



Supporting Information

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Remarkable Low Catalyst Loading in Brønsted Acid Catalyzed Transfer Hydrogenations - Enantioselective Reductions of Benzoxazines, Benzthiazines and Benzoxazinones

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Supporting information

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

¹H-NMR and ¹³C-NMR were recorded on a Bruker AM 250 spectrometer in CDCl₃. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated br s (broadened singlet), s (singlet), d (doublet), t (triplet), m (multiplet)); coupling constants (J) are in Hertz (Hz). Mass spectra (MS-ESI) were conducted on Micromass/ Waters Qtof Ultima 3 instrument. IR spectra were recorded on a Jasco FT/IR-420 spectrometer and are reported in terms of frequency of absorption (cm⁻¹). Optical rotations were measured on a Perkin Elmer 241 polarimeter. The enantiomeric excesses were determined by HPLC analysis using a chiral stationary phase column (column, Daicel Co. CHIRALCEL OD-H; eluent: hexane/2-propanol). The chiral HPLC method were calibrated with the corresponding racemic mixtures. Chemical yields refer to pure isolated substances. The yields and enantiomeric excesses are given in table.

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General procedure for transfer hydrogenation of 3-substituted-2*H*-1,4-benzoxazines and 3-substituted-2*H*-1,4-benzthiazines: The substrate, catalyst (0.01-1 mol%) and Hantzsch dihydropyridine (1.25 equiv.) were suspended in CHCl₃ in a screw-capped vial. The resulting mixture was allowed to stir at RT for 12 h. The solvent was removed under reduced pressure and purification of the crude product by column chromatography on silica gel (ethyl acetate/hexane) afforded the pure product.

General procedure for transfer hydrogenation of 3-substituted-2*H*-1,4-benzoxazin-2-ones: The substrate (20 mg), catalyst (0.1-1 mol%) and Hantzsch dihydropyridine (1.25 equiv.) were suspended in benzene or CHCl₃ (2 mL) in a screw-capped vial. The resulting mixture was allowed to stir at 60°C or RT for 15-24 h. The solvent was removed under reduced pressure and purification of the crude product by column chromatography on silica gel (ethyl acetate/hexane) afforded the pure product.

General procedure for preparation of *N*-benzylamides from 3-substituted-3,4-dihydro-2*H*-1,4-benzoxazin-2-ones: Pyridin-2-ol (3 equivalents) benzylamine (5 equivalents) were added to a solution of 3-substituted-3,4-dihydro-2*H*-1,4-benzoxazin-2-one in THF. The mixture was stirred at ambient temperature for 12 h, diluted with water and extracted with EtOAc. The extracts were dried and evaporated. Purification of the crude product by column chromatography on silica gel (ethyl acetate/hexane) afforded the pure product.

Physical data:

3-phenyl-3,4-dihydro-2*H*-1,4-benzoxazine

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =7.36-7.21 (m, 5H), 6.82-6.69 (m, 2H), 6.67-6.57 (m, 2H), 4.43 (dd, $J(\text{H,H})$ =2.9, 8.6 Hz, 1H), 4.21 (dd, $J(\text{H,H})$ =2.2, 10.6 Hz, 1H), 3.92 ppm (dd, $J(\text{H,H})$ =8.6, 10.6 Hz, 2H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =143.63, 139.02, 133.65, 128.85, 128.39, 127.24, 121.51, 119.14, 116.64, 115.54, 70.92, 54.27 ppm; IR (KBr): $\tilde{\nu}$ =3359, 3059, 3030, 2976, 2919, 2870, 1608, 1591, 1499, 1279, 1210, 744, 700 cm^{-1} ; MS-ESI: m/z : 211.9 $[\text{M}]^+$ ($\text{C}_{14}\text{H}_{13}\text{NO}$); $[\alpha]_{\text{D}}^{\text{RT}}$ =-118.1 (c =1.0 in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min^{-1} , major enantiomer: t_{R} =21.8 min; minor enantiomer: t_{R} =31.7 min.

3-(3-bromophenyl)-3,4-dihydro-2H-1,4-benzoxazine¹

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.53-7.47 (m, 1H), 7.43-7.35 (m, 1H), 7.30-7.12 (m, 2H), 6.82-6.57 (m, 4H), 4.41 (dd, *J*(H,H)=2.9, 8.5 Hz, 1H), 4.20 (dd, *J*(H,H)=2.9, 10.6 Hz, 1H), 3.90 ppm (dd, *J*(H,H)=8.5, 10.6 Hz, 1H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=143.55, 141.46, 133.23, 131.47, 130.40, 130.28, 125.91, 122.97, 121.66, 119.37, 116.71, 115.61, 70.63, 53.76 ppm; IR (KBr): $\tilde{\nu}$ =3368, 3056, 2921, 2859, 1608, 1592, 1499, 1280, 1208, 745 cm⁻¹; MS-ESI: *m/z*: 291.7 [M]⁺ (C₁₄H₁₂⁸¹BrNO), 289.8 [M]⁺ (C₁₄H₁₂⁷⁹BrNO); [α]_D^{RT}=-118.2 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min⁻¹, major enantiomer: *t*_R=19.8 min; minor enantiomer: *t*_R=30.6 min.

3-(4-methylphenyl)-3,4-dihydro-2H-1,4-benzoxazine¹

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.25-7.19 (m, 2H), 7.15-7.08 (m, 2H), 6.80-6.68 (m, 2H), 6.66-6.55 (m, 2H), 4.39 (dd, *J*(H,H)=2.9, 8.7 Hz, 1H), 4.19 (dd, *J*(H,H)=2.9, 10.6 Hz, 1H), 3.90 (dd, *J*(H,H)=8.7, 10.6 Hz, 1H), 2.28 ppm (s, 3H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=143.59, 138.17, 136.04, 133.82, 129.51, 127.12, 121.45, 119.00, 116.60, 115.46, 71.02, 53.98, 21.16 ppm; IR (KBr): $\tilde{\nu}$ =3335, 3025, 2957, 2918, 2868, 2360, 2341, 1607, 1500, 1283, 1200, 815, 736 cm⁻¹; MS-ESI: *m/z*: 225.8 [M]⁺ (C₁₅H₁₅NO); [α]_D^{RT}=-115.8 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=90/10, flow rate=0.6 mL min⁻¹, major enantiomer: *t*_R=17.3 min; minor enantiomer: *t*_R=35.9 min.

¹) The signal of the NH- or/and OH proton was not observed in ¹H-NMR spectrum.

3-(4-bromophenyl)-6-chloro-3,4-dihydro-2H-1,4-benzoxazine¹

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.47-7.41 (m, 2H), 7.21-7.14 (m, 2H), 6.71-6.63 (m, 1H), 6.61-6.51 (m, 2H), 4.39 (m, 1H), 4.16 (dd, *J*(H,H)=2.6, 10.7 Hz, 1H), 3.84 ppm (dd, *J*(H,H)=8.2, 10.7 Hz, 1H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=142.03, 137.80, 134.45, 132.07, 128.77, 128.05, 126.28, 122.41, 118.66, 117.56, 114.87, 70.49, 53.43 ppm; IR (KBr): $\tilde{\nu}$ =3372, 2925, 2854, 1605, 1593, 1496, 1287, 1211, 1008, 817, 797 cm⁻¹; MS-ESI: *m/z*: 325.8 (C₁₄H₁₀⁸¹Br³⁷ClNO) [M-H]⁺, 323.9 (C₁₄H₁₀⁷⁹Br³⁷ClNO and C₁₄H₁₀⁸¹Br³⁵ClNO) [M-H]⁺, 321.9 (C₁₄H₁₀⁷⁹Br³⁵ClNO) [M-H]⁺; [α]_D^{RT} = -56.1 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min⁻¹, major enantiomer: *t*_R=17.6 min; minor enantiomer: *t*_R=30.1 min.

3-(4-methoxyphenyl)-3,4-dihydro-2H-1,4-benzoxazine¹

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.28-7.20 (m, 2H), 6.88-6.81 (m, 2H), 6.80-6.68 (m, 2H), 6.66-6.55 (m, 2H), 4.38 (dd, *J*(H,H)=2.9, 8.8 Hz, 1H), 4.17 (dd, *J*(H,H)=2.9, 10.6 Hz, 1H), 3.89 (dd, *J*(H,H)=8.8, 10.6 Hz, 1H), 3.74 ppm (s, 3H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=159.65, 143.53, 133.96, 131.13, 128.34, 121.43, 118.92, 116.57, 115.37, 114.22, 71.10, 55.35, 53.61 ppm; IR (KBr): $\tilde{\nu}$ =3354, 3045, 3000, 2986, 2920, 2876, 2839, 1610, 1586, 1514, 1501, 1249, 1172, 1030, 833, 745 cm⁻¹; MS-ESI: *m/z*: 241.8 [M]⁺ (C₁₅H₁₅NO₂); [α]_D^{RT} = -55.5 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min⁻¹, major enantiomer *t*_R=17.4 min; minor enantiomer: *t*_R=26.7 min.

3-biphenyl-4-yl-3,4-dihydro-2H-1,4-benzoxazine¹

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.57-7.47 (m, 4H), 7.43-7.26 (m, 5H), 6.82-6.70 (m, 2H), 6.68-6.58 (m, 2H), 4.48 (dd, *J*(H,H)=2.9, 8.5 Hz, 1H), 4.25 (dd, *J*(H,H)=2.9, 10.6 Hz, 1H), 3.97 ppm (dd, *J*(H,H)=8.5, 10.6 Hz, 1H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=143.58, 141.36, 140.61, 138.15, 133.81, 128.96, 128.86, 127.64, 127.57, 127.50, 127.18, 127.13, 121.55, 119.04, 116.65, 115.45, 70.93, 53.98 ppm; IR (KBr): $\tilde{\nu}$ =3378, 3360, 3050, 3028, 2914, 2851, 1605, 1499, 1479, 1281, 1205, 748, 696 cm⁻¹; MS-ESI: *m/z*: 287.9 [M]⁺ (C₂₀H₁₇NO); [α]_D^{RT}=-53.4 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min⁻¹, major enantiomer *t*_R=20.9 min; minor enantiomer: *t*_R=29.0 min.

3-phenyl-3,4-dihydro-2H-1,4-benzoxazin-2-one

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.50-7.35 (m, 5H), 7.12-7.01 (m, 2H), 6.95-6.80 (m, 2H), 5.08 (s, 1H), 4.31 ppm (br s, 1H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=165.24, 140.93, 136.39, 132.40, 129.02, 127.51, 125.22, 120.41, 117.00, 114.91, 59.29 ppm; IR (KBr): $\tilde{\nu}$ =3464, 3367, 3030, 2926, 1742, 1623, 1598, 1503, 1311, 1185, 743 cm⁻¹; MS-ESI: *m/z*: 225.8 [M]⁺ (C₁₄H₁₁NO₂); [α]_D^{RT}=-46.3 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min⁻¹, major enantiomer: *t*_R=18.6 min; minor enantiomer: *t*_R=25.5 min.

3-(4-bromophenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =7.49-7.39 (m, 2H), 7.28-7.17 (m, 2H), 7.02-6.91 (m, 2H), 6.87-6.71 (m, 2H), 4.95 (s, 1H), 4.17 ppm (br s, 1H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =164.73, 140.91, 135.24, 132.17, 132.06, 129.23, 125.31, 123.22, 120.75, 117.09, 115.00, 58.79 ppm; IR (KBr): $\tilde{\nu}$ =3448, 3359, 2925, 2853, 1731, 1621, 1593, 1500, 1309, 1195, 743 cm^{-1} ; MS-ESI: m/z : 304.0 $[\text{M}-\text{H}]^+$ ($\text{C}_{14}\text{H}_9^{81}\text{BrNO}_2$), 301.7 $[\text{M}-\text{H}]^+$ ($\text{C}_{14}\text{H}_9^{79}\text{BrNO}_2$); $[\alpha]_{\text{D}}^{\text{RT}}=-37.8$ ($c=1.0$ in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min^{-1} , major enantiomer: $t_{\text{R}}=10.9$ min; minor enantiomer: $t_{\text{R}}=26.1$ min.

3-(4-methoxyphenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =7.28-7.20 (m, 2H), 7.00-6.90 (m, 2H), 6.85-6.69 (m, 4H), 4.92 (d, $J(\text{H},\text{H})=1.8$ Hz, 1H), 4.12 (br s, 1H), 3.72 ppm (s, 3H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =165.54, 160.08, 141.00, 132.57, 128.81, 128.40, 125.14, 120.39, 116.99, 114.86, 114.39, 58.83, 55.35 ppm; IR (KBr): $\tilde{\nu}$ =3466, 3363, 3330, 3056, 3004, 2933, 2837, 1744, 1614, 1590, 1514, 1501, 1309, 1252, 1183, 1171, 1032, 739 cm^{-1} ; MS-ESI: $m/z(\%)$: 255.8 $[\text{M}]^+$ ($\text{C}_{15}\text{H}_{13}\text{NO}_3$); $[\alpha]_{\text{D}}^{\text{RT}}=-52.7$ ($c=1.0$ in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min^{-1} , major enantiomer: $t_{\text{R}}=13.0$ min; minor enantiomer: $t_{\text{R}}=31.4$ min.

3-(4'-bromobiphenyl-4-yl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =7.53-7.30 (m, 8H), 7.03-6.91 (m, 2H), 6.86-6.72 (m, 2H), 5.03 (d, $J(\text{H,H})$ =1.8 Hz, 1H), 4.19 ppm (br s, 1H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =165.09, 140.98, 140.78, 139.23, 135.68, 132.31, 131.99, 128.70, 128.12, 127.53, 125.25, 121.99, 120.59, 117.07, 114.94, 59.07 ppm; IR (KBr): $\tilde{\nu}$ =3419, 3362, 3056, 3031, 2952, 2926, 2853, 1738, 1609, 1514, 1481, 1444, 1256, 1174, 1002, 816, 742 cm^{-1} ; MS-ESI: m/z : 397.7 $[\text{M}+\text{NH}_3]^+$ ($\text{C}_{20}\text{H}_{17}^{81}\text{BrN}_2\text{O}_2$), 395.9 $[\text{M}+\text{NH}_3]^+$ ($\text{C}_{20}\text{H}_{17}^{79}\text{BrN}_2\text{O}_2$); $[\alpha]_{\text{D}}^{\text{RT}}$ =-61.3 (c =1.0 in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min^{-1} , major enantiomer: t_{R} =16.6 min; minor enantiomer: t_{R} =23.0 min.

3-(2-thienyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =7.19 (dd, $J(\text{H,H})$ =1.2, 4.9 Hz, 1H), 7.02-6.70 (m, 6H), 5.29 (s, 1H), 4.31 ppm (br s, 1H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =164.03, 140.98, 138.70, 131.47, 127.05, 126.87, 126.48, 125.33, 120.92, 117.00, 115.52, 55.23 ppm; IR (KBr): $\tilde{\nu}$ =3329, 3092, 2926, 1763, 1615, 1591, 1500, 1293, 1191, 748, 715 cm^{-1} ; MS-ESI: m/z : 231.8 $[\text{M}]^+$ ($\text{C}_{12}\text{H}_9\text{NO}_2\text{S}$); $[\alpha]_{\text{D}}^{\text{RT}}$ =-14.2 (c =1.0 in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min^{-1} , major enantiomer: t_{R} =18.6 min; minor enantiomer: t_{R} =26.1 min.

***N*-benzyl-2-(3,4-dimethylphenyl)-2-[(2hydroxyphenyl)amino]acetamide¹**

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.24-7.00 (m, 9H), 6.78-6.69 (m, 1H), 6.65-6.50 (m, 3H), 4.61 (s, 1H), 4.50 (dd, *J*(H,H)=6.5, 14.9 Hz, 1H), 4.28 (dd, *J*(H,H)=5.3, 14.9 Hz, 1H), 2.16 (s, 3H), 2.14 ppm (s, 3H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=172.26, 143.88, 138.00, 137.44, 137.06, 135.99, 135.72, 130.31, 128.62, 128.60, 127.68, 127.43, 124.80, 121.47, 119.31, 114.55, 113.14, 64.56, 43.40, 19.78, 19.48 ppm; IR (KBr): $\tilde{\nu}$ =3435, 3302, 3053, 2974, 2924, 1714, 1653, 1525, 1513, 1276, 1257, 737, 712 cm⁻¹; MS-ESI: *m/z*: 361.2 [M+H]⁺ (C₂₃H₂₅N₂O₂); [α]_D^{RT}=-141.3 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min⁻¹, major enantiomer: *t*_R=34.7 min; minor enantiomer: *t*_R=12.2 min.

***N*-benzyl-2-[(5-chloro-2-hydroxyphenyl)amino]-2-phenylacetamide¹**

¹H NMR (250 MHz, CD₃OD, 25 °C, TMS): δ=7.44-7.36 (m, 2H), 7.32-7.20 (m, 3H), 7.17-6.99 (m, 5H), 6.53 (d, *J*(H,H)=8.3 Hz, 1H), 6.39 (dd, *J*(H,H)=2.4, 8.3 Hz, 1H), 6.32 (d, *J*(H,H)=2.4 Hz, 1H), 4.83 (s, 1H), 4.29 ppm (dd, *J*(H,H)=15.0, 22.6 Hz, 2H); ¹³C NMR (62.5 MHz, CD₃OD, 25 °C, TMS): δ=173.89, 144.84, 139.98, 139.66, 138.11, 129.94, 129.50, 129.40, 128.34, 128.31, 128.15, 125.80, 118.15, 115.31, 112.58, 63.45, 44.09 ppm; IR (KBr): $\tilde{\nu}$ =3375, 3281, 3061, 2925, 2853, 1653, 1604, 1510, 1237, 1195, 674 cm⁻¹; MS-ESI: *m/z*: 367.2 (C₂₁H₁₈³⁷ClN₂O₂) [M-H]⁺, 365.0 (C₂₁H₁₈³⁵ClN₂O₂) [M-H]⁺; [α]_D^{RT}=-85.7 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-

propanol=80/20, flow rate=1.0 mL min⁻¹, major enantiomer: t_R=9.5 min; minor enantiomer: t_R=13.2 min.

3-biphenyl-4-yl-3,4-dihydro-2H-1,4-benzthiazine

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.57-7.47 (m, 4H), 7.42-7.23 (m, 5H), 7.00 (dd, *J*(H,H)=1.4, 7.8 Hz, 1H), 6.87 (td, *J*(H,H)=7.5, 1.5 Hz, 1H), 6.60 (td, *J*(H,H)=7.6, 1.2 Hz, 1H), 6.47 (dd, *J*(H,H)=1.0, 8.0 Hz, 1H), 4.65 (dd, *J*(H,H)=2.8, 8.8 Hz, 1H), 4.12 (br s, 1H), 3.13 (dd, *J*(H,H)=8.8, 12.4 Hz, 1H), 2.96 ppm (dd, *J*(H,H)=2.8, 12.4 Hz, 1H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=142.22, 141.87, 141.25, 140.61, 128.93, 128.88, 127.63, 127.54, 127.52, 127.23, 127.18, 127.13, 125.71, 118.42, 115.51, 115.44, 55.92, 33.13 ppm; IR (KBr): $\tilde{\nu}$ =3350, 3061, 3031, 2925, 2850, 1588, 1475, 1306, 758, 737, 694 cm⁻¹; MS-ESI: *m/z*: 304.1 [M+H]⁺ (C₂₀H₁₈NS); [α]_D^{RT}=-51.7 (*c*=1.0 in CHCl₃); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min⁻¹, major enantiomer t_R=31.9 min; minor enantiomer: t_R=38.5 min.

3-(4-bromophenyl)-3,4-dihydro-2H-1,4-benzthiazine

¹H NMR (250 MHz, CDCl₃, 25 °C, TMS): δ=7.45-7.38 (m, 2H), 7.19-7.08 (m, 2H), 6.97 (dd, *J*(H,H)=1.5, 7.7 Hz, 1H), 6.90-6.81 (m, 1H), 6.60 (td, *J*(H,H)=7.5, 1.3 Hz, 1H), 6.44 (dd, *J*(H,H)=1.2, 8.0 Hz, 1H), 4.56 (dd, *J*(H,H)=3.0, 8.2 Hz, 1H), 4.03 (br s, 1H), 3.00 (dd, *J*(H,H)=8.2, 12.6 Hz, 1H), 2.88 ppm (dd, *J*(H,H)=3.0, 12.6 Hz, 1H); ¹³C NMR (62.5 MHz, CDCl₃, 25 °C, TMS): δ=141.91, 141.78, 132.00, 128.44, 127.58, 125.79, 122.07, 118.62, 115.51,

115.47, 55.51, 32.94 ppm; IR (KBr): $\tilde{\nu}$ =3387, 3043, 2920, 2856, 1588, 1480, 1309, 1070, 1010, 818, 744 cm^{-1} ; MS-ESI: m/z : 307.8 $[\text{M}]^+$ ($\text{C}_{14}\text{H}_{12}^{81}\text{BrNS}$), 305.7 $[\text{M}]^+$ ($\text{C}_{14}\text{H}_{12}^{79}\text{BrNS}$); $[\alpha]_{\text{D}}^{\text{RT}}=-48.8$ ($c=1.0$ in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min^{-1} , major enantiomer: $t_{\text{R}}=31.9$ min; minor enantiomer: $t_{\text{R}}=38.5$ min.

3-(4-fluorophenyl)-3,4-dihydro-2H-1,4-benzthiazine

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): $\delta=7.31-7.22$ (m, 2H), 7.04-6.93 (m, 3H), 6.86 (td, $J(\text{H,H})=7.6, 1.5$ Hz, 1H), 6.60 (td, $J(\text{H,H})=7.5, 1.3$ Hz, 1H), 6.46 (dd, $J(\text{H,H})=1.3, 7.9$ Hz, 1H), 4.60 (dd, $J(\text{H,H})=2.9, 8.6$ Hz, 1H), 3.99 (br s, 1H), 3.05 (dd, $J(\text{H,H})=8.6, 12.5$ Hz, 1H), 2.90 ppm (dd, $J(\text{H,H})=2.9, 12.5$ Hz, 1H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): $\delta=162.56$ (d, $J(\text{C,F})=246.9$ Hz, 1C), 141.92, 138.63 (d, $J(\text{C,F})=3.0$ Hz, 1C), 128.37 (d, $J(\text{C,F})=8.2$ Hz, 2C), 127.52, 125.72, 118.59, 115.76 (d, $J(\text{C,F})=21.6$ Hz, 2C), 115.51, 55.45, 33.15 ppm (d, $J(\text{C,F})=1.0$ Hz, 1C); IR (KBr): $\tilde{\nu}$ =3394, 3068, 3043, 2922, 2853, 1589, 1508, 1480, 1309, 1221, 1157, 836, 794, 745, 533 cm^{-1} ; MS-ESI: m/z : 246.1 $[\text{M}+\text{H}]^+$ ($\text{C}_{14}\text{H}_{13}\text{FNS}$); $[\alpha]_{\text{D}}^{\text{RT}}=-60.1$ ($c=1.0$ in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min^{-1} , major enantiomer: $t_{\text{R}}=23.5$ min; minor enantiomer: $t_{\text{R}}=26.5$ min.

3-(4-methylphenyl)-3,4-dihydro-2H-1,4-benzthiazine

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): $\delta=7.23-7.06$ (m, 4H), 6.98 (dd, $J(\text{H,H})=1.4, 7.7$ Hz, 1H), 6.88-6.80 (m, 1H), 6.57 (td,

$J(\text{H,H})=7.5, 1.2 \text{ Hz, 1H})$, 6.43 (dd, $J(\text{H,H})=1.2, 8.0 \text{ Hz, 1H})$, 4.55 (dd, $J(\text{H,H})=2.8, 8.9 \text{ Hz, 1H})$, 3.91 (br s, 1H), 3.07 (dd, $J(\text{H,H})=8.9, 12.5 \text{ Hz, 1H})$, 2.89 (dd, $J(\text{H,H})=2.8, 12.5 \text{ Hz, 1H})$, 2.28 ppm (s, 3H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): $\delta=142.33, 139.91, 138.06, 129.57, 127.47, 126.61, 125.61, 118.28, 115.47, 115.35, 55.95, 33.18, 21.17 \text{ ppm}$; IR (KBr): $\tilde{\nu}=3380, 3012, 2920, 2853, 1591, 1480, 1309, 1105, 818, 743 \text{ cm}^{-1}$; MS-ESI: m/z : 241.6 $[\text{M}]^+$ ($\text{C}_{15}\text{H}_{15}\text{NS}$); $[\alpha]_{\text{D}}^{\text{RT}}=-33.0$ ($c=0.7$ in CHCl_3); HPLC conditions: n-hexane/2-propanol=90/10, flow rate=0.6 mL min $^{-1}$, major enantiomer: $t_{\text{R}}=20.7 \text{ min}$; minor enantiomer: $t_{\text{R}}=30.4 \text{ min}$.

3-(2-naphthyl)-3,4-dihydro-2H-1,4-benzthiazine

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): $\delta=7.81-7.68$ (m, 4H), $\delta=7.46-7.32$ (m, 3H), 7.00 (dd, $J(\text{H,H})=1.5, 7.7 \text{ Hz, 1H})$, 6.91-6.81 (m, 1H), 6.60 (td, $J(\text{H,H})=7.5, 1.3 \text{ Hz, 1H})$, 6.47 (dd, $J(\text{H,H})=1.2, 8.0 \text{ Hz, 1H})$, 4.74 (dd, $J(\text{H,H})=2.8, 8.8 \text{ Hz, 1H})$, 4.19 (br s, 1H), 3.16 (dd, $J(\text{H,H})=8.8, 12.5 \text{ Hz, 1H})$, 2.96 ppm (dd, $J(\text{H,H})=2.8, 12.5 \text{ Hz, 1H})$; ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): $\delta=142.19, 140.13, 133.40, 133.28, 128.82, 128.01, 127.78, 127.59, 126.48, 126.27, 125.74, 125.68, 124.58, 118.49, 115.62, 115.51, 56.36, 33.10 \text{ ppm}$; IR (KBr): $\tilde{\nu}=3386, 3049, 3012, 2922, 2853, 1589, 1480, 1309, 820, 743, 477 \text{ cm}^{-1}$; MS-ESI: m/z : 278.1 $[\text{M}+\text{H}]^+$ ($\text{C}_{18}\text{H}_{16}\text{NS}$); $[\alpha]_{\text{D}}^{\text{RT}}=-28.0$ ($c=1.0$ in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=1.0 mL min $^{-1}$, major enantiomer: $t_{\text{R}}=20.0 \text{ min}$; minor enantiomer: $t_{\text{R}}=31.8 \text{ min}$.

3-(3-bromophenyl)-3,4-dihydro-2H-1,4-benzthiazine

^1H NMR (250 MHz, CDCl_3 , 25 °C, TMS): δ =7.48-7.43 (m, 1H), 7.42-7.35 (m, 1H), 7.25-7.07 (m, 2H), 6.98 (dd, $J(\text{H,H})$ =1.1, 7.7 Hz, 1H), 6.92-6.82 (m, 1H), 6.65-6.56 (m, 1H), 6.54-6.44 (m, 1H), 4.60 (dd, $J(\text{H,H})$ =3.0, 8.4 Hz, 1H), 4.12 (br s, 1H), 3.05 (dd, $J(\text{H,H})$ =8.4, 12.6 Hz, 1H), 2.92 ppm (dd, $J(\text{H,H})$ =3.0, 12.6 Hz, 1H); ^{13}C NMR (62.5 MHz, CDCl_3 , 25 °C, TMS): δ =145.18, 141.76, 132.68, 131.33, 130.44, 129.80, 127.59, 125.80, 125.45, 122.96, 118.63, 115.50, 55.65, 33.00 ppm; IR (KBr): $\tilde{\nu}$ =3381, 3056, 2923, 2853, 1591, 1565, 1475, 1426, 1309, 1071, 745 cm^{-1} ; MS-ESI: m/z : 307.4 ($\text{C}_{14}\text{H}_{12}^{81}\text{BrNS}$) $[\text{M}]^+$, 305.7 ($\text{C}_{14}\text{H}_{12}^{79}\text{BrNS}$) $[\text{M}]^+$; $[\alpha]_{\text{D}}^{\text{RT}}$ = -36.0 (c =1.0 in CHCl_3); HPLC conditions: n-hexane/2-propanol=80/20, flow rate=0.6 mL min^{-1} , major enantiomer t_{R} =32.2 min; minor enantiomer: t_{R} =35.0 min.

Determination of the absolute configuration

The determination of absolute configuration was achieved by an X-ray crystal structure of 3-(2-thienyl)-3,4-dihydro-2*H*-1,4-benzoxazin-2-one and was assigned to be (*S*)-configuration (Figure 1S).

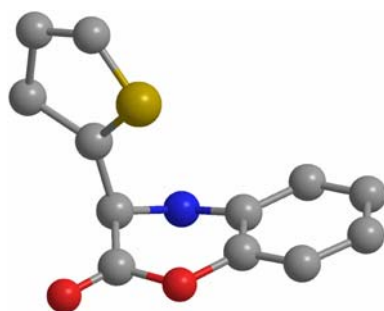


Figure 1S. X-ray crystal structure of 3-(2-thienyl)-3,4-dihydro-2*H*-1,4-benzoxazin-2-one

3-phenyl-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_9010_flow06_acq50

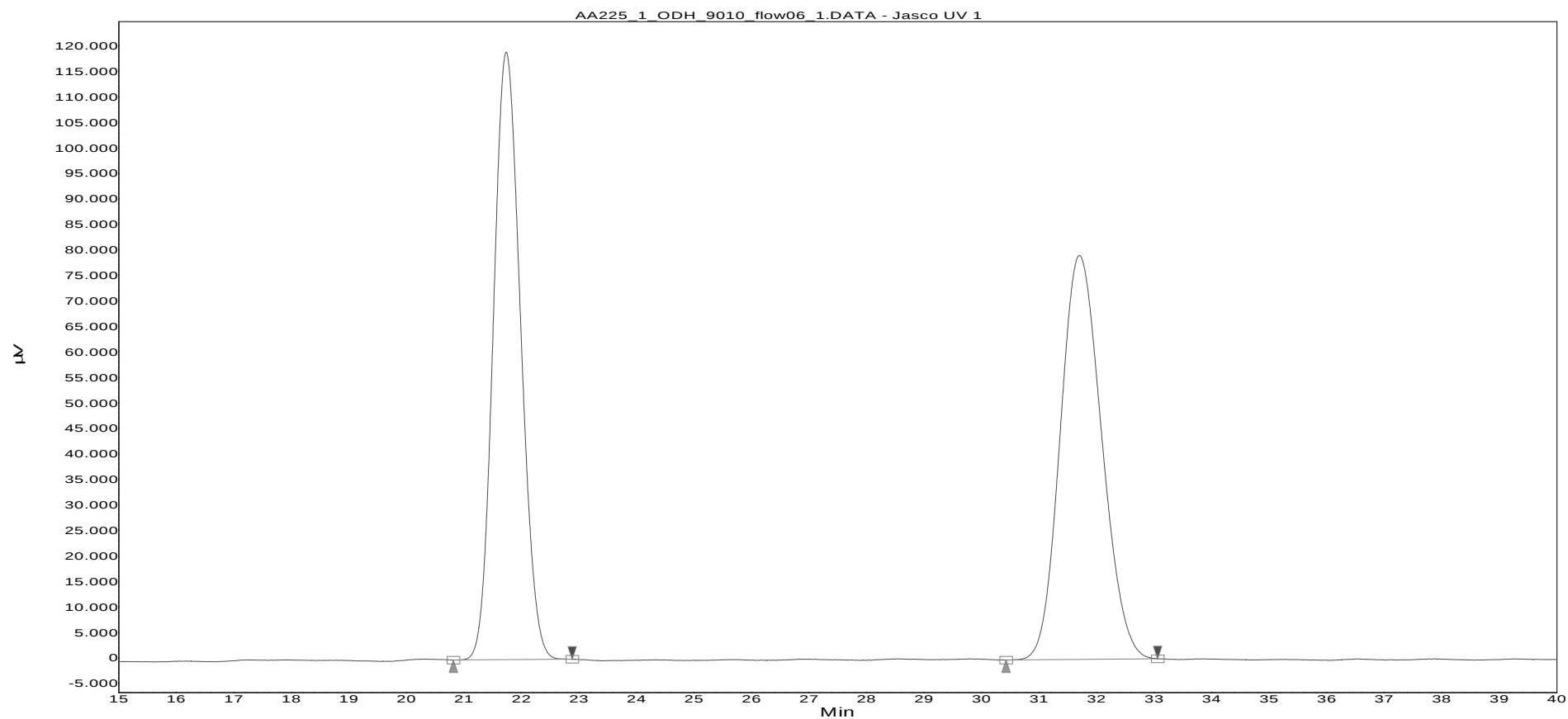
Data file: AA225_1_ODH_9010_flow06_1.DATA

Date: 24.01.2006 20:35:24

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	20,816	21,733	22,882	49,748
2	30,424	31,700	33,058	50,252
Total				100,000

3-phenyl-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_9010_flow06_acq50

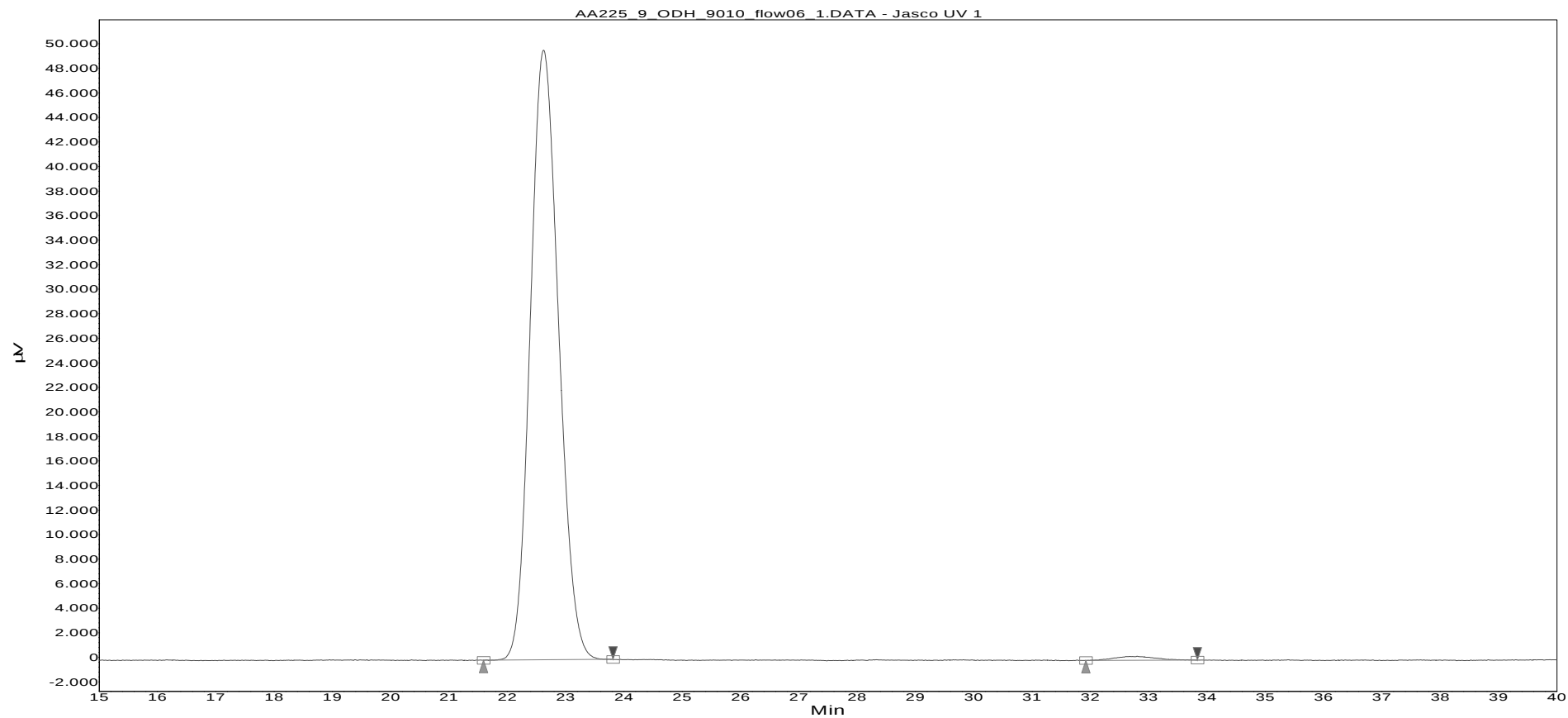
Data file: AA225_9_ODH_9010_flow06_1.DATA

Date: 17.02.2006 06:26:41

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	21,591	22,625	23,812	99,118
2	31,921	32,800	33,833	0,882
Total				100,000

3-(3-bromophenyl)-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow06_acq50

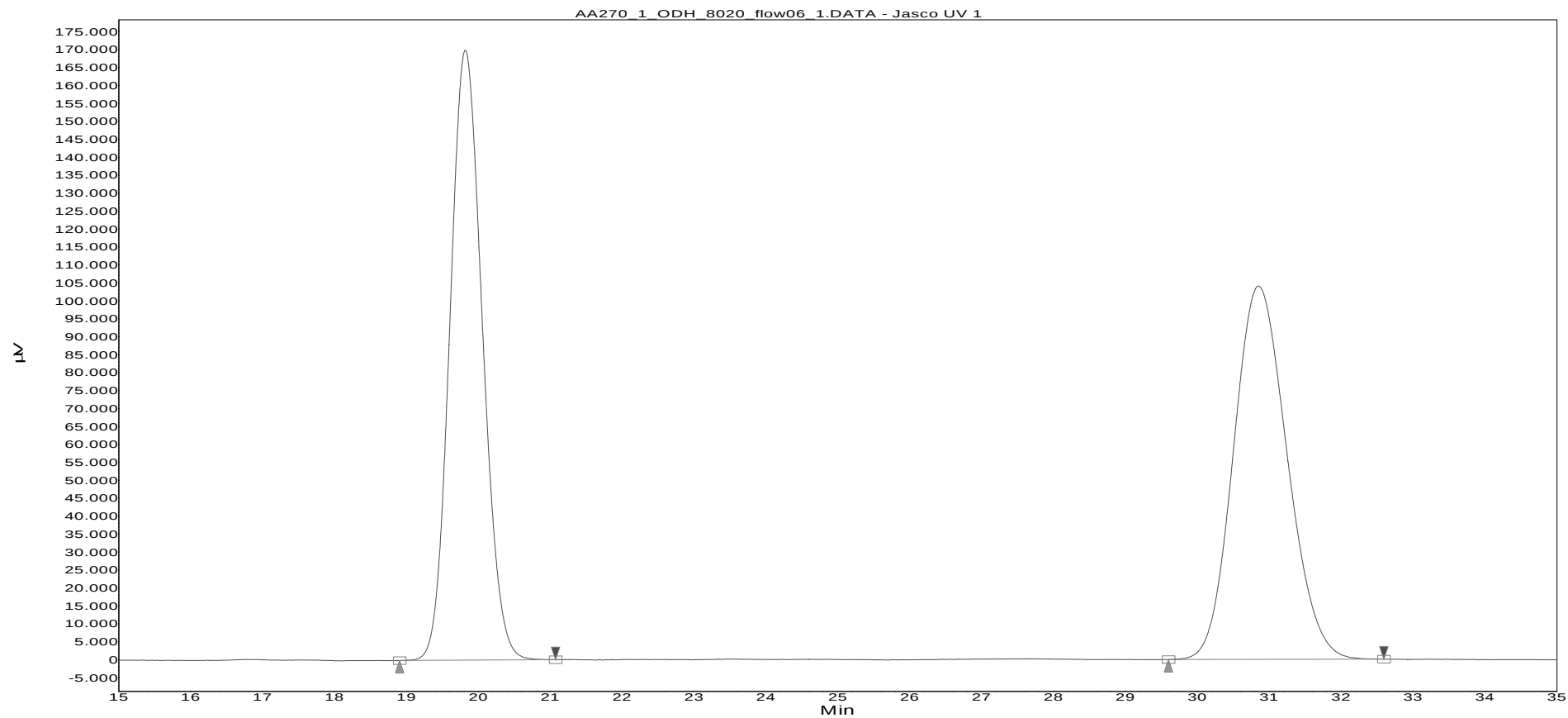
Data file: AA270_1_ODH_8020_flow06_1.DATA

Date: 22.02.2006 14:37:21

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,905	19,817	21,074	49,815
2	29,597	30,850	32,593	50,185
Total				100,000

3-(3-bromophenyl)-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow06_acq50

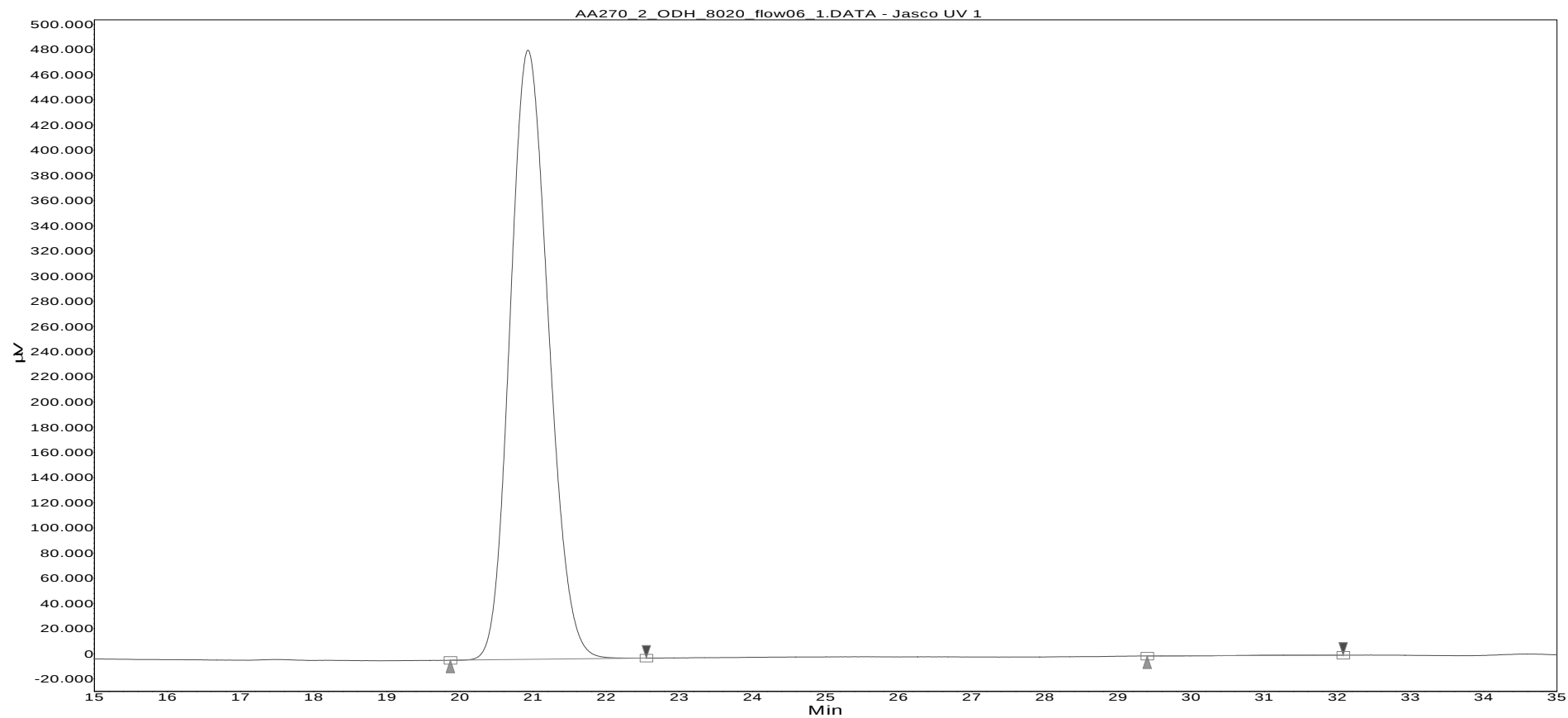
Data file: AA270_2_ODH_8020_flow06_1.DATA

Date: 22.02.2006 15:30:02

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,868	20,925	22,548	99,939
2	29,396	31,075	32,076	0,061
Total				100,000

3-(4-methylphenyl)-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_9010_flow06_acq50

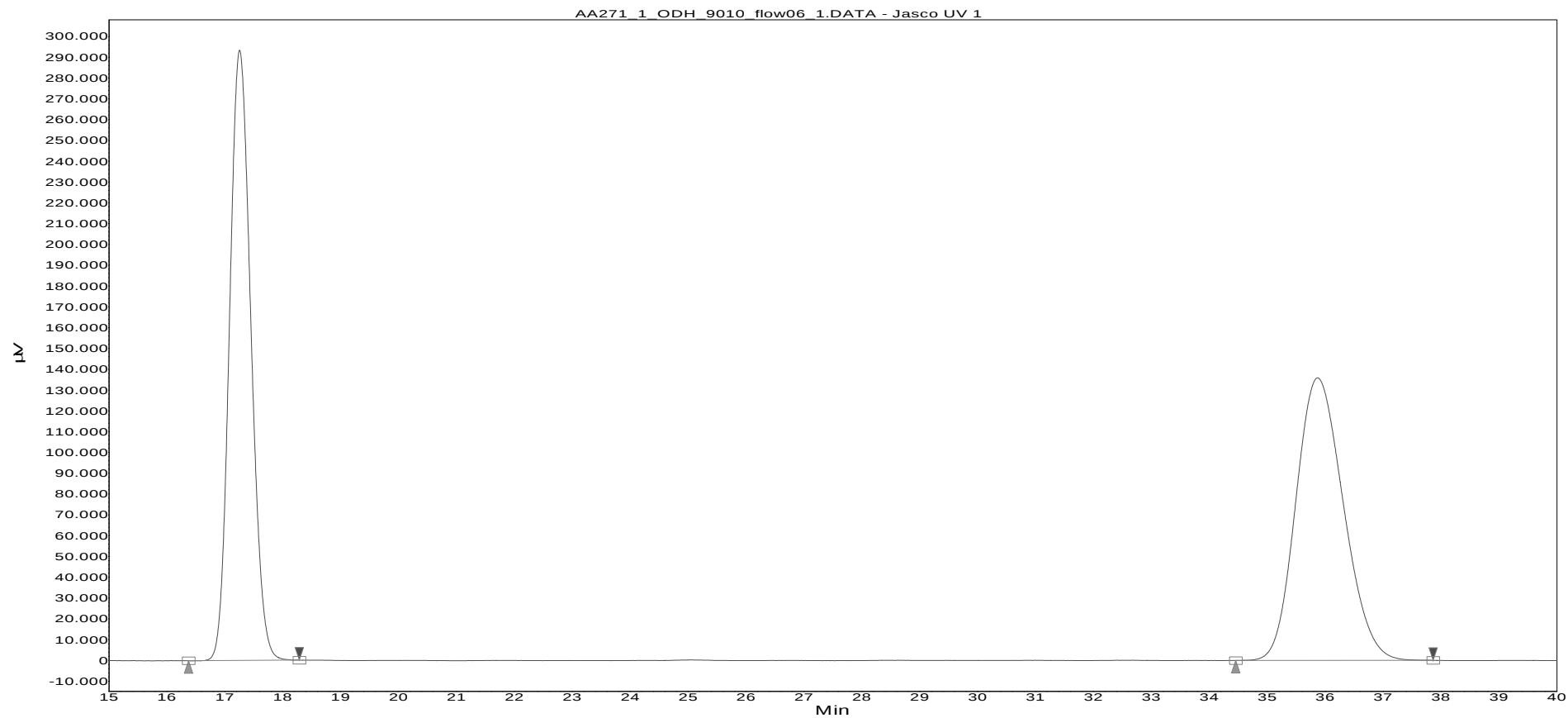
Data file: AA271_1_ODH_9010_flow06_1.DATA

Date: 22.02.2006 05:35:15

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,374	17,258	18,285	49,281
2	34,452	35,867	37,862	50,719
Total				100,000

3-(4-methylphenyl)-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_9010_flow06_acq50

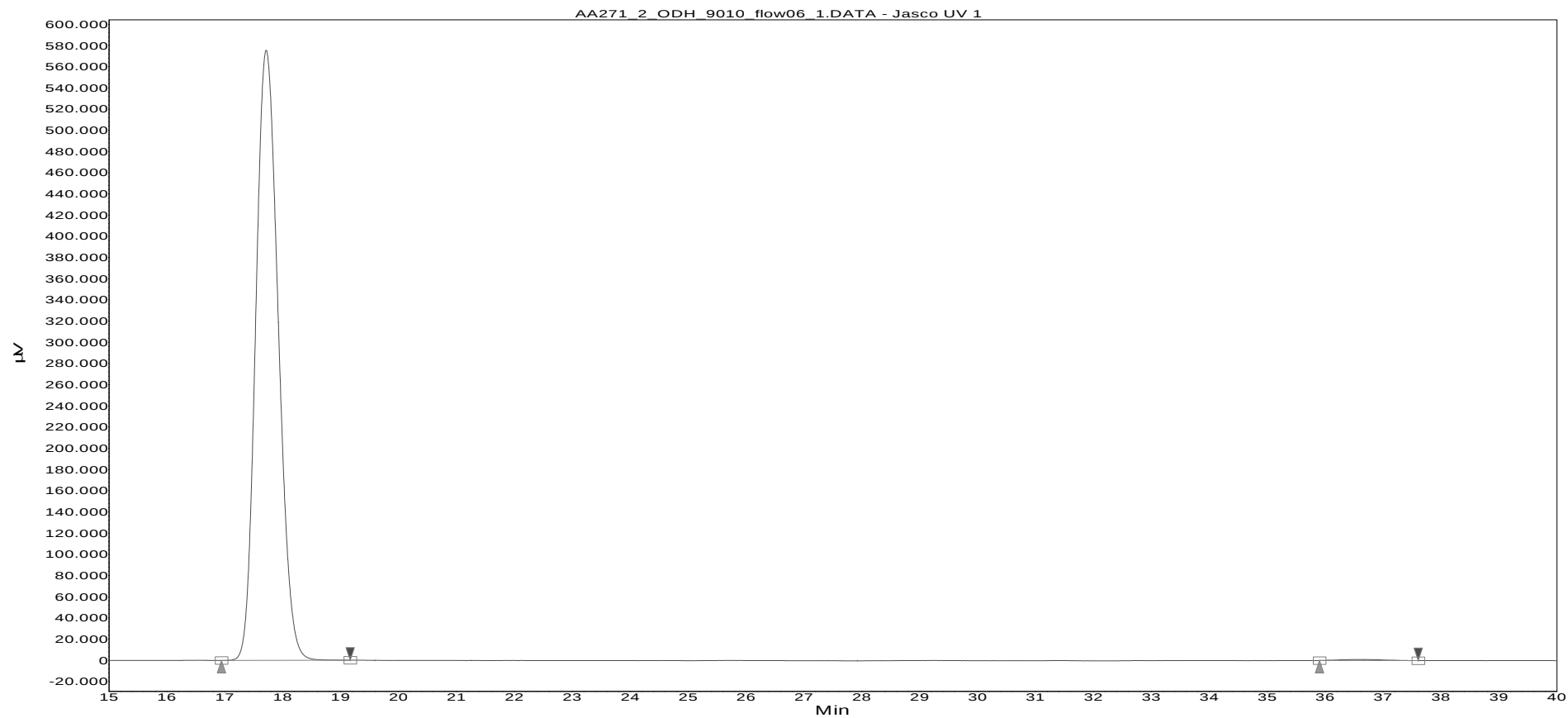
Data file: AA271_2_ODH_9010_flow06_1.DATA

Date: 22.02.2006 06:27:58

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,942	17,717	19,163	99,666
2	35,899	36,633	37,603	0,334
Total				100,000

3-(4-bromophenyl)-6-chloro-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow06_acq50

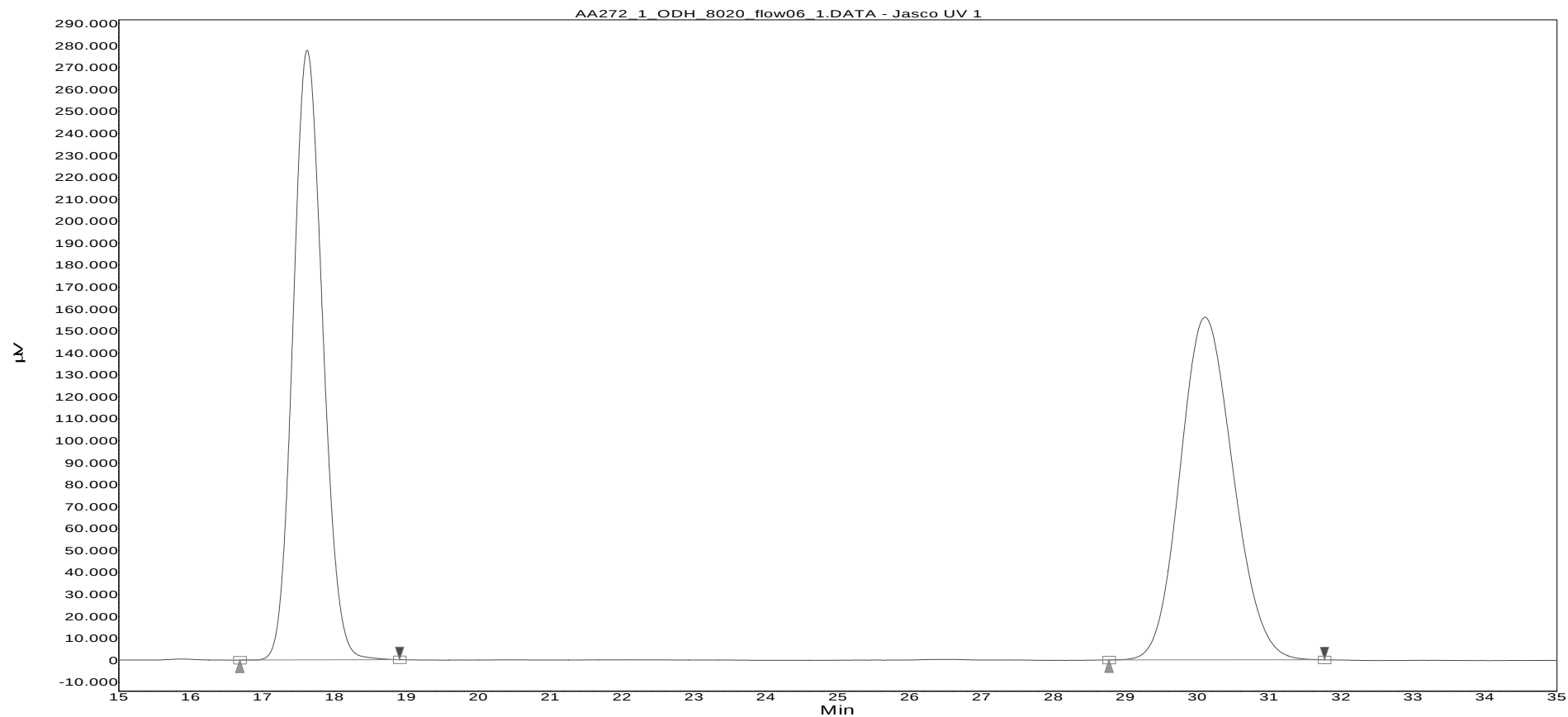
Data file: AA272_1_ODH_8020_flow06_1.DATA

Date: 22.02.2006 12:51:56

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,684	17,617	18,905	49,953
2	28,771	30,108	31,767	50,047
Total				100,000

3-(4-bromophenyl)-6-chloro-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow06_acq50

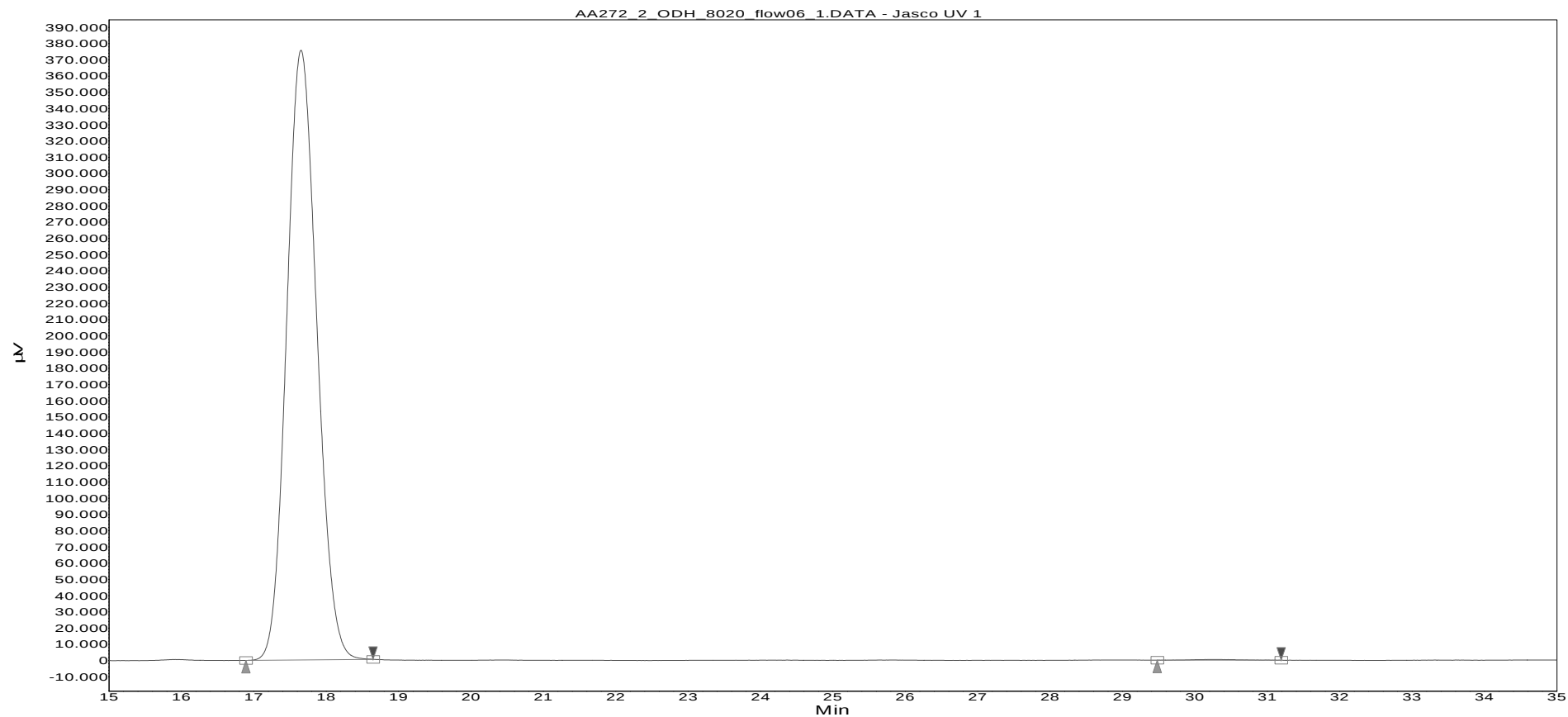
Data file: AA272_2_ODH_8020_flow06_1.DATA

Date: 22.02.2006 13:44:36

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,890	17,650	18,647	99,843
2	29,479	30,258	31,191	0,157
Total				100,000

3-(4-methoxyphenyl)-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow06_acq50

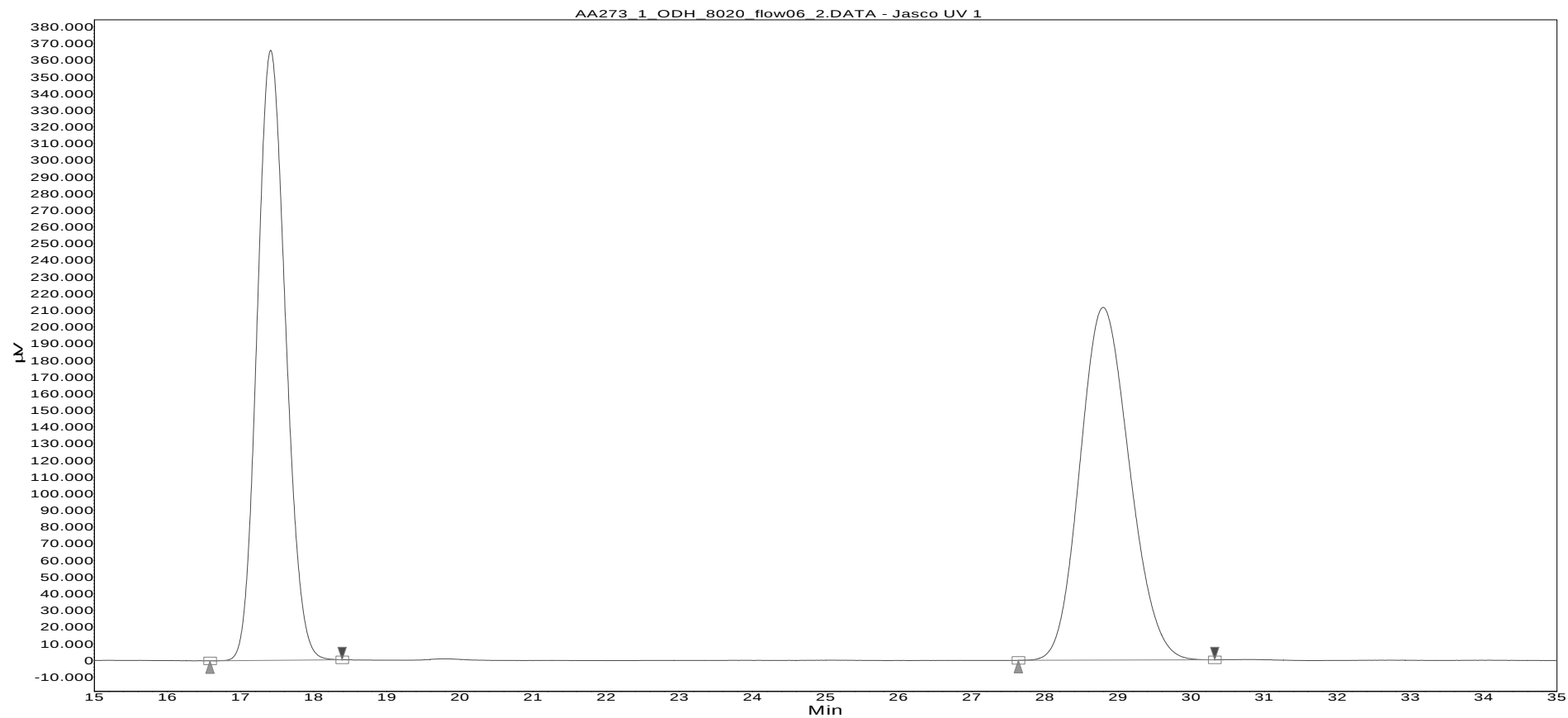
Data file: AA273_1_ODH_8020_flow06_2.DATA

Date: 22.02.2006 11:59:13

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,581	17,408	18,388	49,920
2	27,634	28,792	30,320	50,080
Total				100,000

3-(4-methoxyphenyl)-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow06_acq50

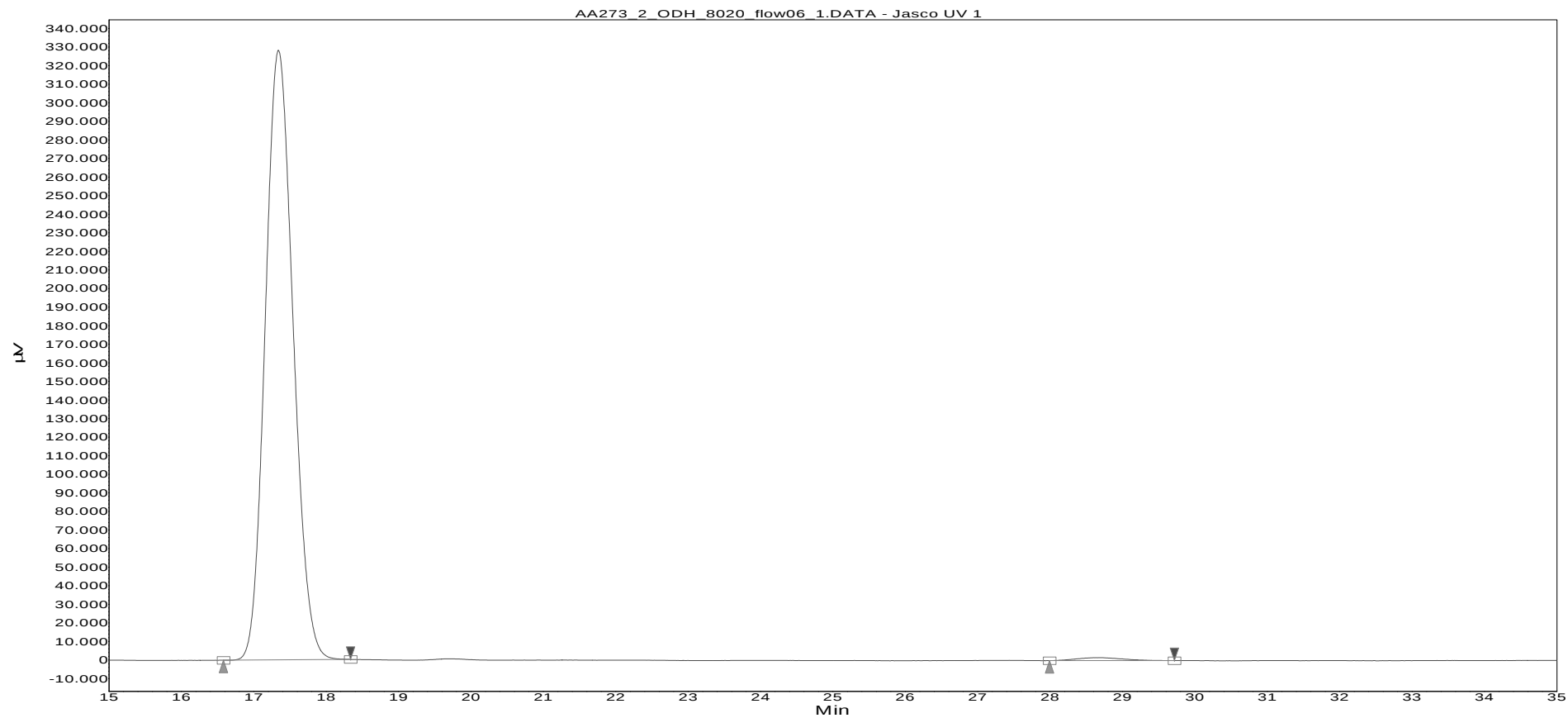
Data file: AA273_2_ODH_8020_flow06_1.DATA

Date: 21.02.2006 23:26:03

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	16,581	17,342	18,337	99,218
1	27,991	28,675	29,715	0,782
Total				100,000

3-biphenyl-4-yl-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow1_acq50

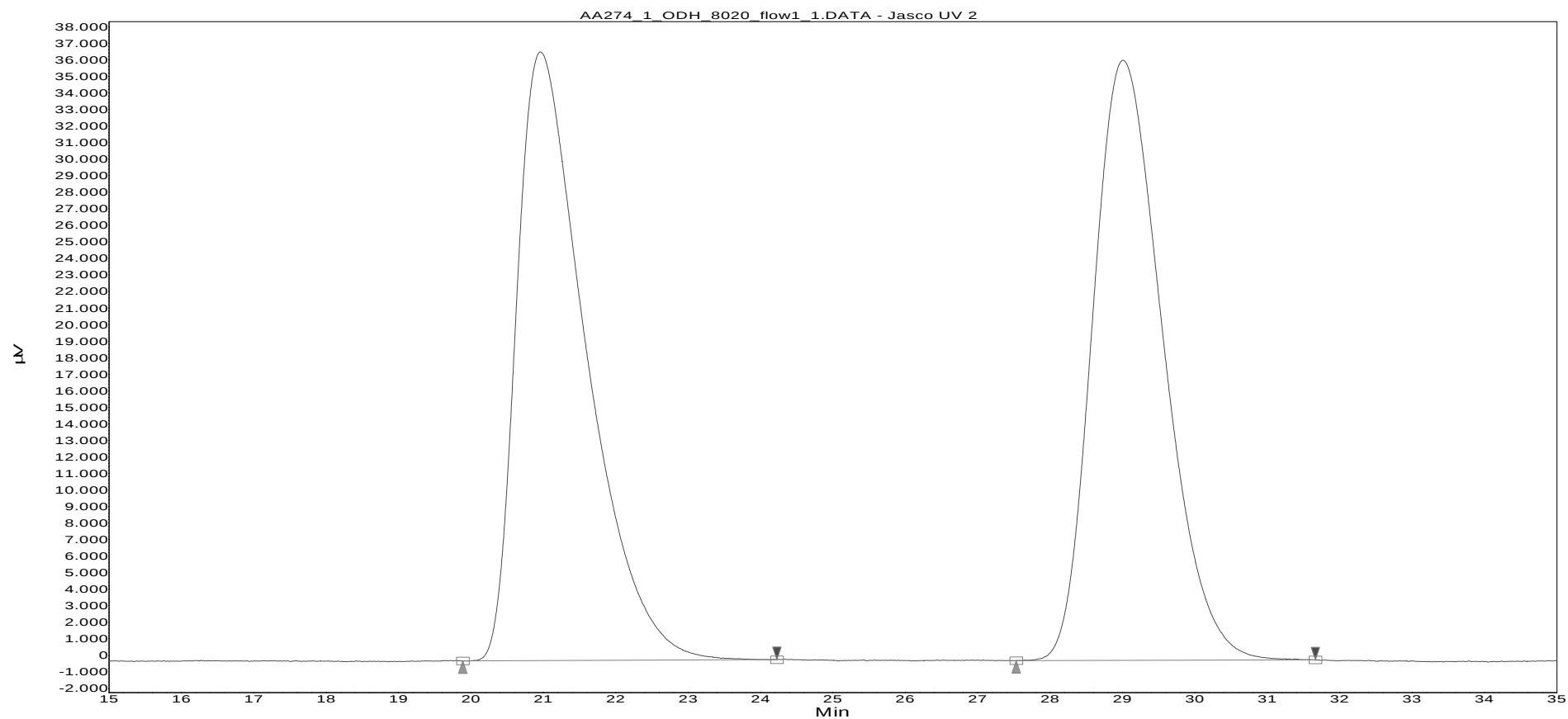
Data file: AA274_1_ODH_8020_flow1_1.DATA

Date: 22.02.2006 17:15:30

Column: ODH

Flow: 1.0 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,886	20,958	24,225	50,107
2	27,531	29,000	31,663	49,893
Total				100,000

3-biphenyl-4-yl-3,4-dihydro-2H-1,4-benzoxazine

Method: ODH_8020_flow1_acq50

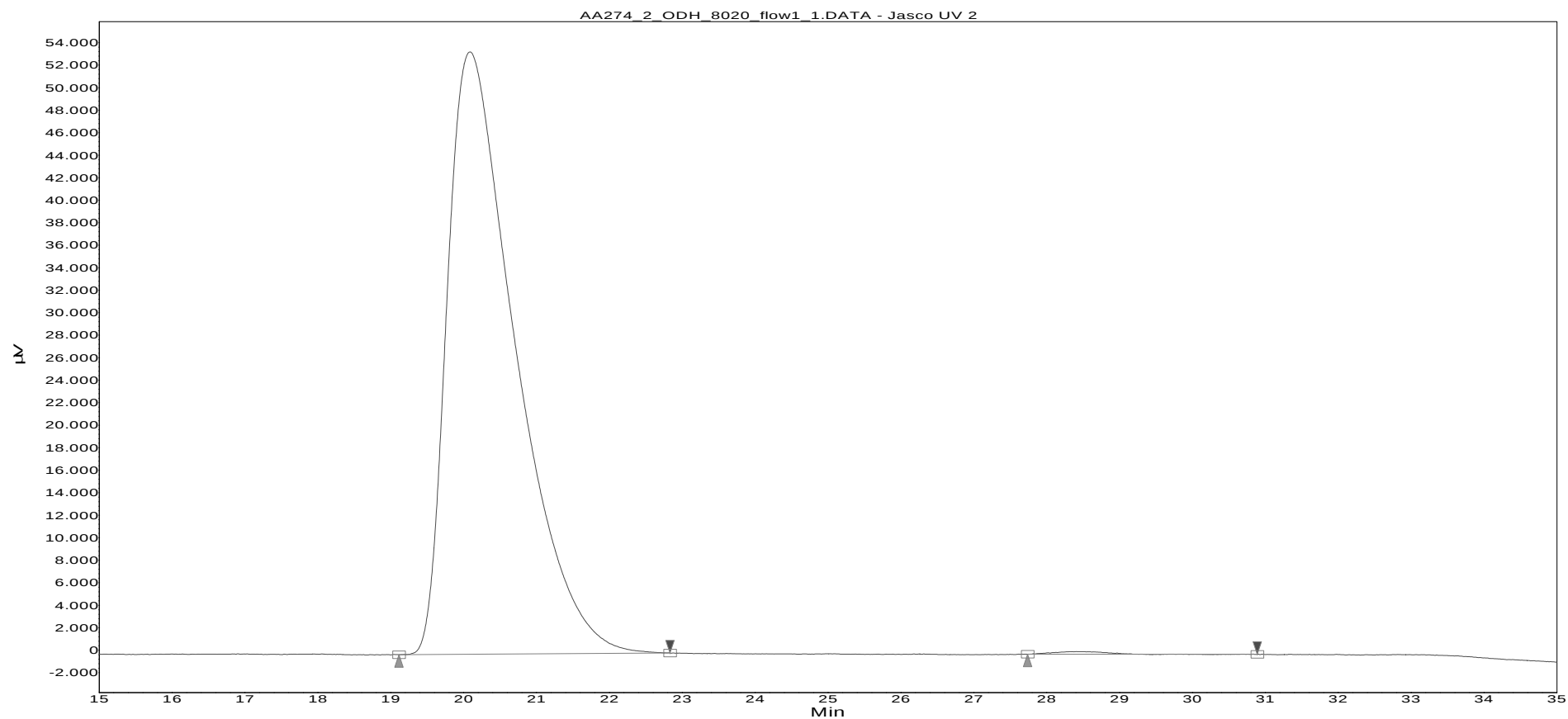
Data file: AA274_2_ODH_8020_flow1_1.DATA

Date: 22.02.2006 18:08:15

Column: ODH

Flow: 1.0 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,112	20,083	22,831	99,697
2	27,738	28,408	30,888	0,303
Total				100,000

3-phenyl-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow06_acq50

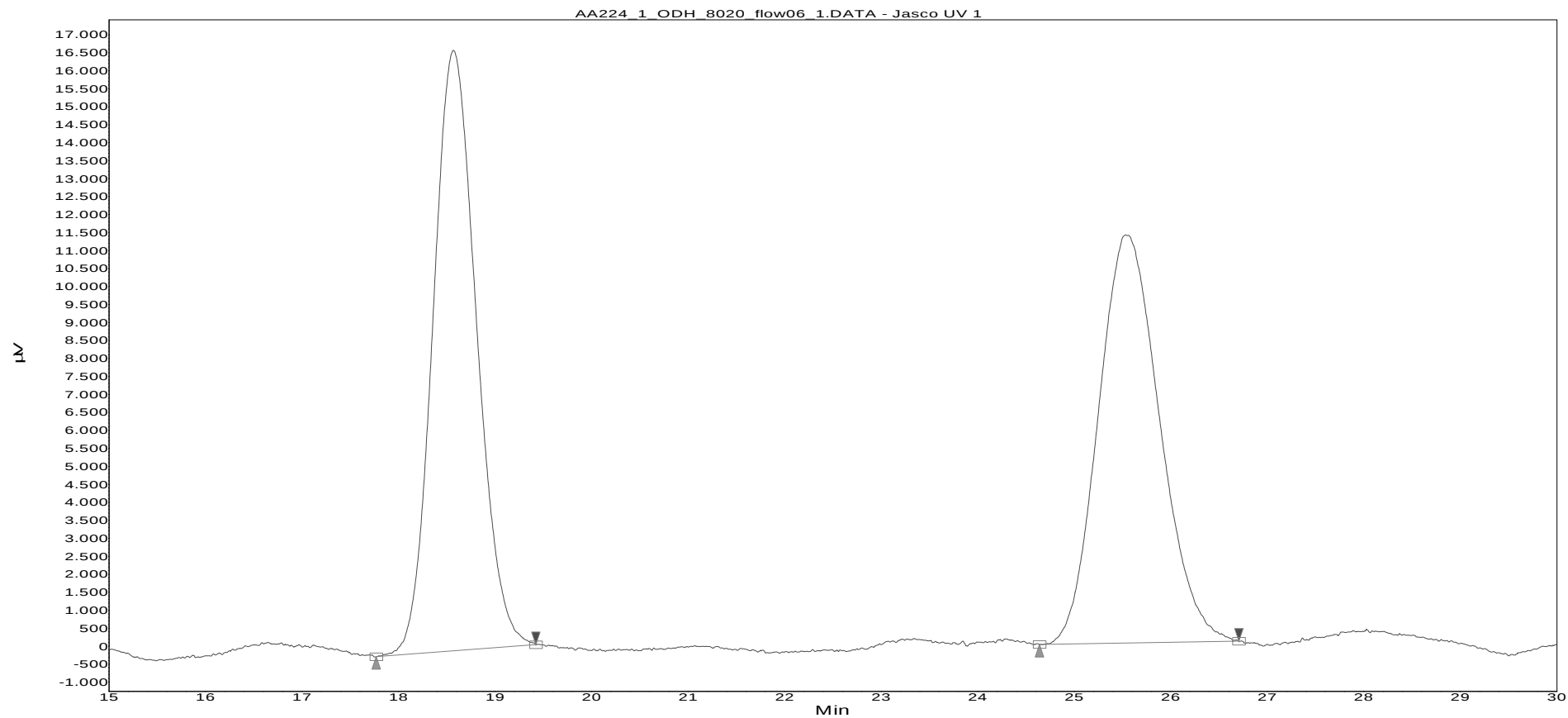
Data file: AA224_1_ODH_8020_flow06_1.DATA

Date: 24.01.2006 12:14:30

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	17,769	18,567	19,421	51,128
2	24,638	25,525	26,705	48,872
Total				100,000

3-phenyl-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow06_acq50

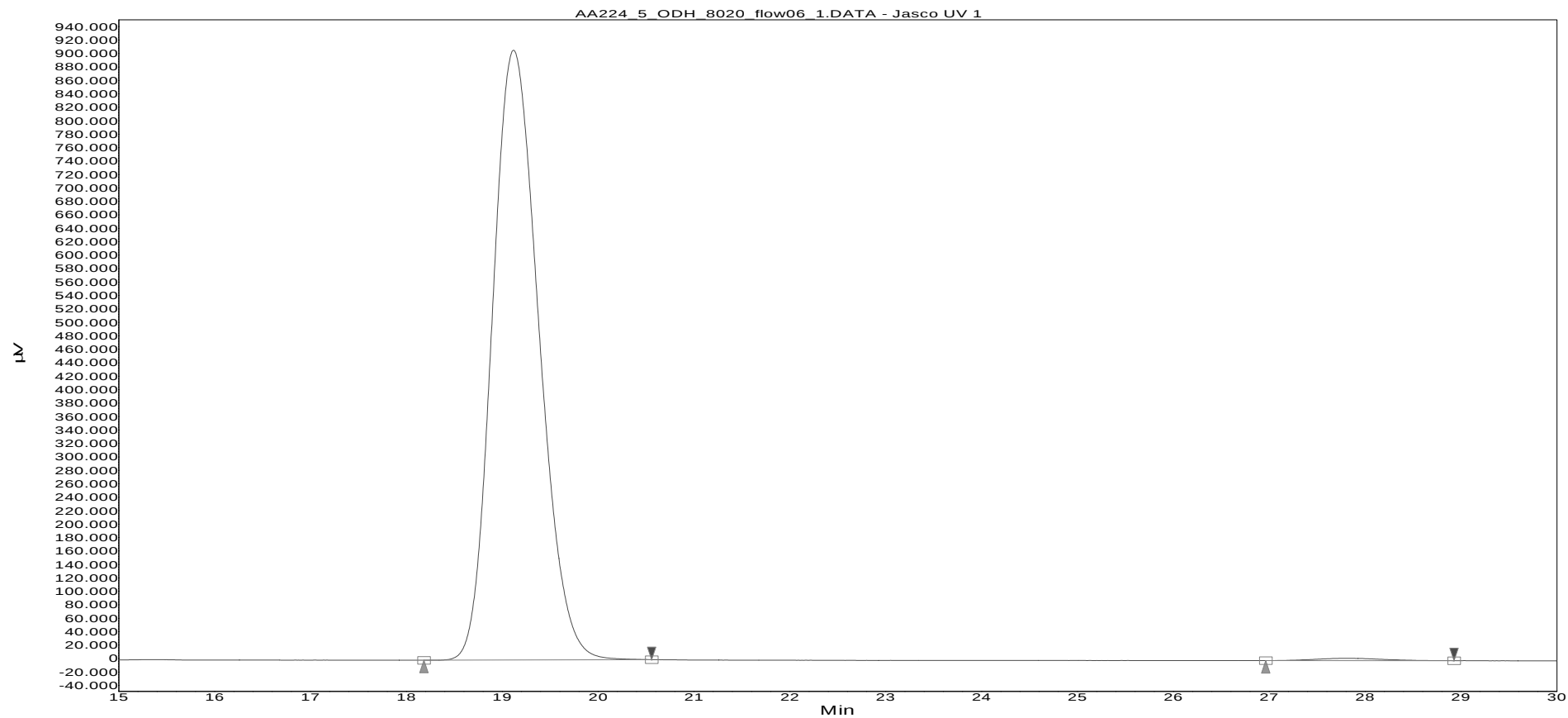
Data file: AA224_5_ODH_8020_flow06_1.DATA

Date: 06.04.2006 00:31:53

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,182	19,117	20,558	99,456
2	26,963	27,817	28,926	0,544
Total				100,000

3-(4-bromophenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

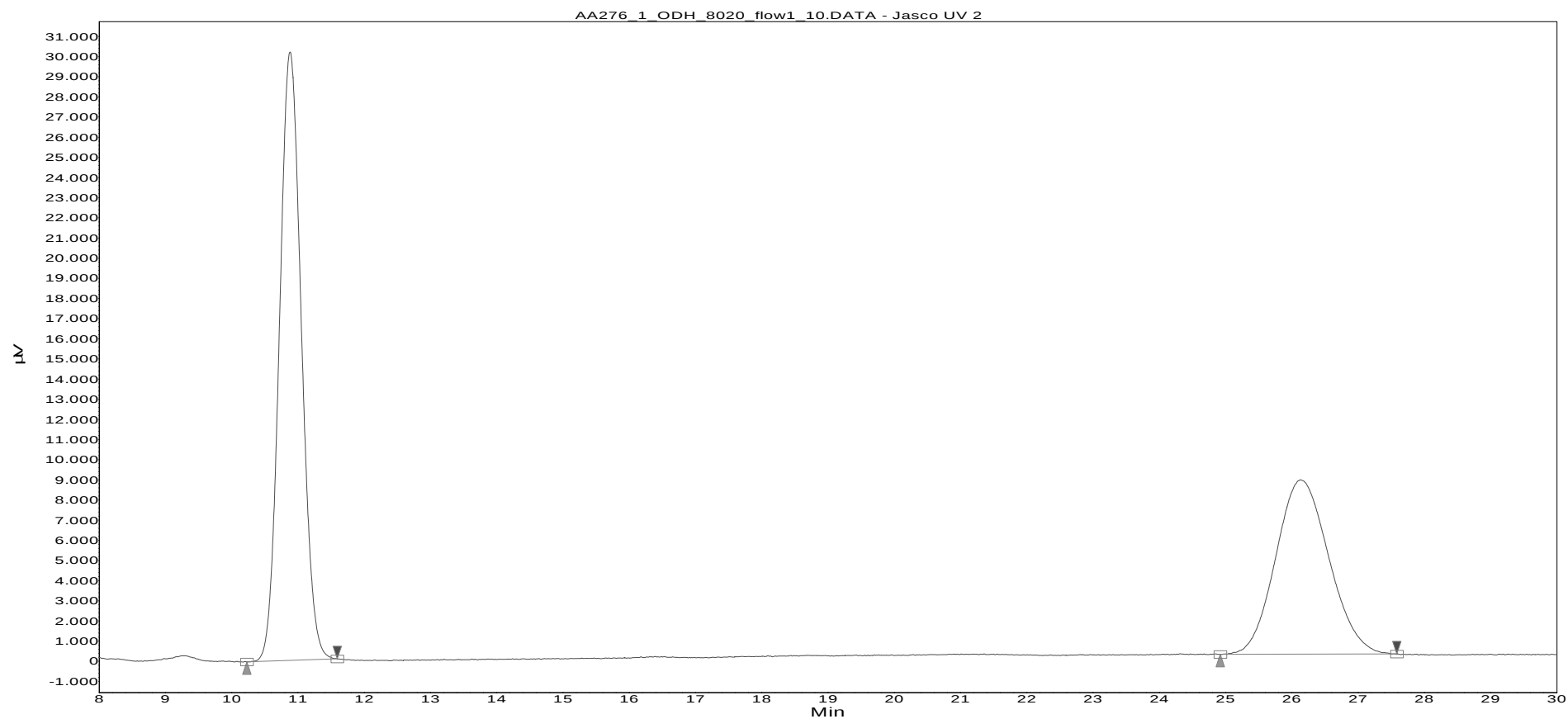
Data file: AA276_1_ODH_8020_flow1_10.DATA

Date: 16.03.2006 16:11:24

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,227	10,883	11,591	59,036
2	24,917	26,125	27,583	40,964
Total				100,000

3-(4-bromophenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

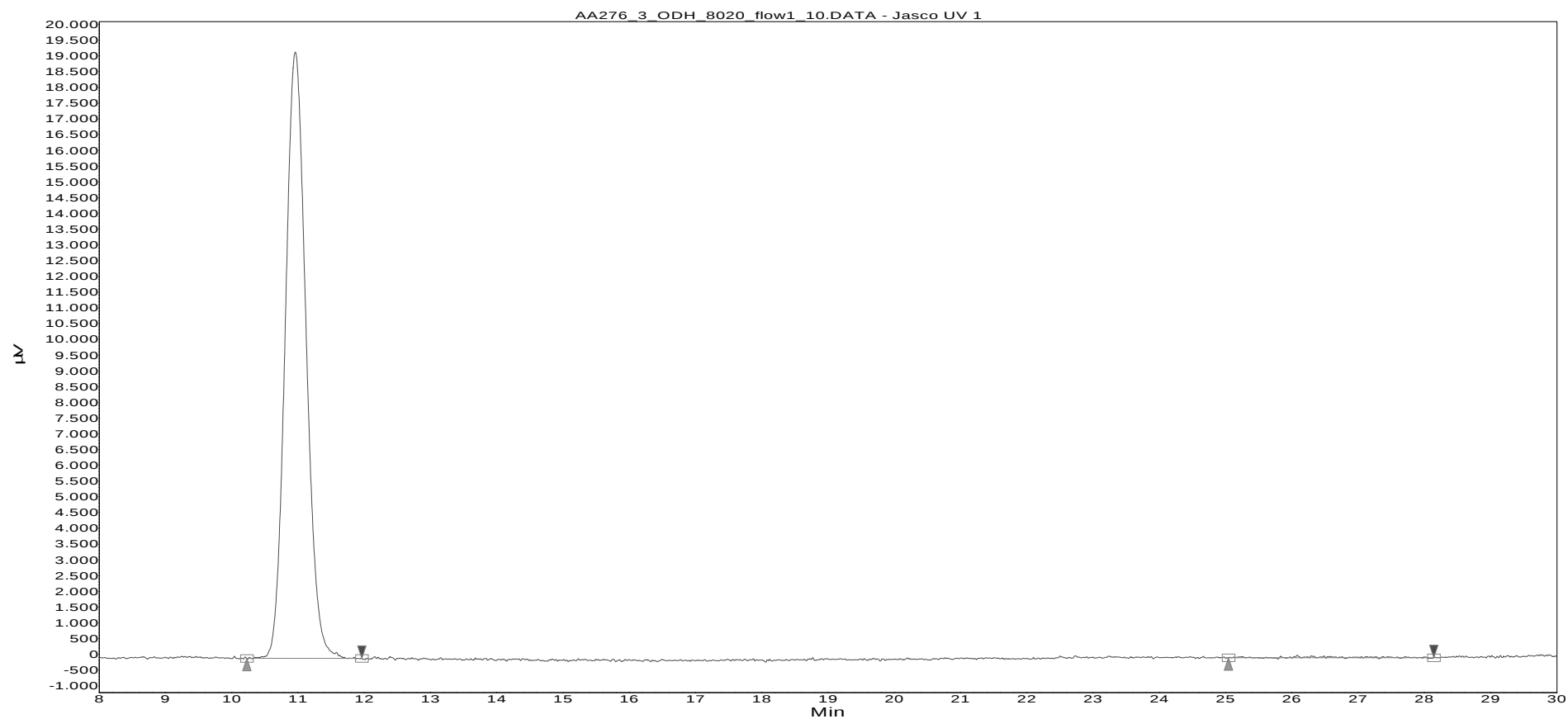
Data file: AA276_3_ODH_8020_flow1_10.DATA

Date: 16.03.2006 17:14:09

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,227	10,958	11,963	99,523
2	25,041	26,075	28,140	0,477
Total				100,000

3-(4-methoxyphenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

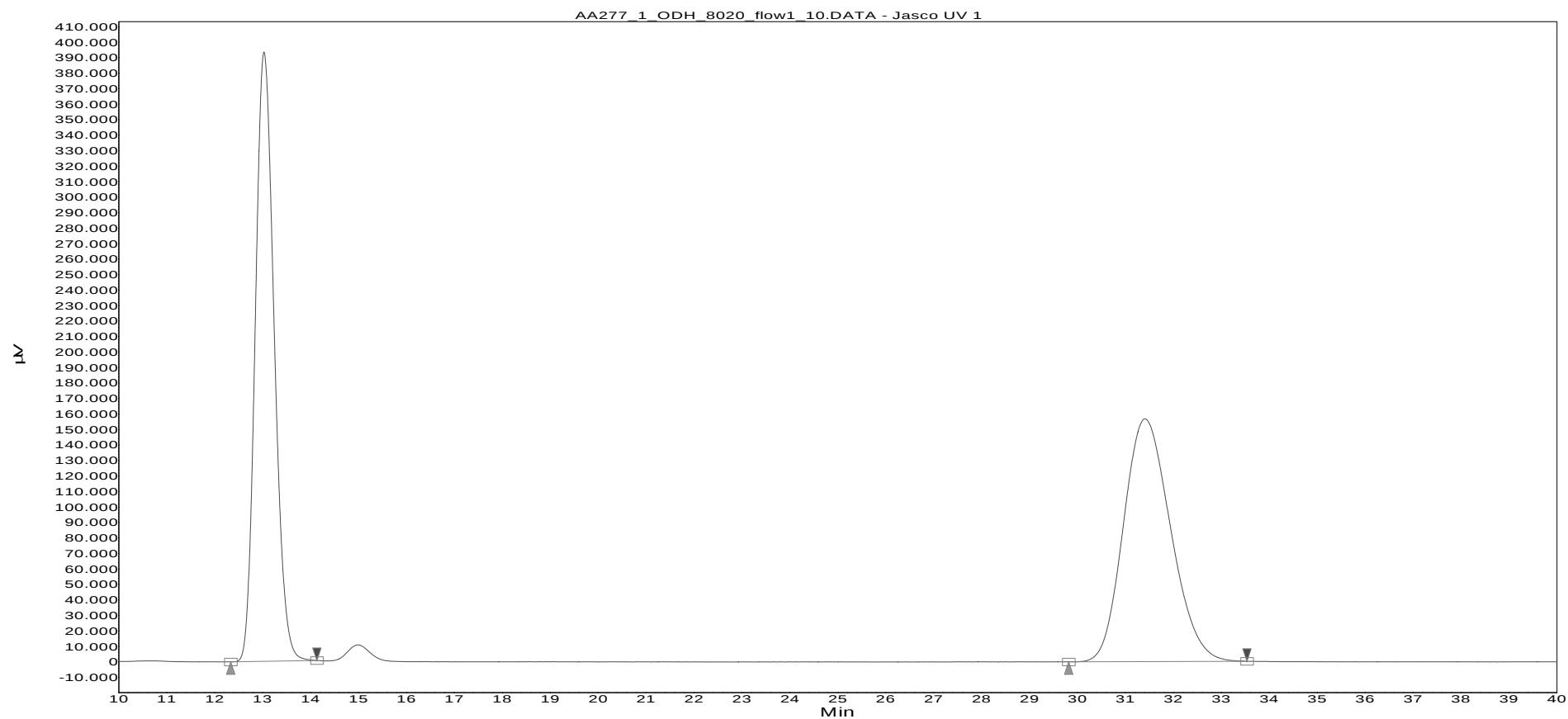
Data file: AA277_1_ODH_8020_flow1_10.DATA

Date: 16.03.2006 18:16:55

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	12,335	13,033	14,132	50,151
2	29,814	31,408	33,533	49,849
Total				100,000

3-(4-methoxyphenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

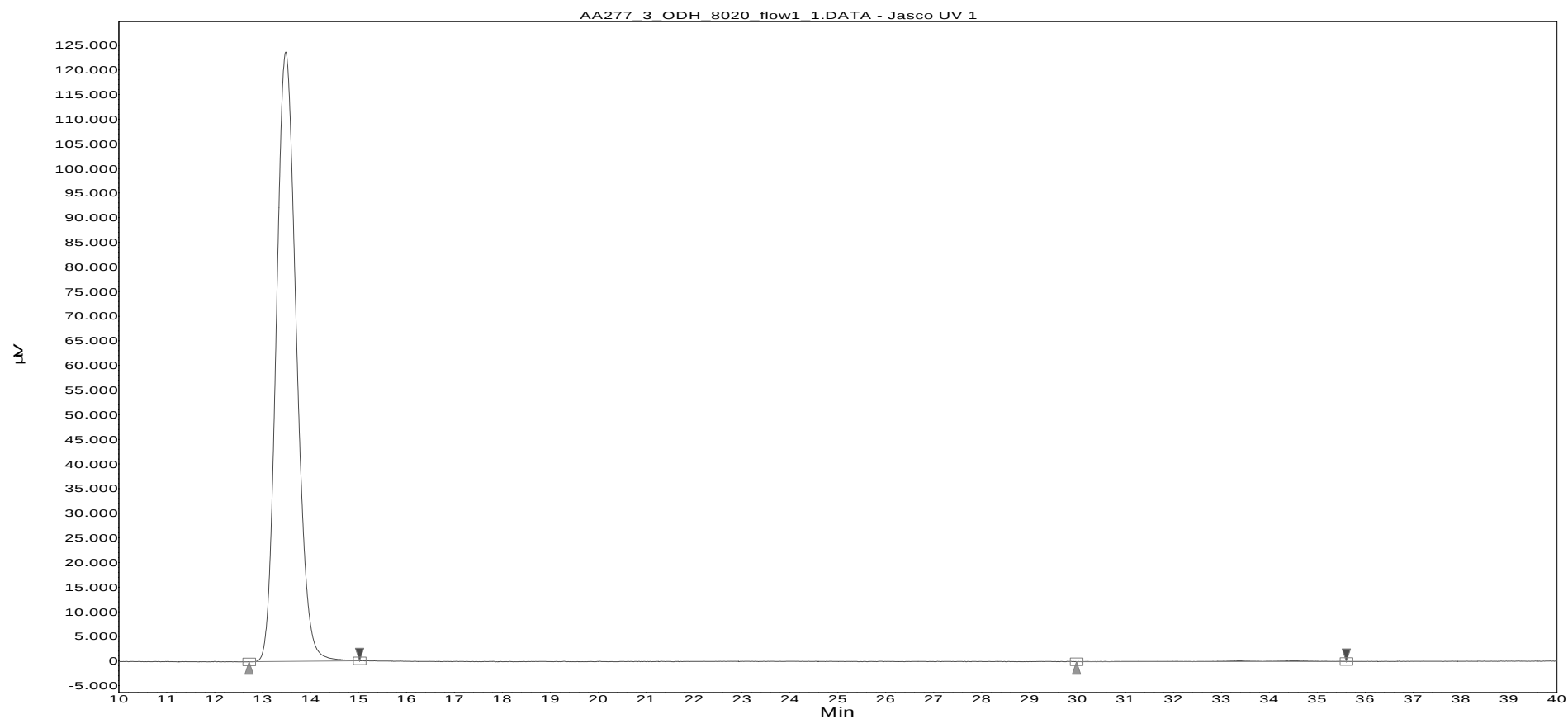
Data file: AA277_3_ODH_8020_flow1_1.DATA

Date: 01.03.2006 04:20:29

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	12,719	13,483	15,022	99,389
2	29,976	33,875	35,605	0,611
Total				100,000

3-(3,4-dimethylphenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq50

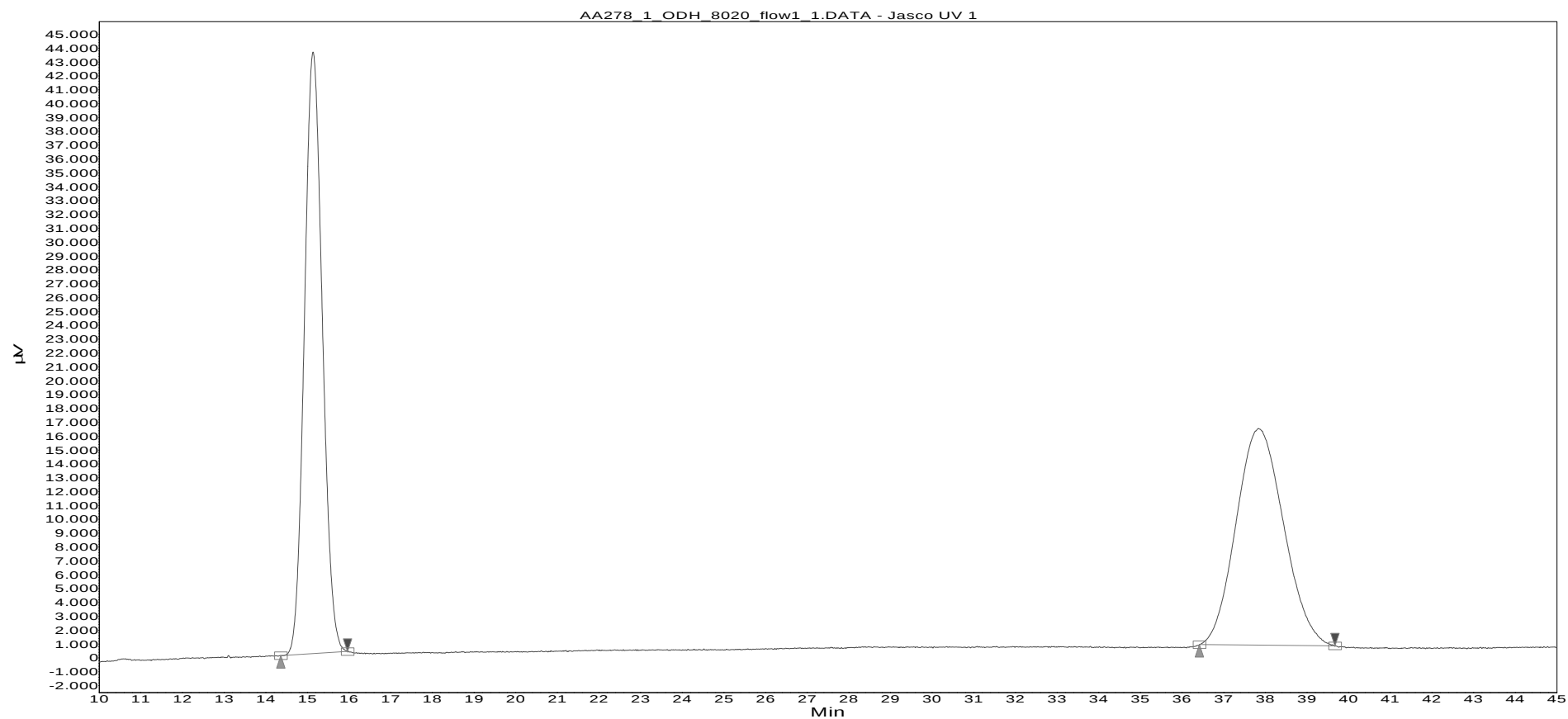
Data file: AA278_1_ODH_8020_flow1_1.DATA

Date: 08.03.2006 14:53:26

Column: ODH

Flow: 1.0 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	14,360	15,133	15,961	51,284
2	36,415	37,833	39,669	48,716
Total				100,000

3-(3,4-dimethylphenyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq50

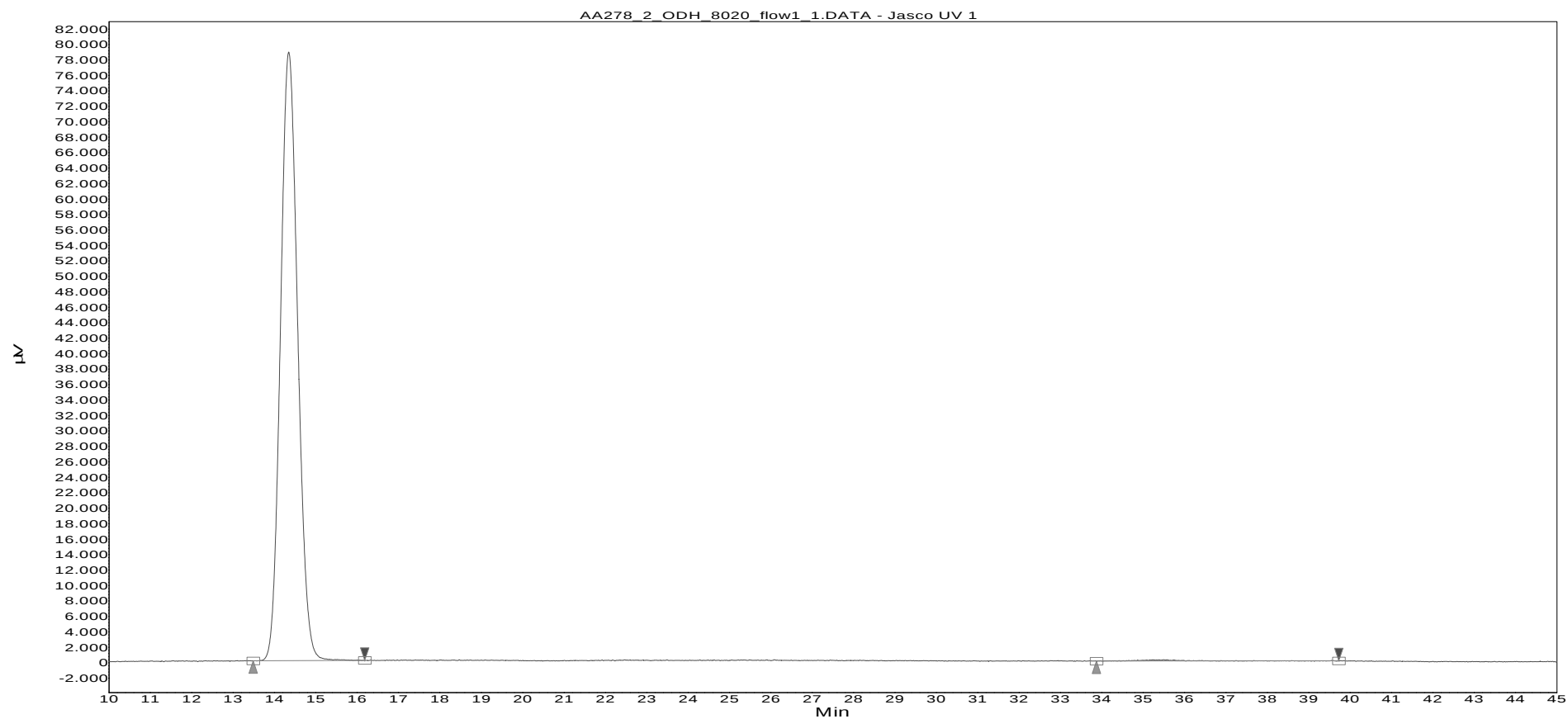
Data file: AA278_2_ODH_8020_flow1_1.DATA

Date: 08.03.2006 15:46:11

Column: ODH

Flow: 1.0 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,485	14,342	16,182	99,556
2	33,867	35,375	39,727	0,444
Total				100,000

N-benzyl-2-(3,4-dimethylphenyl)-2-[(2-hydroxyphenyl)amino]acetamide

Method: ODH_8020_flow1_acq60

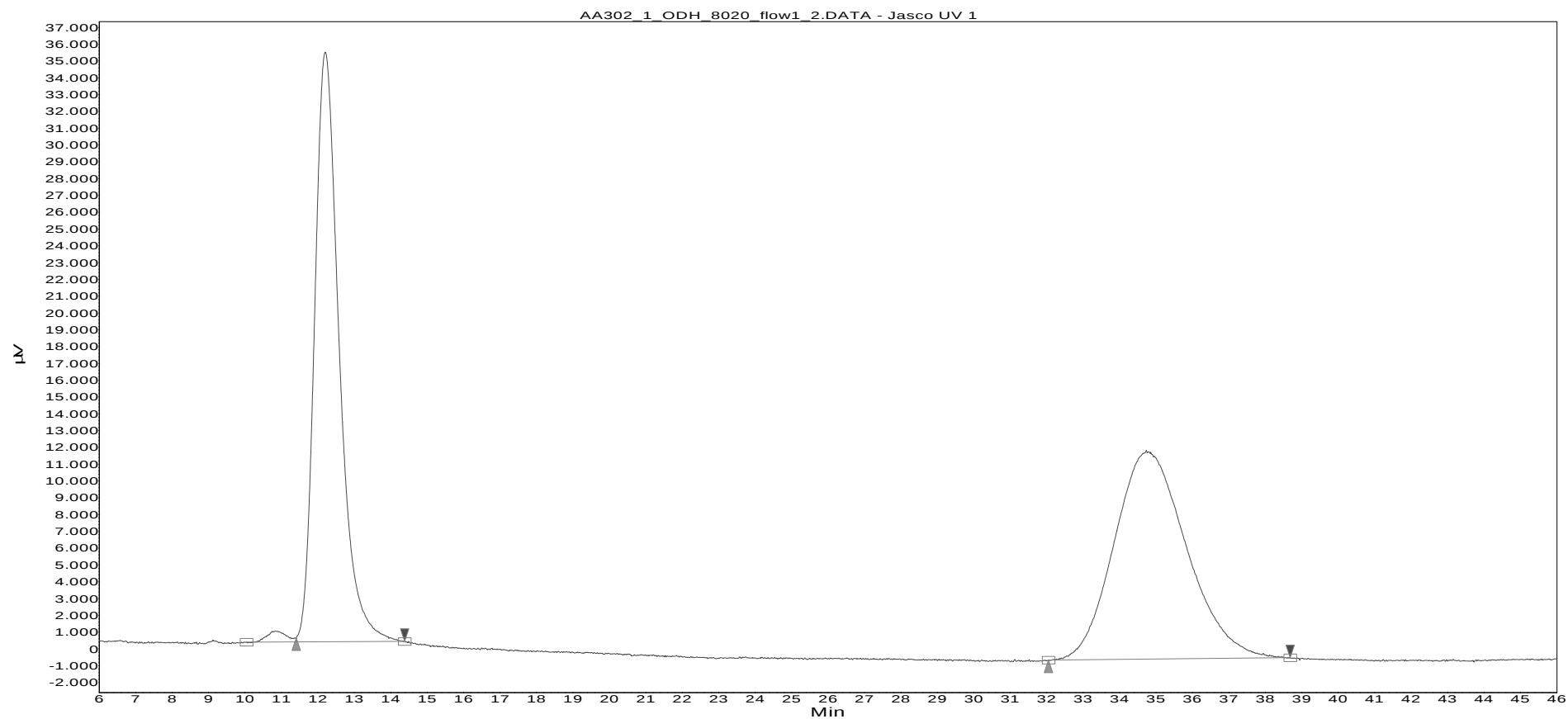
Data file: AA302_1_ODH_8020_flow1_2.DATA

Date: 31.03.2006 14:13:45

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	11,405	12,200	14,380	49,155
1	32,045	34,733	38,678	50,845
Total				100,000

N-benzyl-2-(3,4-dimethylphenyl)-2-[(2-hydroxyphenyl)amino]acetamide

Method: ODH_8020_flow1_acq60

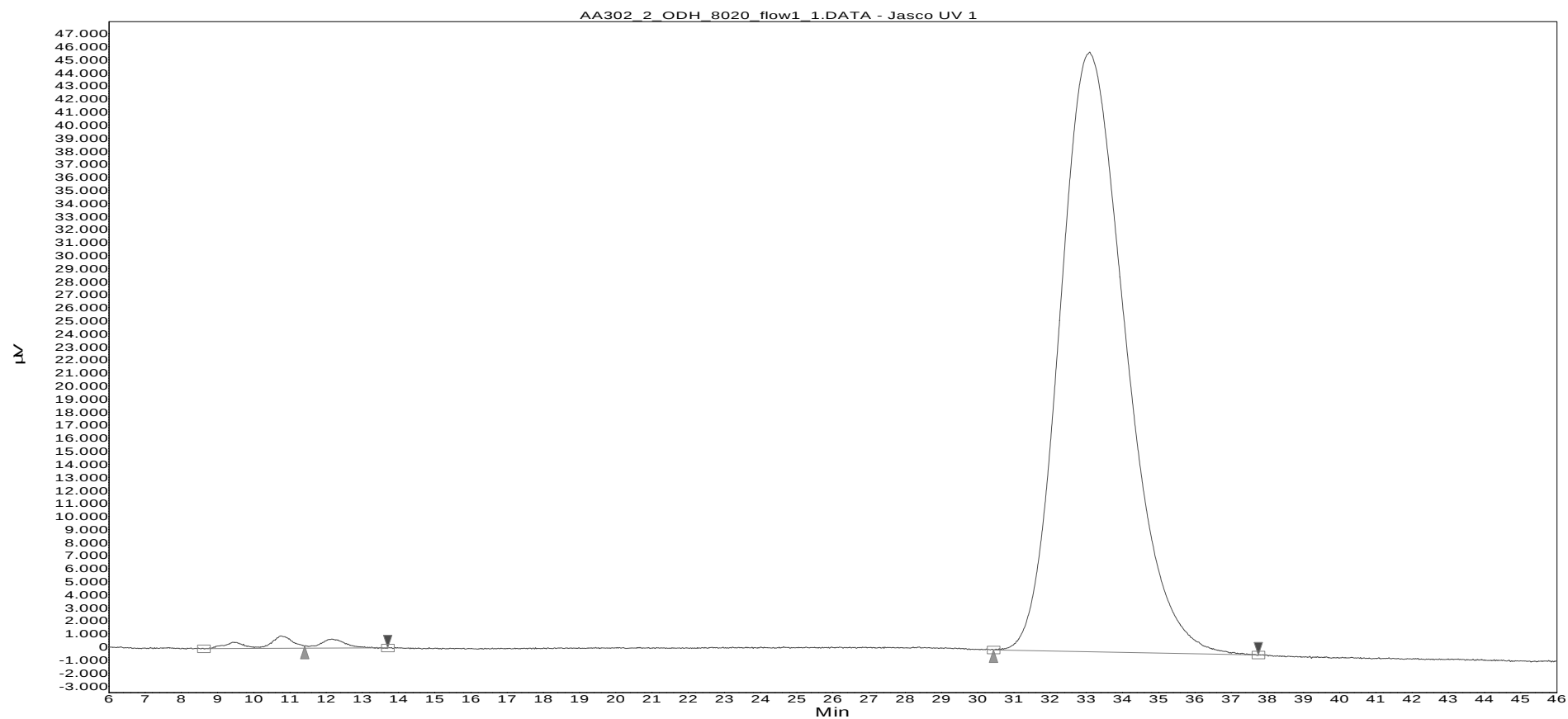
Data file: AA302_2_ODH_8020_flow1_1.DATA

Date: 31.03.2006 13:11:02

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	11,405	12,158	13,698	0,572
1	30,434	33,092	37,748	99,428
Total				100,000

3-(4'-bromobiphenyl-4-yl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

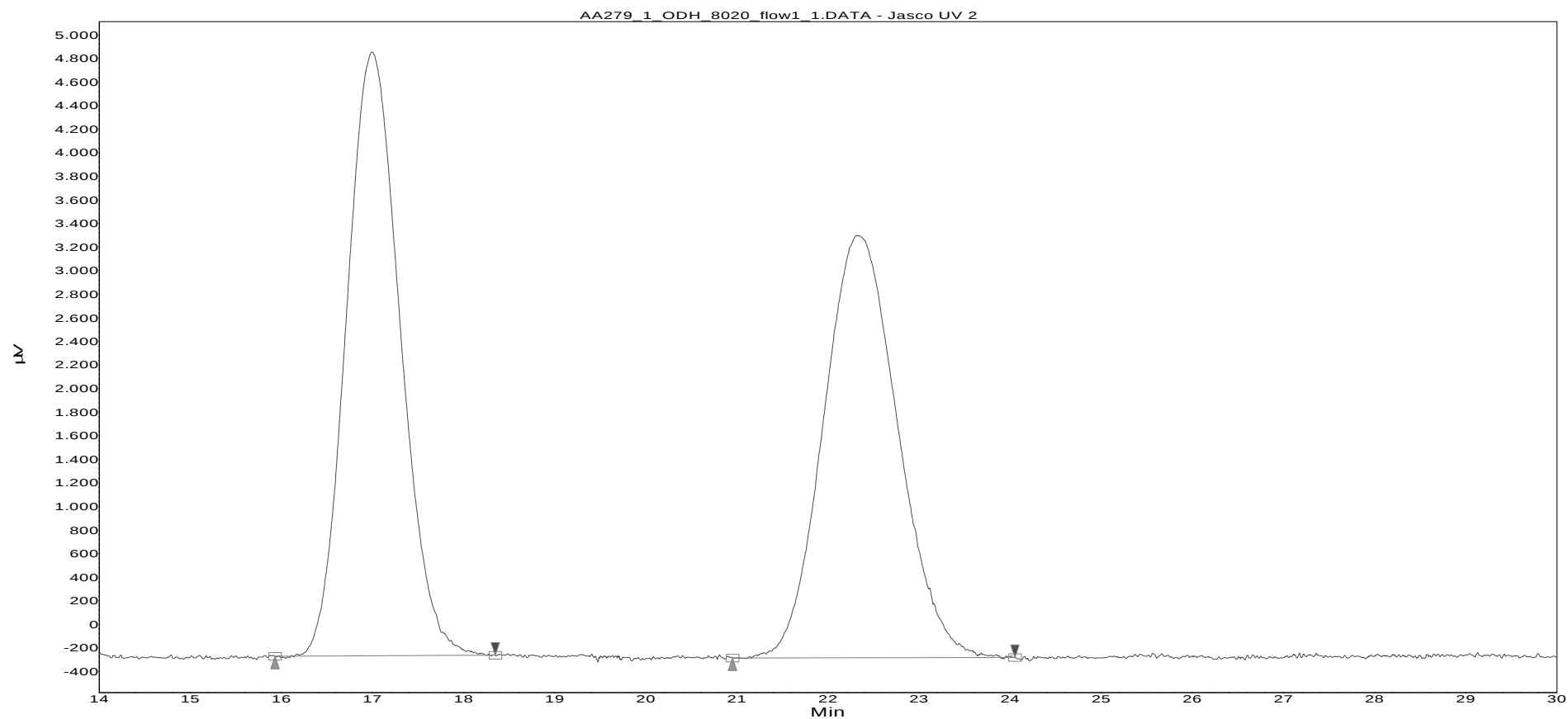
Data file: AA279_1_ODH_8020_flow1_1.DATA

Date: 01.03.2006 05:23:15

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,930	16,992	18,347	50,892
2	20,950	22,325	24,050	49,108
Total				100,000

3-(4'-bromobiphenyl-4-yl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

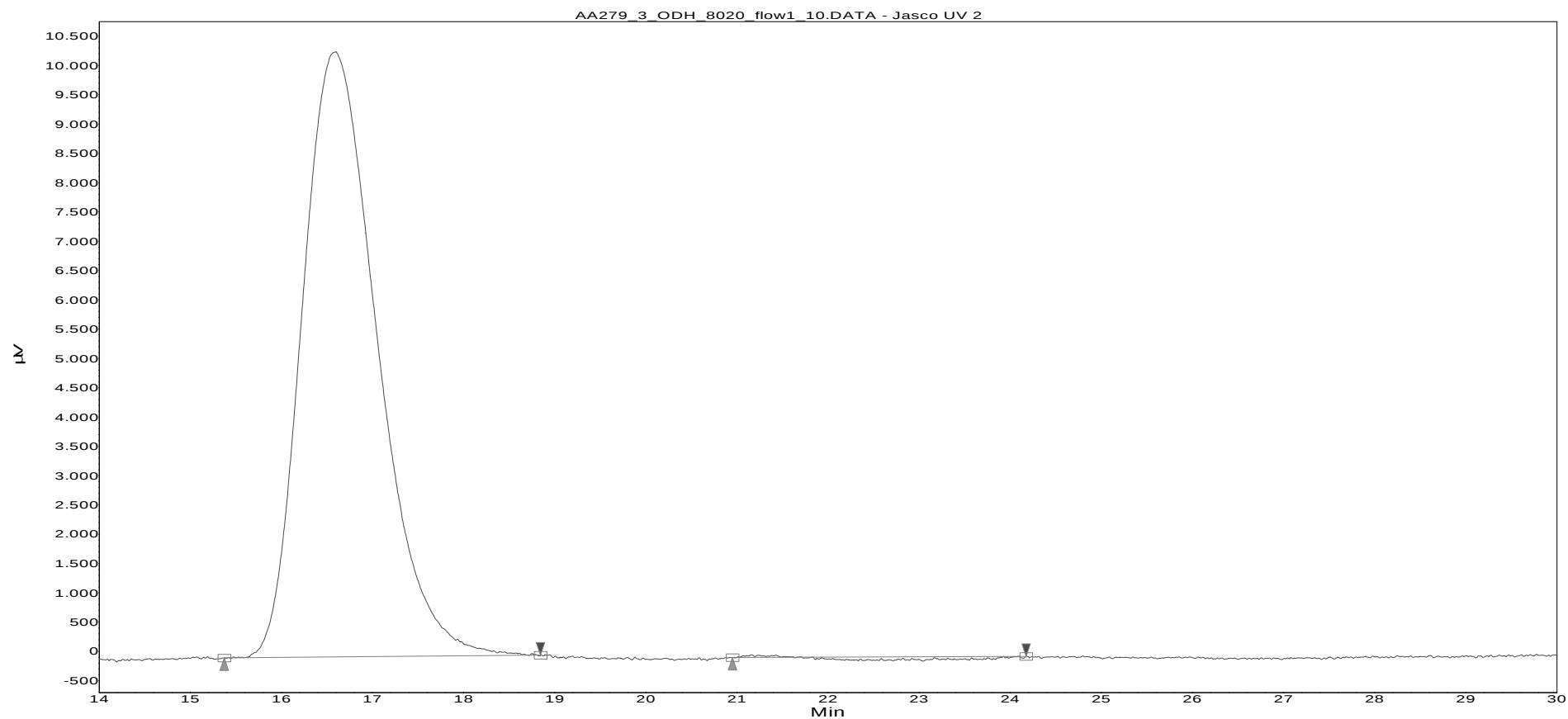
Data file: AA279_3_ODH_8020_flow1_10.DATA

Date: 16.03.2006 19:19:41

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min

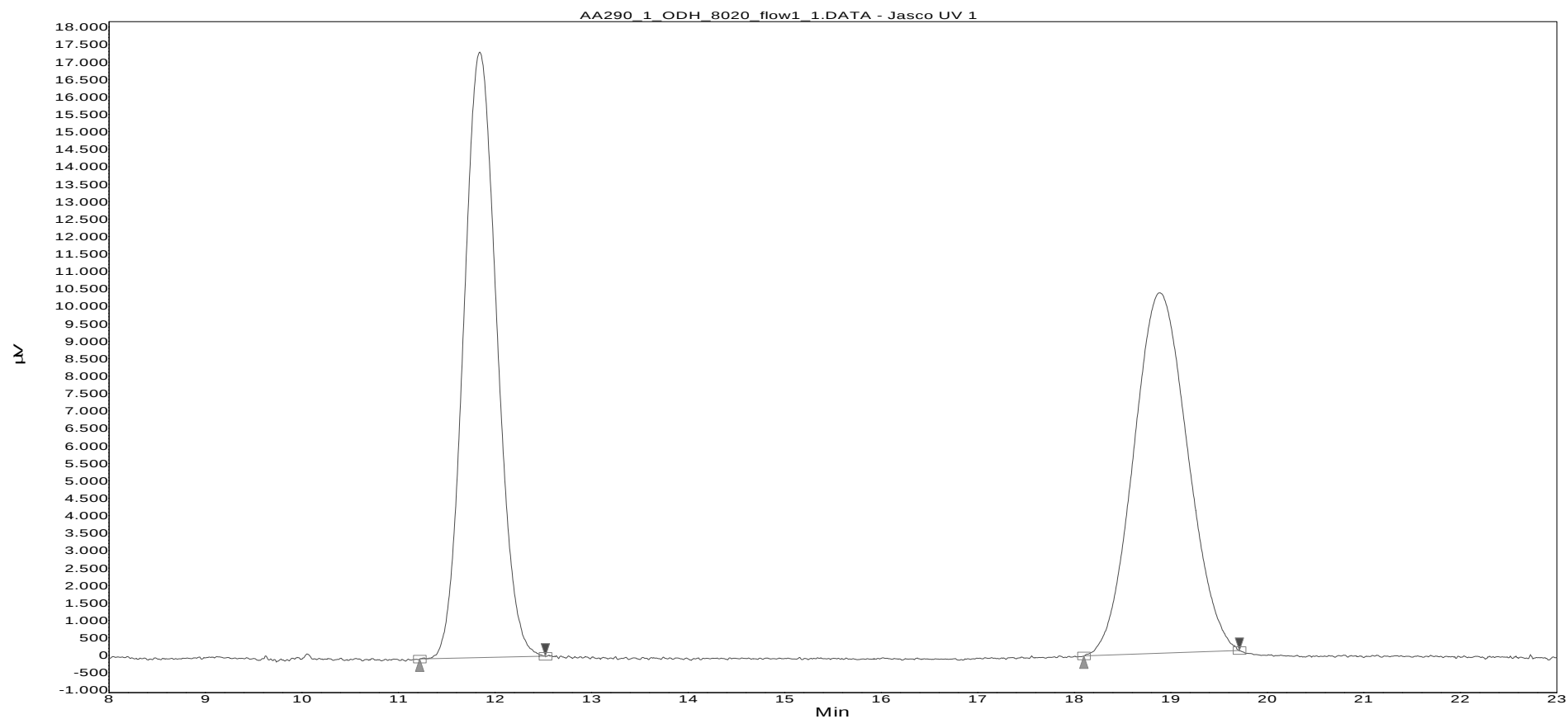


Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,372	16,592	18,843	99,177
2	20,950	23,042	24,174	0,823
Total				100,000

6-chloro-3-phenyl-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60
Data file: AA290_1_ODH_8020_flow1_1.DATA
Date: 02.03.2006 00:35:50

Column: ODH
Flow: 1.0 mL/min
Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	11,219	11,842	12,521	50,714
2	18,099	18,883	19,711	49,286
Total				100,000

6-chloro-3-phenyl-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow1_acq60

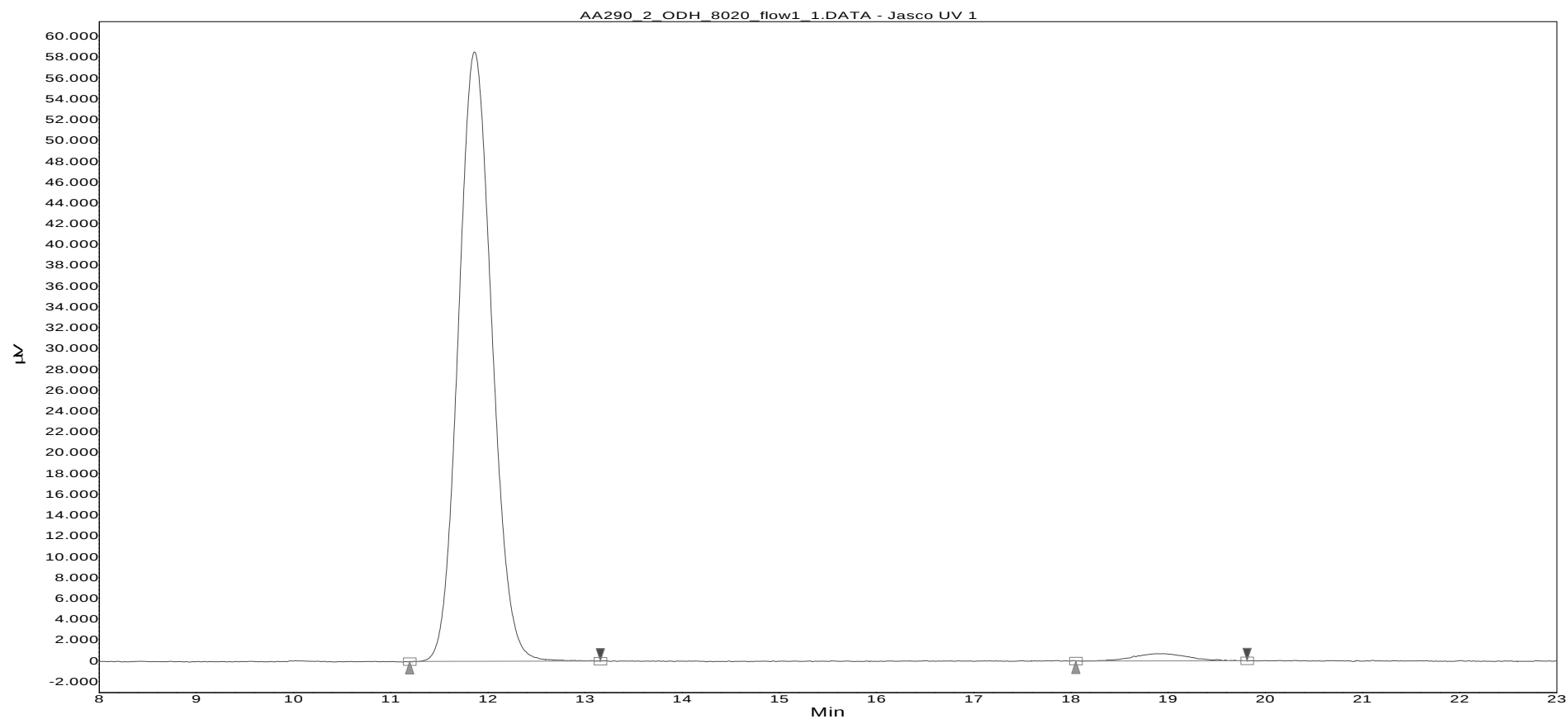
Data file: AA290_2_ODH_8020_flow1_1.DATA

Date: 02.03.2006 01:38:36

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	11,194	11,858	13,157	98,133
2	18,049	18,925	19,812	1,867
Total				100,000

N-benzyl-2-[(5-chloro-2-hydroxyphenyl)amino]-2-phenylacetamide

Method: ODH_8020_flow1_acq60

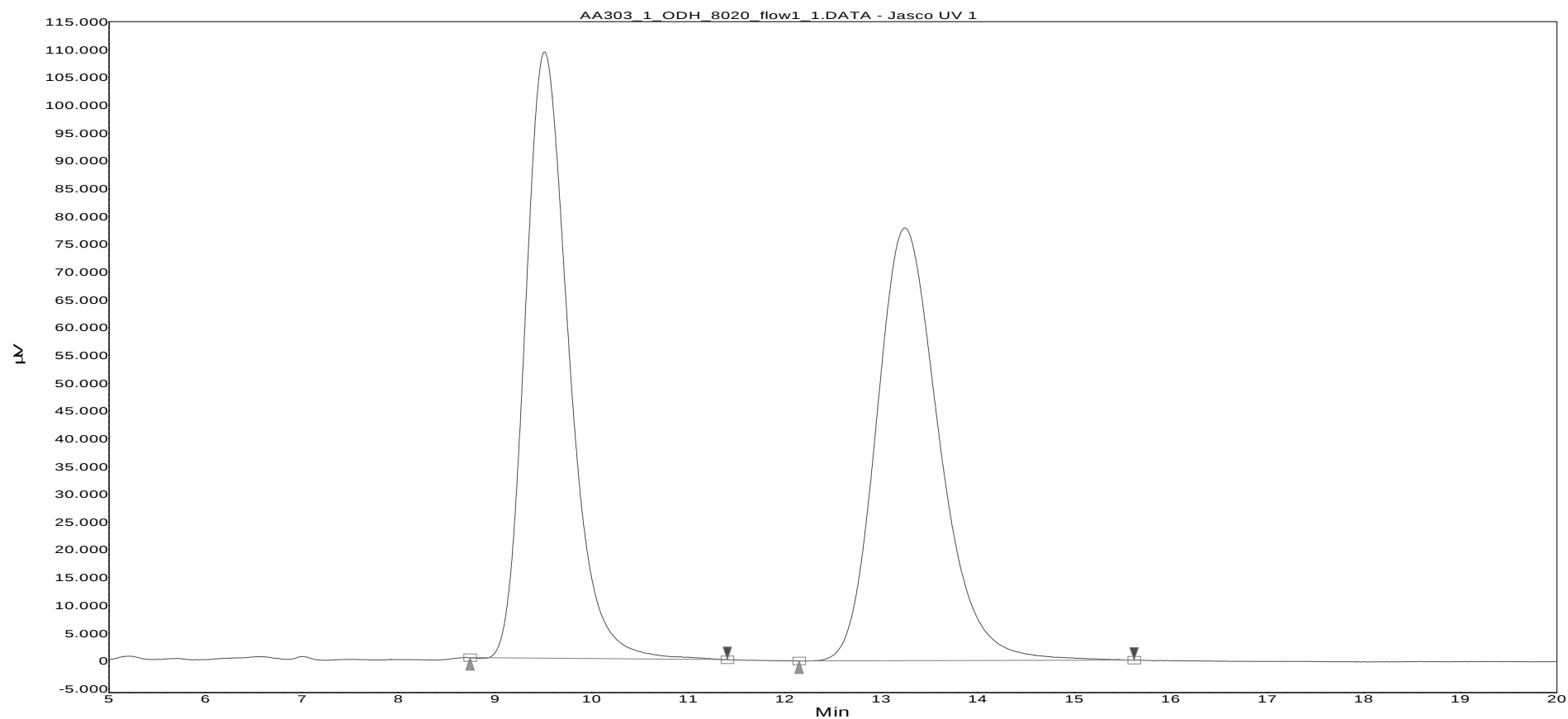
Data file: AA303_1_ODH_8020_flow1_1.DATA

Date: 30.03.2006 23:34:30

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,740	9,508	11,405	49,525
2	12,149	13,242	15,620	50,475
Total				100,000

N-benzyl-2-[(5-chloro-2-hydroxyphenyl)amino]-2-phenylacetamide

Method: ODH_8020_flow1_acq60

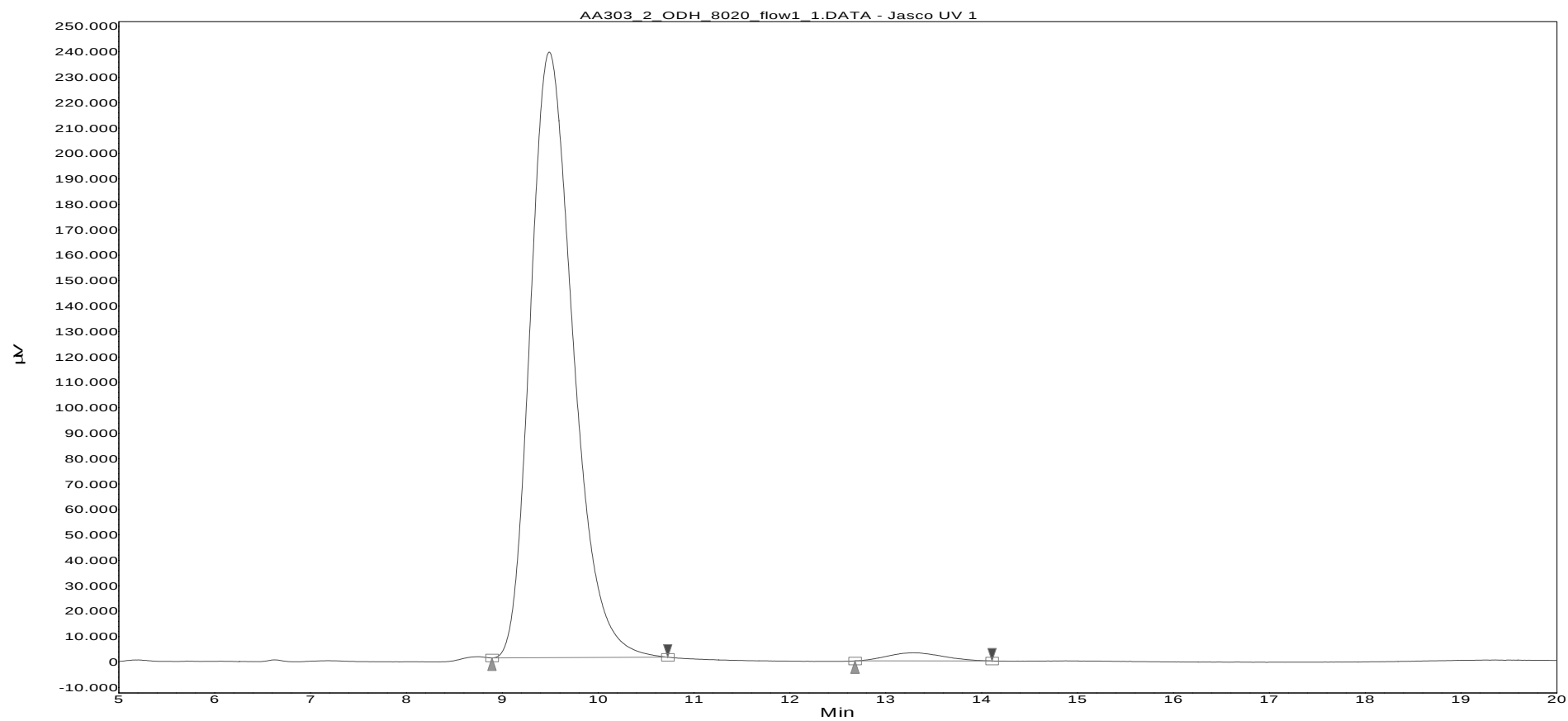
Data file: AA303_2_ODH_8020_flow1_1.DATA

Date: 31.03.2006 00:37:12

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	8,892	9,492	10,726	98,291
2	12,679	13,283	14,108	1,709
Total				100,000

3-(2-thienyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow06_acq50

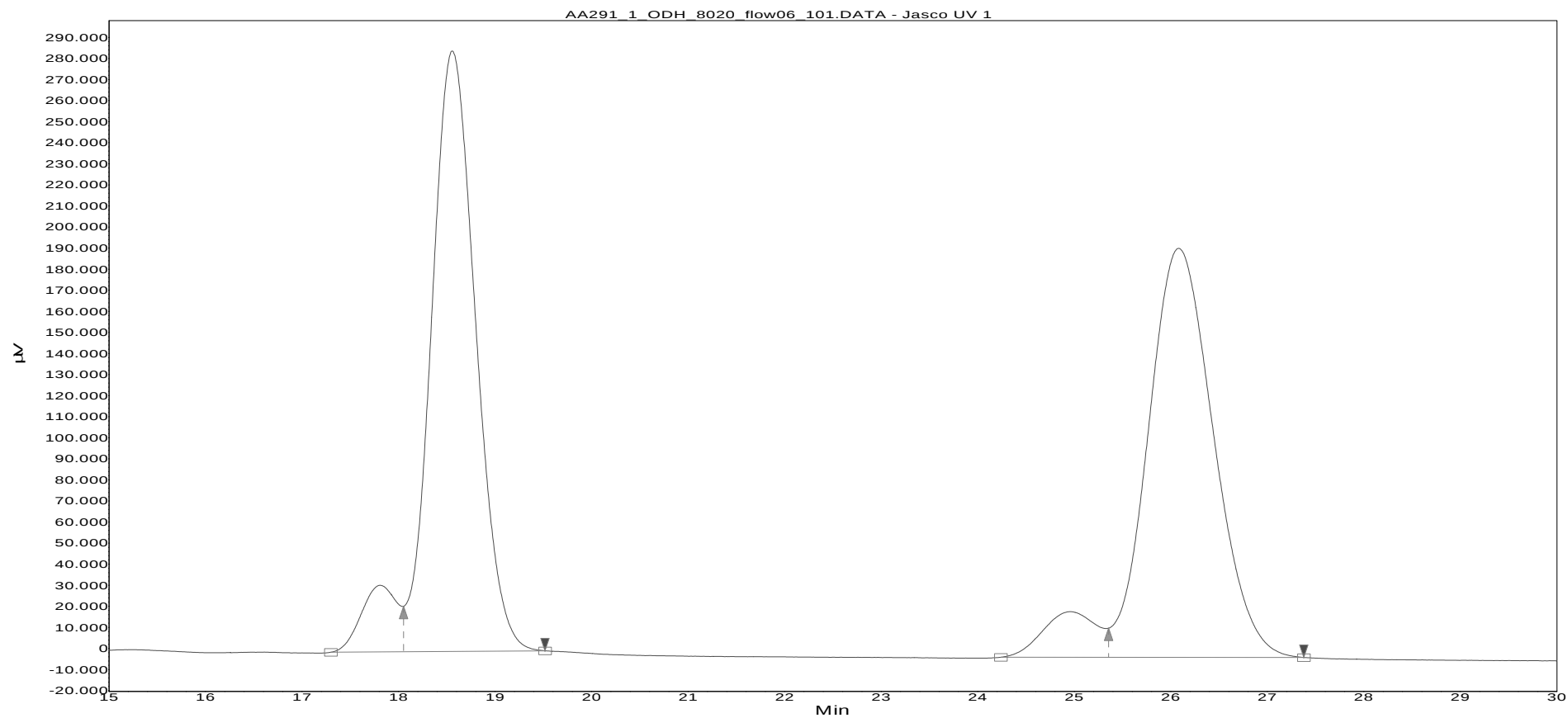
Data file: AA291_1_ODH_8020_flow06_101.DATA

Date: 16.03.2006 13:33:16

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,052	18,558	19,516	49,946
2	25,355	26,083	27,377	50,054
Total				100,000

3-(2-thienyl)-3,4-dihydro-2H-1,4-benzoxazin-2-one

Method: ODH_8020_flow06_acq50

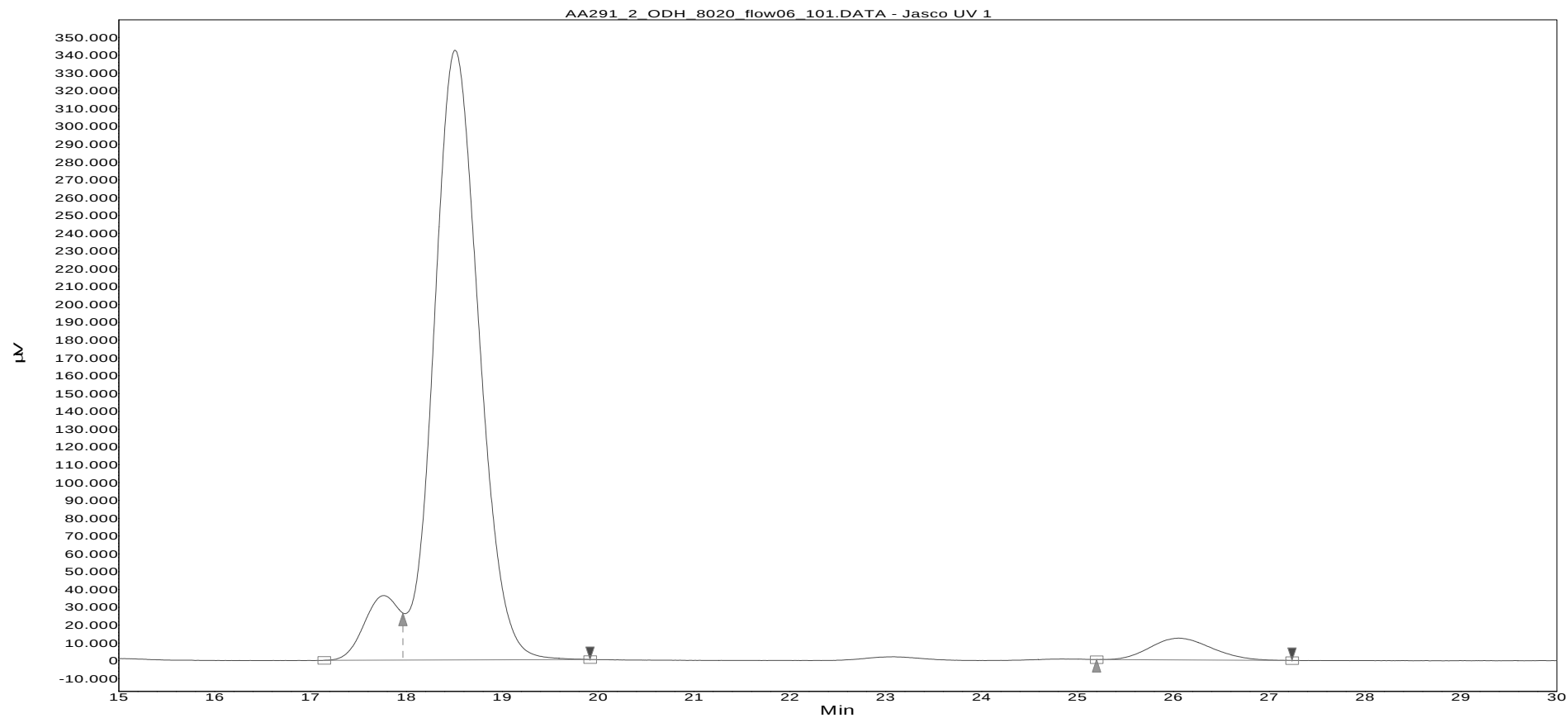
Data file: AA291_2_ODH_8020_flow06_101.DATA

Date: 16.03.2006 14:25:59

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	17,964	18,508	19,915	95,320
1	25,198	26,058	27,237	4,680
Total				100,000

3-biphenyl-4-yl-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow1_acq50

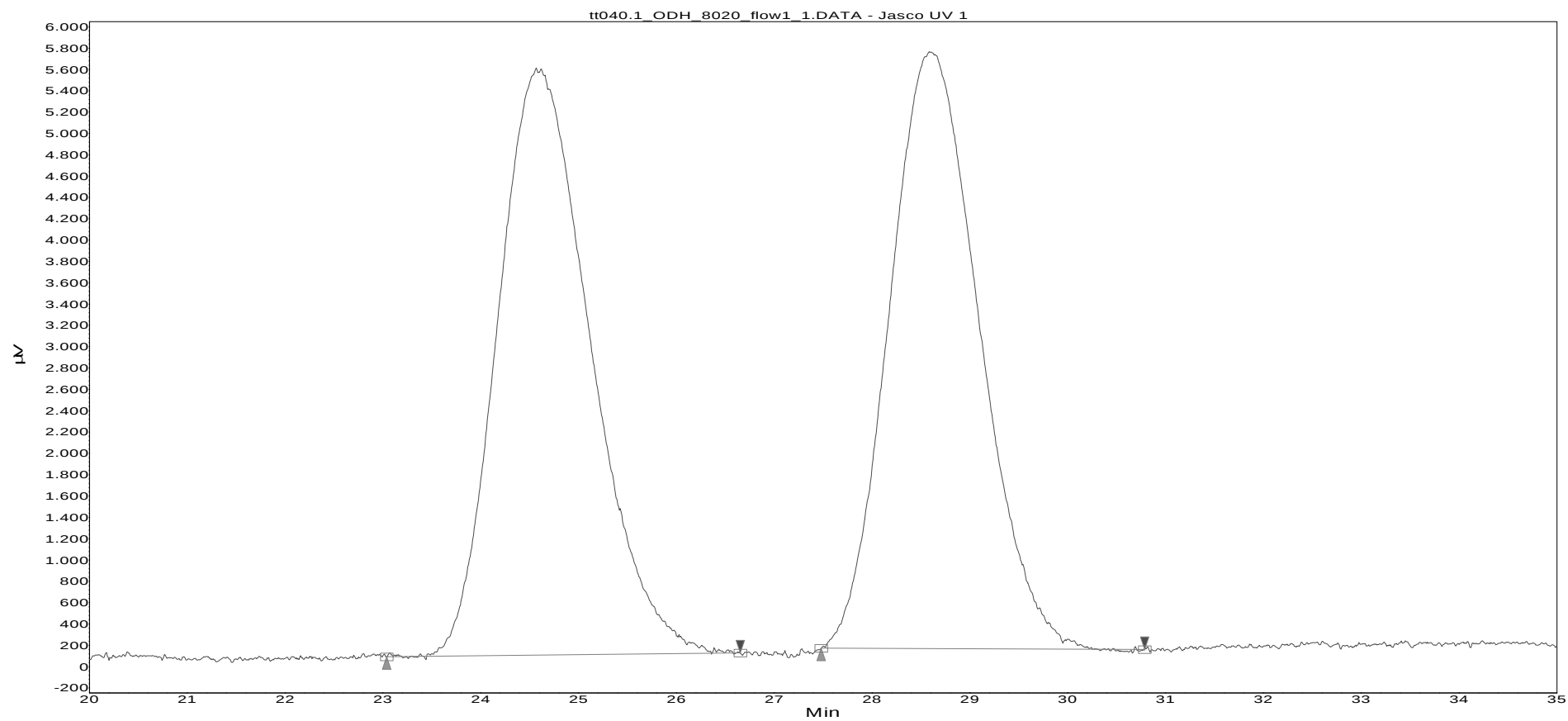
Data file: tt040.1_ODH_8020_flow1_1.DATA

Date: 28.03.2006 22:33:31

Column: ODH

Flow: 1.0 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	23,037	24,558	26,653	50,288
2	27,479	28,600	30,785	49,712
Total				100,000

3-biphenyl-4-yl-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow1_acq50

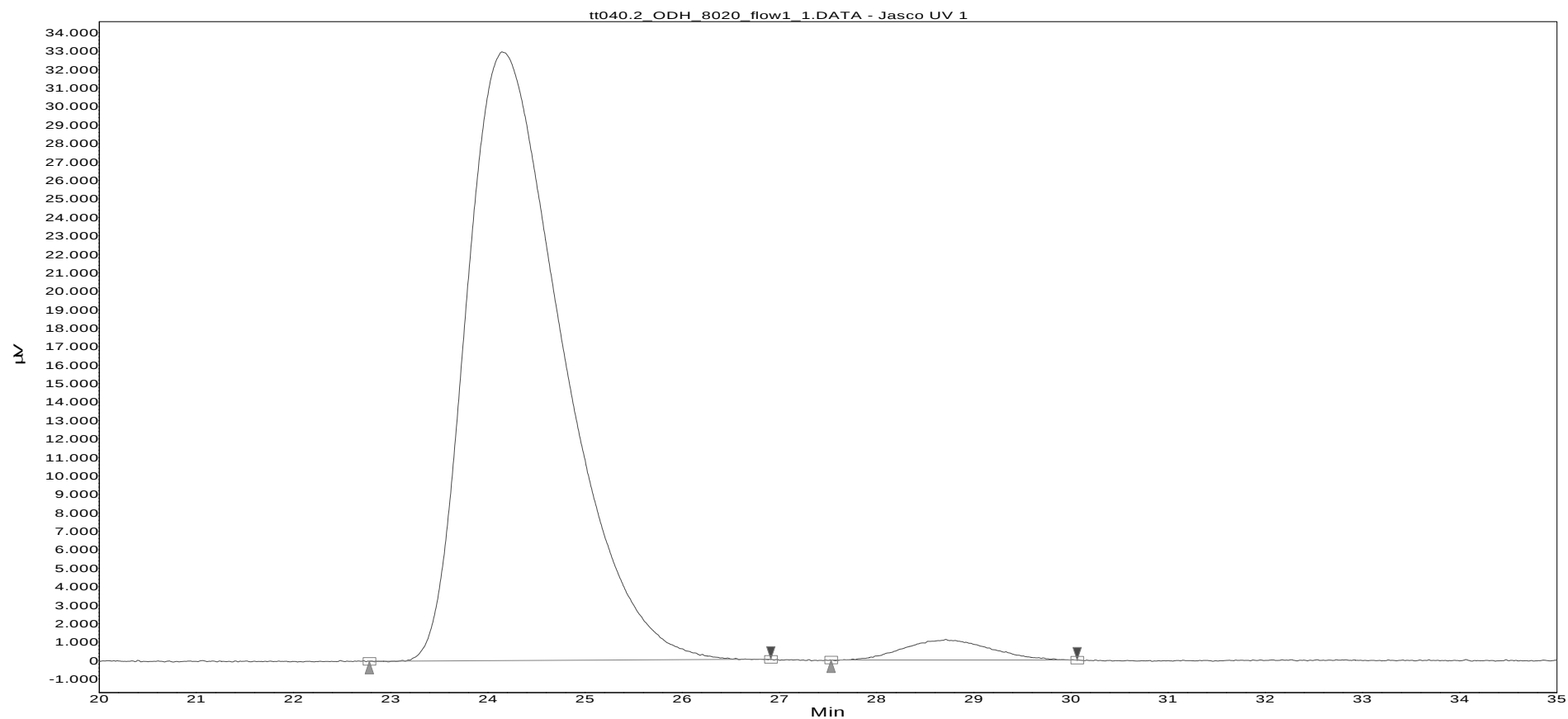
Data file: tt040.2_ODH_8020_flow1_1.DATA

Date: 28.03.2006 23:26:16

Column: ODH

Flow: 1.0 mL/min

Run time: 50,00 min

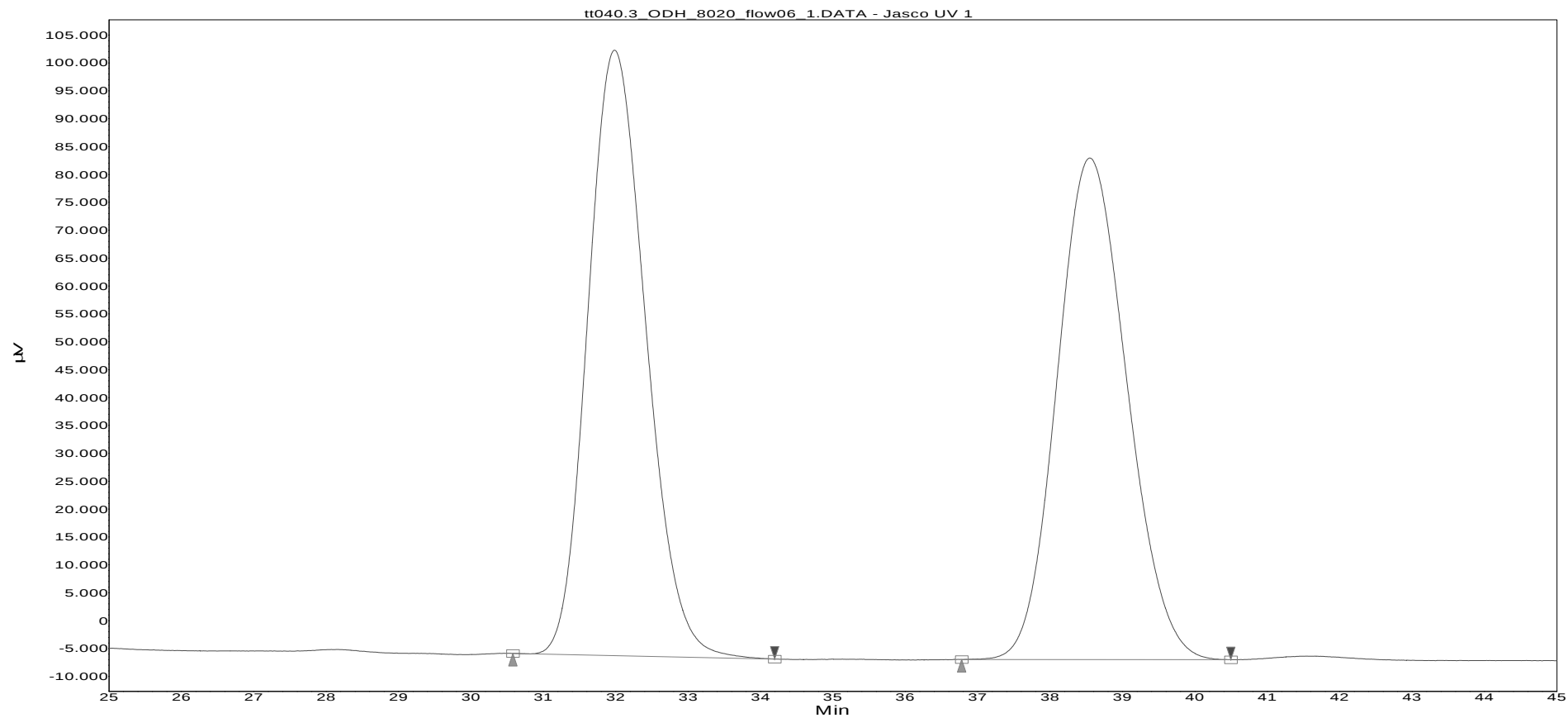


Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	22,779	24,150	26,911	97,105
2	27,531	28,708	30,062	2,895
Total				100,000

3-(4-bromophenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow06_acq50
Data file: tt040.3_ODH_8020_flow06_1.DATA
Date: 29.03.2006 00:18:58

Column: ODH
Flow: 0.6 mL/min
Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	30,579	31,983	34,194	50,049
2	36,777	38,550	40,496	49,951
Total				100,000

3-(4-bromophenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow06_acq50

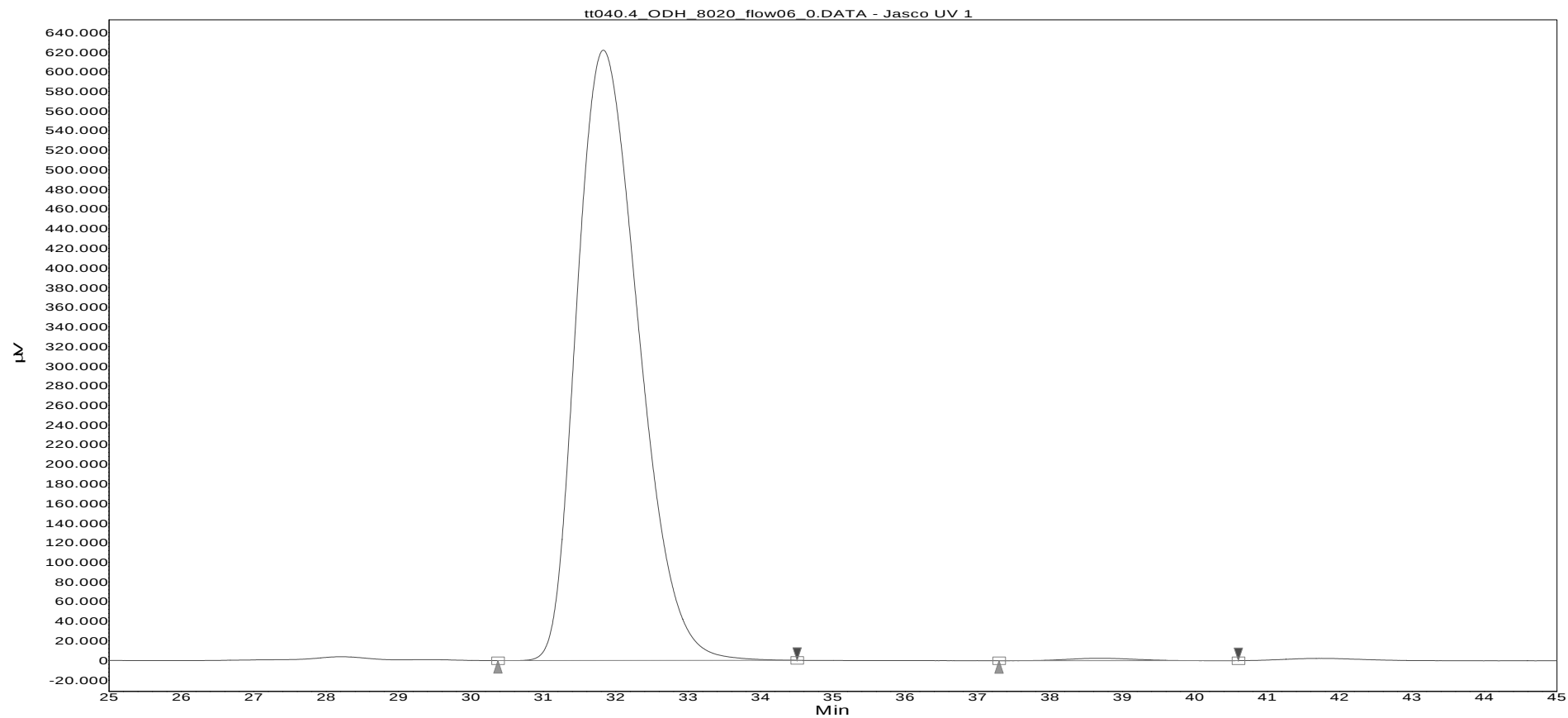
Data file: tt040.4_ODH_8020_flow06_0.DATA

Date: 29.03.2006 01:11:41

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index		Start	Time	End	Area %
		[Min]	[Min]	[Min]	[%]
1		30,372	31,825	34,504	99,566
2		37,293	38,683	40,599	0,434
Total					100,000

3-(4-fluorophenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow06_acq50

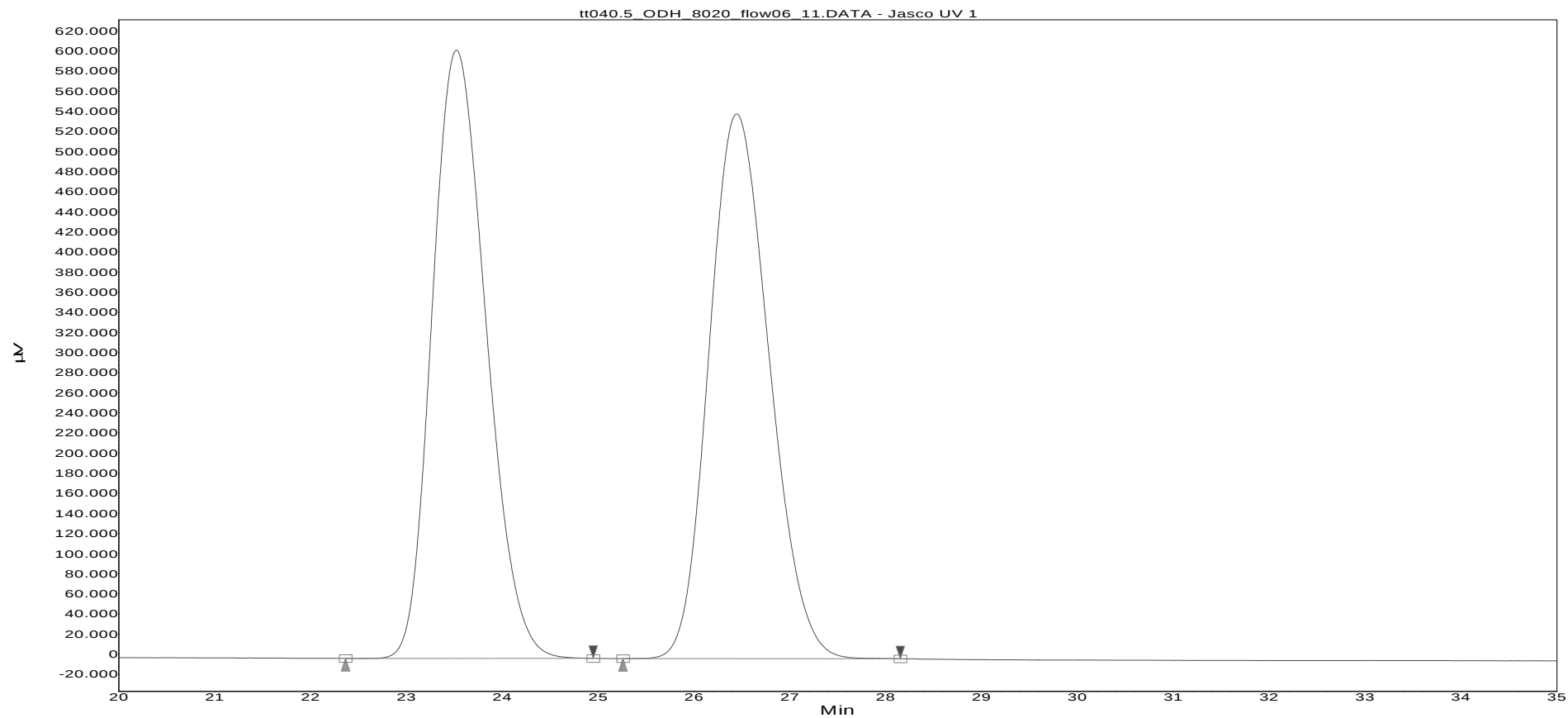
Data file: tt040.5_ODH_8020_flow06_11.DATA

Date: 05.04.2006 12:13:33

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	22,366	23,517	24,948	50,032
2	25,258	26,442	28,151	49,968
Total				100,000

3-(4-fluorophenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow06_acq50

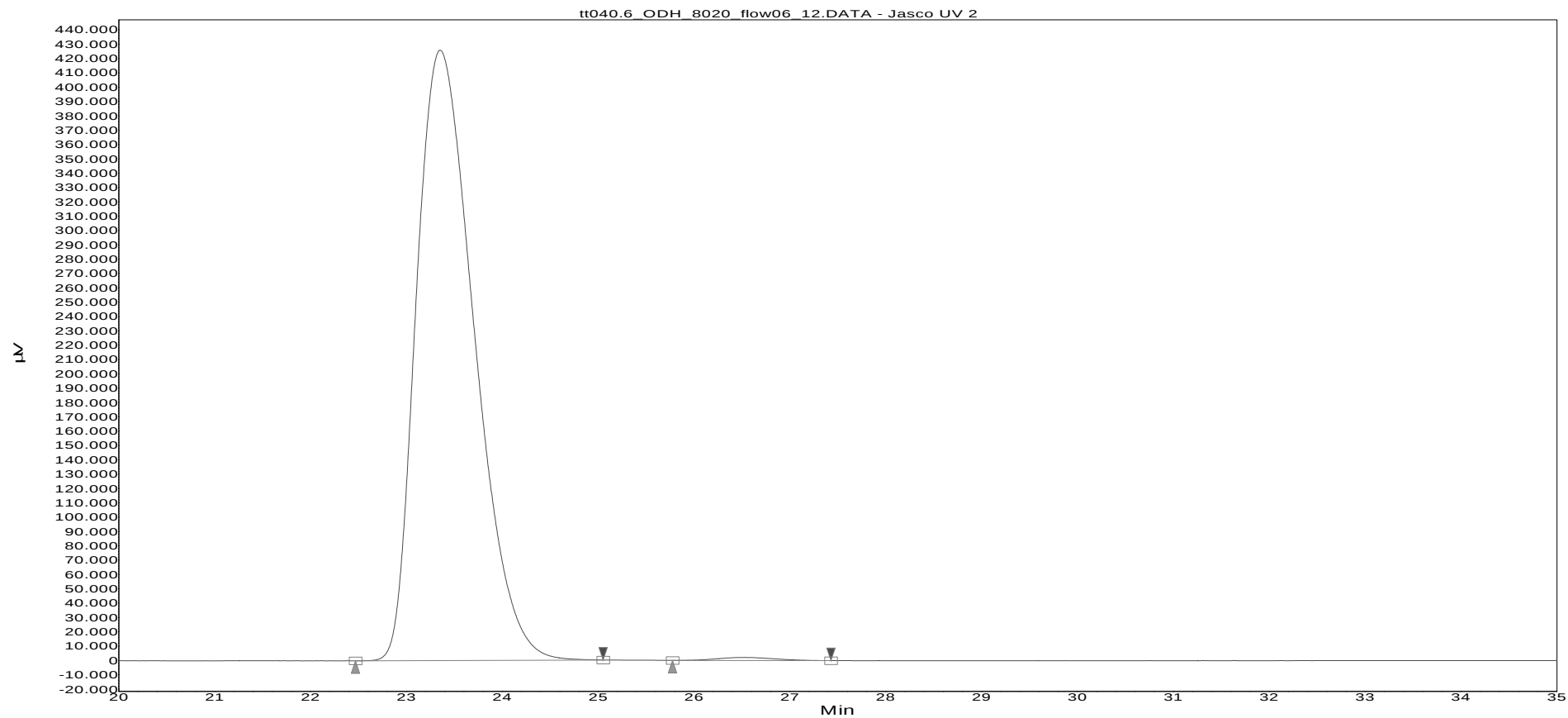
Data file: tt040.6_ODH_8020_flow06_12.DATA

Date: 05.04.2006 13:06:16

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	22,469	23,350	25,052	99,516
2	25,775	26,525	27,428	0,484
Total				100,000

3-(4-methylphenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_9010_flow06_acq50

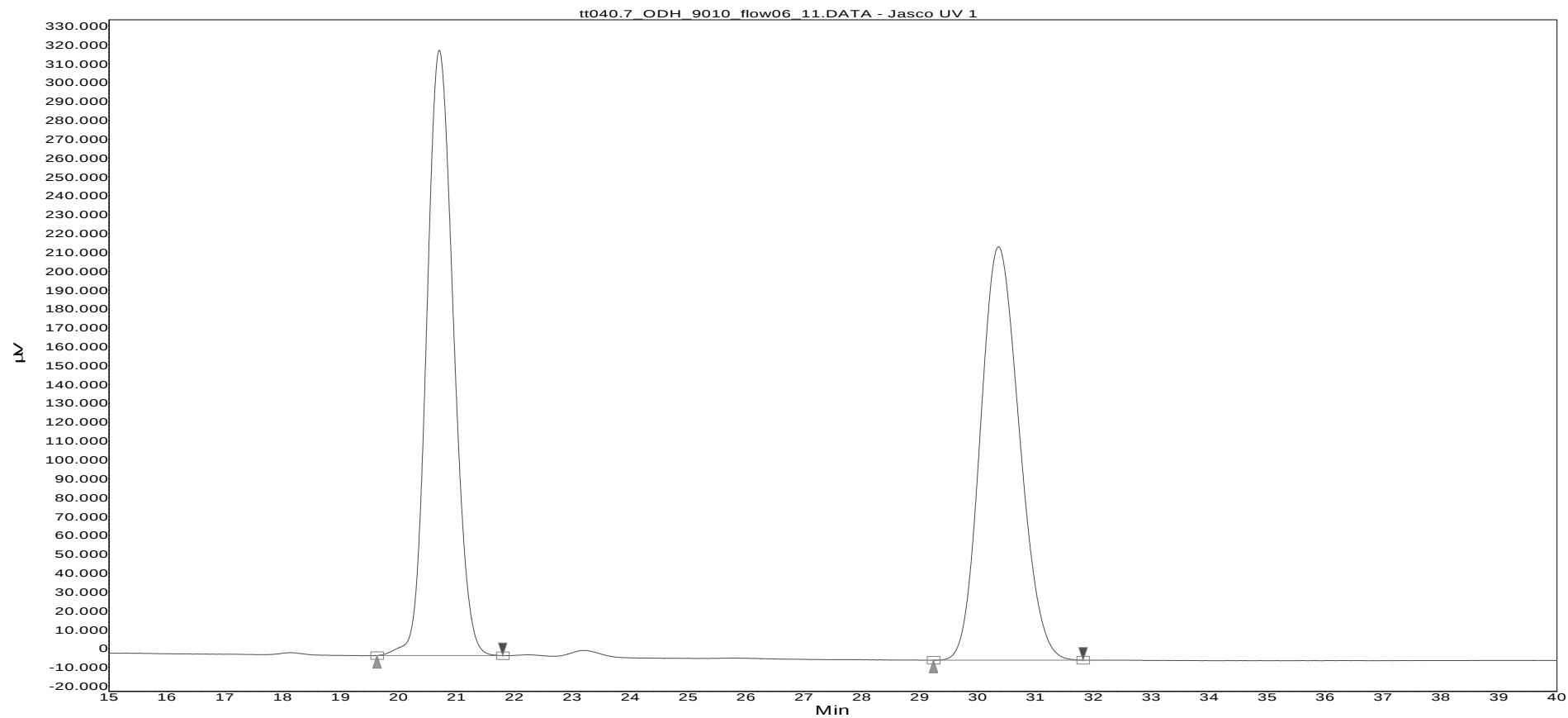
Data file: tt040.7_ODH_9010_flow06_11.DATA

Date: 21.04.2006 18:42:17

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,628	20,700	21,798	50,228
2	29,236	30,358	31,818	49,772
Total				100,000

3-(4-methylphenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_9010_flow06_acq50

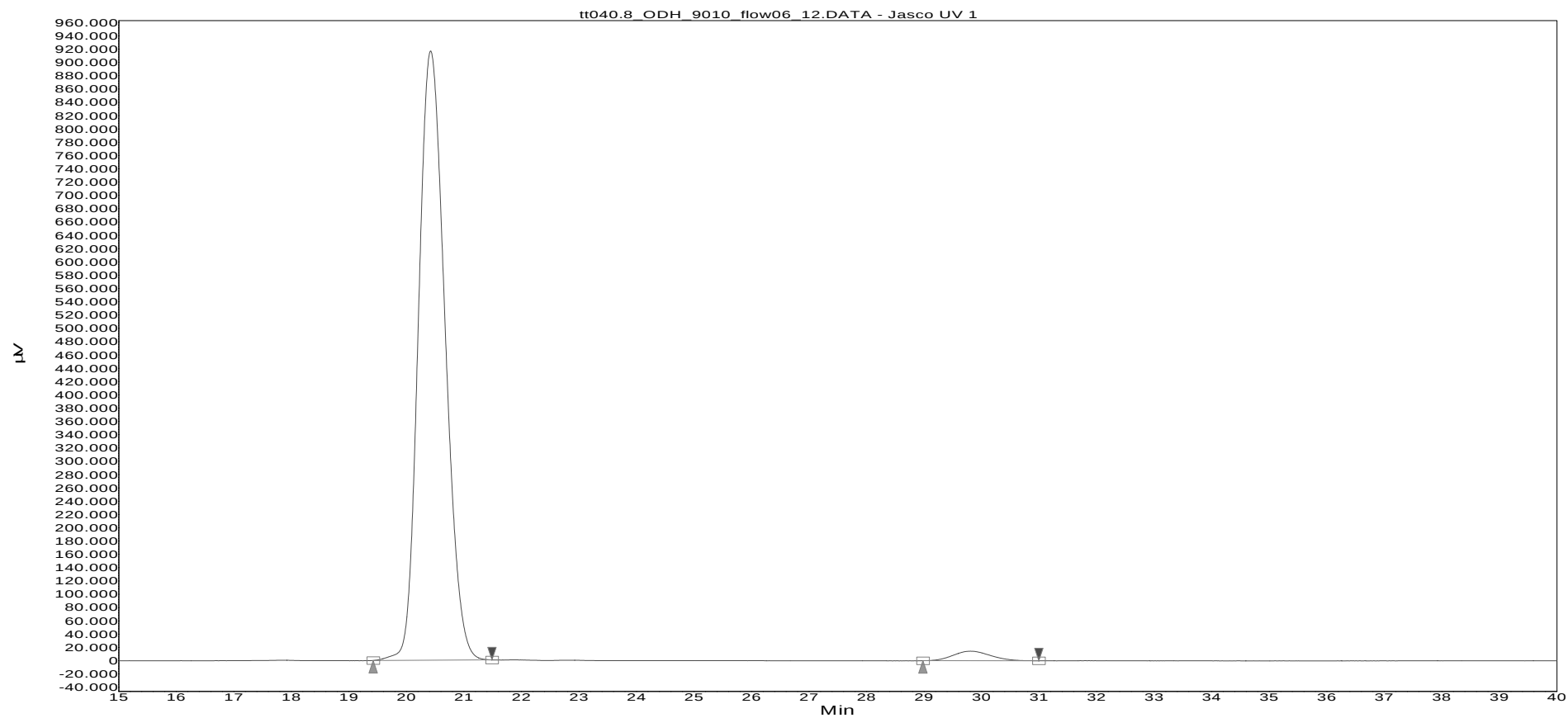
Data file: tt040.8_ODH_9010_flow06_12.DATA

Date: 21.04.2006 19:35:02

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min

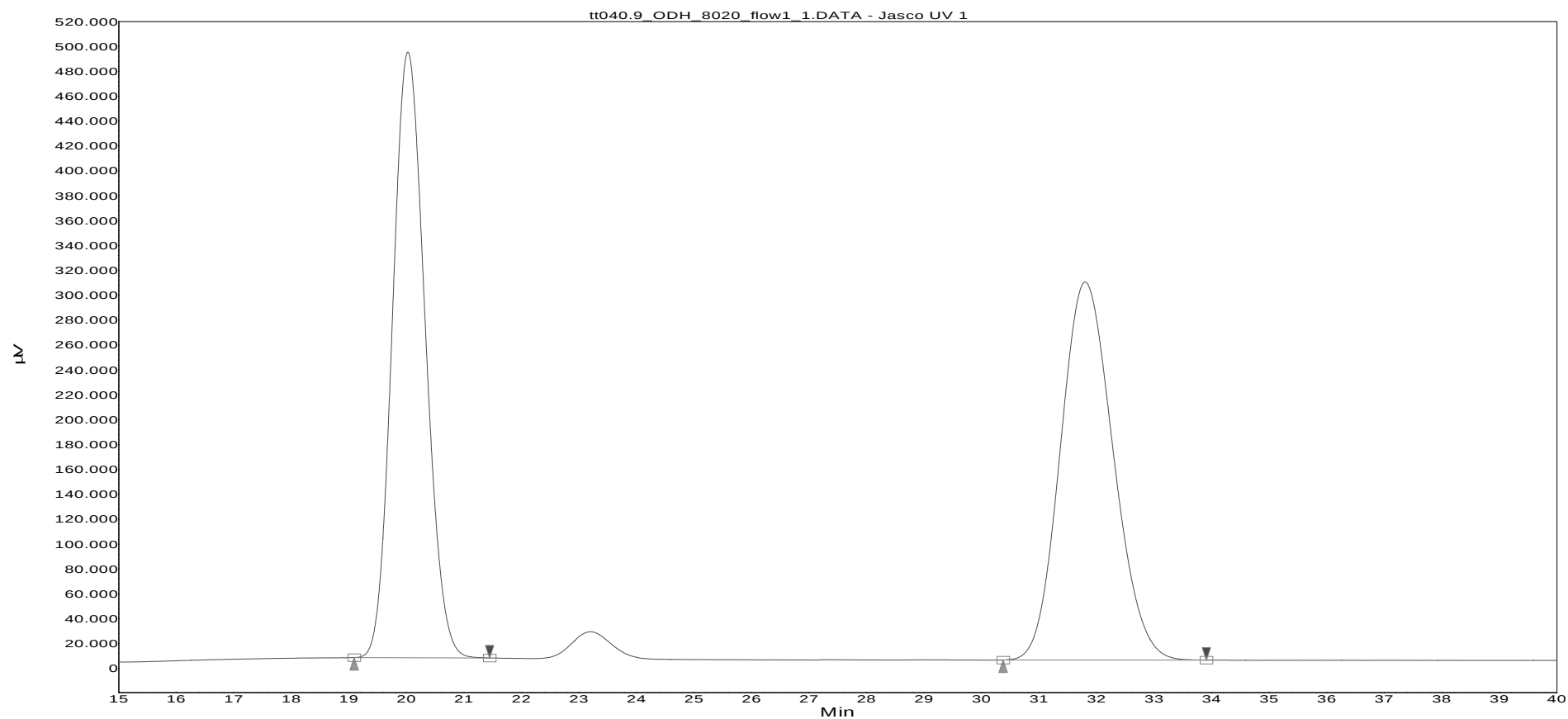


Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,421	20,425	21,488	97,843
2	28,977	29,800	30,992	2,157
Total				100,000

3-(2-naphthyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow1_acq60
Data file: tt040.9_ODH_8020_flow1_1.DATA
Date: 05.04.2006 17:29:57

Column: ODH
Flow: 1.0 mL/min
Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	19,091	20,025	21,446	49,867
2	30,372	31,800	33,905	50,133
Total				100,000

3-(2-naphthyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow1_acq60

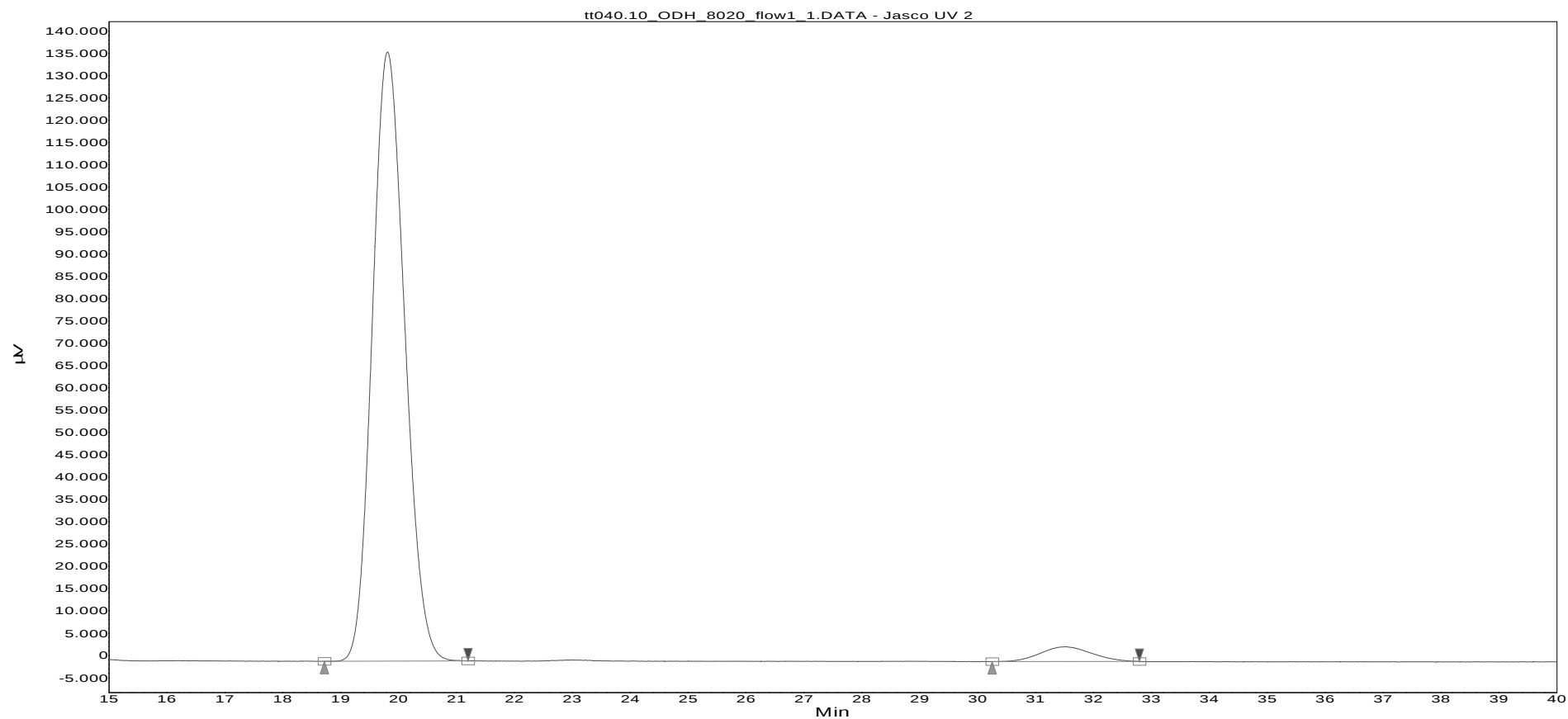
Data file: tt040.10_ODH_8020_flow1_1.DATA

Date: 05.04.2006 18:32:43

Column: ODH

Flow: 1.0 mL/min

Run time: 60,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,719	19,808	21,198	96,349
2	30,248	31,508	32,789	3,651
Total				100,000

3-(3-bromophenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow06_acq50

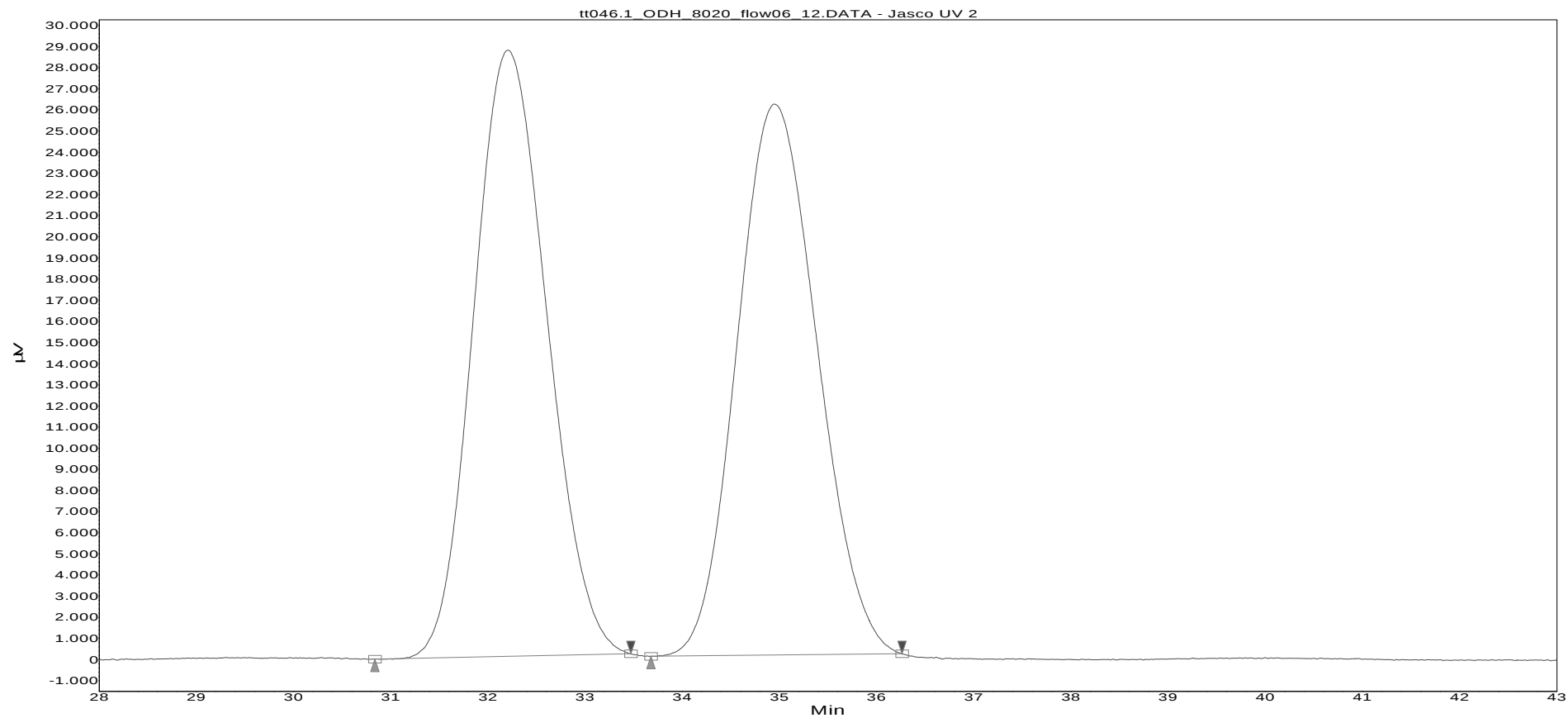
Data file: tt046.1_ODH_8020_flow06_12.DATA

Date: 11.04.2006 16:28:51

Column: ODH

Flow: 0.6 mL/min

Run time: 50,00 min



Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	30,837	32,208	33,471	50,231
2	33,678	34,950	36,260	49,769
Total				100,000

3-(3-bromophenyl)-3,4-dihydro-2H-1,4-benzthiazine

Method: ODH_8020_flow06_acq50

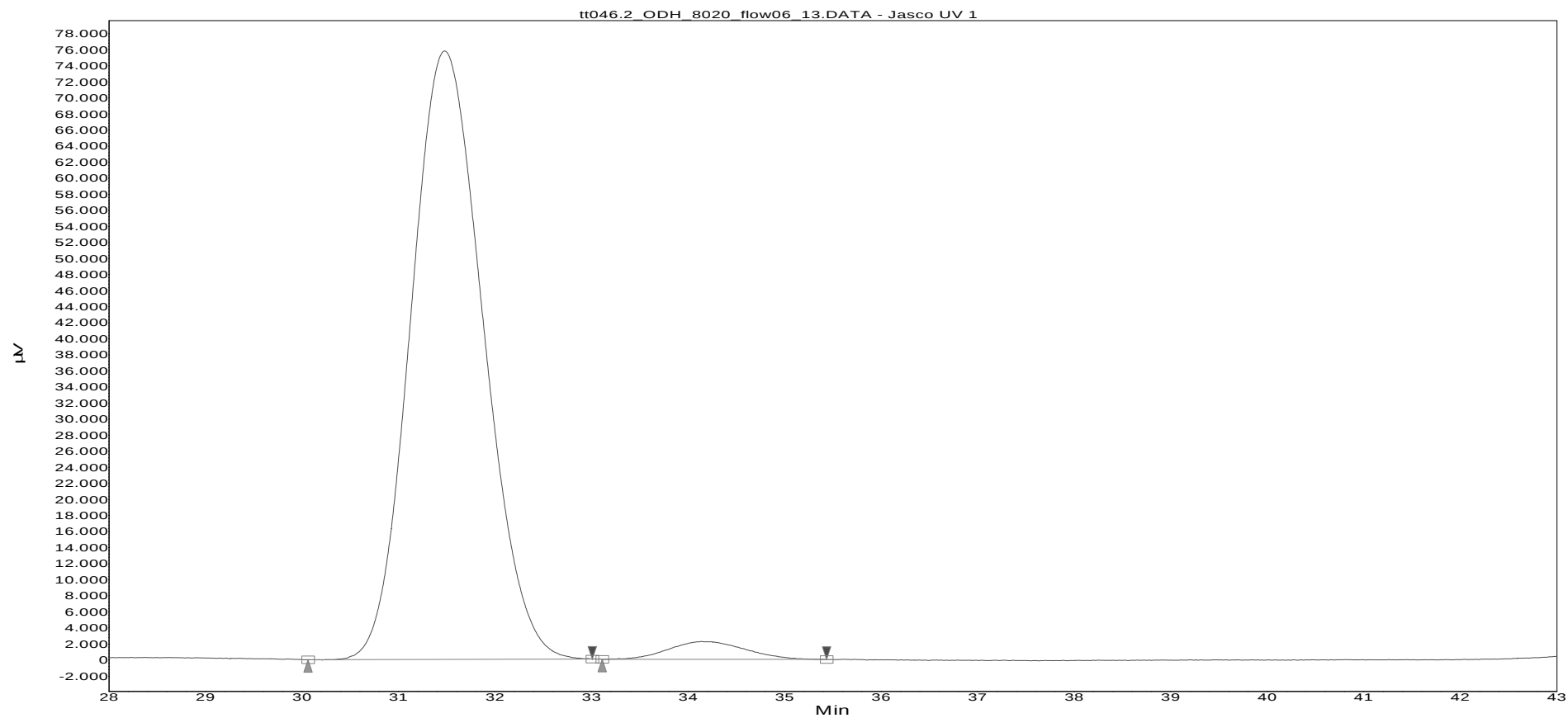
Data file: tt046.2_ODH_8020_flow06_13.DATA

Date: 11.04.2006 17:21:36

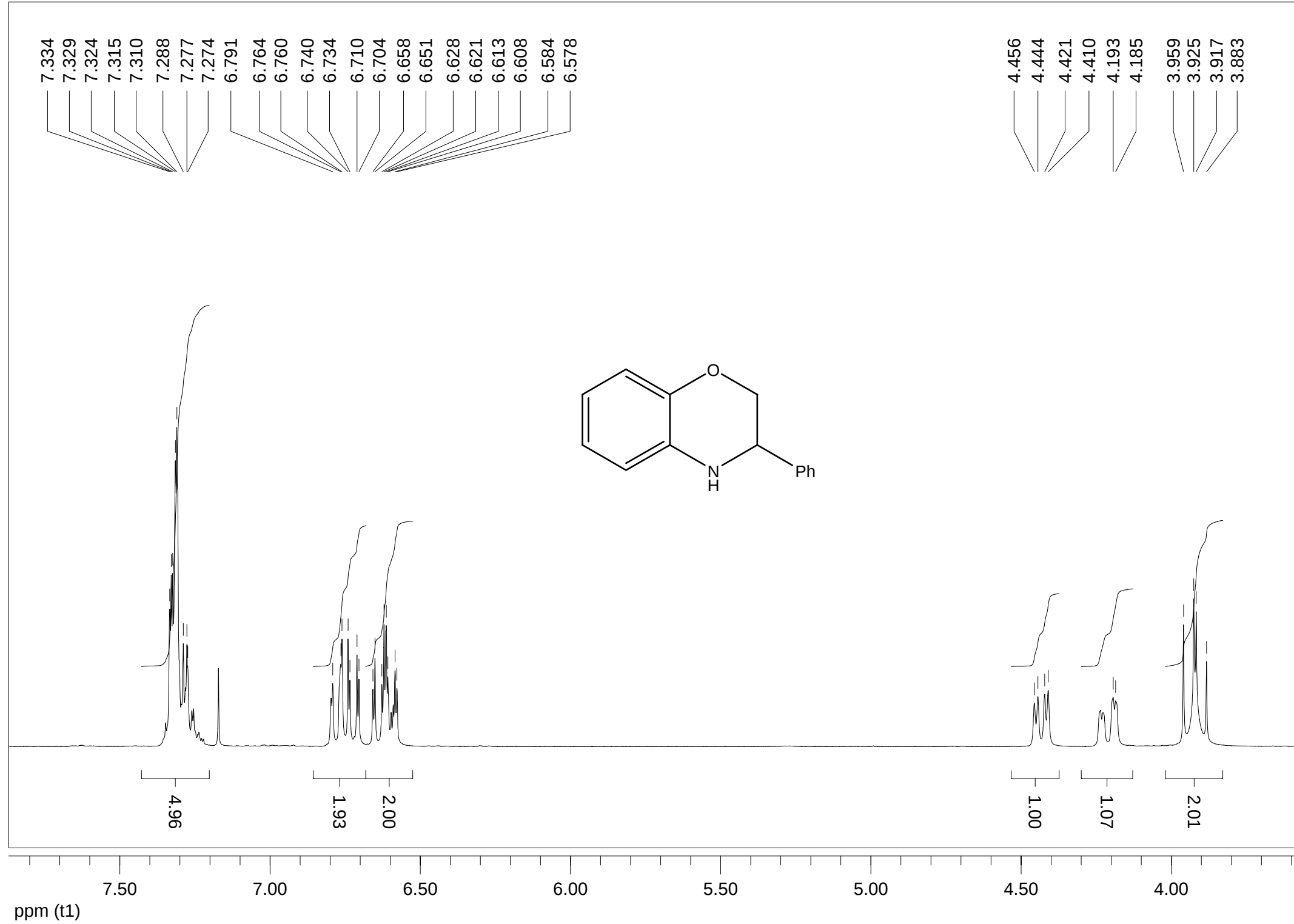
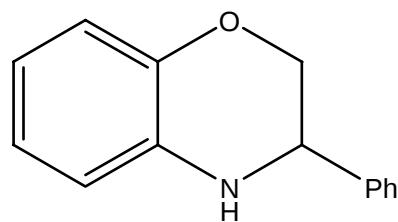
Column: ODH

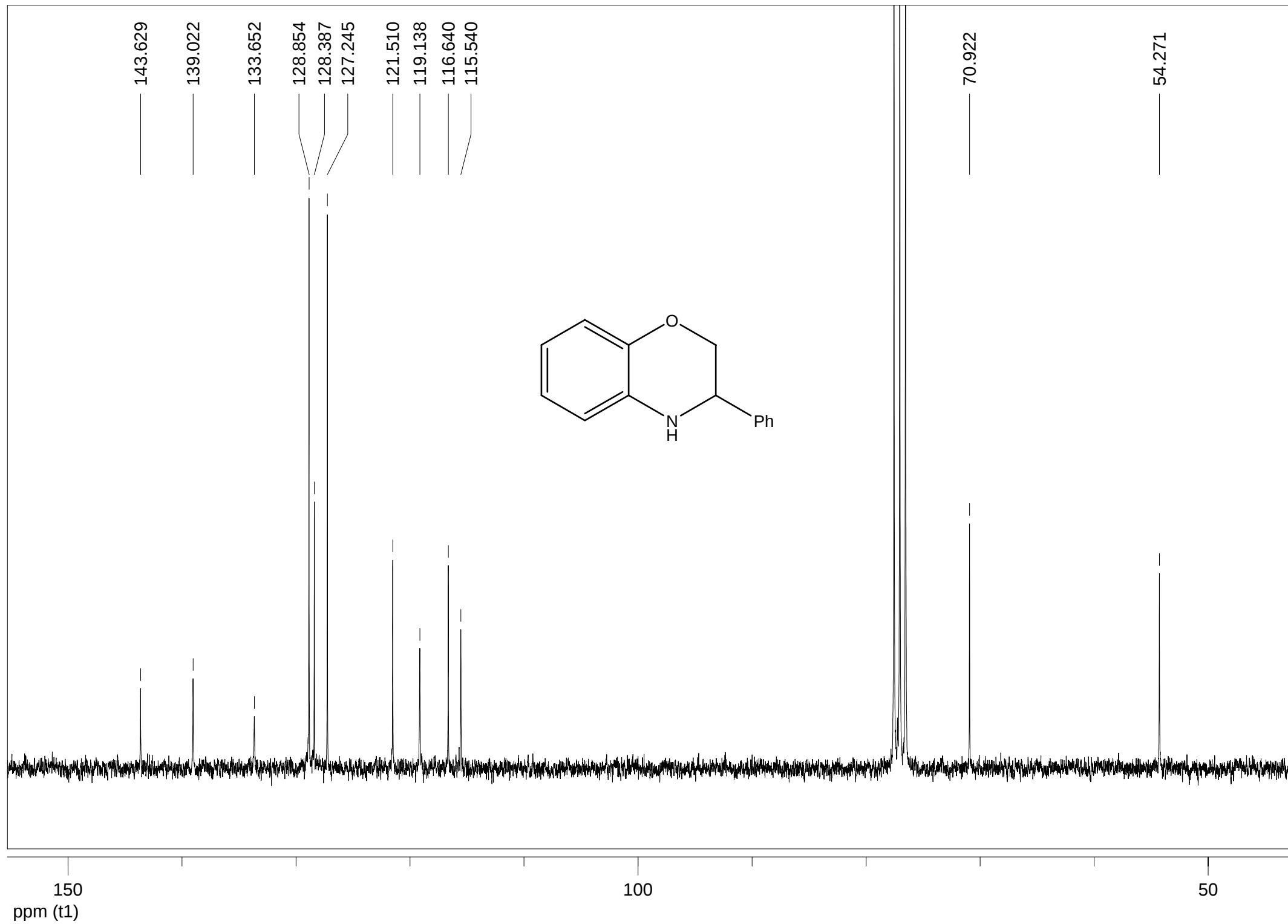
Flow: 0.6 mL/min

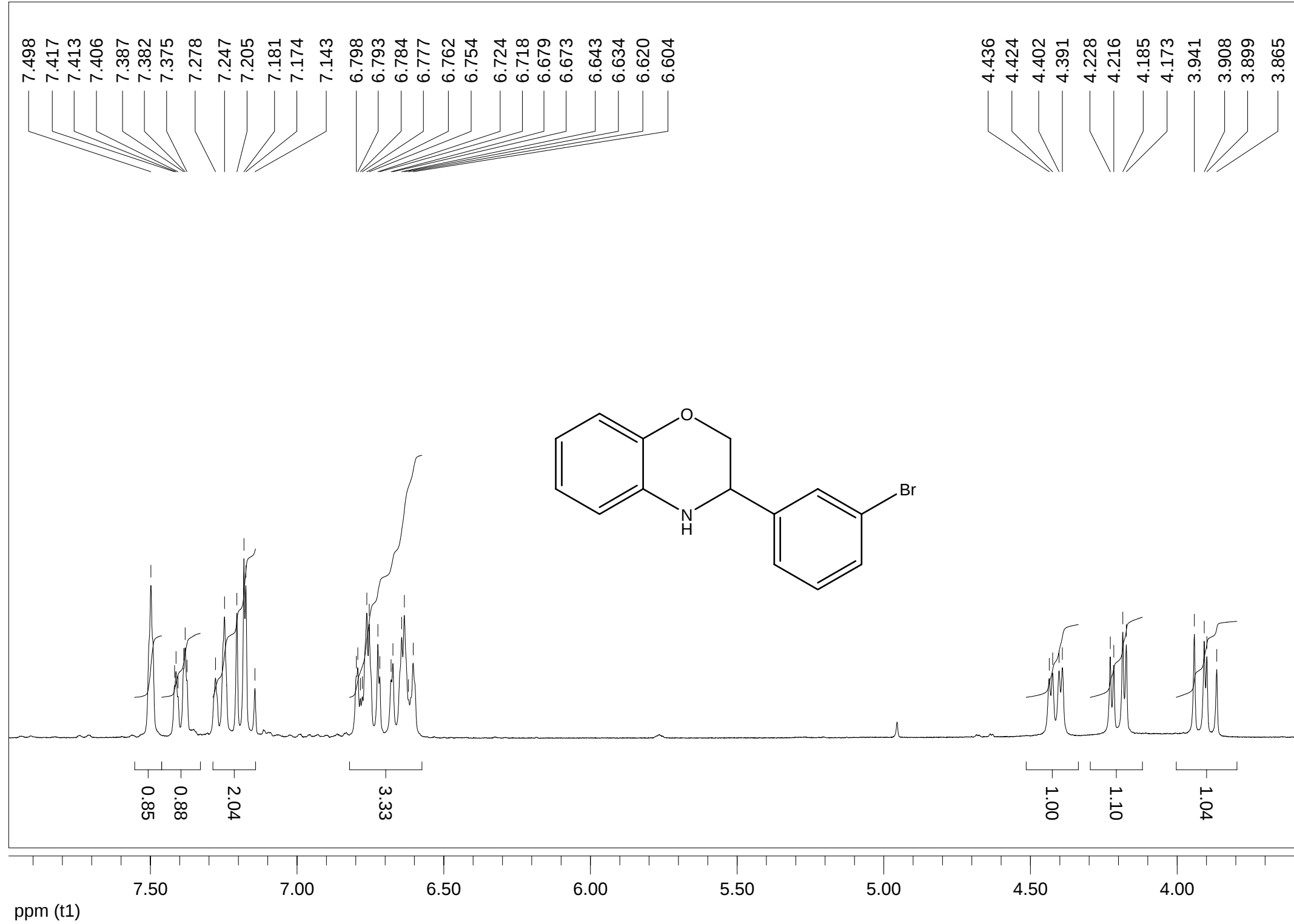
Run time: 50,00 min

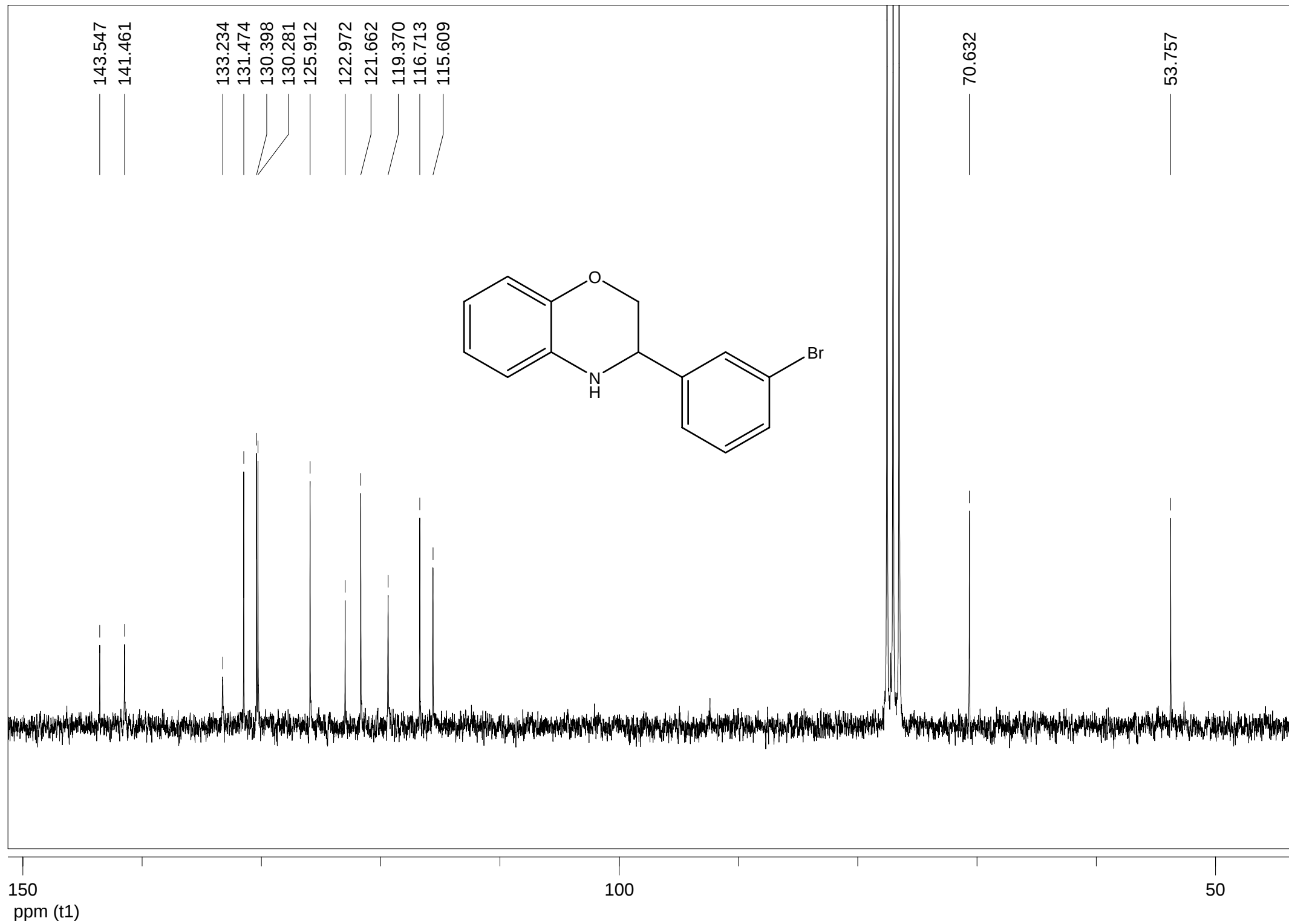


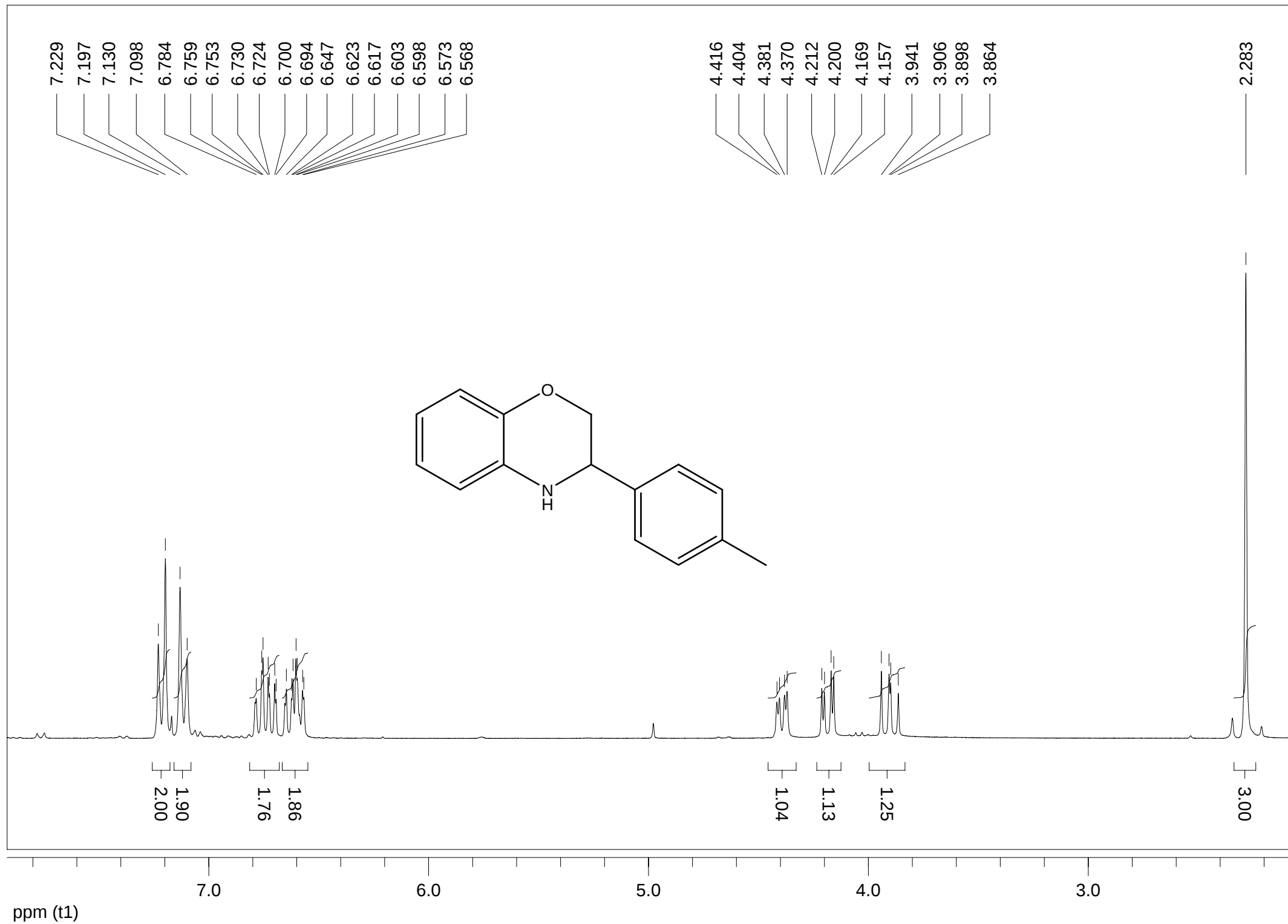
Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	30,062	31,475	33,006	97,007
2	33,110	34,142	35,434	2,993
Total				100,000

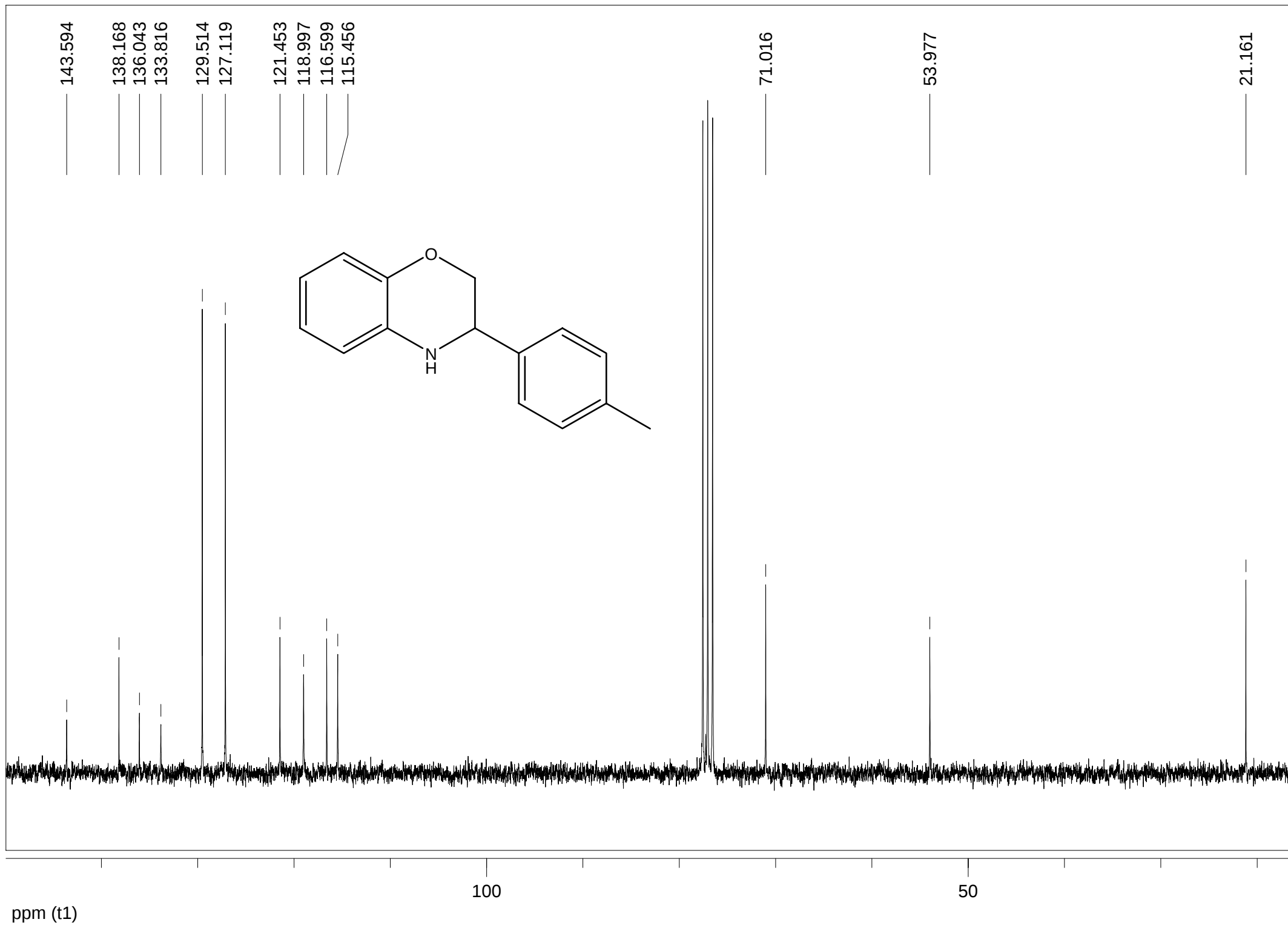


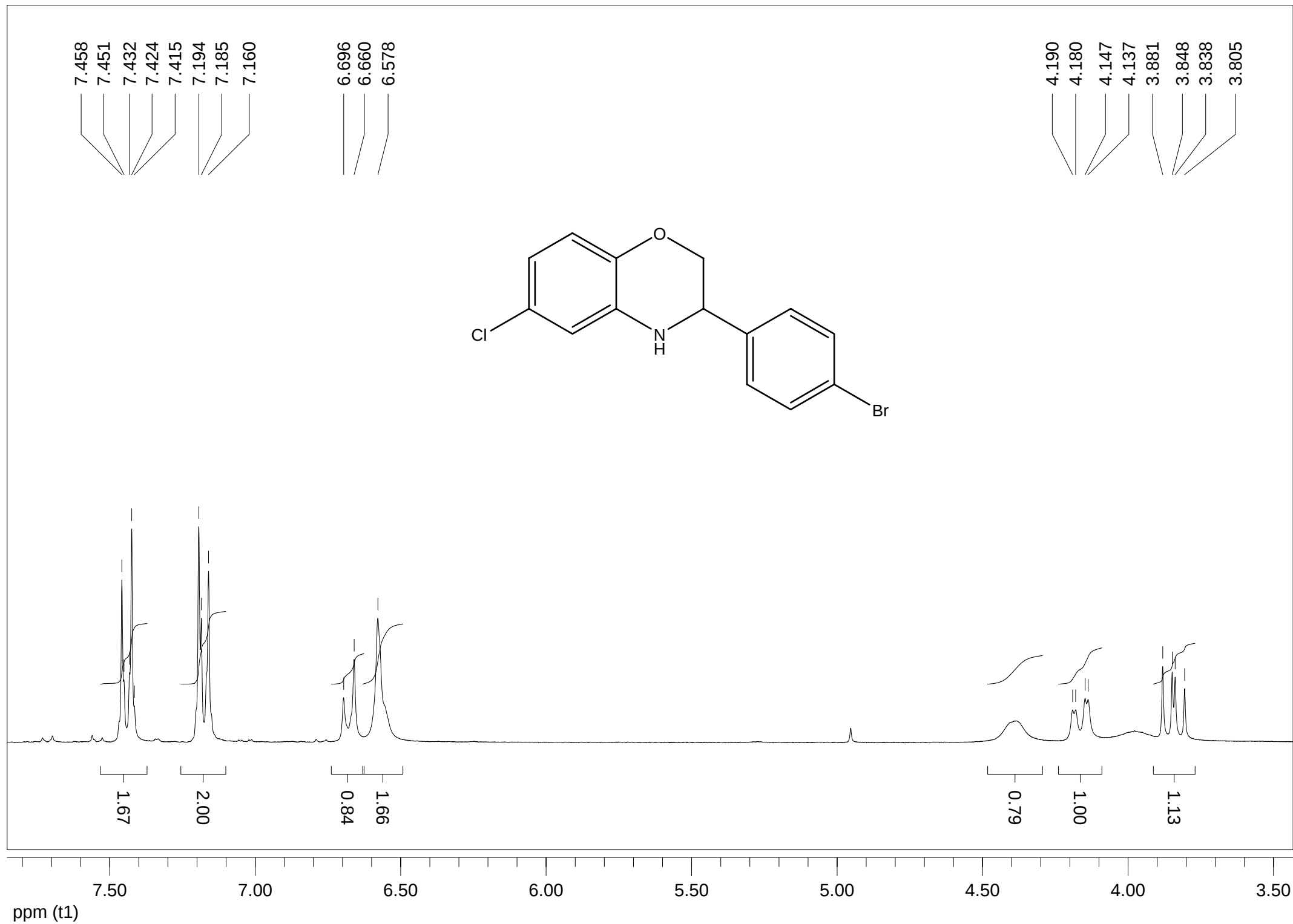


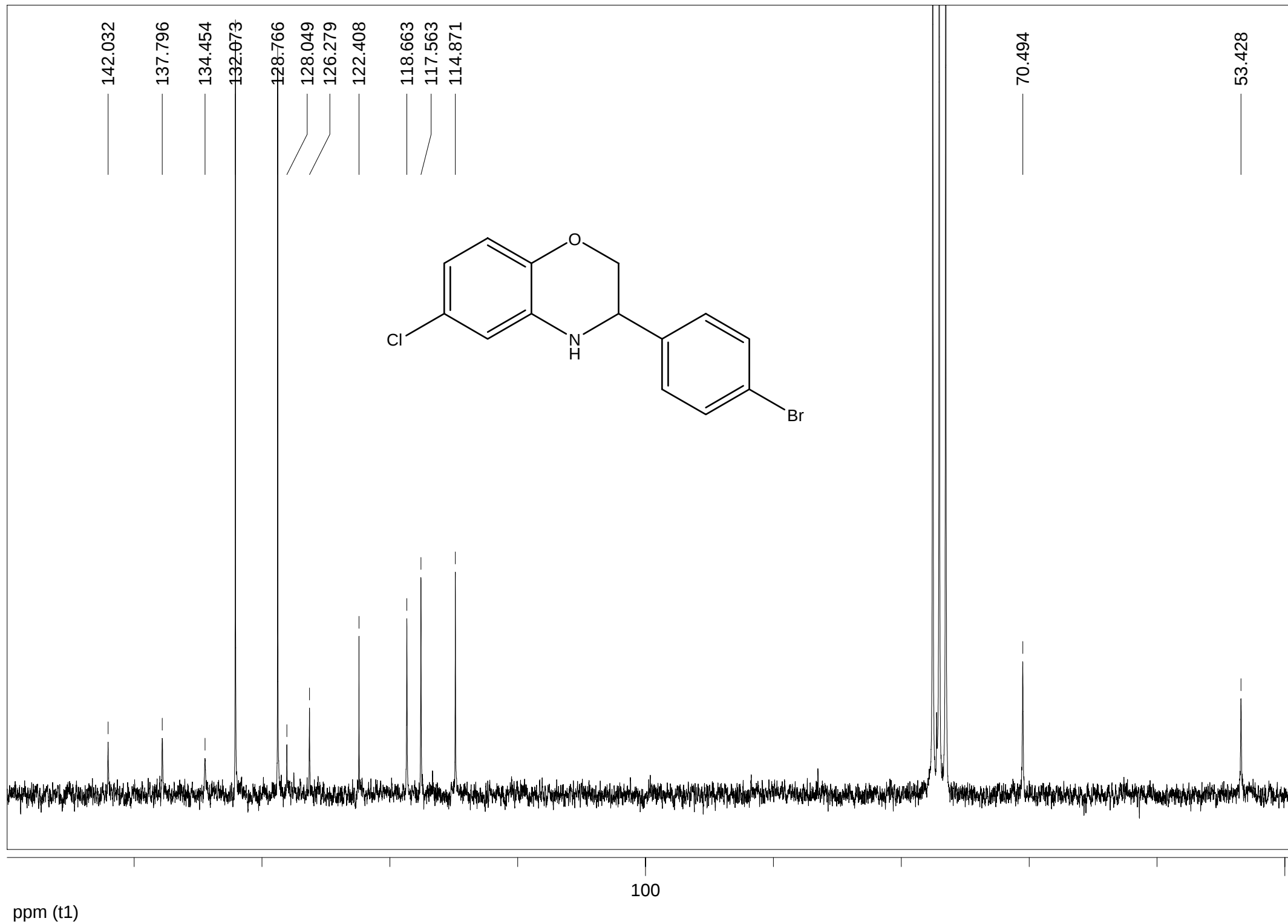


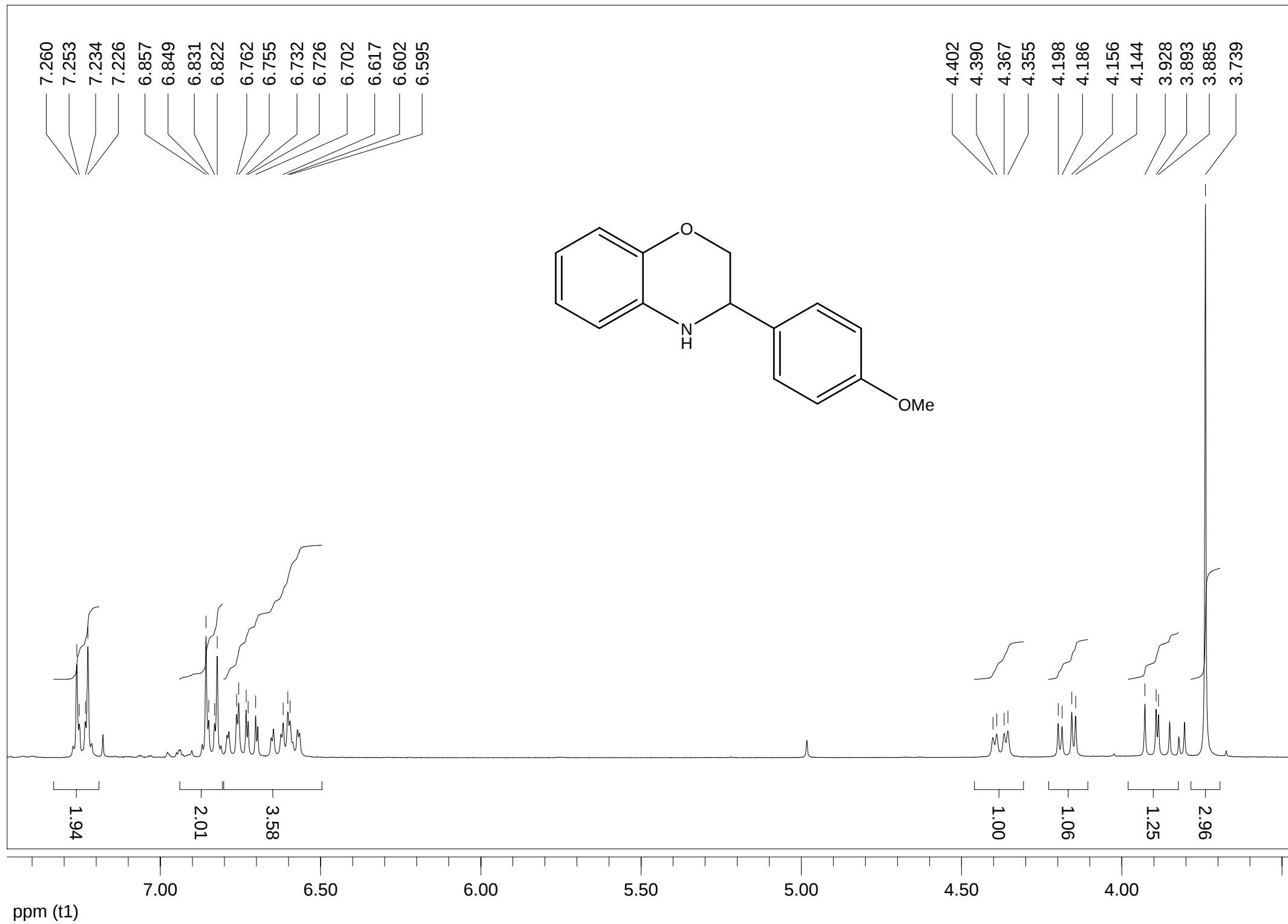


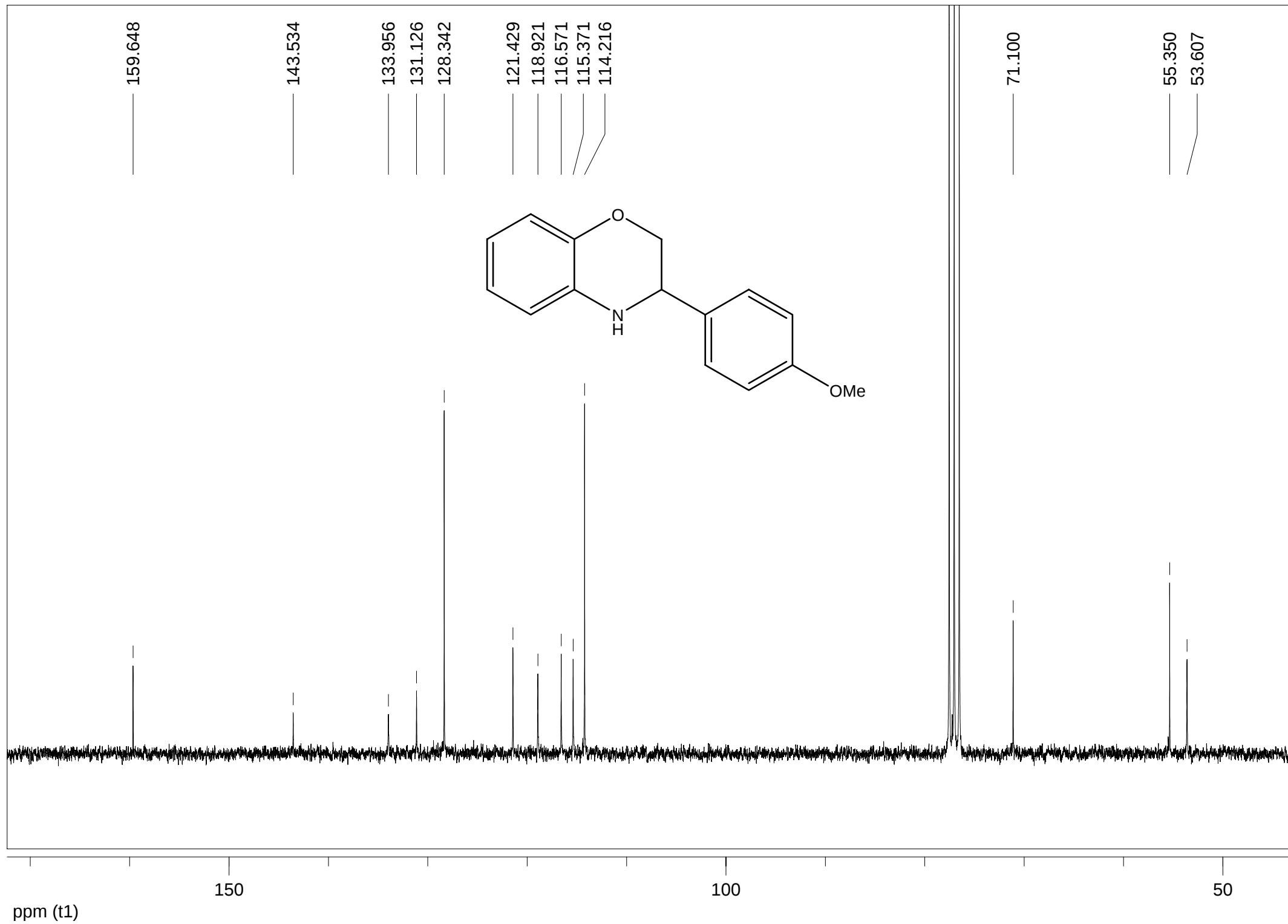


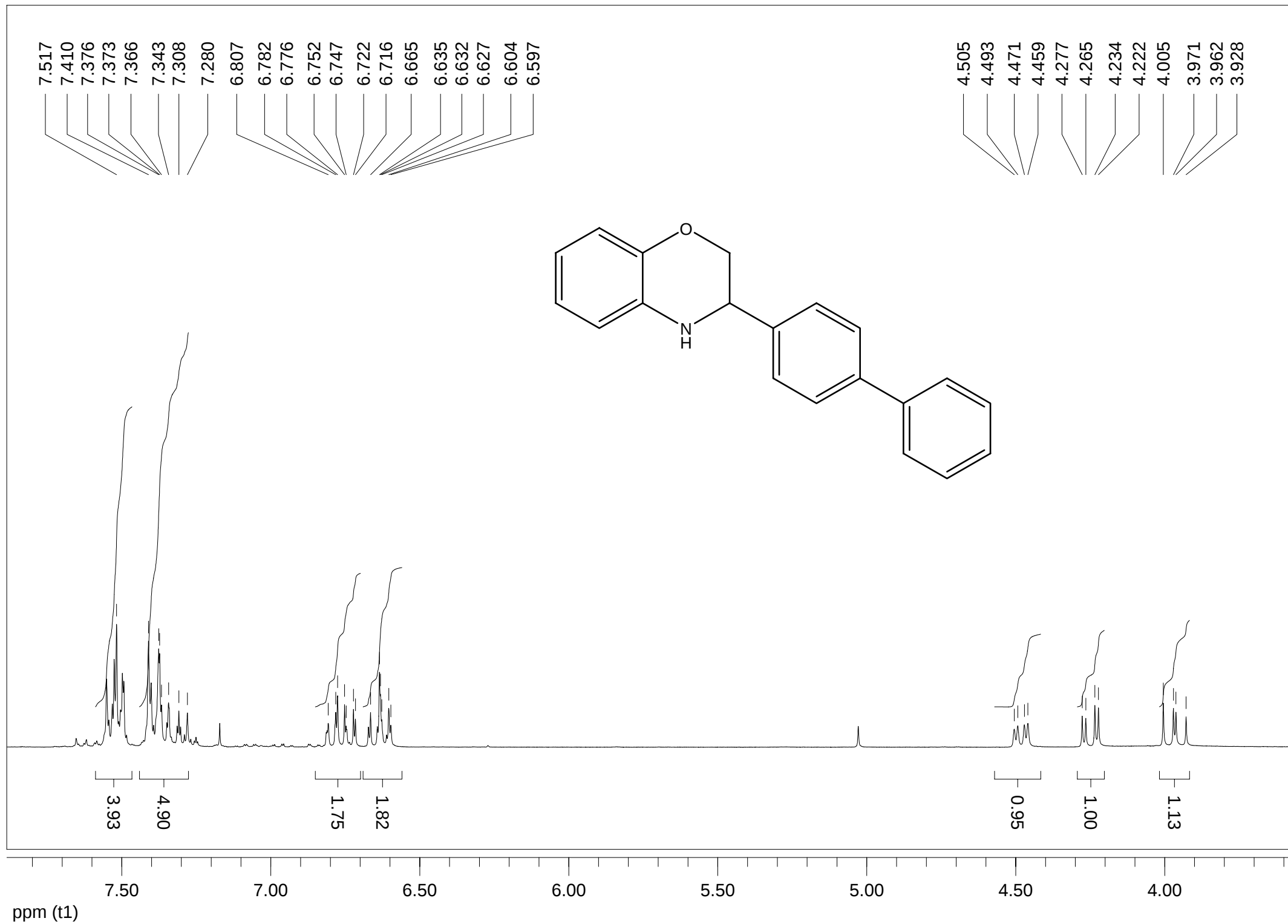


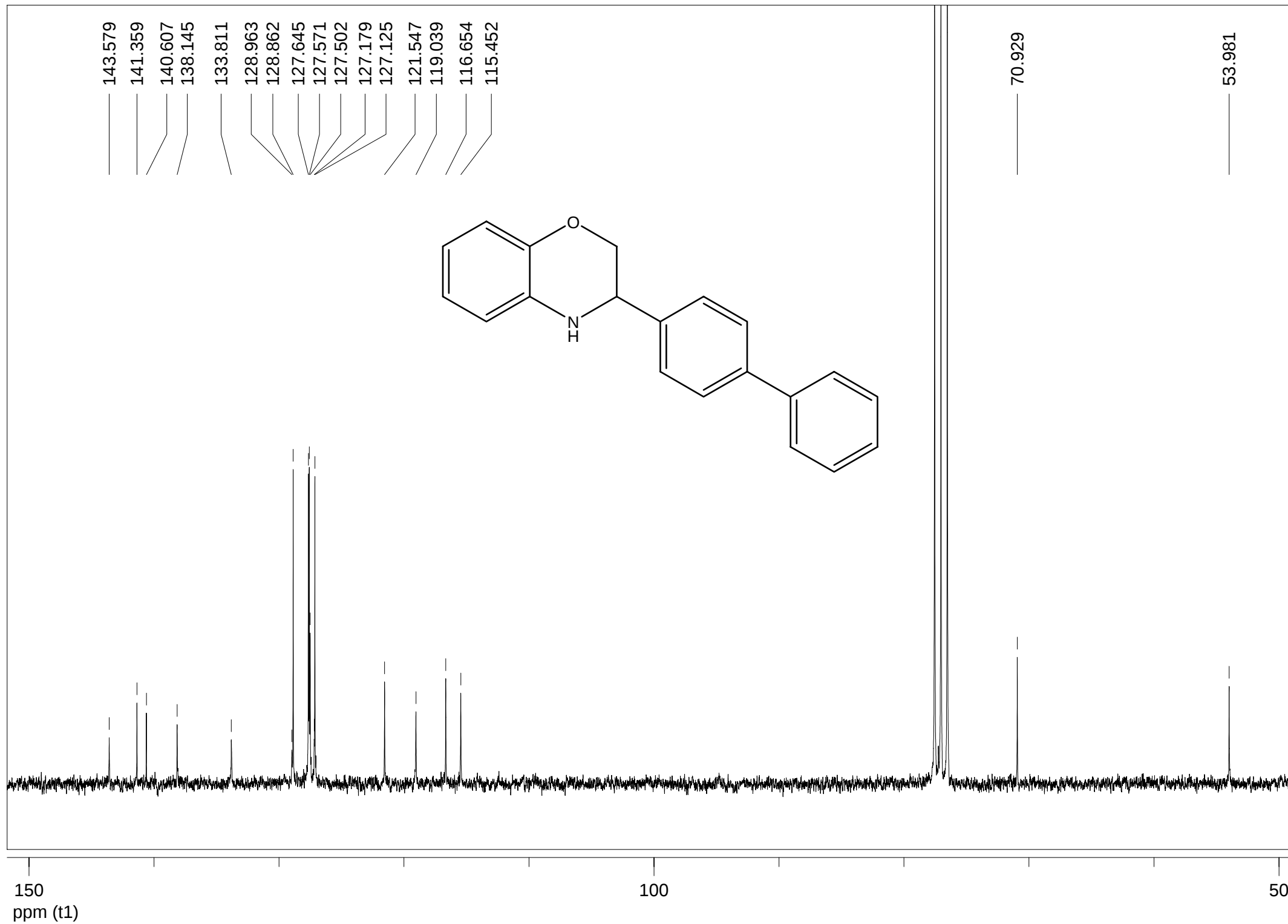


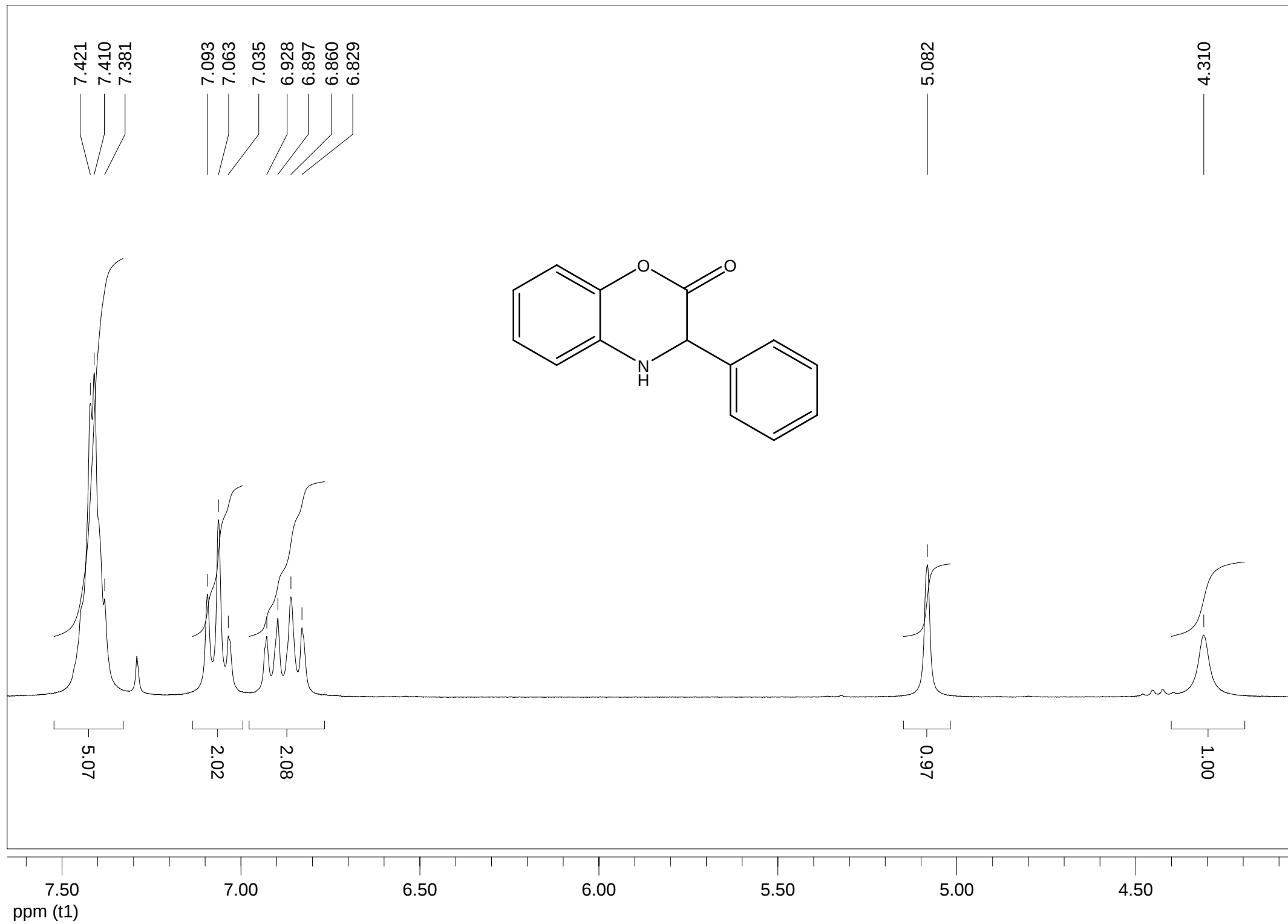


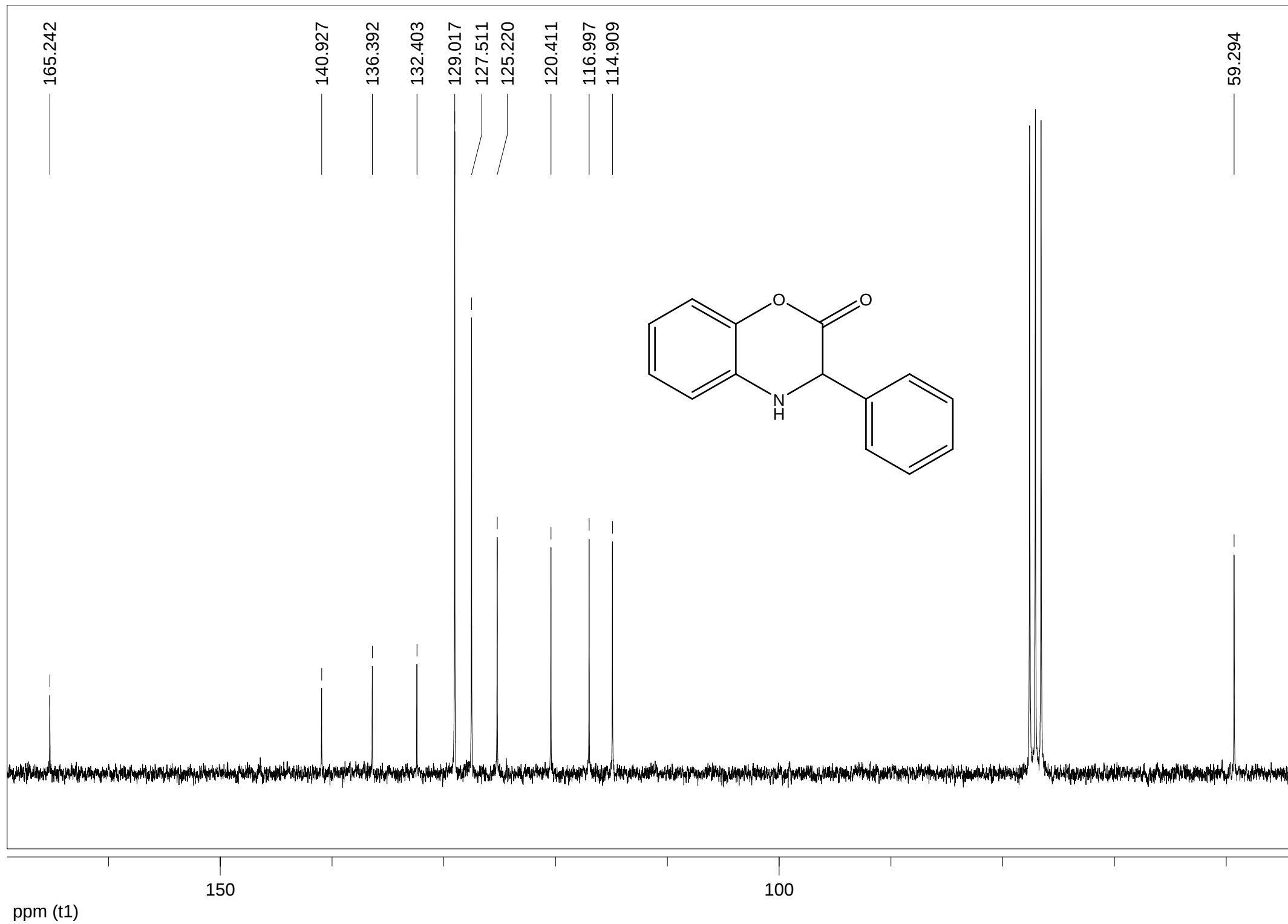


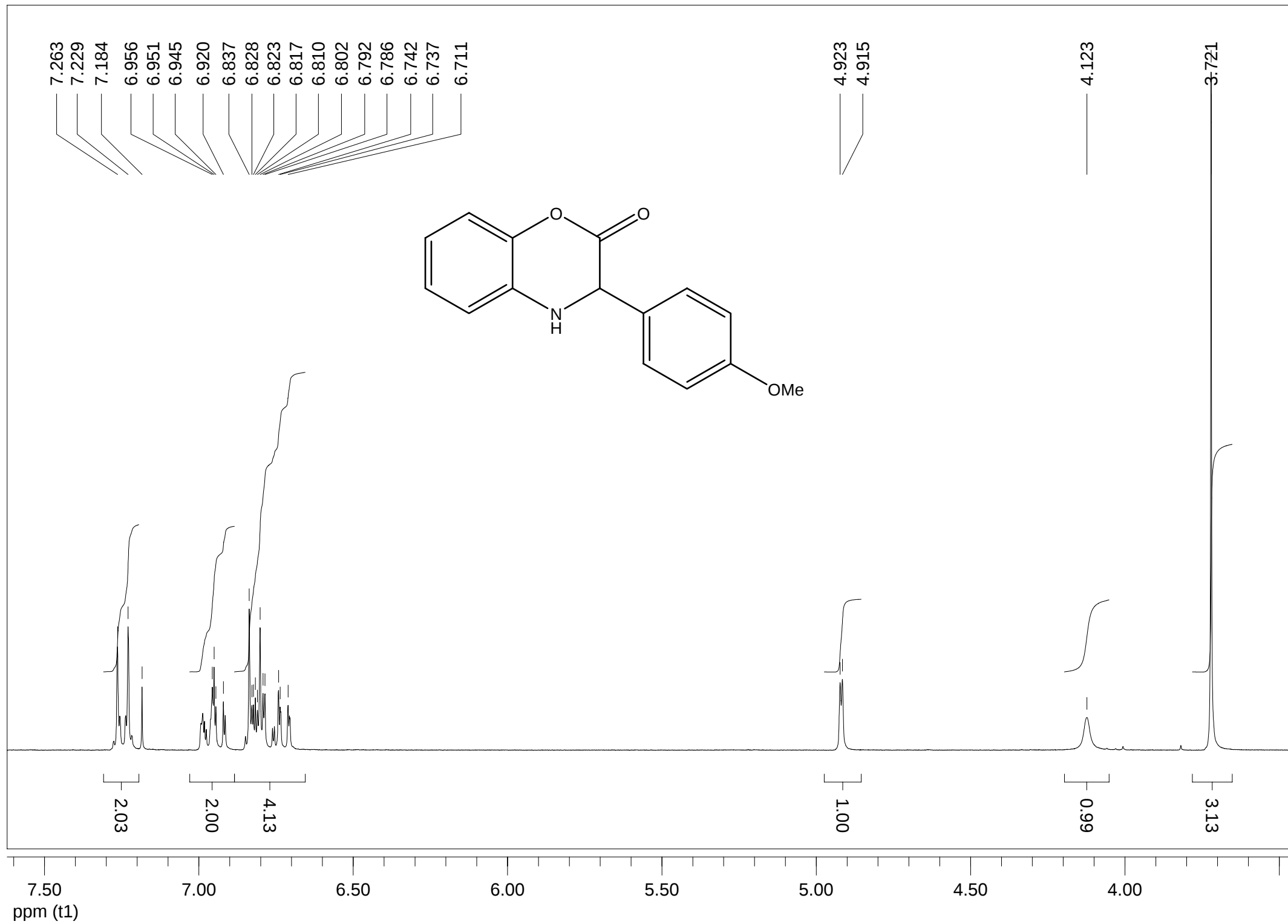


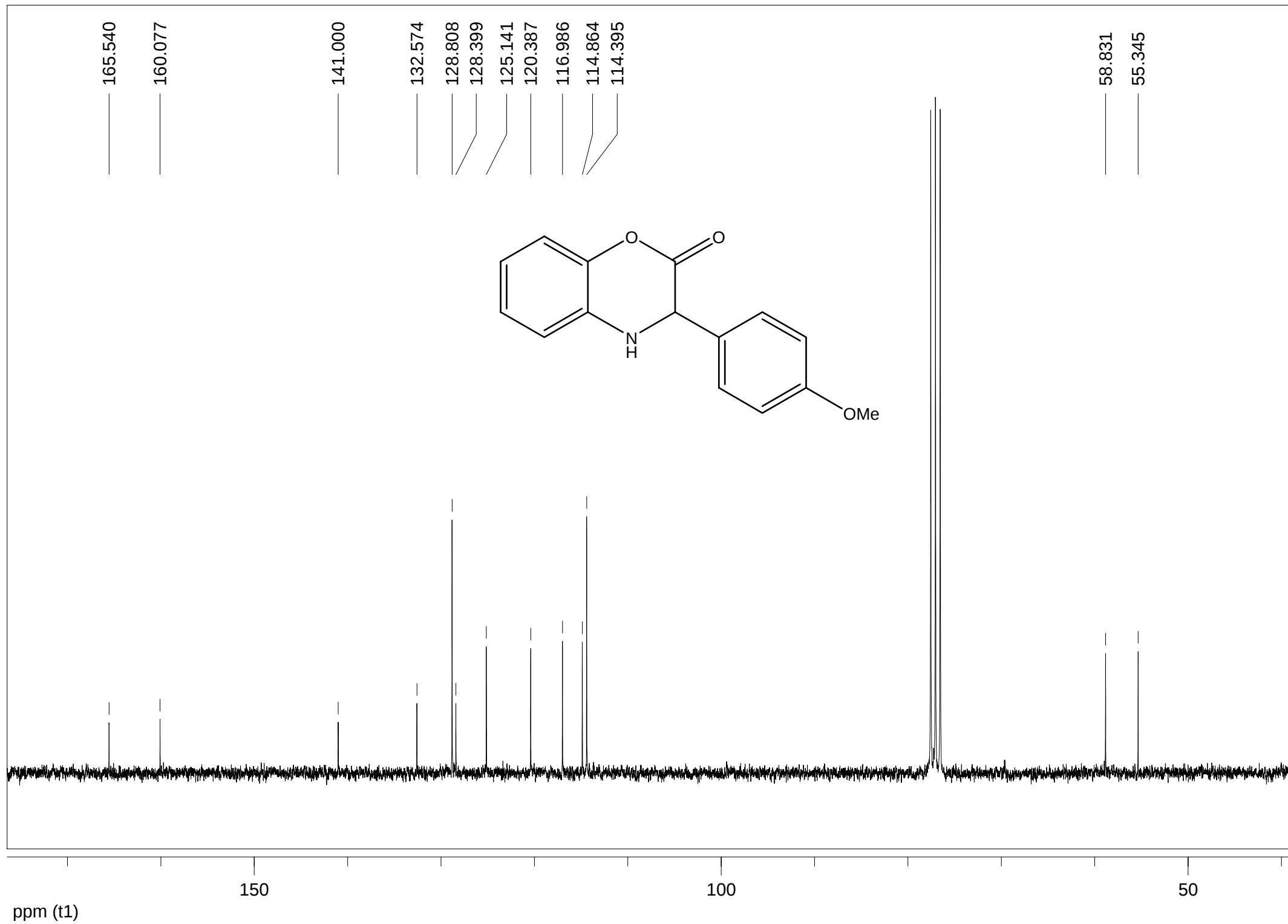


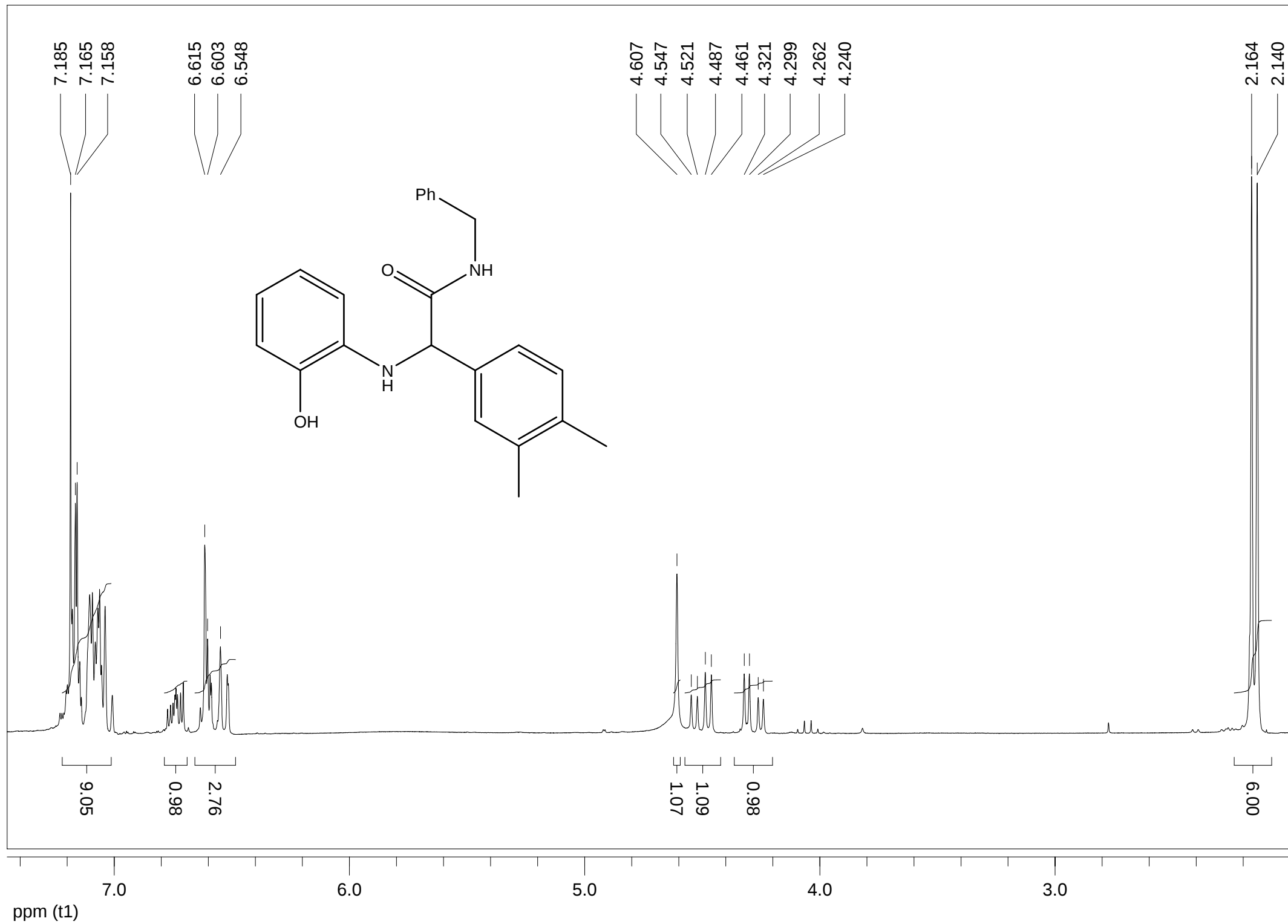


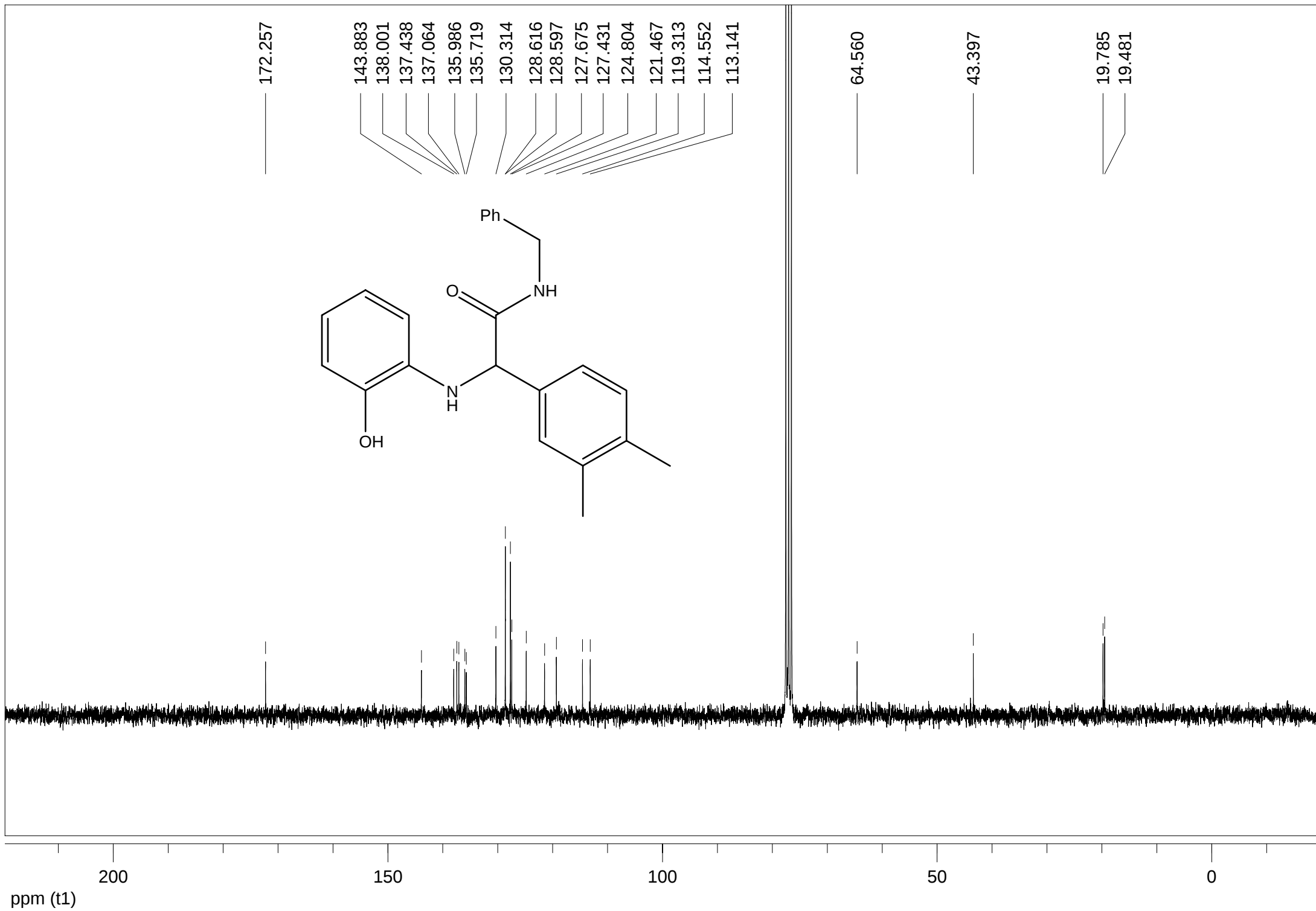


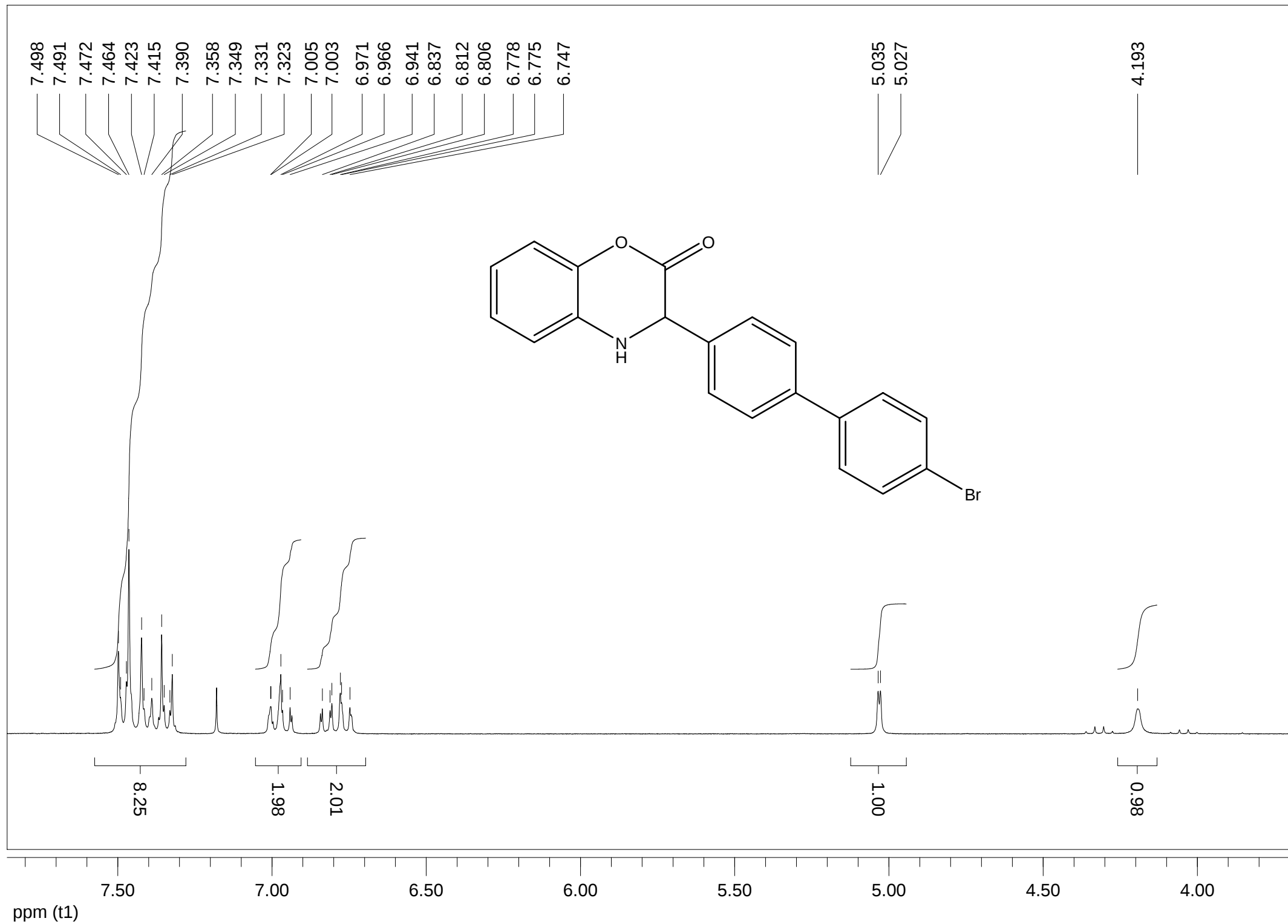


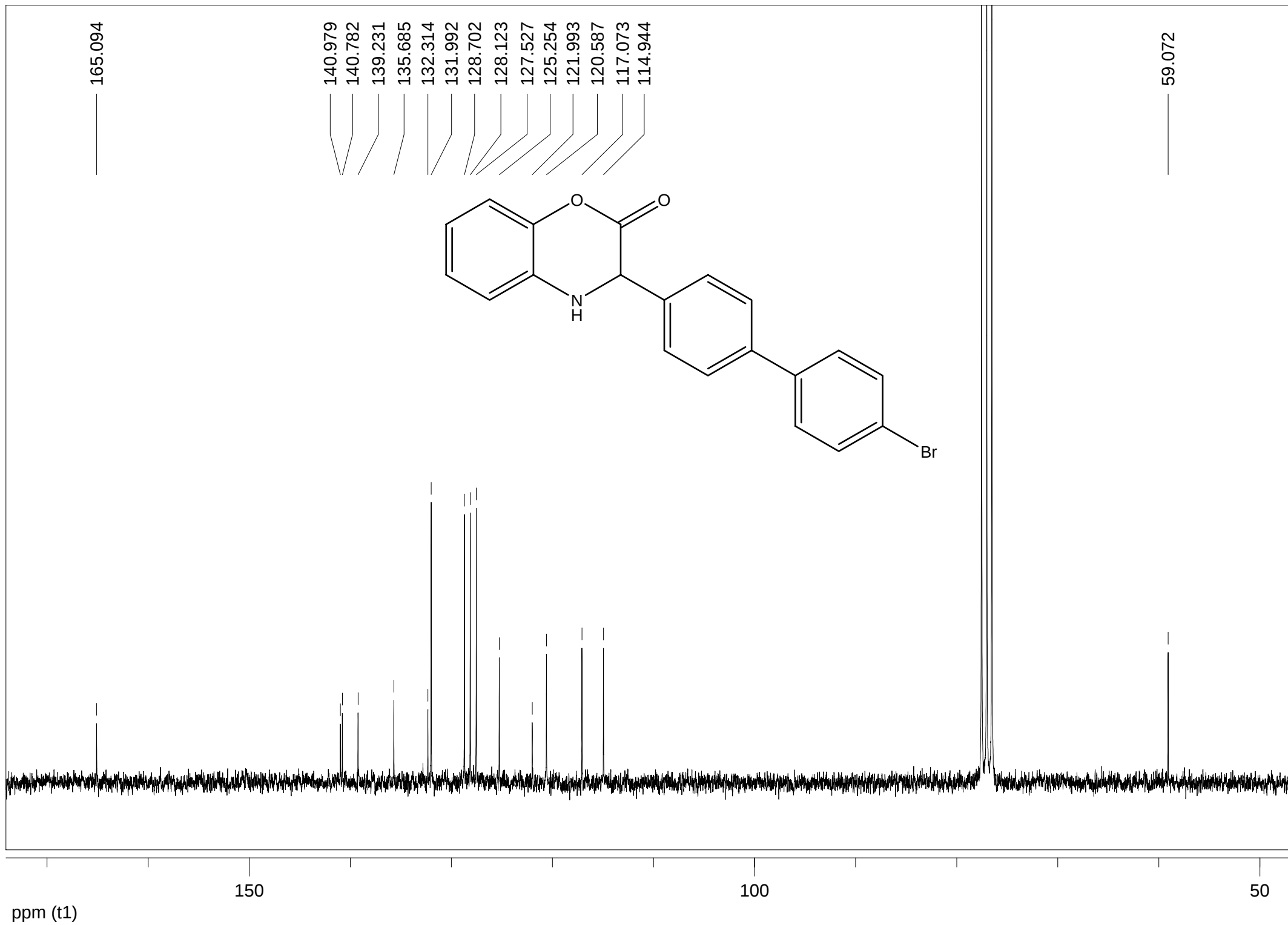


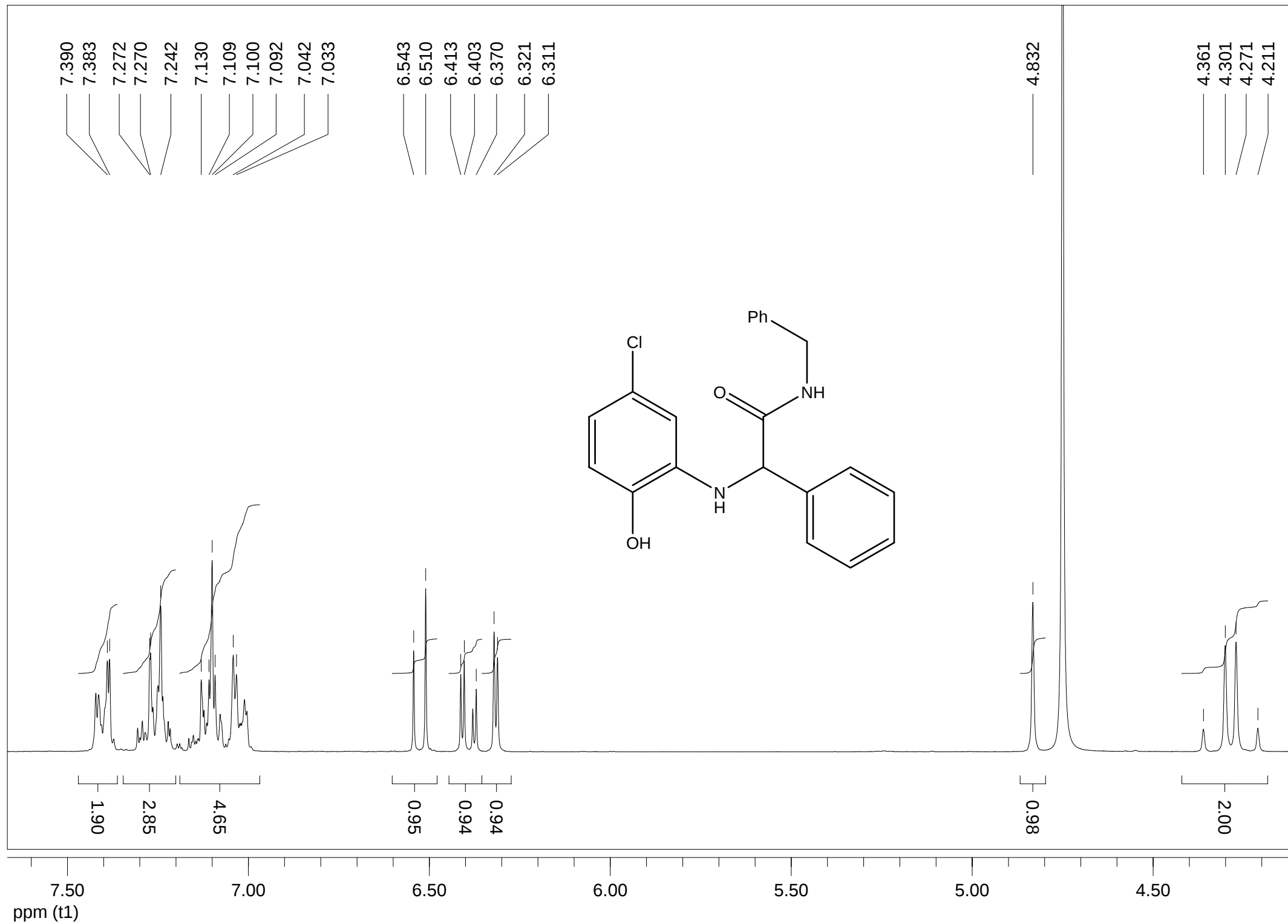


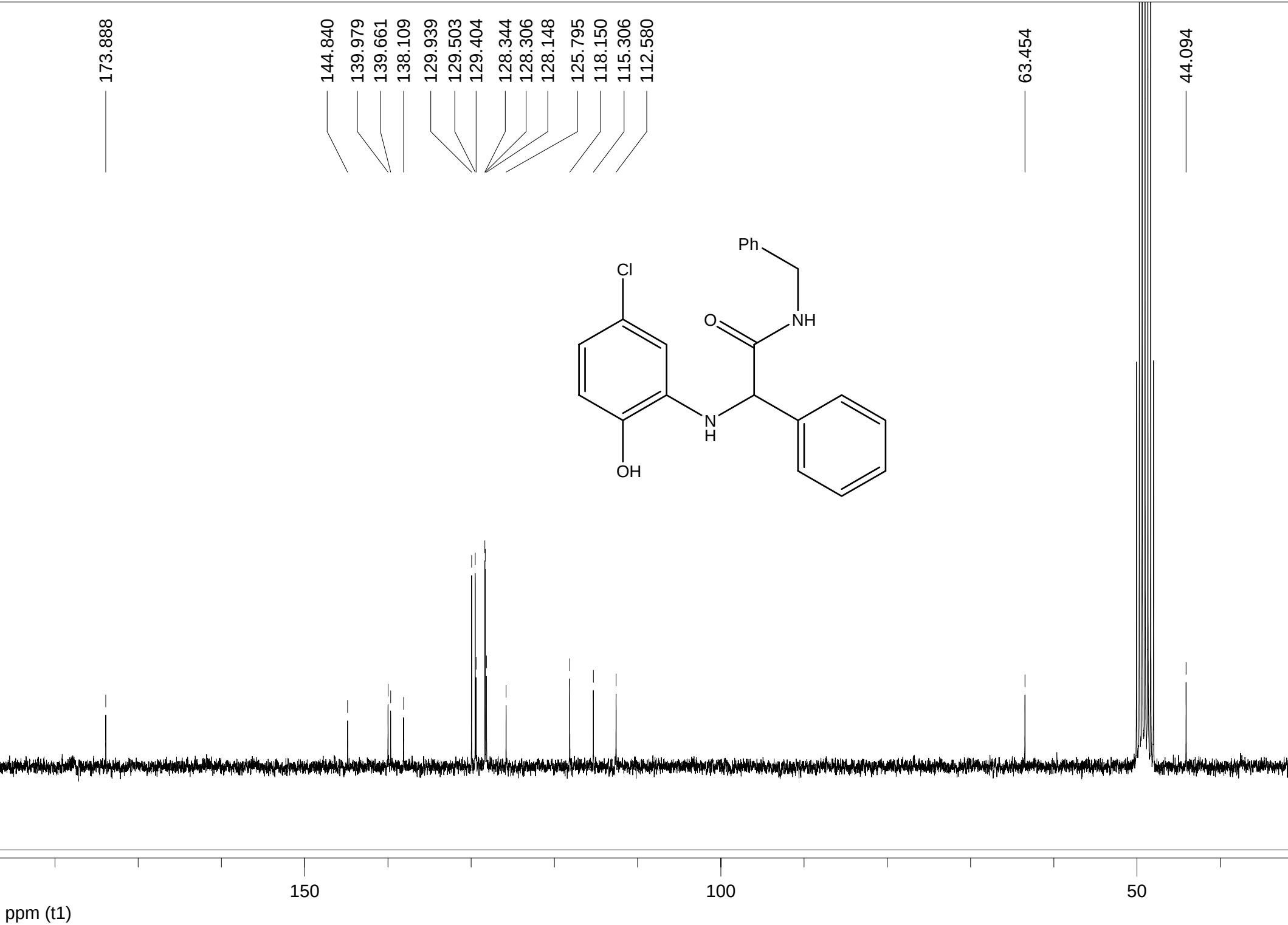


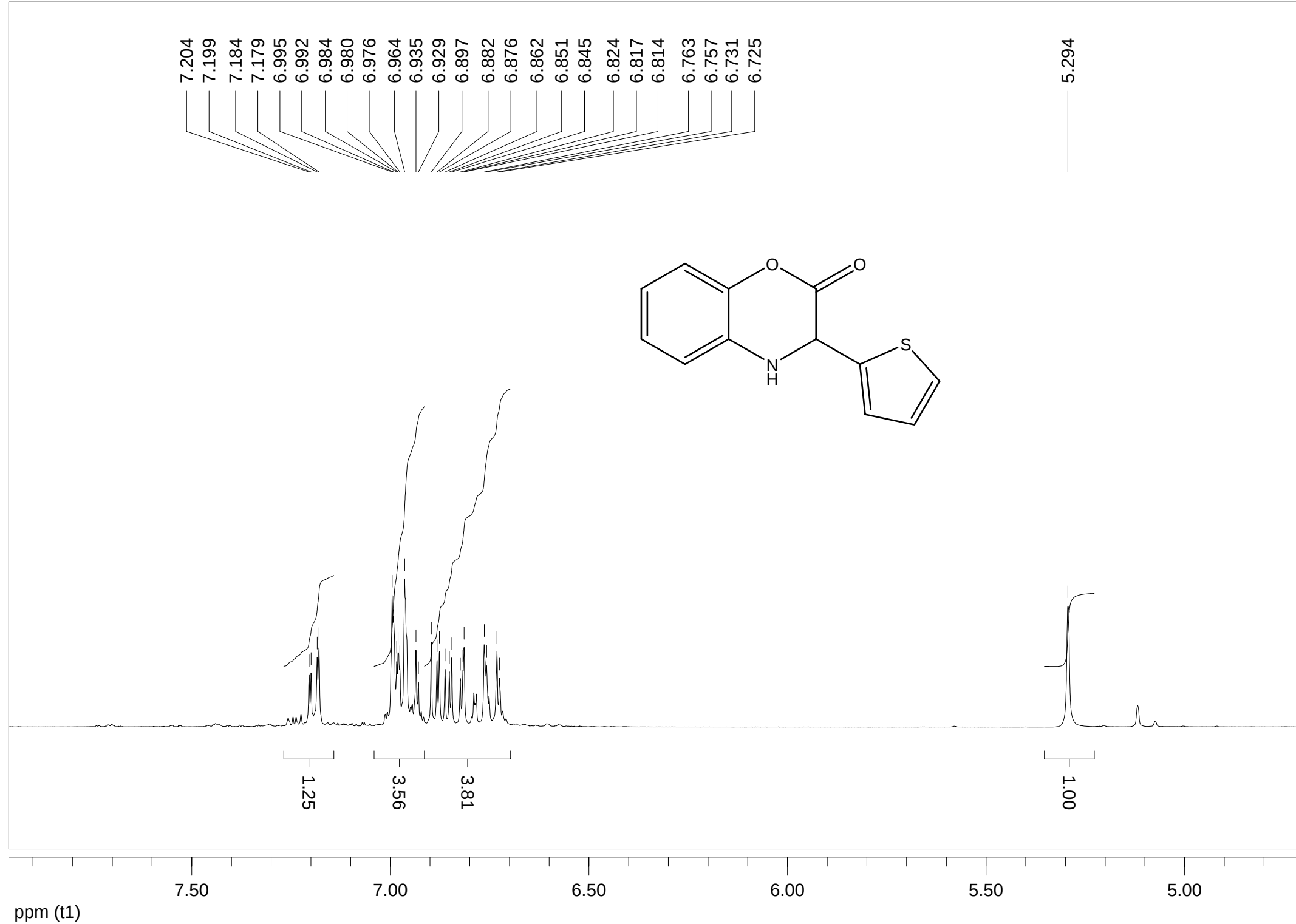
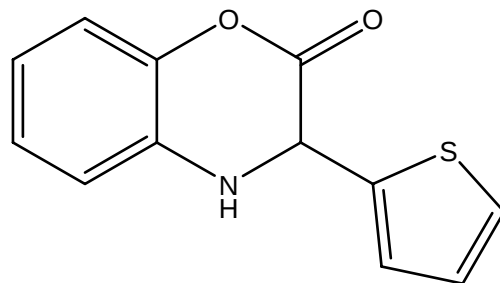












164.031

140.985

138.699

131.465

127.049

126.865

126.476

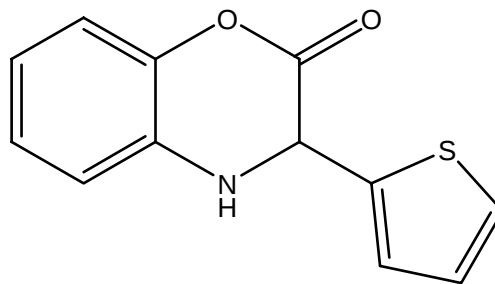
125.327

120.924

116.997

115.521

55.231



ppm (t1)

