Enantioselective Brønsted Acid Catalyzed Transfer Hydrogenation: Organocatalytic Reduction of Imines

Magnus Rueping, Erli Sugiono, Cengiz Azap, Thomas Theissmann and Michael Bolte

Supporting Information

General: All reactions were performed under argon atmosphere. Unless otherwise noted, all materials were obtained from commercial suppliers and were used without further purification. Solvents for extraction and chromatography were technical grade and distilled prior to use. Solvents used in reaction were reagent grade and distilled from the indicated drying agents: CH_2Cl_2 (P_2O_5), benzene (Na). For thin-layer chromatography (TLC), silica gel plates coated aluminium plates (Merck, silica gel 60 F_{254}) were used and chromatograms were visualised by irradiation with UV light at 254 nm. Column chromatography was performed using Merck silica gel 60 (particle size 0.040-0.063 mm). Solvents mixtures are understood as volume/volume.

^{1}H-NMR and ^{13}C-NMR were recorded on a Bruker AM 250 spectrometer in $CDCl_3$. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated (bs (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet)); coupling constants (*J*) are in Hertz (Hz). Field Desorption (FD) mass spectra were obtained with Finnigan MAT 95 instrument. Infrared (IR) spectra were recorded on a Bruker Tensor 27 FT-IR spectrometer and are reported in terms of frequency of absorption (cm^{-1}). The enantiomeric excesses were determined by HPLC analysis using a chiral stationary phase column (column, Daicel Co. CHIRALCEL OD-H and AD-H; eluent: hexane / 2-propanol), by comparing the samples with the corresponding racemic mixtures. Optical rotations were measured on a Perkin Elmer 241 polarimeter.

Transfer Hydrogenation of imines: In a typical experiment the imine **1** (0,20 mmol), catalyst **5** (20 mol%), Hantzsch dihydropyridine **2** (0,27 mmol) and benzene (3,5 mL) were added to a screw-capped vial and the mixture was exposed to an argon atmosphere. The resulting yellow solution was allowed to stir at 60 °C for 3d. The solvent was evaporated in vacuo, and the residue was purified by column chromatography on silica gel to afford the amine. The yields and enantiomeric excesses are given in table 3.

Amine **4a**: eluted from silica gel using hexane/ethyl acetate 40/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.50 (d, *J* = 6.5 Hz, 3H), 3.60 (s, 3H), 3.79 (bs, 1H), 4.50 (q, *J* = 6.5 Hz, 1H), 6.40 – 6.47 (m, 2H), 6.56 – 6.64 (m, 2H), 7.30 – 7.48 (m, 3H), 7.65 – 7.78 (m, 4 H); ^{13}C-NMR ($CDCl_3$): δ= 25.2, 54.6, 55.8, 114.7, 114.8, 124.4, 124.5, 125.5, 126.0, 127.7, 127.9, 128.5, 132.8, 133.6, 141.7, 143.1, 152.0; MS (FD): *m/z* (%) = 277.1 (100) [M^+]; IR (neat): 3405, 3054, 2964, 1515, 1235, 818 cm^{-1}. HPLC conditions: AD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 32,40 min; minor enantiomer: t_R = 28,55 min; $[\alpha]_D^{25}$ = +16.4 (c = 1.8, $CHCl_3$); After one recrystallisation from CH_3OH: mp: 105 °C, major enantiomer: t_R = 31,64 min; minor enantiomer: t_R = 27,89 min; $[\alpha]_D^{25}$ = +23.0 (c = 1.4, $CHCl_3$).

Amine **4b**: eluted from silica gel using toluene/ethyl acetate 15/1 as eluent.^{1}H-NMR ($CDCl_3$): δ= 1.52 (d, *J* = 6.8 Hz, 3H), 4.04 (bs, 1H), 4.56 (q, *J* = 6.8 Hz, 1H), 6.45 - 6.60 (m, 3H), 6.94 – 7.05 (m, 2H), 7.35 – 7.50 (m, 3H), 7.65 – 7.80 (m, 4H); ^{13}C-NMR ($CDCl_3$): δ= 25.1, 53.8, 113.5, 117.4, 124.3, 124.5, 125.6, 126.1, 127.7, 127.9, 128.6, 129.2, 132.8, 133.7, 142.9, 147.4; MS (FD): *m/z* (%) = 247.1 (100) [M^+]; IR (neat): 3413, 3052, 2968, 1602, 1505, 1319, 749 cm^{-1}. HPLC conditions: AD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 18,34 min; minor enantiomer: t_R = 15,56 min; $[\alpha]_D^{25}$ = -12.6 (c = 1.0, CH_3OH).

Amine **4c**: eluted from silica gel using toluene/ethyl acetate 15/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.42 (d, *J* = 6.8 Hz, 3H), 3.62 (s, 3H), 3.72 (bs, 1H), 4.37 (q, $_J$ = 6.8 Hz, 1H), 6.31 – 6.40 (m, 2H), 6.58 – 6.65 (m, 2H), 7.40 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H); ^{13}C-NMR ($CDCl_3$): δ= 26.2, 54.1, 55.7, 114.5, 114.8, 125.6, 125.7, 126.2, 141.1, 149.8, 152.2; MS (FD): *m/z* (%) = 295.1 (100) [M^+]; IR ($CDCl_3$): 3019, 1512, 1326, 758, 669 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 99/1, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 41,48 min; minor enantiomer: t_R = 55,93 min; $[\alpha]_D^{25}$ = -10.2 (c = 1.0, CH_2Cl_2)

Amine **4d**: eluted from silica gel using toluene/ethyl acetate 40/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.44 (d, *J* = 6.8 Hz, 3H), 3.99 (bs, 1H), 4.45 (q, *J* = 6.8 Hz, 1H), 6.36 – 6.43 (m, 2H), 6.56 – 6.64 (m, 1H), 6.98 – 7.07 (m, 2H), 7.41 (d, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H); ^{13}C-NMR ($CDCl_3$): 25.1, 53.3, 113.3, 117.7, 125.6, 125.7, 126.2, 129.2, 146.8; MS (FD): *m/z* (%) = 265.1 (100) [M^+]; IR (neat): 3412, 2963, 2926, 1603, 1505, 1235, 1164, 1123, 1067, 1016,

840, 750, 692 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 26,71 min; minor enantiomer: t_R = 23,49 min; $[\alpha]_D^{25}$= -16.9 (c = 1.0, CH_2Cl_2).

Amine **4e**: eluted from silica gel using toluene/ethyl acetate 15/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.41 (d, *J* = 6.8 Hz, 3H), 3.60 (s, 3H), 3.68 (bs, 1H), 4.32 (q, *J* = 6.8 Hz, 1H), 6.36 – 6.43 (m, 2H), 6.53 – 6.65 (m, 2H), 7.05 – 7.48 (m, 5H); ^{13}C-NMR ($CDCl_3$): 25.2, 54.3, 55.8, 114.6, 114.8, 125.9, 126.8, 128.6, 141.6, 145.5, 151.9 ; MS (FD): *m/z* (%) = 227.2 (100) [M^+]; IR (neat): 3402, 2963, 2928, 2361, 1515, 1235, 1037, 819, 701 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 18,30 min; minor enantiomer: t_R = 20,42 min; $[\alpha]_D^{25}$ = + 2.4 (c = 1.6, $CHCl_3$)[1].

Amine **4f**: eluted from silica gel using hexane/ethyl acetate 10/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.42 (d, *J* = 6.8 Hz, 3H), 3.92 (bs, 1H), 4.40 (q, *J* = 6.8 Hz, 1H), 6.39 – 6.46 (m, 2H), 6.51 – 6.60 (m, 1H), 6.95 – 7.06 (m, 2H), 7.09 – 7.32 (m, 5H); ^{13}C-NMR ($CDCl_3$): 25.1, 53.5, 113.4, 117.3, 125.9, 126.9, 128.7, 129.2, 145.3, 147.3 ; MS (FD): *m/z* (%) = 197.2 (100) [M^+]; IR (neat): 3410, 3023, 1602, 1505, 1318, 750 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 16,22 min; minor enantiomer: t_R = 13,98 min; $[\alpha]_D^{25}$= -12.7 (c = 1.3, CH_3OH)[2].

Amine **4g**: eluted from silica gel using hexane/ethyl acetate 10/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.51 (d, *J* = 6.8 Hz, 3H), 3.69 (s, 3H), 3.78 (bs, 1H), 4.74 (q, *J* = 6.8 Hz, 1H), 6.45 – 6.52 (m, 2H), 6.65 – 6.74 (m, 2H), 6.97 – 7.08 (m, 2H), 7.13 – 7.24 (m, 1H), 7.32 – 7.41 (m, 1H); ^{13}C-NMR ($CDCl_3$): δ= 23.1, 48.9, 55.7, 114.8, 115.3, 115.4, 115.6, 124.4, 124.4, 127.4, 127.5, 128.4, 128.5, 152.6, 158.5, 162.5; MS (FD): *m/z* (%) = 245.1 (100) [M^+]; IR ($CDCl_3$): 3019, 1512, 1215, 759, 669 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 14,55 min; minor enantiomer: t_R = 17,24 min; $[\alpha]_D^{25}$ = + 8.4 (c = 1.0, $CHCl_3$).

Amine **4h**: eluted from silica gel using hexane/ethyl acetate 40/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.35 (d, *J* = 6.6 Hz, 3H), 2.34 (s, 3H), 3.61 (s, 3H), 3.69 (bs, 1H), 4.53 (q, *J* = 6.6 Hz, 1H), 6.30 – 6.40 (m, 2H), 6.55 – 6.70 (m, 2H), 7.05 – 7.15 (m, 3H), 7.30 – 7.45 (m, 1H); ^{13}C-NMR

[1] ($[\alpha]_D^{25}$ = + 7.0 (c = 2.0, $CHCl_3$) (*R*-configuration), Y. Chi, Y.-C. Zhou, X. Zhang, J. Org. Chem. 2003 68 4120)

[2] ($[\alpha]_D^{25}$ = -19.5 (c = 1, CH_3OH) (*R*-configuration), G. Wittig, U. Thiele, Liebigs Ann. Chem. 1969 726 1

($CDCl_3$): δ= 19.0, 23.2, 50.5, 55.8, 114.2, 114.9, 124.7, 126.6, 130.6, 134.6, 141.7, 143.1, 151.9; MS (FD): *m/z* (%) = 241.2 (100) [M^+]; IR (neat): 3403, 2965, 1511, 1235, 1038, 819 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 14,42 min; minor enantiomer: t_R = 18,75 min; $[\alpha]_D^{25}$ = -33.5 (c = 2.2, CH_3OH).

Amine **4i**: eluted from silica gel using hexane/ethyl acetate 40/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.44 (d, *J* = 6.5 Hz, 3H), 2.28 (s, 3H), 2.39 (s, 3H), 3.69 (s, 3H), 3.73 (bs, 1H), 4.57 (q, *J* = 6.5 Hz, 1H), 6.36 – 6.46 (m, 2H), 6.63 – 6.73 (m, 2H), 6.99 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ= 18.9, 21.0, 23.2, 50.3, 55.8, 114,2, 114.8, 124.7, 127.2, 131.4, 134.5, 136.1, 140.0, 141.7, 151.8; MS (FD): *m/z* (%) = 255.2 (100) [M^+]; IR ($CDCl_3$): 2235, 1505, 1241, 908, 735 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 12,26 min; minor enantiomer: t_R = 15,43 min; $[\alpha]_D^{25}$ = -28.7 (c = 1.8, CH_2Cl_2).

Amine **4j**: eluted from silica gel using hexane/ethyl acetate 40/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.43 (d, *J* = 6.8 Hz, 3H), 3.60 (s, 3H), 3.71 (bs, 1H), 4.36 (q, *J* = 6.8 Hz, 1H), 6.39 – 6.45 (m, 2H), 6.58 – 6.65 (m, 2H), 7.21 – 7.37 (m, 5H), 7.42 – 7.52 (m, 4H); ^{13}C-NMR ($CDCl_3$): δ= 25.2, 54.0, 55.8, 114.6, 114.8, 126.4, 127.1, 127.4, 128.8, 139.8, 141.0, 141.6, 144.7, 152.0; MS (FD): *m/z* (%) = 303.2 (100) [M^+]; IR (neat): 3402, 3027, 2963, 1506, 1237, 819, 698 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 35,77 min; minor enantiomer: t_R = 38,32 min; $[\alpha]_D^{25}$ = + 41.9 (c = 2.3, $CHCl_3$). After one recrystallisation from CH_3OH: mp: 98 °C, major enantiomer: t_R = 37,13 min; minor enantiomer: t_R = 39,29 min; $[\alpha]_D^{25}$ = +55.9 (c = 1.7, $CHCl_3$).

Amine **4k**: eluted from silica gel using toluene/ethyl acetate 15/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.40 (d, *J* = 6.7 Hz, 3H), 3.62 (s, 3H), 3.71 (s, 1H), 4.30 (q, *J* = 6.7 Hz, 1H), 6.40 (d, *J* = 8.9 Hz, 2H), 6.62 (d, *J* = 8.9 Hz, 2H), 6.79 (d, *J* = 8.7 Hz, 2H), 7.20 (d, *J* = 8.7 Hz, 2H); ^{13}C-NMR ($CDCl_3$): 25.2, 53.7, 55.3, 55.8, 114.0, 114.6, 114.8, 126.9, 137.6, 141.7, 151.9, 158.5; MS (FD): *m/z* (%) = 257.2 (100); IR ($CDCl_3$): 3853, 3020, 2360, 1653, 1559, 1508, 1216, 759, 669 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 24,08 min; minor enantiomer: t_R = 27,82 min; $[\alpha]_D^{25}$ = +16.7 (c = 1.3, CH_3OH).

Amine **4l**: eluted from silica gel using hexane/ethyl acetate 40/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.39 (d, *J* = 6.7 Hz, 3H), 3.62 (s, 1H), 3.67 (bs, 1H), 4.28 (q, *J* =6.7 Hz, 1H), 6.30 – 6.42 (m, 2H), 6.55 – 6.68 (m, 2H), 7.05 – 7.16 (m, 1H), 7.18 – 7.35 (m, 2H), 7.44 (s, 1H); ^{13}C-NMR ($CDCl_3$): δ= 25.2, 54.0, 55.8, 114.6, 114.8, 122.8, 124.6, 129.1, 130.0, 130.3, 141.2, 148.2, 152.1; MS (FD): *m/z* (%) = 305.0 (100) Br_2 pattern [M^+], 612.0 (39) Br_2 pattern [$2M^+$], 919 (2) Br_2 pattern [$3M^+$]; IR (neat): 3400, 2965, 2831, 1687, 1512, 1235, 1038, 819, 784 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 28,79 min; minor enantiomer: t_R = 35,54 min; $[\alpha]_D^{25}$ = +15.4 (c = 2.0, CH_3OH).

Amine **4m**: eluted from silica gel using hexane/ethyl acetate 5/1 as eluent. ^{1}H-NMR ($CDCl_3$): δ= 1.41 (d, *J* = 6.5 Hz, 3H), 3.60 (s, 3H), 3.79 (bs, 1H), 4.76 (q, *J* = 6.5 Hz, 1H), 6.36 (d, *J* = 8.9 Hz, 2H), 6.58 (d, *J* = 8.9 Hz, 2H), 7.15 – 7.28 (m, 1H), 7.30 – 7.45 (m, 1H), 7.55 – 7.75 (m, 2H); ^{13}C-NMR ($CDCl_3$): δ= 24.5, 49.1, 54.7, 113.4, 113.7, 125.0, 125.6, 125.8, 131.6, 139.8, 151.1; MS (FD): *m/z* (%) = 295.1 (100) [M^+]; IR (neat): 3405, 2982, 1596, 1513, 1285, 1040, 820, 772 cm^{-1}. HPLC conditions: OD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R = 14.16 min; minor enantiomer: t_R = 16.09 min; $[\alpha]_D^{25}$ = -20.6 (c = 0.6, $CHCl_3$).

X-Ray crystallography:

Crystal data for **5c**: $C_{40}H_{25}O_4P$ * 3 CH_3OH * H_2O, M = 714.71, orthorhombic, space group $P2_12_12_1$, a = 8.2655(4) Å, b = 13.7986(6) Å, c = 32.804(2) Å, V = 3741.4(3) Å^3, T = 173 K, Z = 4, D_c = 1.269 g cm^{-3}, λ (Mo Kα) = 0.71073 Å, 31567 reflections measured, 6560 unique (R_{int} = 0.075) which were used in all calculations. An empirical absorption correction was performed using the MULABS option [1] in the PLATON program [4]. The structure was solved by direct methods (SHELXS-97) [2] and refined by full-matrix least-squares methods on F^2 with 488 parameters (SHELXL-97)) [3]. R_1 = 0.0874 ($I > 2\sigma(I)$) and wR_2 = 0.2378, GOF = 1.021; max/min residual density 0.785/-0.753 e Å^{-3}. The absolute structure could be determined: Flack-x-parameter = 0.0(2). H atoms bonded to C were refined using a riding model. H atoms bonded to O could not be located and were omitted from the refinement. One methanol molecule is disordered over two sites with occupation factors of 0.63(1) and 0.37(1). CCDC reference number 273898.

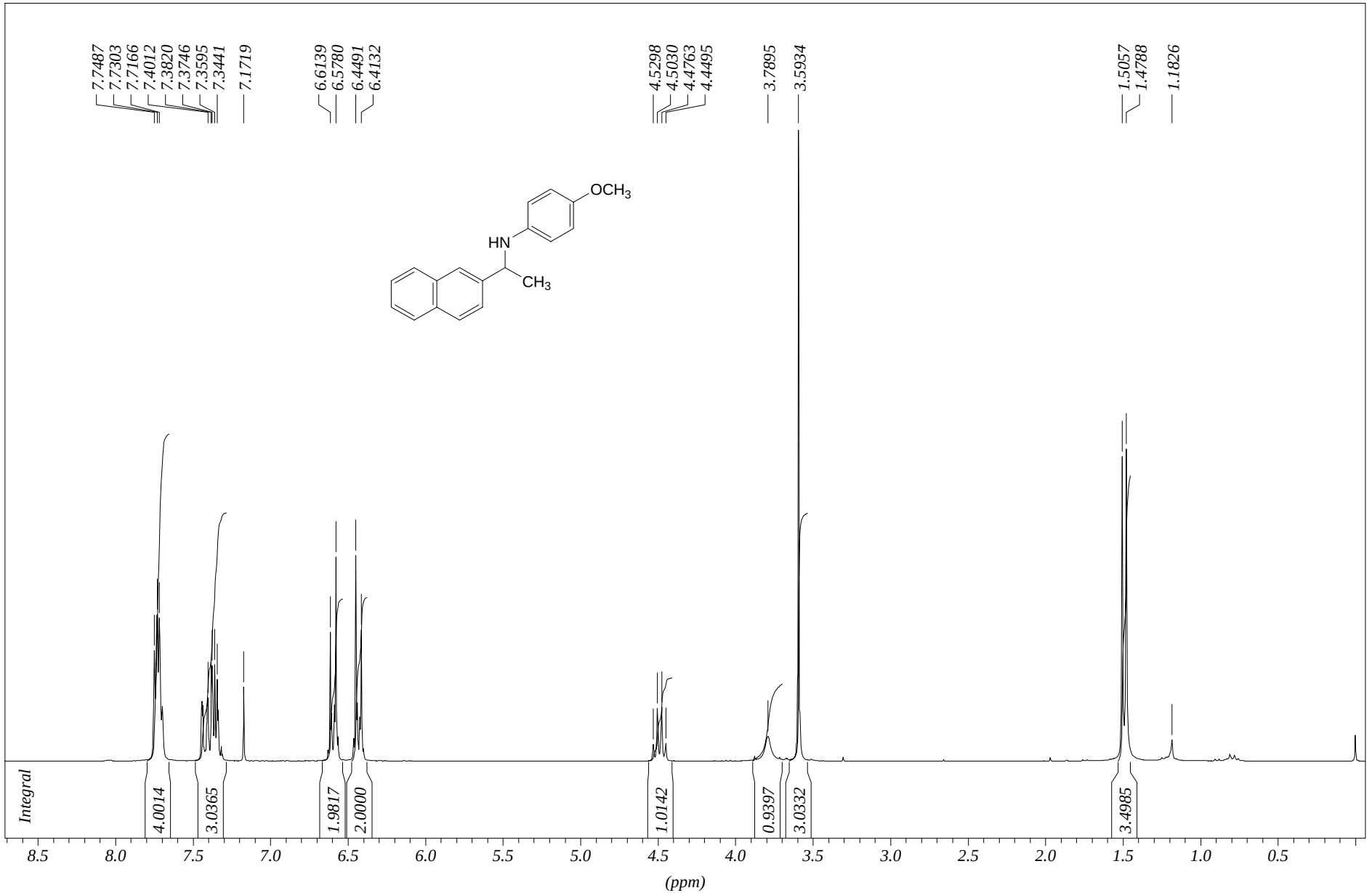
[1] Blessing, R.H. (1995). Acta Cryst. A51, 33-38.

[2] Sheldrick, G.M. (1990). Acta Cryst. A46, 467 - 473.

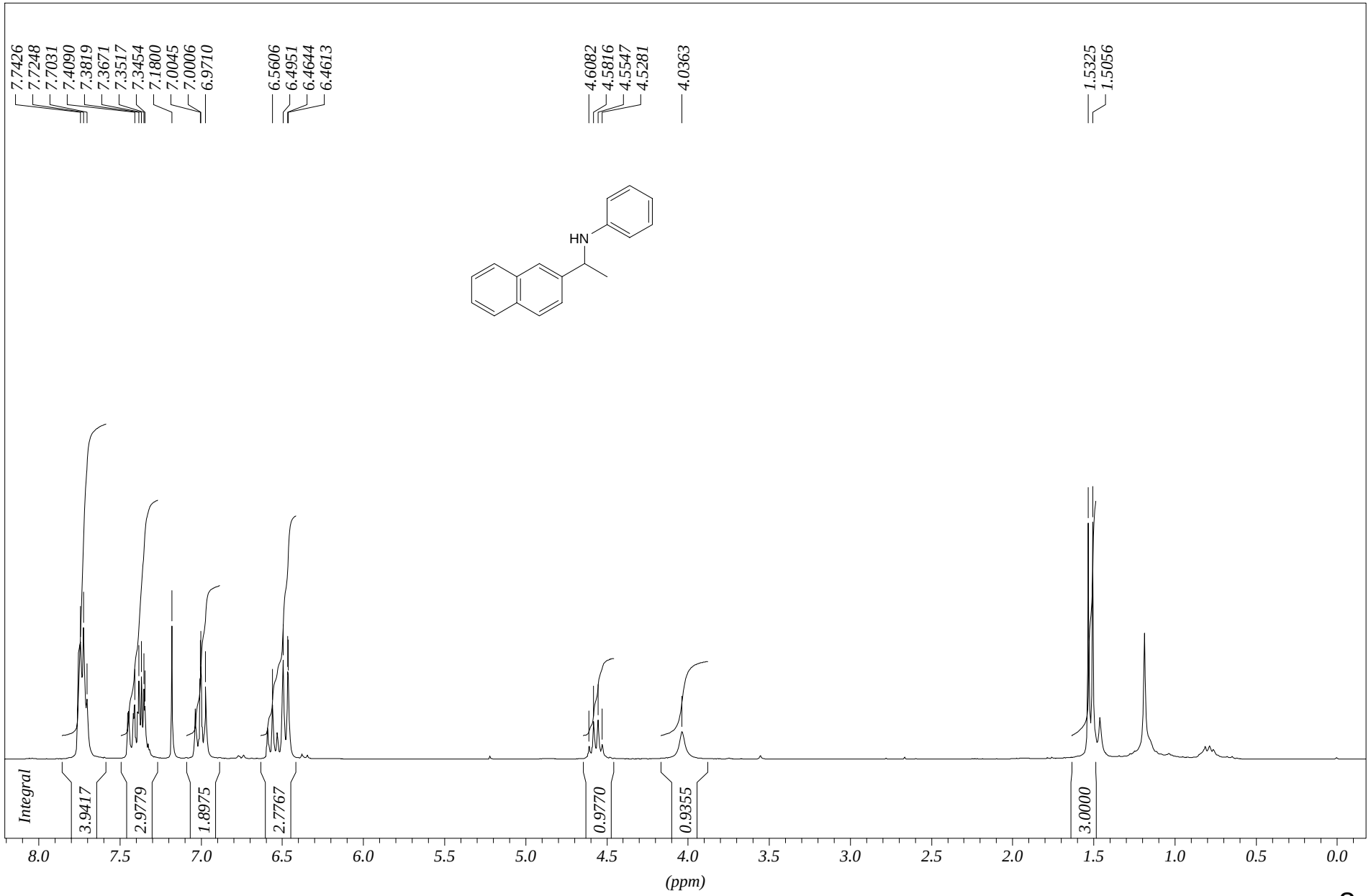
[3] Sheldrick, G.M. (1997). SHELXL-97. Program for the refinement of crystal structures. Univ. of Göttingen, Federal Republic of Germany.

[4] Spek, A.L. (2003). J. Appl. Cryst. 36, 7-13.

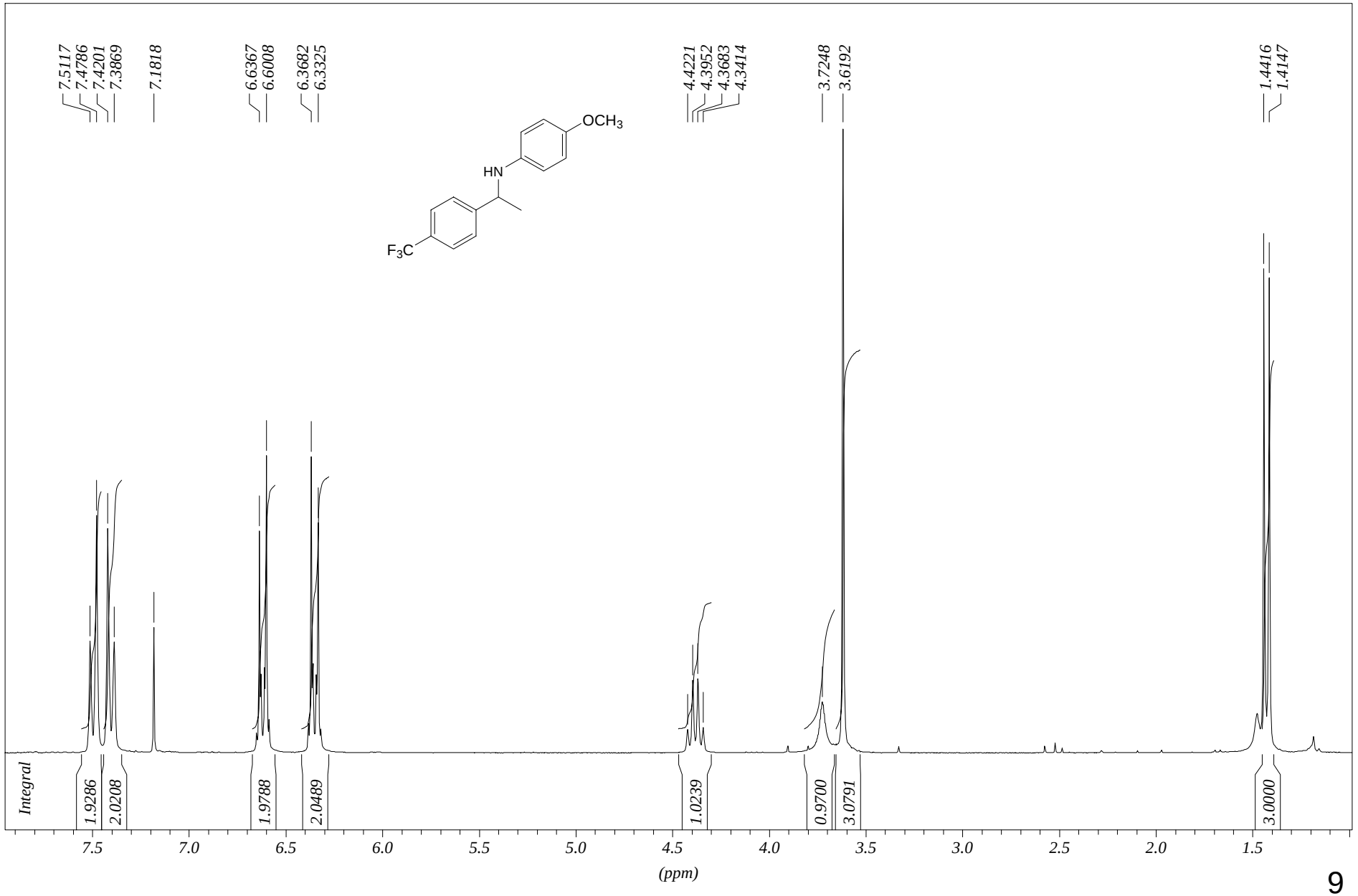
ES210 1H_16scans CDCl3 v serv 44



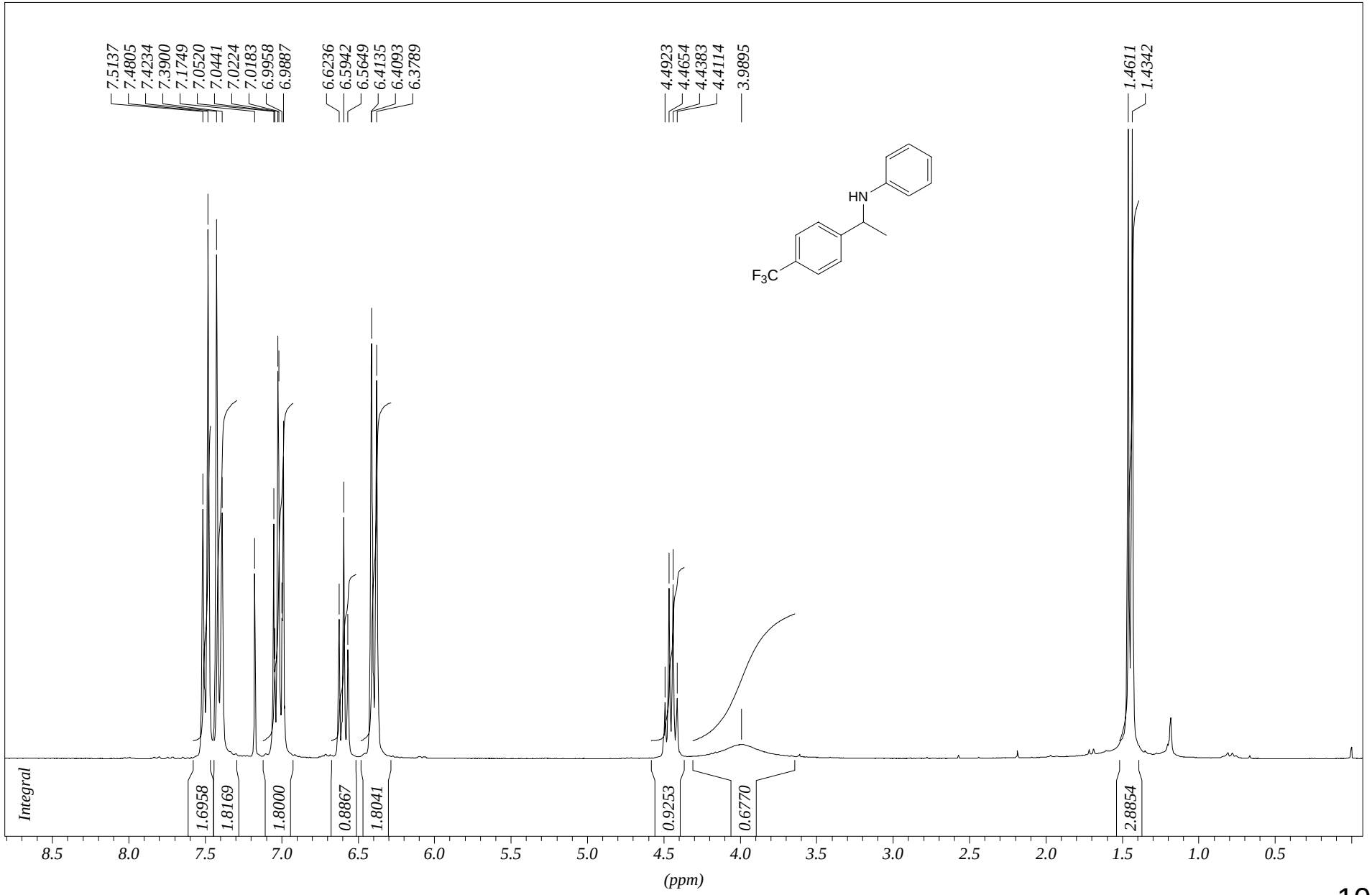
ES193 1H_16scans CDCl3 v serv 26



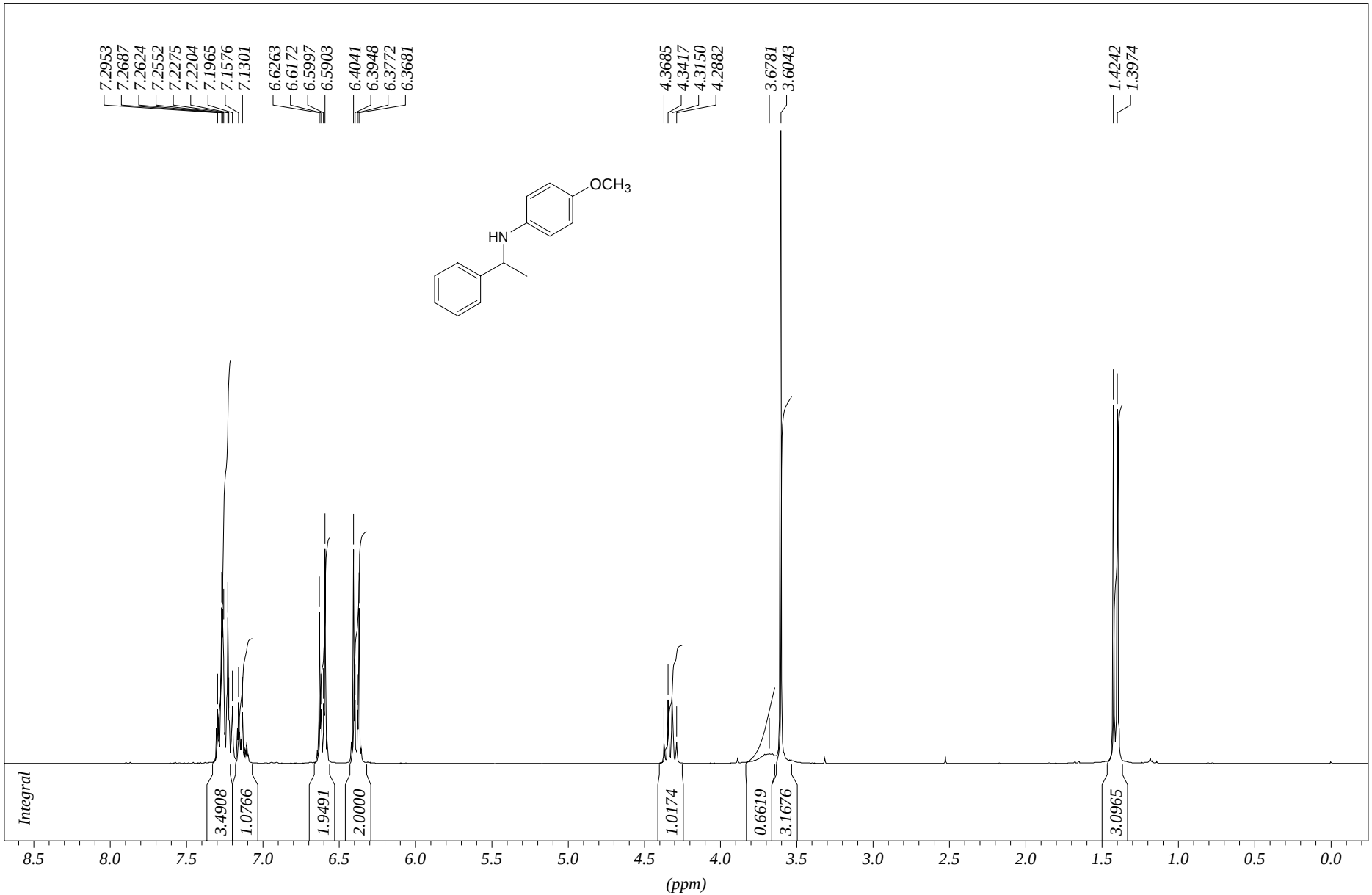
ES203 1H_16scans CDCl3 v serv 7



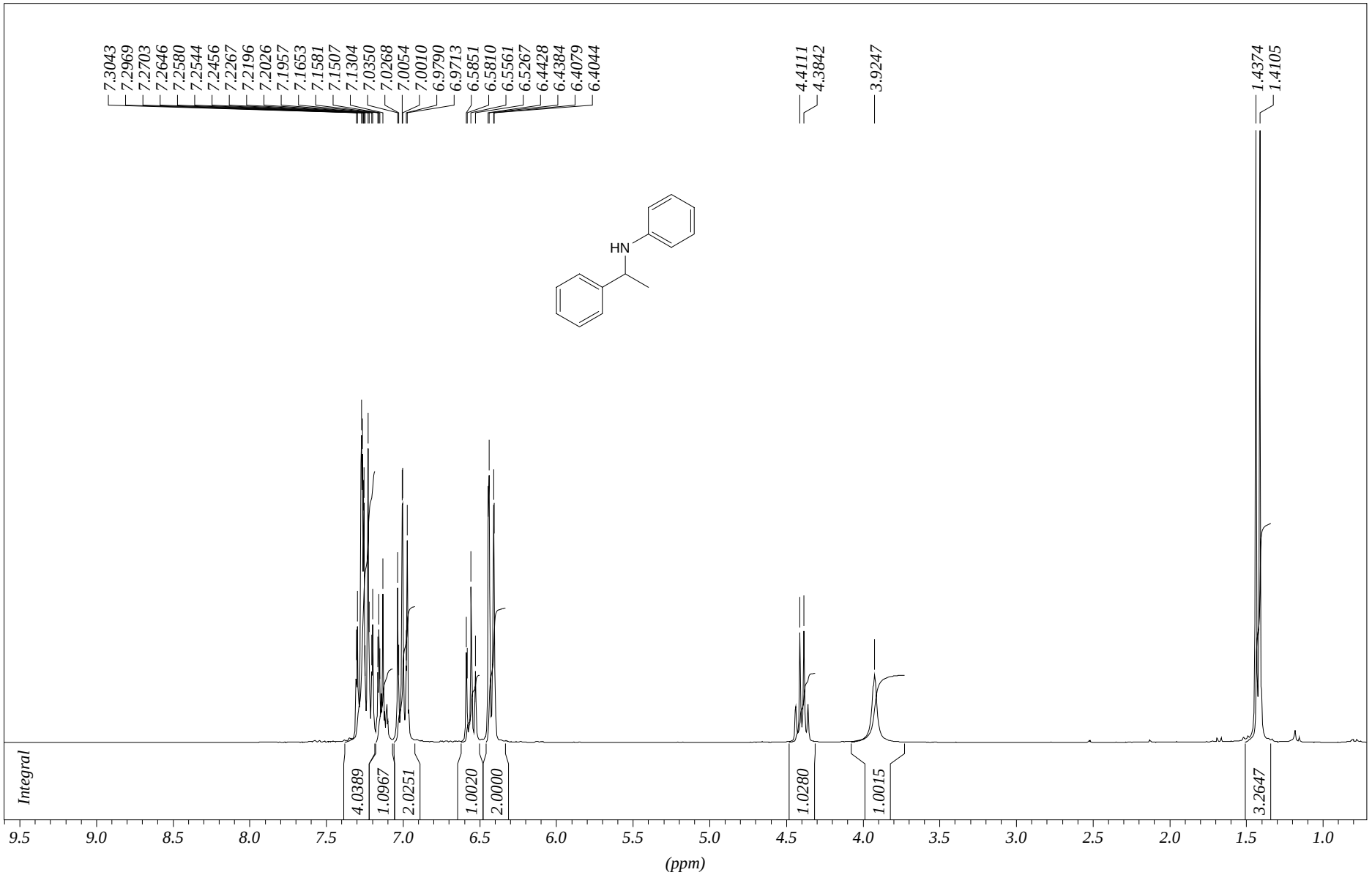
ES208 16scans CDCl3 v serv 31



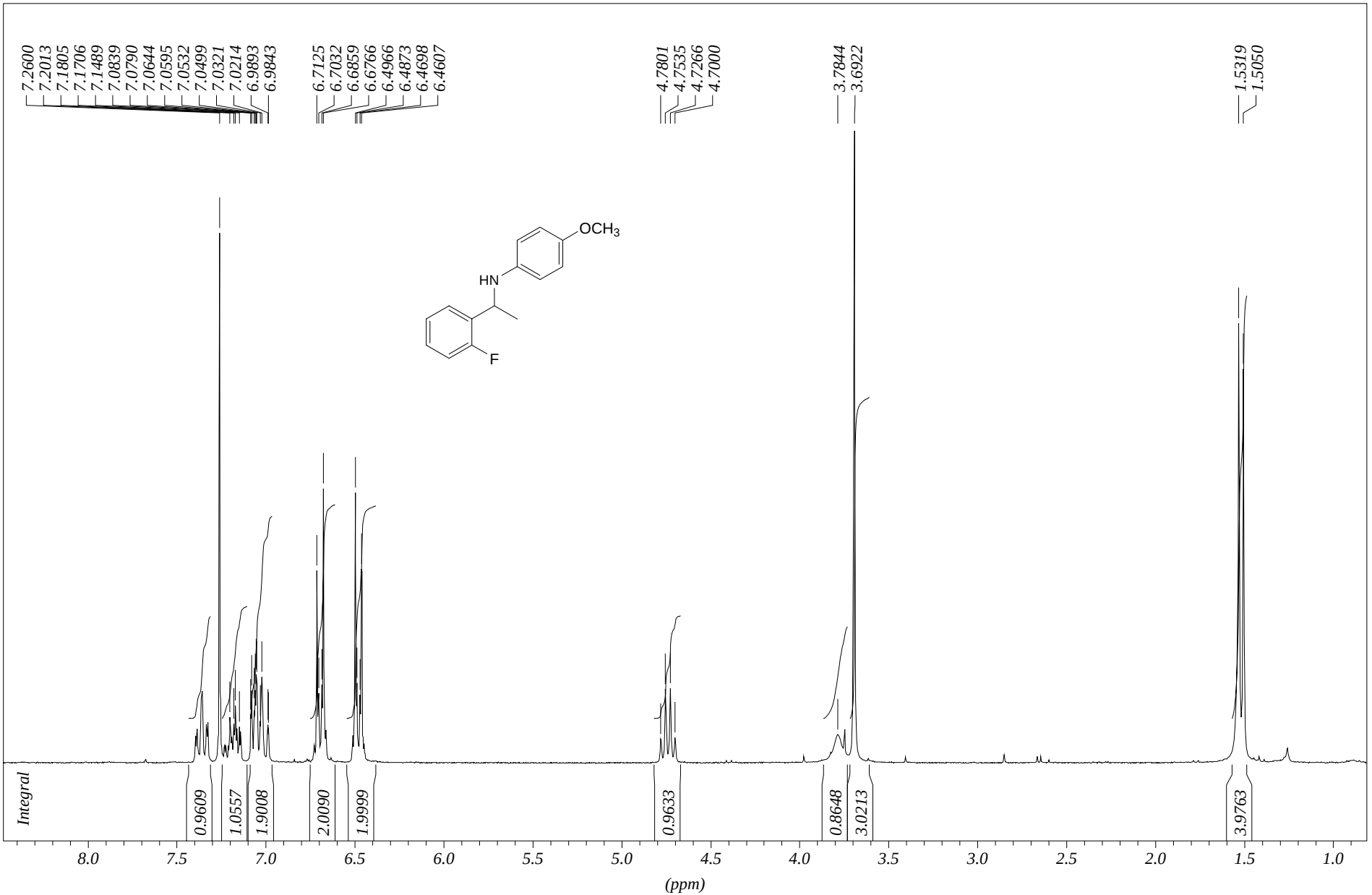
ES209 1H_16scans CDCl3 v serv 44



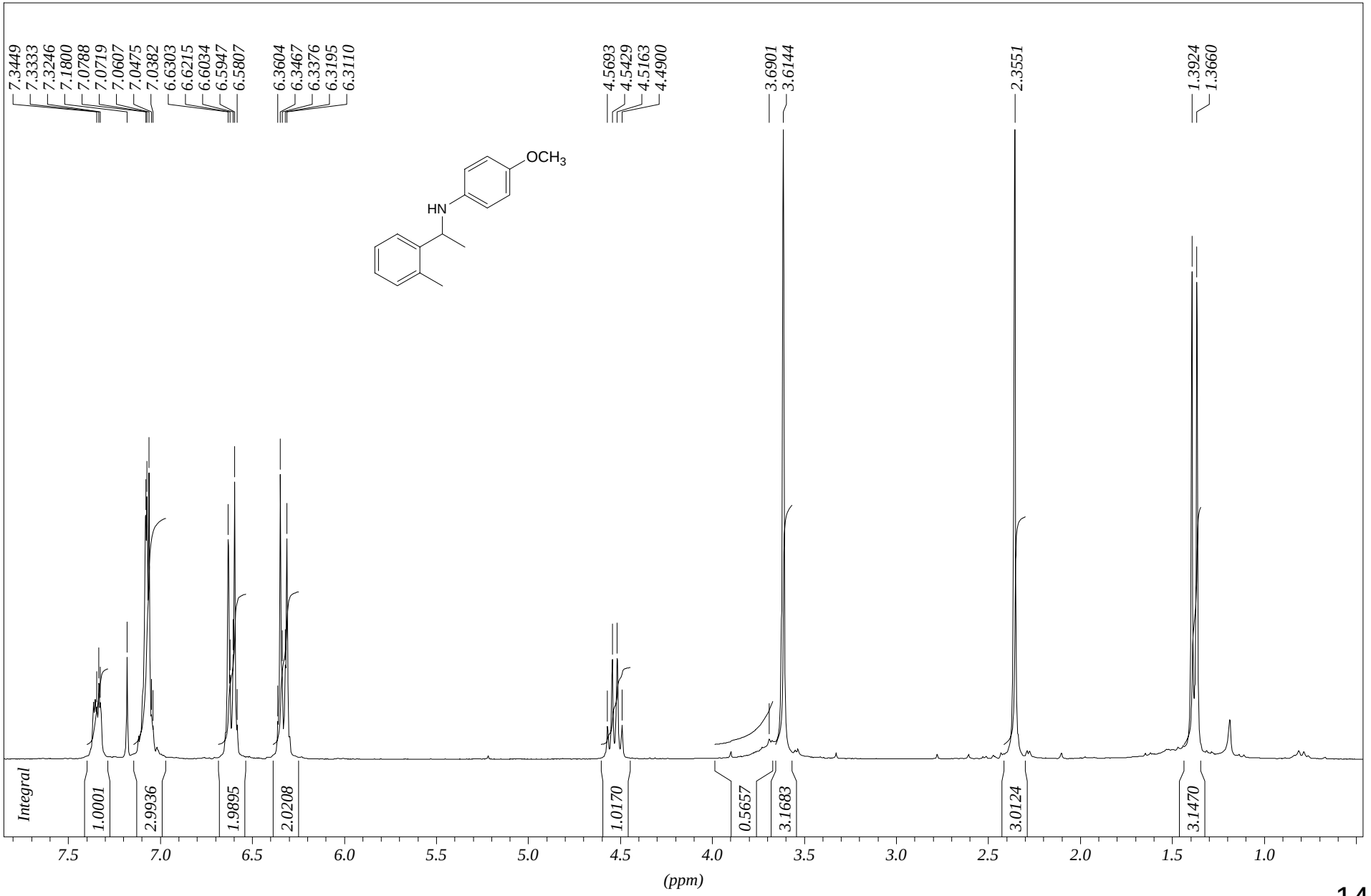
ES207 1H_16scans CDCl3 v serv 19



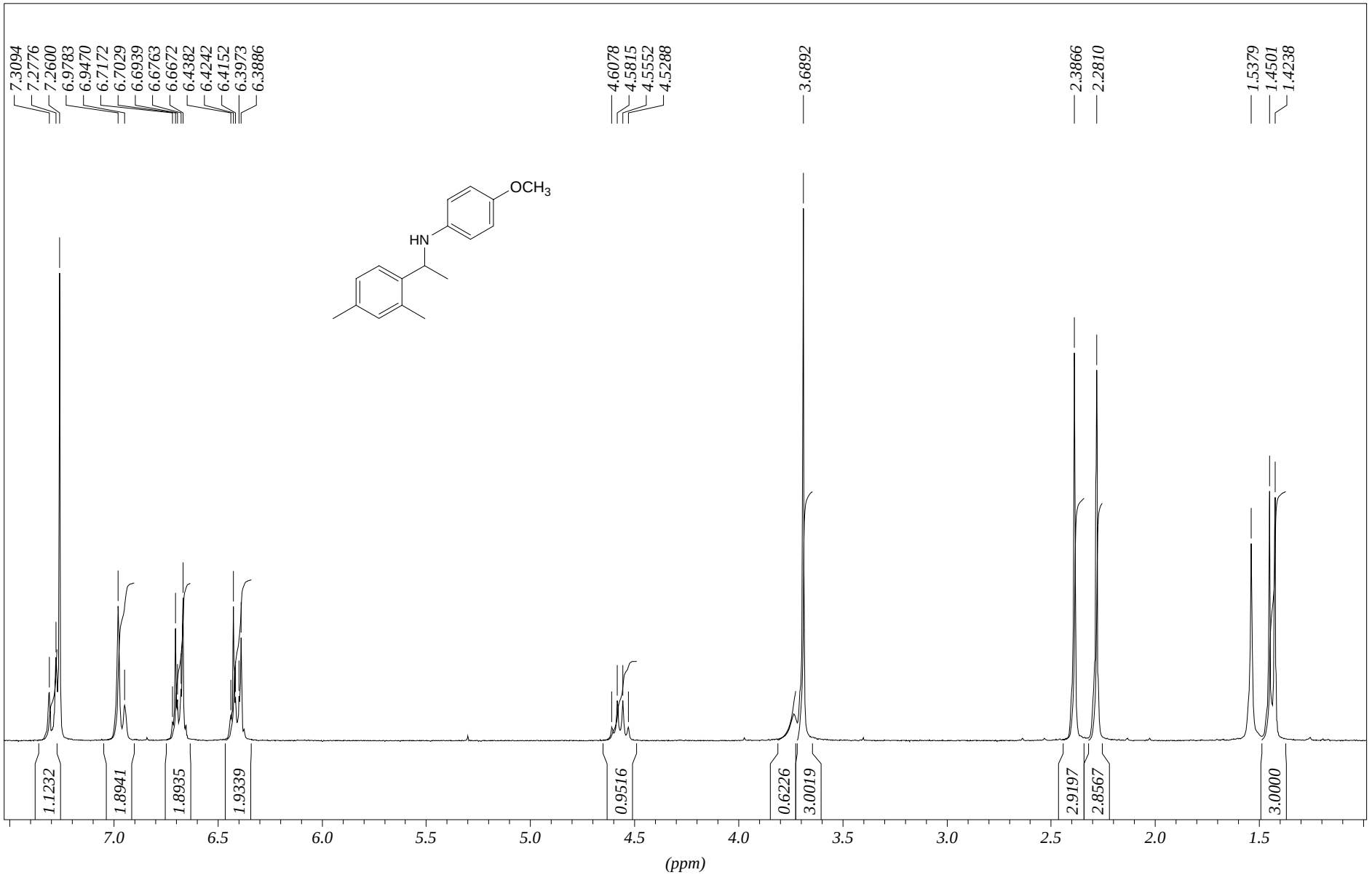
ES195 1H_16scans CDCl3 v serv 17



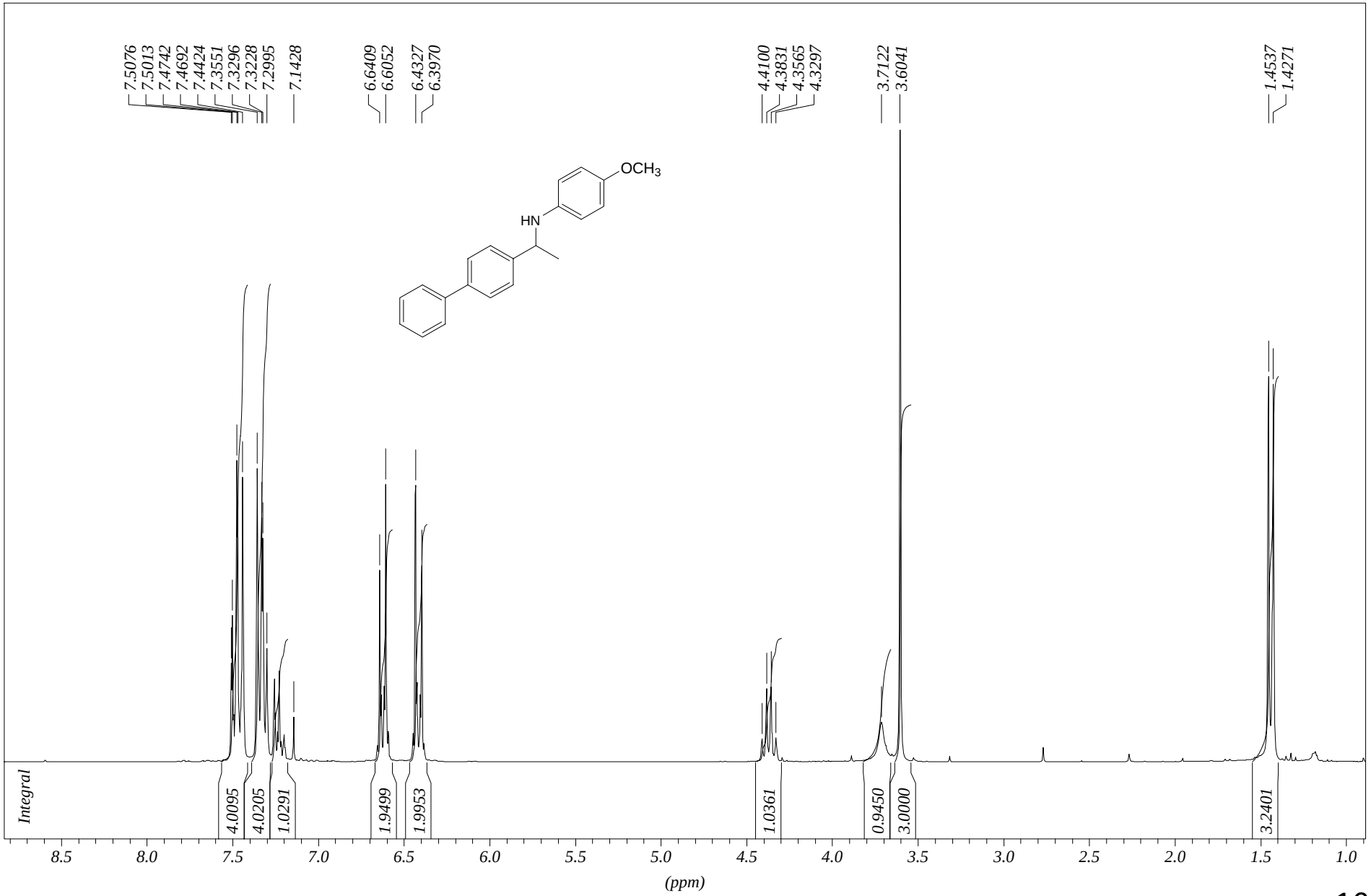
ES205 1H_16scans CDCl3 v serv 46



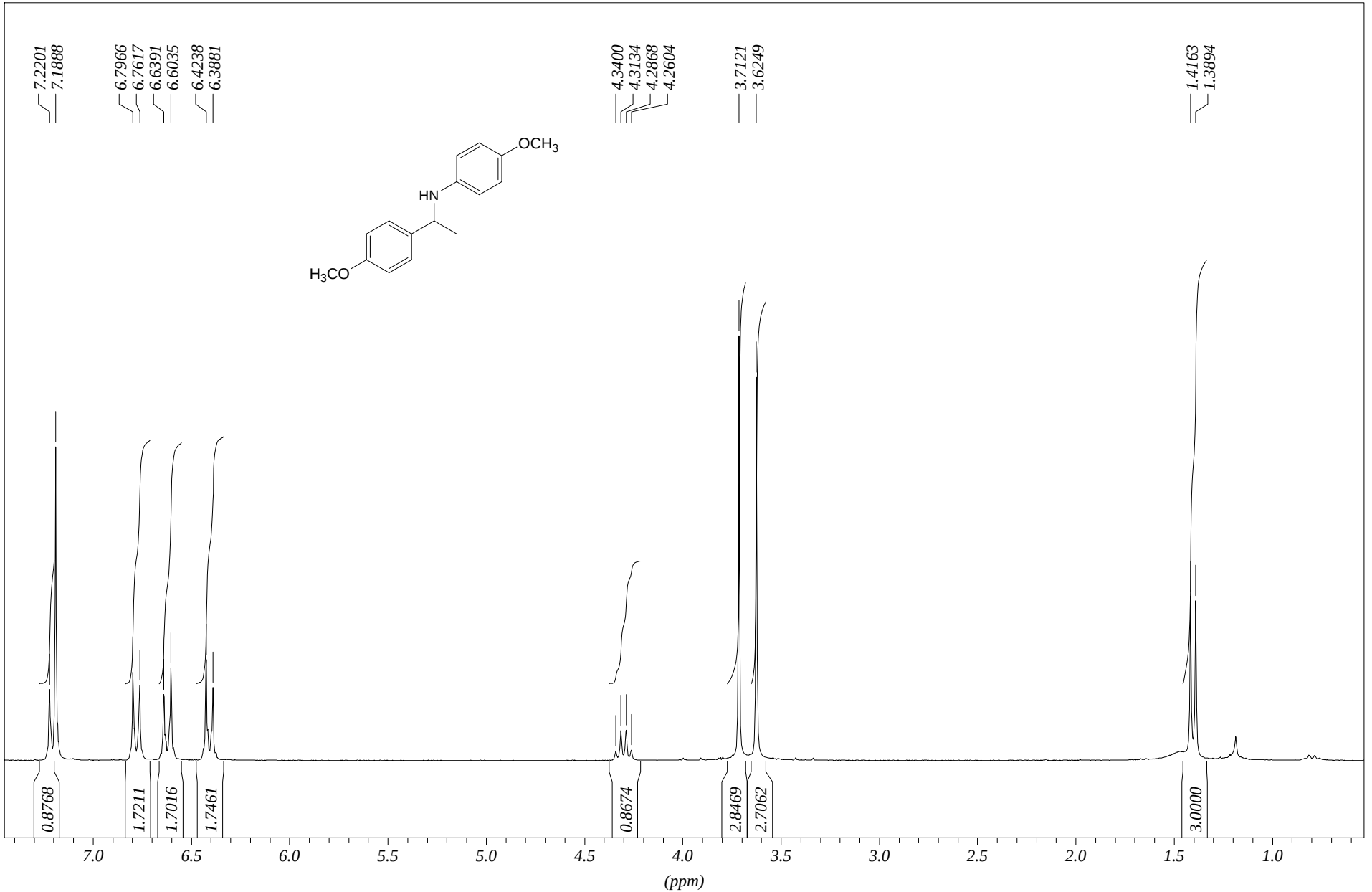
ES211 1H_16scans CDCl3 v serv 44



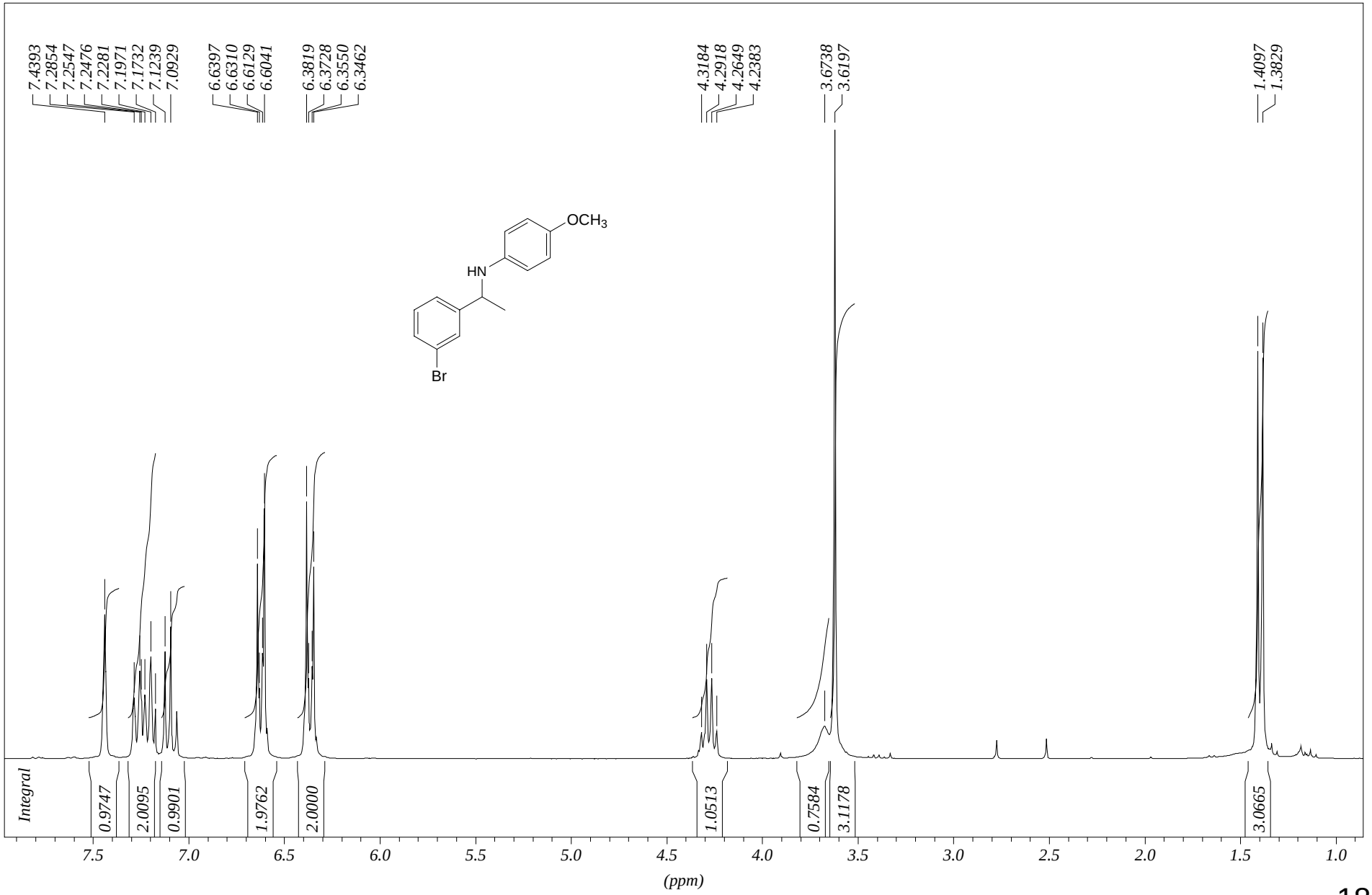
ES206 1H_16scans CDCl3 v serv 9

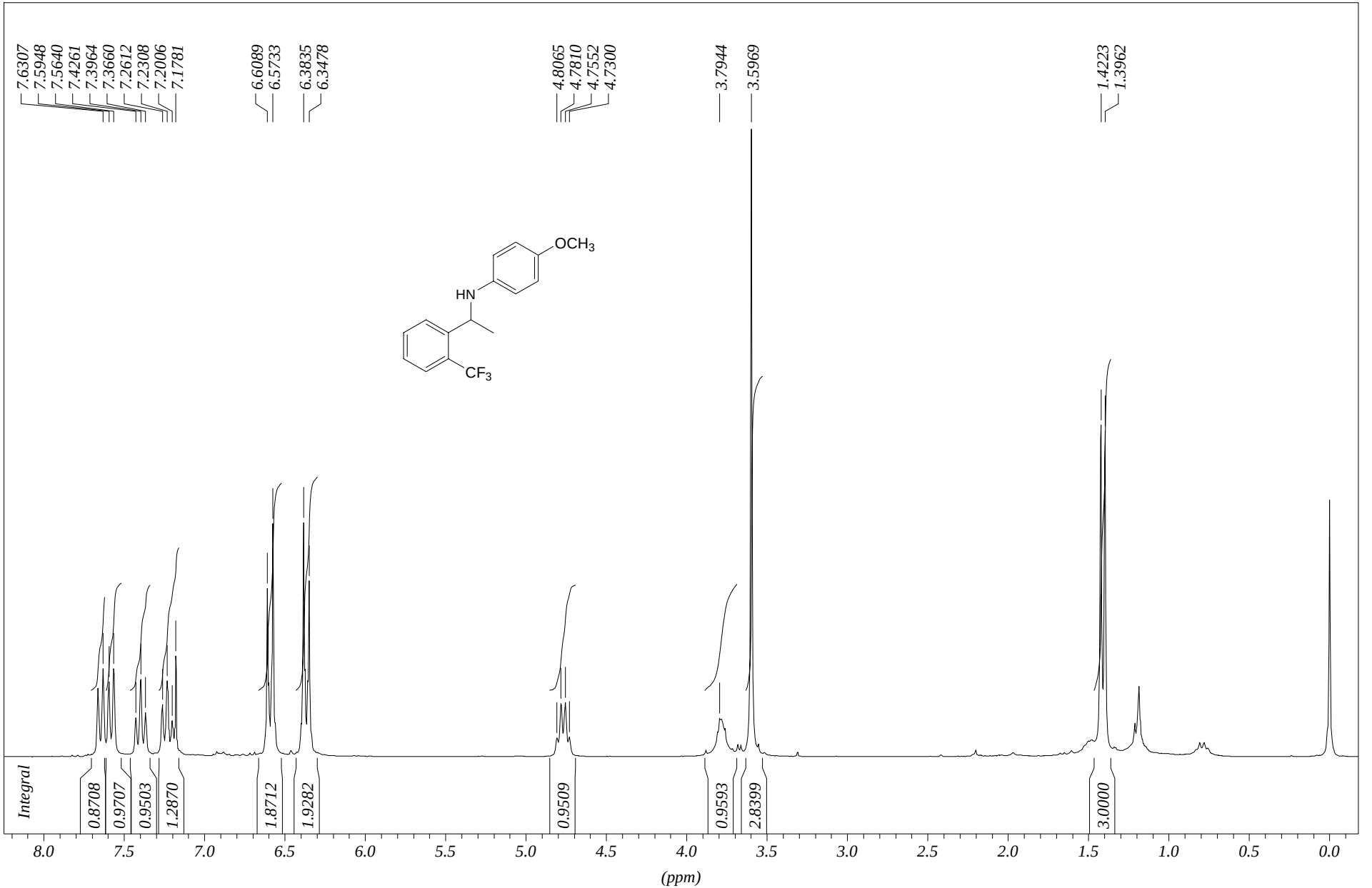


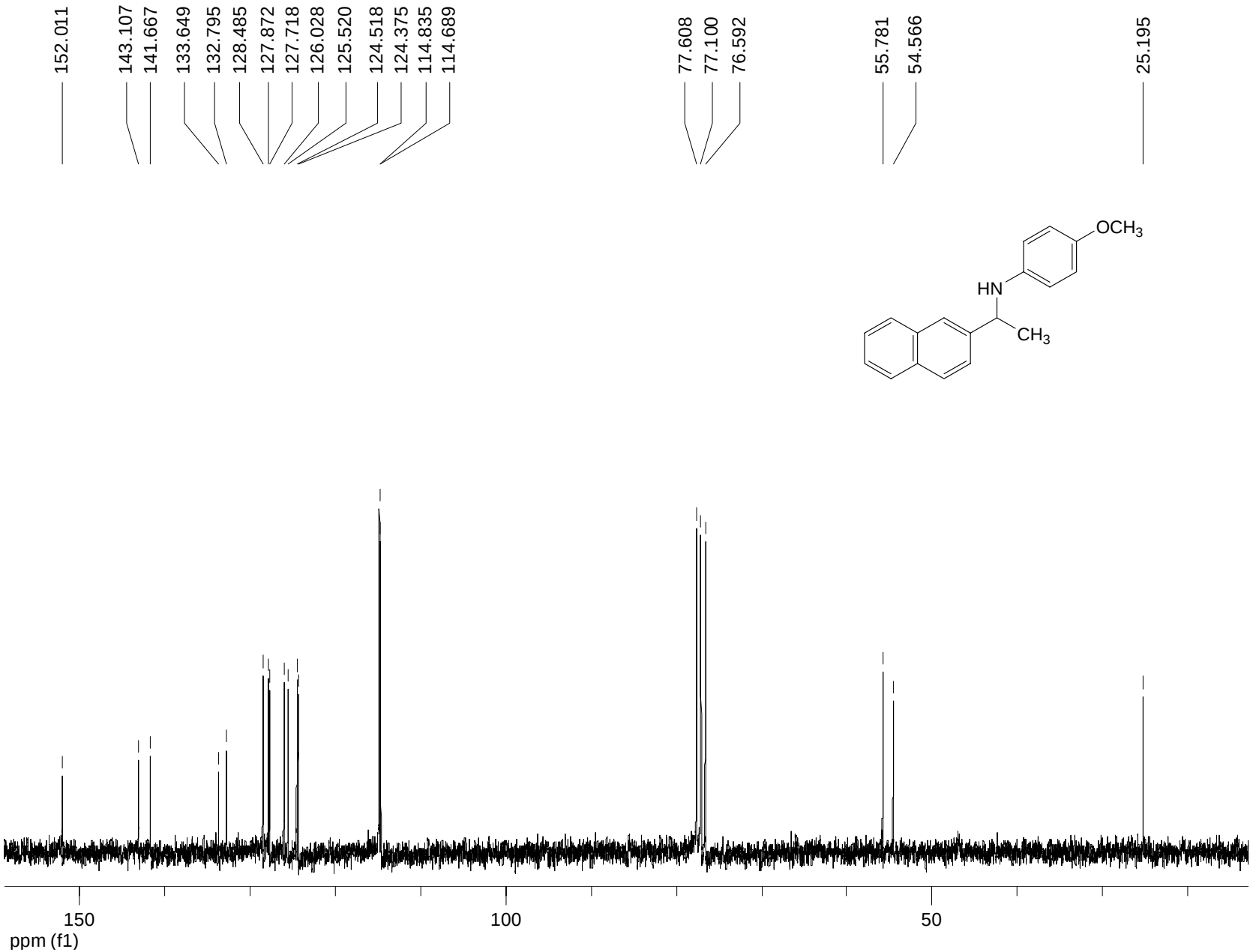
ES200 1H_16scans CDCl3 v serv 7

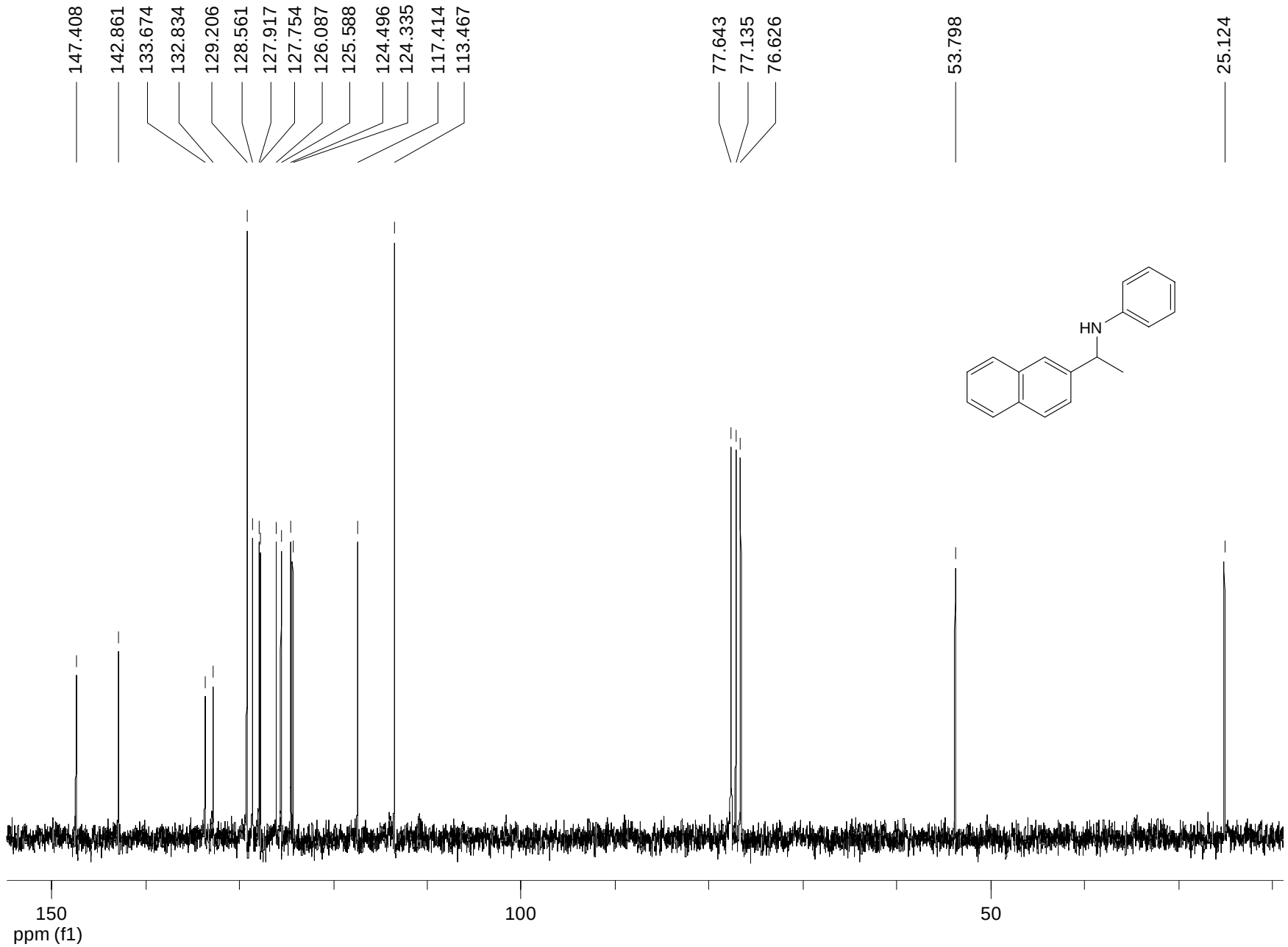


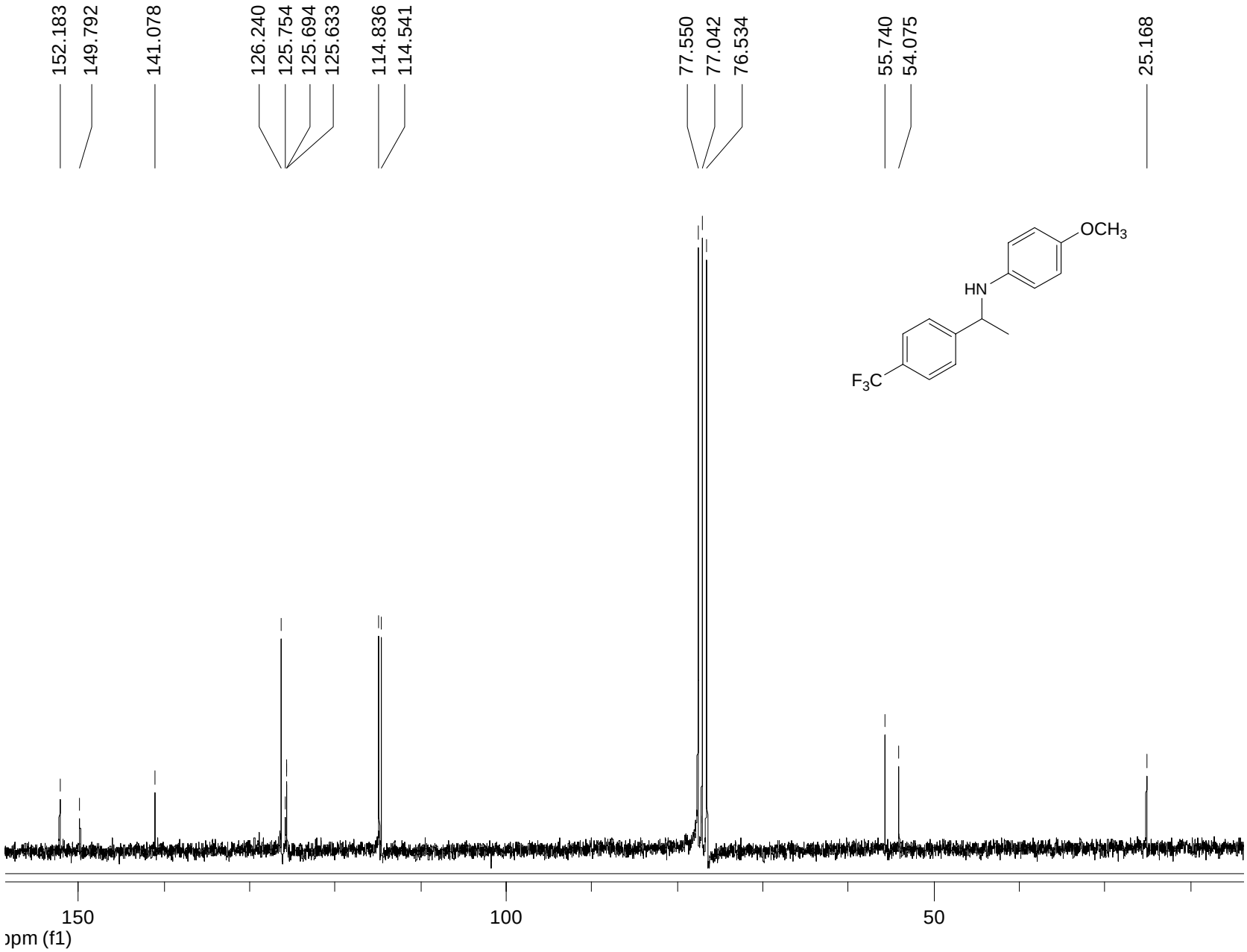
ES204 1H_16scans CDCl3 v serv 13

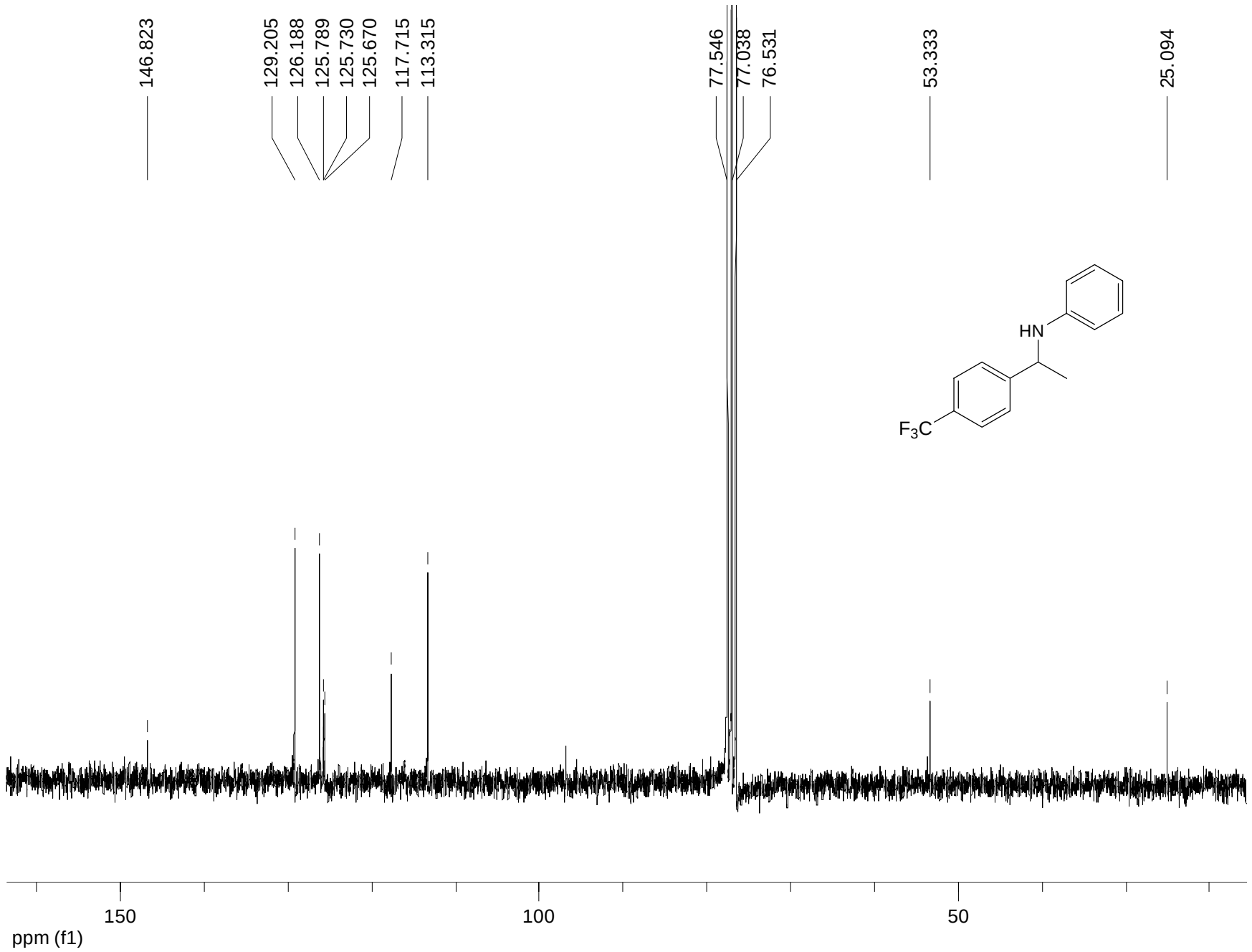


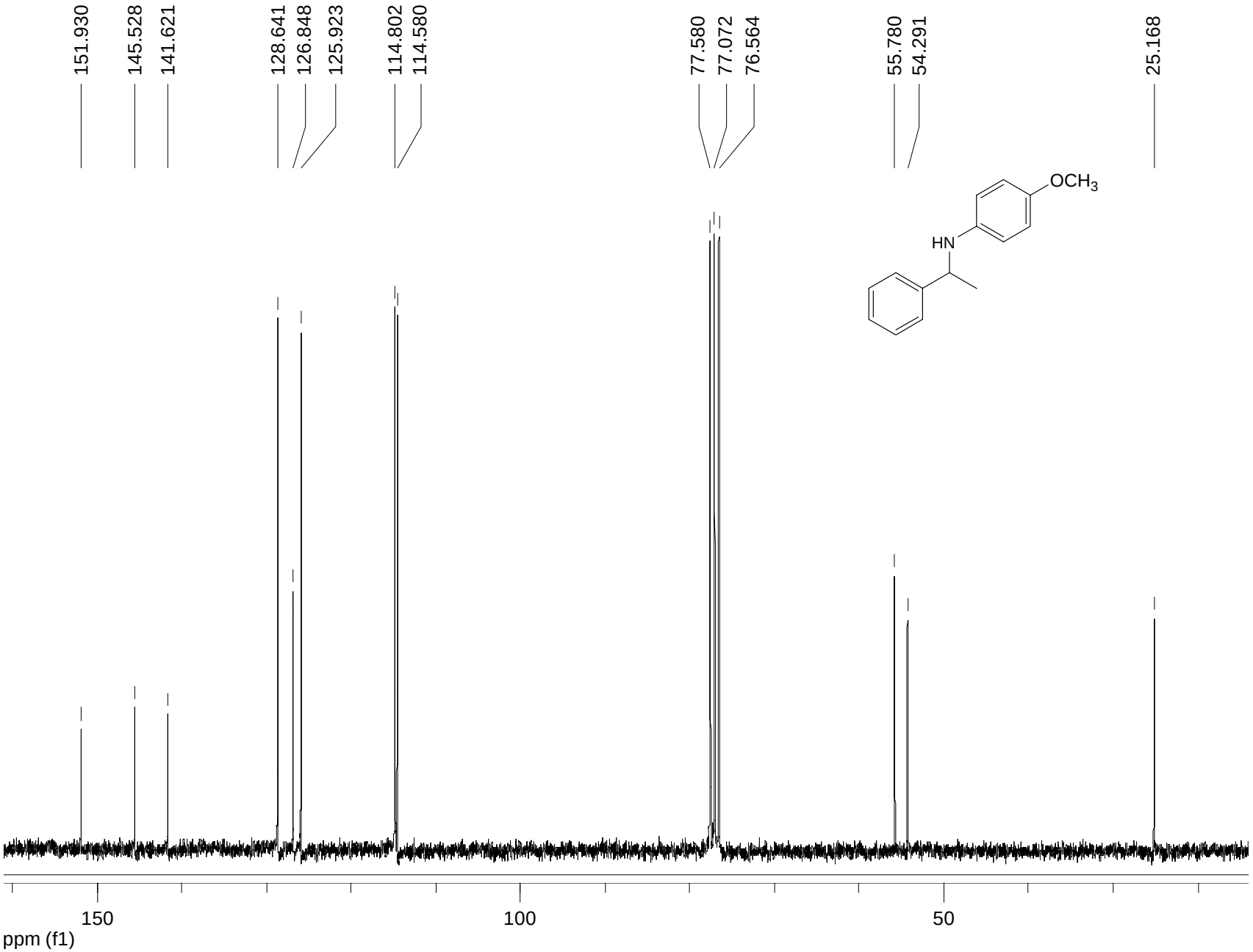

7.6307
7.5948
7.5640
7.4261
7.3964
7.3660
7.2612
7.2308
7.2006
7.1781
6.6089
6.5733
6.3835
6.3478
4.8065
4.7810
4.7552
4.7300
3.7944
3.5969
1.4223
1.3962
OCH3
HN
CF3
Integral
0.8708
0.9707
0.9503
1.2870
1.8712
1.9282
0.9509
0.9593
2.8399
3.0000
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

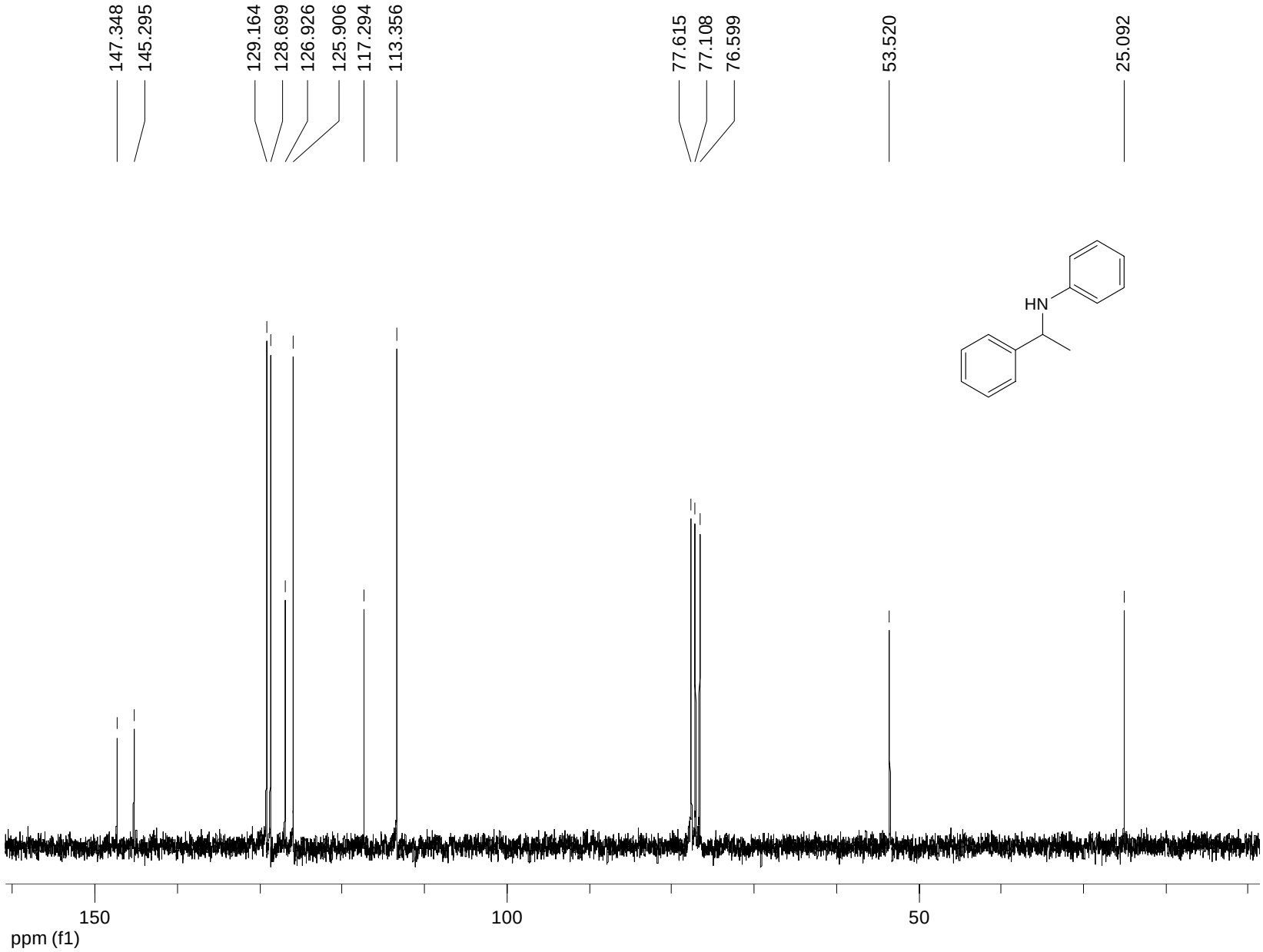

152.011
143.107
141.667
133.649
132.795
128.485
127.872
127.718
126.028
125.520
124.518
124.375
114.835
114.689
77.608
77.100
76.592
55.781
54.566
25.195
OCH3
HN
CH3
150
100
50
ppm (f1)

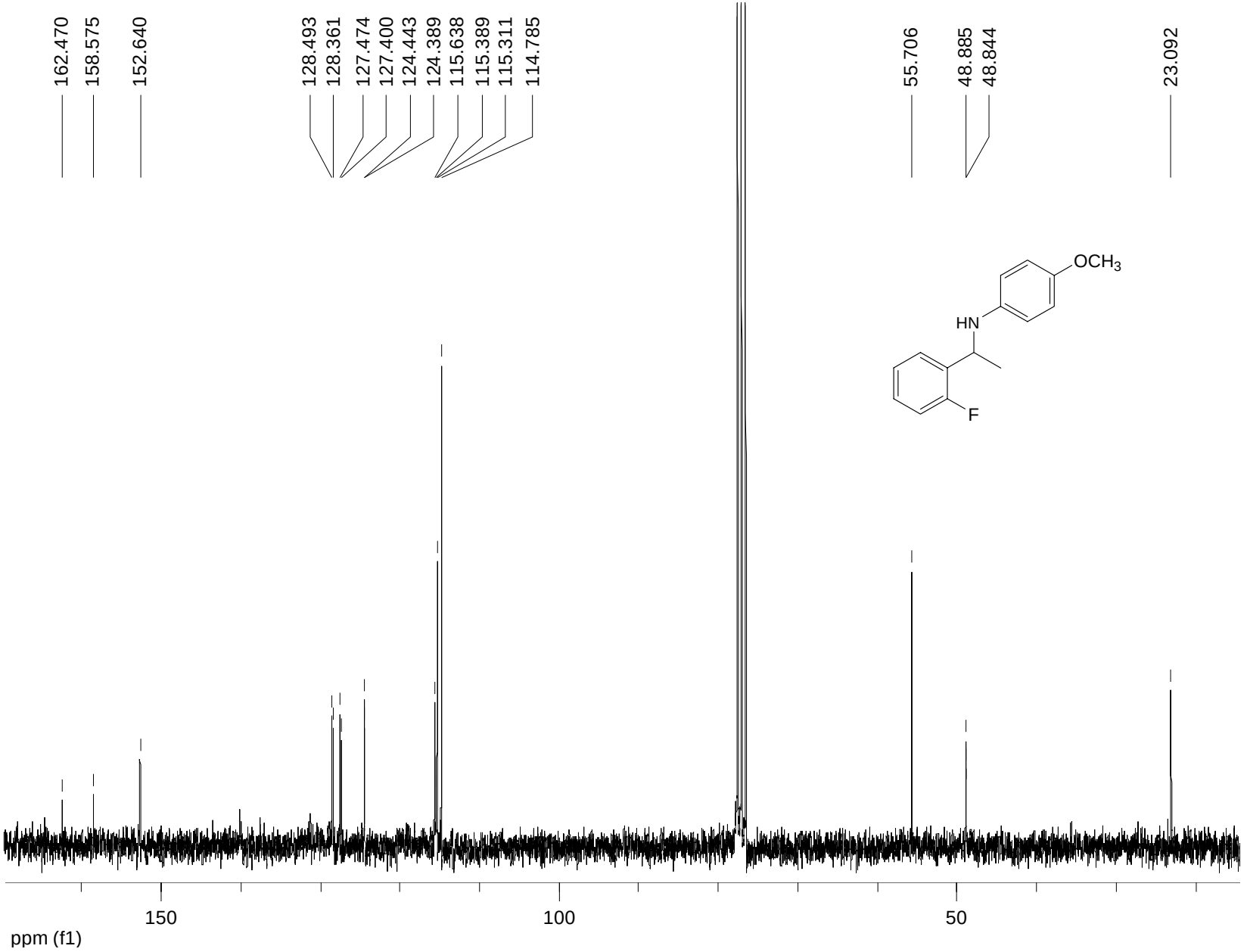

147.408
142.861
133.674
132.834
129.206
128.561
127.917
127.754
126.087
125.588
124.496
124.335
117.414
113.467
77.643
77.135
76.626
53.798
25.124
HN
150
100
50
ppm (f1)

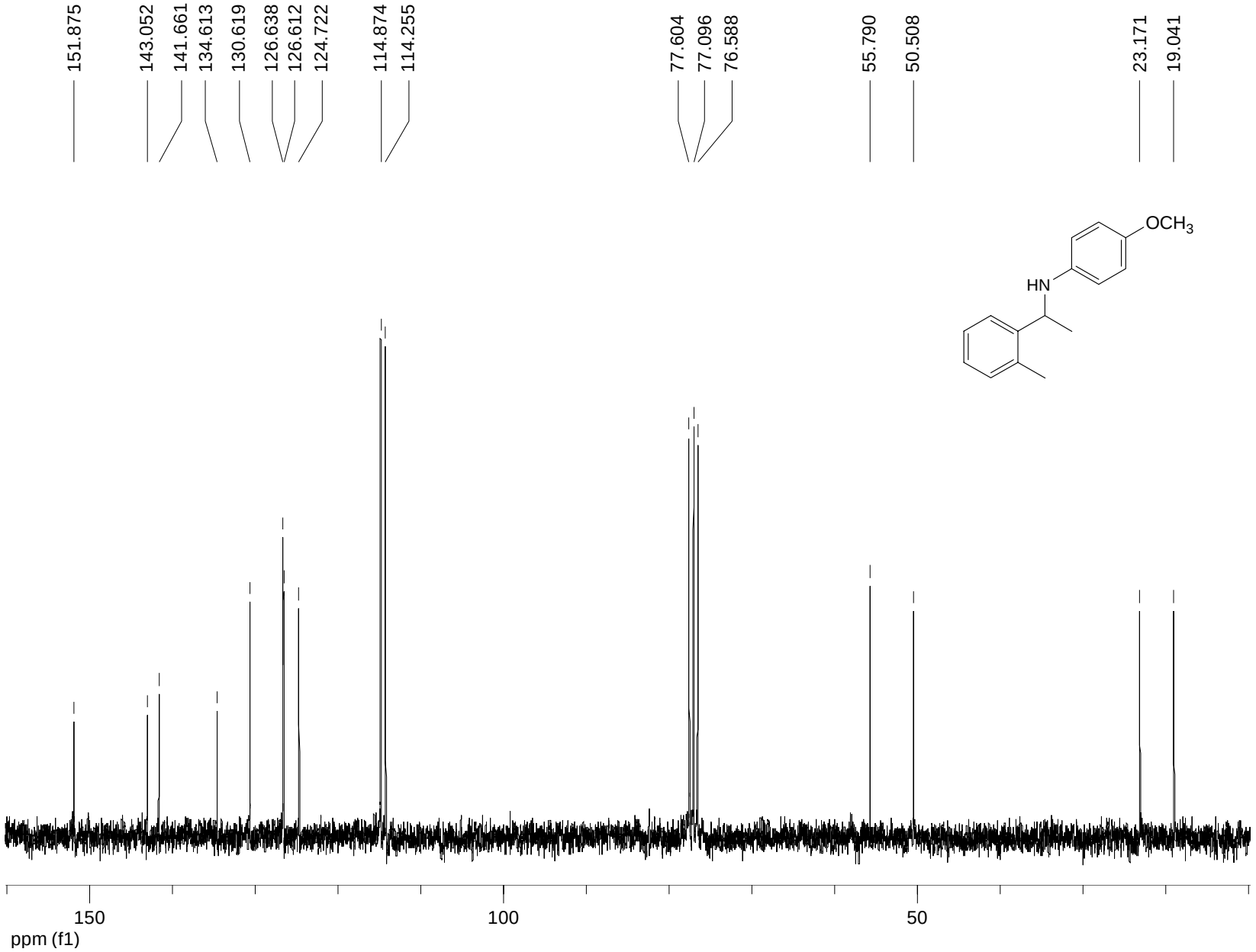

152.183
149.792
141.078
126.240
125.754
125.694
125.633
114.836
114.541
77.550
77.042
76.534
55.740
54.075
25.168
OCH3
HN
F3C
150
100
50
ppm (f1)

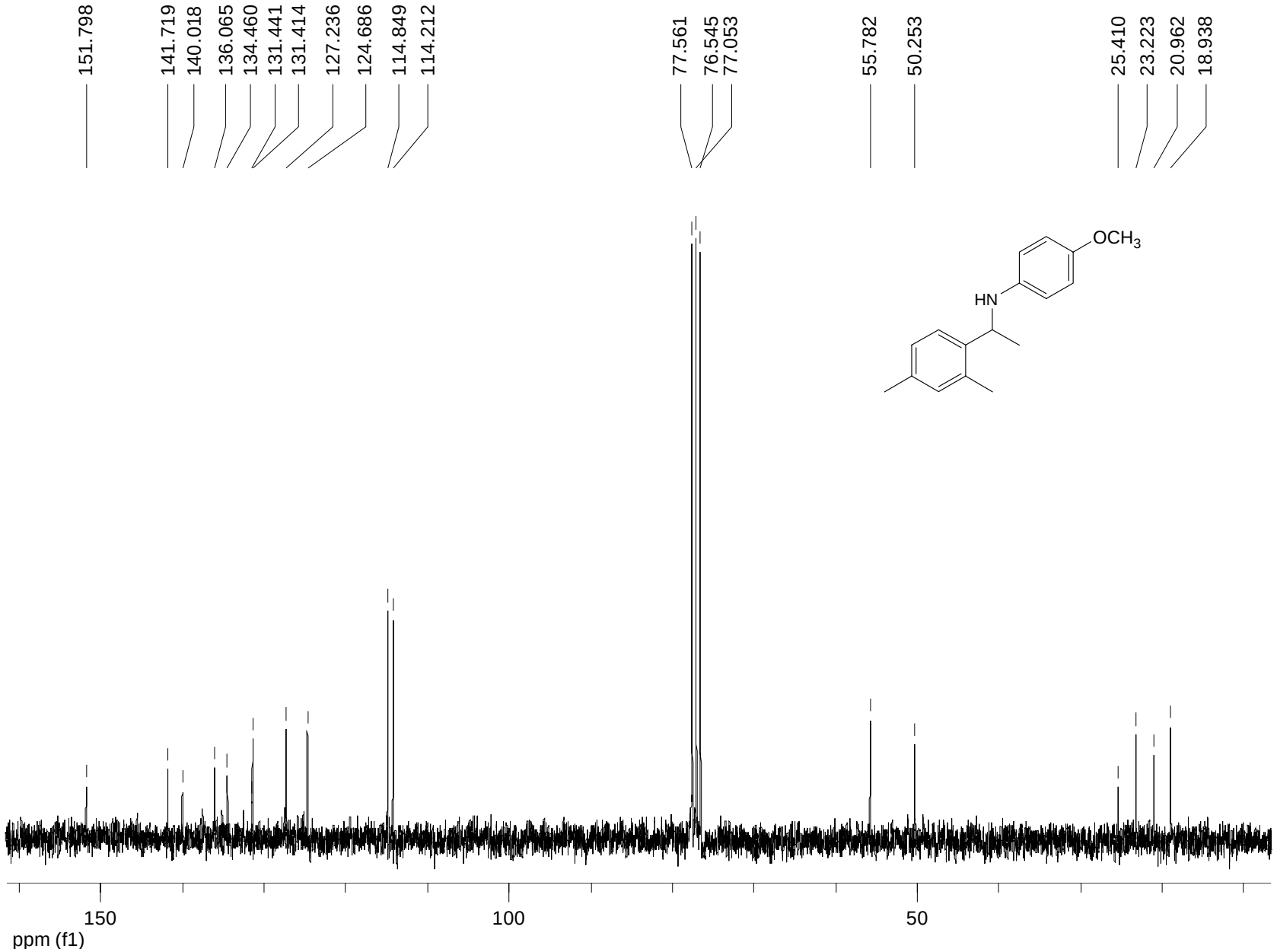

146.823
129.205
126.188
125.789
125.730
125.670
117.715
113.315
77.546
77.038
76.531
53.333
25.094
HN
F3C
150
100
50
ppm (f1)

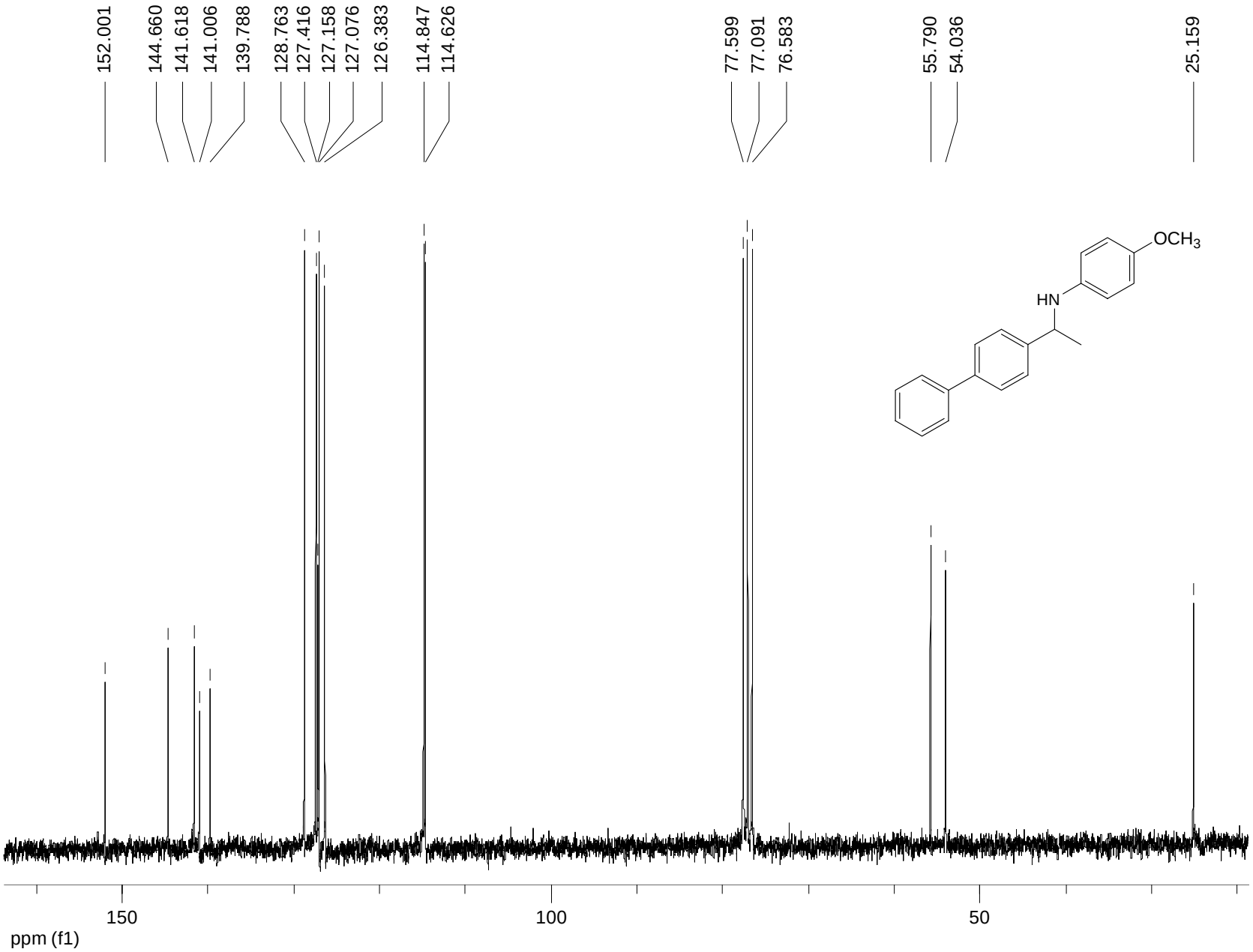

151.930
145.528
141.621
128.641
126.848
125.923
114.802
114.580
77.580
77.072
76.564
55.780
54.291
25.168
OCH3
HN
150
100
50
ppm (f1)

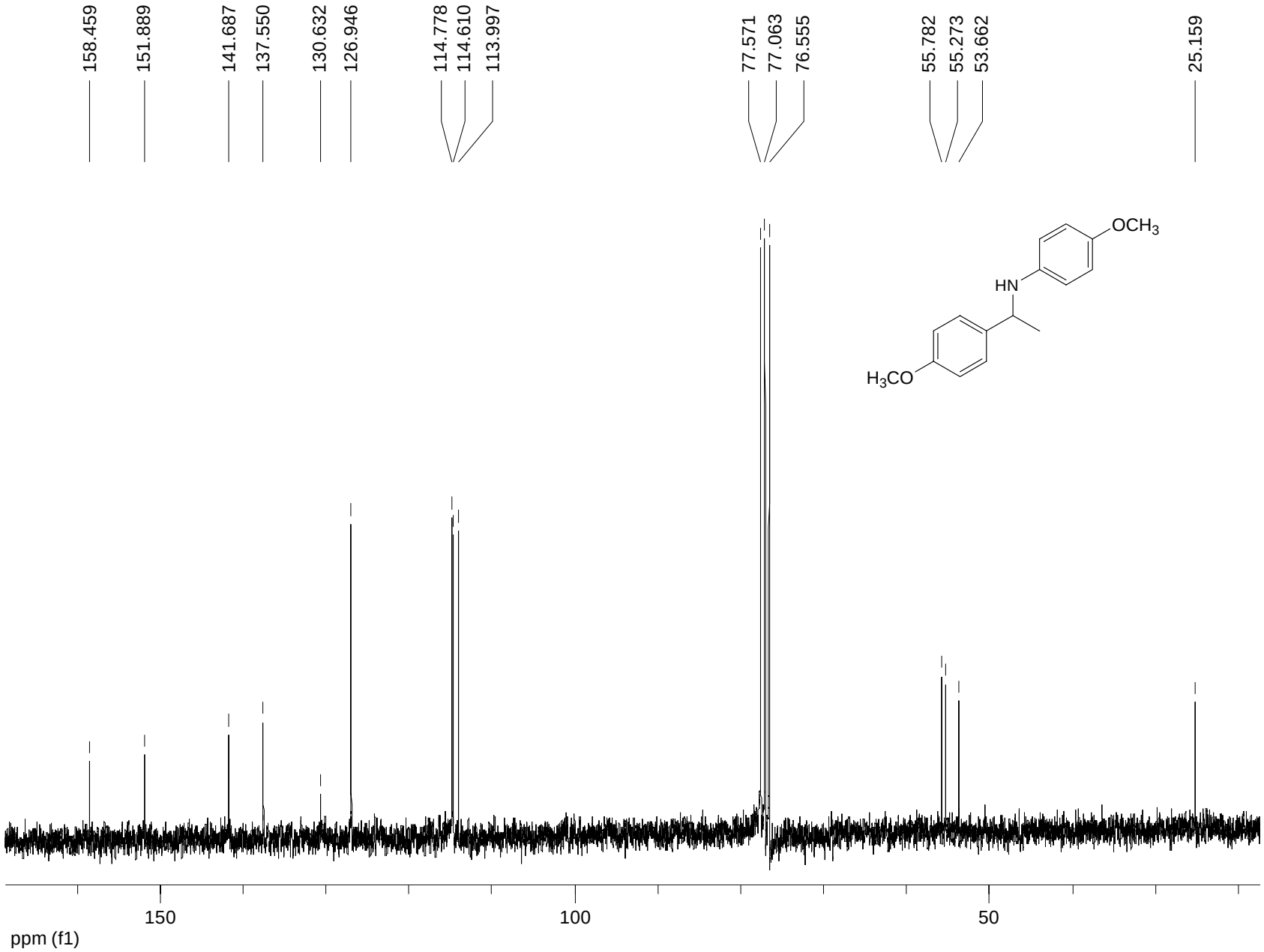

147.348
145.295
129.164
128.699
126.926
125.906
117.294
113.356
77.615
77.108
76.599
53.520
25.092
HN
150
100
50
ppm (f1)

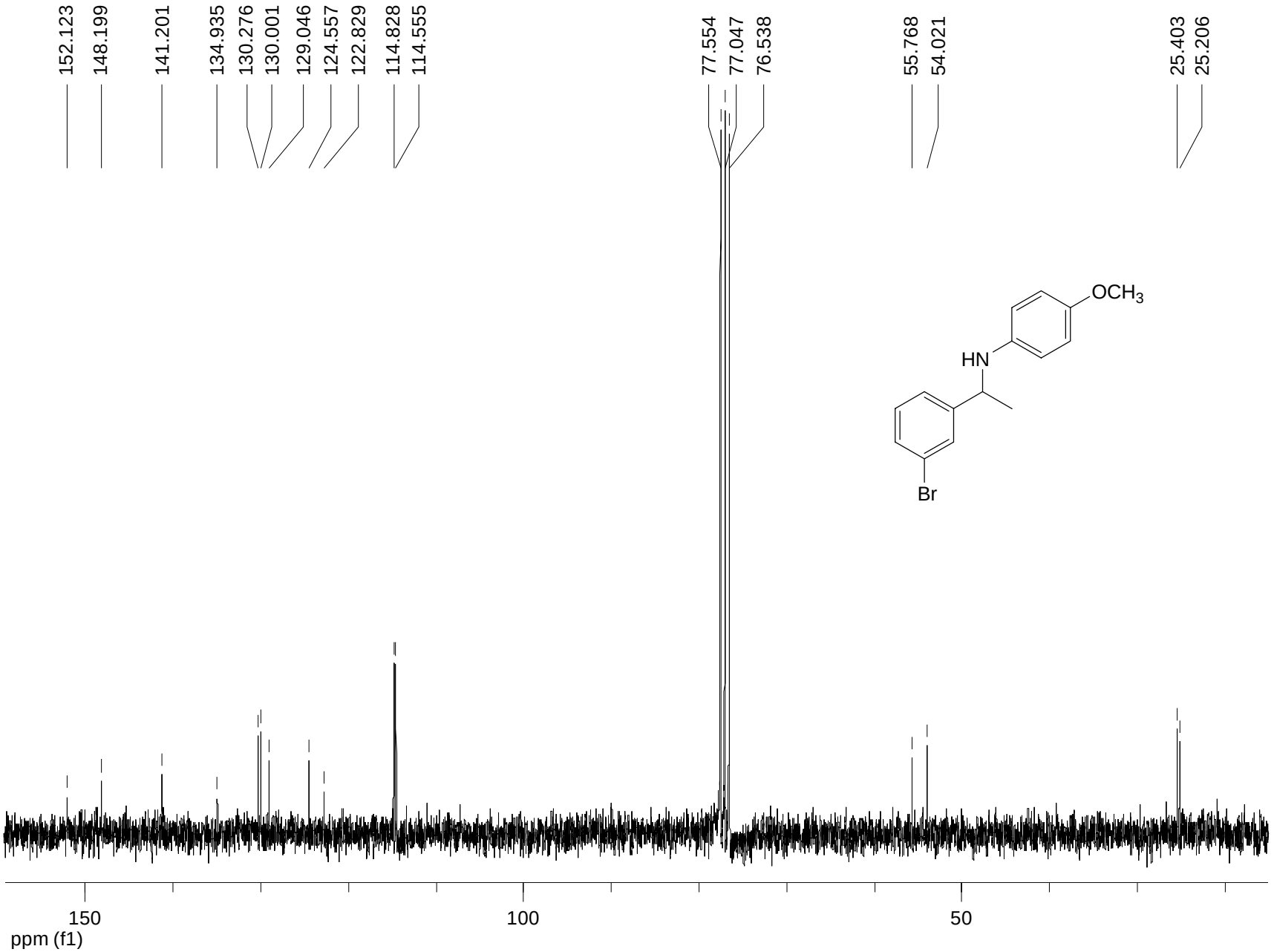

162.470
158.575
152.640
128.493
128.361
127.474
127.400
124.443
124.389
115.638
115.389
115.311
114.785
55.706
48.885
48.844
23.092
OCH3
HN
F
150
100
50
ppm (f1)

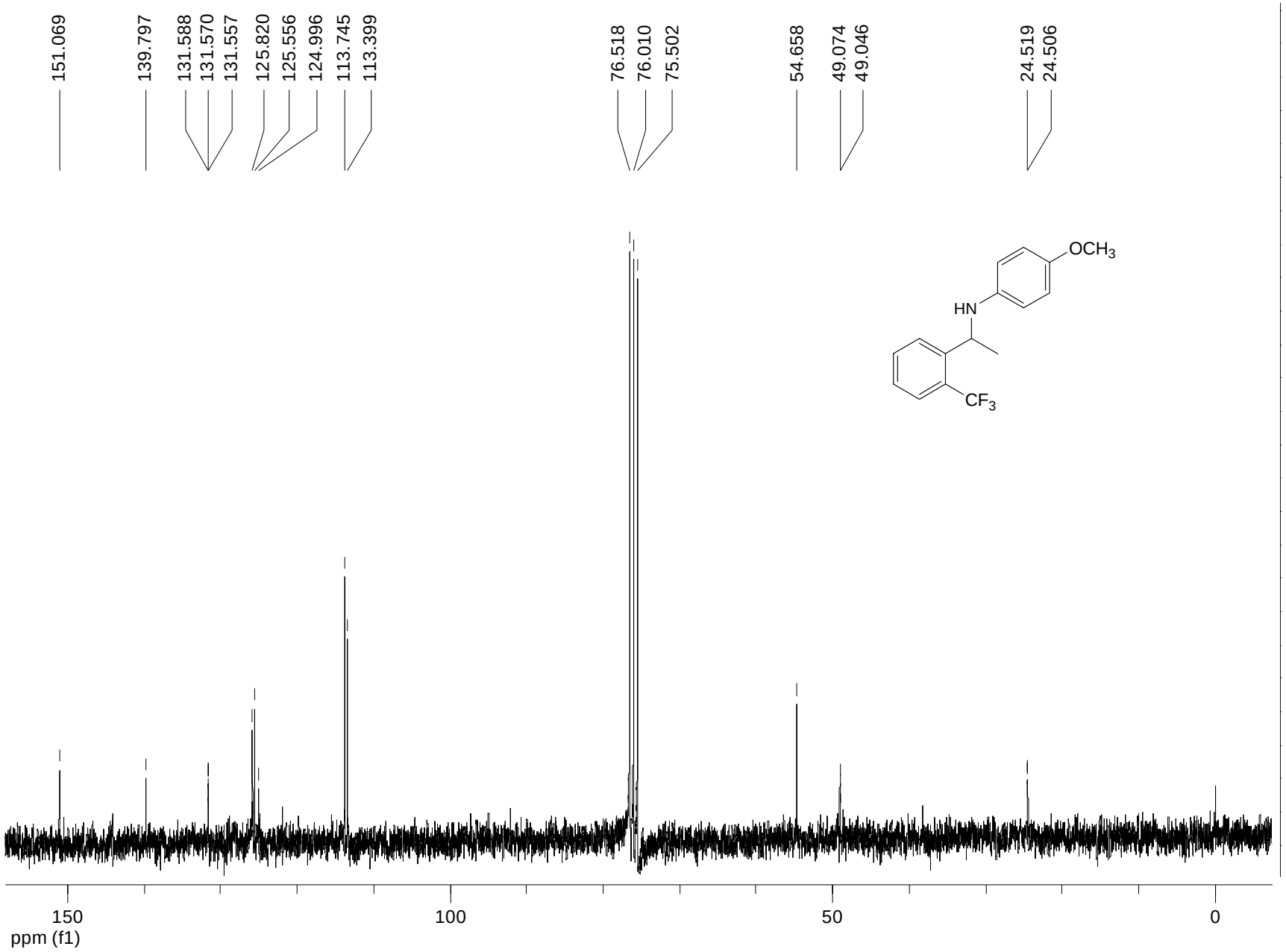

151.875
143.052
141.661
134.613
130.619
126.638
126.612
124.722
114.874
114.255
77.604
77.096
76.588
55.790
50.508
23.171
19.041
OCH3
HN
150
100
50
ppm (f1)


151.798
141.719
140.018
136.065
134.460
131.441
131.414
127.236
124.686
114.849
114.212
77.561
76.545
77.053
55.782
50.253
25.410
23.223
20.962
18.938
OCH3
HN
150
100
50
ppm (f1)


152.001
144.660
141.618
141.006
139.788
128.763
127.416
127.158
127.076
126.383
114.847
114.626
77.599
77.091
76.583
55.790
54.036
25.159
OCH3
HN
150
100
50
ppm (f1)


158.459
151.889
141.687
137.550
130.632
126.946
114.778
114.610
113.997
77.571
77.063
76.555
55.782
55.273
53.662
25.159
OCH3
HN
H3CO
150
100
50
ppm (f1)

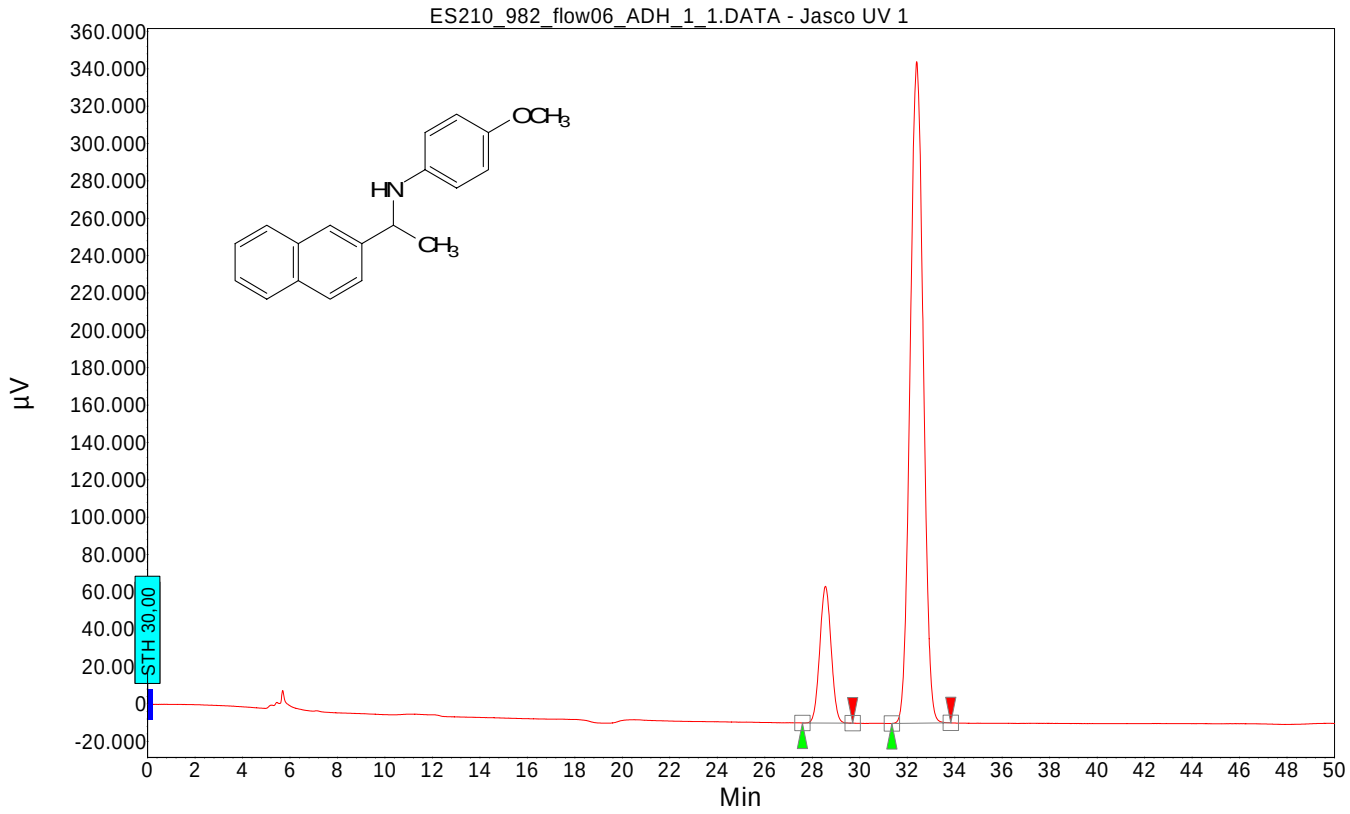

152.123
148.199
141.201
134.935
130.276
130.001
129.046
124.557
122.829
114.828
114.555
77.554
77.047
76.538
55.768
54.021
25.403
25.206
OCH3
HN
Br
150
100
50
ppm (f1)


151.069
139.797
131.588
131.570
131.557
125.820
125.556
124.996
113.745
113.399
76.518
76.010
75.502
54.658
49.074
49.046
24.519
24.506
OCH3
HN
CF3
150
100
50
0
ppm (f1)

Chromatogram : ES210_982_flow06_ADH_1_1

Column: AD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



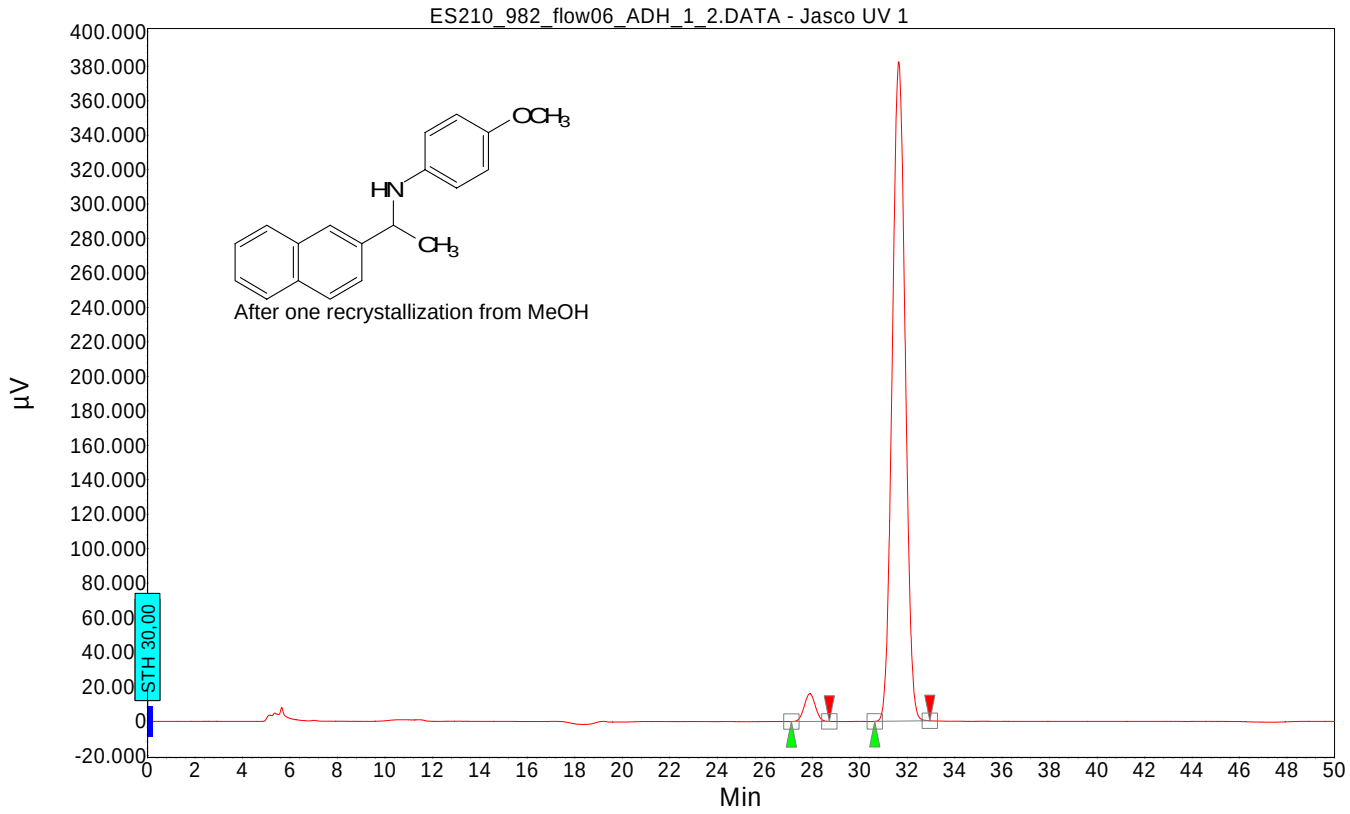
Peak Results

Index	Start	Time	End	Height	Area %
	[Min]	[Min]	[Min]	[µV]	[%]
1	27,583	28,550	29,700	73221,8	15,183
2	31,353	32,400	33,833	354115,9	84,817
Total				427337,7	100,000

Chromatogram : ES210_982_flow06_ADH_1_2

Column: AD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



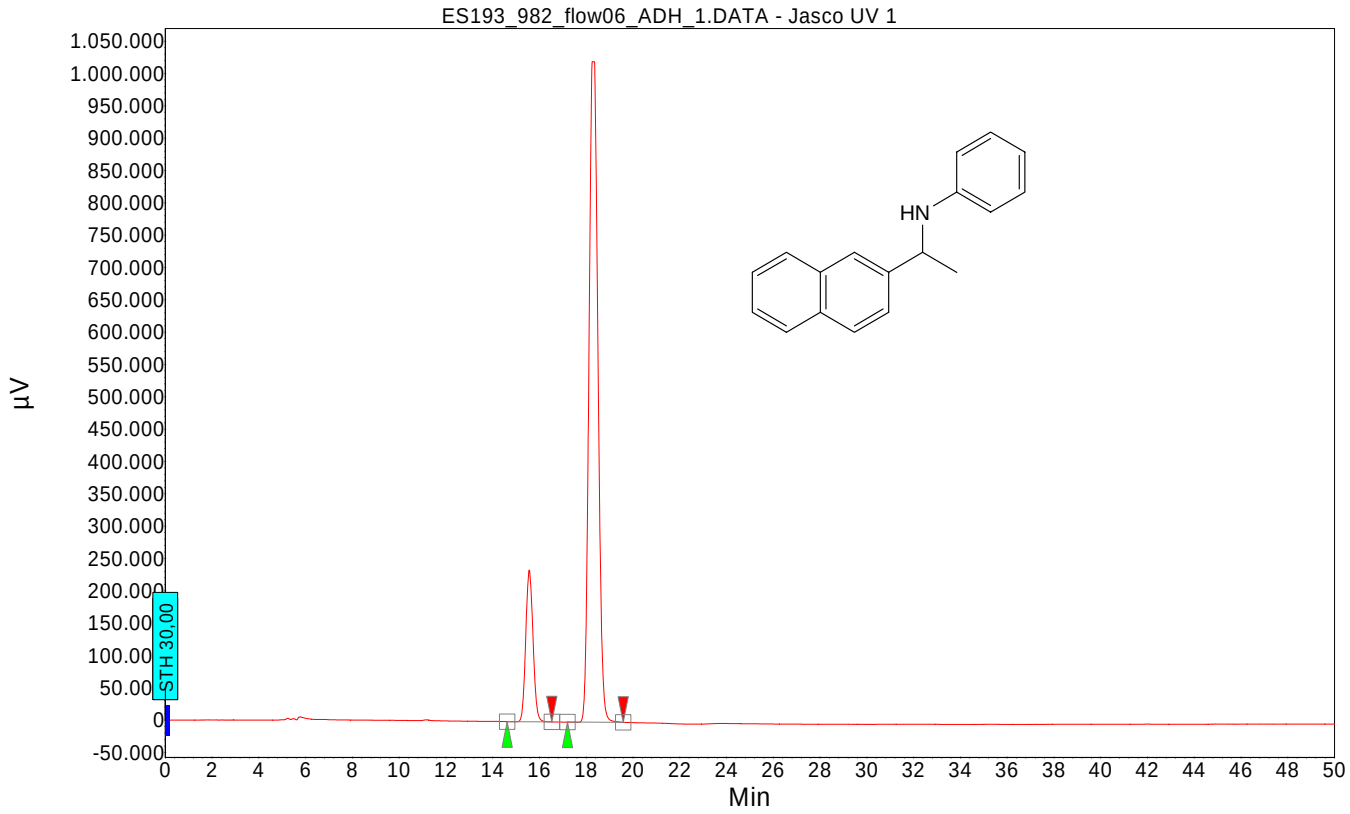
Peak Results

Index	Start	Time	End	Height	Area %
	[Min]	[Min]	[Min]	[µV]	[%]
1	27,118	27,892	28,719	16172,5	3,471
2	30,630	31,642	32,955	382827,3	96,529
Total				398999,8	100,000

Chromatogram : ES193_982_flow06_ADH_1

Column: AD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



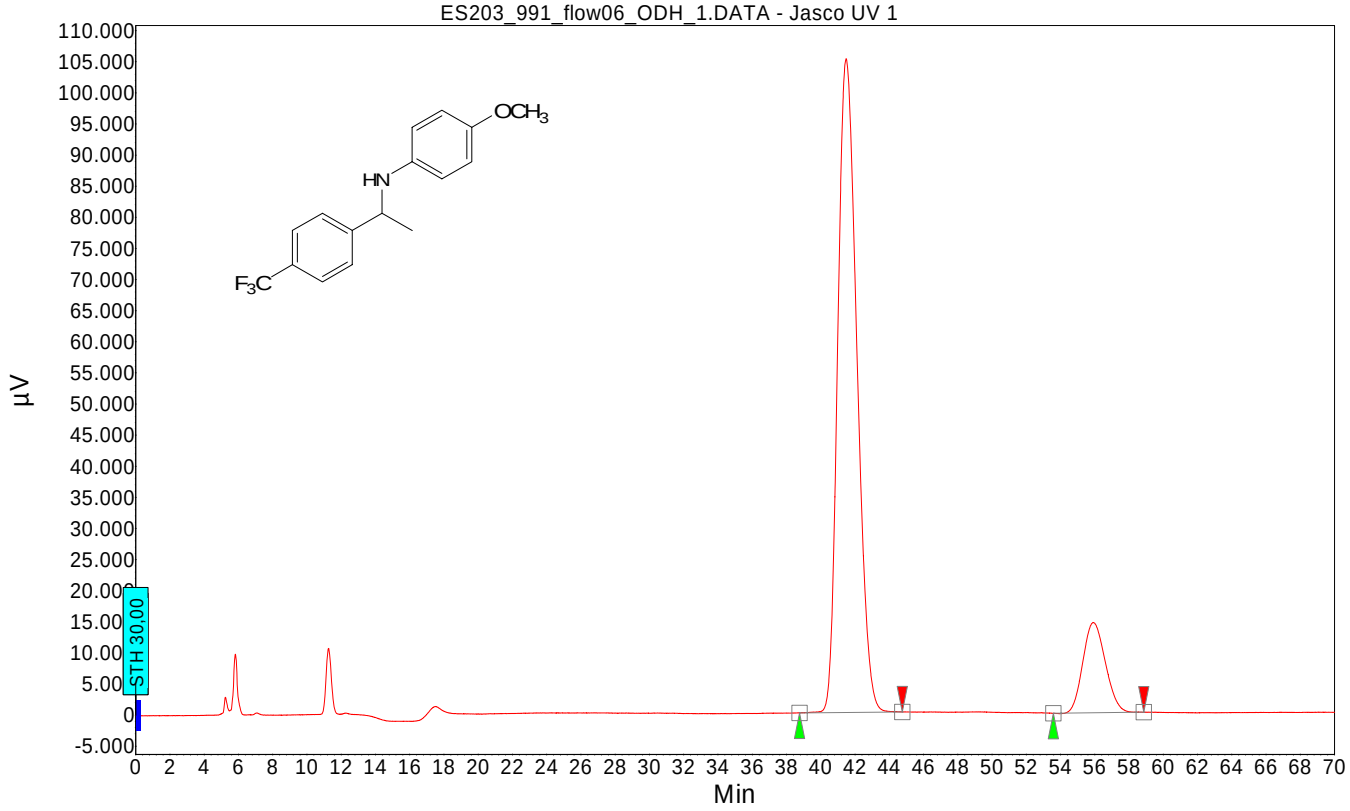
Peak Results

Index	Start	Time	End	Height	Area %
	[Min]	[Min]	[Min]	[µV]	[%]
1	14,618	15,558	16,529	234566,8	15,741
2	17,200	18,342	19,576	1021451,2	84,259
Total				1256018,0	100,000

Chromatogram : ES203_991_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 99/1 (0.6 ml/min)



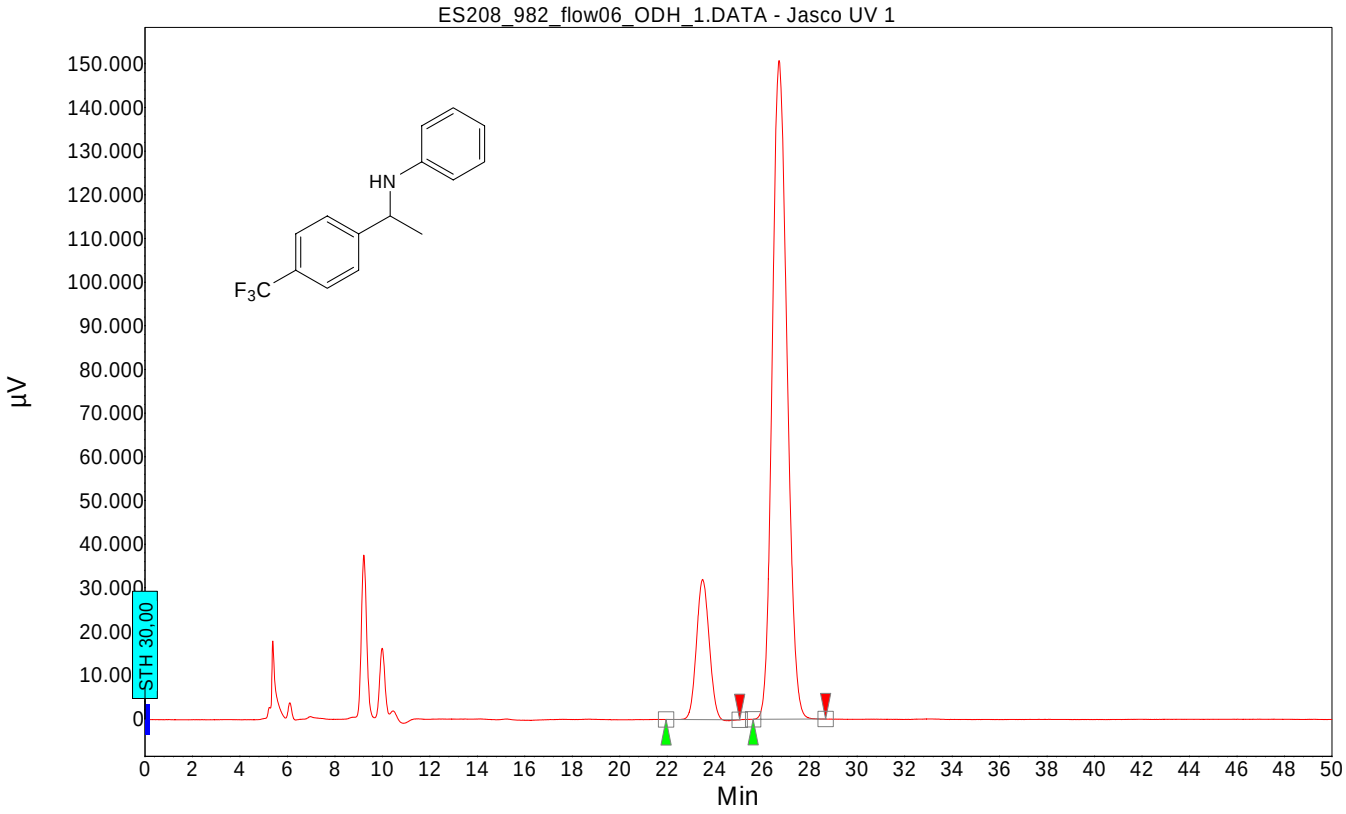
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	38,760	41,483	44,762	86,037
2	53,585	55,933	58,864	13,963
Total				100,000

Chromatogram : ES208_982_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



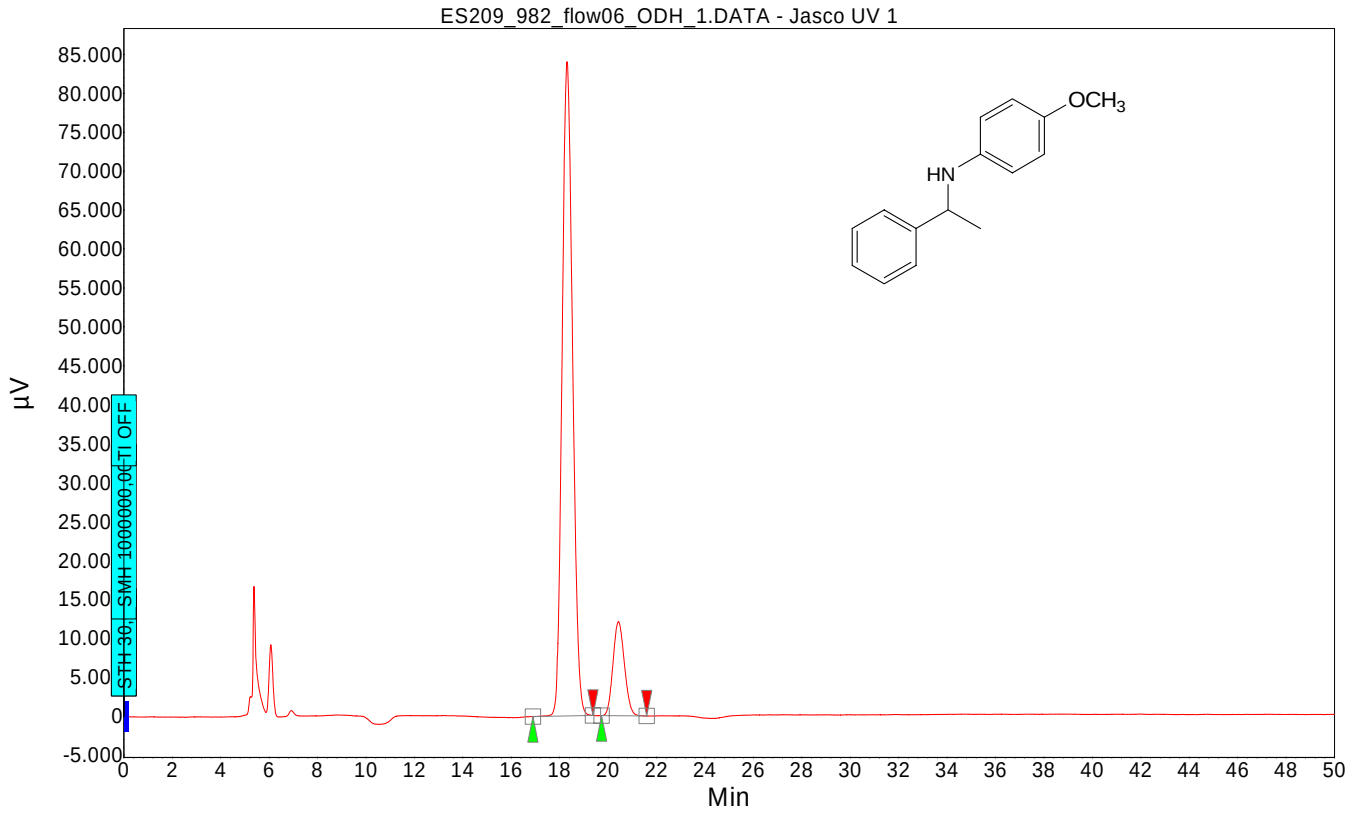
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	21,952	23,492	25,052	15,404
2	25,620	26,708	28,667	84,596
Total				100,000

Chromatogram : ES209_982_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



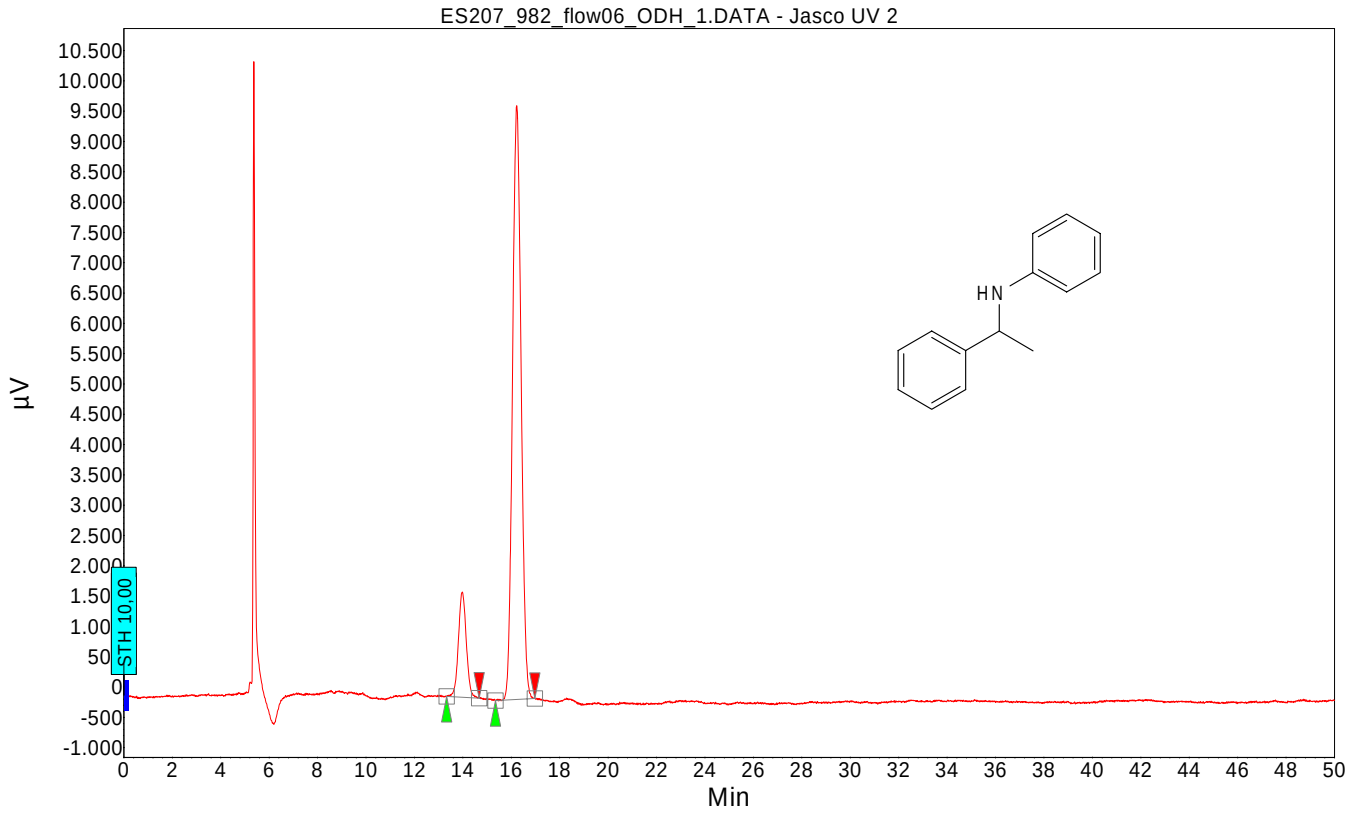
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,890	18,300	19,370	86,523
2	19,731	20,425	21,591	13,477
Total				100,000

Chromatogram : ES207_982_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



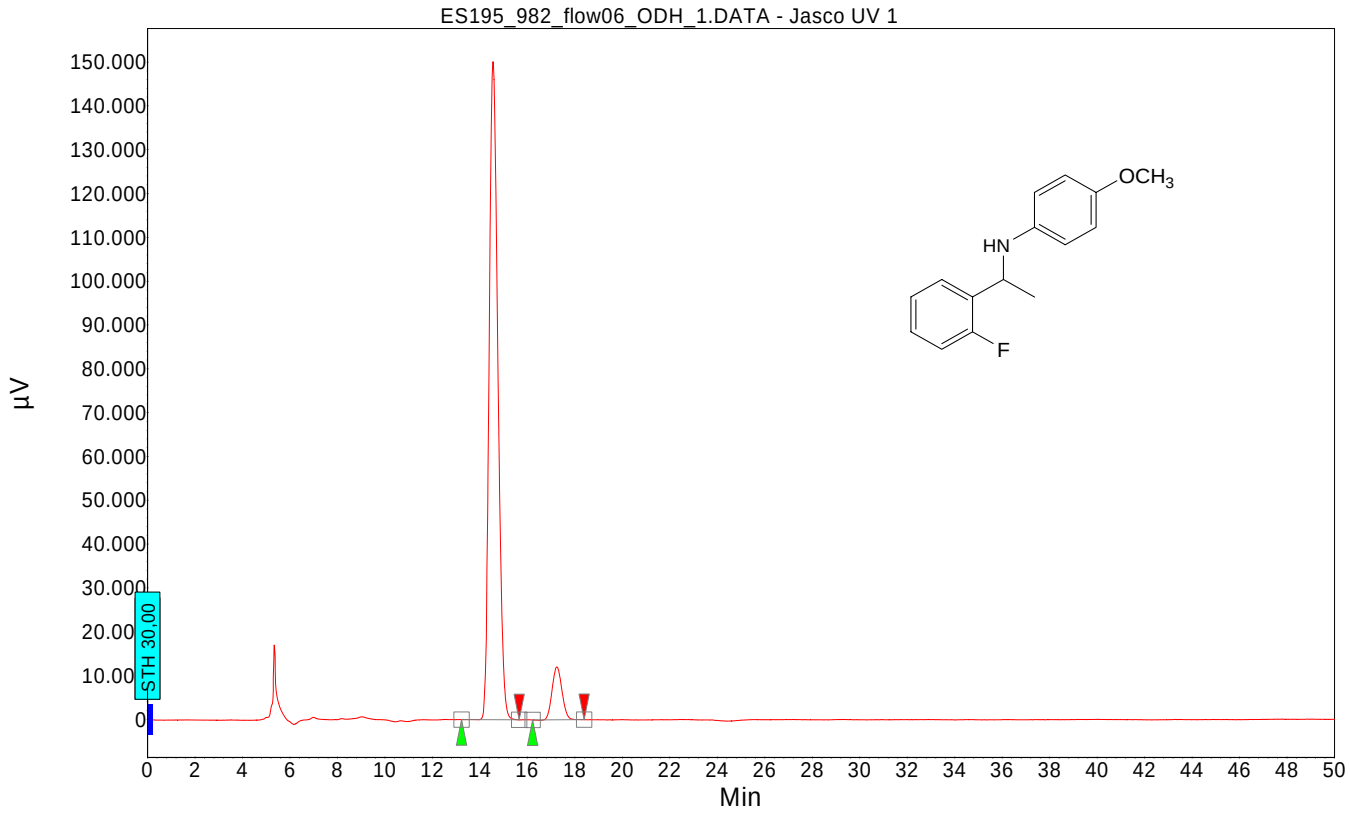
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,328	13,975	14,676	14,008
2	15,346	16,225	16,975	85,992
Total				100,000

Chromatogram : ES195_982_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



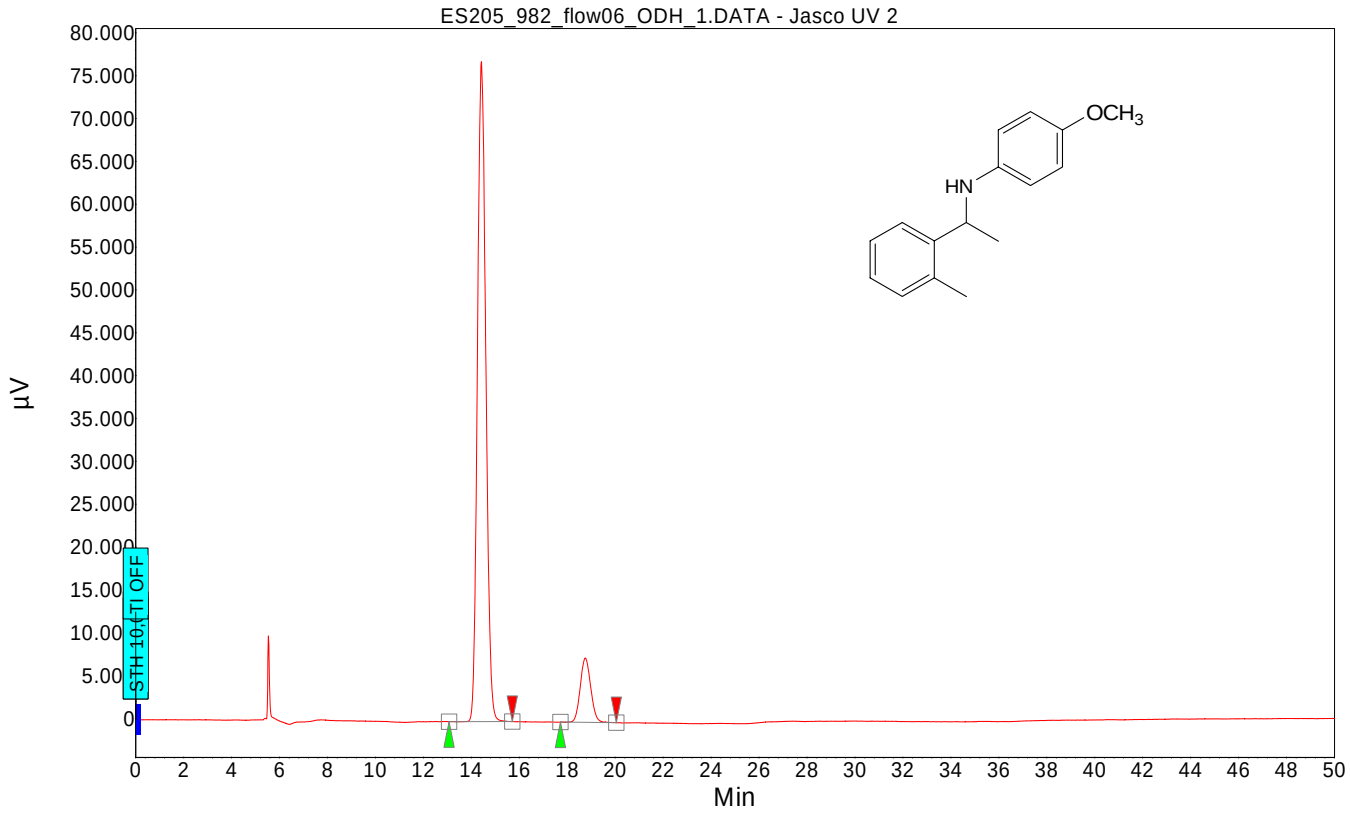
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,223	14,550	15,651	91,708
2	16,219	17,242	18,388	8,292
Total				100,000

Chromatogram : ES205_982_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



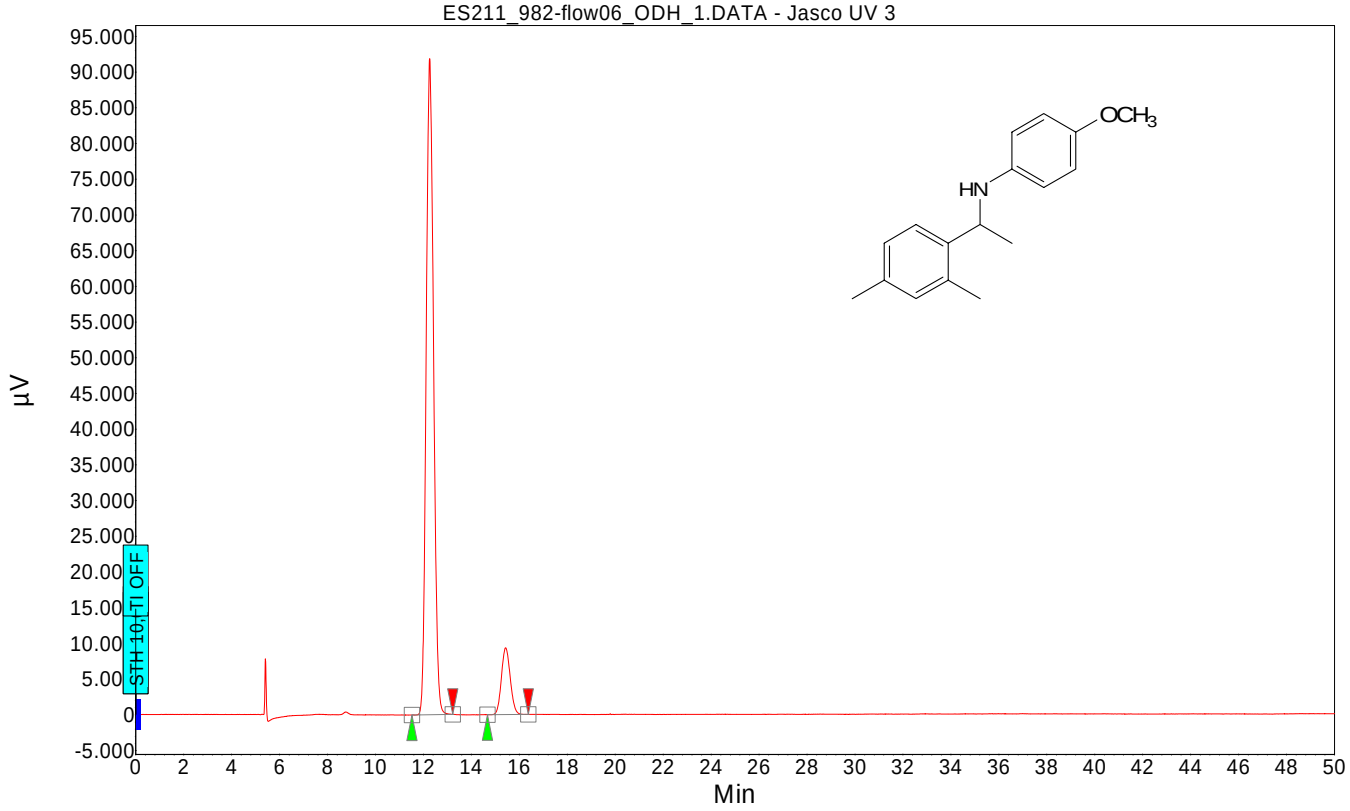
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,068	14,417	15,702	89,152
2	17,717	18,750	20,041	10,848
Total				100,000

Chromatogram : ES211_982-flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



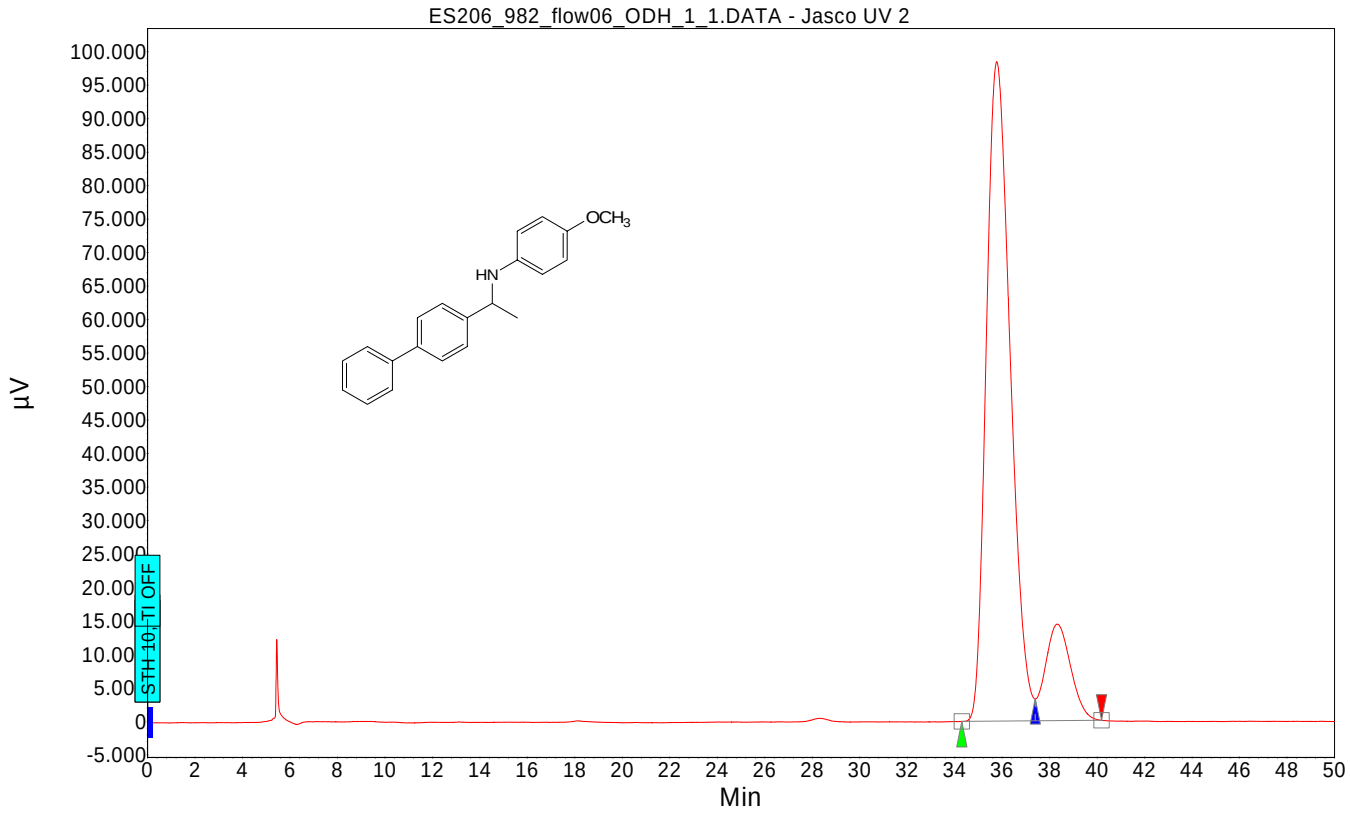
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	11,519	12,258	13,223	89,091
2	14,669	15,425	16,374	10,909
Total				100,000

Chromatogram : ES206_982_flow06_ODH_1_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



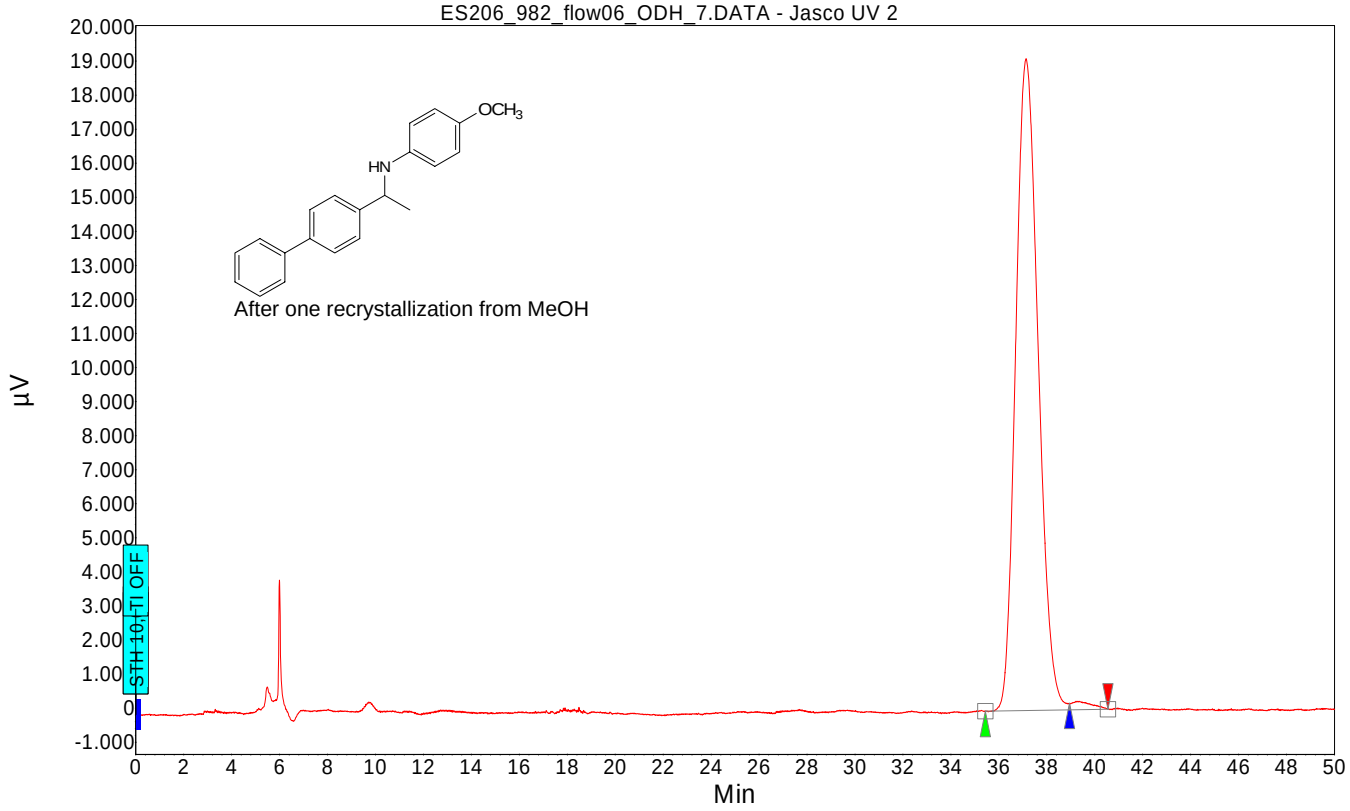
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	34,298	35,767	37,397	86,632
2	37,397	38,317	40,186	13,368
Total				100,000

Chromatogram : ES206_982_flow06_ODH_7

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



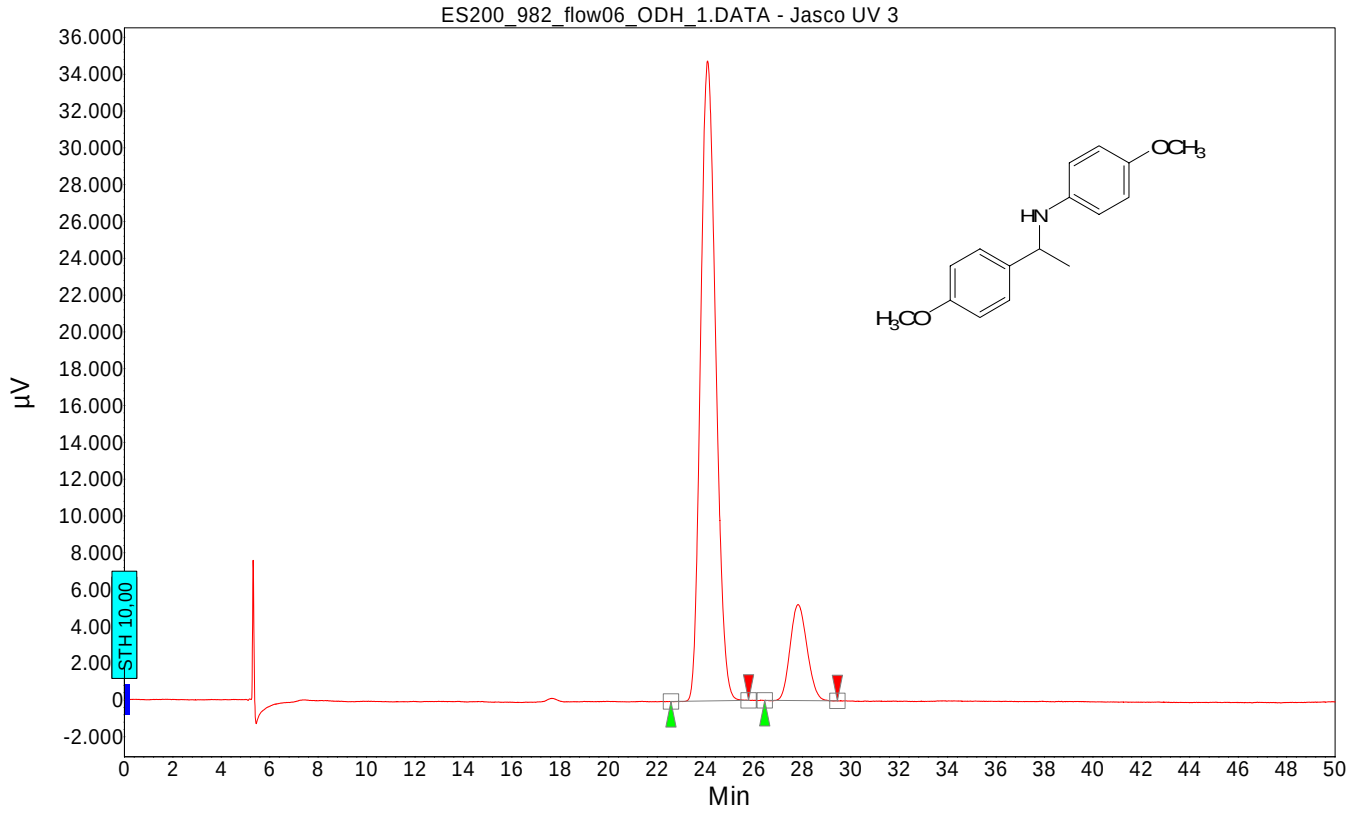
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	35,434	37,133	38,946	98,861
2	38,946	39,292	40,548	1,139
Total				100,000

Chromatogram : ES200_982_flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)



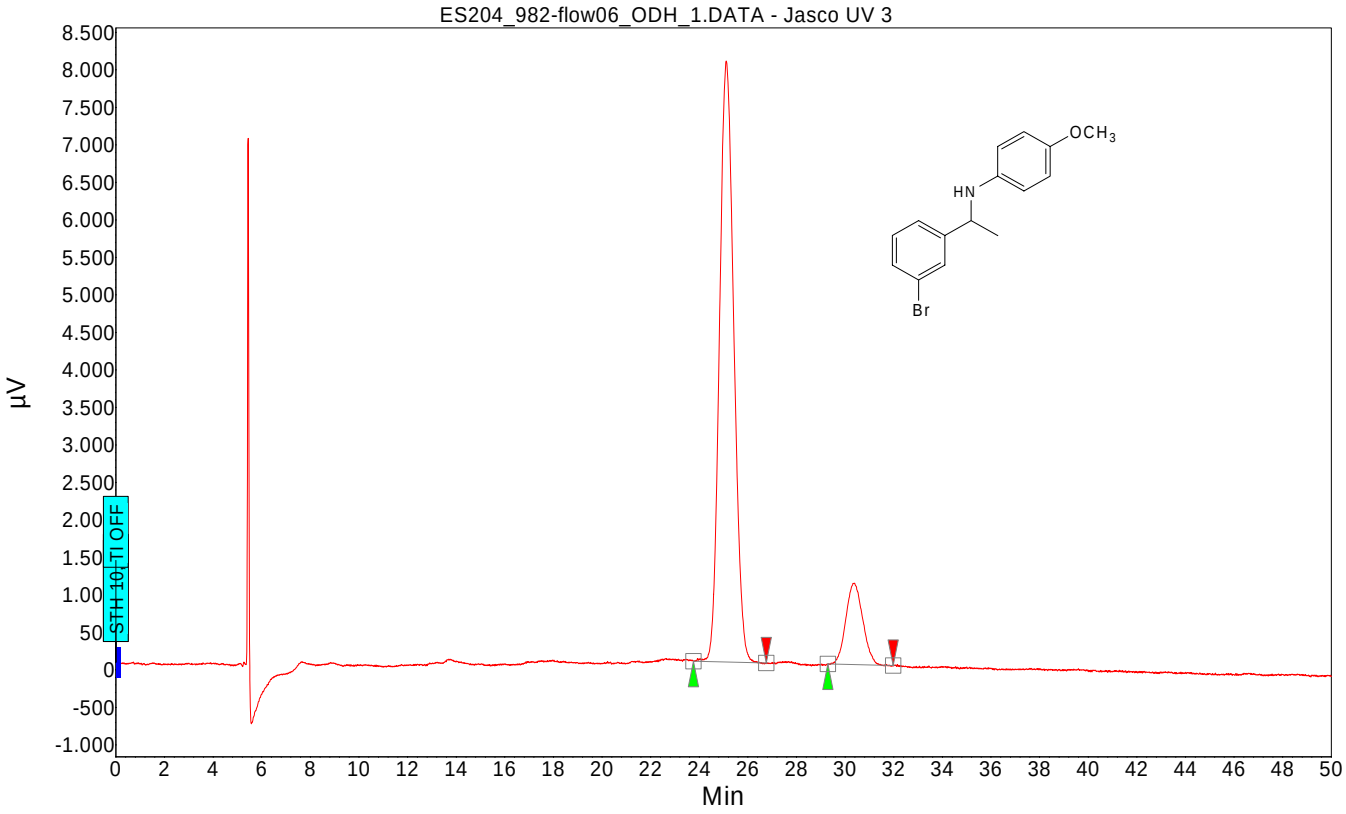
Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	22,572	24,083	25,775	85,658
2	26,446	27,817	29,442	14,342
Total				100,000

Chromatogram : ES204_982-flow06_ODH_1

Column: OD-H

n-hexane/ 2-propanol 98/2 (0.6 ml/min)

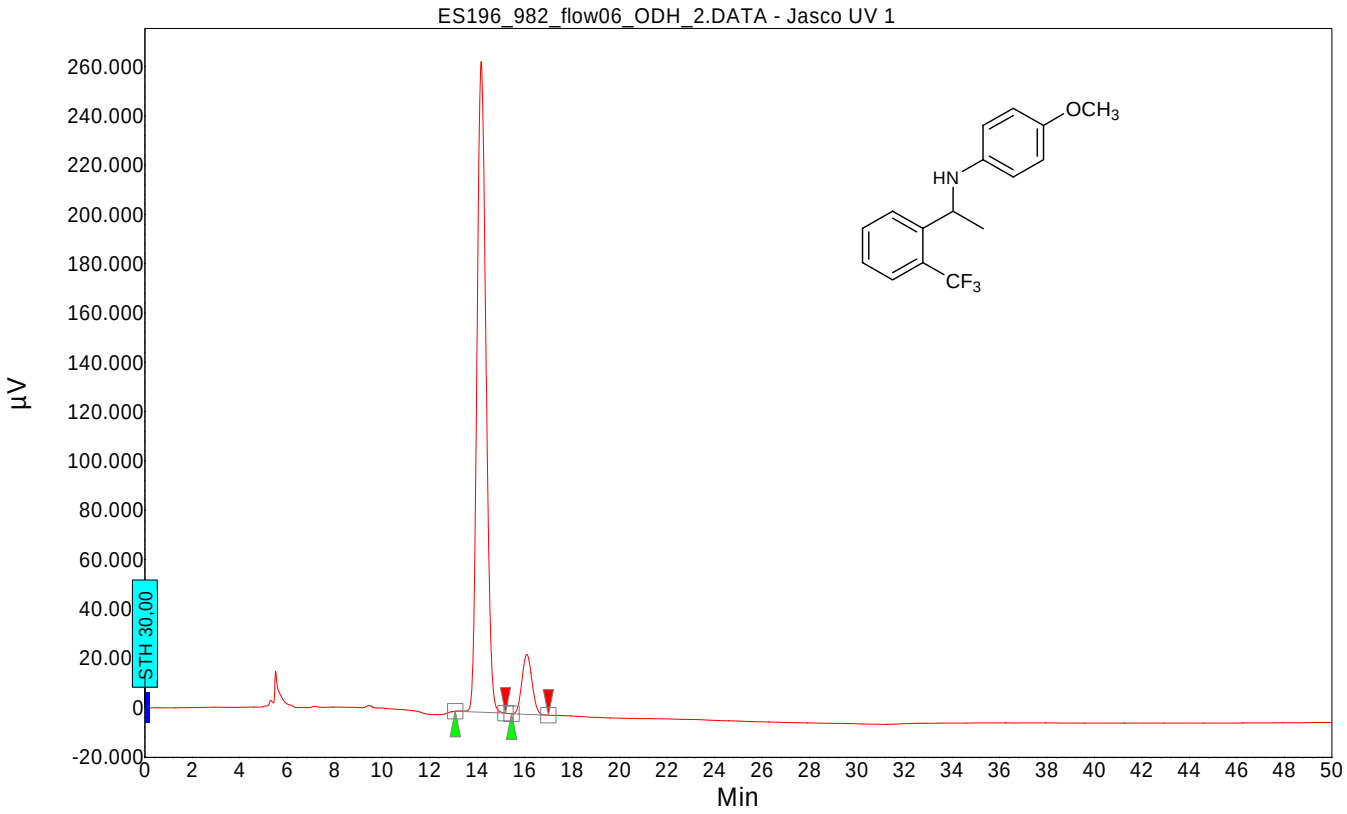


Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	23,760	25,108	26,756	86,252
2	29,287	30,358	31,973	13,748
Total				100,000

Chromatogram : ES196_982_flow06_ODH_2

Column: OD-H
n-hexane/ 2-propanol 98/2 (0.6 ml/min)



Peak Results

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,068	14,167	15,186	90,677
2	15,444	16,092	16,994	9,323
Total				100,000