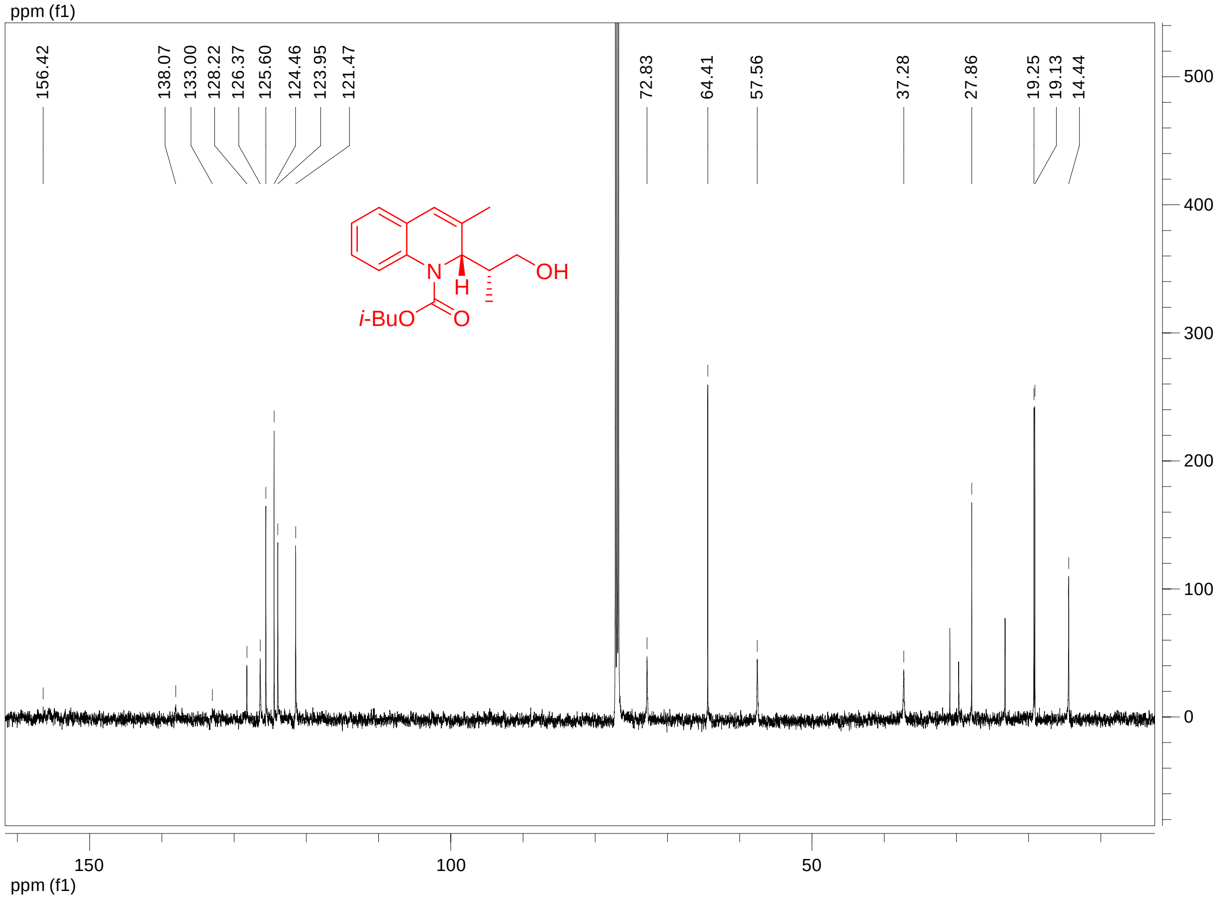
159.04
135.38
134.28
127.36
126.11
125.61
124.75
124.46
124.23
63.53
63.30
55.04
38.23
14.28
12.95
Cl
Cl
N
H
OH
Me
EtO
O
ppm (t1)


Br
N
H
OH
Me
EtO
O
0.95
1.21
1.95
0.91
1.00
1.99
1.08
1.05
1.04
3.08
3.09
4000
3000
2000
1000
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

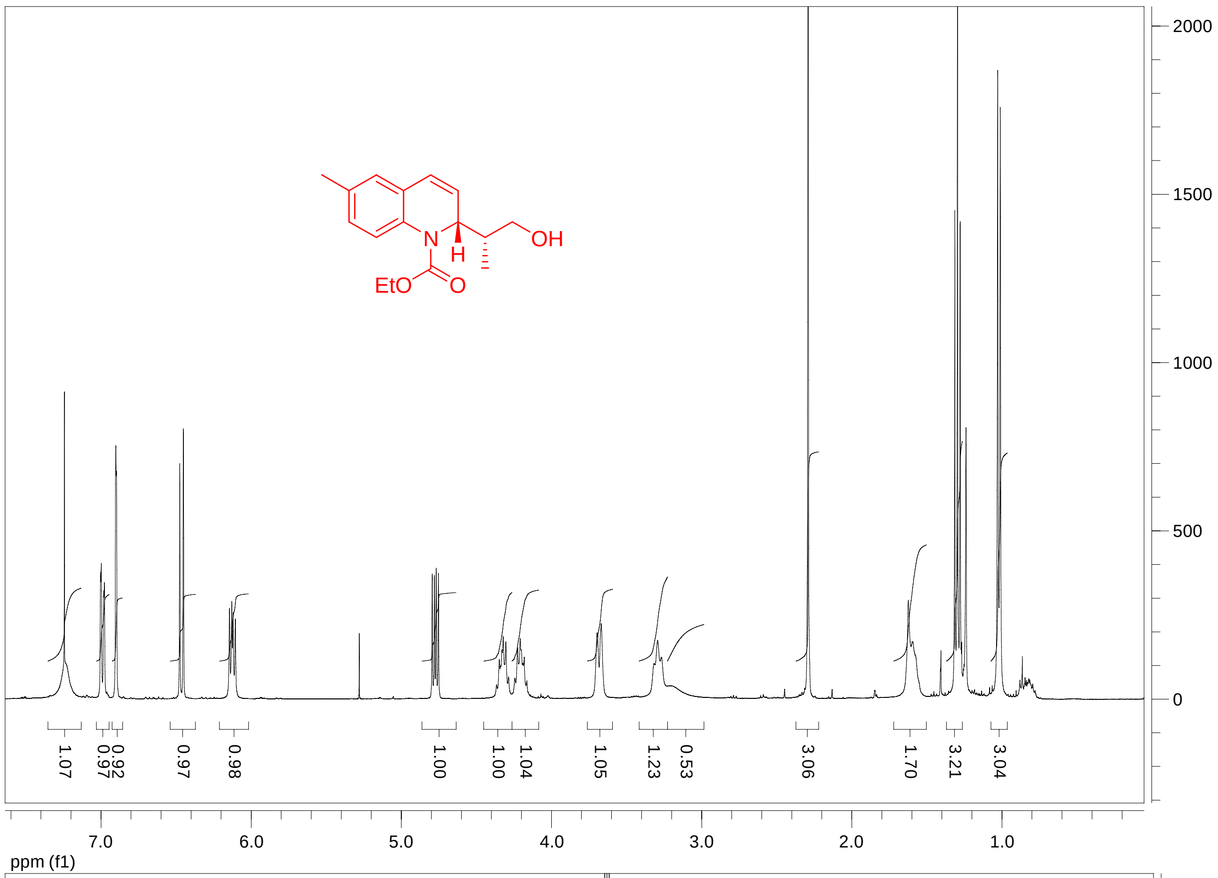

154.73
132.97
127.84
127.70
127.36
125.79
124.86
124.40
119.38
64.08
62.99
60.29
37.75
14.35
Br
N
H
OH
EtO
O
600
500
400
300
200
100
0
-100
150
100
50
ppm (f1)



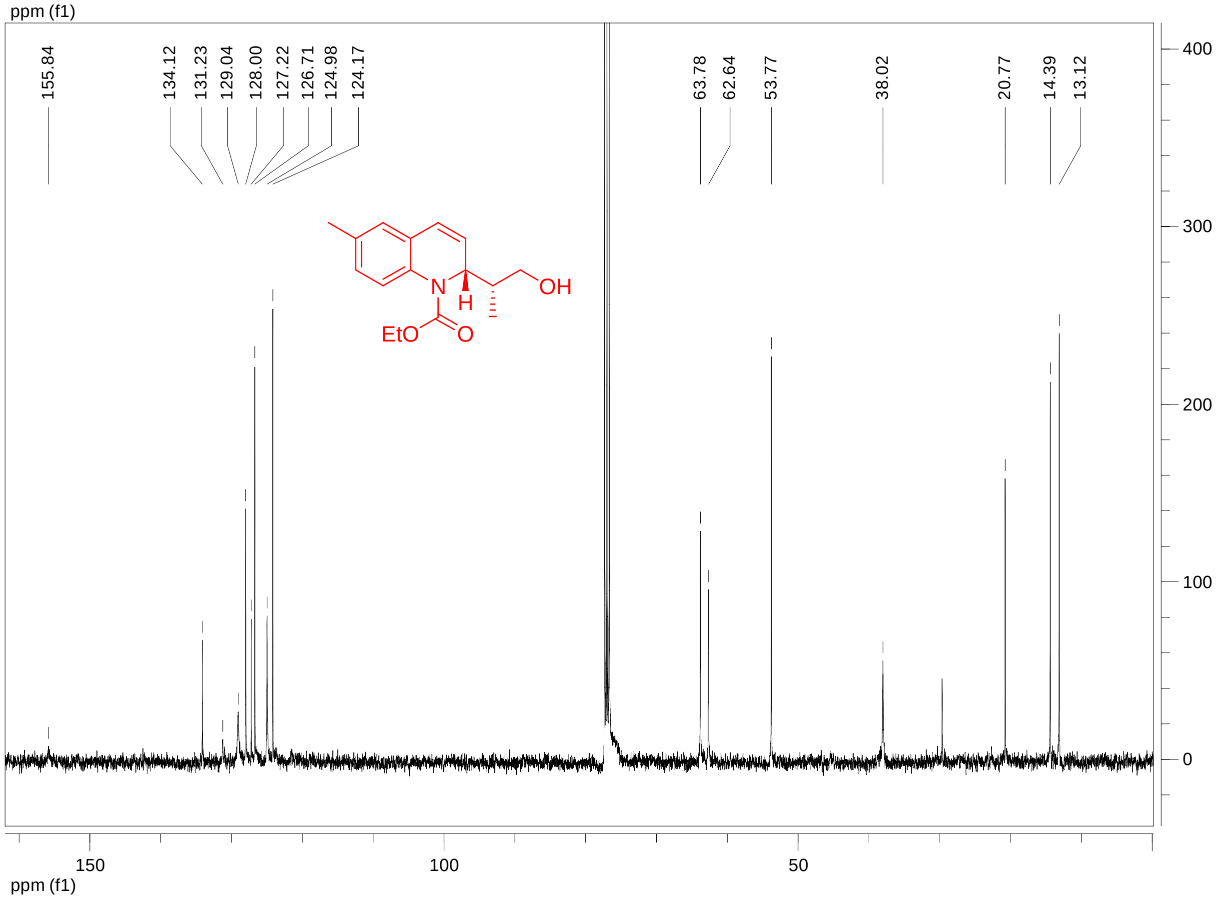
N
H
OH
i-BuO
O
0.80
1.01
1.85
0.93
1.00
1.08
1.06
1.13
1.10
0.68
3.03
1.12
1.09
3.33
6.34
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)
2000
1500
1000
500
0



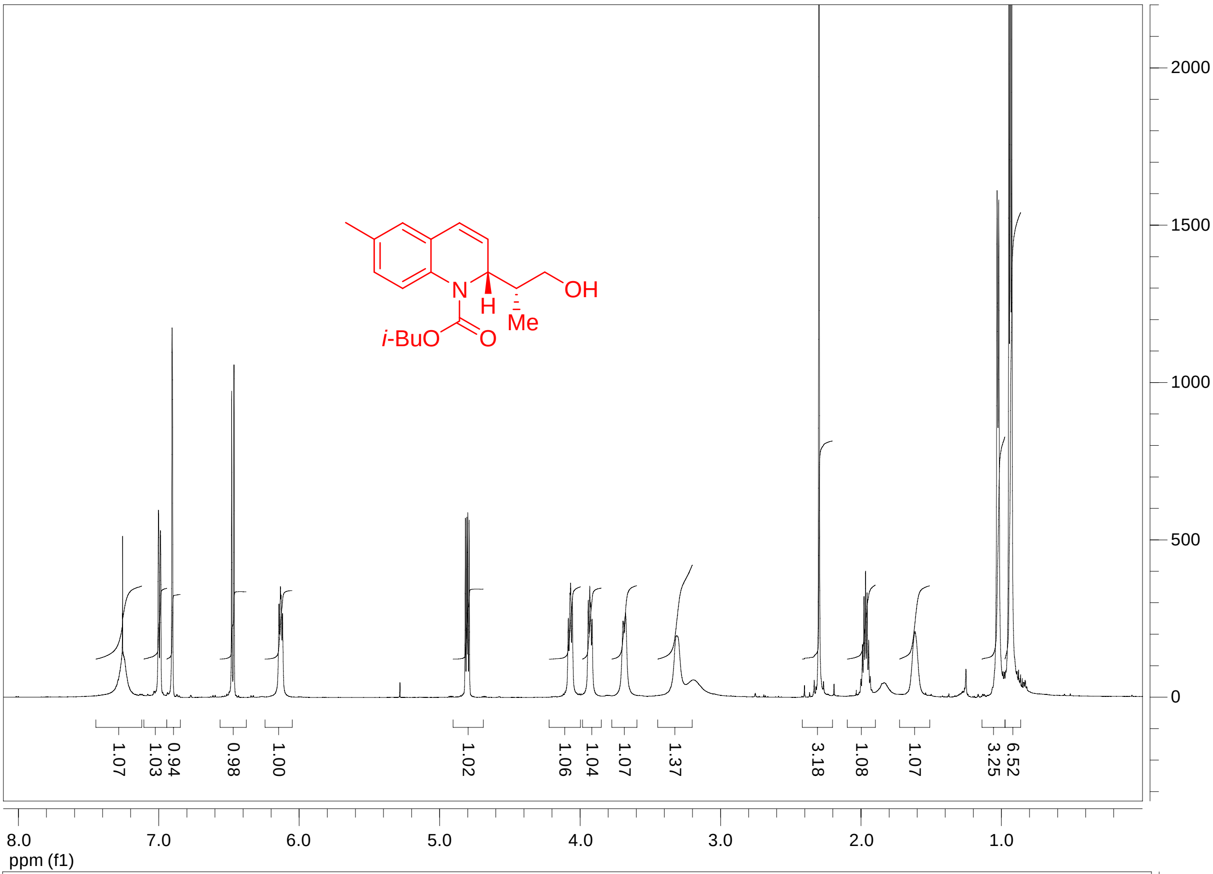
156.42
138.07
133.00
128.22
126.37
125.60
124.46
123.95
121.47
72.83
64.41
57.56
37.28
27.86
19.25
19.13
14.44
N
H
OH
i-BuO
O
150
100
50
ppm (f1)
500
400
300
200
100
0



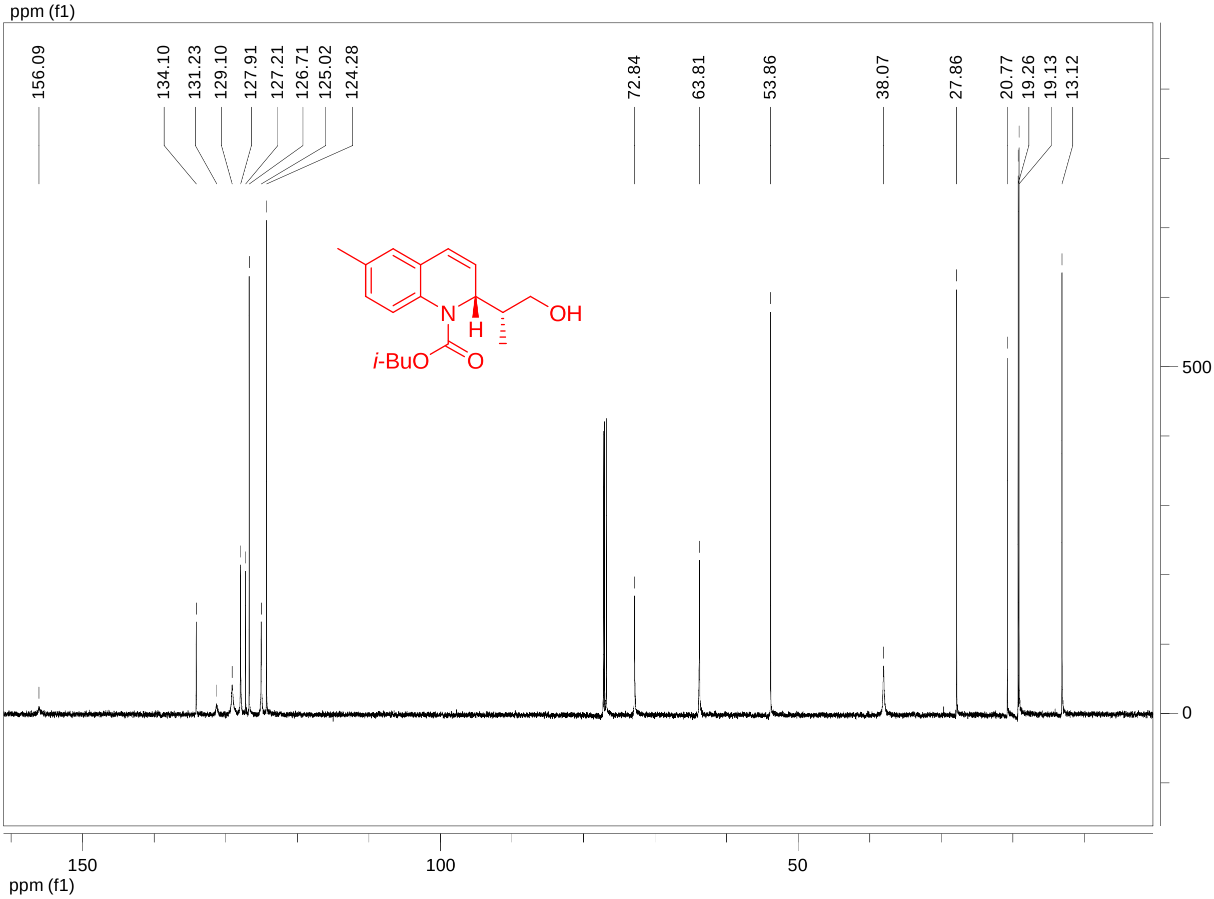
OH
N
H
EtO
O
1.07
0.97
0.92
0.97
0.98
1.00
1.00
1.04
1.05
1.23
0.53
3.06
1.70
3.21
3.04
2000
1500
1000
500
0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



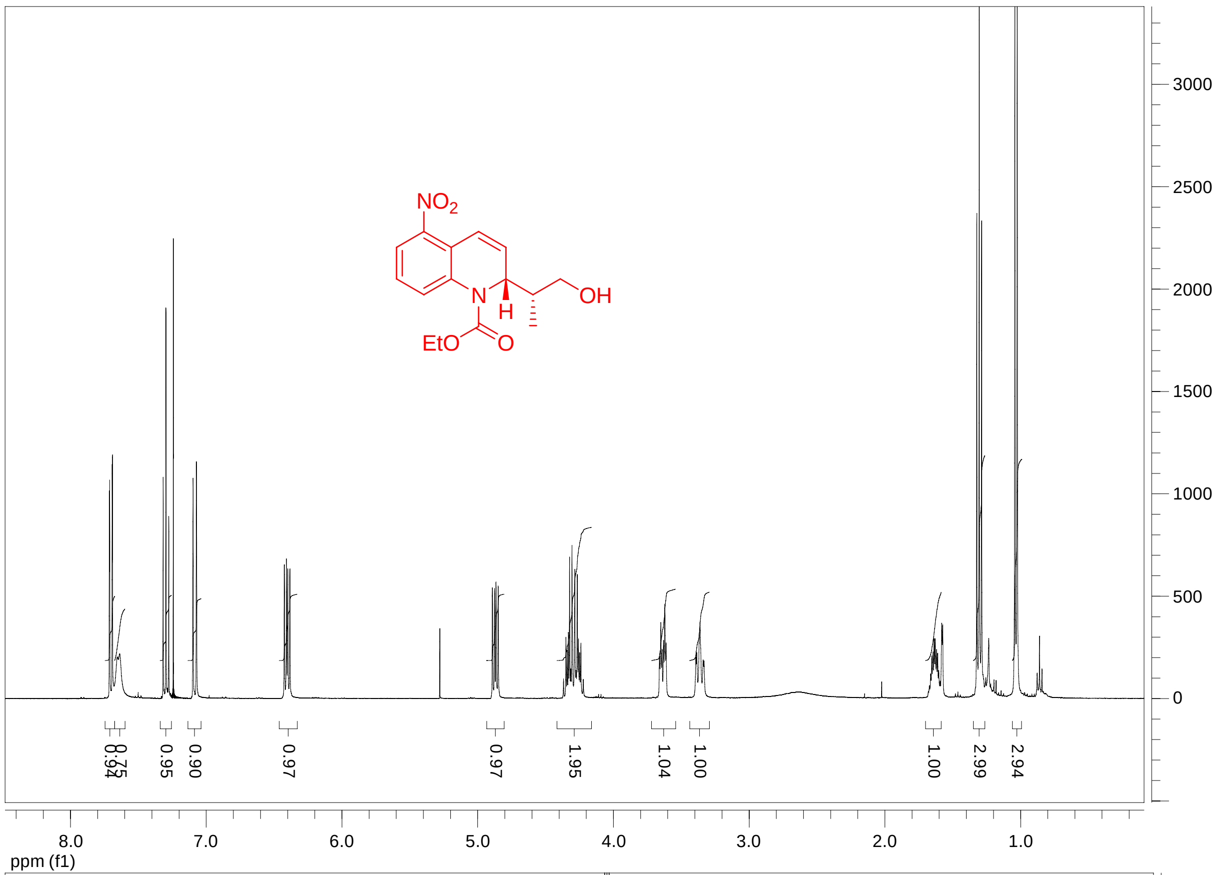
155.84
134.12
131.23
129.04
128.00
127.22
126.71
124.98
124.17
63.78
62.64
53.77
38.02
20.77
14.39
13.12
OH
N
H
EtO
O
400
300
200
100
0
150
100
50
ppm (f1)



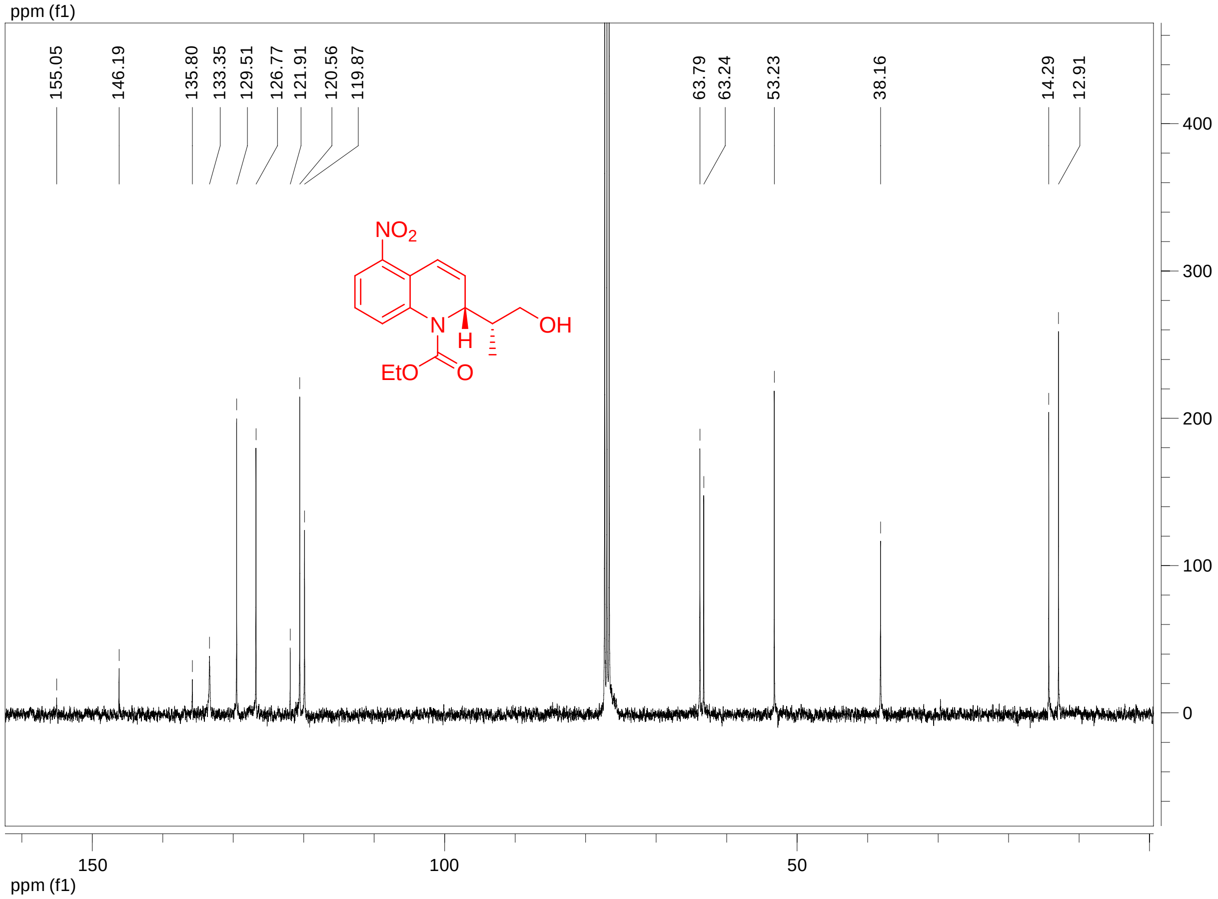
OH
N
H
Me
i-BuO
O
1.07
1.03
0.94
0.98
1.00
1.02
1.06
1.04
1.07
1.37
3.18
1.08
1.07
3.25
6.52
2000
1500
1000
500
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



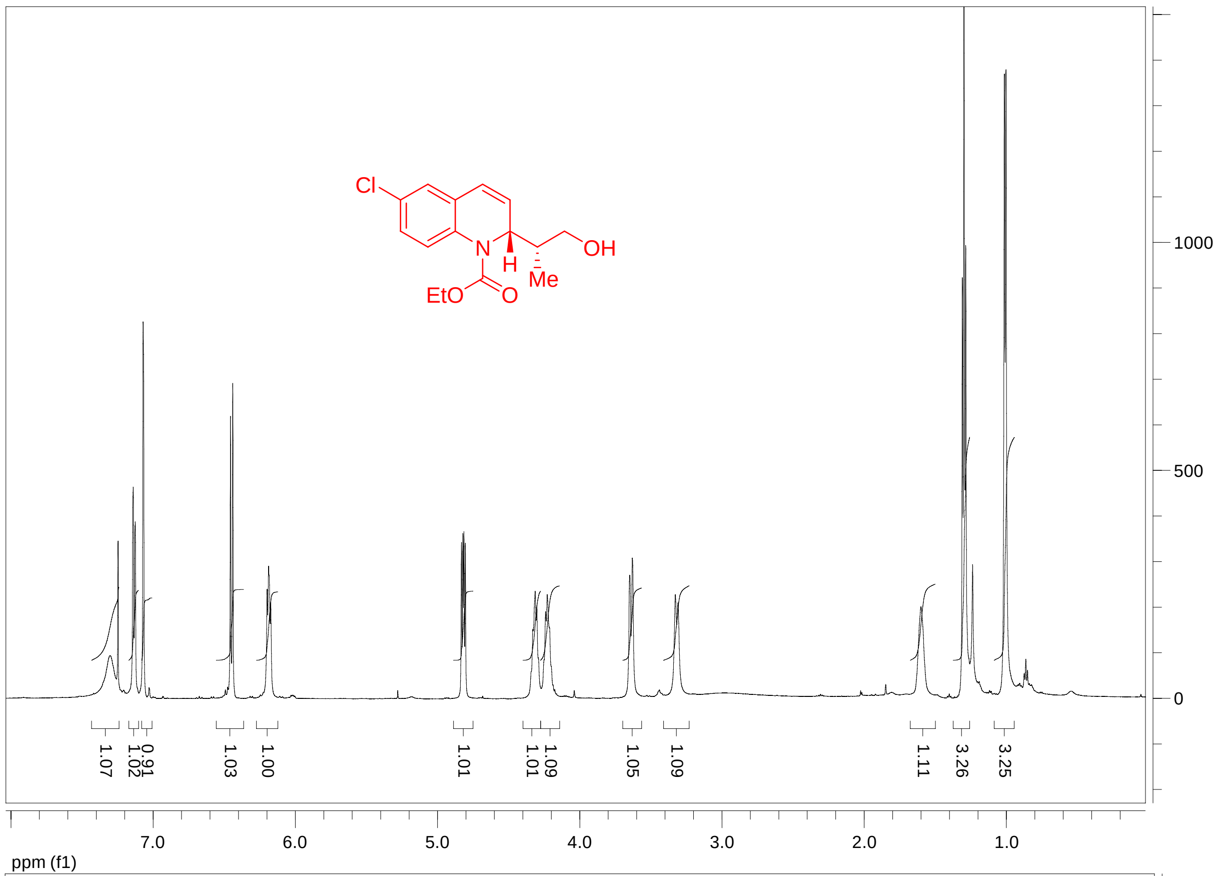
156.09
134.10
131.23
129.10
127.91
127.21
126.71
125.02
124.28
72.84
63.81
53.86
38.07
27.86
20.77
19.26
19.13
13.12
N
H
OH
i-BuO
O
500
0
150
100
50
ppm (f1)

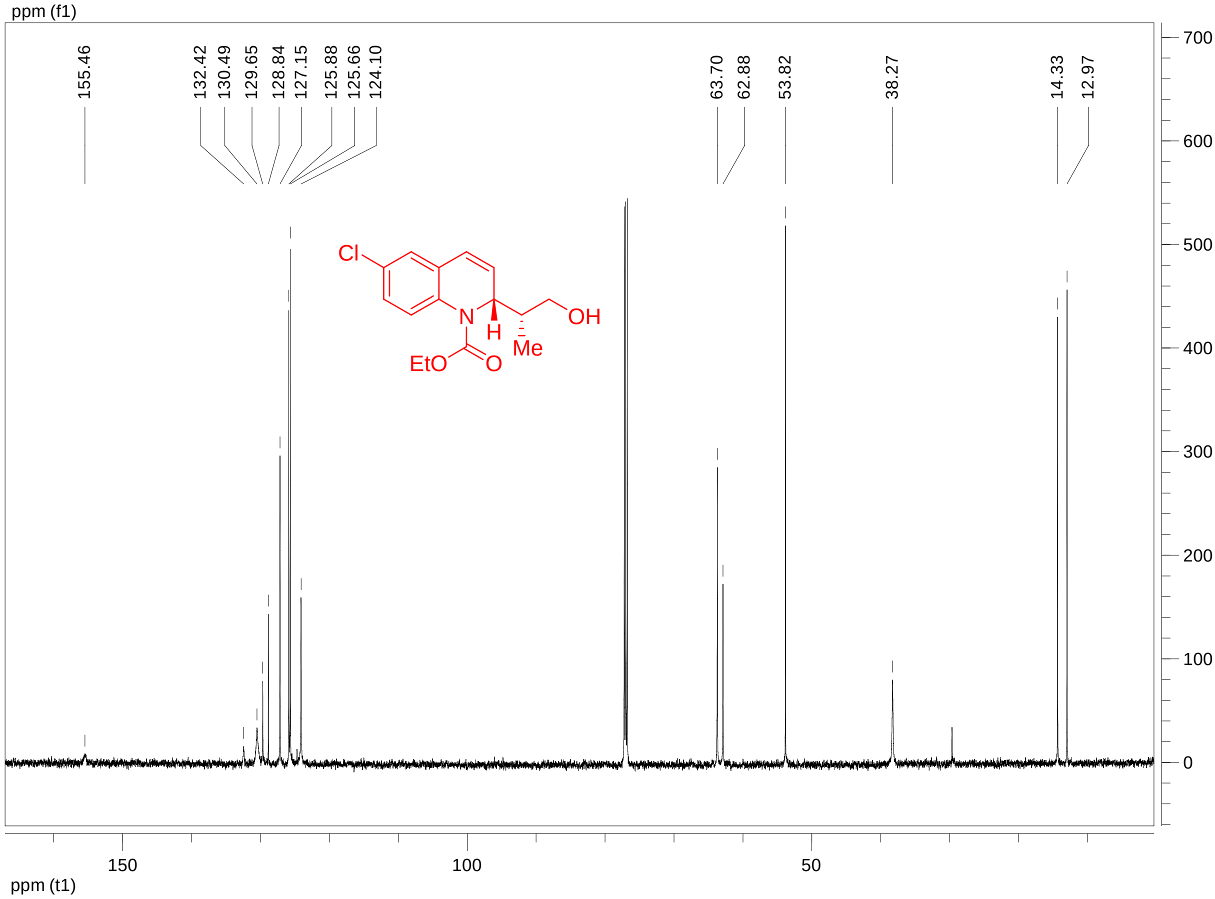


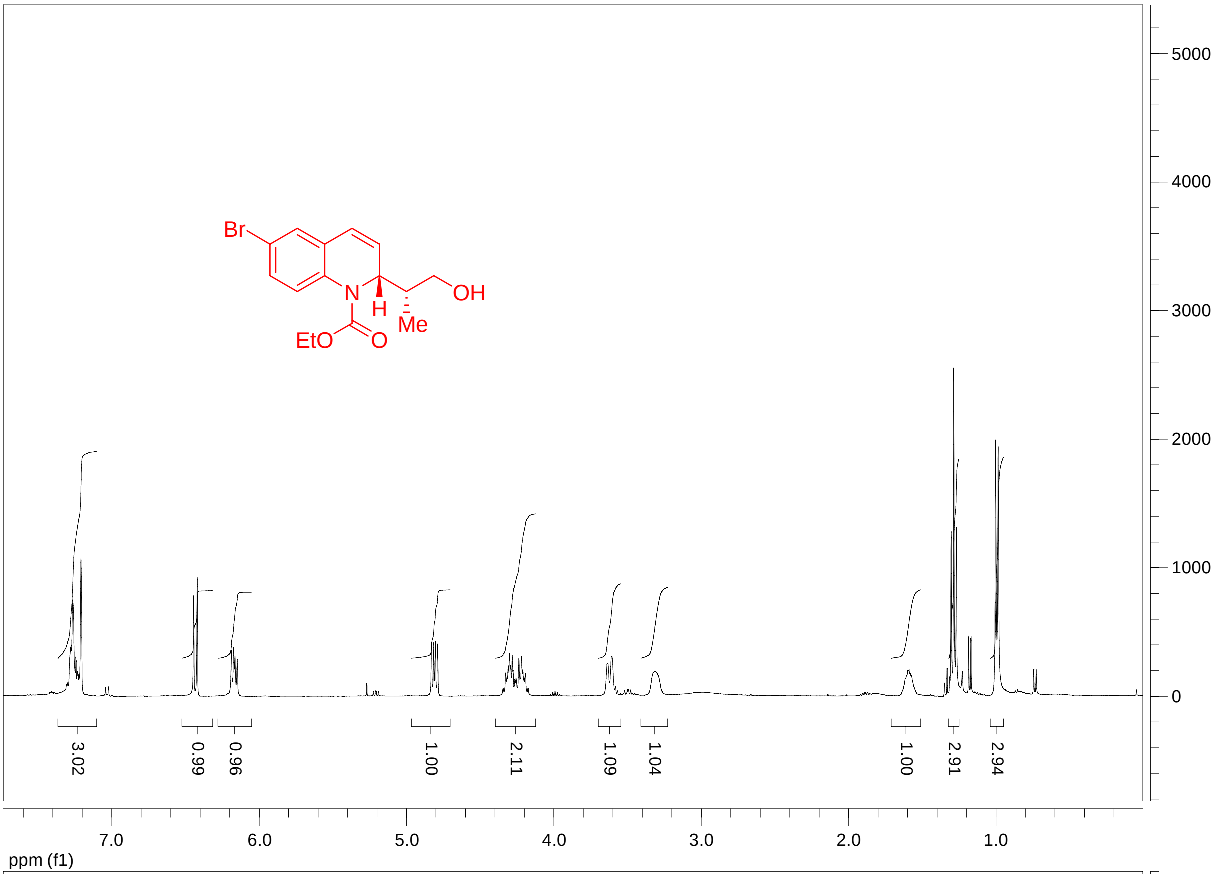
NO2
N
H
OH
EtO
O
0.94
0.75
0.95
0.90
0.97
0.97
1.95
1.04
1.00
1.00
2.99
2.94
3000
2500
2000
1500
1000
500
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



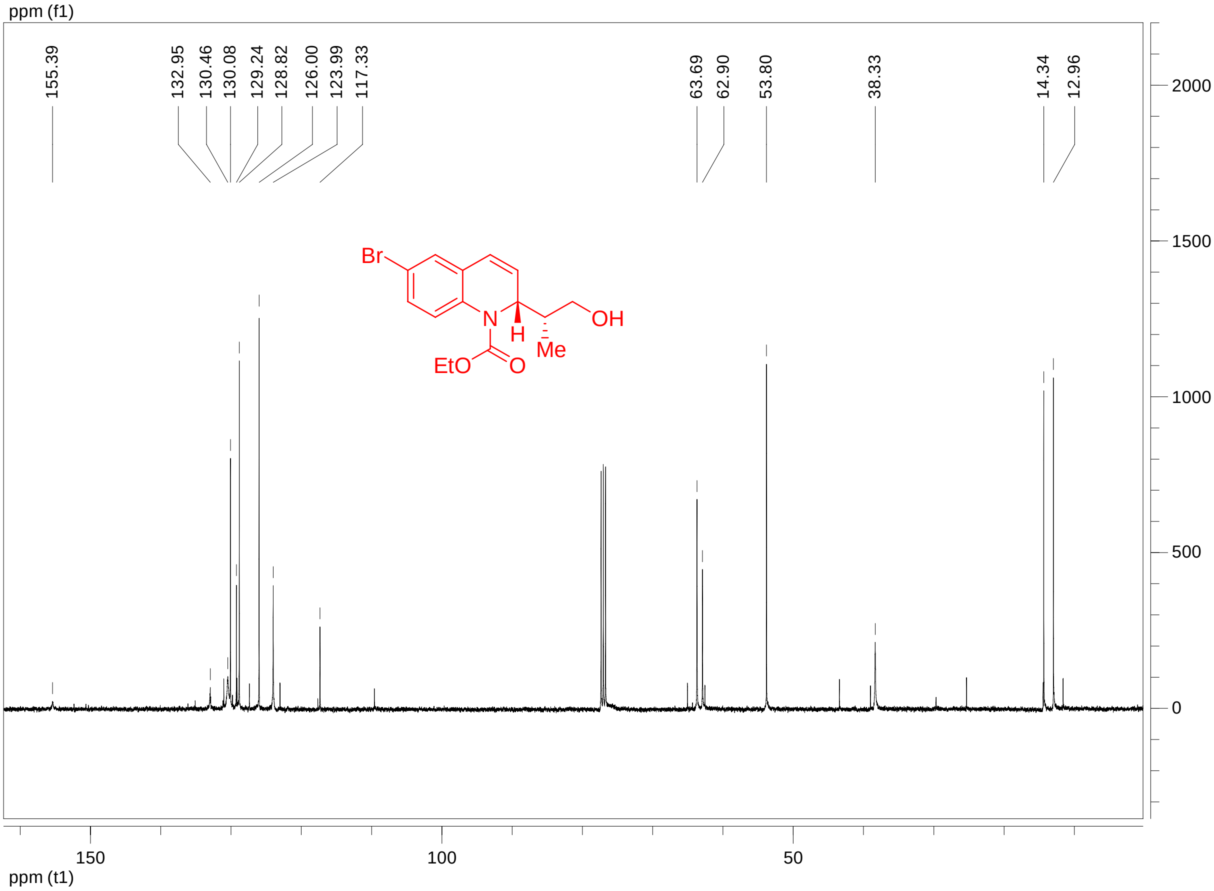
155.05
146.19
135.80
133.35
129.51
126.77
121.91
120.56
119.87
63.79
63.24
53.23
38.16
14.29
12.91
NO2
N
H
OH
EtO
O
400
300
200
100
0
150
100
50
ppm (f1)


Cl
N
H
OH
Me
EtO
O
1.07
1.02
0.91
1.03
1.00
1.01
1.01
1.09
1.05
1.09
1.11
3.26
3.25
1000
500
0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

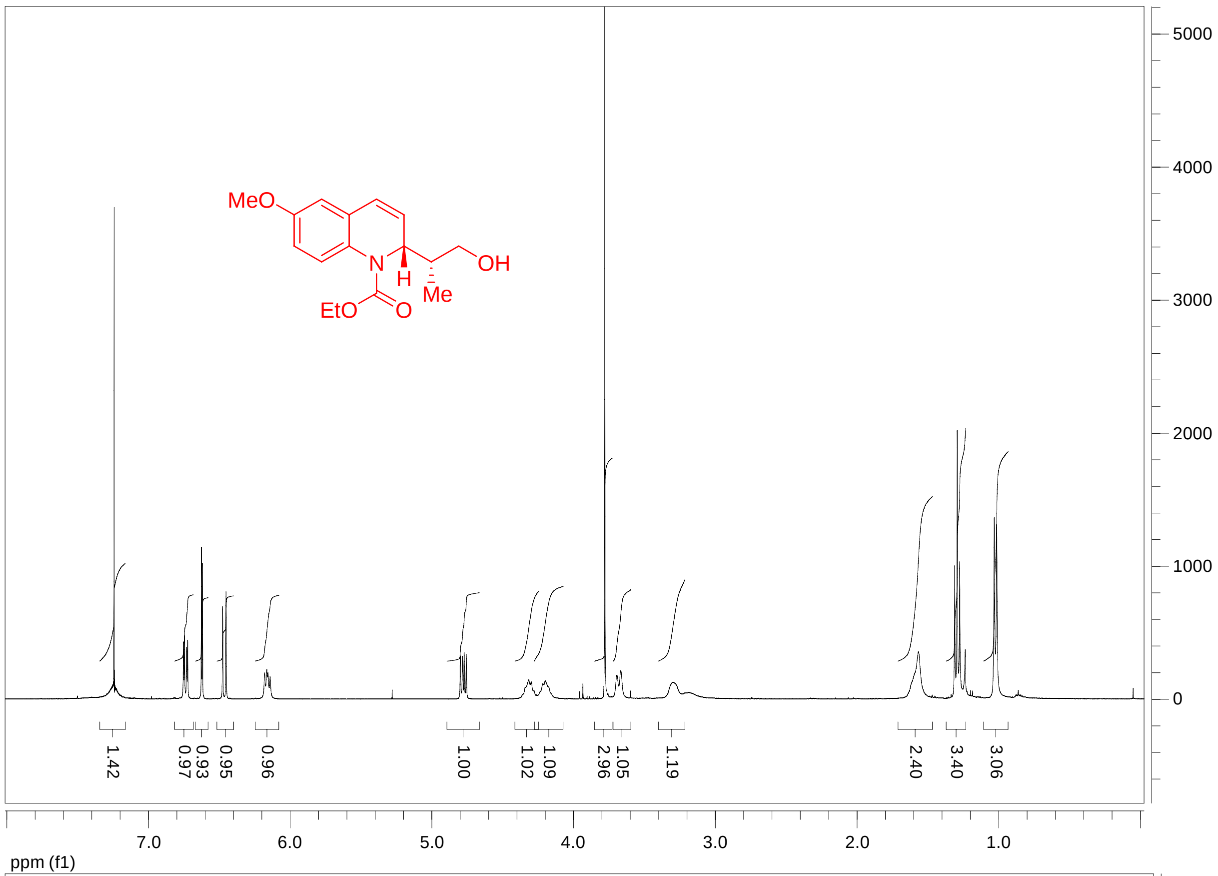

155.46
132.42
130.49
129.65
128.84
127.15
125.88
125.66
124.10
63.70
62.88
53.82
38.27
14.33
12.97
Cl
N
H
OH
Me
EtO
O
700
600
500
400
300
200
100
0
150
100
50
ppm (t1)



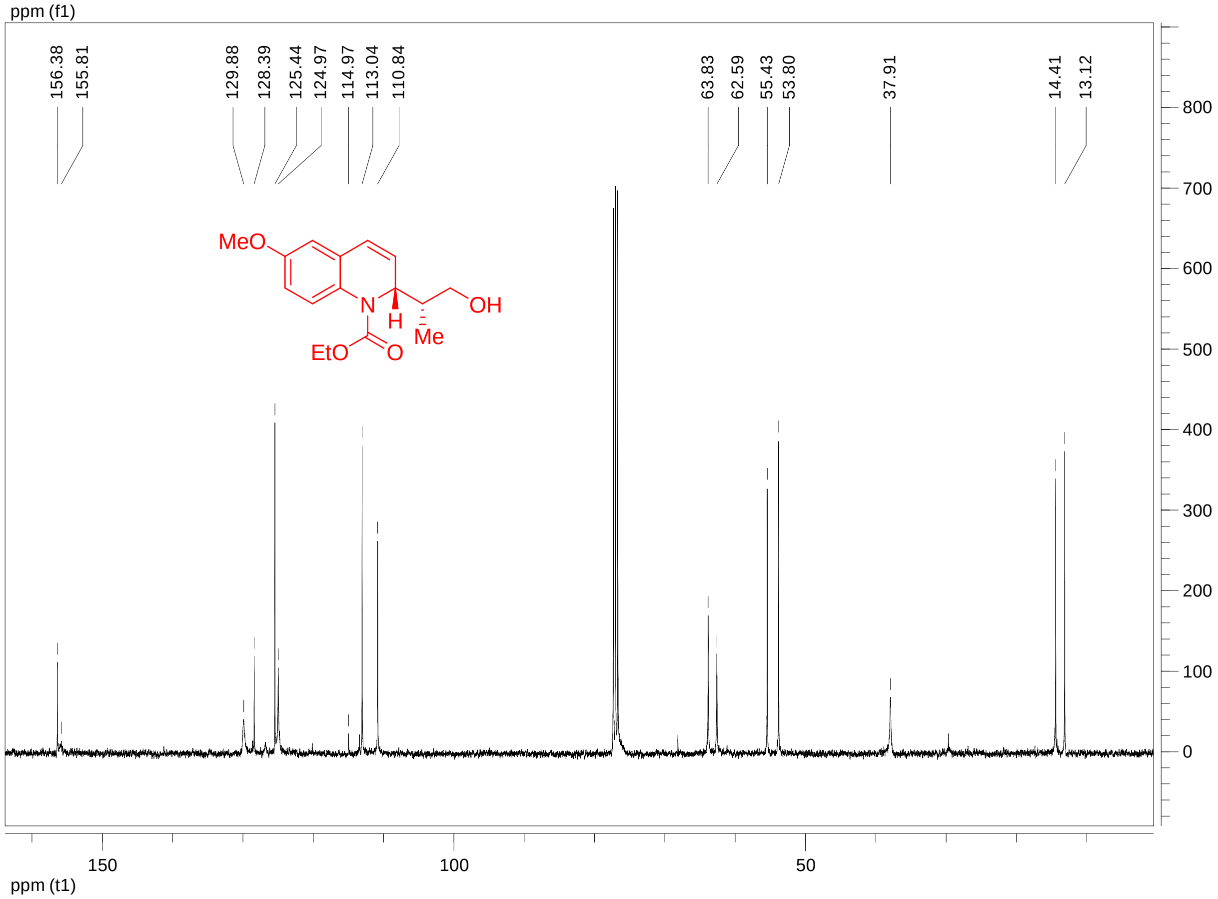
Br
N
H
OH
Me
EtO
O
3.02
0.99
0.96
1.00
2.11
1.09
1.04
1.00
2.91
2.94
5000
4000
3000
2000
1000
0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



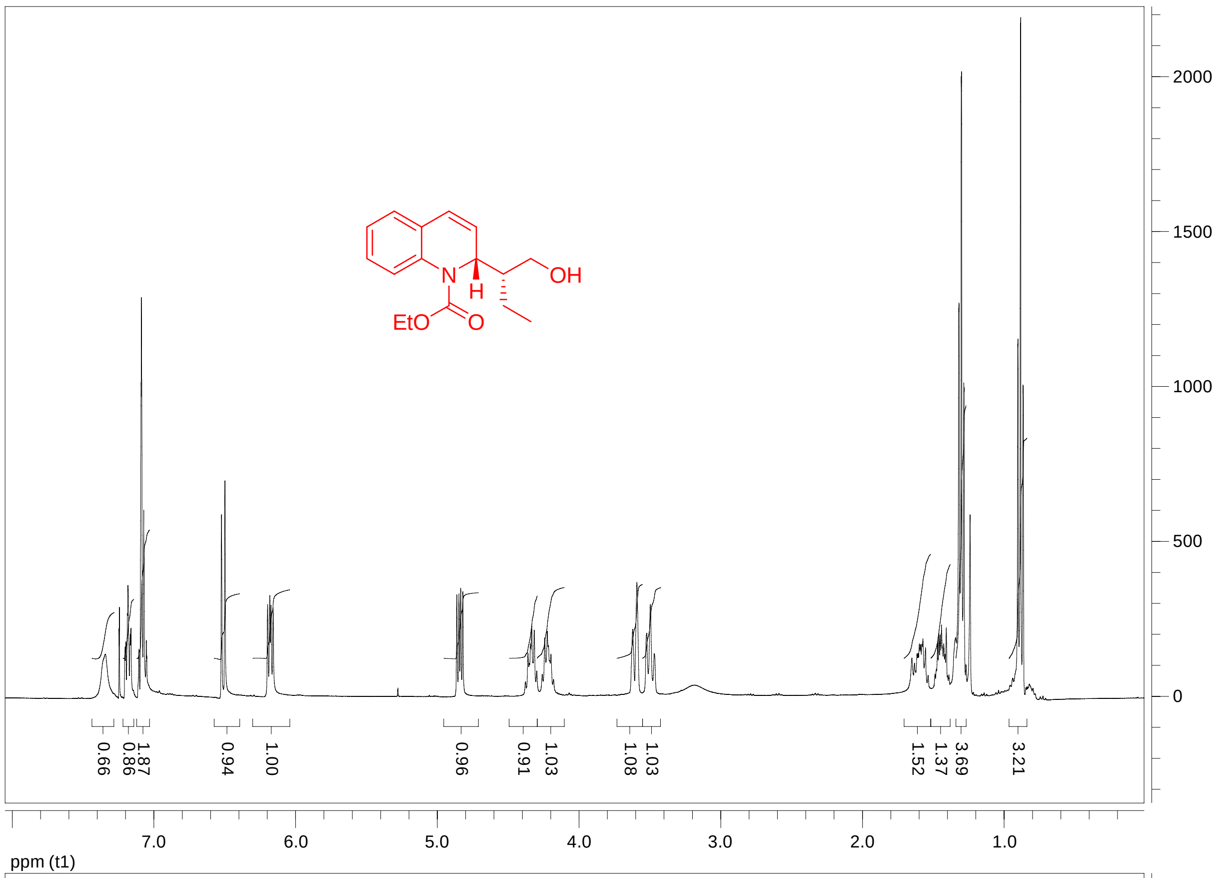
155.39
132.95
130.46
130.08
129.24
128.82
126.00
123.99
117.33
63.69
62.90
53.80
38.33
14.34
12.96
Br
N
H
OH
Me
EtO
O
2000
1500
1000
500
0
150
100
50
ppm (t1)



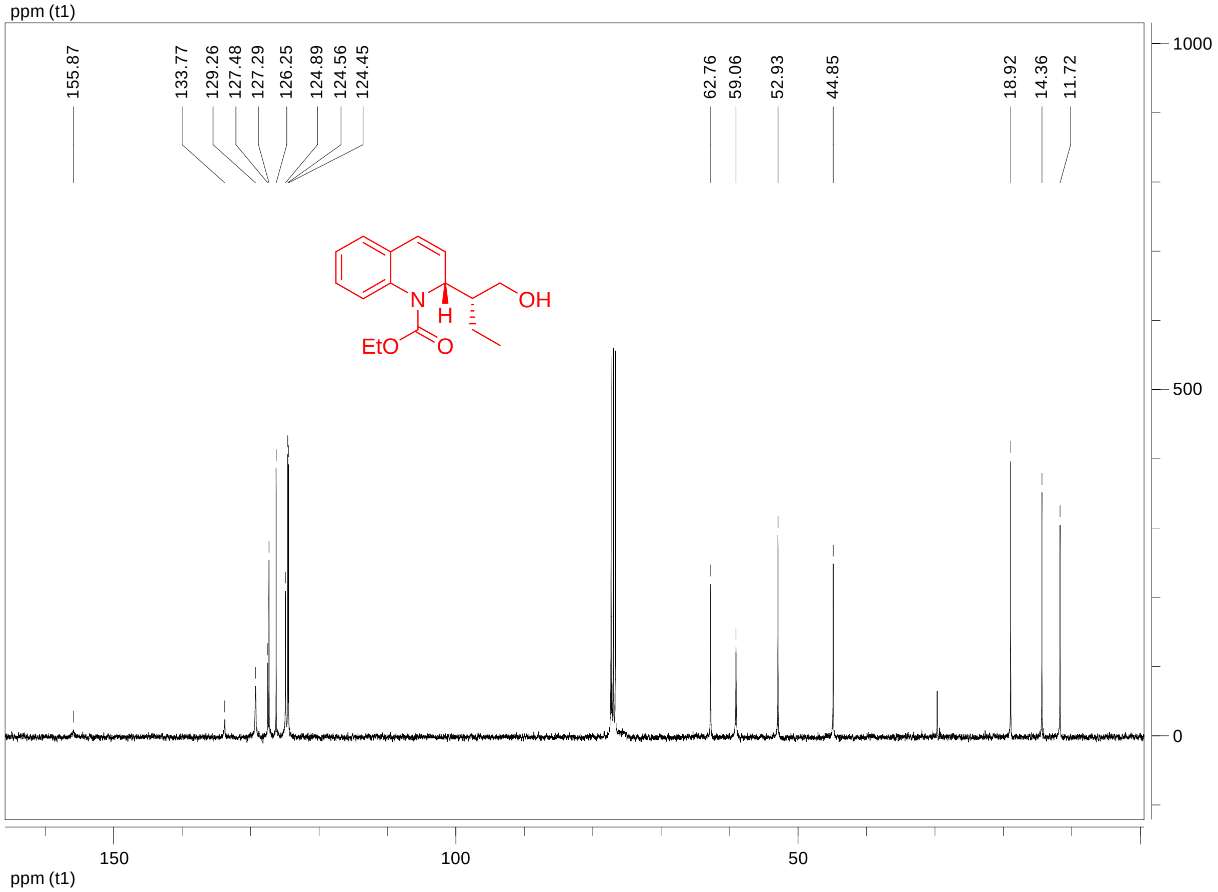
MeO
N
H
OH
Me
EtO
O
1.42
0.97
0.93
0.95
0.96
1.00
1.02
1.09
2.96
1.05
1.19
2.40
3.40
3.06
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)
5000
4000
3000
2000
1000
0



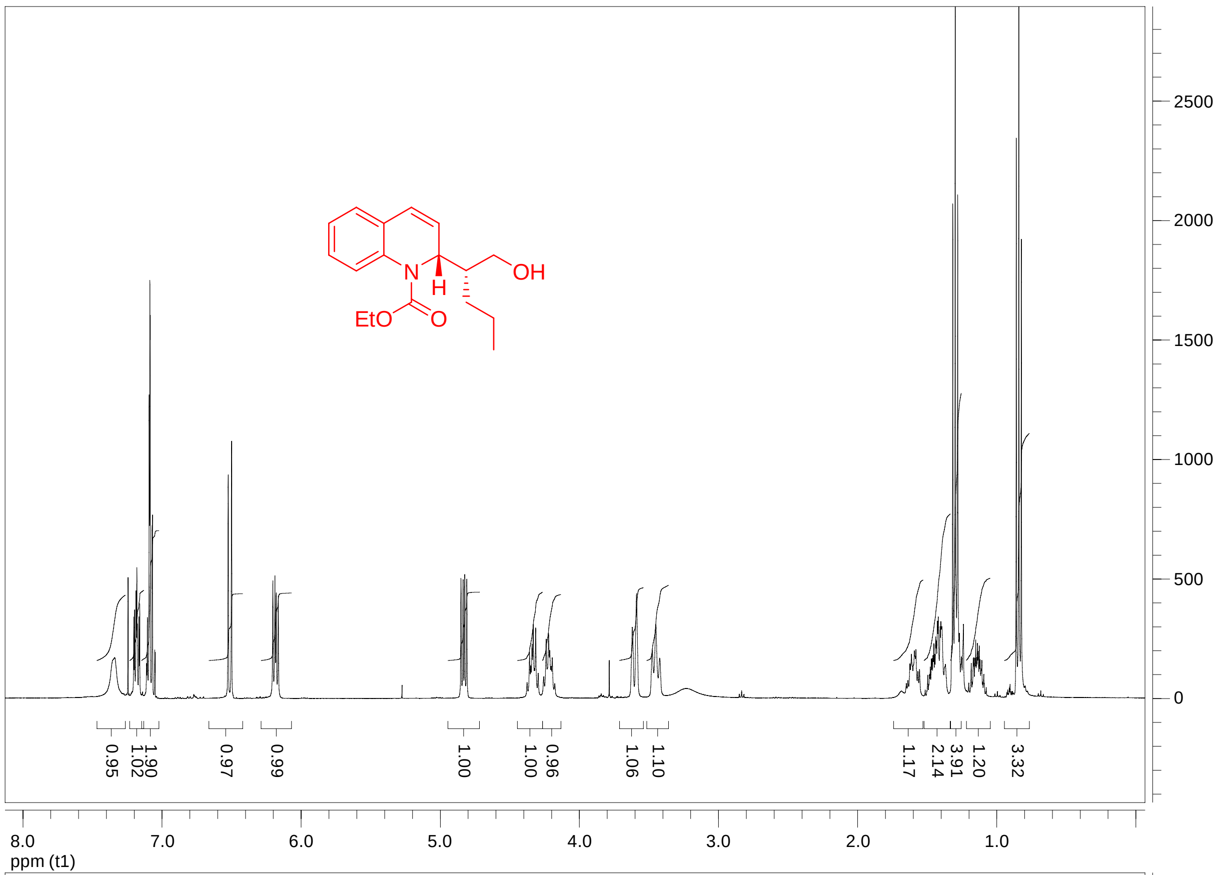
156.38
155.81
129.88
128.39
125.44
124.97
114.97
113.04
110.84
63.83
62.59
55.43
53.80
37.91
14.41
13.12
MeO
N
H
OH
Me
EtO
O
150
100
50
ppm (t1)
800
700
600
500
400
300
200
100
0

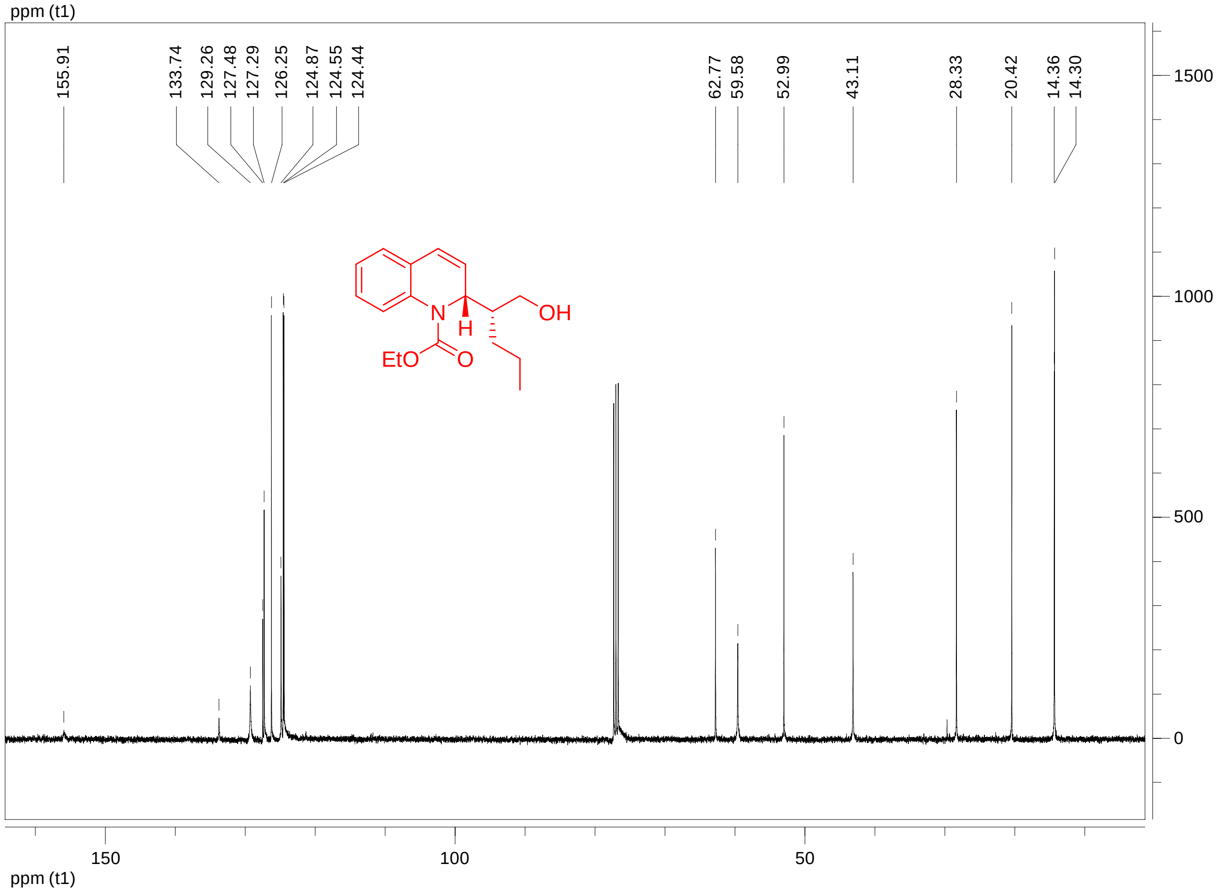


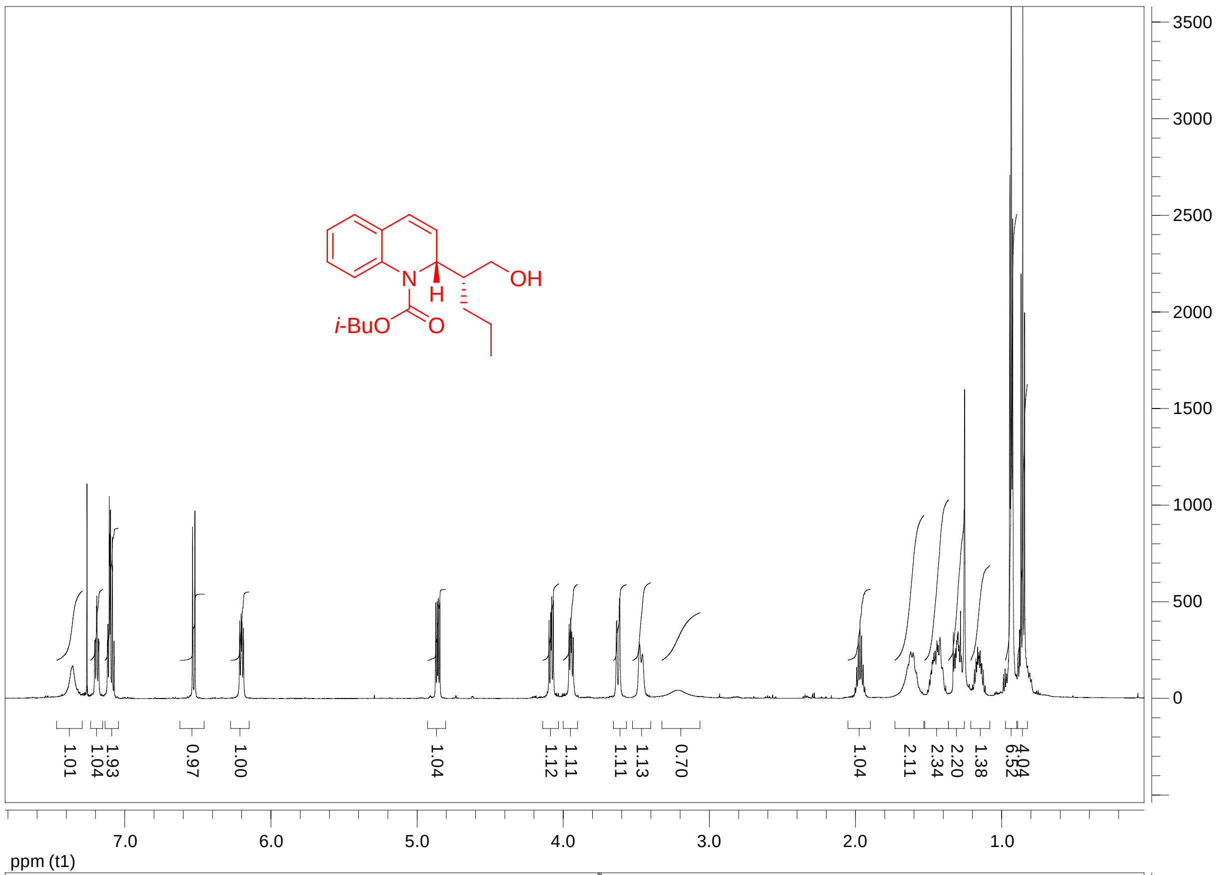
N
H
OH
EtO
O
0.66
0.86
1.87
0.94
1.00
0.96
0.91
1.03
1.08
1.03
1.52
1.37
3.69
3.21
2000
1500
1000
500
0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

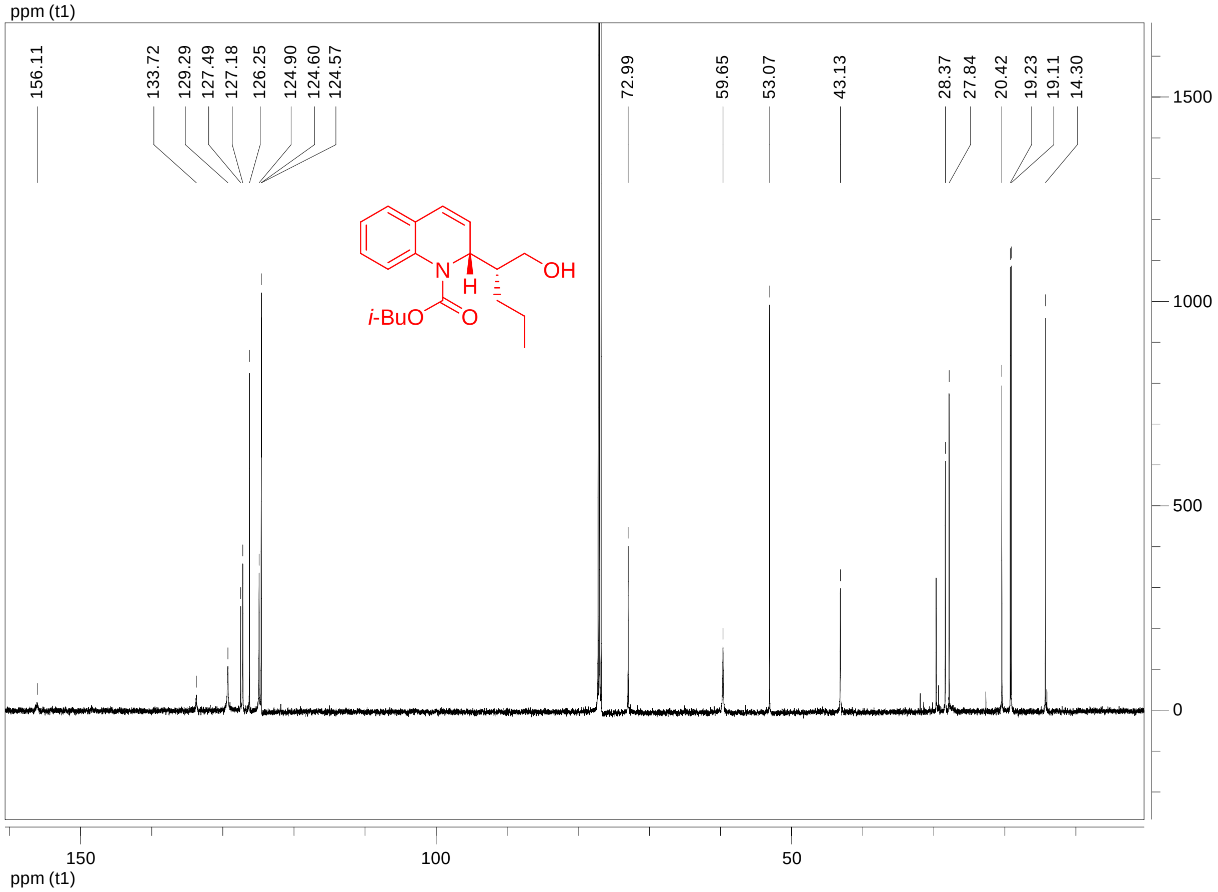


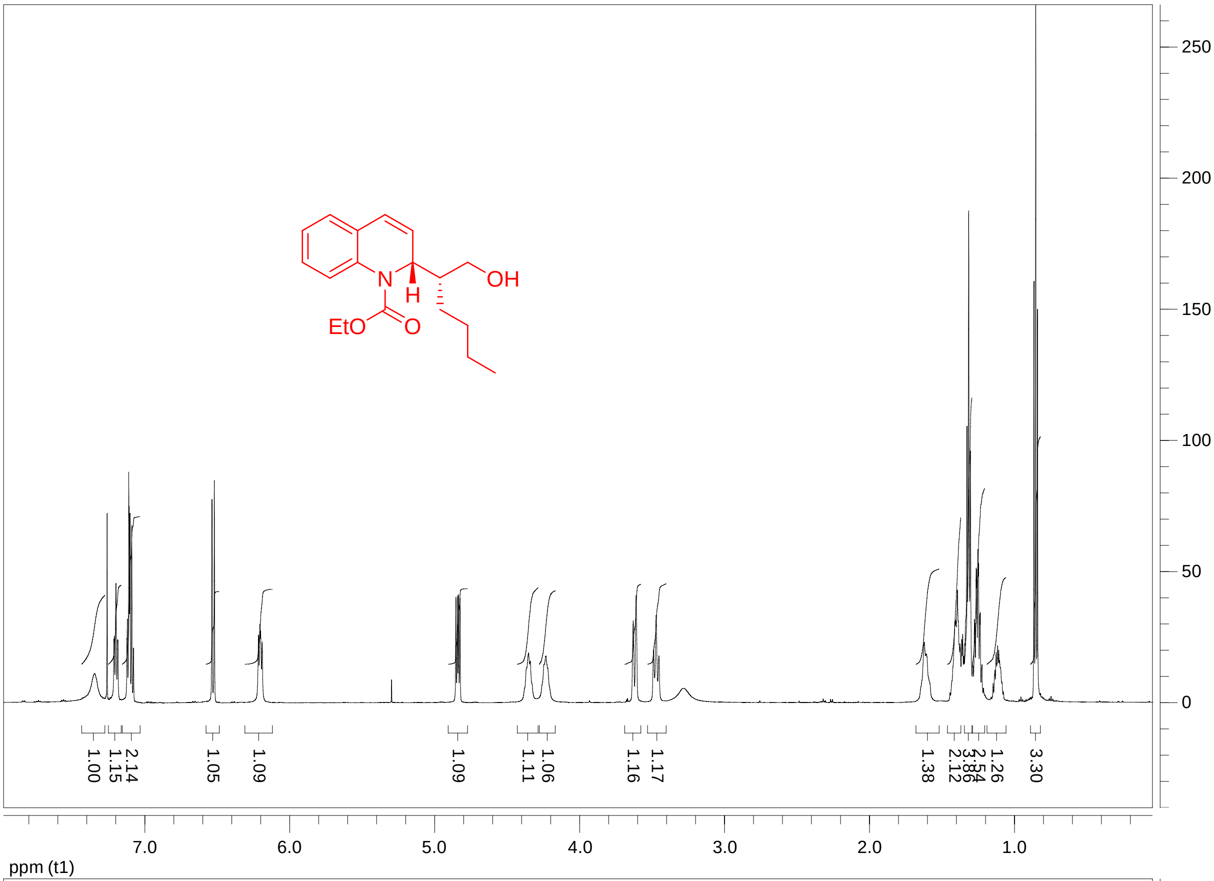
155.87
133.77
129.26
127.48
127.29
126.25
124.89
124.56
124.45
62.76
59.06
52.93
44.85
18.92
14.36
11.72
N
H
OH
EtO
O
1000
500
0
150
100
50
ppm (t1)


N
H
OH
EtO
O
0.95
1.02
1.90
0.97
0.99
1.00
1.00
0.96
1.06
1.10
1.17
2.14
3.91
1.20
3.32
2500
2000
1500
1000
500
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

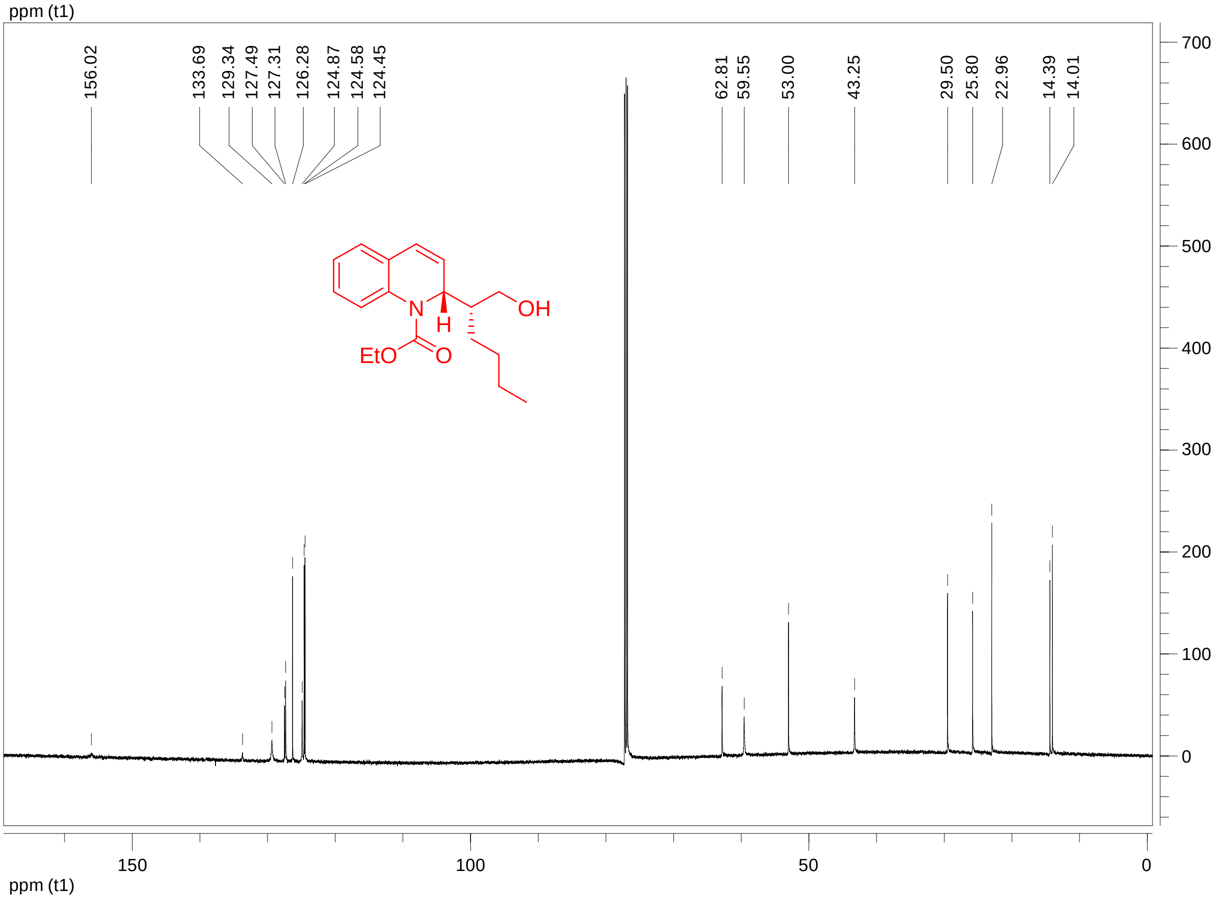

155.91
133.74
129.26
127.48
127.29
126.25
124.87
124.55
124.44
62.77
59.58
52.99
43.11
28.33
20.42
14.36
14.30
N
H
OH
EtO
O
1500
1000
500
0
150
100
50
ppm (t1)


N
H
OH
i-BuO
O
1.01
1.04
1.93
0.97
1.00
1.04
1.12
1.11
1.11
1.13
0.70
1.04
2.11
2.34
2.20
1.38
6.52
4.04
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)
3500
3000
2500
2000
1500
1000
500
0

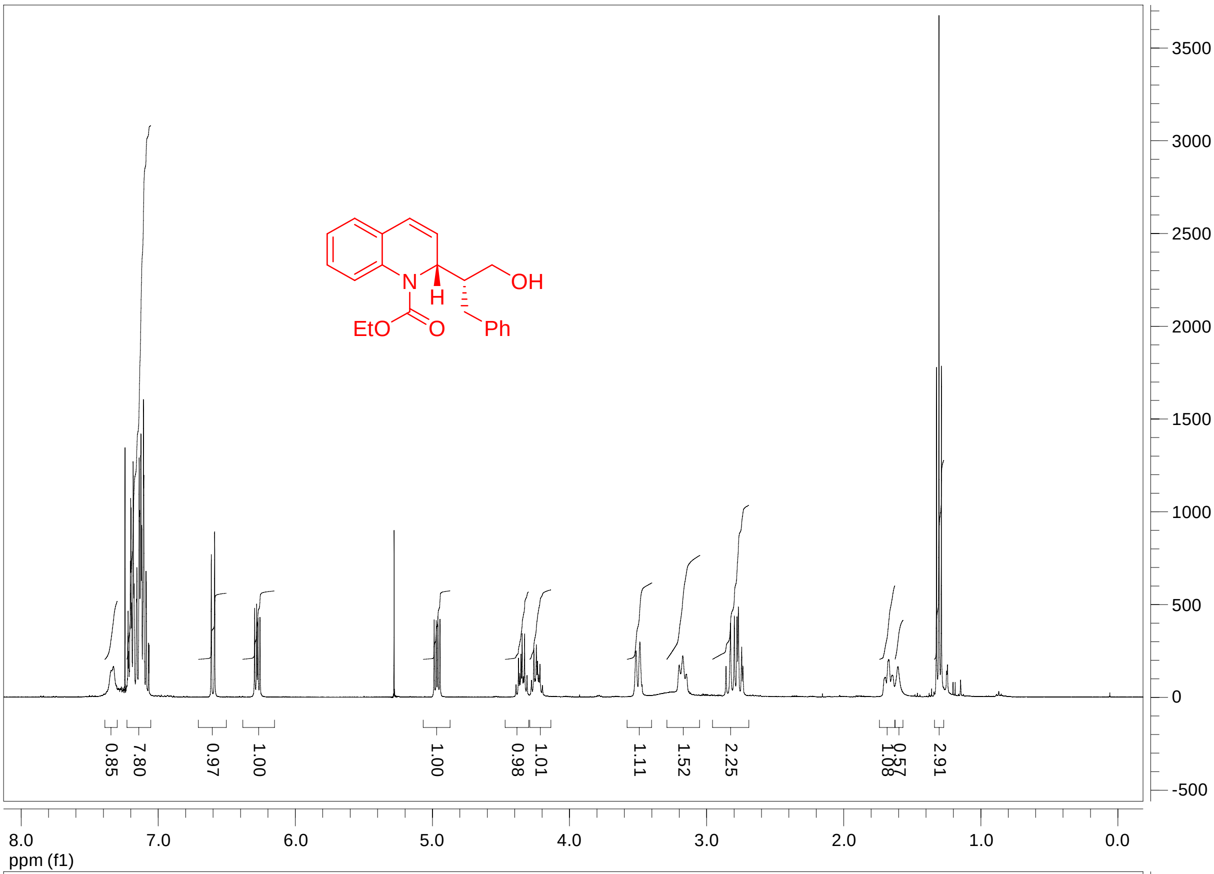

156.11
133.72
129.29
127.49
127.18
126.25
124.90
124.60
124.57
72.99
59.65
53.07
43.13
28.37
27.84
20.42
19.23
19.11
14.30
N
H
OH
i-BuO
O
150
100
50
ppm (t1)
1500
1000
500
0



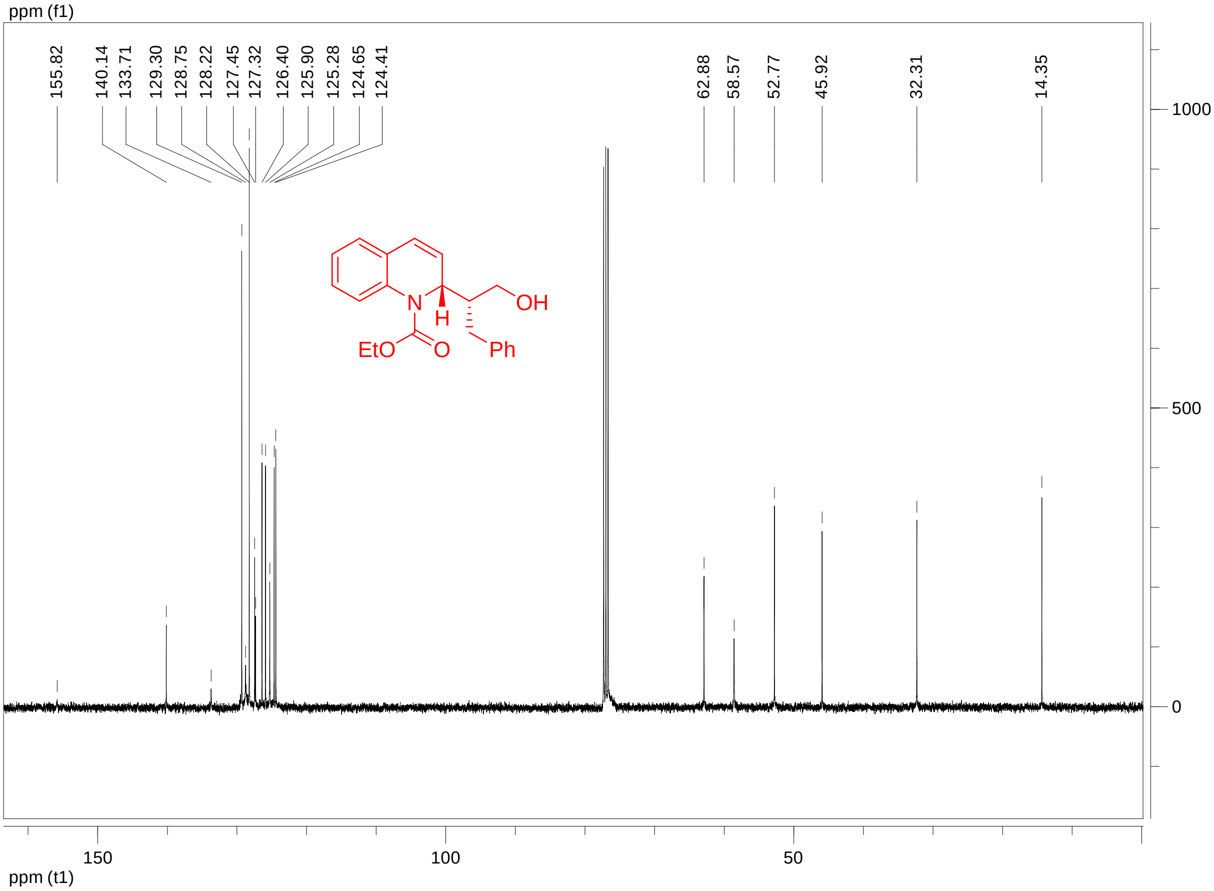
N
H
OH
EtO
O
1.00
1.15
2.14
1.05
1.09
1.09
1.11
1.06
1.16
1.17
1.38
2.12
3.86
2.54
1.26
3.30
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)
250
200
150
100
50
0



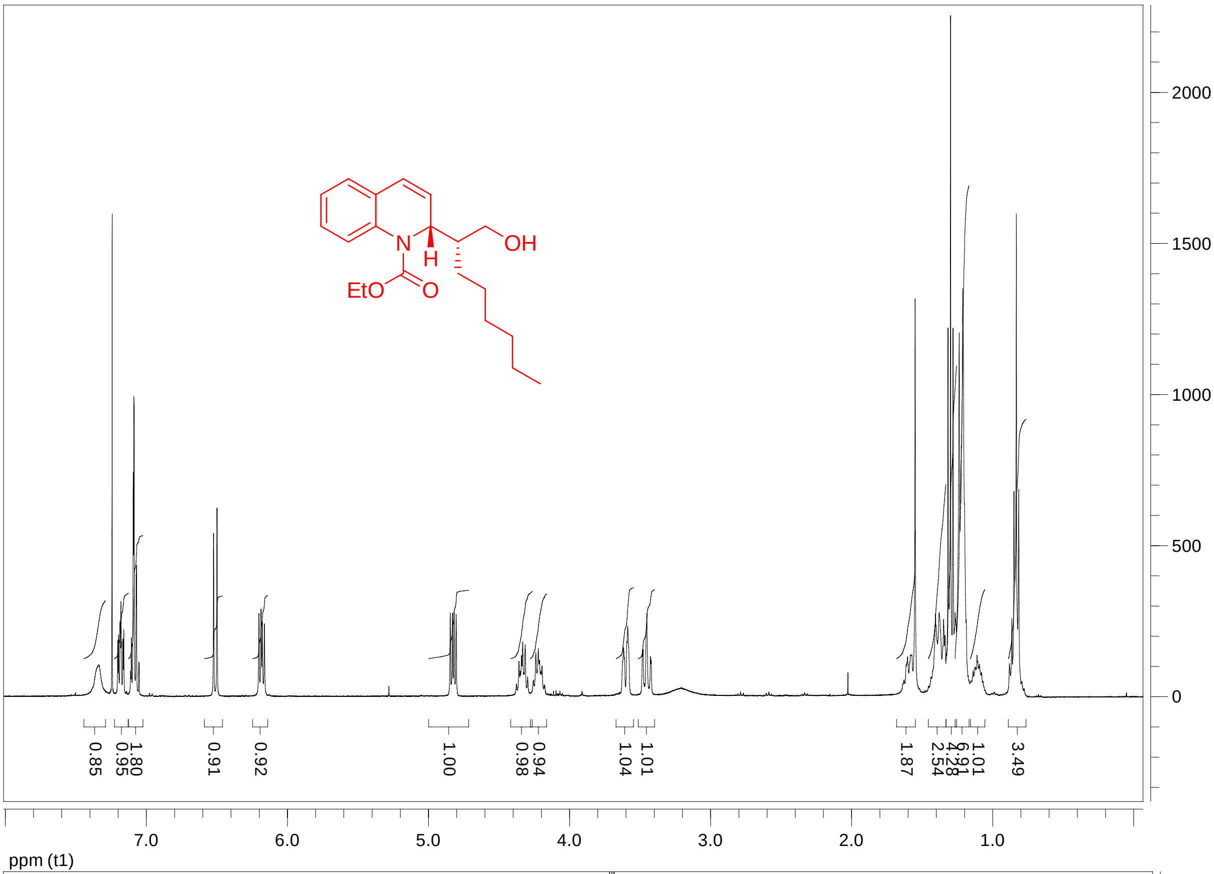
156.02
133.69
129.34
127.49
127.31
126.28
124.87
124.58
124.45
62.81
59.55
53.00
43.25
29.50
25.80
22.96
14.39
14.01
N
H
OH
EtO
O
150
100
50
0
ppm (t1)
700
600
500
400
300
200
100
0



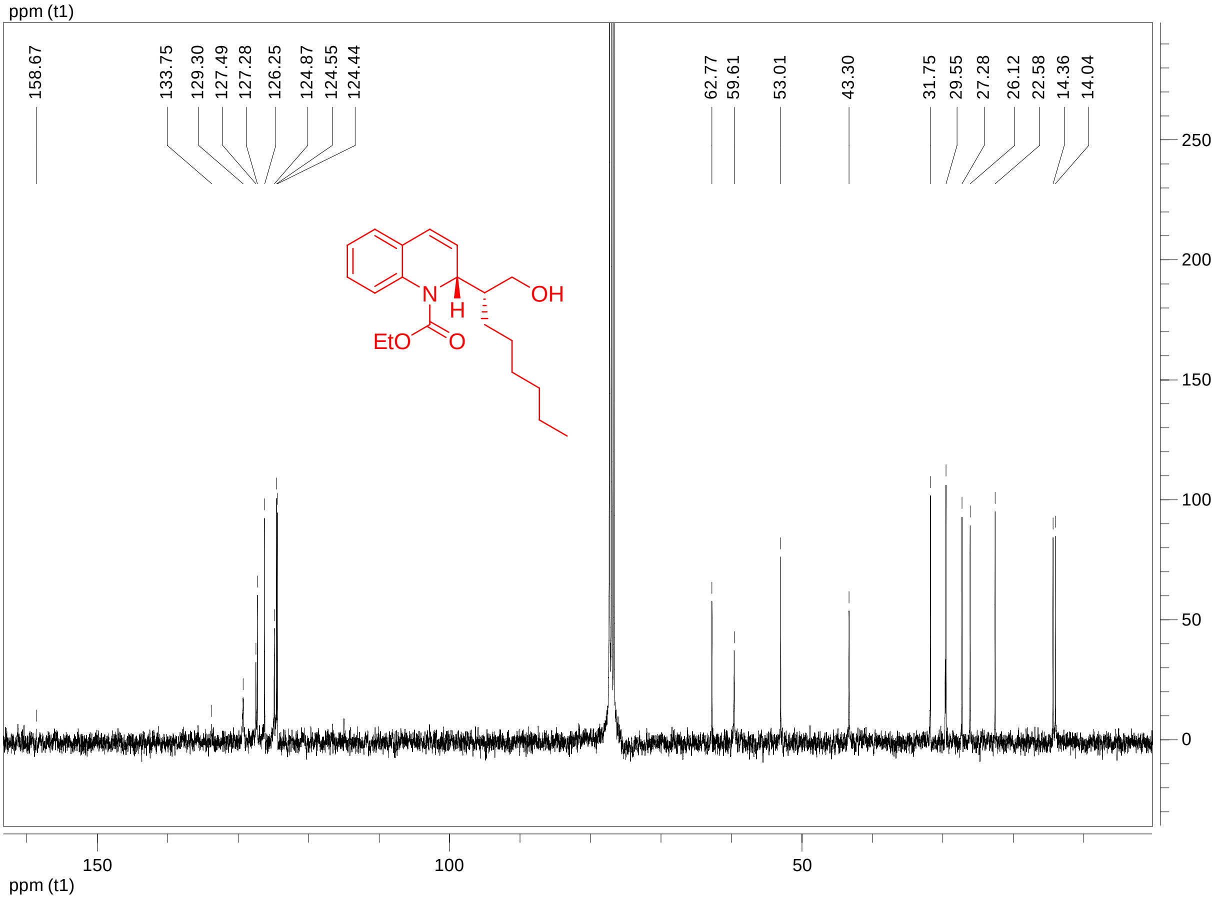
EtO
O
N
H
OH
Ph
0.85
7.80
0.97
1.00
1.00
0.98
1.01
1.11
1.52
2.25
1.08
0.57
2.91
3500
3000
2500
2000
1500
1000
500
0
-500
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)



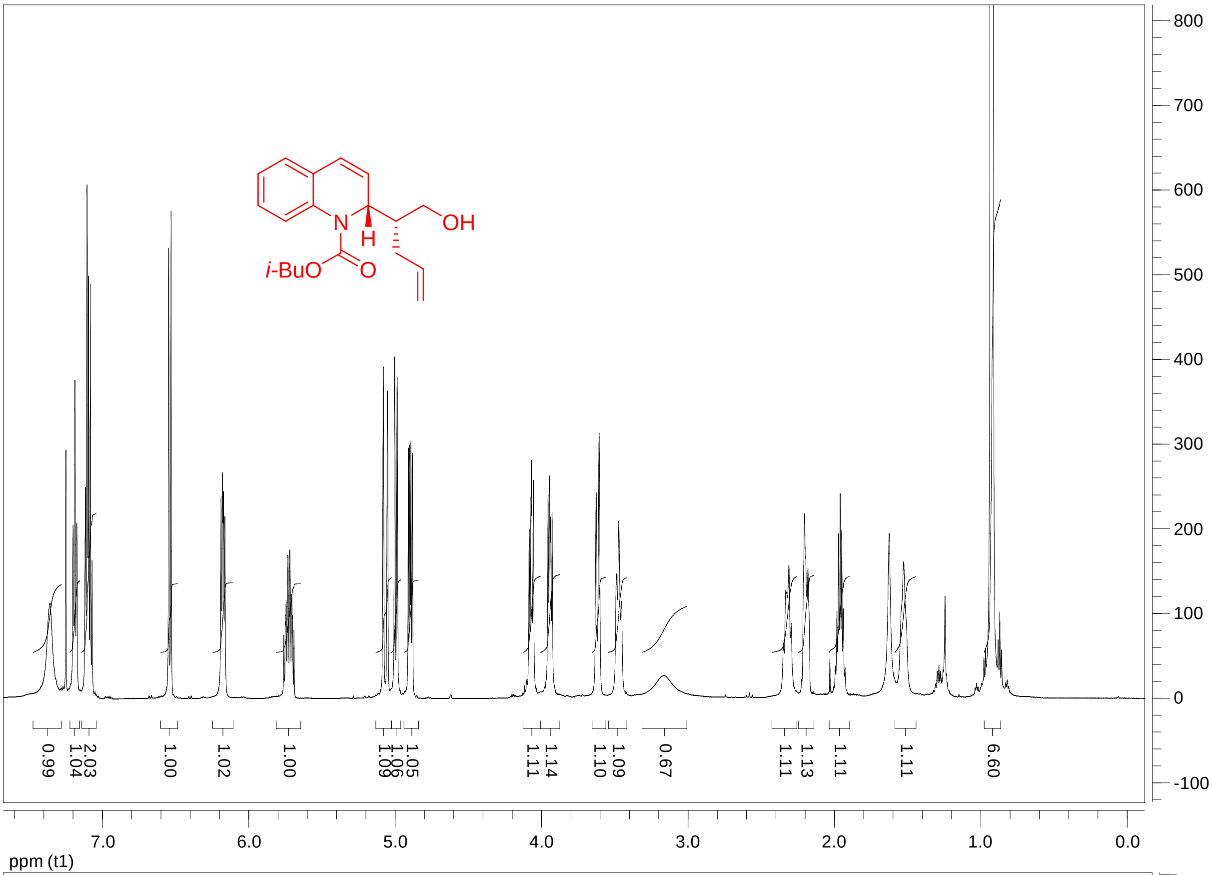
155.82
140.14
133.71
129.30
128.75
128.22
127.45
127.32
126.40
125.90
125.28
124.65
124.41
62.88
58.57
52.77
45.92
32.31
14.35
EtO
O
N
H
OH
Ph
1000
500
0
150
100
50
ppm (t1)

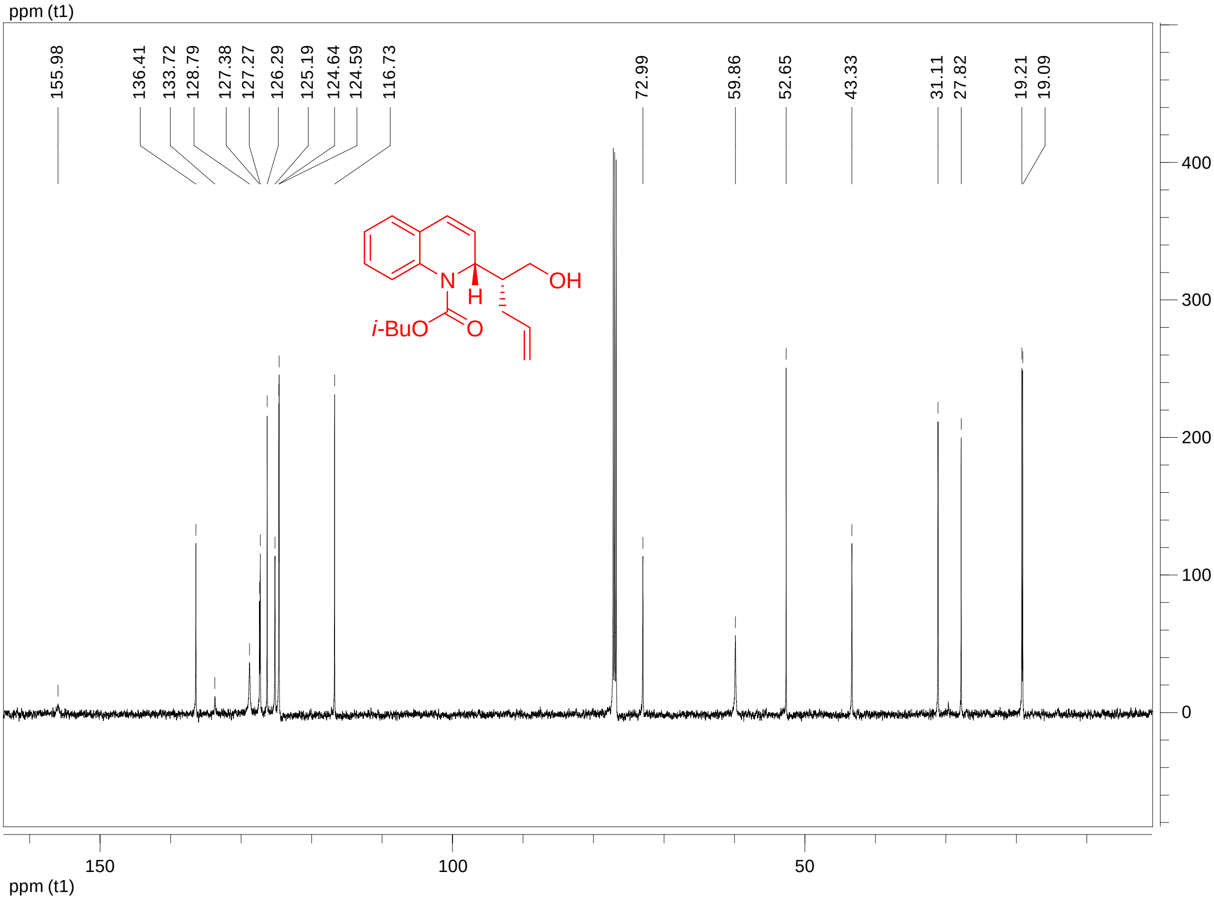


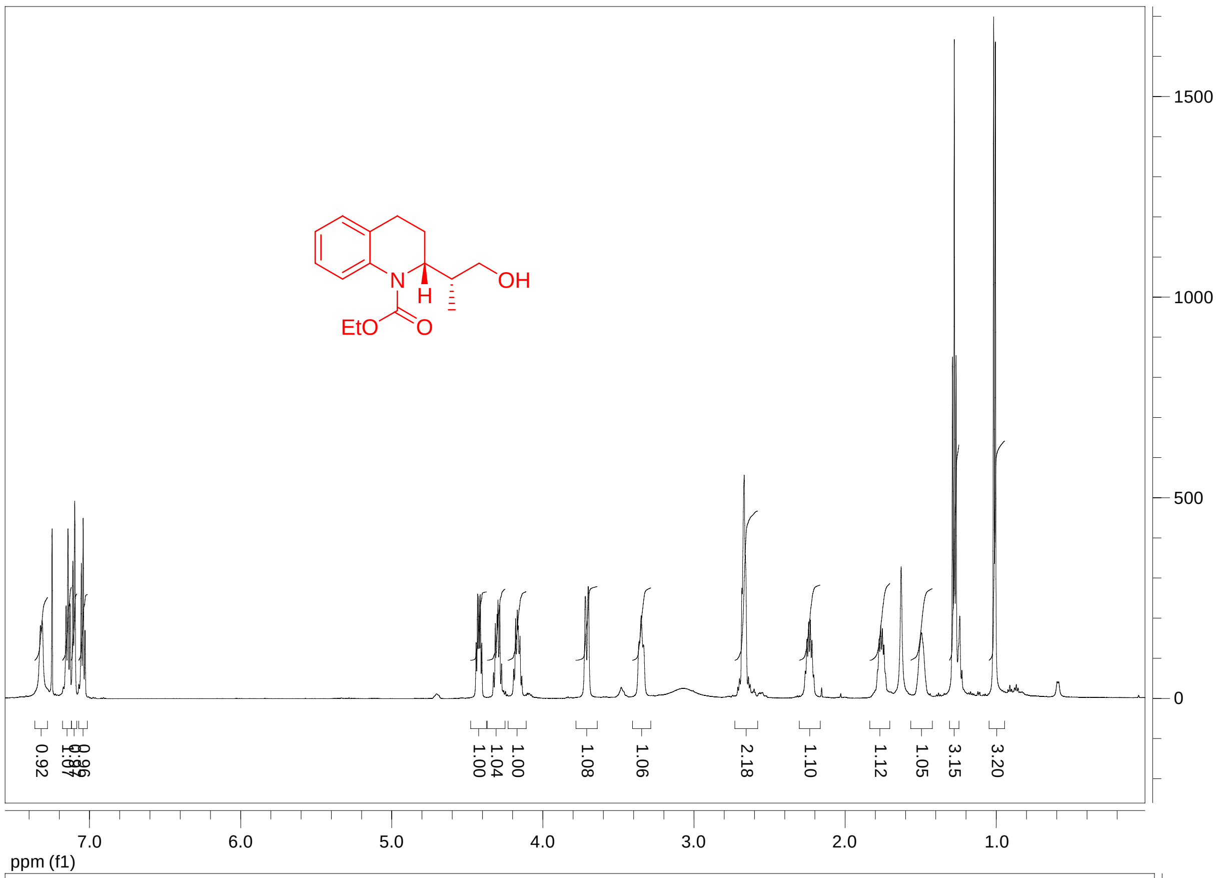
N
H
OH
EtO
O
0.85
0.95
1.80
0.91
0.92
1.00
0.98
0.94
1.04
1.01
1.87
2.54
4.28
6.91
1.01
3.49
2000
1500
1000
500
0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)



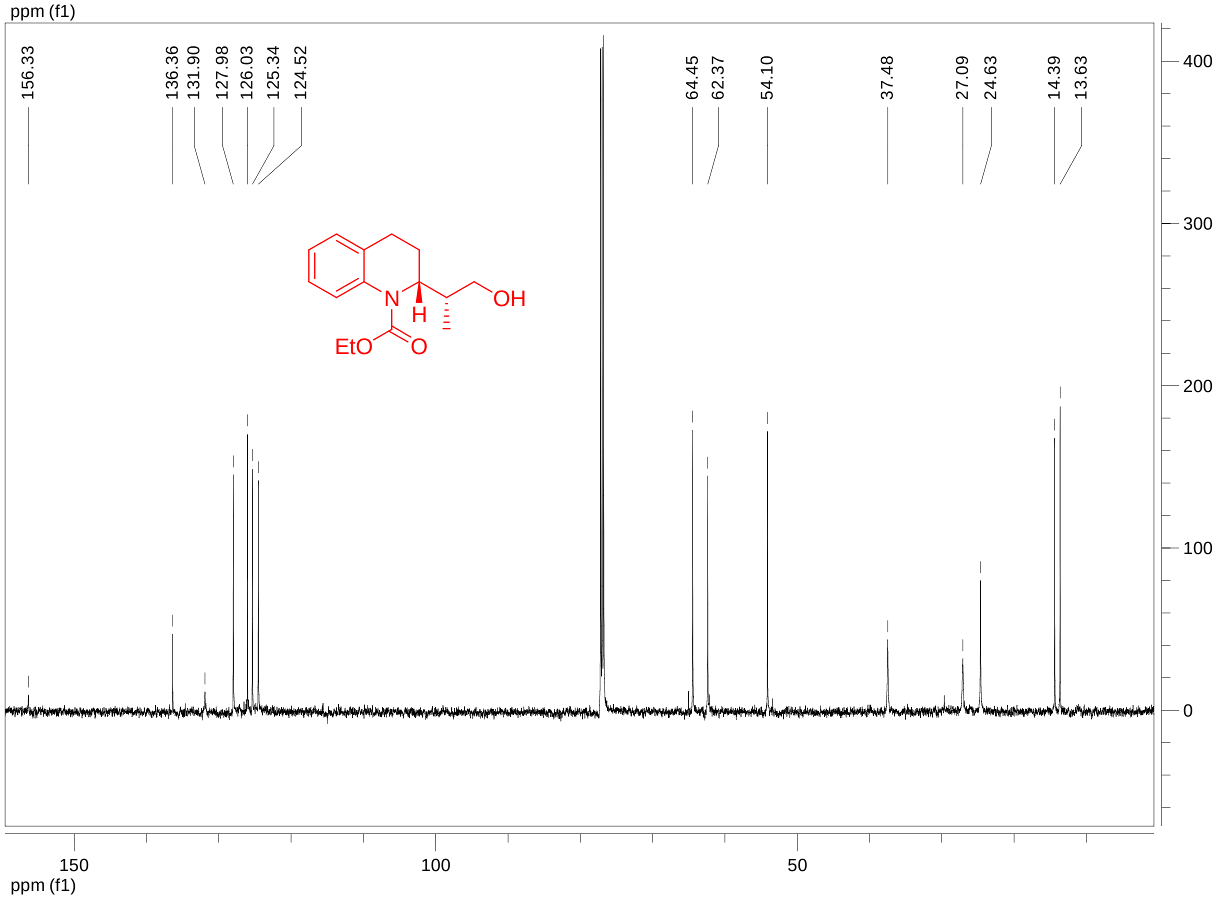
158.67
133.75
129.30
127.49
127.28
126.25
124.87
124.55
124.44
62.77
59.61
53.01
43.30
31.75
29.55
27.28
26.12
22.58
14.36
14.04
N
H
OH
EtO
O
250
200
150
100
50
0
150
100
50
ppm (t1)


i-BuO
N
H
O
OH
0.99
1.04
2.03
1.00
1.02
1.00
1.09
1.06
1.05
1.11
1.14
1.10
1.09
0.67
1.11
1.13
1.11
1.11
6.60
ppm (t1)

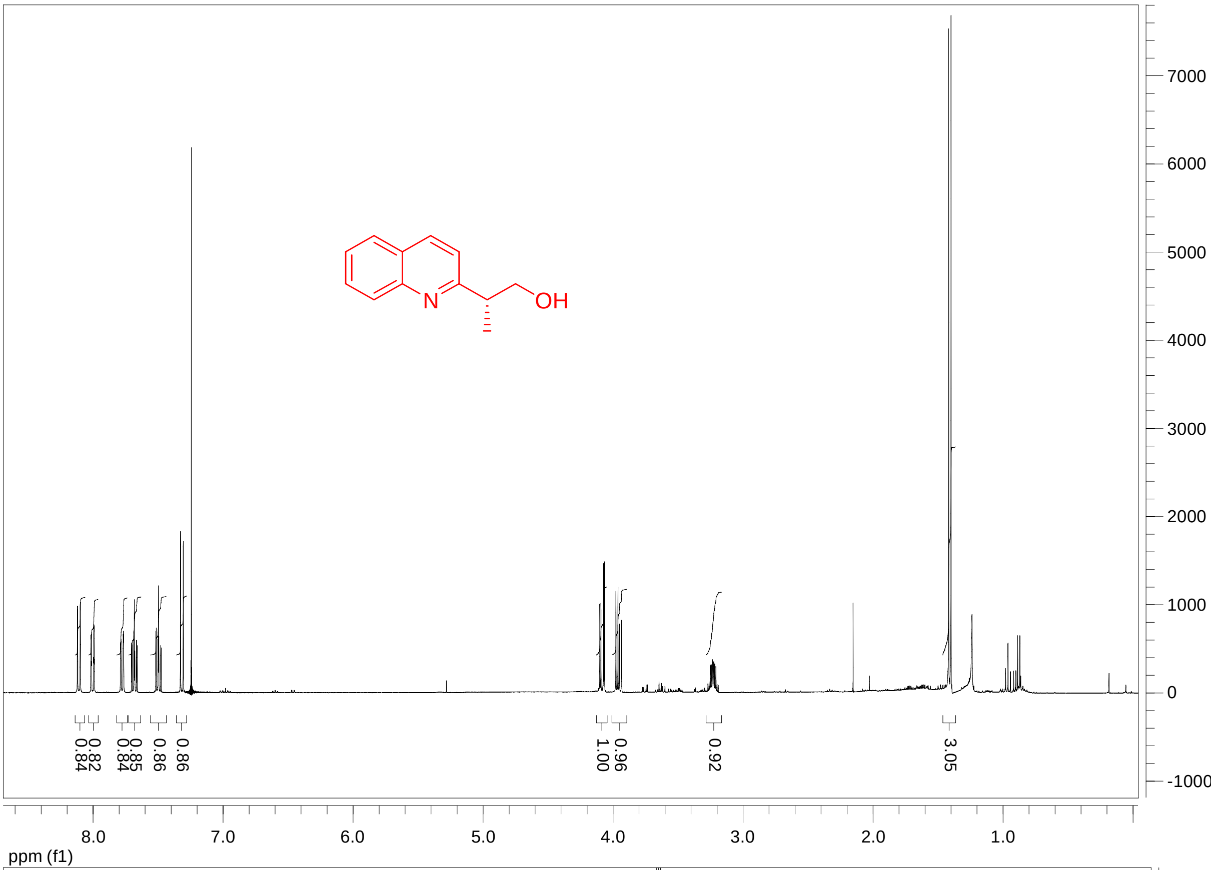

155.98
136.41
133.72
128.79
127.38
127.27
126.29
125.19
124.64
124.59
116.73
72.99
59.86
52.65
43.33
31.11
27.82
19.21
19.09
i-BuO
N
H
O
OH
ppm (t1)



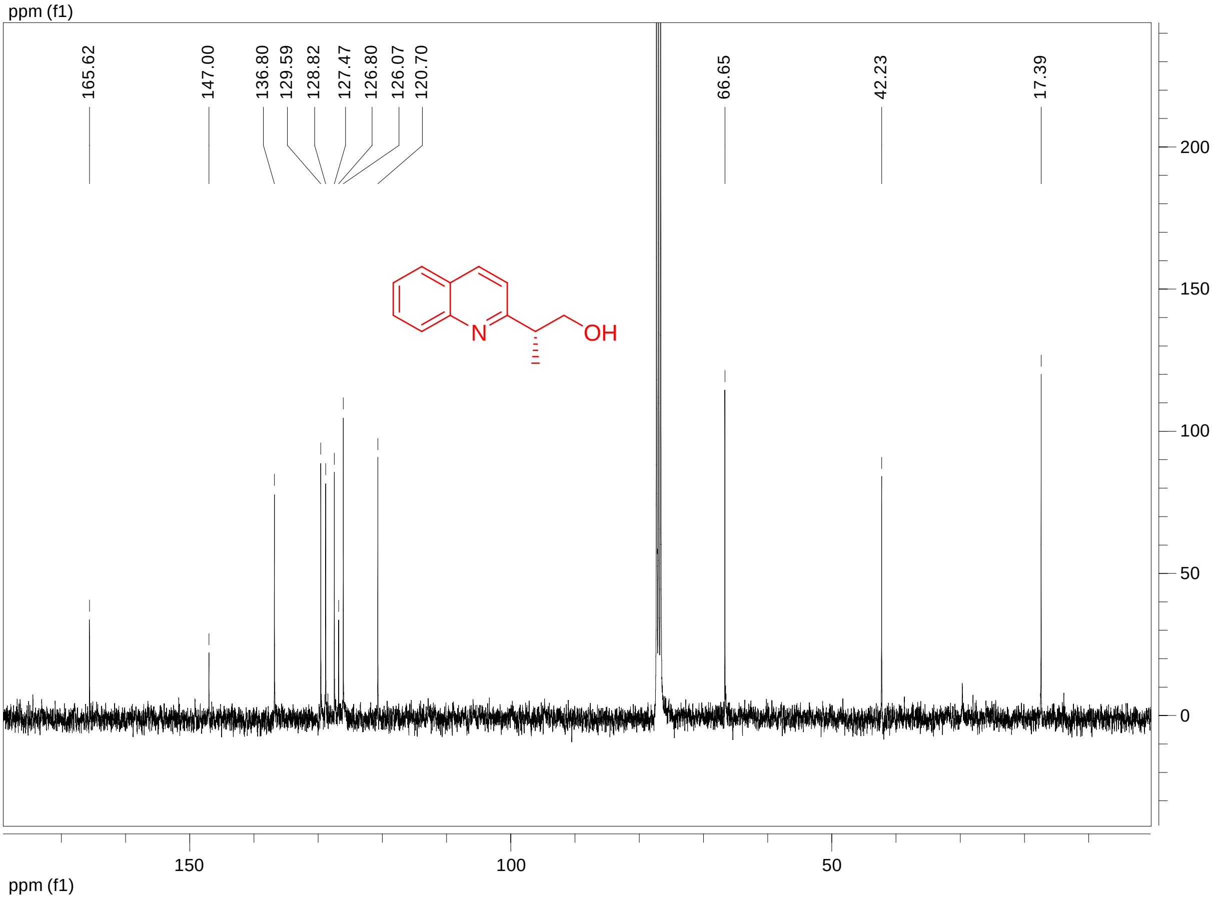
OH
EtO
O
N
H
ppm (f1)



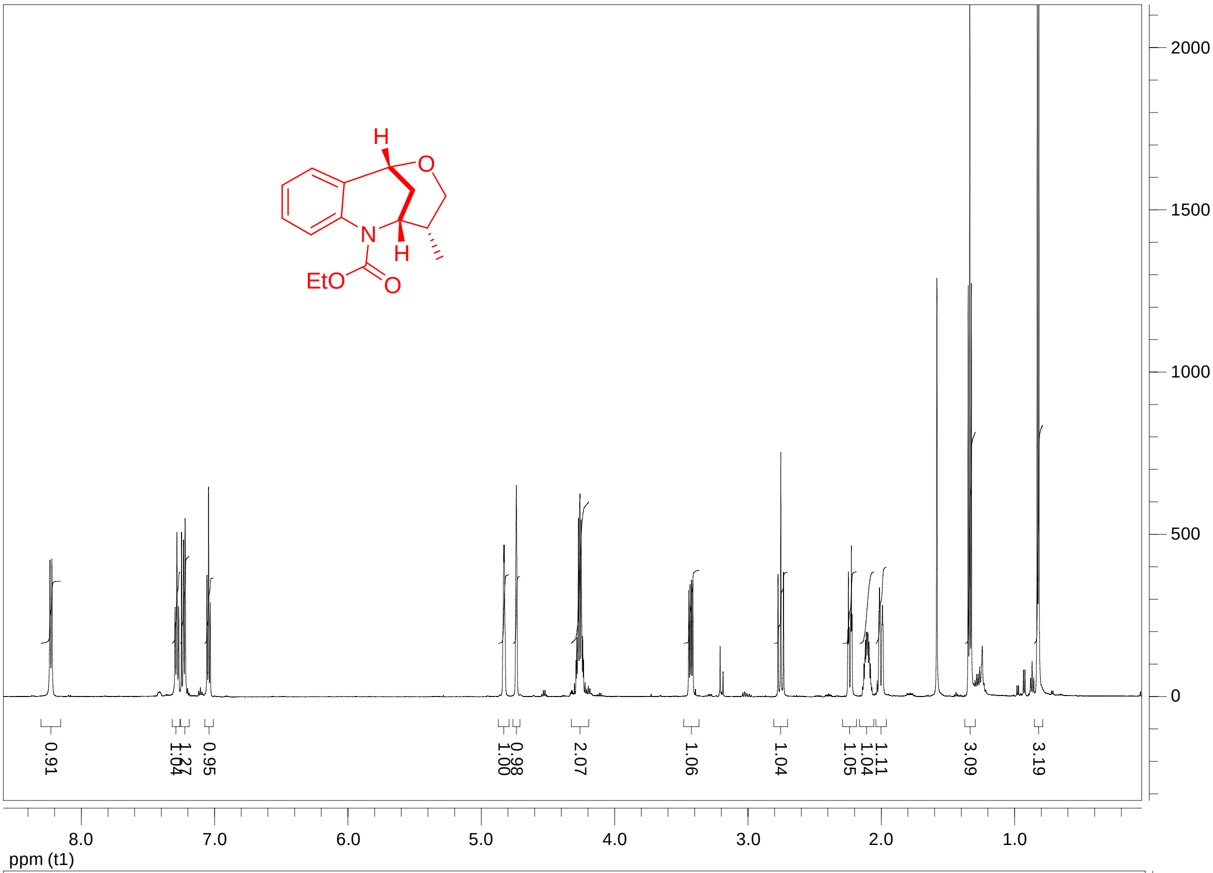
156.33
136.36
131.90
127.98
126.03
125.34
124.52
64.45
62.37
54.10
37.48
27.09
24.63
14.39
13.63
OH
EtO
O
N
H
ppm (f1)



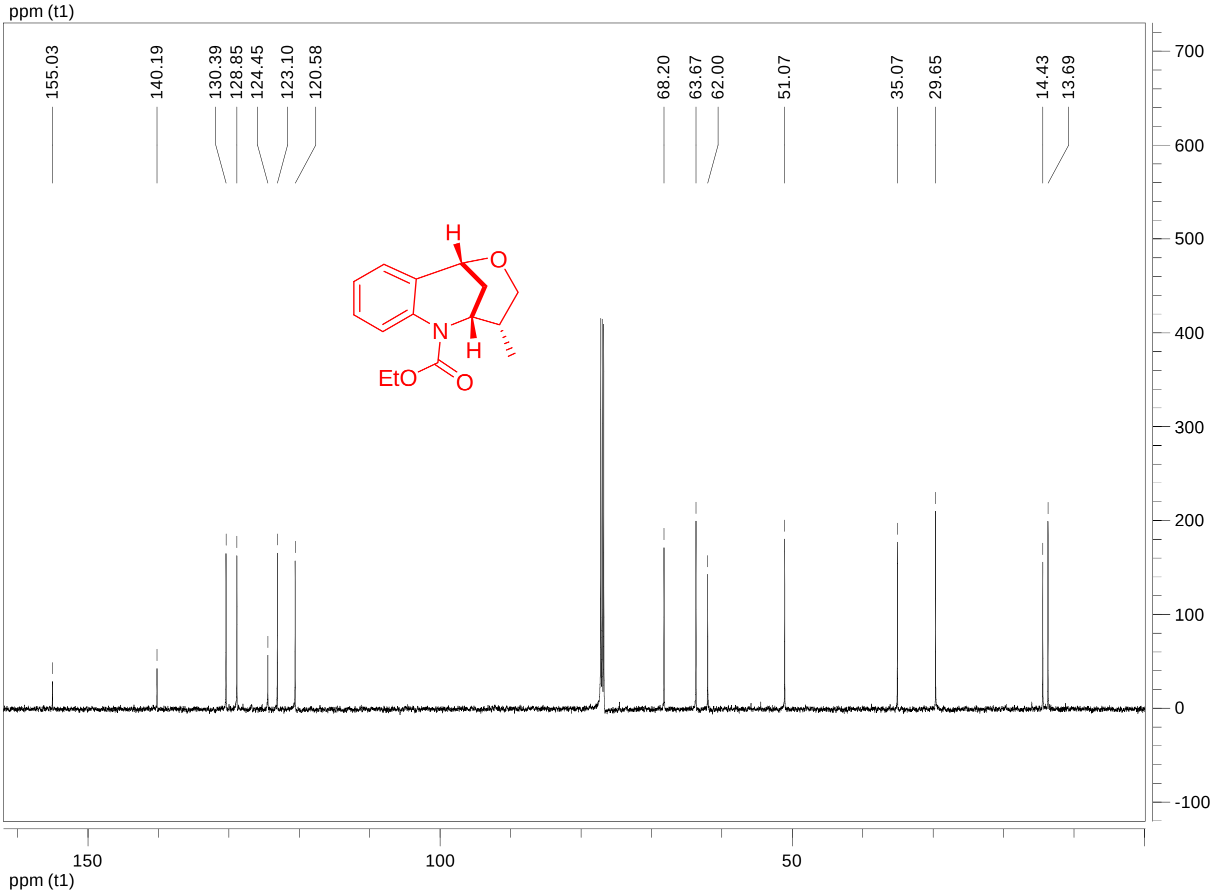
OH
N
0.84
0.82
0.84
0.85
0.86
0.86
1.00
0.96
0.92
3.05
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



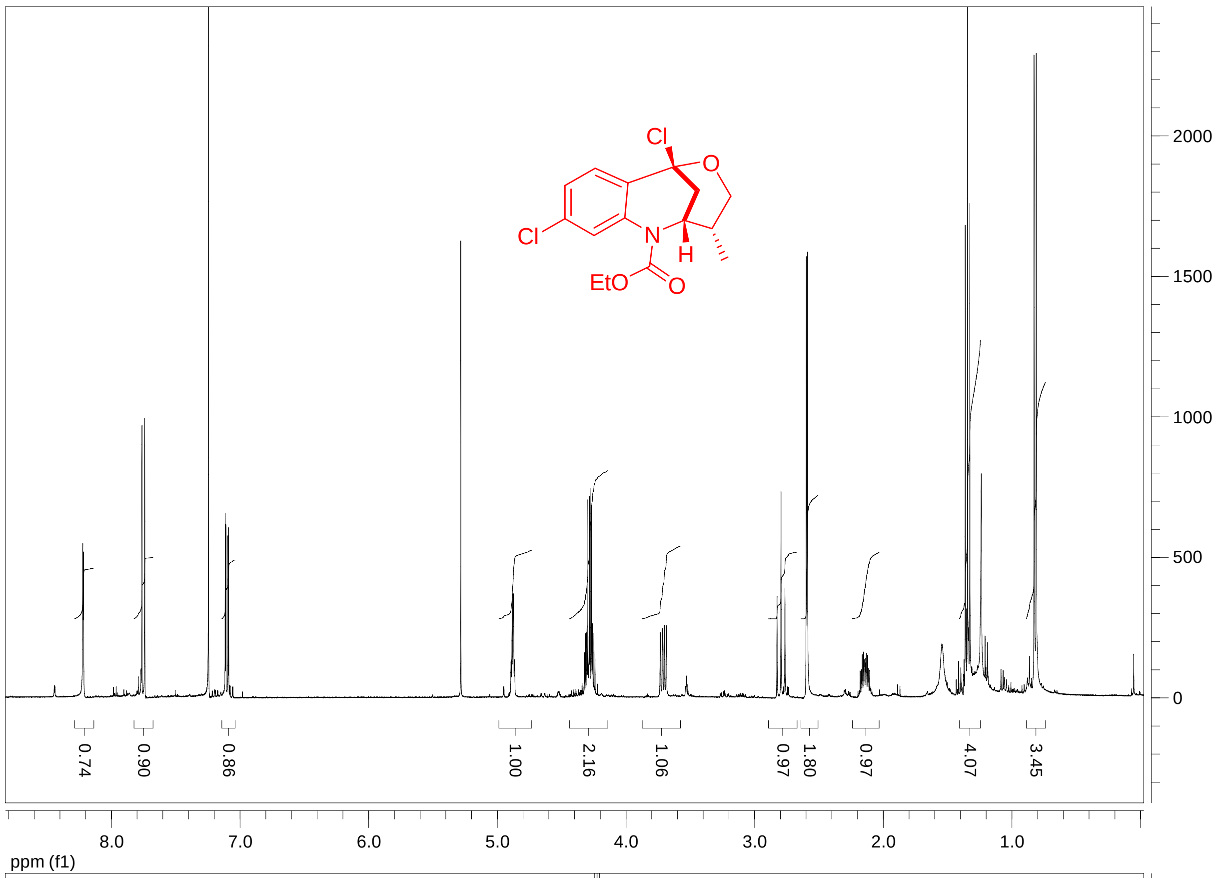
165.62
147.00
136.80
129.59
128.82
127.47
126.80
126.07
120.70
66.65
42.23
17.39
OH
N
150
100
50
ppm (f1)



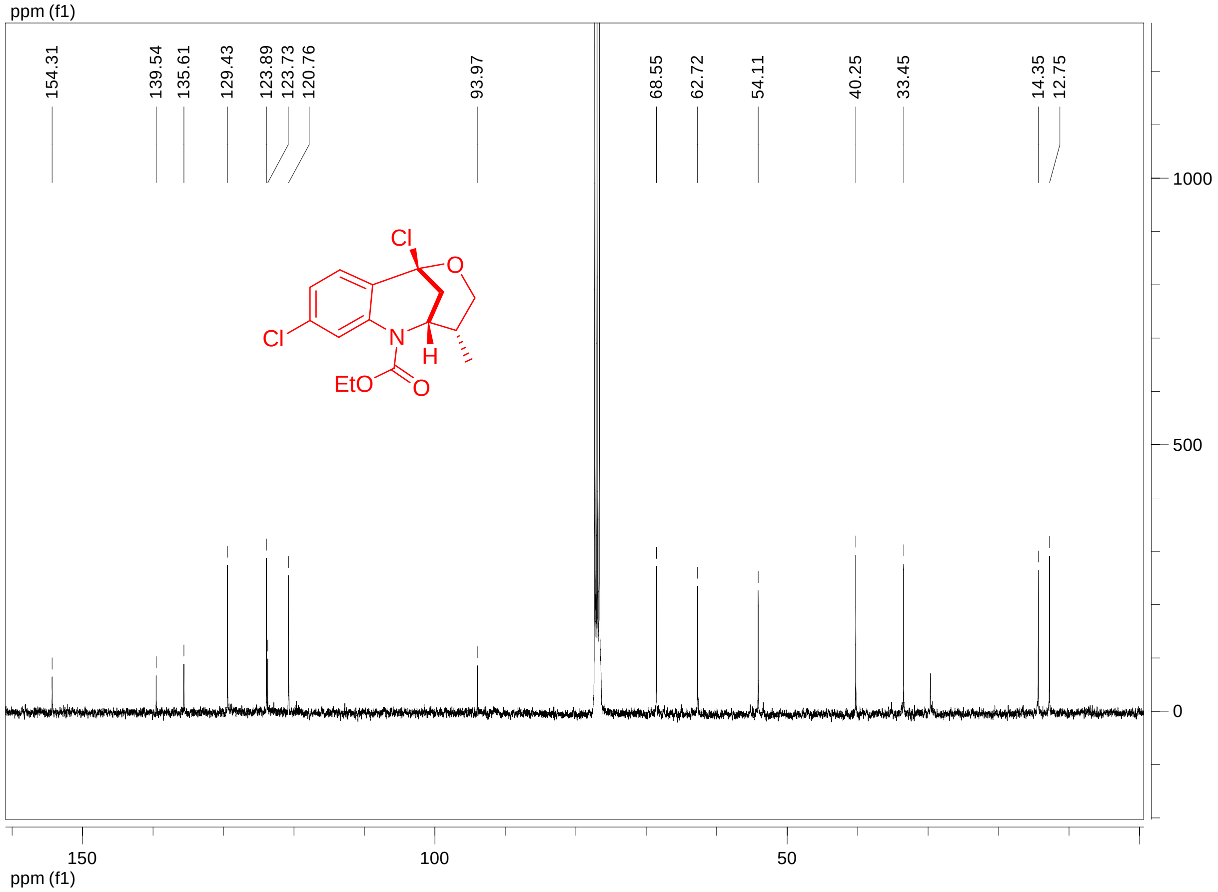
EtO
0.91
1.04
1.27
0.95
1.00
0.98
2.07
1.06
1.04
1.05
1.04
1.11
3.09
3.19
ppm (t1)



155.03
140.19
130.39
128.85
124.45
123.10
120.58
68.20
63.67
62.00
51.07
35.07
29.65
14.43
13.69
EtO
ppm (t1)



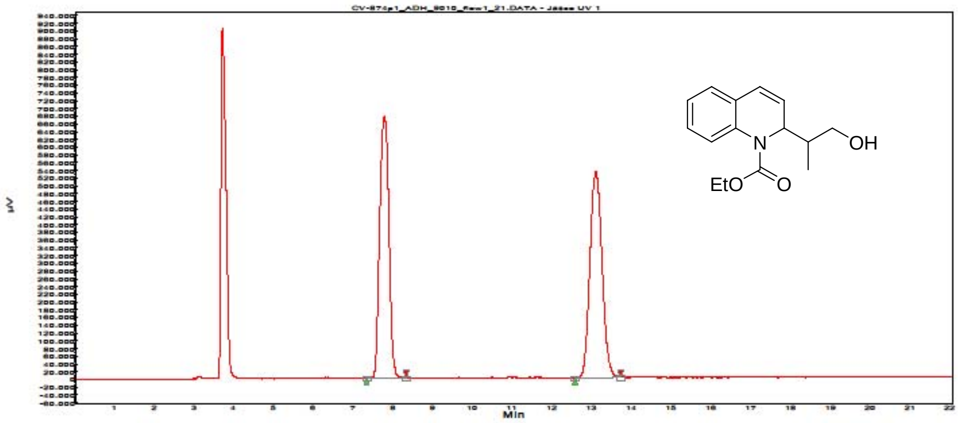
Cl
O
Cl
N
H
EtO
O
0.74
0.90
0.86
1.00
2.16
1.06
0.97
1.80
0.97
4.07
3.45
2000
1500
1000
500
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)



154.31
139.54
135.61
129.43
123.89
123.73
120.76
93.97
68.55
62.72
54.11
40.25
33.45
14.35
12.75
Cl
O
Cl
N
H
EtO
O
1000
500
0
150
100
50
ppm (f1)

Chromatogram : CV-874p1_ADH_9010_flow1_21

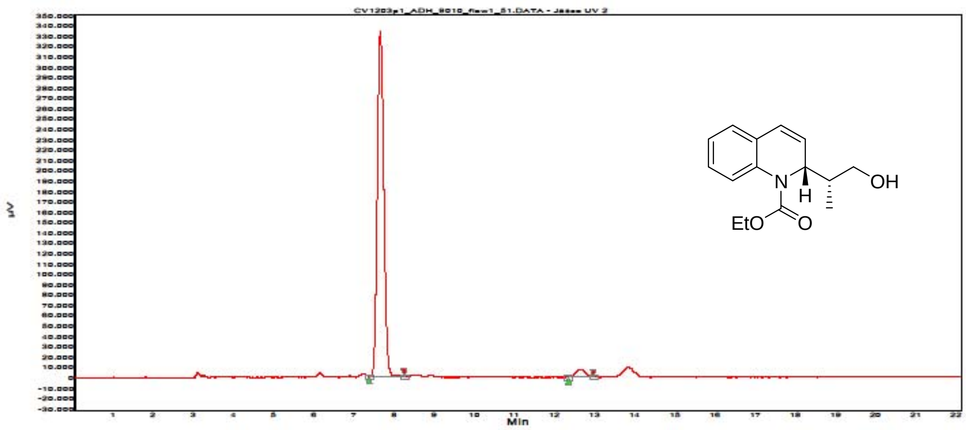
Data file: CV-874p1_ADH_9010_flow1_21.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 06.01.2012 19:58:51



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,349	7,800	8,343	49,127
2	12,580	13,108	13,731	50,873
Total				100,000

Chromatogram : CV1203p1_ADH_9010_flow1_51

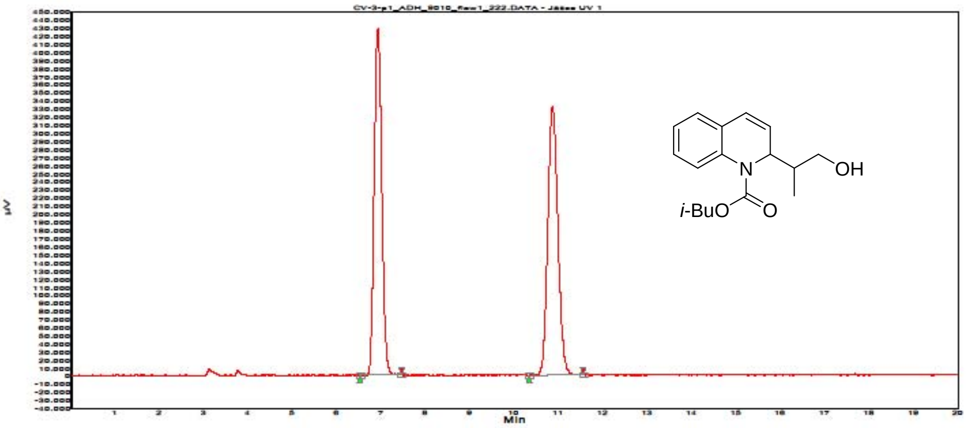
Data file: CV1203p1_ADH_9010_flow1_51.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 12.09.2012 12:35:01



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,390	7,667	8,253	96,815
2	12,348	12,642	12,964	3,185
Total				100,000

Chromatogram : CV-3-p1_ADH_9010_flow1_222

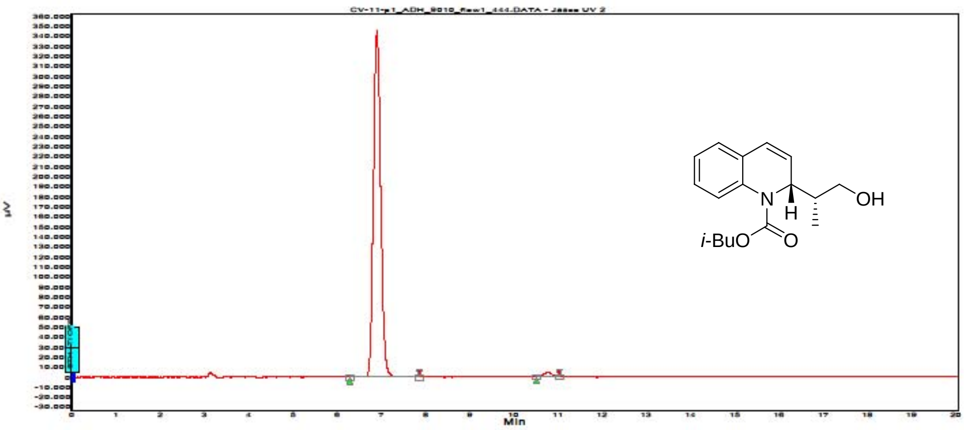
Data file: CV-3-p1_ADH_9010_flow1_222.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 24.04.2012 12:49:03



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,536	6,942	7,485	49,102
2	10,351	10,883	11,567	50,898
Total				100,000

Chromatogram : CV-11-p1_ADH_9010_flow1_444

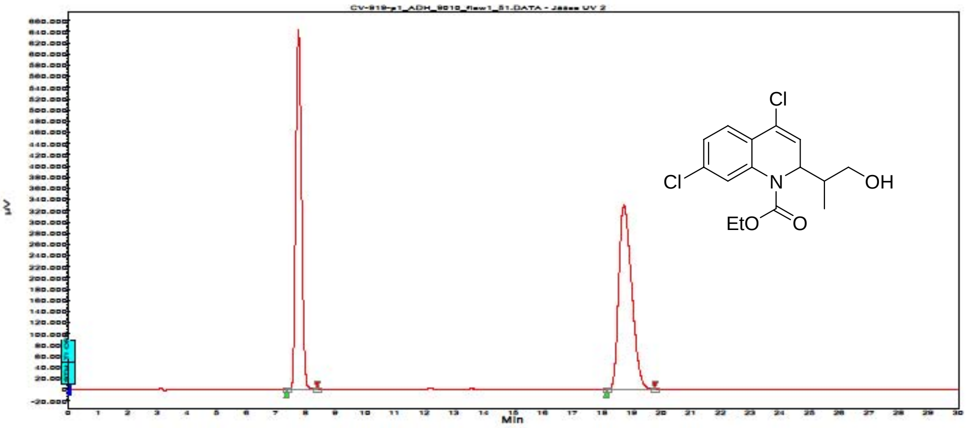
Data file: CV-11-p1_ADH_9010_flow1_444.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 24.04.2012 14:14:28



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,293	6,908	7,866	98,551
2	10,516	10,783	11,034	1,449
Total				100,000

Chromatogram : CV-919-p1_ADH_9010_flow1_51

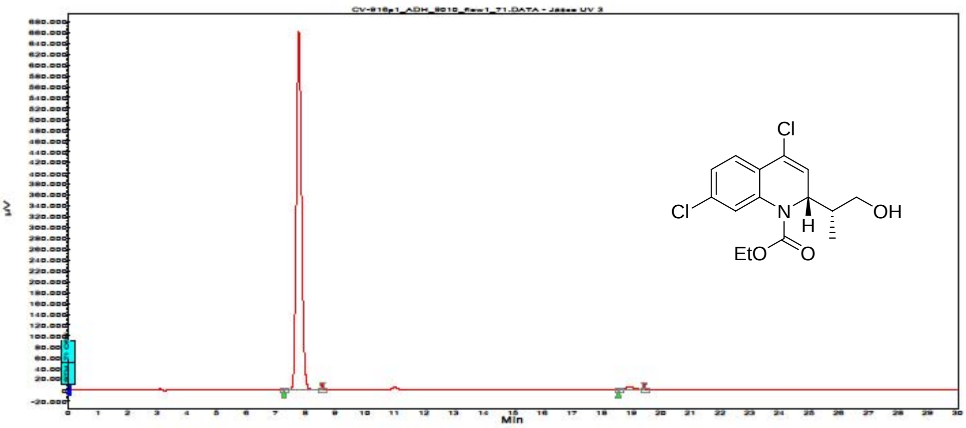
Data file: CV-919-p1_ADH_9010_flow1_51.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 06.01.2012 20:59:17



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,376	7,775	8,399	46,561
2	18,161	18,733	19,773	53,439
Total				100,000

Chromatogram : CV-916p1_ADH_9010_flow1_71

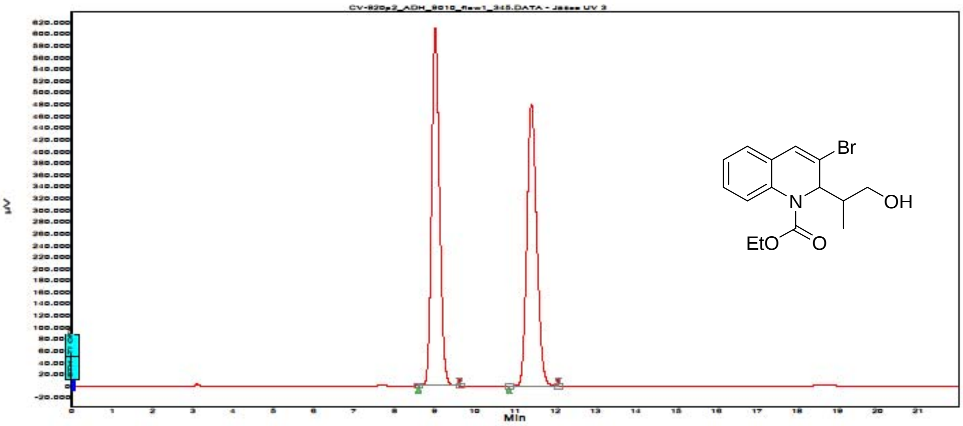
Data file: CV-916p1_ADH_9010_flow1_71.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 06.01.2012 22:04:43



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,283	7,783	8,585	98,344
2	18,564	18,967	19,432	1,656
Total				100,000

Chromatogram : CV-920p2_ADH_9010_flow1_345

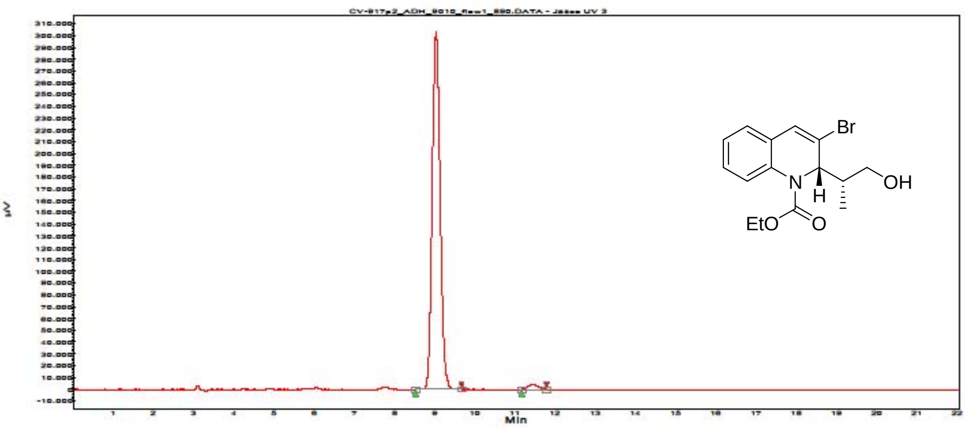
Data file: CV-920p2_ADH_9010_flow1_345.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 11.01.2012 20:56:36



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8,601	9,025	9,623	0,000	50,61	608362,2	138781,2	50,613
2	UNKNOWN	10,845	11,408	12,067	0,000	49,39	480330,4	135419,4	49,387
Total						100,00	1088692,6	274200,6	100,000

Chromatogram : CV-917p2_ADH_9010_flow1_890

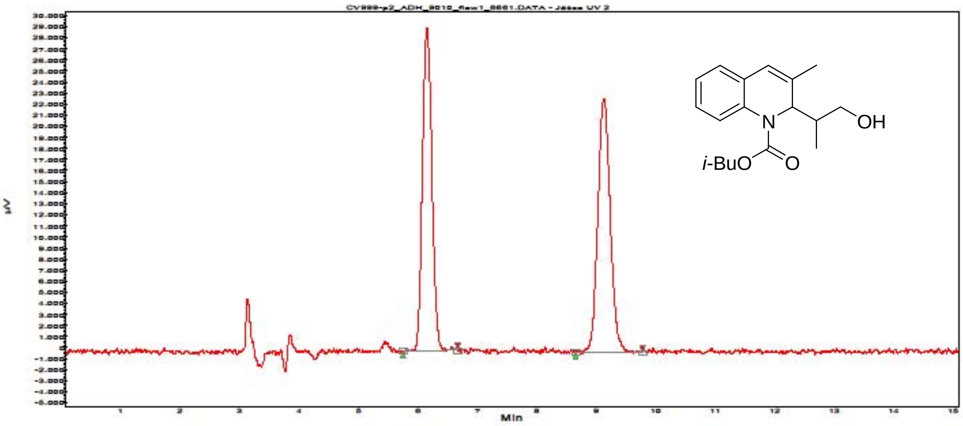
Data file: CV-917p2_ADH_9010_flow1_890.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 11.01.2012 22:28:56



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,533	9,033	9,669	98,335
2	11,169	11,450	11,783	1,665
Total				100,000

Chromatogram : CV999-p2_ADH_9010_flow1_6661

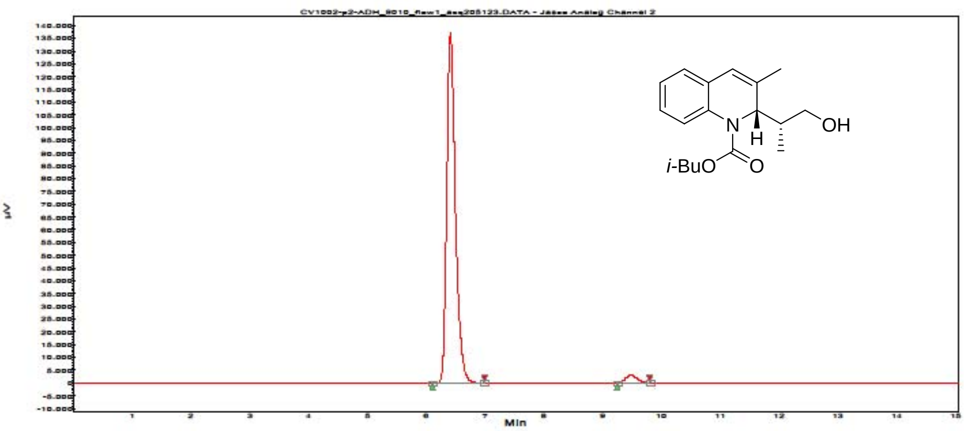
Data file: CV999-p2_ADH_9010_flow1_6661.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 09.05.2012 21:34:17



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	5,754	6,158	6,668	49,396
2	8,652	9,125	9,783	50,604
Total				100,000

Chromatogram : CV1002-p2-ADH_9010_flow1_acq205123

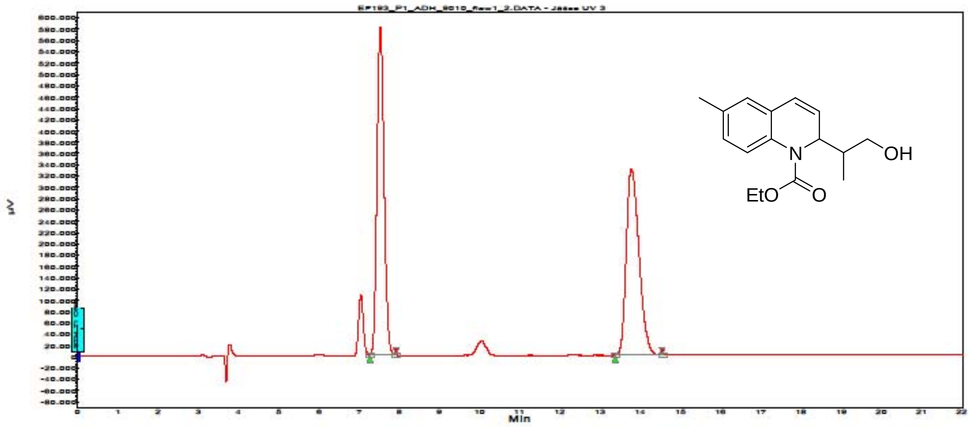
Data file: CV1002-p2-ADH_9010_flow1_acq205123.DATA
Method: HPLC2_ADH_9010_flow1_acq20
Date: 11.05.2012 12:33:23



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μV]	[μV.Min]	[%]
1	UNKNOWN	6,112	6,417	6,996	0,000	97,06	137418,1	23439,6	97,060
2	UNKNOWN	9,245	9,475	9,803	0,000	2,94	3072,5	710,0	2,940
Total						100,00	140490,6	24149,6	100,000

Chromatogram : EF193_P1_ADH_9010_flow1_2

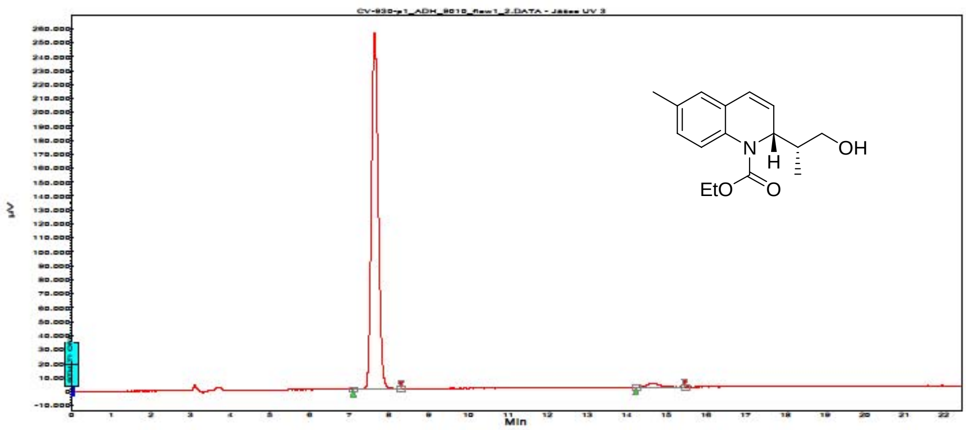
Data file: EF193_P1_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 27.01.2012 16:19:01



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7,275	7,533	7,924	0,000	49,68	580357,9	119587,5	49,685
2	UNKNOWN	13,378	13,783	14,541	0,000	50,32	329890,5	121105,6	50,315
Total						100,00	910248,4	240693,1	100,000

Chromatogram : CV-930-p1_ADH_9010_flow1_2

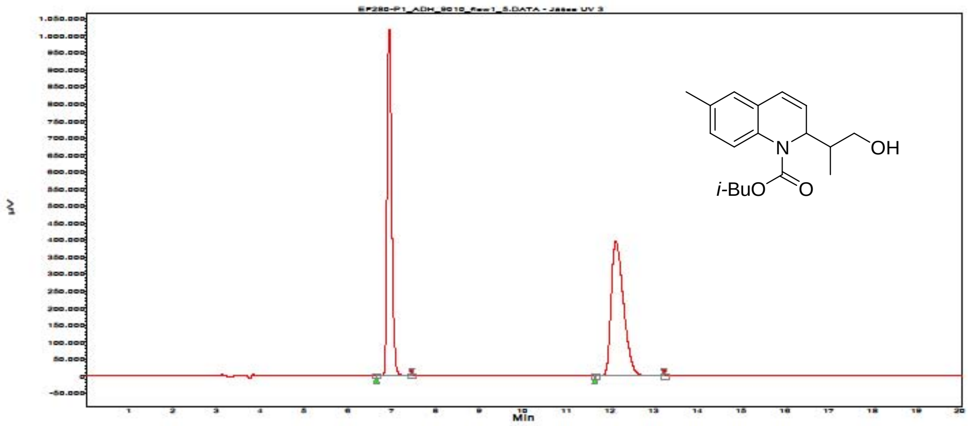
Data file: CV-930-p1_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 03.02.2012 20:23:23



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,106	7,642	8,313	98,146
2	14,225	14,658	15,454	1,854
Total				100,000

Chromatogram : EF280-P1_ADH_9010_flow1_5

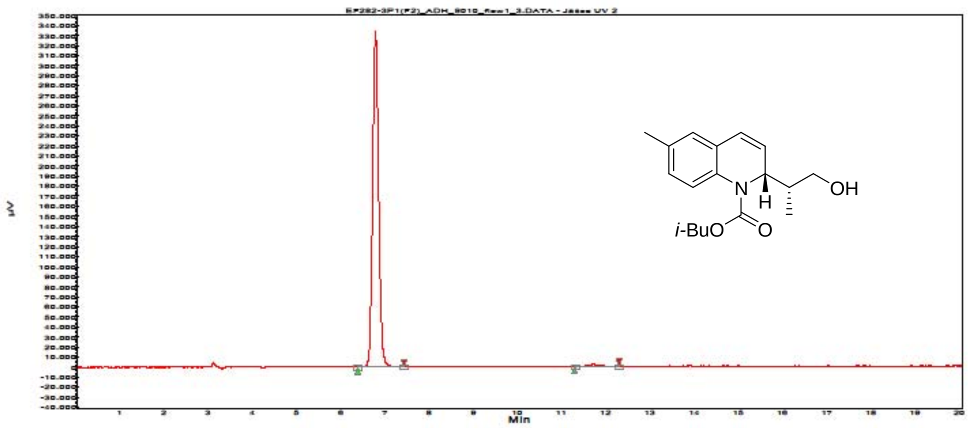
Data file: EF280-P1_ADH_9010_flow1_5.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 17.05.2012 21:10:15



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,663	6,967	7,469	49,391
2	11,653	12,133	13,233	50,609
Total				100,000

Chromatogram : EF282-3P1(F2)_ADH_9010_flow1_3

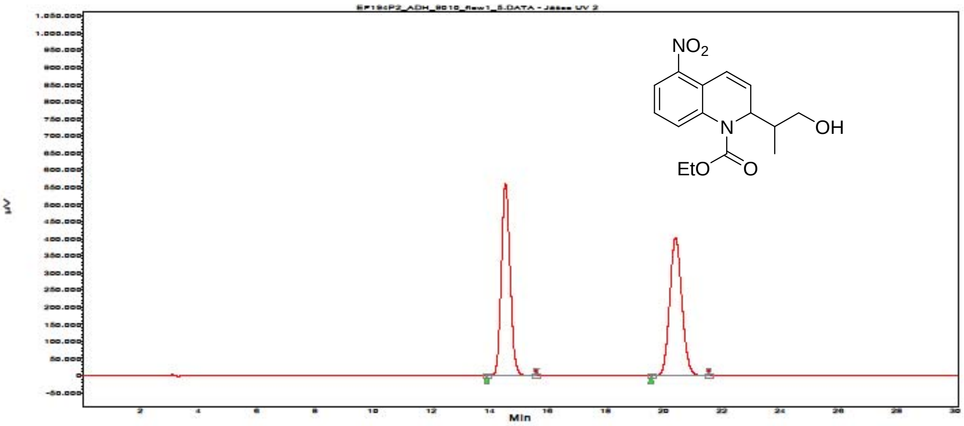
Data file: EF282-3P1(F2)_ADH_9010_flow1_3.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 21.05.2012 20:34:35



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,398	6,792	7,434	98,704
2	11,293	11,725	12,303	1,296
Total				100,000

Chromatogram : EF194P2_ADH_9010_flow1_5

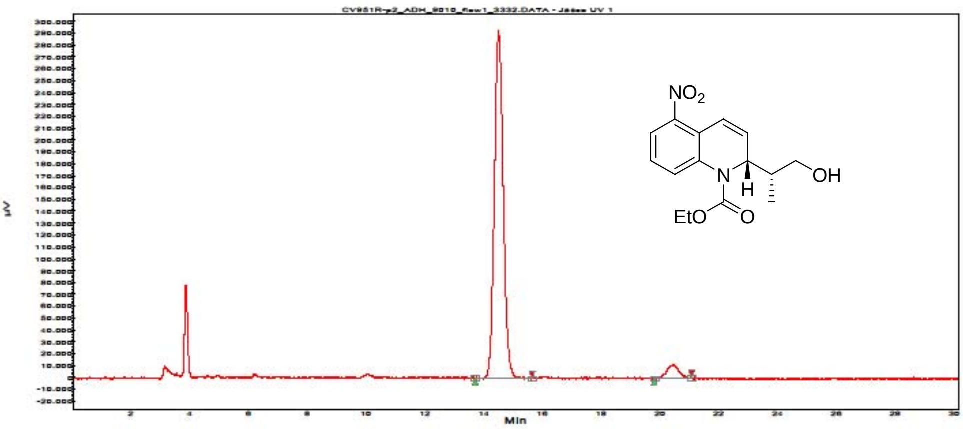
Data file: EF194P2_ADH_9010_flow1_5.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 27.01.2012 18:02:31



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,916	14,558	15,612	49,986
2	19,558	20,400	21,531	50,014
Total				100,000

Chromatogram : CV951R-p2_ADH_9010_flow1_3332

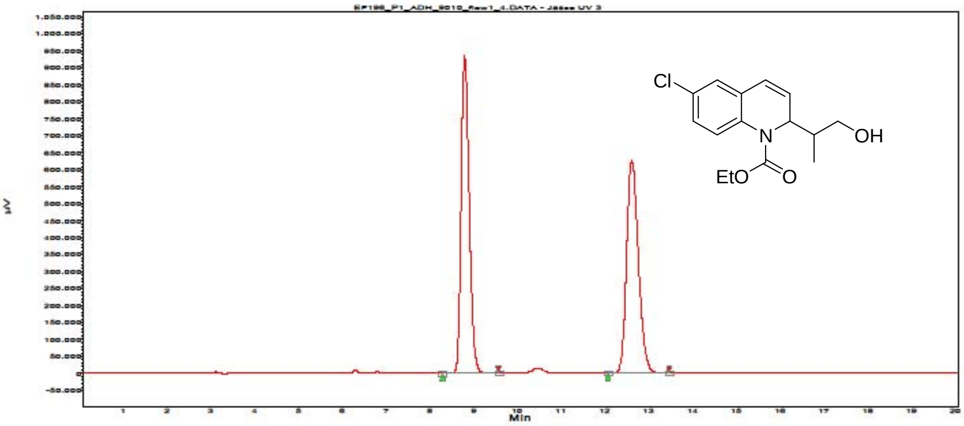
Data file: CV951R-p2_ADH_9010_flow1_3332.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 27.02.2012 17:48:17



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,719	14,508	15,646	95,114
2	19,804	20,442	21,070	4,886
Total				100,000

Chromatogram : EF196_P1_ADH_9010_flow1_4

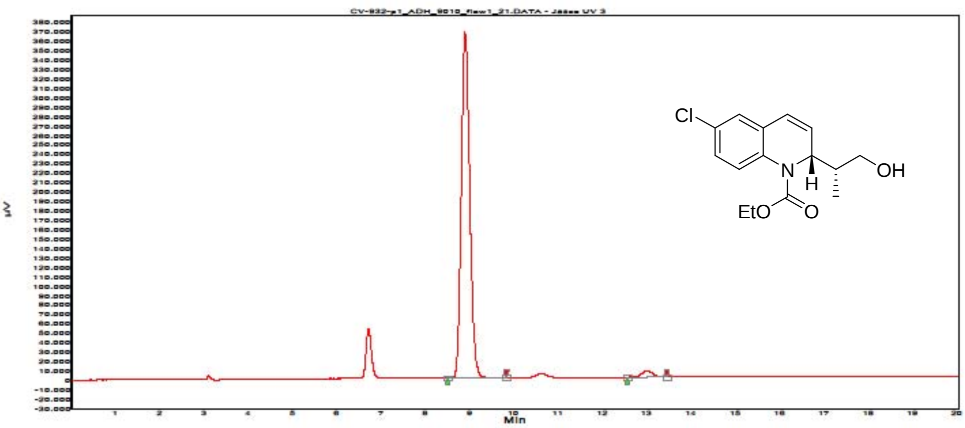
Data file: EF196_P1_ADH_9010_flow1_4.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 26.01.2012 19:33:38



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8,295	8,800	9,574	0,000	50,49	936978,3	198949,7	50,486
2	UNKNOWN	12,074	12,617	13,466	0,000	49,51	626505,4	195122,9	49,514
Total						100,00	1563483,7	394072,6	100,000

Chromatogram : CV-932-p1_ADH_9010_flow1_21

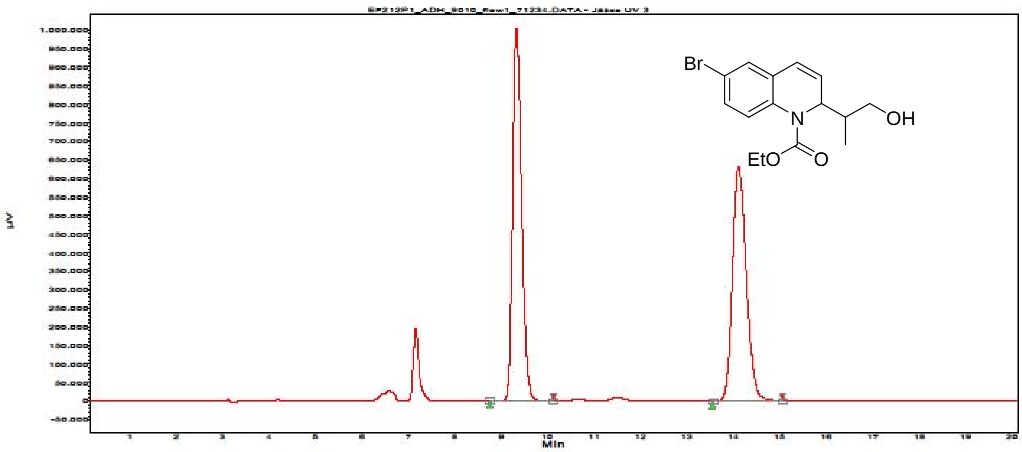
Data file: CV-932-p1_ADH_9010_flow1_21.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 06.02.2012 17:52:33



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	8,509	8,908	9,833	97,501
1	12,562	13,017	13,459	2,499
Total				100,000

Chromatogram : EF212P1_ADH_9010_flow1_71234

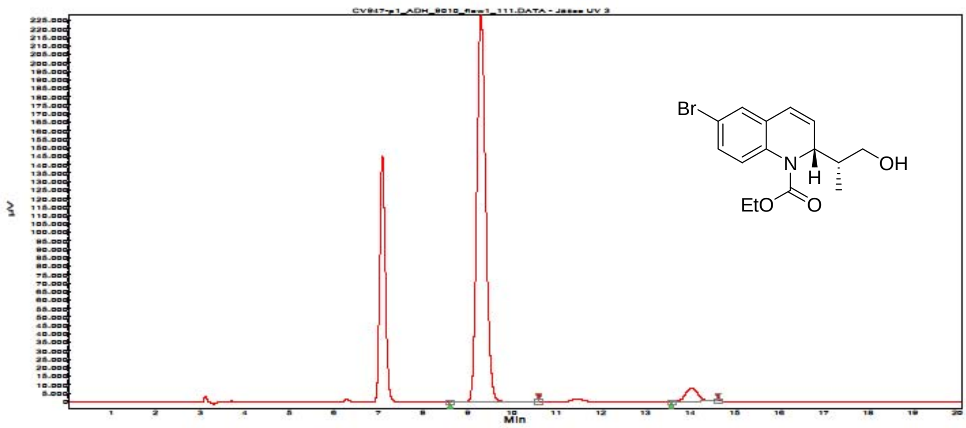
Data file: EF212P1_ADH_9010_flow1_71234.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 12.02.2012 17:17:49



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μV]	[μV.Min]	[%]
1	UNKNOWN	8,763	9,333	10,124	0,000	50,82	1003759,2	223612,4	50,818
2	UNKNOWN	13,547	14,108	15,052	0,000	49,18	627804,8	216412,7	49,182
Total						100,00	1631564,0	440025,1	100,000

Chromatogram : CV947-p1_ADH_9010_flow1_111

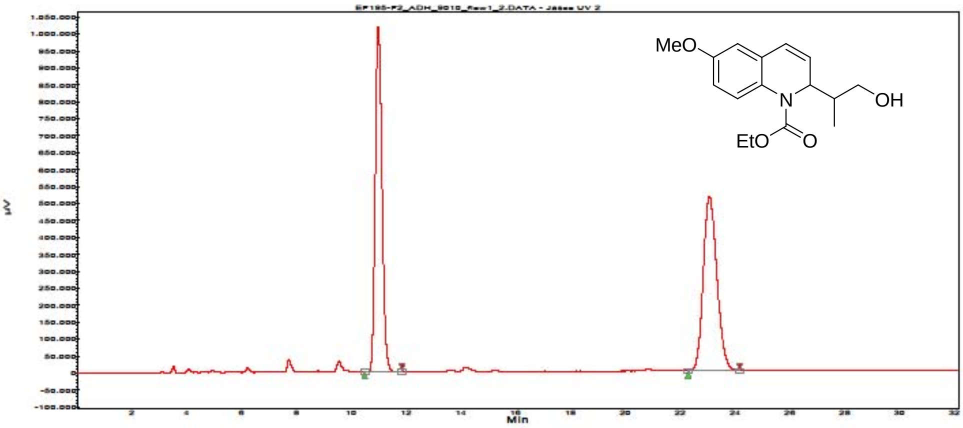
Data file: CV947-p1_ADH_9010_flow1_111.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 10.02.2012 21:07:00



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μV]	[μV.Min]	[%]
1	UNKNOWN	8,616	9,308	10,599	0,000	95,39	228609,1	52391,3	95,386
2	UNKNOWN	13,574	14,025	14,628	0,000	4,61	8009,8	2534,2	4,614
Total						100,00	236618,8	54925,5	100,000

Chromatogram : EF195-F2_ADH_9010_flow1_2

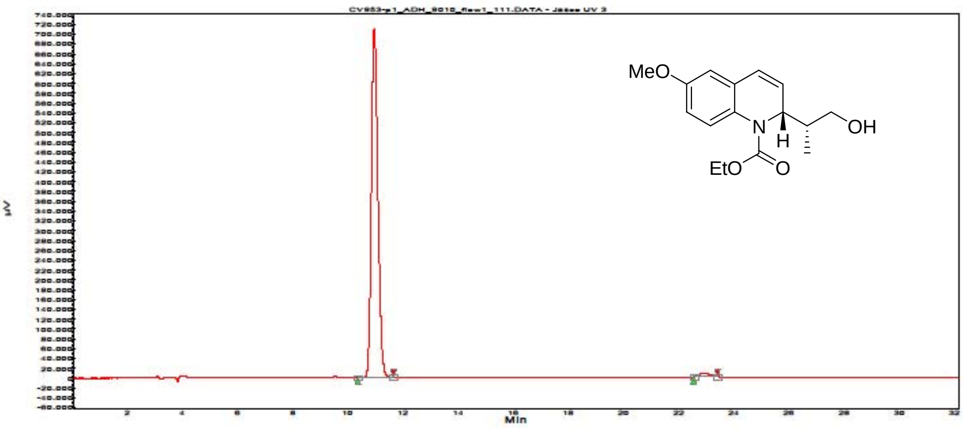
Data file: EF195-F2_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 28.02.2012 15:40:33



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,505	10,992	11,855	49,441
2	22,285	23,050	24,161	50,559
Total				100,000

Chromatogram : CV953-p1_ADH_9010_flow1_111

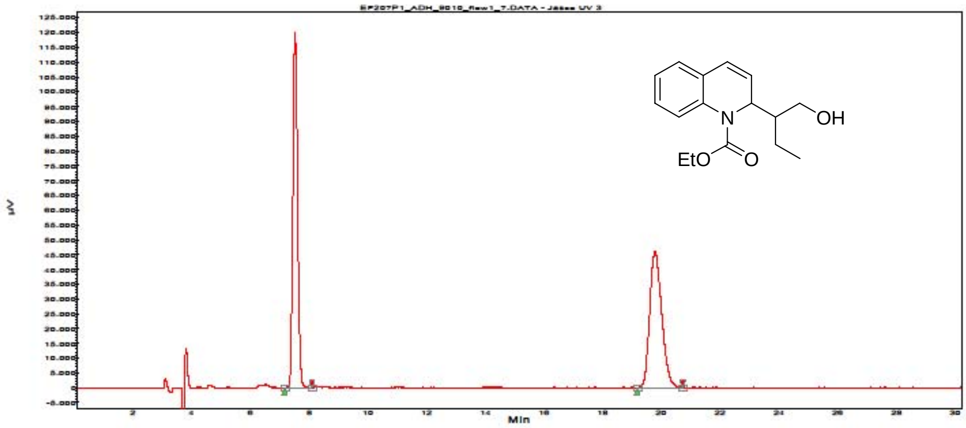
Data file: CV953-p1_ADH_9010_flow1_111.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 28.02.2012 16:19:20



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10,376	10,967	11,669	0,000	98,34	709439,0	193273,1	98,337
2	UNKNOWN	22,538	22,950	23,400	0,000	1,66	7315,6	3268,0	1,663
Total						100,00	716754,6	196541,1	100,000

Chromatogram : EF207P1_ADH_9010_flow1_7

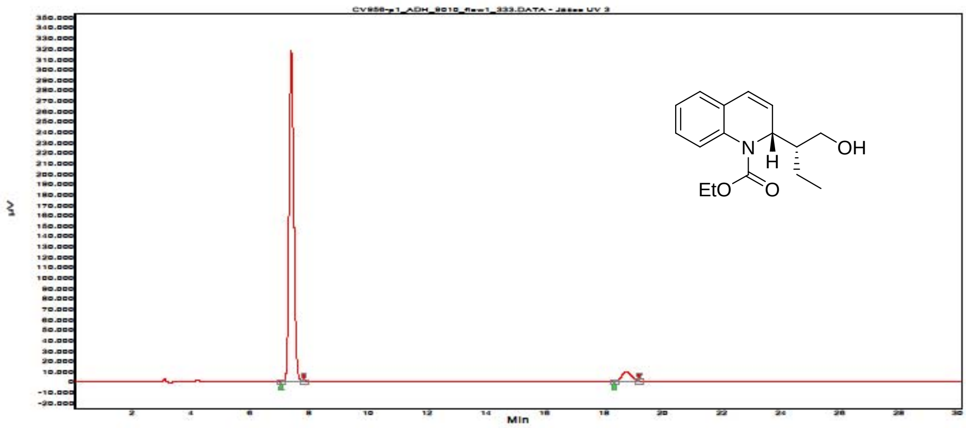
Data file: EF207P1_ADH_9010_flow1_7.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 11.02.2012 01:17:58



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7,174	7,533	8,105	0,000	50,38	119978,4	22148,4	50,377
2	UNKNOWN	19,161	19,775	20,714	0,000	49,62	46037,1	21817,3	49,623
Total						100,00	166015,5	43965,7	100,000

Chromatogram : CV956-p1_ADH_9010_flow1_333

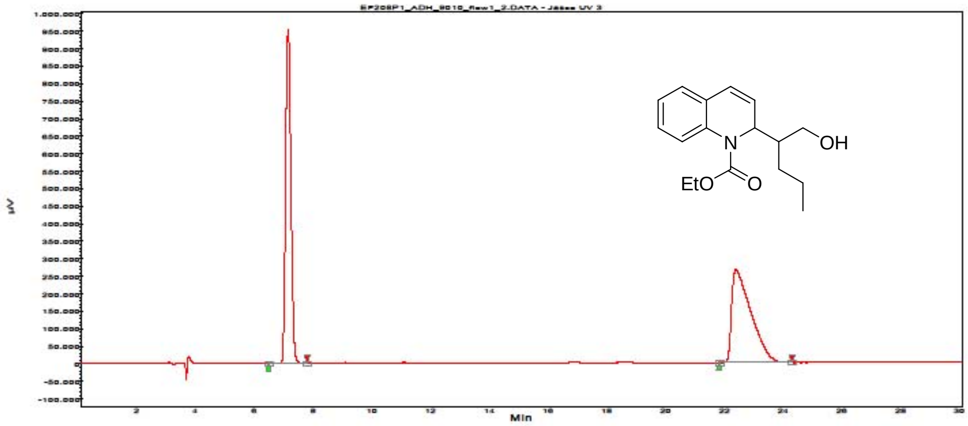
Data file: CV956-p1_ADH_9010_flow1_333.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 28.02.2012 18:03:50



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,059	7,408	7,835	94,334
2	18,350	18,758	19,187	5,666
Total				100,000

Chromatogram : EF208P1_ADH_9010_flow1_2

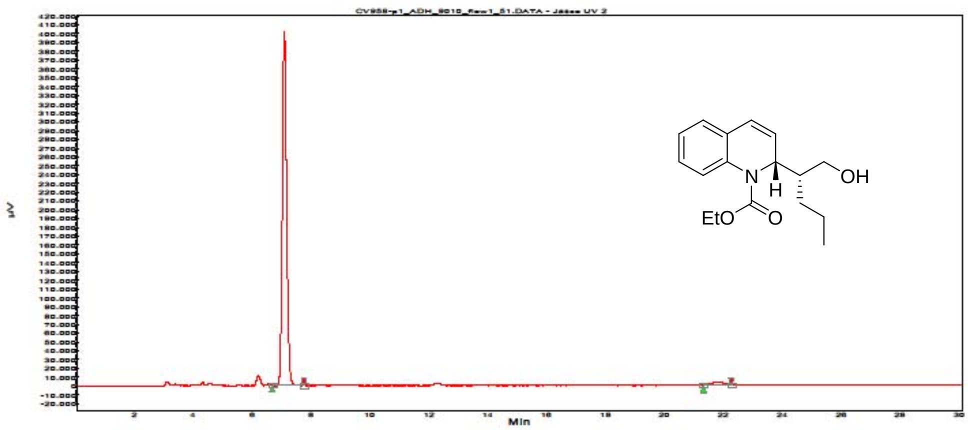
Data file: EF208P1_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 10.02.2012 19:08:56



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6,508	7,167	7,810	0,000	49,48	954779,4	196833,4	49,482
2	UNKNOWN	21,818	22,392	24,298	0,000	50,52	265380,7	200954,4	50,518
Total						100,00	1220160,1	397787,8	100,000

Chromatogram : CV958-p1_ADH_9010_flow1_51

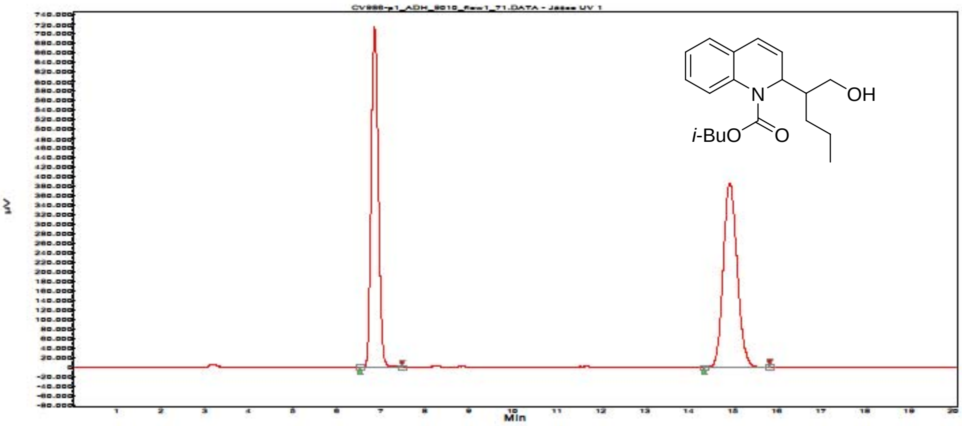
Data file: CV958-p1_ADH_9010_flow1_51.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 27.02.2012 12:18:17



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,694	7,108	7,769	97,488
2	21,322	21,858	22,273	2,512
Total				100,000

Chromatogram : CV986-p1_ADH_9010_flow1_71

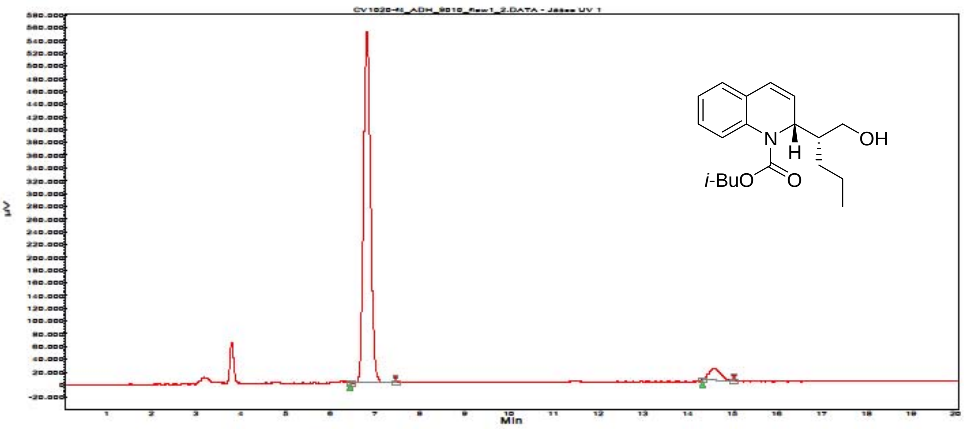
Data file: CV986-p1_ADH_9010_flow1_71.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 20.05.2012 20:45:09



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,535	6,867	7,488	48,846
2	14,345	14,925	15,836	51,154
Total				100,000

Chromatogram : CV1020-f4_ADH_9010_flow1_2

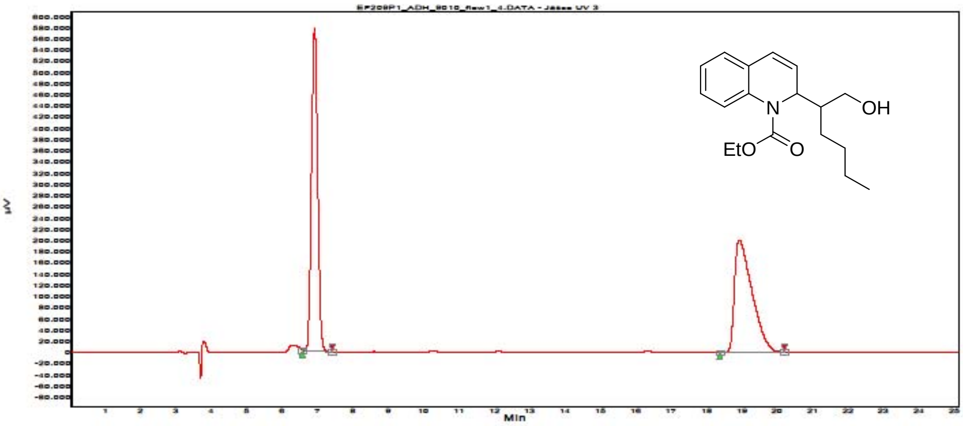
Data file: CV1020-f4_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 01.06.2012 15:56:28



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,454	6,825	7,464	94,690
2	14,330	14,600	15,031	5,310
Total				100,000

Chromatogram : EF209P1_ADH_9010_flow1_4

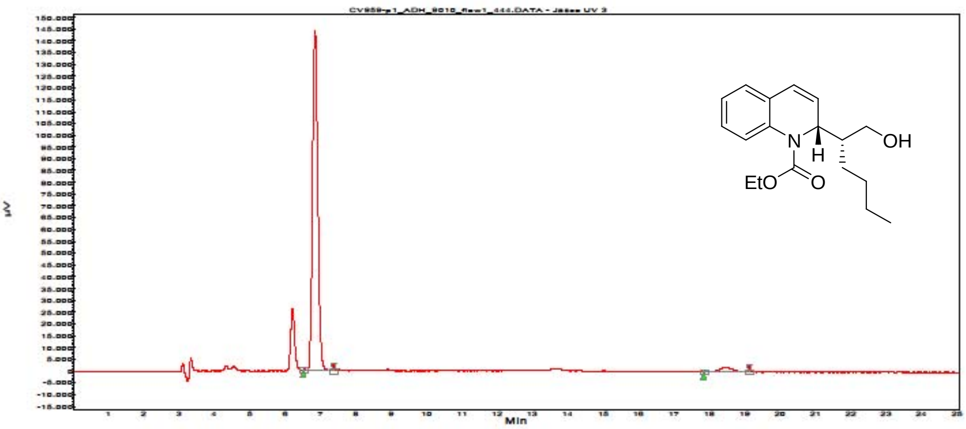
Data file: EF209P1_ADH_9010_flow1_4.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 10.02.2012 23:12:29



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6,580	6,933	7,420	0,000	50,47	574520,1	116734,2	50,474
2	UNKNOWN	18,377	18,925	20,193	0,000	49,53	200756,5	114539,6	49,526
Total						100,00	775276,6	231273,8	100,000

Chromatogram : CV959-p1_ADH_9010_flow1_444

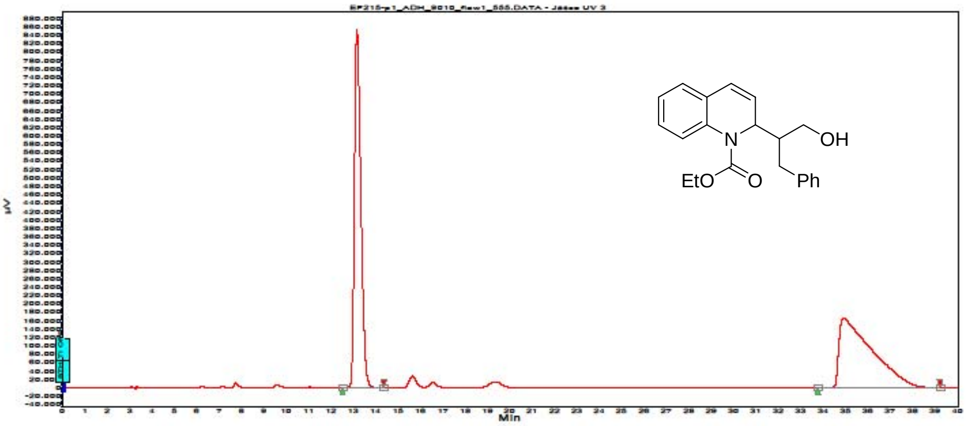
Data file: CV959-p1_ADH_9010_flow1_444.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 27.02.2012 18:51:01



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,524	6,858	7,376	96,748
2	17,836	18,467	19,127	3,252
Total				100,000

Chromatogram : EF215-p1_ADH_9010_flow1_555

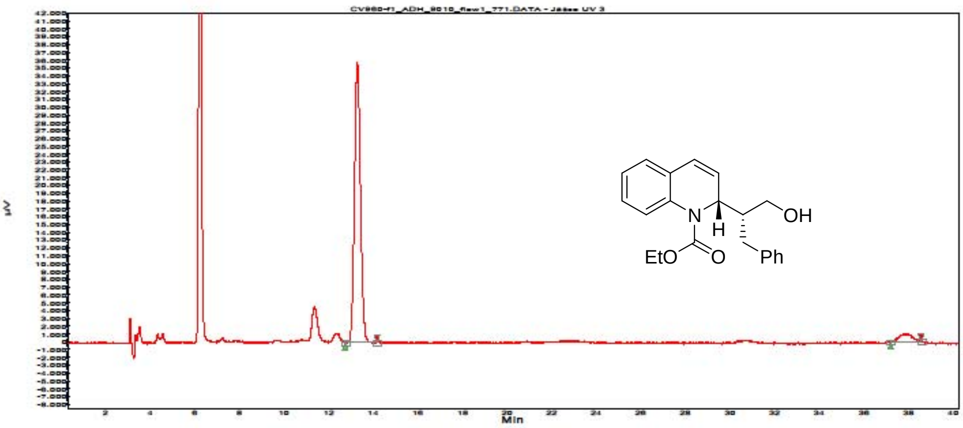
Data file: EF215-p1_ADH_9010_flow1_555.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 28.02.2012 19:07:23



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	12,521	13,167	14,380	49,851
2	33,760	34,892	39,215	50,149
Total				100,000

Chromatogram : CV960-f1_ADH_9010_flow1_771

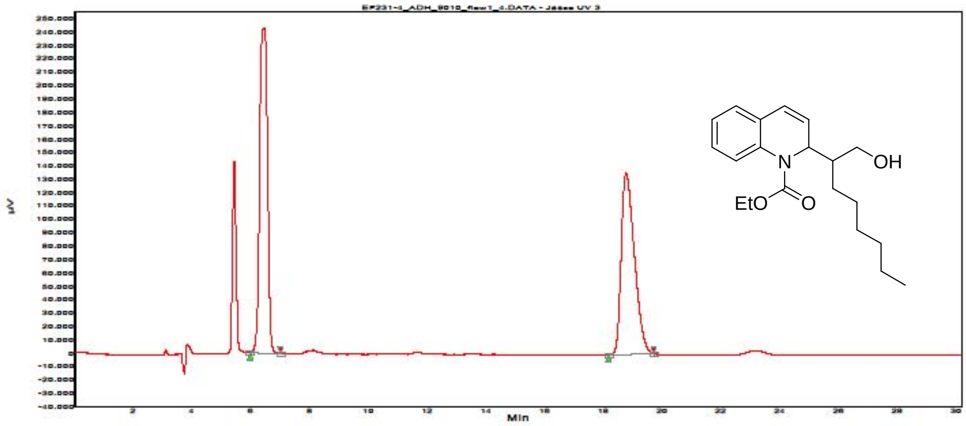
Data file: CV960-f1_ADH_9010_flow1_771.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 28.02.2012 00:19:34



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12,756	13,292	14,166	0,000	94,28	35850,6	11182,5	94,281
2	UNKNOWN	37,183	37,792	38,559	0,000	5,72	973,5	678,3	5,719
Total						100,00	36824,1	11860,8	100,000

Chromatogram : EF231-4_ADH_9010_flow1_4

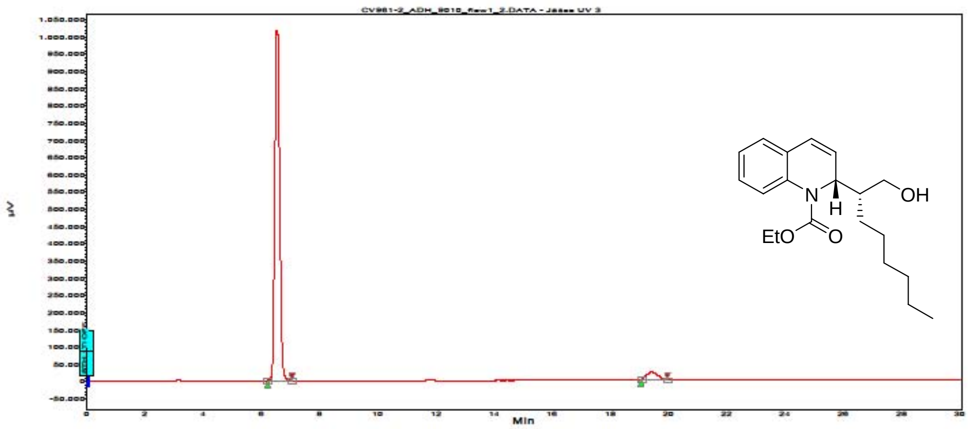
Data file: EF231-4_ADH_9010_flow1_4.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 06.03.2012 23:53:54



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	5,992	6,483	7,025	51,871
2	18,182	18,775	19,711	48,129
Total				100,000

Chromatogram : CV961-2_ADH_9010_flow1_2

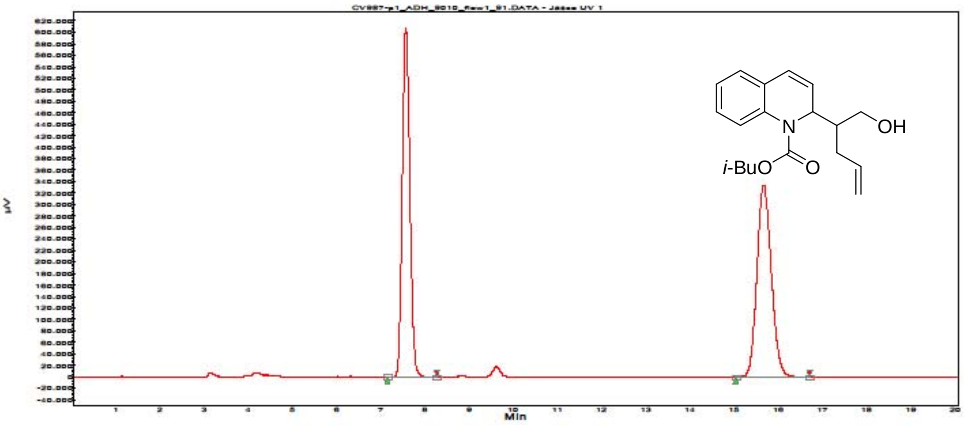
Data file: CV961-2_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 14.03.2012 10:56:36



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,240	6,575	7,066	95,594
2	19,050	19,417	19,959	4,406
Total				100,000

Chromatogram : CV987-p1_ADH_9010_flow1_91

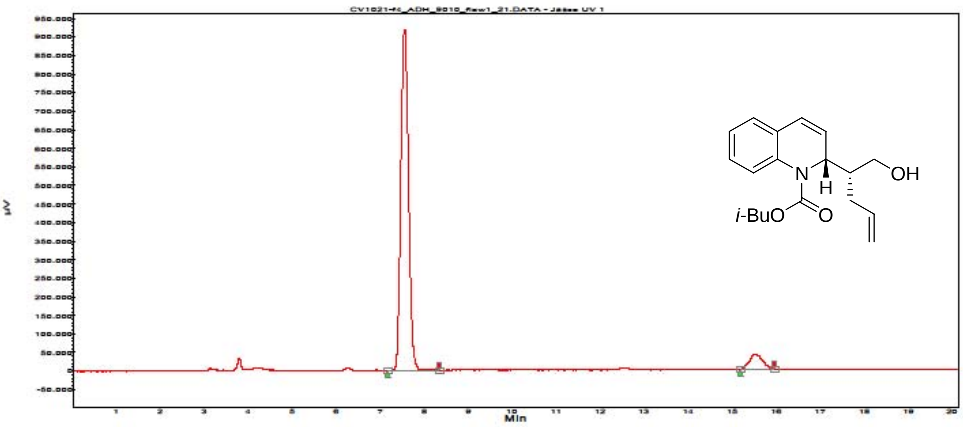
Data file: CV987-p1_ADH_9010_flow1_91.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 20.05.2012 21:50:35



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,159	7,575	8,275	48,961
2	15,031	15,667	16,705	51,039
Total				100,000

Chromatogram : CV1021-f4_ADH_9010_flow1_21

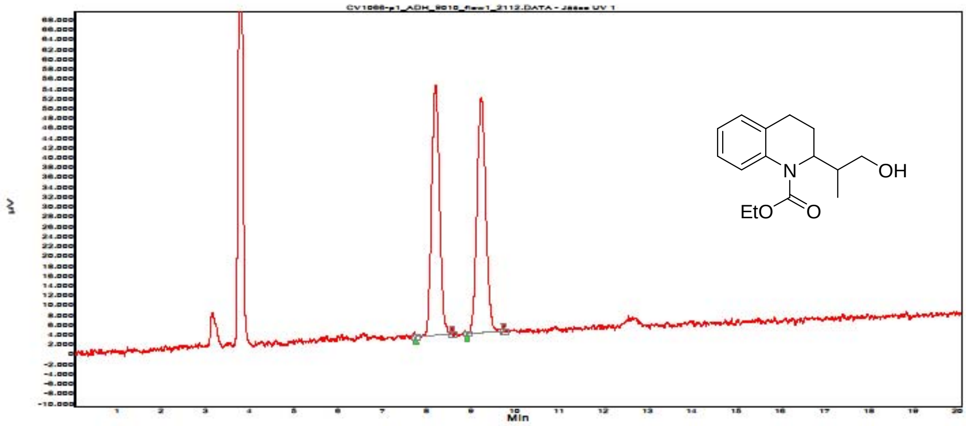
Data file: CV1021-f4_ADH_9010_flow1_21.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 01.06.2012 22:40:00



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,178	7,558	8,340	93,092
2	15,186	15,550	15,954	6,908
Total				100,000

Chromatogram : CV1066-p1_ADH_9010_flow1_2112

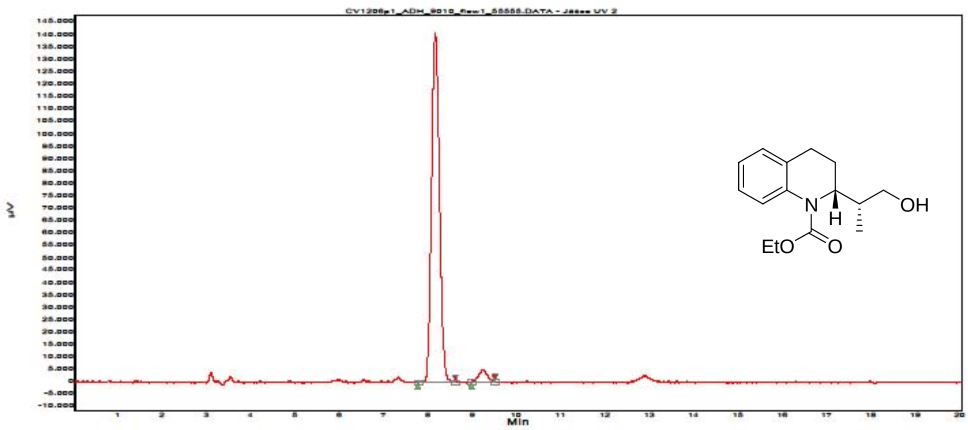
Data file: CV1066-p1_ADH_9010_flow1_2112.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 29.06.2012 11:35:40



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,764	8,200	8,578	49,080
2	8,922	9,242	9,747	50,920
Total				100,000

Chromatogram : CV1206p1_ADH_9010_flow1_55555

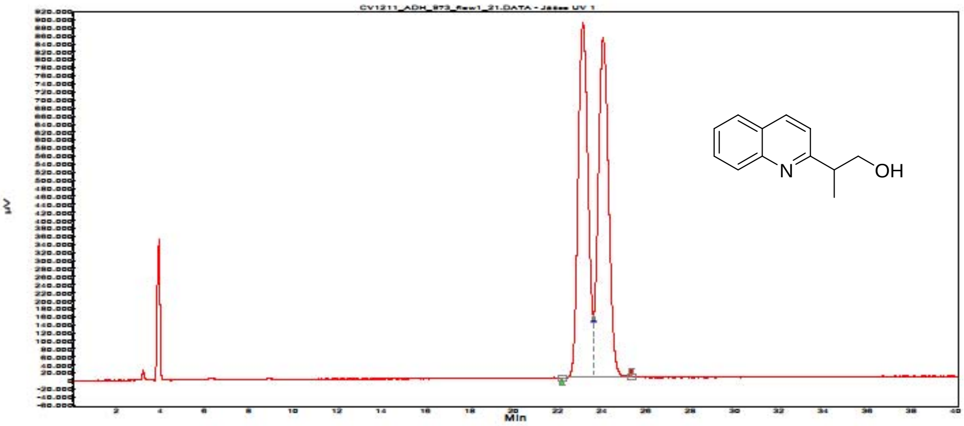
Data file: CV1206p1_ADH_9010_flow1_55555.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 05.09.2012 16:49:17



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,781	8,175	8,627	96,584
2	8,989	9,242	9,521	3,416
Total				100,000

Chromatogram : CV1211_ADH_973_flow1_21

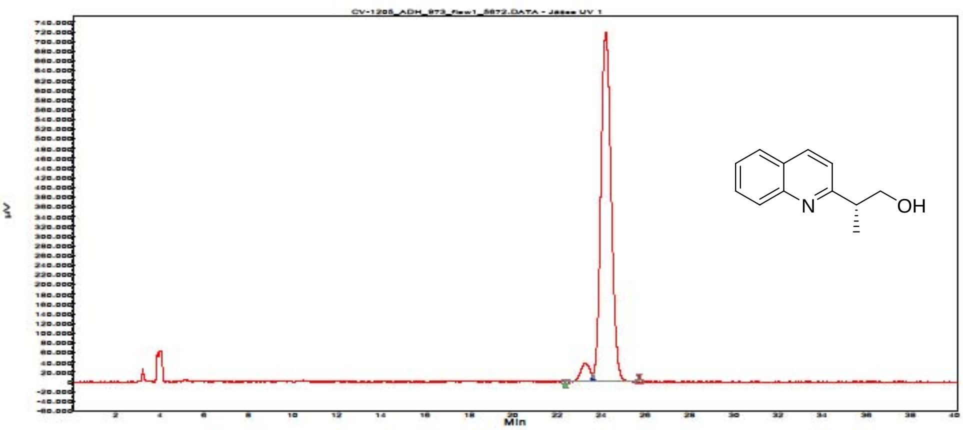
Data file: CV1211_ADH_973_flow1_21.DATA
Method: HPLC1_ADH_973_flow1_acq_50
Date: 12.09.2012 11:11:31



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	22,180	23,125	23,611	49,878
2	23,611	24,050	25,312	50,122
Total				100,000

Chromatogram : CV-1205_ADH_973_flow1_5672

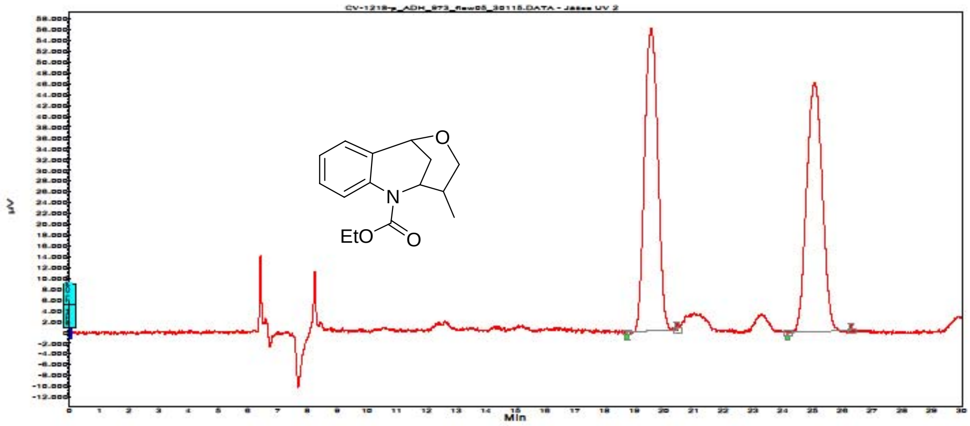
Data file: CV-1205_ADH_973_flow1_5672.DATA
Method: HPLC1_ADH_973_flow1_acq_50
Date: 06.09.2012 22:03:09



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	22,358	23,267	23,602	4,238
2	23,602	24,200	25,697	95,762
Total				100,000

Chromatogram : CV-1218-p_ADH_973_flow05_30115

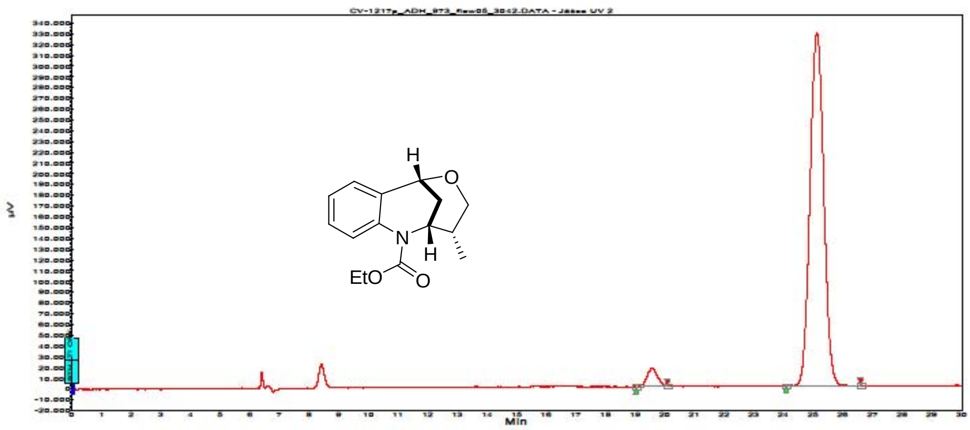
Data file: CV-1218-p_ADH_973_flow05_30115.DATA
Method: HPLC1_ADH_973_flow05_acq_30
Date: 17.09.2012 19:26:09



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,750	19,567	20,424	50,568
2	24,143	25,058	26,281	49,432
Total				100,000

Chromatogram : CV-1217p_ADH_973_flow05_3042

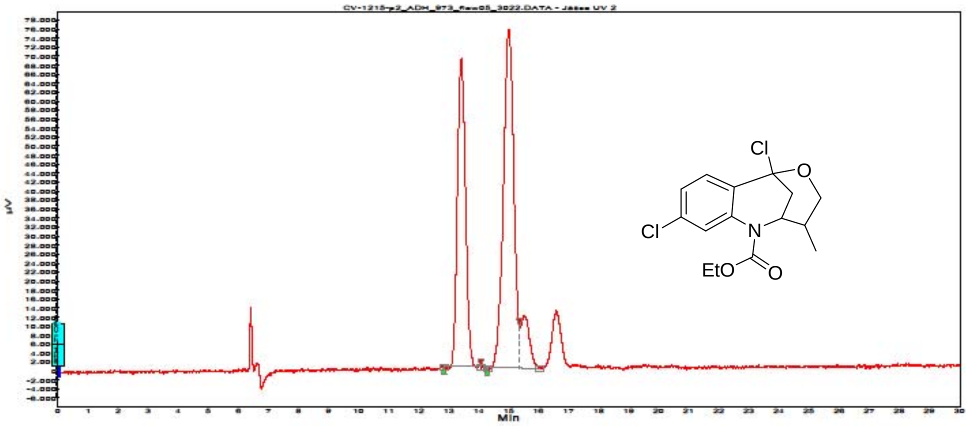
Data file: CV-1217p_ADH_973_flow05_3042.DATA
Method: HPLC1_ADH_973_flow05_acq_30
Date: 17.09.2012 15:47:36



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,029	19,558	20,083	3,661
2	24,081	25,125	26,591	96,339
Total				100,000

Chromatogram : CV-1215-p2_ADH_973_flow05_3022

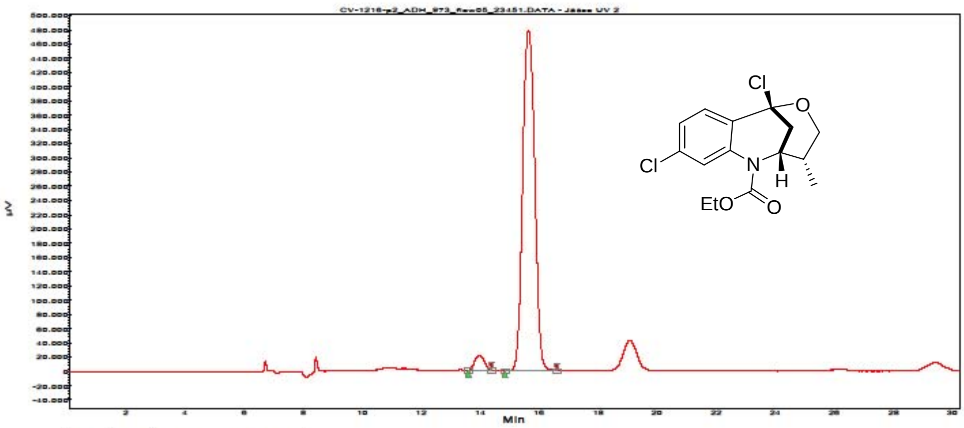
Data file: CV-1215-p2_ADH_973_flow05_3022.DATA
Method: HPLC1_ADH_973_flow05_acq_30
Date: 17.09.2012 16:20:19



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	12,838	13,425	14,066	41,241
2	14,293	15,008	15,366	58,759
Total				100,000

Chromatogram : CV-1216-p2_ADH_973_flow05_23451

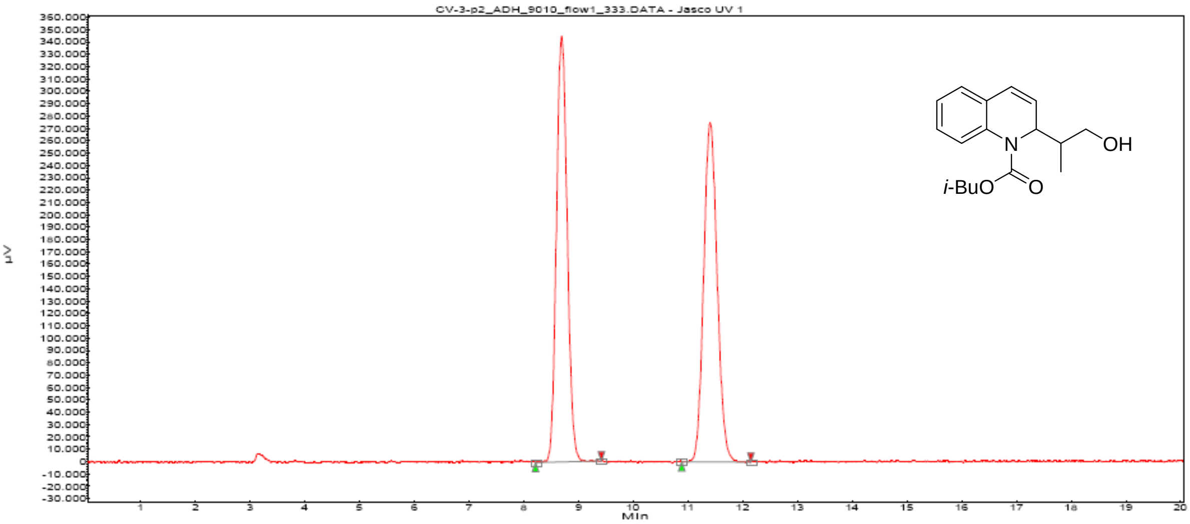
Data file: CV-1216-p2_ADH_973_flow05_23451.DATA
Method: HPLC1_ADH_973_flow05_acq_90
Date: 14.09.2012 12:06:02



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,599	13,975	14,377	3,339
2	14,844	15,633	16,586	96,661
Total				100,000

Chromatogram : CV-3-p2_ADH_9010_flow1_333

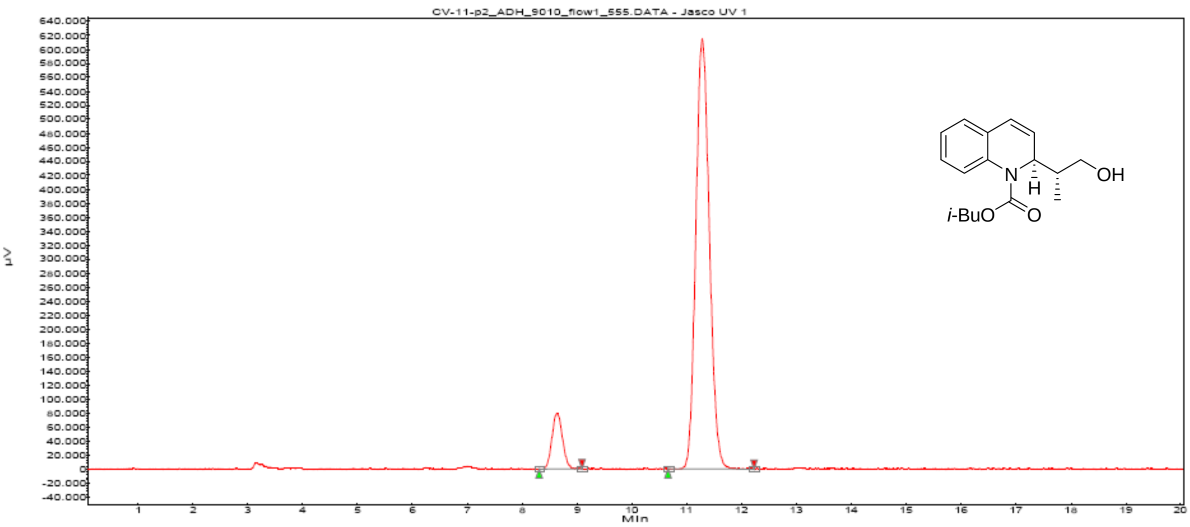
Data file: CV-3-p2_ADH_9010_flow1_333.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 24.04.2012 13:31:46



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,223	8,700	9,421	50,022
2	10,888	11,408	12,149	49,978
Total				100,000

Chromatogram : CV-11-p2_ADH_9010_flow1_555

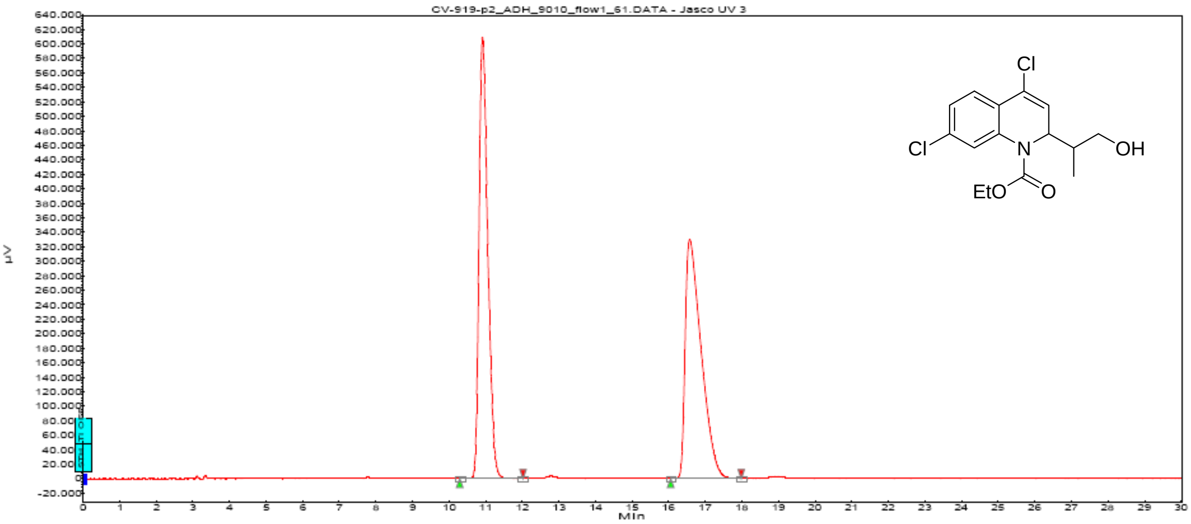
Data file: CV-11-p2_ADH_9010_flow1_555.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 24.04.2012 14:39:43



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,309	8,633	9,093	9,078
2	10,660	11,283	12,227	90,922
Total				100,000

Chromatogram : CV-919-p2_ADH_9010_flow1_61

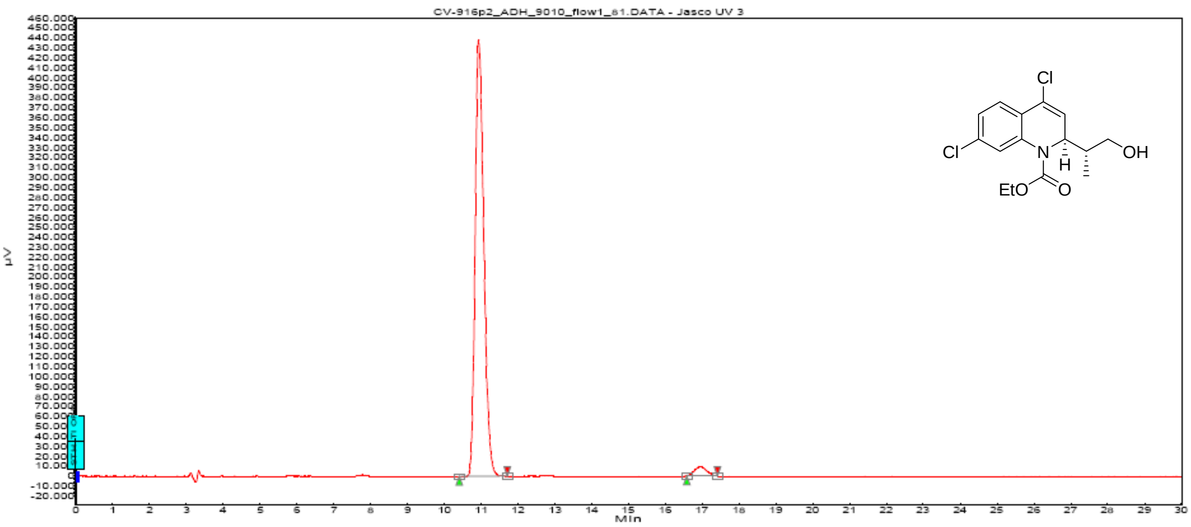
Data file: CV-919-p2_ADH_9010_flow1_61.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 06.01.2012 21:32:01



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,289	10,908	12,025	50,641
2	16,054	16,567	17,975	49,359
Total				100,000

Chromatogram : CV-916p2_ADH_9010_flow1_81

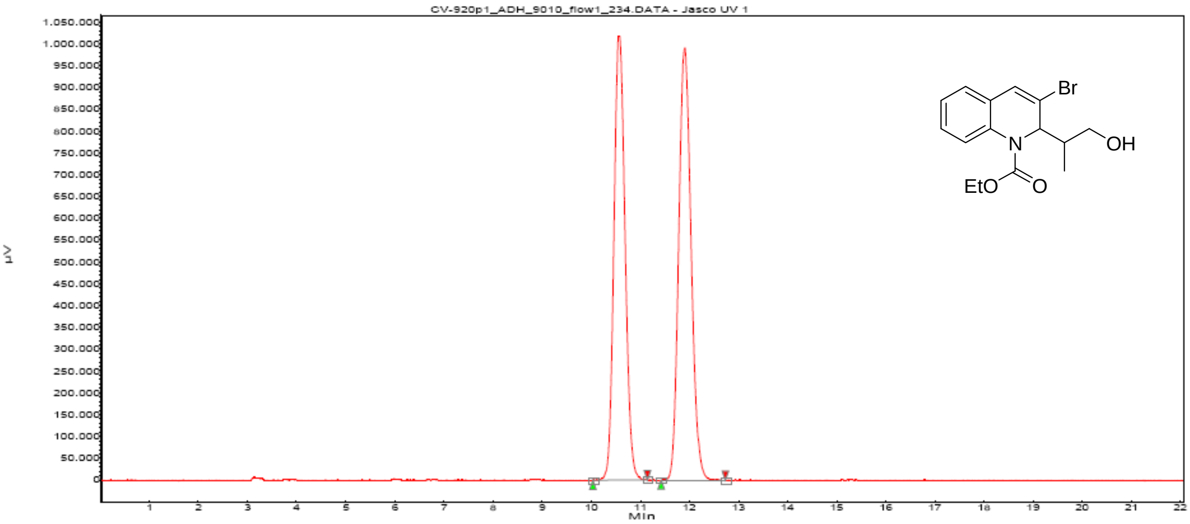
Data file: CV-916p2_ADH_9010_flow1_81.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 06.01.2012 22:37:27



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,413	10,933	11,715	97,091
2	16,581	16,958	17,417	2,909
Total				100,000

Chromatogram : CV-920p1_ADH_9010_flow1_234

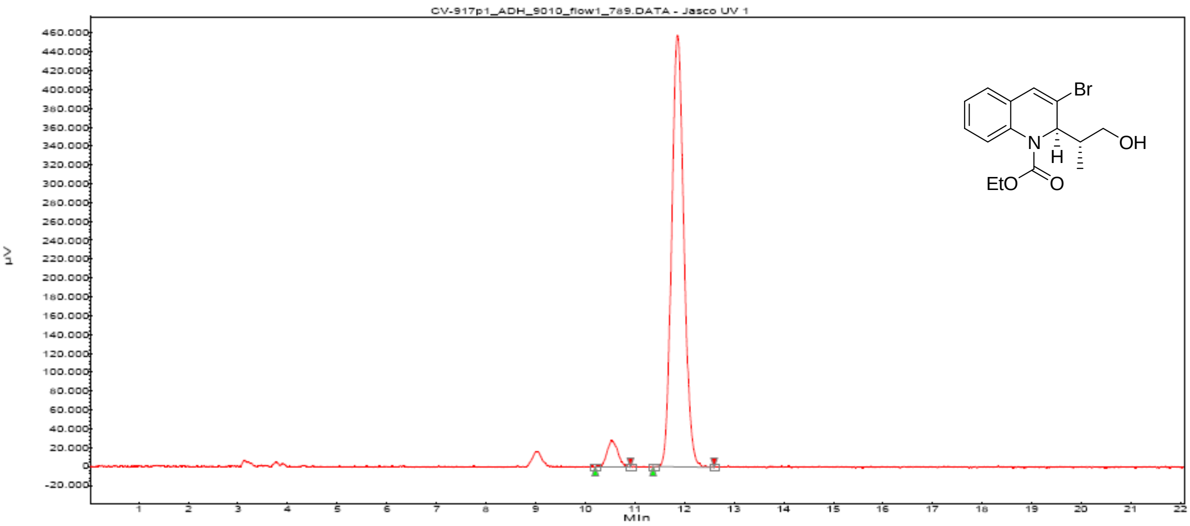
Data file: CV-920p1_ADH_9010_flow1_234.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 11.01.2012 20:31:15



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,031	10,550	11,142	48,102
2	11,426	11,908	12,727	51,898
Total				100,000

Chromatogram : CV-917p1_ADH_9010_flow1_789

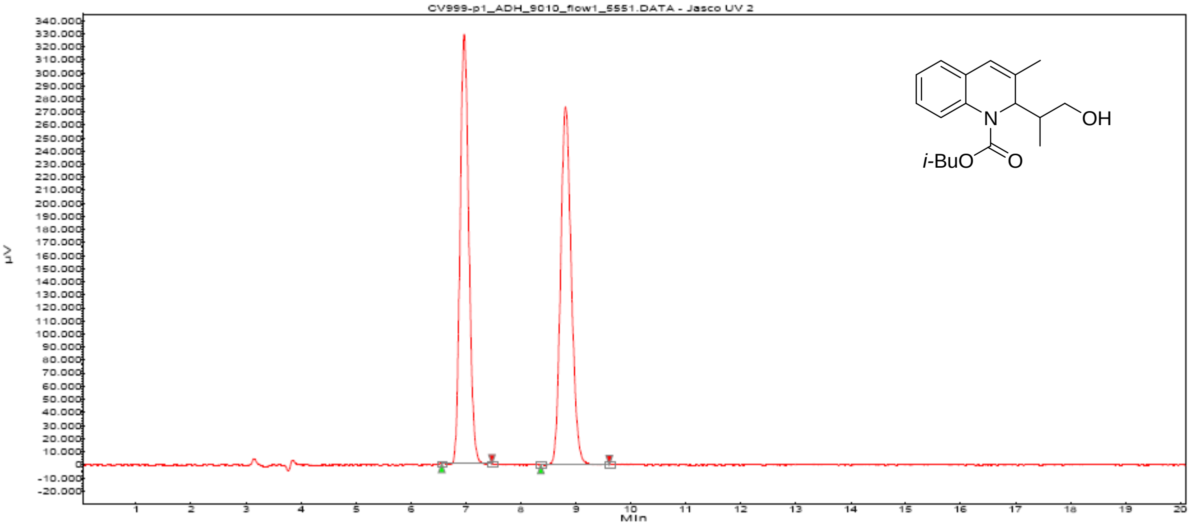
Data file: CV-917p1_ADH_9010_flow1_789.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 11.01.2012 21:56:13



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,206	10,542	10,912	5,077
2	11,367	11,867	12,596	94,923
Total				100,000

Chromatogram : CV999-p1_ADH_9010_flow1_5551

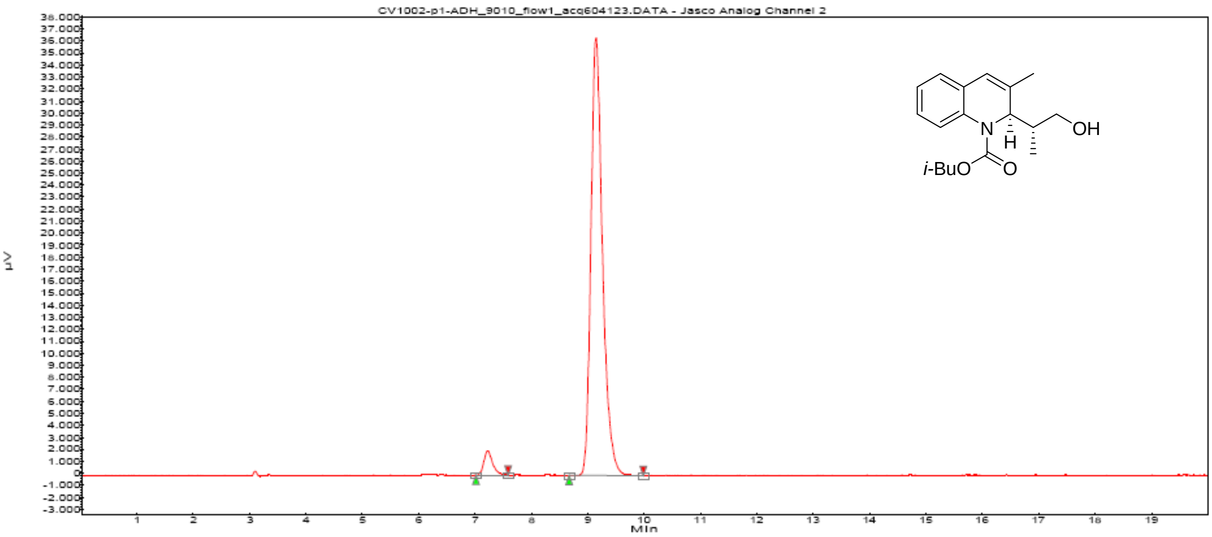
Data file: CV999-p1_ADH_9010_flow1_5551.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 09.05.2012 20:51:33



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	6,563	6,975	7,474	49,388
2	8,364	8,817	9,606	50,612
Total				100,000

Chromatogram : CV1002-p1-ADH_9010_flow1_acq604123

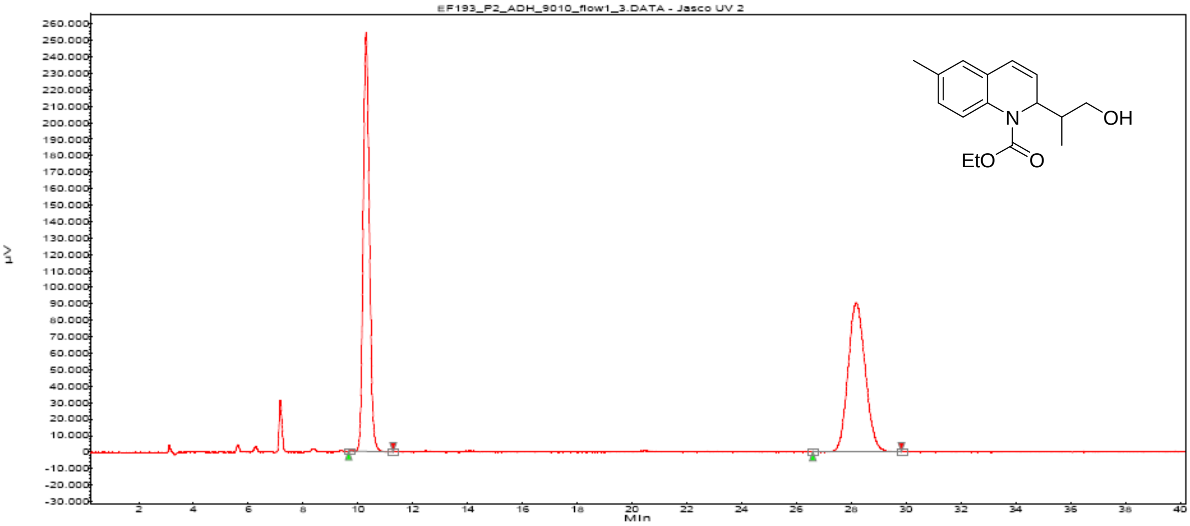
Data file: CV1002-p1-ADH_9010_flow1_acq604123.DATA
Method: HPLC2_ADH_9010_flow1_acq60
Date: 11.05.2012 12:07:52



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7,015	7,225	7,589	0,000	4,01	2011,7	362,6	4,007
2	UNKNOWN	8,667	9,150	9,983	0,000	95,99	36425,1	8687,8	95,993
Total						100,00	38436,8	9050,4	100,000

Chromatogram : EF193_P2_ADH_9010_flow1_3

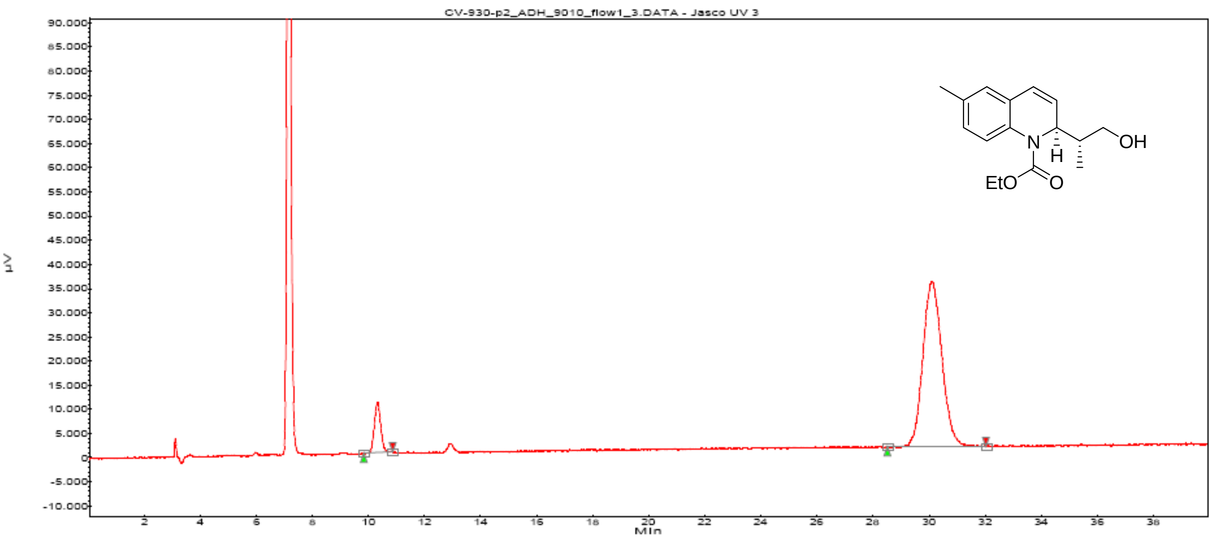
Data file: EF193_P2_ADH_9010_flow1_3.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 26.01.2012 18:30:55



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,669	10,300	11,281	50,199
2	26,591	28,167	29,814	49,801
Total				100,000

Chromatogram : CV-930-p2_ADH_9010_flow1_3

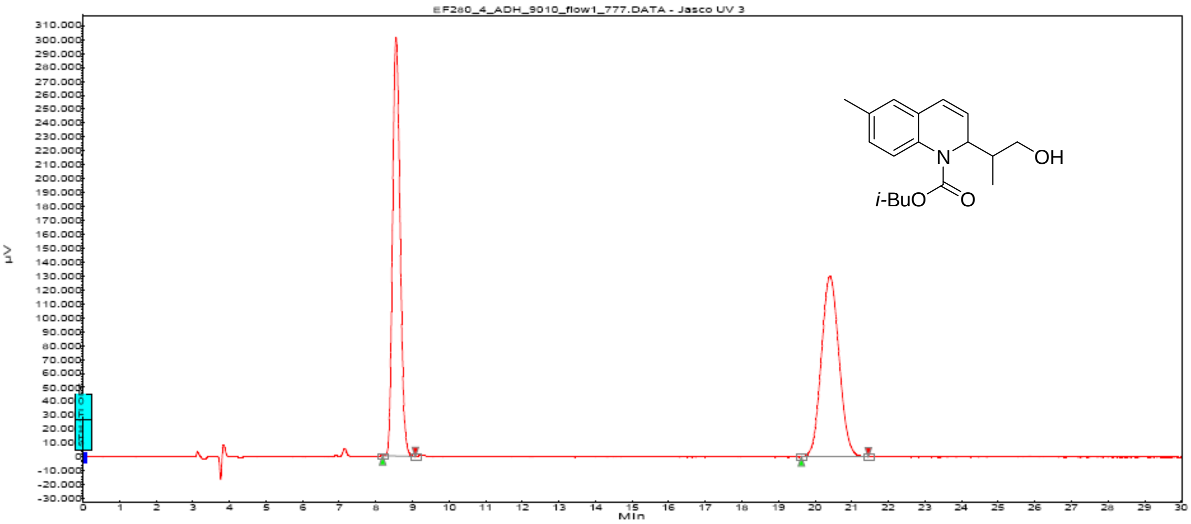
Data file: CV-930-p2_ADH_9010_flow1_3.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 03.02.2012 20:48:19



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,835	10,325	10,868	9,172
2	28,512	30,100	32,025	90,828
Total				100,000

Chromatogram : EF280_4_ADH_9010_flow1_777

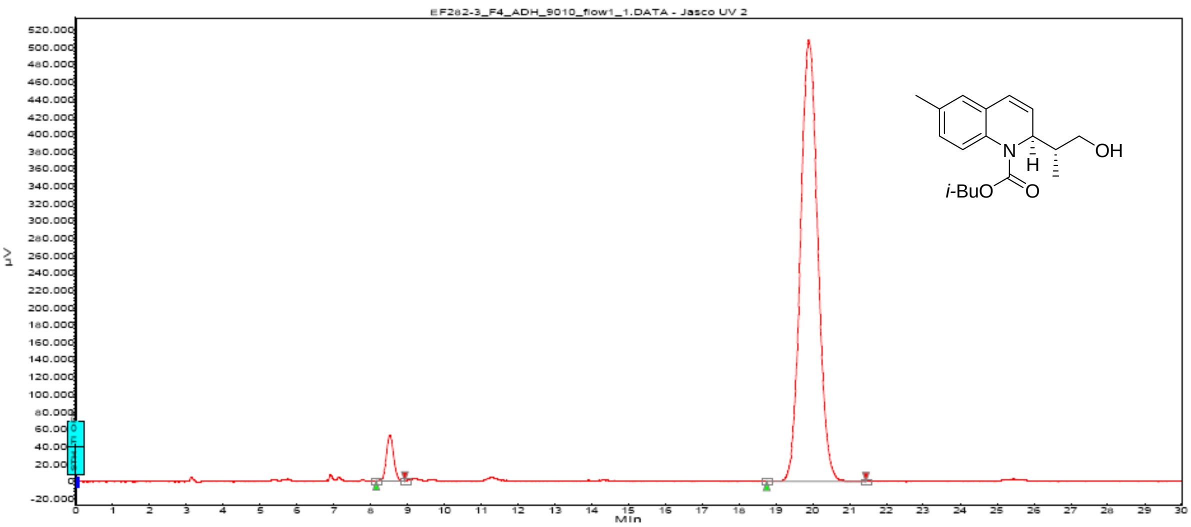
Data file: EF280_4_ADH_9010_flow1_777.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 16.05.2012 22:33:02



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,182	8,550	9,081	49,692
2	19,618	20,408	21,446	50,308
Total				100,000

Chromatogram : EF282-3_F4_ADH_9010_flow1_1

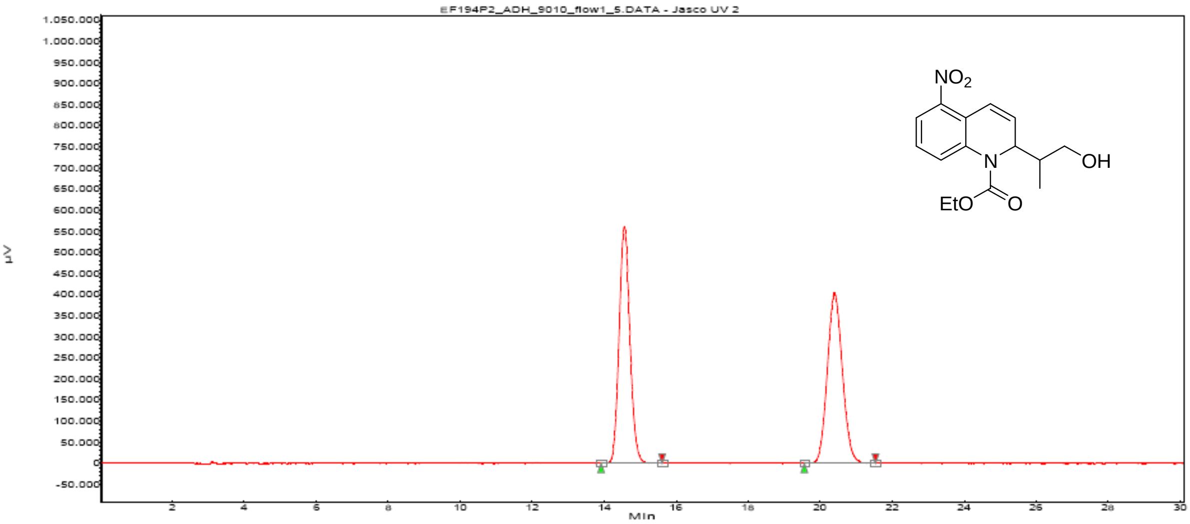
Data file: EF282-3_F4_ADH_9010_flow1_1.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 21.05.2012 00:01:32



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,151	8,525	8,926	4,236
2	18,750	19,892	21,446	95,764
Total				100,000

Chromatogram : EF194P2_ADH_9010_flow1_5

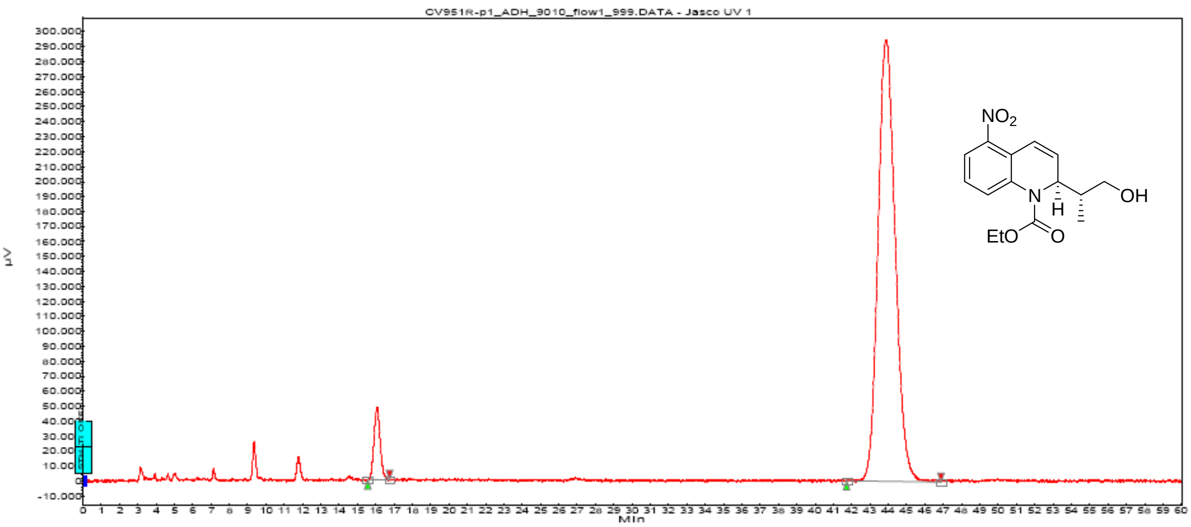
Data file: EF194P2_ADH_9010_flow1_5.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 27.01.2012 18:02:31



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,916	14,558	15,612	49,986
2	19,558	20,400	21,531	50,014
Total				100,000

Chromatogram : CV951R-p1_ADH_9010_flow1_999

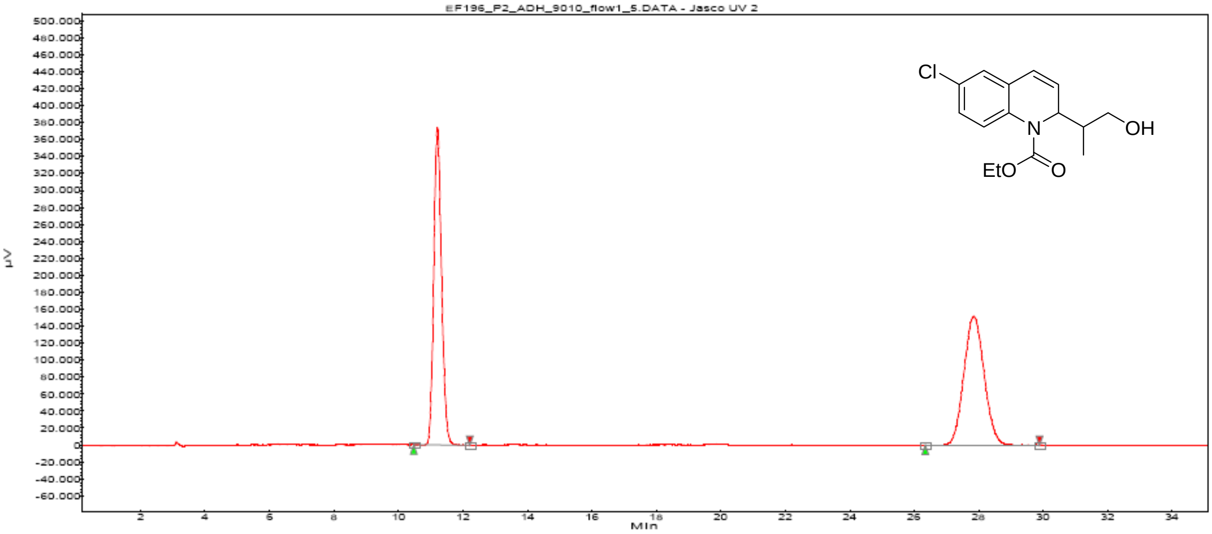
Data file: CV951R-p1_ADH_9010_flow1_999.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 27.02.2012 21:11:22



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,558	16,067	16,736	5,535
2	41,715	43,875	46,860	94,465
Total				100,000

Chromatogram : EF196_P2_ADH_9010_flow1_5

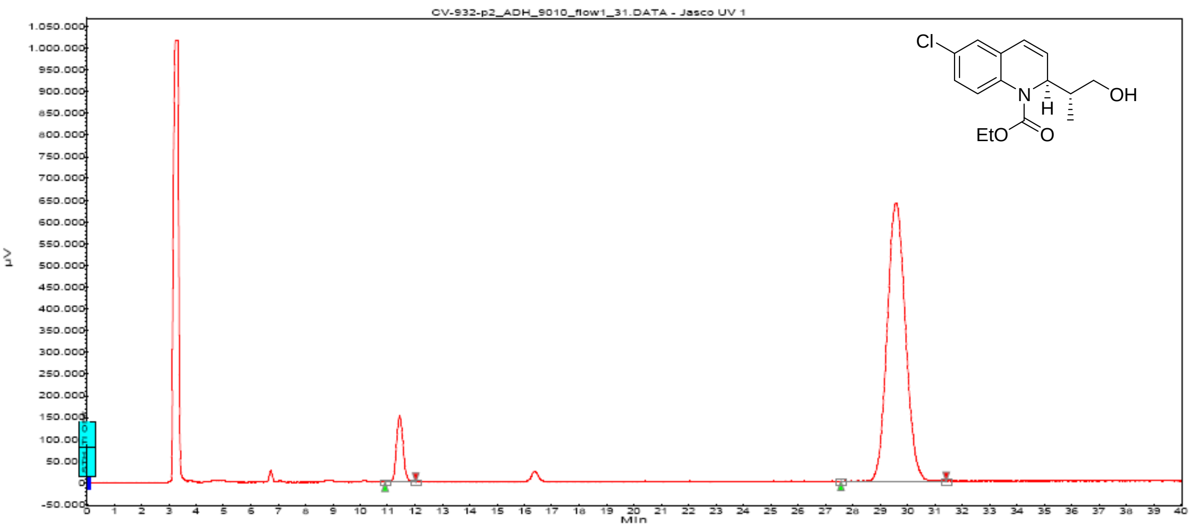
Data file: EF196_P2_ADH_9010_flow1_5.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 26.01.2012 20:03:37



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,475	11,208	12,211	48,615
2	26,343	27,842	29,876	51,385
Total				100,000

Chromatogram : CV-932-p2_ADH_9010_flow1_31

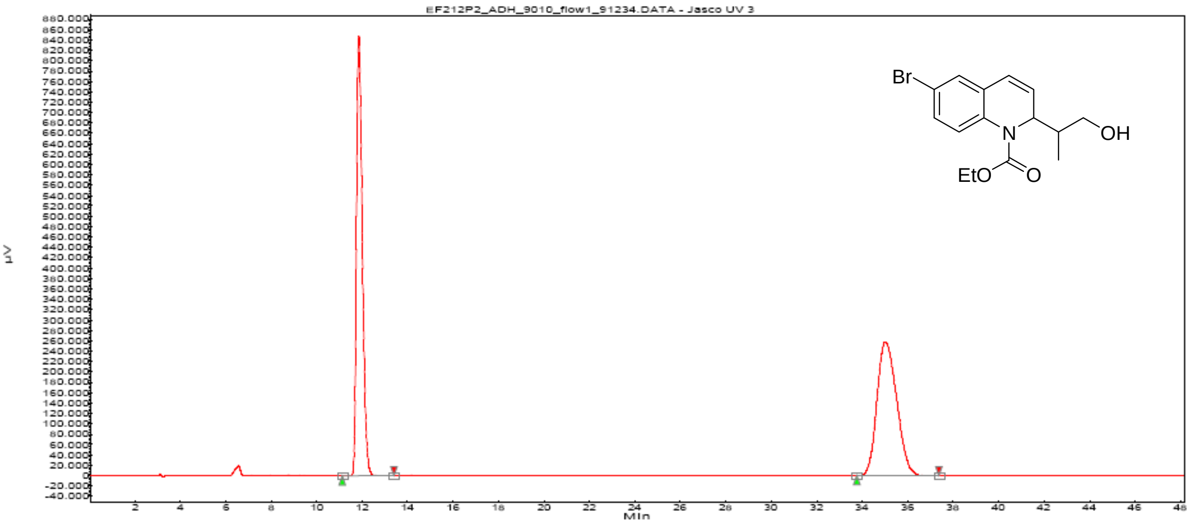
Data file: CV-932-p2_ADH_9010_flow1_31.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 06.02.2012 18:17:09



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,909	11,442	12,025	7,893
2	27,562	29,558	31,405	92,107
Total				100,000

Chromatogram : EF212P2_ADH_9010_flow1_91234

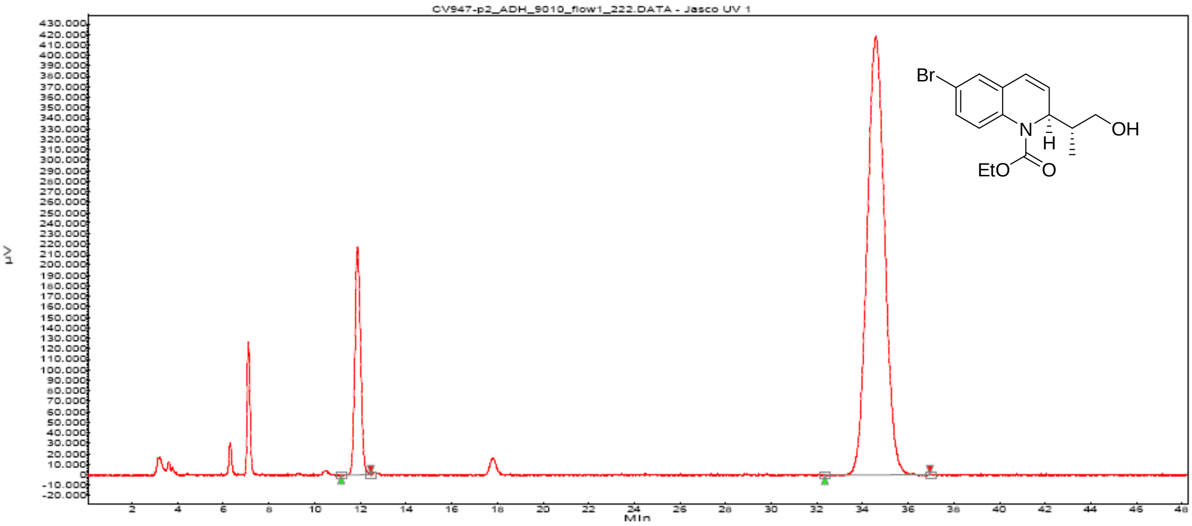
Data file: EF212P2_ADH_9010_flow1_91234.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 12.02.2012 18:20:32



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11,135	11,858	13,403	0,000	49,42	848302,9	252650,9	49,421
2	UNKNOWN	33,765	35,017	37,373	0,000	50,58	257600,7	258573,8	50,579
Total						100,00	1105903,6	511224,8	100,000

Chromatogram : CV947-p2_ADH_9010_flow1_222

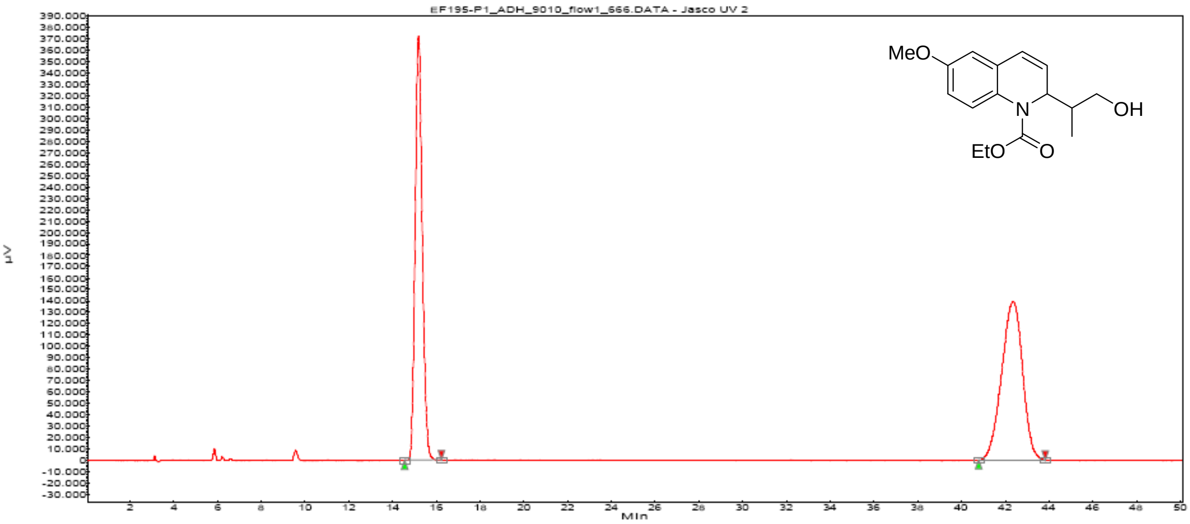
Data file: CV947-p2_ADH_9010_flow1_222.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 10.02.2012 22:09:43



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	11,157	11,883	12,459	14,599
2	32,355	34,592	36,942	85,401
Total				100,000

Chromatogram : EF195-P1_ADH_9010_flow1_666

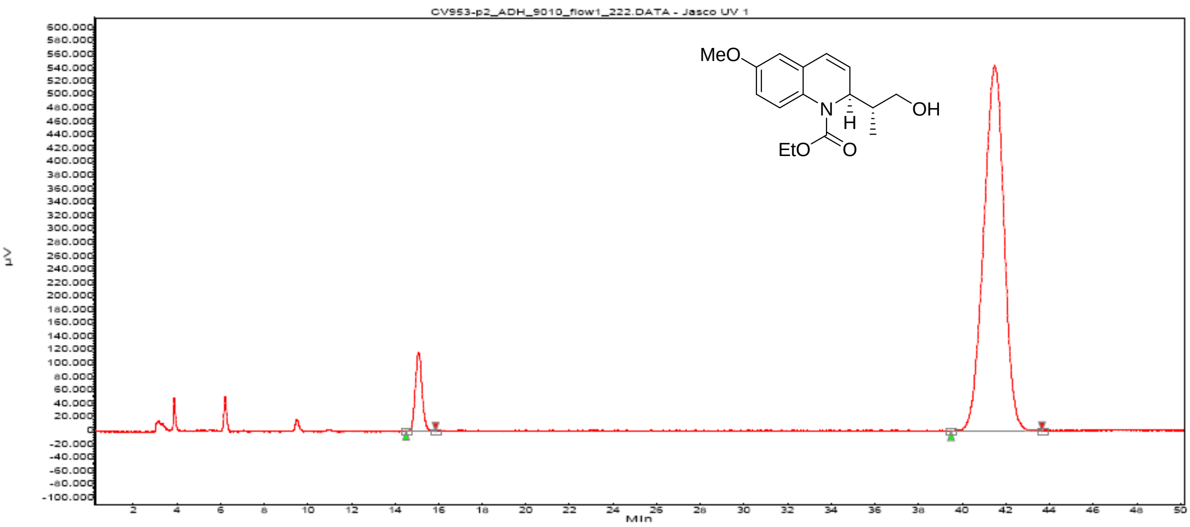
Data file: EF195-P1_ADH_9010_flow1_666.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 27.02.2012 22:14:06



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	14,566	15,192	16,240	47,612
2	40,785	42,367	43,822	52,388
Total				100,000

Chromatogram : CV953-p2_ADH_9010_flow1_222

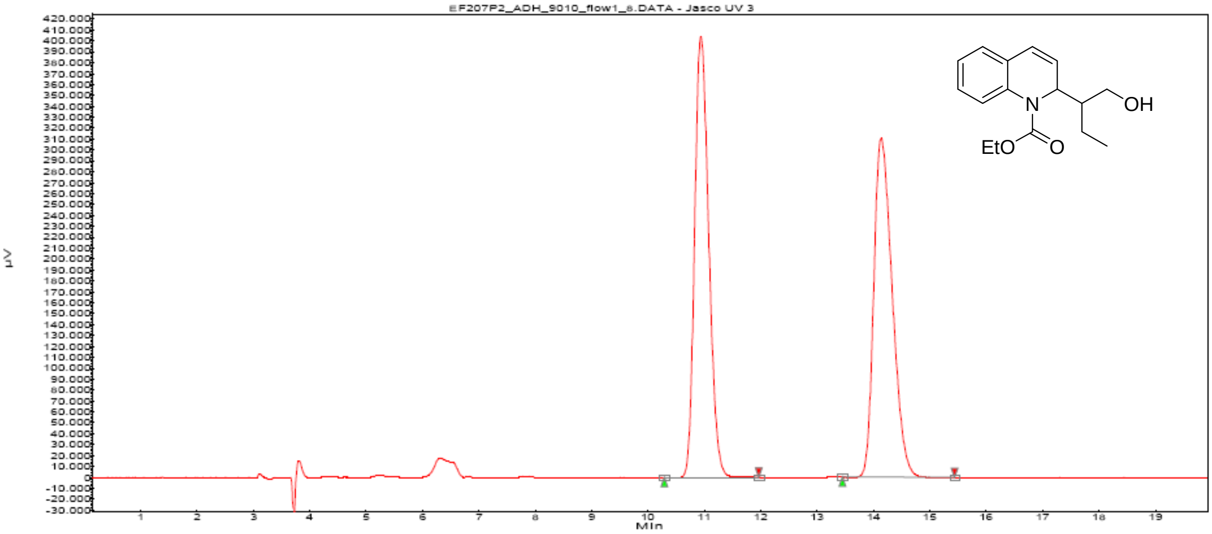
Data file: CV953-p2_ADH_9010_flow1_222.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 28.02.2012 17:01:07



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	14,504	15,083	15,868	6,618
1	39,483	41,483	43,636	93,382
Total				100,000

Chromatogram : EF207P2_ADH_9010_flow1_8

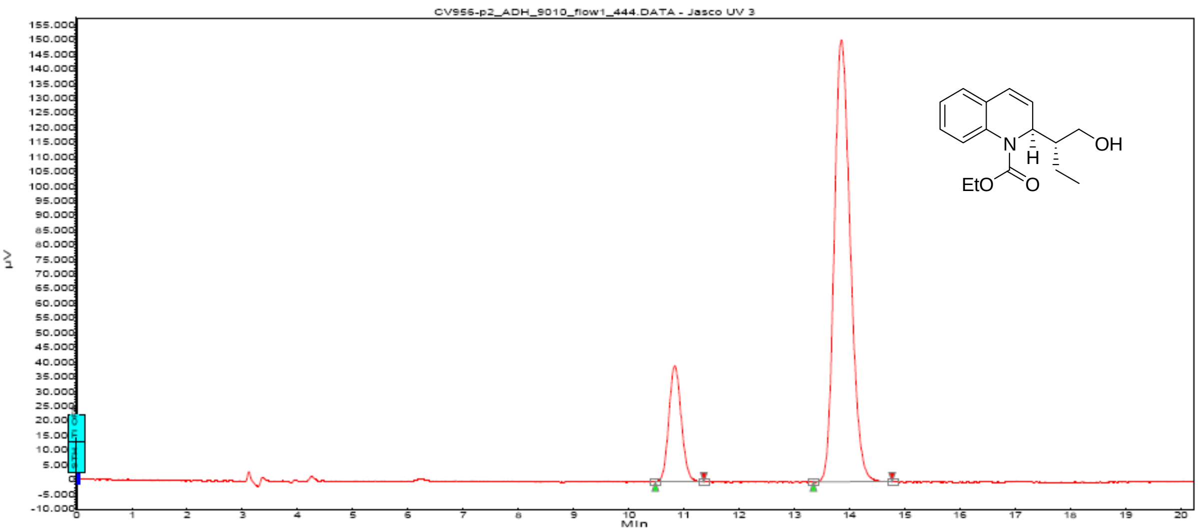
Data file: EF207P2_ADH_9010_flow1_8.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 11.02.2012 02:20:42



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10,289	10,933	11,963	0,000	49,86	404241,3	116907,1	49,857
2	UNKNOWN	13,450	14,133	15,434	0,000	50,14	310458,6	117579,9	50,143
Total						100,00	714699,9	234487,0	100,000

Chromatogram : CV956-p2_ADH_9010_flow1_444

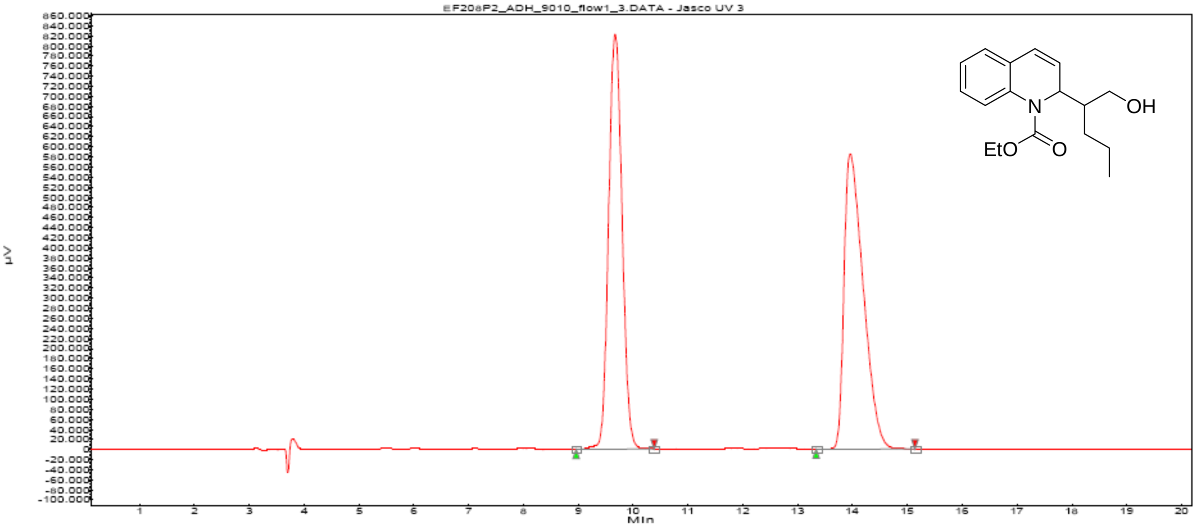
Data file: CV956-p2_ADH_9010_flow1_444.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 28.02.2012 18:44:38



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,484	10,833	11,361	16,873
2	13,346	13,850	14,766	83,127
Total				100,000

Chromatogram : EF208P2_ADH_9010_flow1_3

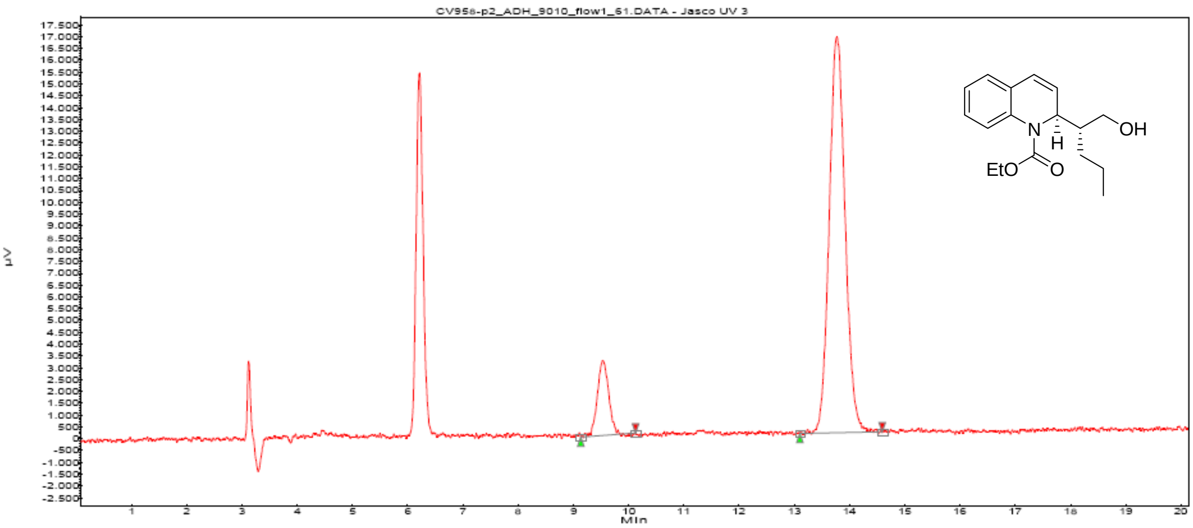
Data file: EF208P2_ADH_9010_flow1_3.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 10.02.2012 20:11:39



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8,962	9,675	10,383	0,000	49,71	822659,7	235067,1	49,711
2	UNKNOWN	13,334	13,958	15,138	0,000	50,29	585982,9	237797,5	50,289
Total						100,00	1408642,6	472864,6	100,000

Chromatogram : CV958-p2_ADH_9010_flow1_61

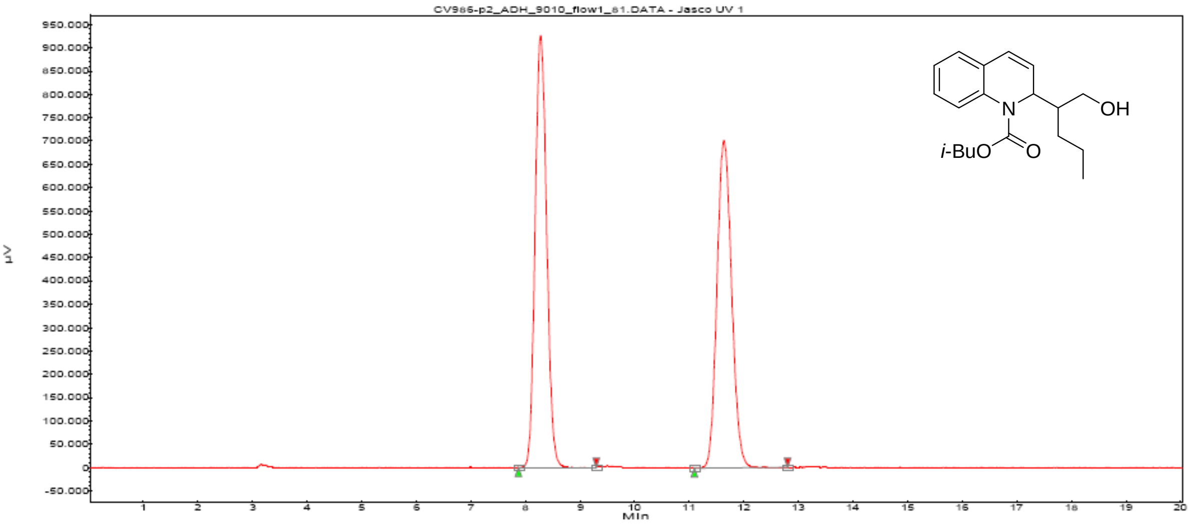
Data file: CV958-p2_ADH_9010_flow1_61.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 27.02.2012 13:01:00



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,132	9,533	10,124	11,735
2	13,099	13,767	14,587	88,265
Total				100,000

Chromatogram : CV986-p2_ADH_9010_flow1_81

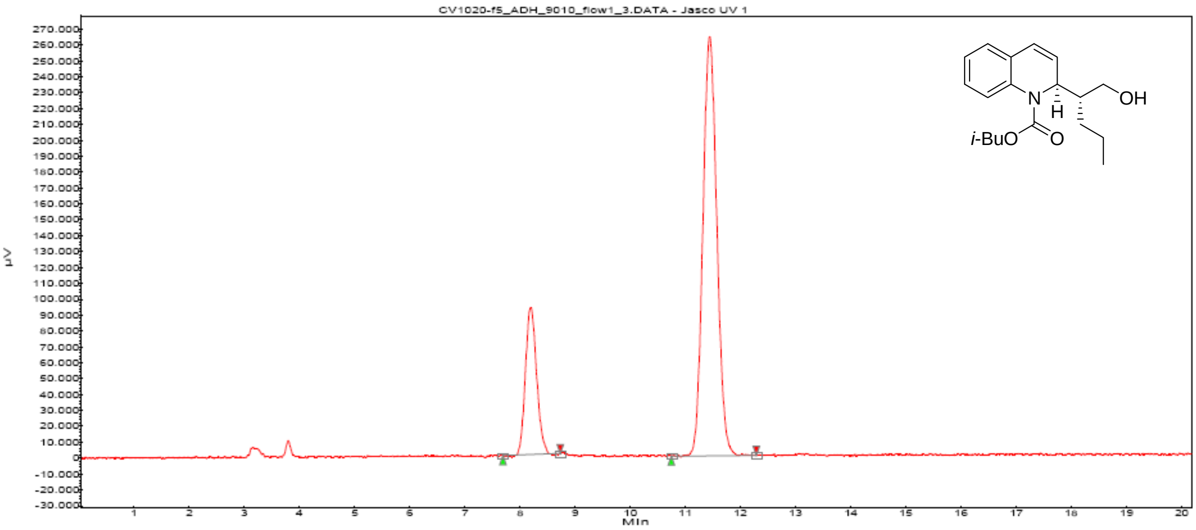
Data file: CV986-p2_ADH_9010_flow1_81.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 20.05.2012 21:17:52



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,872	8,275	9,298	51,083
2	11,095	11,633	12,800	48,917
Total				100,000

Chromatogram : CV1020-f5_ADH_9010_flow1_3

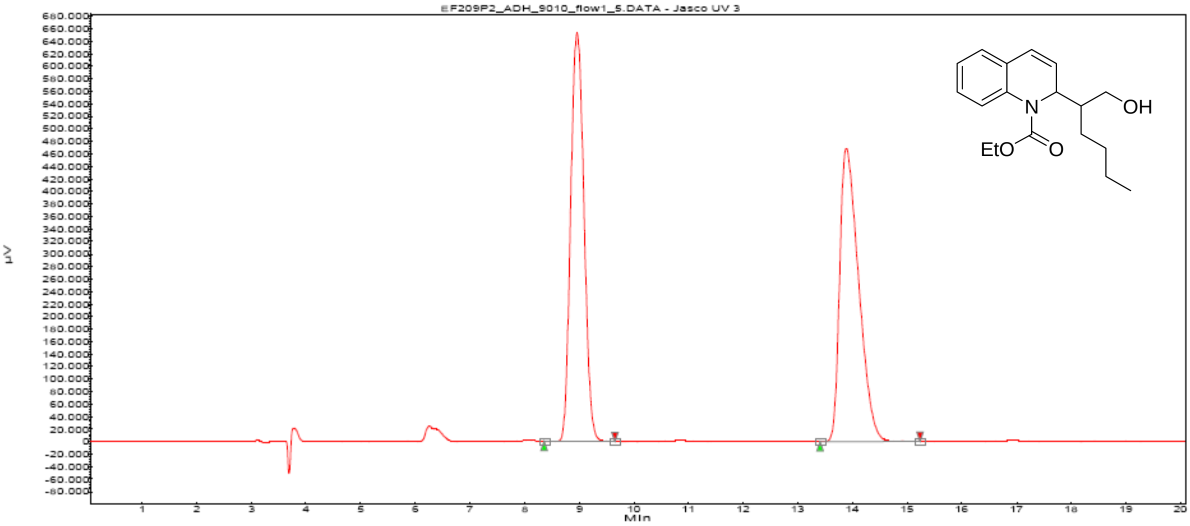
Data file: CV1020-f5_ADH_9010_flow1_3.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 01.06.2012 16:39:12



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,692	8,192	8,731	21,863
2	10,748	11,442	12,286	78,137
Total				100,000

Chromatogram : EF209P2_ADH_9010_flow1_5

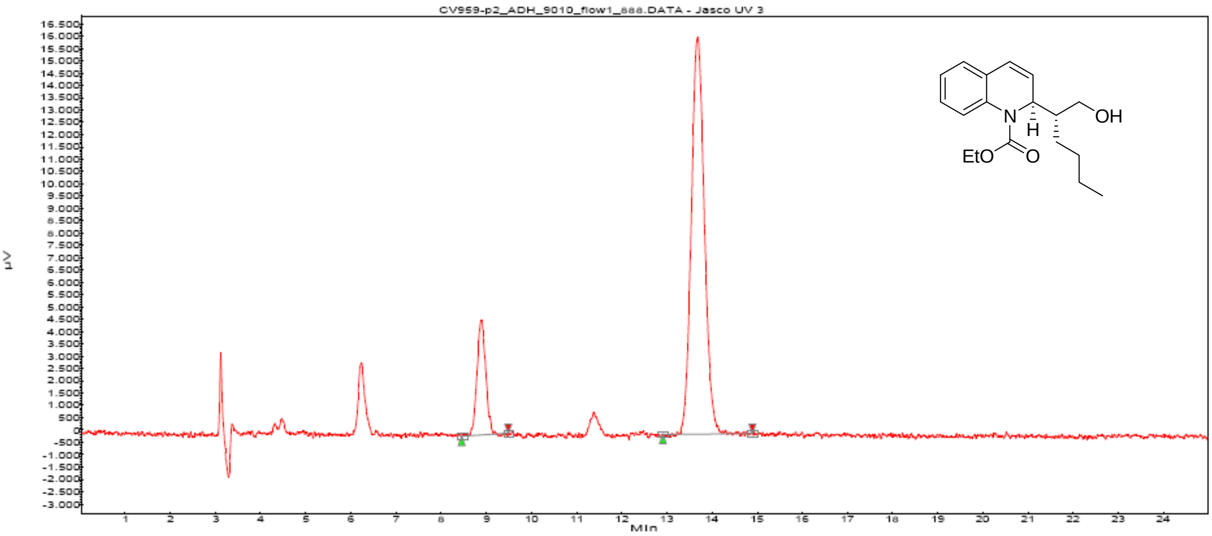
Data file: EF209P2_ADH_9010_flow1_5.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 11.02.2012 00:15:15



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8,356	8,967	9,655	0,000	49,44	653113,5	183988,9	49,444
2	UNKNOWN	13,406	13,883	15,235	0,000	50,56	468755,7	188129,6	50,556
Total						100,00	1121869,2	372118,5	100,000

Chromatogram : CV959-p2_ADH_9010_flow1_888

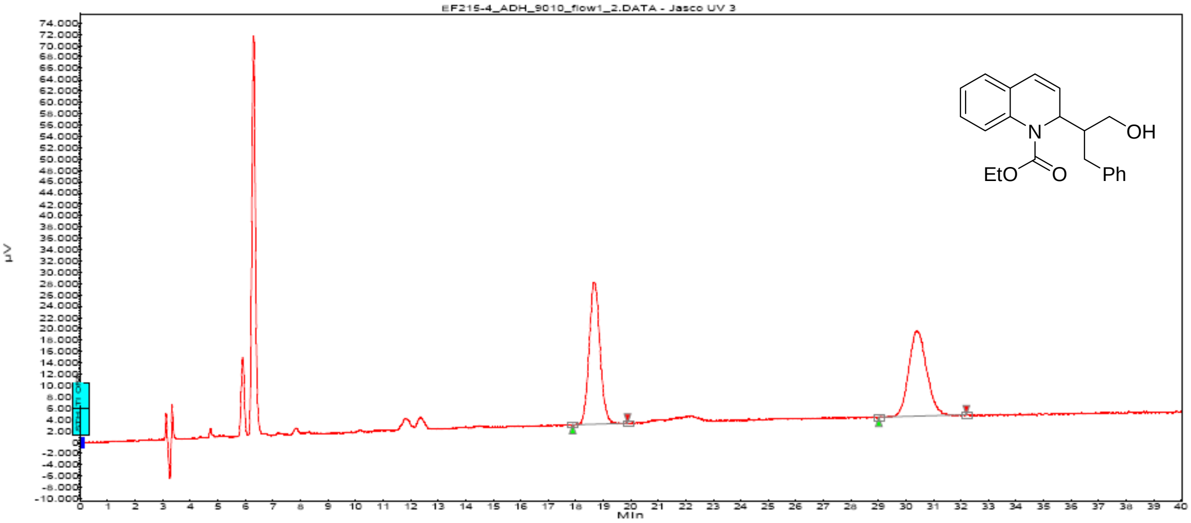
Data file: CV959-p2_ADH_9010_flow1_888.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 27.02.2012 20:36:27



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,457	8,883	9,487	16,134
2	12,913	13,683	14,896	83,866
Total				100,000

Chromatogram : EF215-4_ADH_9010_flow1_2

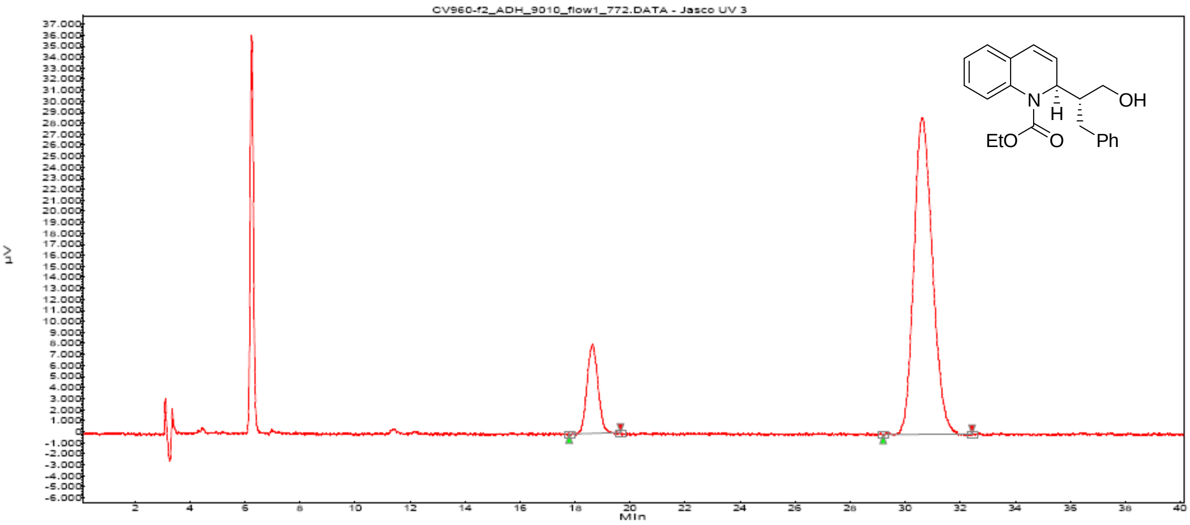
Data file: EF215-4_ADH_9010_flow1_2.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 23.03.2012 17:12:15



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	17,893	18,675	19,876	50,229
2	29,008	30,408	32,190	49,771
Total				100,000

Chromatogram : CV960-f2_ADH_9010_flow1_772

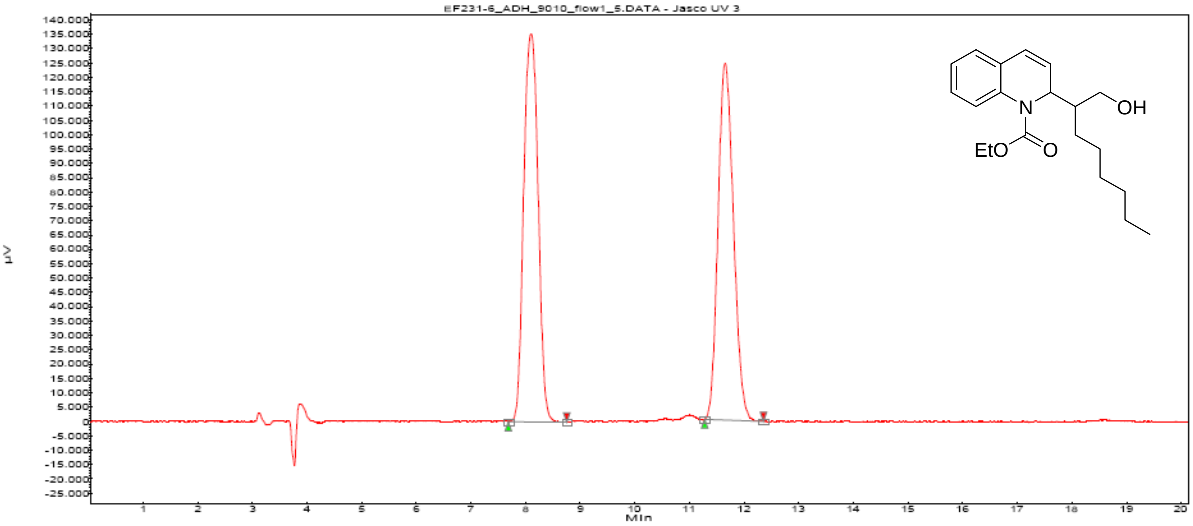
Data file: CV960-f2_ADH_9010_flow1_772.DATA
Method: HPLC1_ADH_9010_flow1_acq_60
Date: 28.02.2012 01:22:18



Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	17,789	18,633	19,649	0,000	14,12	8132,2	3696,7	14,122
2	UNKNOWN	29,194	30,625	32,417	0,000	85,88	28675,1	22480,7	85,878
Total						100,00	36807,3	26177,4	100,000

Chromatogram : EF231-6_ADH_9010_flow1_5

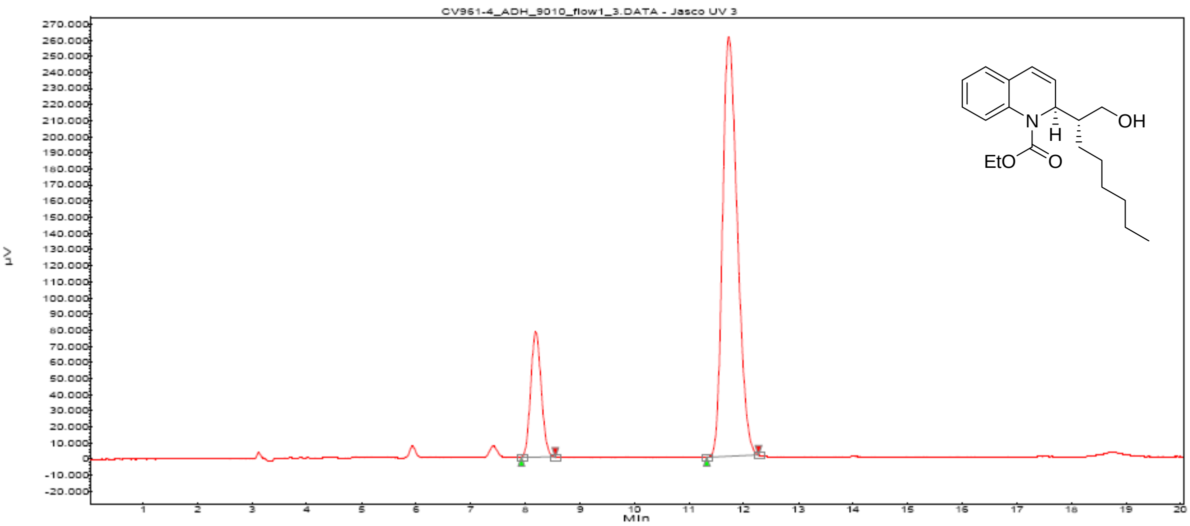
Data file: EF231-6_ADH_9010_flow1_5.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 07.03.2012 00:36:37



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,686	8,108	8,760	50,312
2	11,281	11,658	12,355	49,688
Total				100,000

Chromatogram : CV961-4_ADH_9010_flow1_3

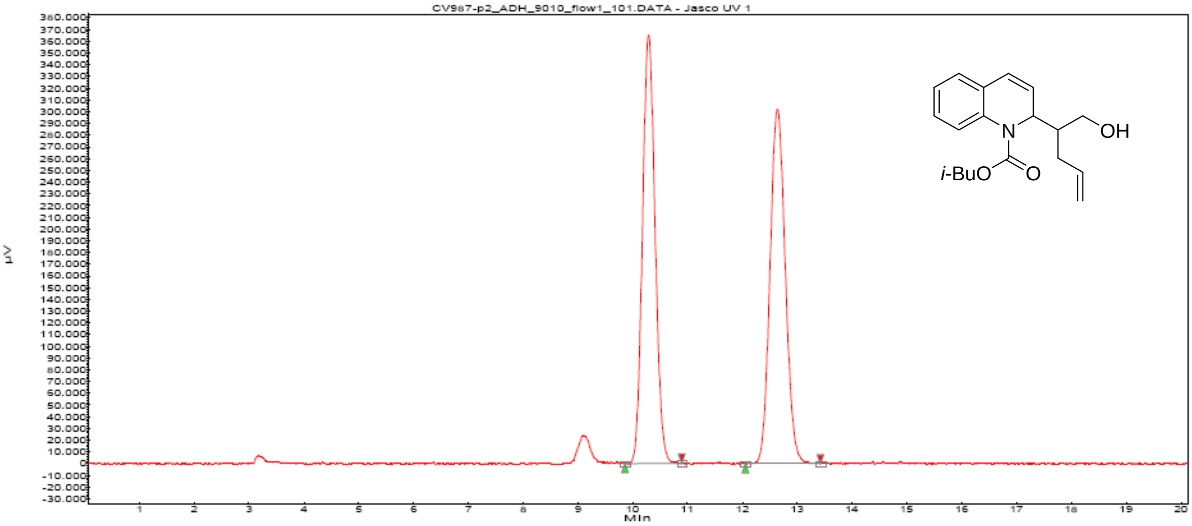
Data file: CV961-4_ADH_9010_flow1_3.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 14.03.2012 11:39:20



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	7,934	8,192	8,554	17,505
2	11,322	11,725	12,273	82,495
Total				100,000

Chromatogram : CV987-p2_ADH_9010_flow1_101

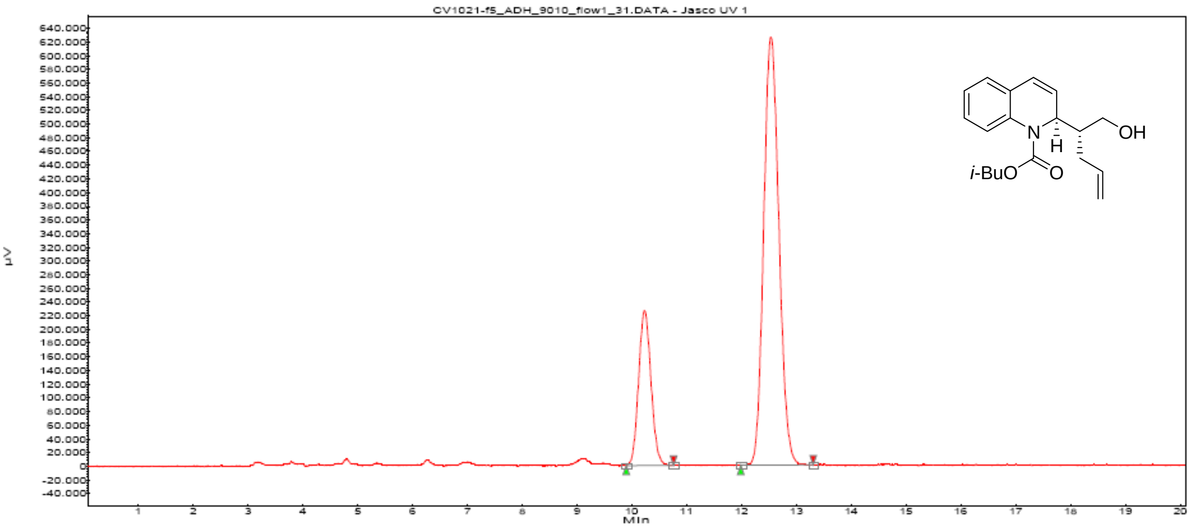
Data file: CV987-p2_ADH_9010_flow1_101.DATA
Method: HPLC1_ADH_9010_flow1_acq_30
Date: 20.05.2012 22:23:18



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,860	10,283	10,896	49,854
2	12,056	12,633	13,423	50,146
Total				100,000

Chromatogram : CV1021-f5_ADH_9010_flow1_31

Data file: CV1021-f5_ADH_9010_flow1_31.DATA
Method: HPLC1_ADH_9010_flow1_acq_40
Date: 01.06.2012 23:22:43



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,897	10,233	10,764	22,641
2	11,983	12,533	13,306	77,359
Total				100,000