Electronic Supplementary Information

Facile Synthesis of the Spiro-Pyridoindolone Scaffold *via* a Gold-Catalysed Intramolecular Alkynol Cyclisation/Hydroindolylation

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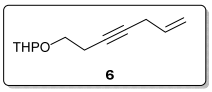
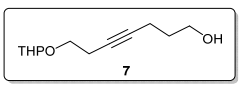
Experimental

General Information: Standard inert atmosphere techniques were used in handling all air and moisture sensitive reagents. Reactions were carried out under an Argon atmosphere in oven dried glassware. All solvents were dried according to standard procedures and techniques before use. Reactions were monitored by thin–layer chromatography (TLC Silica gel 60 F254from Merck), visualised under dual short/long wave UV fluorescence (λ_{max}= 254 and 365 nm) and developed with *p*–anisaldehyde stains, followed by heating. Column chromatography was performed on silica (60–120, 100–200 and 230–400 mesh size). Melting Points were measured and are uncorrected. NMR spectroscopy: ^{1}H NMR spectroscopy measurements were carried out on 400 and 500 MHz NMR spectrometers with $CDCl_3$ (δ 7.27) as an internal standard unless otherwise stated. The ^{13}C NMR spectra were recorded on at 400 (100 MHz) or 500 (125 MHz) NMR spectrometer with $CDCl_3$ (δ 77.0) as an internal standard unless otherwise stated. Chemical shifts (*δ*) are given in ppm downfield from TMS, and coupling constants (*J*) are in Hertz (Hz). The ^{1}H NMR spectra are reported as follows: δ/ppm (multiplicity, coupling constant followed by number of protons). Multiplicity of ^{1}H NMR is abbreviated as follows: s = singlet, d = doublet, dd = doublet of doublets, t = triplet, dt = doublet of triplet, q = quartet, dq = doublet of quartet, m = multiplet. The multiplicity of ^{13}C NMR signals was assigned with the help of DEPT spectra and the abbreviations used: s = singlet, d = doublet, t = triplet, q = quartet represents C (quaternary), CH, CH_2 and CH_3 respectively. High Resolution Mass Spectra (HRMS) were recorded on a Q Exactive Hybrid Quadrupole Orbitrap Mass Spectrometer.

2-(Hept-6-en-3-yn-1-yloxy)tetrahydro-2*H*-pyran (**6**)**:**[1] At rt, a solution of THP-protected but-3-ynol (8.0 g, 51.9 mmol) in DMF (60 ml) was treated with allyl bromide (8.97 mL, 103.8 mmol), K_2CO_3 (10.75 g, 77.8 mmol), Na_2SO_3 (3.27 g, 25.9 mmol), CuI (988 mg, 5.2 mmol), and cat. DBU sequentially and the reaction mixture was stirred for 12 h at rt. The reaction mixture was filtered through celite pad (washed with EtOAc), diluted with EtOAc (200 mL X 2), washed with water (2 x 100 mL), brine (50 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The resulting crude was purified by column chromatography to afford alkene **6** (9.2 g, 91% yield) as a colourless liquid. R_f = 0.8 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 1.49–1.65 (m, 4H), 1.71 (tt, *J* = 13.1, 3.5 Hz, 1H), 1.80–1.88 (m, 1H), 2.50 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.89–2.98 (m, 2H), 3.45–3.60 (m, 2H), 3.77–3.94 (m, 2H), 4.65 (t, *J* = 3.0 Hz, 1H), 5.05 (dq, *J* = 10.0, 1.6 Hz, 1H), 5.32 (dq, *J* = 10.0, 1.6 Hz, 1H), 5.81 (ddt, *J* = 10.0, 5.0, 1.6 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): *δ* 19.4 (t), 20.2 (t), 23.1 (t), 25.4 (t), 30.6 (t), 62.2 (t), 66.1 (t), 77.7 (s), 79.4 (s), 98.7 (d), 115.7 (t), 133.1 (d) ppm; HRMS (ESI) calcd for $C_{12}H_{18}NaO_2$: 217.1199 [M + Na]$^+$; found 217.1197.

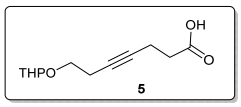


7-((Tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-ol (**7**)**:**[2] A mixture of alkene **6** (9.2 g, 47.36 mmol) and 9-BBN dimer (5.78 g, 23.7 mmol) was heated at 50 °C for 10 min. The contents were cooled to 0 °C, diluted with THF (100 mL) and stirred at rt for 5 h. The reaction mixture was treated with 30% H_2O_2 (37.0 mL, 473.6 mmol) and 3M NaOH (157.8 mL, 473.6 mmol) and stirring was continued at rt for 12 h. The reaction mixture was diluted with cold water (200 mL) and was extracted with EtOAc (200 mL X 2). The combined organic layer was washed with brine (100 mL), dried (Na_2SO_4) and concentrated under reduced pressure. Purification of the resulting crude by column chromatography gave the alcohol **7** (4.94 g, 76% yield brsm) as colourless liquid along with the starting olefin **6** (3.3 g). R_f = 0.4 (50% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 1.49–1.64 (m, 4H),

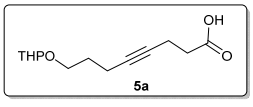


1.69–1.77 (m, 4H), 1.80–1.88 (m, 1H), 2.28 (tt, *J* = 6.8, 2.3 Hz, 2H), 2.46 (tt, *J* = 6.8, 2.4 Hz, 2H), 3.53 (dt, *J* = 9.6, 7.1 Hz, 2H), 3.74–3.83 (m, 3H), 3.89 (tt, *J* = 8.3, 3.0 Hz, 1H), 4.64 (t, *J* = 4.6 Hz, 1Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): *δ* 15.5 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.6 (t), 31.4 (t), 62.0 (t), 62.3 (t), 66.1 (t), 77.7 (s), 80.4 (s), 98.8 (d) ppm; HRMS (ESI) calcd for $C_{12}H_{20}NNaO_3$: 235.1305 $[M + Na]^+$; found 235.1299.

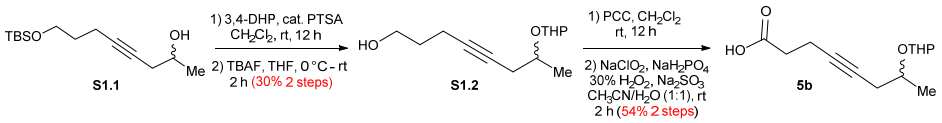
7-((Tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-ynoic acid (**5**):[2–3] A solution of alcohol **7** (4.9 g, 23.1 mmol) in CH_2Cl_2 (100 mL) was treated with PCC (9.95 g, 46.2 mmol) and stirred for 12 h at rt. The reaction mixture was filtered through silica bed using CH_2Cl_2 as eluent to obtain the crude aldehyde as colourless liquid which was used directly for the next step. The above crude aldehyde was dissolved in a 1:1 CH_3CN/H_2O (50 mL) and treated with $NaClO_2$ (1.88 g, 20.8 mmol), NaH_2PO_4 (2.5 g, 20.8 mmol) and 30% H_2O_2 (1.63 mL, 20.8 mmol) and the contents were stirred at rt for 2 h. The reaction mixture was quenched by adding Na_2SO_3 (2.63 g, 20.8 mmol) and then extracted with EtOAc (100 X 2 mL). The combined organic layer was washed with water (50 mL), brine (50 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The resulting crude was purified by column chromatography to afford acid **5** as colourless liquid (3.62 g, 69% yield, 2 steps). R_f = 0.3 (50% ethyl acetate in petroleum ether); ^1H NMR (400 MHz, $CDCl_3$): *δ* 1.47–1.65 (m, 4H), 1.72 (tt, *J* = 13.0, 3.1 Hz, 1H), 1.79–1.88 (m, 1H), 2.43–2.49 (m, 4H), 2.56 (td, *J* = 8.2, 7.5 Hz, 2H), 3.49–3.55 (m, 2H), 3.78 (dt, *J* = 9.7, 7.1 Hz, 1H), 3.89 (tt, *J* = 8.2, 3.0 Hz, 1H), 4.65 (t, *J* = 3.0 Hz, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): *δ* 14.5 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.5 (t), 33.7 (t), 62.2 (t), 66.0 (t), 78.0 (s), 78.9 (s), 98.7 (d), 177.4 (s) ppm; HRMS (ESI) calcd for $C_{12}H_{18}NaO_4$: 249.1097 $[M + Na]^+$; found 249.1091



8-((Tetrahydro-2*H*-pyran-2-yl)oxy)oct-4-ynoic acid (**5a**)**:** prepared by employing OTHP-pent-4-ynol (2.0 g, 10.97 mmol) following 4 steps.[1–3] Acid **5a** (720 mg, 61% yield, 2 steps) was obtained as a colourless liquid; R_f = 0.3 (30% ethyl acetate in petroleum ether); ^1H NMR (400 MHz, $CDCl_3$): *δ* 1.47–1.62 (m, 4H), 1.67–1.85 (m, 4H), 2.25 (tt, *J* = 6.9, 2.2 Hz, 2H), 2.42–2.50 (m, 2H), 2.50–2.58 (m, 2H), 3.46 (dt, *J* = 9.7, 6.2 Hz, 1H), 3.50–3.55 (m, 1H), 3.80 (dt, *J* = 9.7, 6.3 Hz, 1H), 3.84–3.90 (m, 1H), 4.62 (t, *J* = 3.3 Hz, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): *δ* 14.5 (t), 15.5 (t), 19.3 (t), 25.4 (t), 28.9 (t), 30.6 (t), 33.8 (t), 62.0 (t), 66.0 (t), 78.2 (s), 80.5 (s), 98.7 (d), 177.3 (s) ppm; HRMS (ESI) calcd for $C_{13}H_{20}NaO_4$: 241.1434 $[M + Na]^+$; found 241.1428.

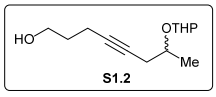


Scheme S1. Synthesis of Acid **5b**



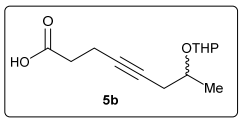
A solution of alcohol **S1.1**[4] (1.5 g, 5.85 mmol), 3,4-dihydropyran (0.64 mL, 7.02 mmol) and pTSA (100 mg, 0.58 mmol) in CH_2Cl_2 (30 mL) was stirred at rt for 12 h. After completion of the reaction (indicated by TLC), the reaction mixture was neutralised with the addition of Et_3N. The volatiles were removed under reduced pressure. The resulting crude was dissolved in THF (50 mL) and cooled to 0 °C. To this, tetra-*n*-butyl ammonium fluoride (TBAF 1.38 g, 5.29 mmol) was added at stirred at rt for 2 h. After completion of reaction (indicated by TLC), the reaction was diluted with water (20 mL) and extracted with EtOAc (50 mL X 2), dried (Na_2SO_4) and concentrated under reduced pressure. The crude was purified by column chromatography to afford alkynol **S1.2** (400 mg, 30% yield, 2 steps) as colourless liquid.

7-((Tetrahydro-2*H*-pyran-2-yl)oxy)oct-4-yn-1-ol (**S1.2**)**:** Colourless liquid (400 mg, 40%); R_f = 0.2 (20% ethyl acetate in petroleum ether); ^1H NMR (500 MHz, $CDCl_3$): *δ* 1.21 (d, *J* = 6.1 Hz, 3H), 1.29 (d, *J* = 6.1 Hz, 3H), 1.47–1.59 (m, 8H), 1.66–1.76 (m, 6H), 1.78–1.86 (m, 2H), 1.96 (br. s. 2H), 2.23–2.31 (m, 5.6H), 2.32–2.39 (m, 1.6H), 2.45–2.53 (m, 1H), 3.45–3.53 (m, 2H), 3.69–3.77 (m, 4H), 3.83–3.96 (m, 4H), 4.69 (s, 1H), 4.74 (s, 1H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): *δ* 15.4 (t), 15.4 (t), 18.9 (q), 19.6 (t), 19.7 (t), 21.0 (q), 25.4 (t), 25.4

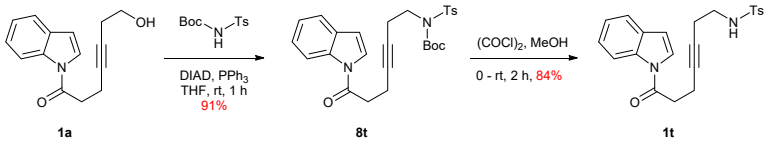


(t), 26.0 (t), 27.4 (t), 30.9 (t), 31.0 (t), 31.4 (t), 31.5 (t), 61.8 (t), 62.4 (t), 62.6 (t), 71.0 (d), 71.3 (d), 77.4 (s), 77.8 (s), 80.9 (s), 80.9 (s), 96.7 (d), 97.7 (d) ppm; HRMS (ESI) calcd for $C_{13}H_{22}NaO_3$: 249.1461 $[M + Na]^+$; found 249.1459.

7-((Tetrahydro-2*H*-pyran-2-yl)oxy)oct-4-ynoic acid (**5b**)**:** The alcohol **S1.2** was oxidised to corresponding acid **5b** by following the established procedure[2–3] (54% yield, 2 steps). Colourless liquid; R_f = 0.2 (30% ethyl acetate in petroleum ether); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.22 (d, *J* = 6.1 Hz, 2.37H), 1.29 (d, *J* = 6.1 Hz, 3H), 1.48–1.61 (m, 7.5H), 1.69–1.75 (m, 2H), 1.80–1.87 (m, 2H), 2.20–2.39 (m, 3H), 2.44–2.52 (m, 4H), 2.53–2.60 (m, 4H), 3.47–3.54 (m, 2H), 3.85–3.98 (m, 3.71H), 4.72 (t, *J* = 4.2 Hz, 1H), 4.76 (m, 1H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 14.5 (t), 19.0 (q), 19.5 (t), 19.8 (t), 21.1 (q), 25.4 (t), 26.1 (t), 27.4 (t), 30.9 (t), 31.0 (t), 33.6 (t), 62.4 (t), 62.7 (t), 71.1 (d), 71.5 (d), 78.0 (s), 78.3 (s), 79.4 (s, 2C), 96.7 (d), 97.8 (d), 176.7 (s) ppm; HRMS (ESI) calcd for $C_{13}H_{20}NaO_4$: 263.1254 $[M + Na]^+$; found 263.1246.

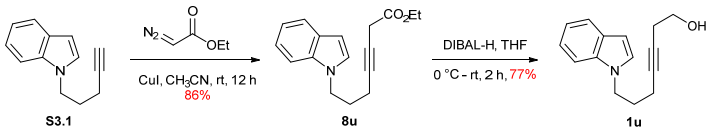


Scheme S2. Synthesis of alkynylamine **1t**



***tert*-Butyl (7-(1*H*-indol-1-yl)-7-oxohept-3-yn-1-yl)(tosyl)carbamate** (**8t**)**:**[5] At 0 °C, a solution of alcohol **1a** (100 mg, 0.42 mmol), TsNHBoc (146 mg, 0.54 mmol), PPh_3 (141 mg, 0.54 mmol) in THF (10 mL) was treated with DIAD (106 µL, 0.54 mmol) and stirring was continued at rt for 1 h. The reaction mixture was and purified by column chromatography to afford alkynylamine **8t** (188 mg, 91% yield) as a colourless syrup colourless sticky liquid (188 mg, 91%); R_f = 0.7 (40% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.34 (s, 9H), 2.43 (s, 3H), 2.62 (tt, *J* = 7.3, 2.2 Hz, 2H), 2.69 (tt, *J* = 7.8, 2.1 Hz, 2H), 3.16 (dd, *J* = 8.4, 6.5 Hz, 2H), 3.93–3.98 (m, 2H), 6.66 (d, *J* = 3.3 Hz, 1H), 7.26–7.32 (m, 3H), 7.36 (td, *J* = 8.3, 1.1 Hz, 1H), 7.49 (d, *J* = 3.7 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 1H), 7.80 (d, *J* = 8.3 Hz, 2H), 8.46 (d, *J* = 8.2 Hz, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 14.2 (t), 20.2 (t), 21.6 (q), 27.8 (q, 3C), 35.2 (t), 45.6 (t), 77.5 (s), 80.4 (s), 84.4 (s), 109.3 (d), 116.5 (d), 120.8 (d), 123.7 (d), 124.5 (d), 125.1 (d), 127.8 (d, 2C), 129.2 (d, 2C), 130.3 (s), 135.5 (s), 137.3 (s), 144.2 (s), 150.7 (s), 169.8 (s) ppm; HRMS (ESI) calcd for $C_{27}H_{30}N_2O_5SNa$: 517.1768 $[M + Na]^+$; found 517.1758.

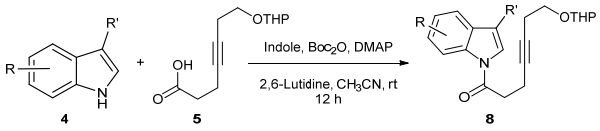
Scheme S3. Synthesis of Alkynol **1u**



Ethyl 7-(1*H*-indol-1-yl)hept-3-ynoate (8u):[6] A solution of alkyne **S3.1**[7] (1 g, 5.46 mmol), ethyl diazoacetate (3.11 g, 27.28 mmol) and CuI (519 mg, 2.73 mmol) in CH_3CN (30 mL) was stirred at rt for 12 h. After completion of reaction (indicated by TLC), the reaction mixture was filtered through celite and washed with EtOAc (30 mL X 2). The combined organic layer was washed with water (2 x 25 mL), brine (25 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The crude was purified by column chromatography to afford alkyne **8u** (1.27 g, 86% yield) as a colourless liquid. R_f = 0.5 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.20 (t, *J* = 7.1 Hz, 3H), 1.92 (quin, *J* = 6.6 Hz, 2H), 2.02–2.09 (m, 2H), 3.19 (t, *J* = 2.3 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 4.19 (t, *J* = 6.6 Hz, 2H), 6.39 (d, *J* = 3.0 Hz, 1H), 7.0 (t, *J* = 7.7 Hz, 1H), 7.05–7.14 (m, 2H), 7.29 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 14.1 (q), 16.0 (t), 26.0 (t), 28.7 (t), 44.6

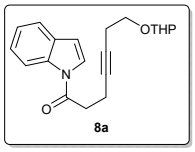
(t), 61.4 (t), 73.1 (s), 82.1 (s), 100.9 (d), 109.3 (d), 119.2 (d), 120.8 (d), 121.3 (d), 128.1 (d), 128.6 (s), 135.8 (s), 168.7 (s) ppm; HRMS (ESI) calcd for $C_{17}H_{20}N_2O_2$: 270.1489 [M + H]$^+$; found 270.1484.

General Procedure for Coupling of Indoles and Acid 5:[8]

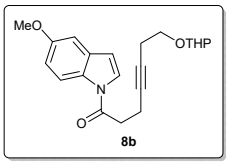


The coupling reactions are carried out employing the acid 0.5 (200 mg, 0.88 mmol) and used the corresponding indole in excess (2.21 mmol, 2.5 equiv). A representative coupling procedure as follows. To a solution of acid **5** (1 equiv) and indole **4** (2.5 equiv) in CH_3CN (5 mL) were added Boc_2O (2.5 equiv), DMAP (0.15 equiv), and 2,6-lutidine (0.5 equiv) sequentially at rt and the stirring was continued for additional12 h. After completion of reaction as indicated by TLC, the reaction mixture was concentrated under reduced pressure and the crude was purified by column chromatography to afford THP protected alkynol **8**.

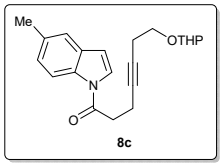
1-(1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8a**)**:** Following the general procedure, the coupling of indole **4a** (258 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8a** (219 mg, 76%) as a colourless solid. R_f = 0.2 (10% ethyl acetate in petroleum ether); mp: 88–90 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.46–1.61 (m, 4H), 1.63–1.71 (m, 1H), 1.81 (dt, *J* = 13.0, 3.8 Hz, 1H), 2.47 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.71 (tt, *J* = 7.2, 2.3 Hz, 2H), 3.14 (dd, *J* = 8.1, 6.9 Hz, 2H), 3.44–3.56 (dt, *J* = 9.6, 7.1 Hz, 2H), 3.79 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.87 (td, *J* = 8.2, 3.0 Hz, 1H), 4.62 (t, *J* = 3.0 Hz, 1H), 6.66 (dd, *J* = 3.8, 0.4 Hz, 1H), 7.28 (td, *J* = 7.4, 1.0 Hz, 1H), 7.36 (td, *J* = 7.7, 1.2 Hz, 1H), 7.47 (d, *J* = 3.7 Hz, 1H), 7.57 (d, *J* = 7.7 Hz, 1H), 8.47 (d, *J* = 8.2 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.4 (t), 62.2 (t), 65.9 (t), 78.2 (s), 79.0 (s), 98.8 (d), 109.3 (d), 116.6 (d), 120.8 (d), 123.7 (d), 124.4 (d), 125.2 (d), 130.3 (s), 135.6 (s), 169.7 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{23}NNaO_3$: 348.1570 [M + Na]$^+$; found 348.1560.



1-(5-Methoxy-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8b**)**:** Following the procedure, the coupling of indole **4b** (325 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8b** (213 mg, 67%) as a pale yellow solid. R_f = 0.2 (30% ethyl acetate in petroleum ether); mp: 64–66 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.44–1.58 (m, 4H), 1.71 (tt, *J* = 13.0, 3.6 Hz, 1H), 1.78–1.87 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.69 (tt, *J* = 7.6, 2.3 Hz, 2H), 3.11 (dd, *J* = 8.1, 6.6 Hz, 2H), 3.46–3.55 (m, 2H), 3.79 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.84–3.91 (m, 4H), 4.62 (dd, *J* = 4.0, 3.0 Hz, 1H), 6.59 (dd, *J* = 3.7, 0.6 Hz, 1H), 6.96 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.03 (d, *J* = 2.5 Hz, 1H), 7.44 (d, *J* = 3.7 Hz, 1H), 8.35 (d, *J* = 8.8 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.6 (t), 35.1 (t), 55.6 (q), 62.3 (t), 65.9 (t), 78.2 (s), 79.1 (s), 98.8 (d), 103.6 (d), 109.2 (d), 113.5 (d), 117.3 (d), 125.0 (d), 130.3 (s), 131.3 (s), 156.5 (s), 169.33 (s) ppm; HRMS (ESI) calcd for $C_{21}H_{25}NNaO_4$: 378.1676 [M + Na]$^+$; found 378.1660.

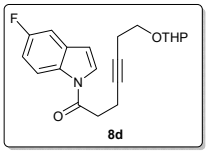


1-(5-Methyl-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8c**)**:** Following the general procedure, the coupling of indole **4c** (289 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8c** (199 mg, 66%) as a colourless liquid. R_f = 0.3 (20% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45–1.59 (m, 4H), 1.66–1.75 (tt, *J* = 13.0, 3.1 Hz, 1H), 1.82 (dd, *J* = 9.2, 3.6 Hz, 1H), 2.45 (s, 3H), 2.46–2.49 (m, 2H), 2.70 (tt, *J* = 7.8, 2.2 Hz, 2H), 3.12 (dd, *J* = 8.1, 6.6 Hz, 2H) 3.52 (dt, *J* = 9.6, 7.1 Hz, 2H), 3.79 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.85–3.90 (m, 1H), 4.62 (t, *J* = 3.1 Hz, 1H), 6.59 (d, *J* = 3.7 Hz, 1H), 7.18 (dd, *J* = 8.3, 1.2 Hz, 1H), 7.36 (s, 1H), 7.44 (d, *J* = 3.7 Hz, 1H), 8.32 (d, *J* = 8.3 Hz, 1H); ^{13}C{^{1}H} NMR (100

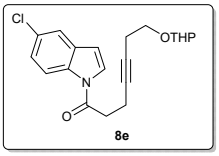


MHz, $CDCl_3$): δ 14.3 (t), 19.5 (t), 20.2 (t), 21.3 (q), 25.4 (t), 30.5 (t), 35.3 (t), 62.3 (t), 66.0 (t), 78.2 (s), 79.1 (s), 98.8 (d), 109.1 (d), 116.2 (d), 120.8 (d), 124.4 (d), 126.5 (d), 130.6 (s), 133.3 (s), 133.8 (s), 169.5 (s) ppm; HRMS (ESI) calcd for $C_{21}H_{25}NNaO_3$: 362.1727 $[M + Na]^+$; found 362.1715.

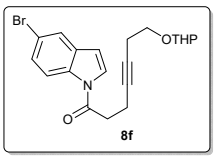
1-(5-Fluoro-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8d**)**:** Following the general procedure, the coupling of indole **4d** (298 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded **8d** (203 mg, 67%) as a colourless solid. R_f = 0.5 (40% ethyl acetate in petroleum ether); mp: 68–70 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.56 (m, 4H), 1.70 (tt, *J* = 12.8, 3.1 Hz, 1H), 1.78–1.84 (m, 1H), 2.46 (tt, *J* = 7.2, 2.2 Hz, 1H), 2.70 (tt, *J* = 7.8, 2.2 Hz, 2H), 3.13 (dd, *J* = 7.0, 1.5 Hz, 2H), 3.47–3.54 (m, 2H), 3.71–3.82 (m, 2H), 3.87 (ddd, *J* = 11.1, 8.1, 3.2 Hz, 1H), 4.62 (t, *J* = 3.0 Hz, 1H), 6.63 (d, *J* = 3.7 Hz, 1H), 7.08 (td, *J* = 9.1, 2.6 Hz, 1H), 7.22 (dd, *J* = 8.6, 2.5 Hz, 1H), 7.52 (d, *J* = 3.7 Hz, 1H), 8.43 (dd, *J* = 9.0, 4.7 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.5 (t), 20.2 (t), 25.4 (t), 30.6 (t), 35.1 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.9 (s), 98.8 (d), 106.3 (dd, $J_{C\text{-}F}$ = 24.4 Hz), 109.5 (dd, $J_{C\text{-}F}$ = 3.8 Hz), 112.9 (dd, $J_{C\text{-}F}$ = 25.1 Hz), 117.6 (dd, $J_{C\text{-}F}$ = 9.1 Hz), 125.9 (d), 131.3 (sd, $J_{C\text{-}F}$ = 9.9 Hz), 132.0 (s), 159.7 (sd, $J_{C\text{-}F}$ = 240.3 Hz), 169.5 (s); ^{19}F NMR (376 MHz, $CDCl_3$): δ –119.25 ppm; HRMS (ESI) calcd for $C_{20}H_{22}NFNaO_3$: 366.1476 $[M + Na]^+$; found 366.1462.



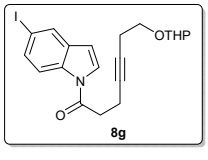
1-(5-Chloro-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8e**)**:** Following the general procedure, the coupling of indole **4e** (335 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8e** (253 mg, 83%) as a colourless liquid. R_f = 0.5 (30% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.60 (m, 4H), 1.69 (tt, *J* = 13.0, 3.1 Hz, 1H) 1.78–1.83 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.70 (tt, *J* = 7.6, 2.3 Hz, 2H), 3.12 (dd, *J* = 8.0, 6.6 Hz, 2H), 3.46–3.55 (m, 2H), 3.78 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.83–3.91 (m, 1H), 4.62 (dd, *J* = 2.8, 1.0 Hz, 1H), 6.60 (dd, *J* = 3.8, 0.6 Hz, 1H), 7.31 (dd, *J* = 8.7, 2.0 Hz, 1H), 7.49 (d, *J* = 3.7 Hz, 1H), 7.54 (d, *J* = 1.7 Hz, 1H), 8.40 (d, *J* = 8.8 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.5 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.2 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.9 (s), 98.8 (d), 108.6 (d), 117.6 (d), 120.5 (d), 125.3 (d), 125.6 (d), 129.3 (s), 131.5 (s), 133.9 (s), 169.6 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}NClNaO_3$: 382.1180 $[M + Na]^+$; found 382.1169.



1-(5-Bromo-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8f**)**:** Following the general procedure, the coupling indole **4f** (433 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8f** (306 mg, 85%) as a colourless solid. R_f = 0.5 (30% ethyl acetate in petroleum ether); mp: 78–80 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.60 (m, 4H), 1.70 (tt, *J* = 13.0, 3.1 Hz, 1H), 1.77–1.88 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.65–2.75 (m, 2H), 3.12 (dd, *J* = 8.1, 6.6 Hz, 2H), 3.46–3.55 (m, 2H), 3.78 (dt, *J* = 9.7, 7.1 Hz, 1H), 3.83–3.92 (m, 1H), 4.61 (dd, *J* = 2.8, 1.3 Hz, 1H), 6.60 (dd, *J* = 3.8, 0.6 Hz, 1H), 7.45 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.48 (d, *J* = 3.7 Hz, 1H), 7.70 (d, *J* = 1.7 Hz, 1H), 8.35 (d, *J* = 8.8 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.5 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.2 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.8 (s), 98.8 (d), 108.4 (d), 117.0 (s), 118.0 (d), 123.5 (d), 125.5 (d), 128.0 (d), 132.0 (s), 134.3 (s), 169.6 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}NBrNaO_3$: 426.0675 $[M + Na]^+$; found 426.0667.

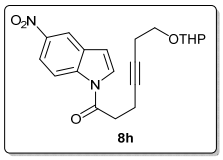


1-(5-Iodo-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8g**)**:** Following the general procedure, the coupling of indole **4g** (537 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) provided compound **8g** (245 mg, 61%) as a pale yellow liquid. R_f = 0.3 (20% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.57 (m, 4H), 1.66–1.74 (m, 1H), 1.77–1.85 (m, 1H), 2.46 (tt, *J* = 7.1, 2.2 Hz, 2H), 2.67–2.73 (m, 2H), 3.12 (t, *J* = 7.3 Hz, 2H), 3.46–3.55 (m, 2H), 3.77 (td, *J* = 9.6, 2.5 Hz, 1H), 3.82–3.91 (m, 1H), 4.61 (t, *J* = 3.0 Hz 1H), 6.59 (d, *J* = 3.7 Hz, 1H), 7.45 (d, *J* = 3.7 Hz, 1H), 7.63 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.92 (d, *J* = 1.6 Hz, 1H), 8.24 (d, *J* = 8.7 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.5 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.3



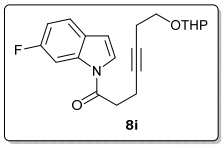
(t), 62.3 (t), 65.9 (t), 78.4 (s), 78.8 (s), 87.9 (s), 98.8 (d), 108.2 (d), 118.4 (d), 125.1 (d), 129.7 (d), 132.6 (s), 133.7 (d), 134.9 (s), 169.7 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}NINaO_3$: 474.0537 [M + Na]$^+$; found 474.0521.

1-(5-Nitro-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8h**)**:** Following the general procedure, the coupling of indole **4h** (358 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) gave compound **8h** (197 mg, 60%) as an yellow solid. R_f = 0.3 (40% ethyl acetate in petroleum ether); mp: 80 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.46–1.59 (m, 4H), 1.70 (tt, *J* = 12.88, 3.50 Hz, 1H), 1.76–1.87 (m, 1H), 2.46 (tt, *J* = 7.13, 2.38 Hz, 2H), 2.73 (tt, *J* = 7.63, 2.38 Hz, 2H), 3.18 (dd, *J* = 7.88, 6.63 Hz, 2H), 3.51 (dt, *J* = 9.76, 7.13 Hz, 2H), 3.78 (dt, *J* = 9.69, 7.16 Hz, 1H), 3.87 (ddd, *J* = 11.29, 8.10, 3.25 Hz, 1H), 4.62 (dd, *J* = 2.88, 1.21 Hz, 1H), 6.82 (dd, *J* = 3.88, 0.63 Hz, 1H), 7.65 (d, *J* = 3.88 Hz, 1H), 8.25 (dd, *J* = 9.19, 2.31 Hz, 1H), 8.50 (d, *J* = 2.13 Hz, 1H), 8.60 (d, *J* = 9.13 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.2 (t), 19.5 (t), 20.16 (t), 25.4 (t), 30.5 (t), 35.4 (t), 62.3 (t), 65.9 (t), 78.5 (s), 78.7 (s), 98.8 (d), 109.7 (d), 116.8 (d), 117.1 (d), 120.5 (d), 127.2 (d), 130.2 (s), 138.6 (s), 144.3 (s), 169.9 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}N_2NaO_5$: 393.1421 [M + Na]$^+$; found 393.1411.



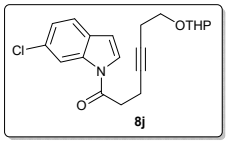
8h

1-(6-Fluoro-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (8i): Following the general procedure, the coupling of indole **4i** (298 mg, 2.21) and acid **5** (200 mg, 0.88 mmol) provided compound **8i** (167 mg, 55%) as a colourless syrup. R_f = 0.2 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.60 (m, 4H), 1.71 (tt, *J* = 13.0, 3.2 Hz, 1H), 1.77–1.85 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.70 (ddt, *J* = 8.3, 6.3, 2.3 Hz, 2H), 3.12 (dd, *J* = 8.1, 6.6 Hz, 2H), 3.47–3.54 (m, 2H), 3.79 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.87 (ddd, *J* = 11.1, 8.0, 3.1 Hz, 1H), 4.62 (t, *J* = 3.0 Hz, 1H), 6.62 (d, *J* = 3.8 Hz, 1H), 7.03 (td, *J* = 8.8, 2.3 Hz, 1H), 7.44–7.50 (m, 2H), 8.22 (dd, *J* = 10.3, 2.1 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.2 (t), 19.5 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.2 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.9 (s), 98.8 (d), 104.0 (dd, $J_{C\text{-}F}$ = 28.9 Hz), 109.0 (d), 111.9 (dd, $J_{C\text{-}F}$ = 24.4 Hz), 121.3 (dd, $J_{C\text{-}F}$ = 9.9 Hz), 124.6 (dd, $J_{C\text{-}F}$ = 3.8 Hz), 126.5 (s), 135.7 (sd, $J_{C\text{-}F}$ = 12.9 Hz),161.3 (sd, $J_{C\text{-}F}$ = 241.0 Hz), 169.8 (s); ^{19}F NMR (376 MHz, $CDCl_3$): δ –116.35 ppm; HRMS (ESI) calcd for $C_{20}H_{22}NFNaO_3$: 366.1476 [M + Na]$^+$; found 366.1467.



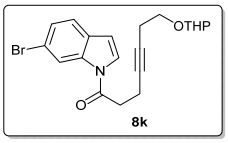
8i

1-(6-Chloro-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8j**)**:** Following the general procedure, the coupling of indole **4j** (335 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) gave compound **8j** (217 mg, 68%) as a pale yellow liquid. R_f = 0.4 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45–1.62 (m, 4H), 1.63–1.74 (m, 1H), 1.76–1.87 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.70 (tt, *J* = 7.7, 2.2 Hz, 2H), 3.11 (t, *J* = 7.3 Hz, 2H), 3.52 (dt, *J* = 9.7, 7.1 Hz, 2H), 3.78 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.87 (ddd, *J* = 11.1, 8.0, 3.2 Hz, 1H), 4.58–4.65 (m, 1H), 6.62 (d, *J* = 3.8 Hz, 1H), 7.22–7.30 (m, 1H), 7.42–7.50 (m, 2H), 8.53 (s, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.3 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.8 (s), 98.8 (d), 109.0 (d), 116.9 (d), 121.4 (d), 124.3 (d), 124.9 (d), 128.7 (s), 131.1 (s), 135.9 (s), 169.7 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}NClNaO_3$: 382.1180 [M + Na]$^+$; found 382.1185.



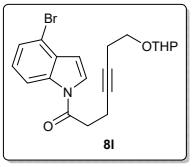
8j

1-(6-Bromo-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8k**)**:** Following the procedure, the coupling of indole **4k** (433 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) gave compound **8k** (201 mg, 56%) as a colourless syrup. R_f = 0.2 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.60 (m, 4H), 1.740 (tt, *J* = 12.8, 3.2 Hz, 1H), 1.768–1.86 (m, 1H), 2.46 (tt, *J* = 7.1, 2.2 Hz, 2H), 2.69 (tt, *J* = 7.3, 2.3 Hz, 2H), 3.11 (t, *J* = 7.3 Hz, 2H), 3.44–3.56 (m, 2H), 3.78 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.87 (ddd, *J* = 11.1, 8.0, 3.1 Hz, 1H), 4.62 (t, *J* = 3.0 Hz, 1H), 6.62 (d, *J* = 3.8 Hz, 1H), 7.36–7.42 (m, 2H), 7.43 (d, *J* = 3.7 Hz, 1H), 8.70 (s, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.2 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.2 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.8 (s), 98.8 (d), 109.0 (d), 118.9 (s), 119.7 (d), 121.8 (d), 124.8 (d), 127.0 (d), 129.1 (s), 136.2 (s), 169.7 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}NBrNaO_3$: 426.0675 [M + Na]$^+$; found 426.0663.



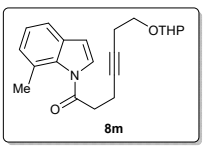
8k

1-(4-Bromo-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8l**)**:** Prepared by following procedure of coupling indole **4l** (433 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8l** as colourless sticky liquid (210 mg, 58%); R_f = 0.2 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.55 (m, 4H), 1.71 (tt, *J* = 12.8, 3.2 Hz, 1H), 1.83 (dd, *J* = 9.8, 3.1 Hz, 1H), 2.47 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.72 (ddt, *J* = 8.3, 6.3, 2.3 Hz, 2H), 3.15 (dd, *J* = 8.0, 6.6 Hz, 2H), 3.46–3.56 (m, 2H), 3.80 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.84–3.94 (m, 1H), 4.62 (dd, *J* = 4.0, 3.0 Hz, 1H), 6.73 (d, *J* = 3.8 Hz, 1H), 7.22 (t, *J* = 8.0 Hz, 1H), 7.45 (dd, *J* = 7.7, 0.5 Hz, 1H), 7.54 (d, *J* = 3.7 Hz, 1H), 8.44 (d, *J* = 8.3 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 19.5 (t), 20.2 (t), 25.4 (t), 30.5 (t), 35.4 (t), 62.3 (t), 65.9 (t), 78.4 (s), 78.8 (s), 98.8 (d), 109.1 (d), 114.6 (s), 115.6 (d), 124.9 (d), 126.2 (d), 126.7 (d), 131.0 (s), 135.9 (s), 169.8 (s) ppm; HRMS (ESI) calcd for $C_{20}H_{22}NBrNaO_3$: 426.0675 [M + Na]$^+$; found 426.0666.



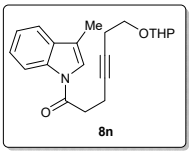
8l

1-(7-Methyl-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8m**)**:** Following the general procedure, the coupling of indole **4m** (290 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8m** (107 mg, 35%) as a colourless oil; R_f = 0.3 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.49–1.57 (m, 4H), 1.70 (tt, *J* = 9.8, 3.1 Hz, 1H), 1.81 (dd, *J* = 9.1, 3.5 Hz, 1H), 2.46 (tt, *J* = 7.2, 2.3 Hz, 2H), 2.56 (s, 3H), 2.71 (tt, *J* = 7.9, 2.3 Hz, 2H), 3.15 (t, *J* = 7.2 Hz, 2H), 3.4–3.54 (m, 2H), 3.78 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.86 (ddd, *J* = 11.1, 7.8, 3.1 Hz, 1H), 4.6 (dd, *J* = 3.0, 1.5 Hz, 1H), 6.65 (d, *J* = 3.8 Hz, 1H), 7.15 (d, *J* = 7.3 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.41 (d, J = 7.3 Hz, 1H), 7.44 (d, *J* = 3.7 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 15.1 (t), 19.4 (t), 20.15 (t), 22.6 (q), 25.4 (t), 30.5 (t), 36.1 (t), 62.3 (t), 65.9 (t), 78.4 (s), 79.0 (s), 98.8 (d), 109.2 (d), 118.5 (d), 124.1 (d), 125.7 (d), 126.7 (s), 128.2 (d), 131.9 (s), 135.1 (s), 169.2 (s) ppm; HRMS (ESI) calcd for $C_{21}H_{25}NNaO_3$: 362.1727 [M + Na]$^+$; found 362.1717.



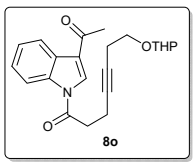
8m

1-(3-Methyl-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8n**)**:** Following the general procedure, coupling of indole **4n** (290 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8n** (183 mg, 61%) as a colourless syrup. R_f = 0.8 (30% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48–1.52 (m, 4H), 1.70 (tt, *J* = 12.8, 3.2 Hz, 1H), 1.78–1.87 (m, 1H), 2.29 (d, *J* = 1.2 Hz, 3H), 2.47 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.69 (tt, *J* = 7.4, 2.3 Hz, 2H), 3.09 (dd, *J* = 8.3, 6.6 Hz, 2H), 3.46–3.56 (m, 2H), 3.80 (ddd, *J* = 9.6, 7.2, 2.3 Hz, 1H), 3.88 (ddd, *J* = 11.1, 8.0, 3.1 Hz, 1H), 4.62 (t, *J* = 3.1 Hz, 1H), 7.23 (s, 1H), 7.30 (td, *J* = 7.4, 1.1 Hz, 1H), 7.36 (td, *J* = 7.6, 1.3 Hz, 1H), 7.50 (dd, *J* = 7.6, 0.6 Hz, 1H), 8.44 (d, *J* = 7.7 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 9.7 (q), 14.3 (d), 19.4 (d), 20.2 (d), 25.4 (d), 30.5 (d), 35.4 (d), 62.2 (d), 65.9 (d), 78.1 (s), 79.2 (s), 98.7 (d), 116.6 (d), 118.6 (s), 118.8 (d), 121.3 (d), 123.4 (d), 125.2 (d), 131.3 (s), 135.8 (s), 169.3 (s) ppm; HRMS (ESI) calcd for $C_{21}H_{25}NNaO_3$: 362.1737 [M + Na]$^+$; found 362.1718.



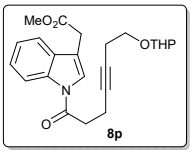
8n

1-(3-Acetyl-1*H*-indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8o**)**:** Following the general procedure, the coupling of indole **4o** (351 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) gave the compound **8o** (179 mg, 55%) as a pale yellow oil. R_f = 0.6 (40% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.46–1.60 (m, 4H), 1.69 (dt, *J* = 8.7, 3.3 Hz, 1H), 1.75–1.85 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.58 (s, 3H), 2.73 (tt, *J* = 7.6, 2.3 Hz, 2H), 3.19 (dd, *J* = 8.0, 6.6 Hz, 2H), 3.50 (dt, *J* = 9.6, 7.1 Hz, 2H), 3.79 (dt, *J* = 9.7, 7.1 Hz, 1H), 3.86 (ddd, *J* = 11.2, 8.0, 3.2 Hz, 1H), 4.56–4.64 (dd, *J* = 2.8, 1.5 Hz, 1H), 7.36–7.46 (m, 2H), 8.07 (s, 1H), 8.34 (ddd, *J* = 6.3, 2.6, 0.6 Hz, 1H), 8.39 (ddd, *J* = 7.0, 2.6, 0.6 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.2 (t), 19.4 (t), 20.14 (t), 25.3 (t), 27.9 (q), 30.5 (t), 35.4 (t), 62.3 (t), 65.9 (t), 78.6 (s), 78.7 (s), 98.8 (d), 116.1 (d), 121.9 (s), 122.5 (d), 125.2 (d), 126.3 (d), 127.1 (s), 130.4 (d), 136.0 (s), 169.9 (s), 193.6 (s) ppm; HRMS (ESI) calcd for $C_{22}H_{25}NNaO_4$: 390.1676 [M + Na]$^+$; found 390.1663.

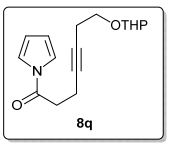


8o

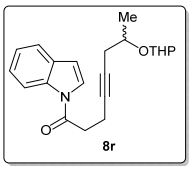
Methyl 2-(1-(7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-ynoyl)-1*H*-indol-3-yl)acetate (**8p**)**:** Following the procedure, the coupling of indole **4p** (418 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) afforded compound **8p** (162 mg, 46%) as a colourless syrup. R_f = 0.3 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45–1.62 (m, 5H), 1.67–1.69 (m, 1H), 1.69–1.74 (m, 1H), 1.77–1.86 (m, 1H), 2.46 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.66–2.73 (m, 2H), 3.13 (dd, *J* = 8.2, 6.6 Hz, 2H), 3.47–3.55 (m, 2H), 3.74 (s, 3H), 3.78 (dt, *J* = 9.7, 7.2 Hz, 1H), 3.87 (ddd, *J* = 11.2, 8.0, 3.2 Hz, 1H), 4.62 (t, *J* = 2.8 Hz, 1H), 7.31 (td, *J* = 7.4, 1.0 Hz, 1H), 7.38 (td, *J* = 7.7, 1.3 Hz, 1H), 7.49–7.55 (m, 2H), 8.45 (d, *J* = 8.1 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.2 (t), 19.4 (t), 20.2 (t), 25.4 (t), 30.5 (t), 30.7 (t), 35.3 (t), 52.2 (q), 62.2 (t), 65.9 (t), 78.2 (s), 79.0 (s), 98.7 (d), 115.1 (s), 116.7 (d), 118.8 (d), 122.9 (d), 123.7 (d), 125.5 (d), 129.9 (s), 135.7 (s), 169.5 (s), 171.2 (s) ppm; HRMS (ESI) calcd for $C_{23}H_{27}NNaO_5$: 420.1781 [M + Na]$^+$; found 420.1778.



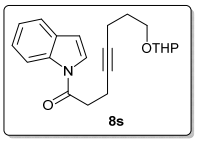
1-(1*H*-Pyrrol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)hept-4-yn-1-one (**8q**)**:** Following the procedure, the coupling of pyrrole **4q** (148 mg, 2.21 mmol) and acid **5** (200 mg, 0.88 mmol) gave compound **8q** (80 mg, 33%) as a colourless syrup. R_f = 0.6 (30% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45–1.65 (m, 4H), 1.65–1.75 (m, 1H), 1.76–1.89 (m, 1H), 2.45 (tt, *J* = 7.1, 2.3 Hz, 2H), 2.64 (ddt, *J* = 8.4, 6.4, 2.3 Hz, 2H), 3.04 (dd, *J* = 8.1, 6.6 Hz, 2H), 3.45–3.57 (m, 2H), 3.78 (dt, *J* = 9.6, 7.1 Hz, 1H), 3.87 (tt, *J* = 8.3, 3.0 Hz, 1H), 4.60–4.65 (m, 1H), 6.30 (t, *J* = 2.3 Hz, 2H), 7.32 (br. s., 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.1 (t), 19.4 (t), 20.1 (t), 25.4 (t), 30.5 (t), 34.2 (t), 62.2 (t), 65.9 (t), 78.2 (s), 78.8 (s), 98.7 (d), 113.2 (d, 2C), 118.9 (d, 2C), 168.9 (s) ppm; HRMS (ESI) calcd for $C_{16}H_{21}N\,Na\,O_3$: 298.1414 [M + Na]$^+$; found 298.1406.



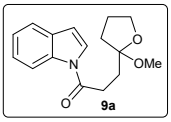
1-(1*H*-Indol-1-yl)-7-((tetrahydro-2*H*-pyran-2-yl)oxy)oct-4-yn-1-one (**8r**)**:** Following the procedure, the coupling of indole **4a** (244 mg, 2.08 mmol) and acid **5b** (200 mg, 0.83 mmol) gave compound **8r** (198 mg, 70%) as a colourless oil; R_f = 0.5 (10% ethyl acetate in petroleum ether); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.23 (d, *J* = 6.1 Hz, 3H), 1.31 (d, *J* = 6.4 Hz, 2.85H), 1.47–1.61 (m, 8H), 1.68–1.76 (m, 3H), 1.78–1.89 (m, 2H), 2.26–2.33 (m, 1.5H), 2.35–2.45 (m, 1.5H), 2.53–2.57 (m, 1H), 2.73 (tt, *J* = 8.6, 2.5 Hz, 3.8H), 3.16 (td, *J* = 7.3, 3.6 Hz, 3.7H), 3.46–3.55 (m, 1.95H), 3.85–4.00 (m, 3.79H), 4.71–4.76 (m, 1.87H), 6.68 (d, *J* = 3.8 Hz, 1.79H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.38 (7.6 Hz, 1.82H), 7.49 (m, 1.76H), 7.59 (d, *J* = 7.6 Hz, 1.77H), 8.48 (d, *J* = 8.3 Hz, 1.75H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 14.3 (t), 14.4 (t), 19.0 (q), 19.5 (t), 19.8 (t), 21.1 (q), 25.4 (t), 25.5 (t), 26.1 (t), 27.5 (t), 30.9 (t), 31.0 (t), 35.4 (t), 62.4 (t), 62.7 (t), 71.0 (d), 71.4 (d), 78.2 (s), 78.5 (s), 79.5 (s) 96.7 (d), 97.8 (d), 109.3 (d), 116.6 (d), 120.8 (d), 123.7 (d), 124.4 (d), 125.2 (d), 130.3 (s), 135.6 (s), 169.7 (s), 169.7 (s) ppm; HRMS (ESI) calcd for $C_{21}H_{25}N\,Na\,O_3$: 362.1727 [M + Na]$^+$; found 362.1720.



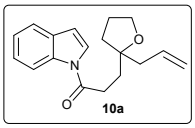
1-(1*H*-Indol-1-yl)-8-((tetrahydro-2*H*-pyran-2-yl)oxy)oct-4-yn-1-one (**8s**)**:** Following the procedure, the coupling of indole **4a** (244 mg, 2.08 mmol) and acid **5a** (200 mg, 0.83 mmol) gave compound **8s** (117 mg, 41%) as a colourless oil. R_f = 0.5 (10% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45–1.61 (m, 4H), 1.66–1.85 (m, 4H), 2.22–2.31 (m, 2H), 2.70 (tt, *J* = 7.8, 2.3 Hz, 2H), 3.14 (dd, *J* = 8.2, 6.6 Hz, 2H), 3.41–3.53 (m, 2H), 3.79 (dt, *J* = 9.7, 3.3 Hz, 1H), 3.6 (tt, *J* = 11.2, 3.2 Hz, 1H), 4.57 (t, *J* = 3.4 Hz, 1H), 6.66 (d, *J* = 3.7 Hz, 1H), 7.24–7.32 (m, 1H), 7.33–7.40 (m, 1H), 7.48 (d, *J* = 3.8 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 1H), 8.47 (d, *J* = 8.2 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 14.3 (t), 15.6 (t), 19.5 (t), 25.4 (t), 29.0 (t), 30.7 (t), 35.5 (t), 62.2 (t), 66.0 (t), 78.2 (s), 80.9 (s), 98.8 (d), 109.3 (d), 116.6 (d), 120.8 (d), 123.7 (d), 124.4 (d), 125.2 (d), 130.3 (s), 135.6 (s), 169.8 (s) ppm; HRMS (ESI) calcd for $C_{21}H_{25}N\,Na\,O_3$: 362.1727 [M + Na]$^+$; found 362.1717.



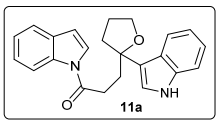
1-(1*H*-Indol-1-yl)-3-(2-methoxytetrahydrofuran-2-yl)propan-1-one (**9a**): Cyclization of alkynol **1a** (30 mg, 0.12 mmol) in the presence of methanol (10 µL, 0.24 mmol) provided compound **9a** (22.5 mg, 66%) as a colourless syrup. R_f = 0.5 (20% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.68–1.73 (m, 1H), 1.81–1.91 (m, 1H), 1.92–2.02 (m, 2H), 2.11 (ddd, *J* = 6.7, 14.3, 9.0 Hz, 1H), 2.32 (ddd, *J* = 14.5, 8.9, 7.0 Hz, 1H), 2.92–2.96 (m, 2H), 3.15 (s, 3H), 3.81–3.90 (m, 2H), 6.56 (dd, *J* = 3.9, 0.5 Hz, 1H), 7.20 (td, *J* = 7.5, 1.1 Hz, 1H), 7.28 (td, *J* = 7.7, 1.3 Hz, 1H), 7.44 (d, *J* = 3.8 Hz, 1H), 7.49 (d, *J* = 7.7 Hz, 1H), 8.39 (d, *J* = 8.2 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 24.5 (t), 29.8 (t), 31.2 (t), 35.0 (t), 48.4 (q), 67.9 (t), 108.7 (s), 109.1 (d), 116.6 (d), 120.8 (d), 123.6 (d), 124.7 (d), 125.1 (d), 130.3 (s), 135.6 (s), 171.2 (s) ppm; HRMS (ESI) calcd for $C_{16}H_{19}NNaO_3$: 296.1257 [M + Na]$^+$; found 296.1252.



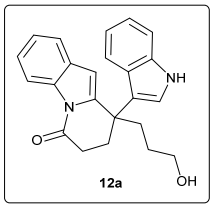
3-(2-Allyltetrahydrofuran-2-yl)-1-(1*H*-indol-1-yl)propan-1-one (**10a**): Cyclization of alkynol **1a** (30 mg, 0.12 mmol) in the presence of trimethylallyl silane (0.2 mL, 1.2 mmol) gave compound **10a** (22.2 mg, 63%)as a colourless liquid. R_f = 0.8 (40% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.73 (dt, *J* = 12.5, 6.6 Hz, 1H), 1.84–1.92 (m, 1H), 1.92–2.00 (m, 2H), 2.05 (t, *J* = 8.0 Hz, 2H), 2.30–2.41 (m, 2H), 3.02 (dt, *J* = 13.3, 7.8 Hz, 2H), 3.87 (t, *J* = 6.6 Hz, 2H), 5.08–5.19 (m, 2H), 5.83 (ddt, *J* = 17.1, 10.1, 7.2 Hz, 1H), 6.65 (dd, *J* = 3.7, 0.6 Hz, 1H), 7.24–7.31 (m, 1H), 7.36 (td, *J* = 7.7, 1.3 Hz, 1H), 7.49–7.60 (m, 2H), 8.47 (d, *J* = 8.2 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 26.4 (t), 31.1 (t), 33.6 (t), 35.1 (t), 43.7 (t), 68.1 (t), 83.8 (s), 109.4 (d), 116.9 (d), 118.4 (t), 121.1 (d), 123.9 (d), 125.1 (d), 125.4 (d), 130.7 (s), 134.5 (d), 135.9 (s), 172.1 (s) ppm; HRMS (ESI) calcd for $C_{18}H_{21}NNaO_2$: 306.1465 [M + Na]$^+$; found 306.1472.



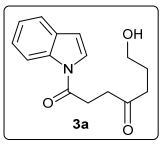
3-(2-(1*H*-Indol-3-yl)tetrahydrofuran-2-yl)-1-(1*H*-indol-1-yl)propan-1-one (**11a**): Cyclization of alkynol **1a** (30 mg, 0.12 mmol) in the presence of indole **4a** (15 mg, 0.13 mmol) gave compound **11a** as colourless syrup (32.1 mg, 72%); R_f = 0.7 (40% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.89–2.00 (m, 1H), 2.00–2.11 (m, 1H), 2.22 (dt, *J* = 12.0, 7.8 Hz, 1H), 2.39–2.48 (m, 2H), 2.59–2.74 (m, 2H), 3.10 (tt, *J* = 12.0, 5.0 Hz, 1H), 3.97–4.06 (m, 2H), 6.51 (dd, *J* = 3.7, 0.5 Hz, 1H), 7.11–7.16 (m, 1H), 7.18 (d, *J* = 2.3 Hz, 1H), 7.19–7.26 (m, 2H), 7.27–7.33 (m, 2H), 7.35–7.40 (m, 1H), 7.48–7.54 (m, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 8.07 (br. s., 1H), 8.38 (d, *J* = 8.1 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 25.9 (t), 31.9 (t), 35.6 (t), 38.3 (t), 67.5 (t), 83.7 (s), 108.6 (d), 111.3 (d), 116.5 (d), 119.7 (d), 120.0 (d), 120.6 (d), 120.7 (s), 121.5 (d), 122.1 (d), 123.4 (d), 124.8 (s), 124.9 (d), 124.9 (s), 130.3 (s), 135.5 (s), 137.1 (s), 172.0 (s) ppm; HRMS (ESI) calcd for $C_{23}H_{22}N_2NaO_2$: 381.1573 [M + Na]$^+$; found 381.12585.



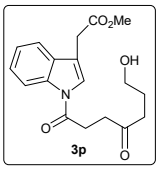
9-(3-Hydroxypropyl)-9-(1*H*-indol-3-yl)-8,9-dihydropyrido[1,2-*a*]indol-6(7*H*)-one (**12a**): Cyclization of alkynol **1a** (30 mg, 0.12 mmol) in the presence of indole **4a** (15 mg, 0.13 mmol) in CH_3CN solvent gave compound **12a** as pale brown gum (33 mg, 74%); R_f = 0.2 (50% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.47 –1.64 (m, 2H), 2.13 –2.31 (m, 2H), 2.34–2.49 (m, 2H), 2.69 (dt, *J* = 13.2, 4.9 Hz, 1H), 2.80 (dt, *J* = 17.7, 5.0 Hz, 1H), 3.42 (q, *J* = 6.0 Hz, 2H), 4.45 (t, *J* = 5.0 Hz, 1H), 6.56 (s, 1H), 6.72 (d, *J* = 2.2 Hz, 1H), 6.88–6.95 (m, 1H), 7.06 (t, *J* = 7.3 Hz, 1H), 7.25–7.31 (m, 2H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.56–7.61 (m, 1H), 8.35 (dd, *J* = 6.3, 2.0 Hz, 1H), 10.92 (s, 1H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6): δ 28.1 (s), 30.3 (s), 31.0 (s), 35.2 (s), 39.6 (s), 61.1 (s), 105.0 (d), 112.0 (d), 115.7 (d), 117.9 (s), 118.5 (d), 119.9 (d), 120.3 (d), 120.9 (d), 123.7 (d), 123.9 (d), 124.9 (s), 129.2 (s), 134.4 (s), 137.2 (s), 144.7 (s), 169.2 (s), ppm; HRMS (ESI) calcd for $C_{23}H_{22}N_2NaO_2$: 381.1573 [M + Na]$^+$; found 381.1583.



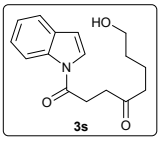
7-Hydroxy-1-(1*H*-indol-1-yl)heptane-1,4-dione (3a): Colourless syrup (5 mg, 15%); R_f = 0.2 (40% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.82 (br. s., 1H), 1.92 (quin, *J* = 6.5 Hz, 2H), 2.73 (t, *J* = 6.9 Hz, 2H), 2.97 (t, *J* = 6.2 Hz, 2H), 3.26 (t, *J* = 6.2 Hz, 2H), 3.69 (t, *J* = 6.0 Hz, 2H), 6.66 (d, *J* = 3.7 Hz, 1H), 7.23–7.29 (m, 1H) 7.31–7.37 (m, 1H), 7.53 (d, *J* = 3.7 Hz, 1H), 7.57 (d, *J* = 7.5 Hz, 1H), 8.40 (d, *J* = 8.1 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 26.5 (t), 29.7 (t), 36.5 (t), 39.5 (t), 62.1 (t), 109.4 (d), 116.5 (d), 120.9 (d), 123.7 (d), 124.4 (d), 125.1 (d), 130.3 (s), 135.6 (s), 170.3 (s), 209.4 (s) ppm; HRMS (ESI) calcd for $C_{15}H_{17}NNaO_3$: 282.1101 [M + Na]$^+$; found 282.1106.



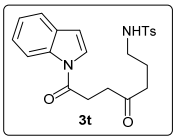
Methyl 2-(1-(7-hydroxy-4-oxoheptanoyl)-1*H*-indol-3-yl)acetate (**3p**)**:** Colourless liquid (12 mg, 37%); R_f = 0.1 (50% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.83–1.96 (m, 2H), 2.73 (t, *J* = 6.9 Hz, 2H), 2.96 (t, *J* = 6.1 Hz, 2H), 3.25 (t, *J* = 6.2 Hz, 2H), 3.67–3.71 (m, 2H), 3.71–3.76 (m, 5H), 7.28–7.33 (m, 1H), 7.33–7.39 (m, 1H), 7.49–7.57 (m, 2H), 8.39 (d, *J* = 8.1 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 26.5 (t), 29.7 (t), 30.8 (t), 36.5 (t), 39.5 (t), 52.2 (q), 62.1 (t), 115.2 (s), 116.6 (d), 118.8 (d), 123.1 (d), 123.7 (d), 125.5 (d), 130.0 (s), 135.8 (s), 170.1 (s), 171.3 (s), 209.3 (s) ppm; HRMS (ESI) calcd for $C_{18}H_{21}NNaO_5$: 354.1312 [M + Na]$^+$; found 354.1307.



8-Hydroxy-1-(1*H*-indol-1-yl)octane-1,4-dione (**3s**)**:** Colourless oil (24.5 mg, 76%); R_f = 0.2 (40% ethyl acetate in petroleum ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.63 (br. s., 1H), 1.82–1.91 (m, 2H), 2.13 (quin, *J* = 6.9 Hz, 2H), 2.59 (t, *J* = 6.8 Hz, 2H), 2.68 (t, *J* = 6.8 Hz, 2H), 2.98 (t, *J* = 7.0 Hz, 2H), 3.67 (t, *J* = 6.0 Hz, 2H), 6.65 (d, *J* = 3.7 Hz, 1H), 7.28 (td, *J* = 1.0, 7.4 Hz, 1H), 7.34–7.39 (m, 1H), 7.51 (d, *J* = 3.8 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 1H), 8.46 (d, *J* = 8.2 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 18.6 (t), 26.5 (t), 34.7 (t), 39.6 (t), 41.3 (t), 62.2 (t), 109.3 (d), 116.5 (d), 120.8 (d), 123.7 (d), 124.7 (d), 125.1 (d), 130.4 (s), 135.6 (s), 171.1 (s), 210.8 (s) ppm; HRMS (ESI) calcd for $C_{16}H_{19}NNaO_3$: 296.1257 [M + Na]$^+$; found 296.1252.



N-(7-(1*H*-indol-1-yl)-4,7-dioxoheptyl)-4-methylbenzenesulfonamide (**3t**)**:** Prepared from alkynylamine **1t** (50 mg, 0.12 mmol). Pale brown solid (31 mg, 60%); R_f = 0.5 (40% ethyl acetate in petroleum ether); mp: 94–96 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.83 (quin, *J* = 6.6 Hz, 1H), 2.41 (s, 3H), 2.69 (t, *J* = 6.6 Hz, 2H), 2.91 (dd, *J* = 6.7, 5.8 Hz, 2H), 2.99 (q, *J* = 6.4 Hz, 2H) 3.19–3.30 (m, 2H), 4.72 (t, *J* = 6.3 Hz, 1H), 6.67 (d, *J* = 3.7 Hz, 1H), 7.28–7.32 (m, 3H), 7.32–7.37 (m, 1H), 7.50–7.60 (m, 2H), 7.74 (d, *J* = 8.2 Hz, 2H), 8.38 (d, *J* = 8.1 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 21.5 (q), 23.3 (t), 29.8 (t), 36.5 (t), 39.4 (t), 42.4 (t), 109.5 (d), 116.5 (d), 120.9 (d), 123.7 (d), 124.5 (d), 125.1 (d), 127.0 (d, 2C), 129.7 (d, 2C), 130.3 (s), 135.6 (s), 137.0 (s), 143.4 (s), 170.3 (s), 208.7 (s) ppm; HRMS (ESI) calcd for $C_{22}H_{24}N_2SNaO_4$: 435.1349 [M + Na]$^+$; found 435.1360.



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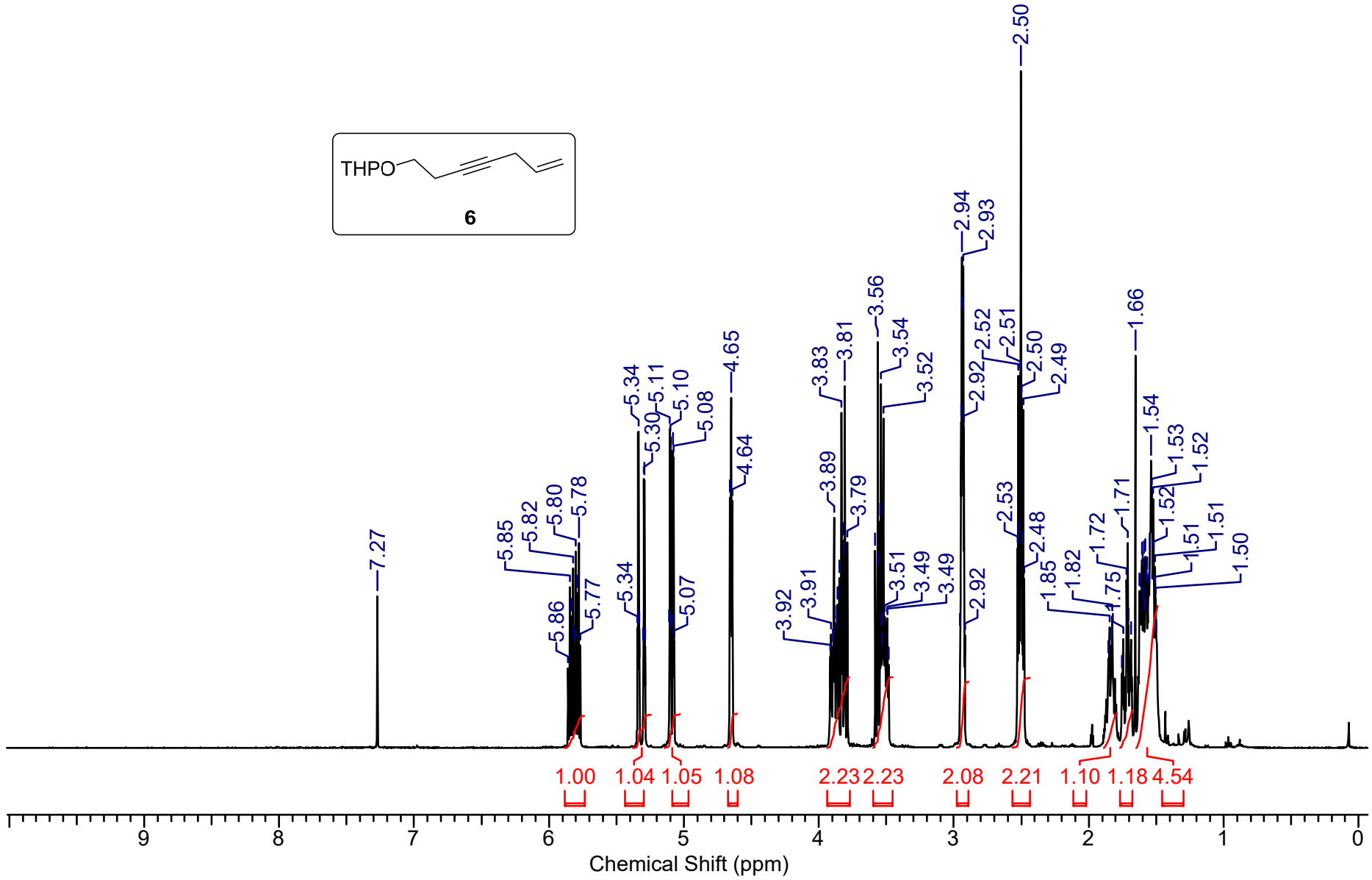
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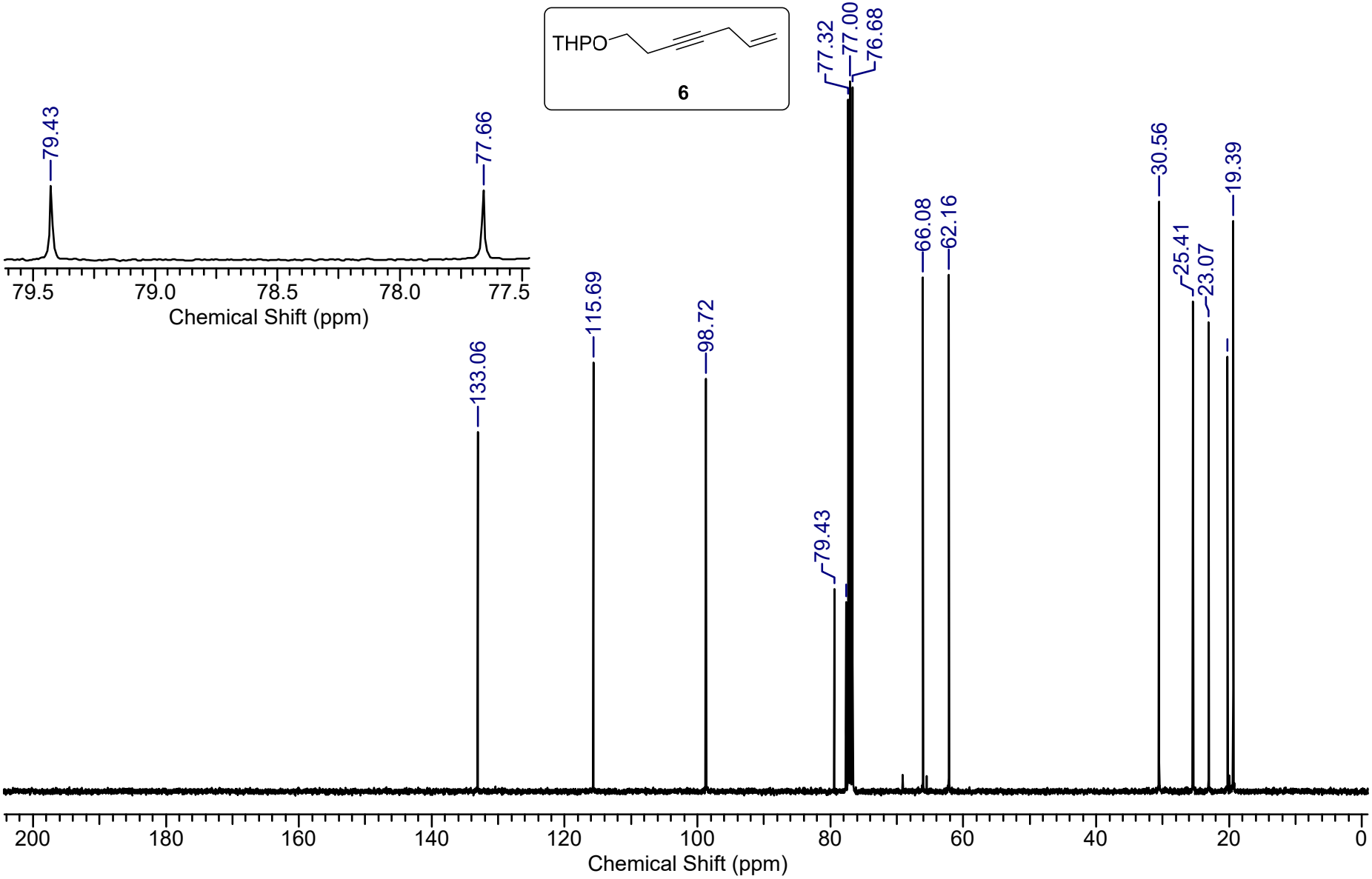
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100
80
60
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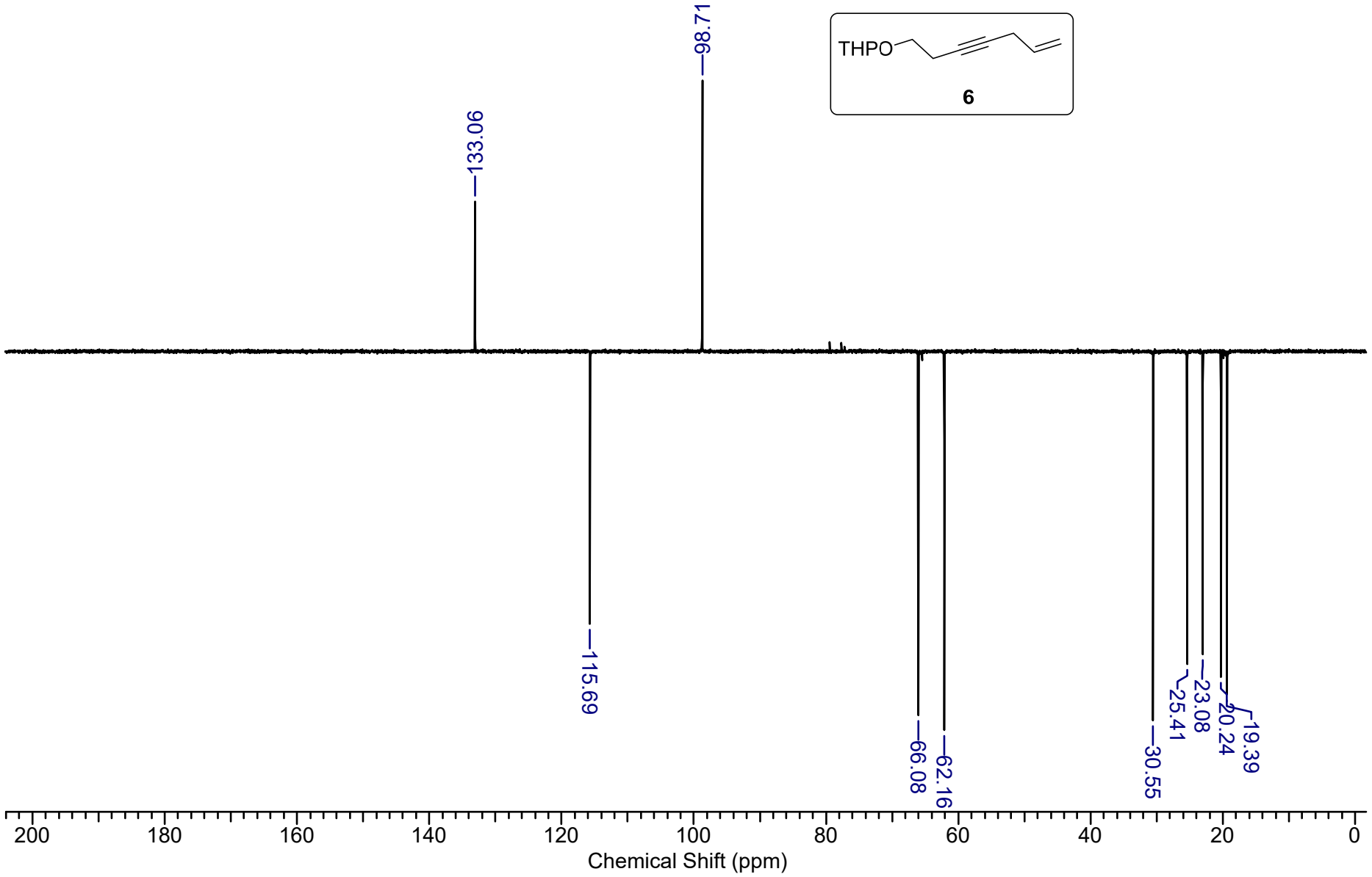
Chemical Shift (ppm)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120
100
80
60
40
20
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Chemical Shift (ppm)

M-1 #522 RT: 3.03 AV: 1 NL: 1.04E8
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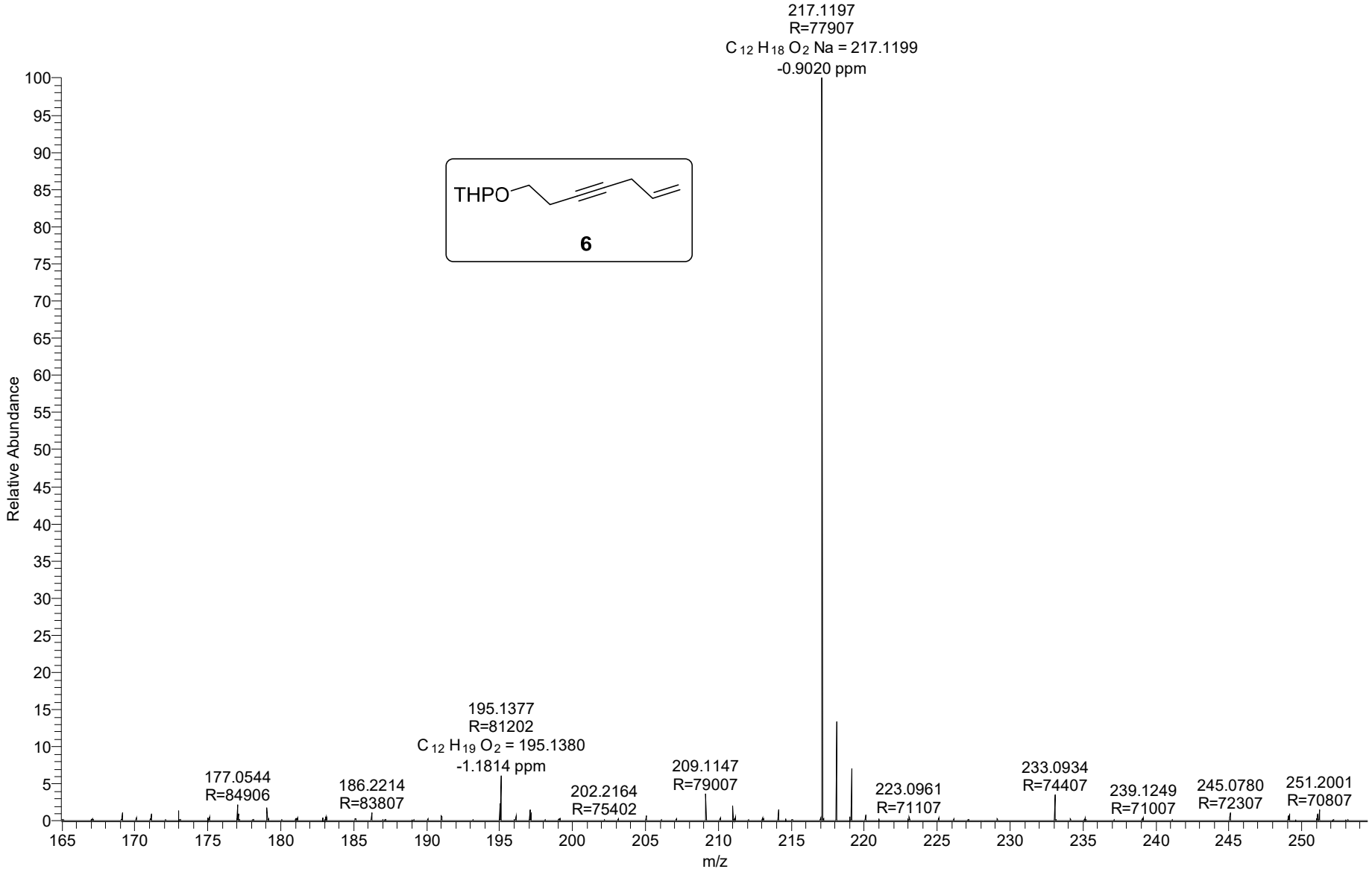

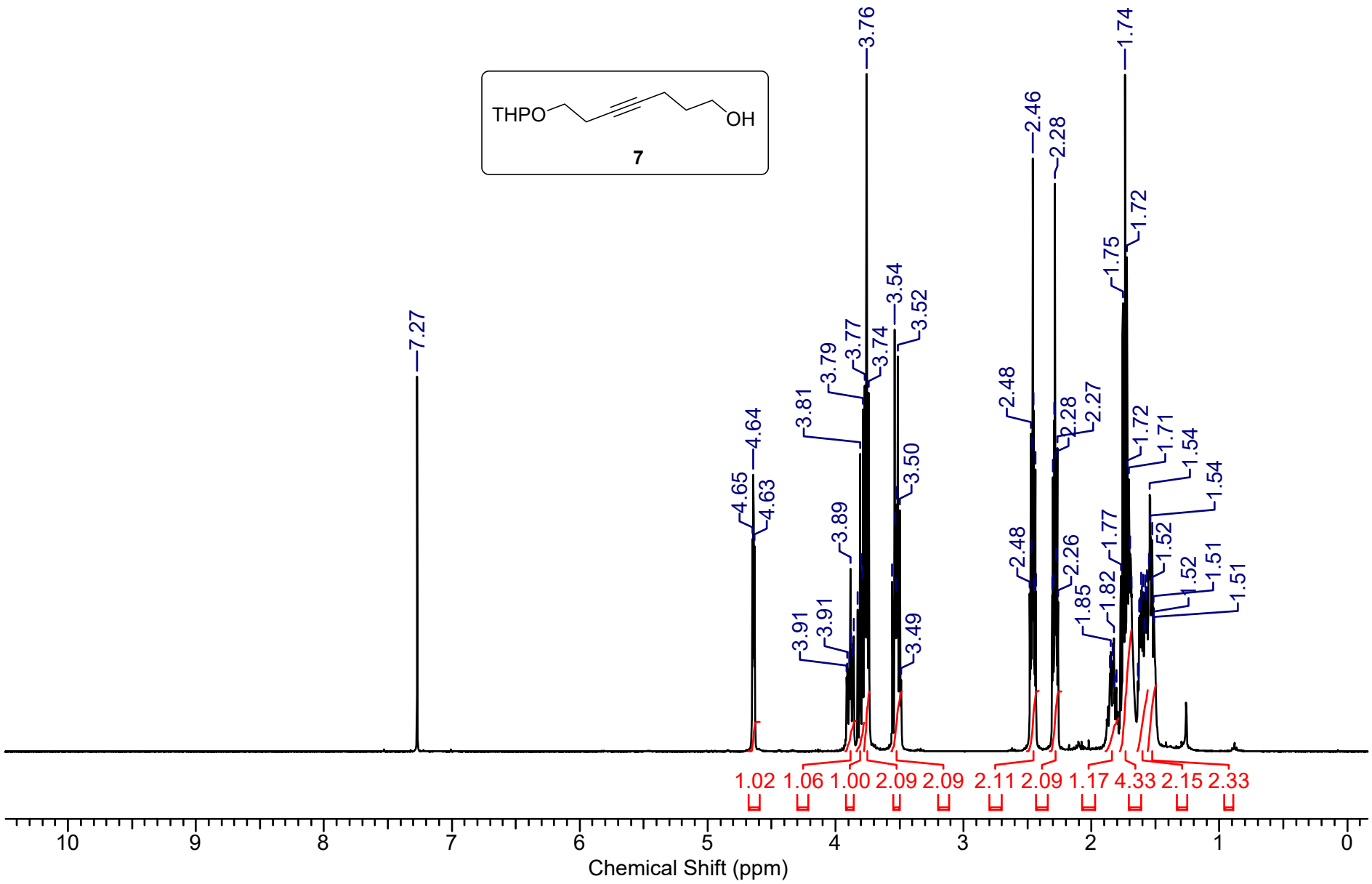
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1.52
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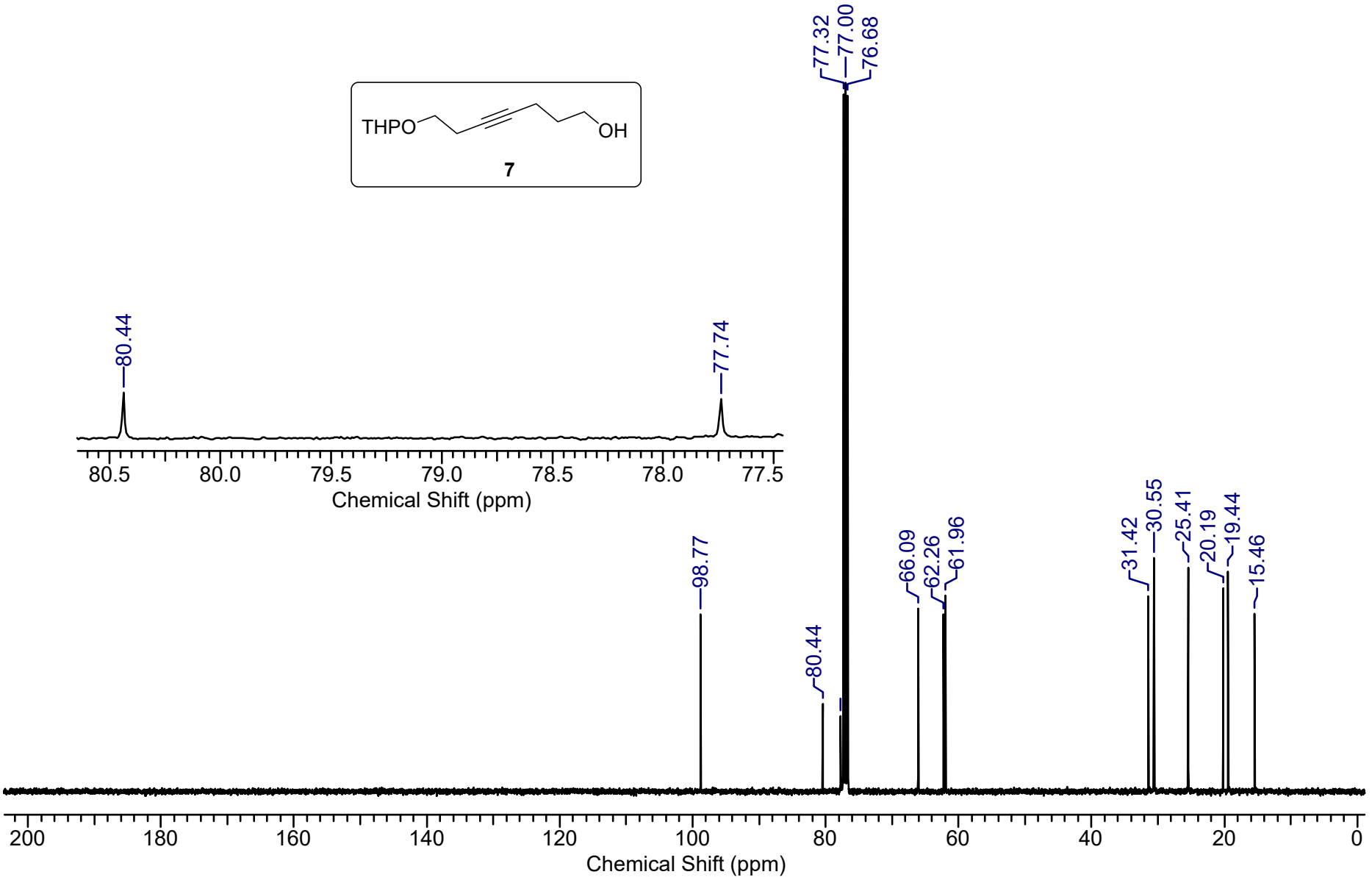
Chemical Shift (ppm)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78.0
77.5
Chemical Shift (ppm)
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61.96
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30.55
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80
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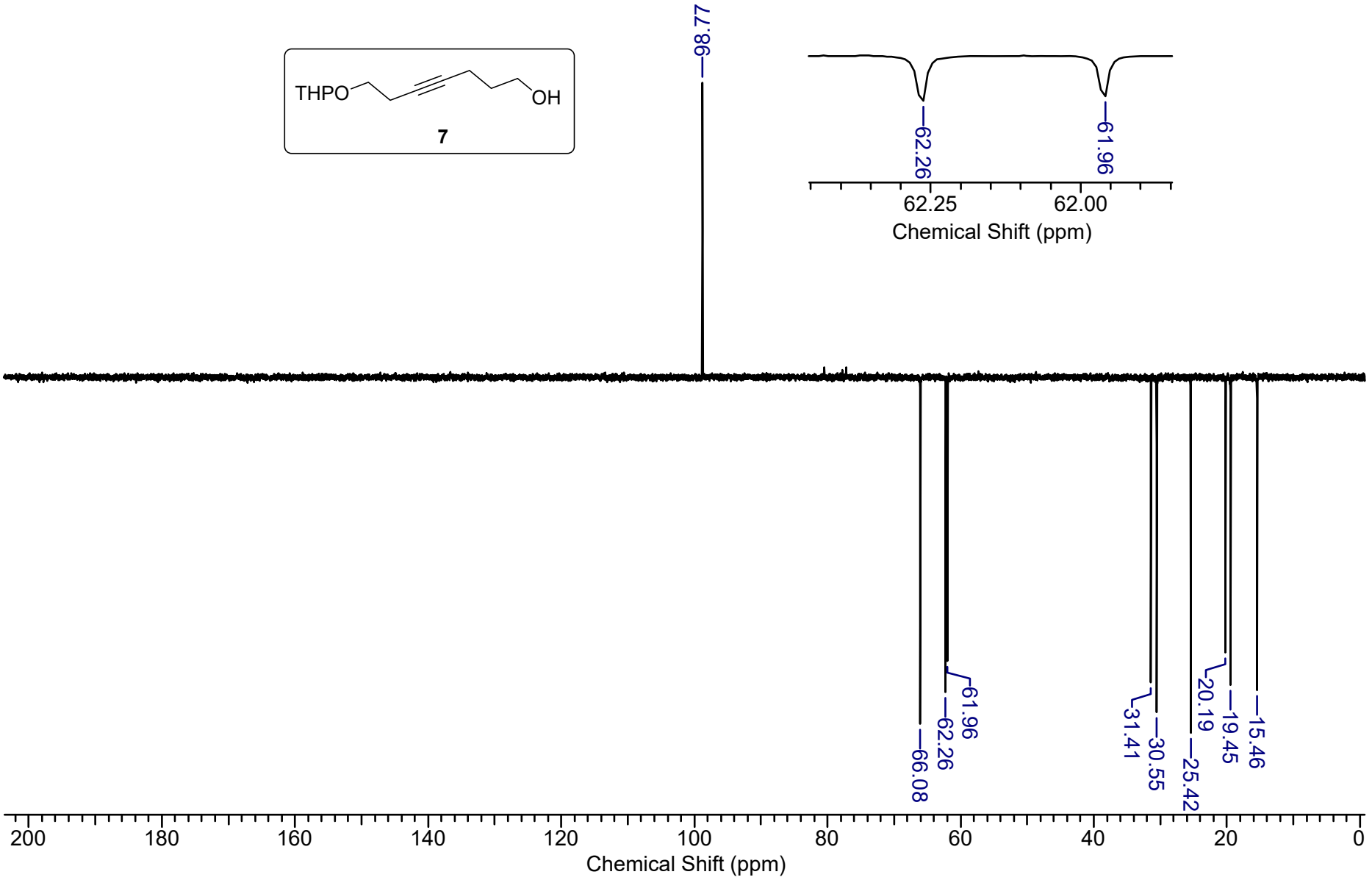
Chemical Shift (ppm)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80
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40
20
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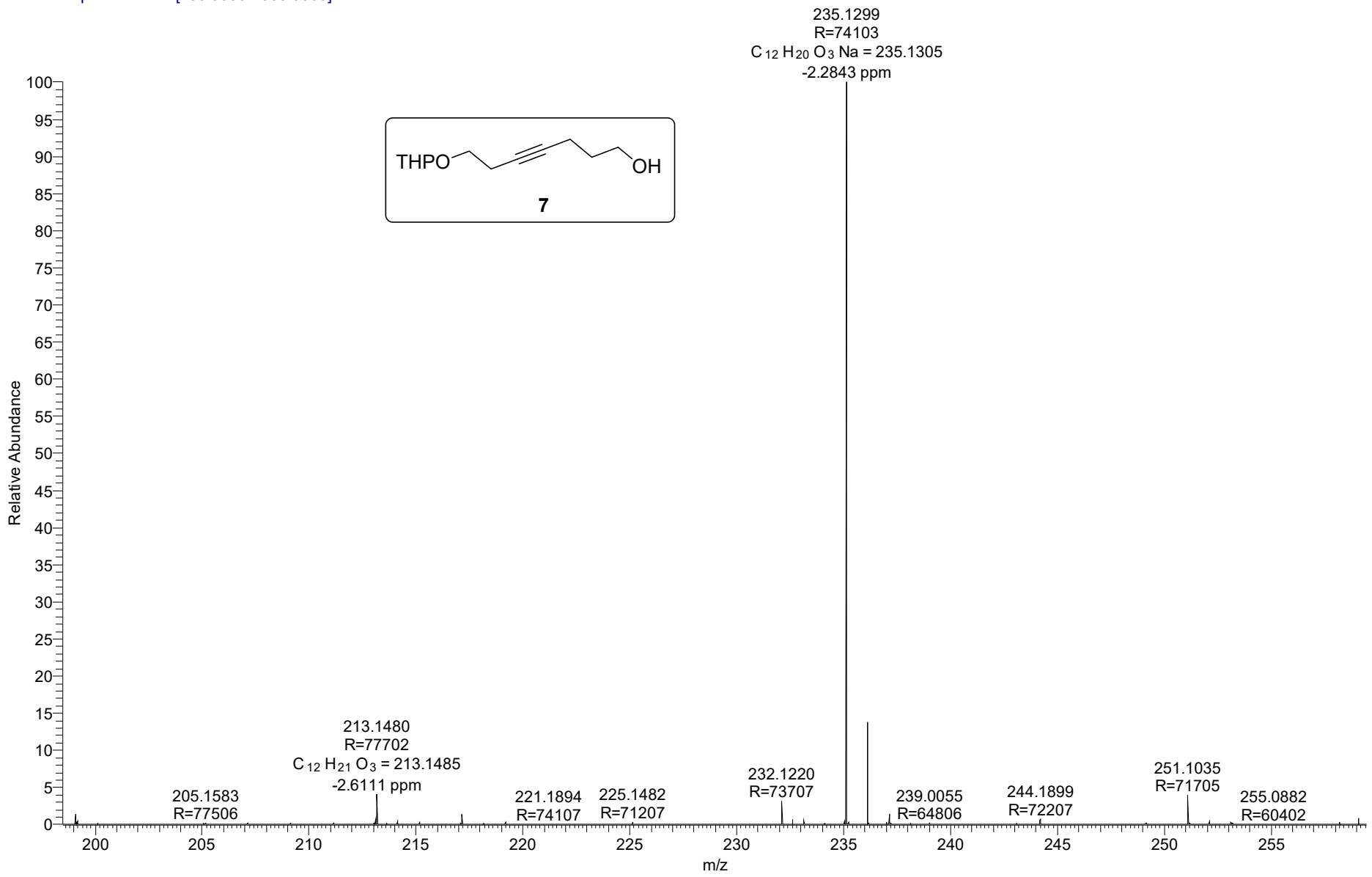
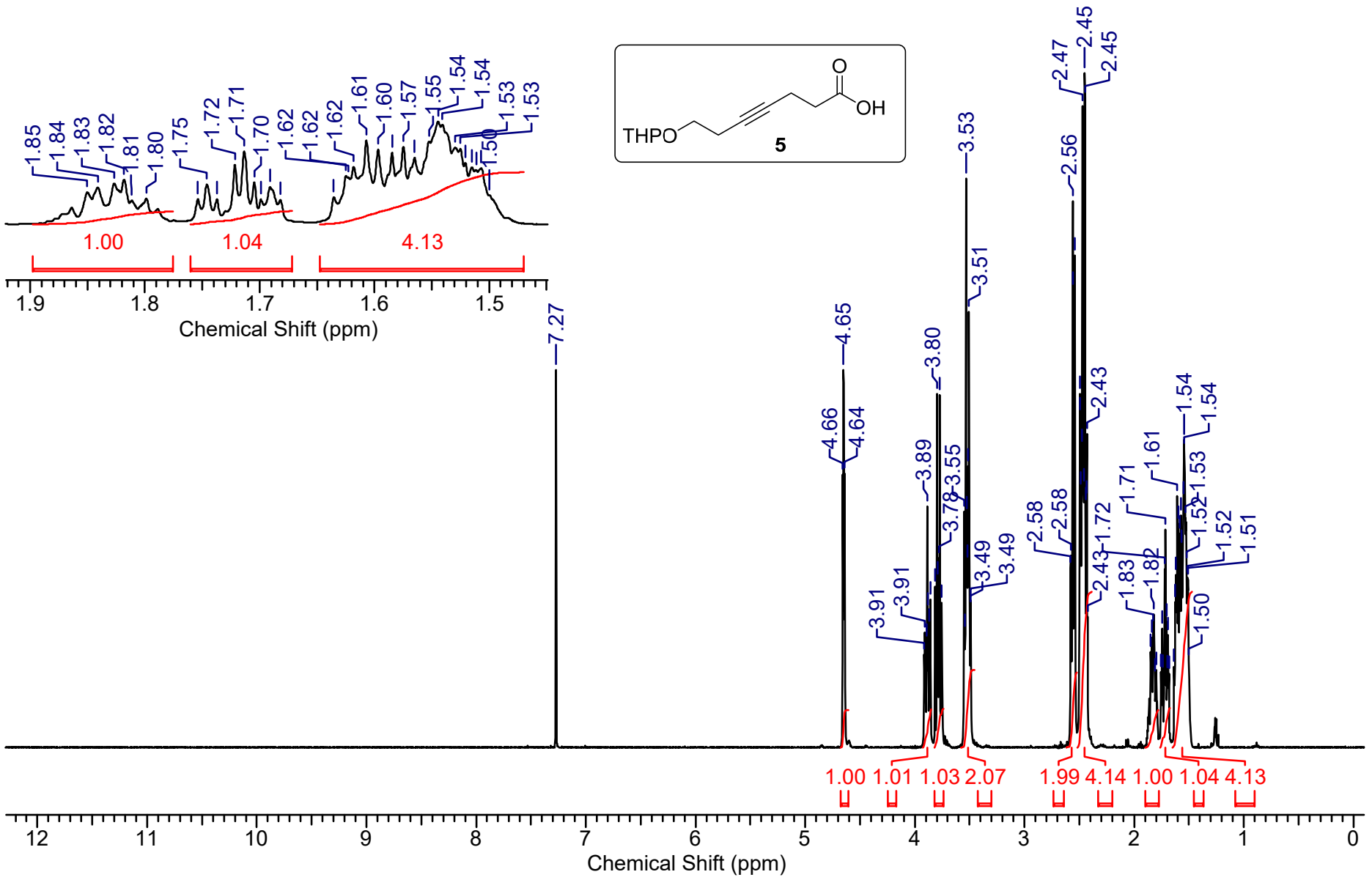
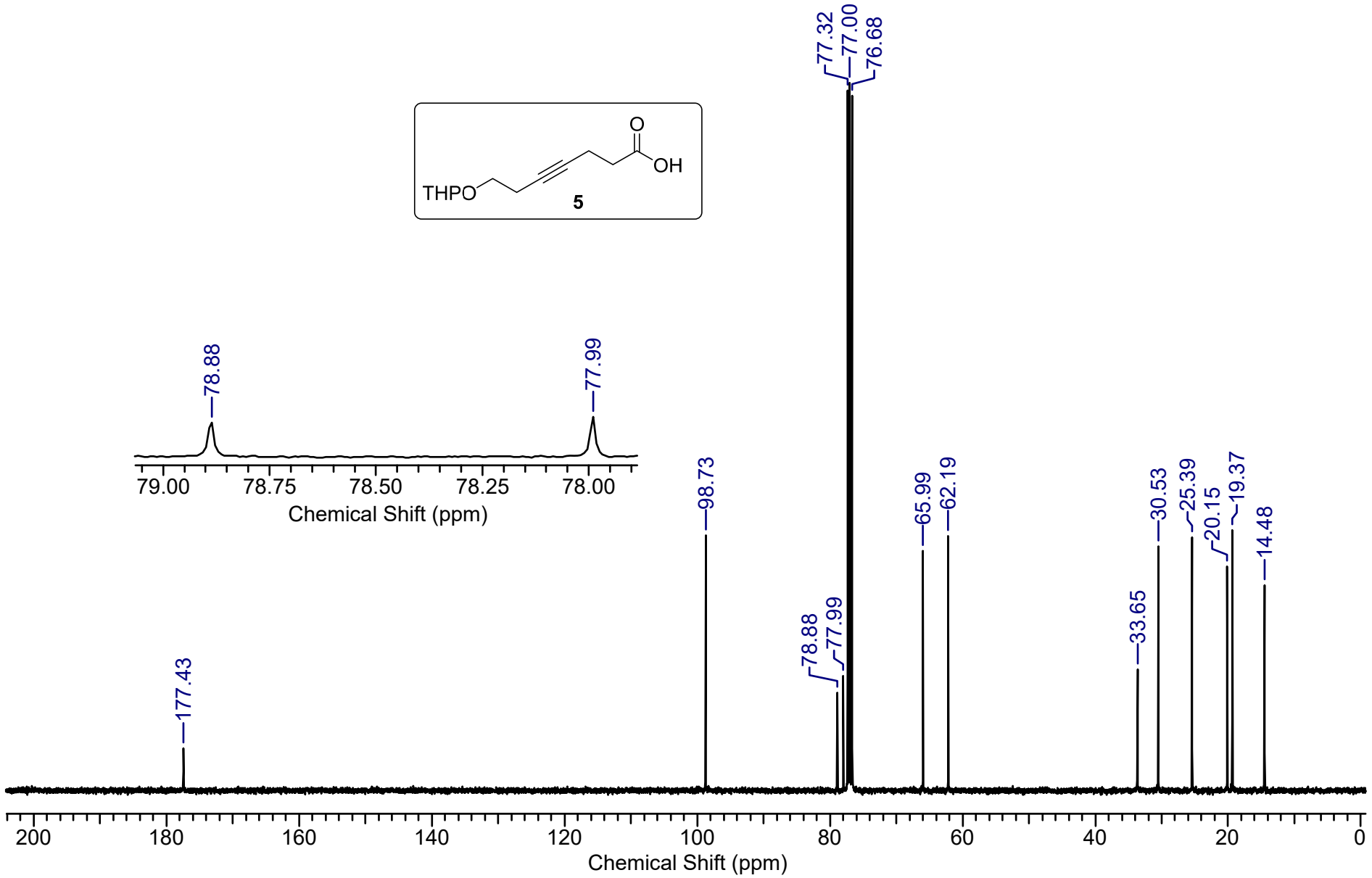
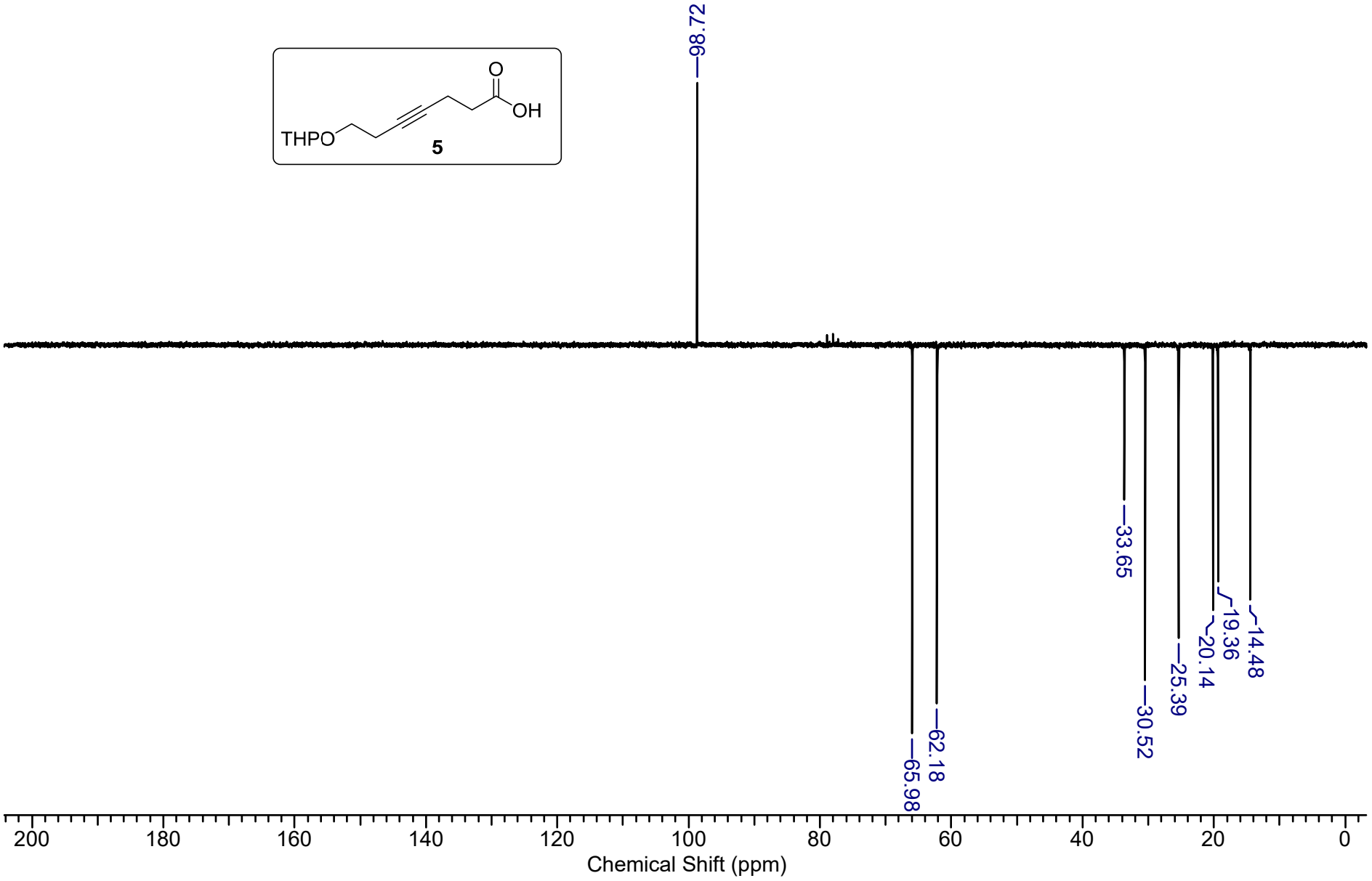
Chemical Shift (ppm)

M-2 #458 RT: 2.62 AV: 1 NL: 3.97E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

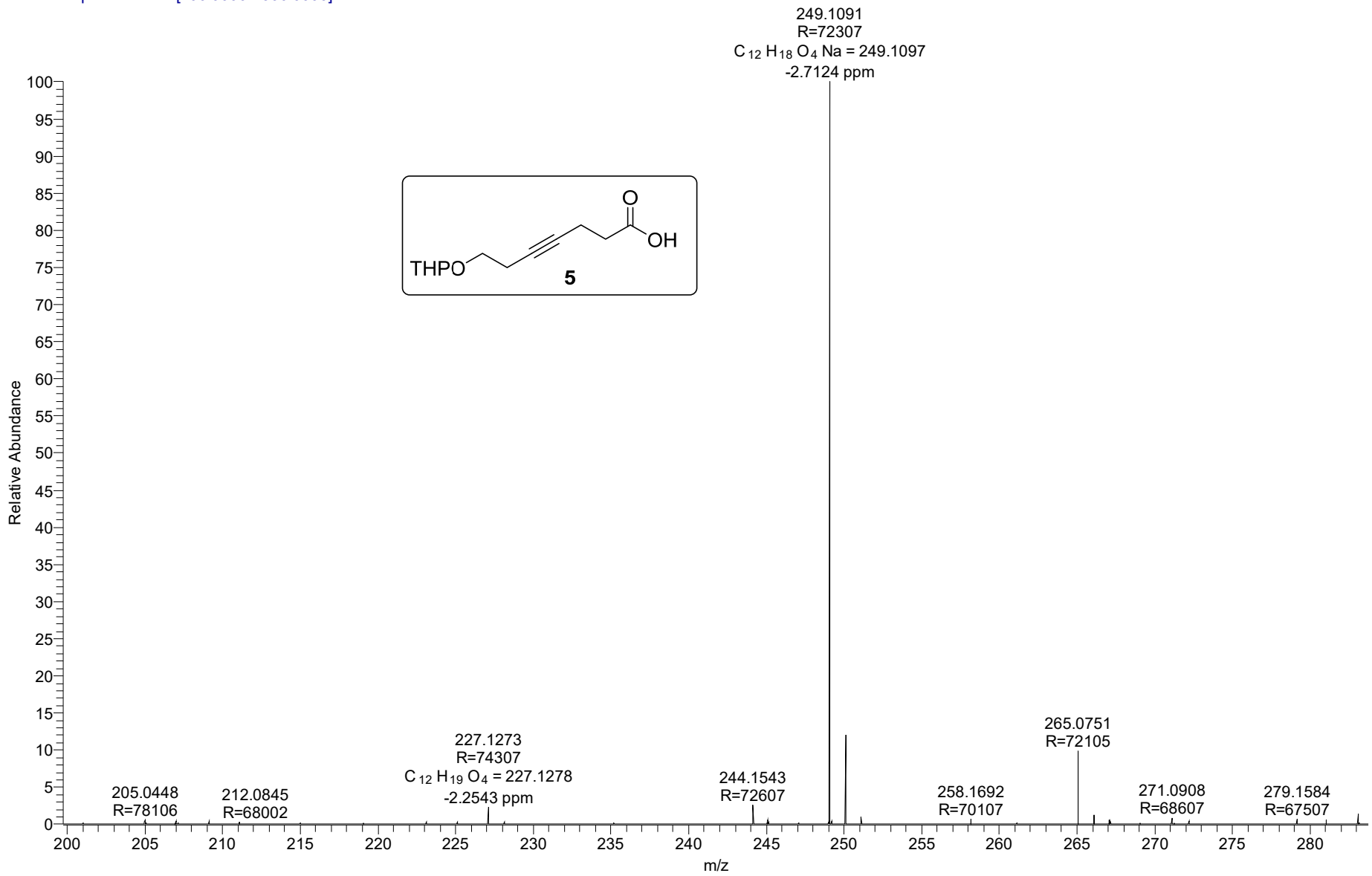


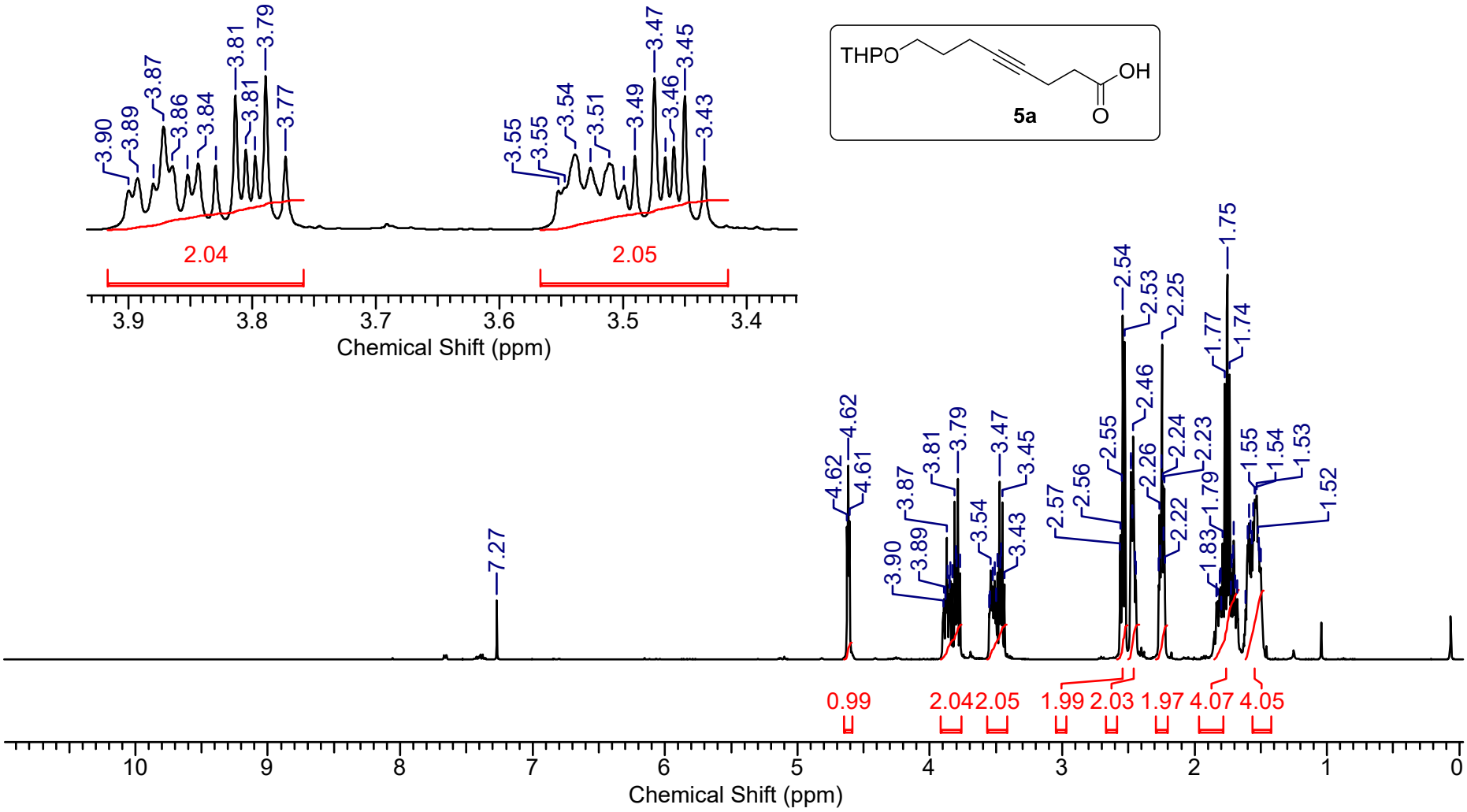

THPO
O
OH
5
Chemical Shift (ppm)
Chemical Shift (ppm)
1.00 1.04 4.13
1.00 1.01 1.03 2.07 1.99 4.14 1.00 1.04 4.13

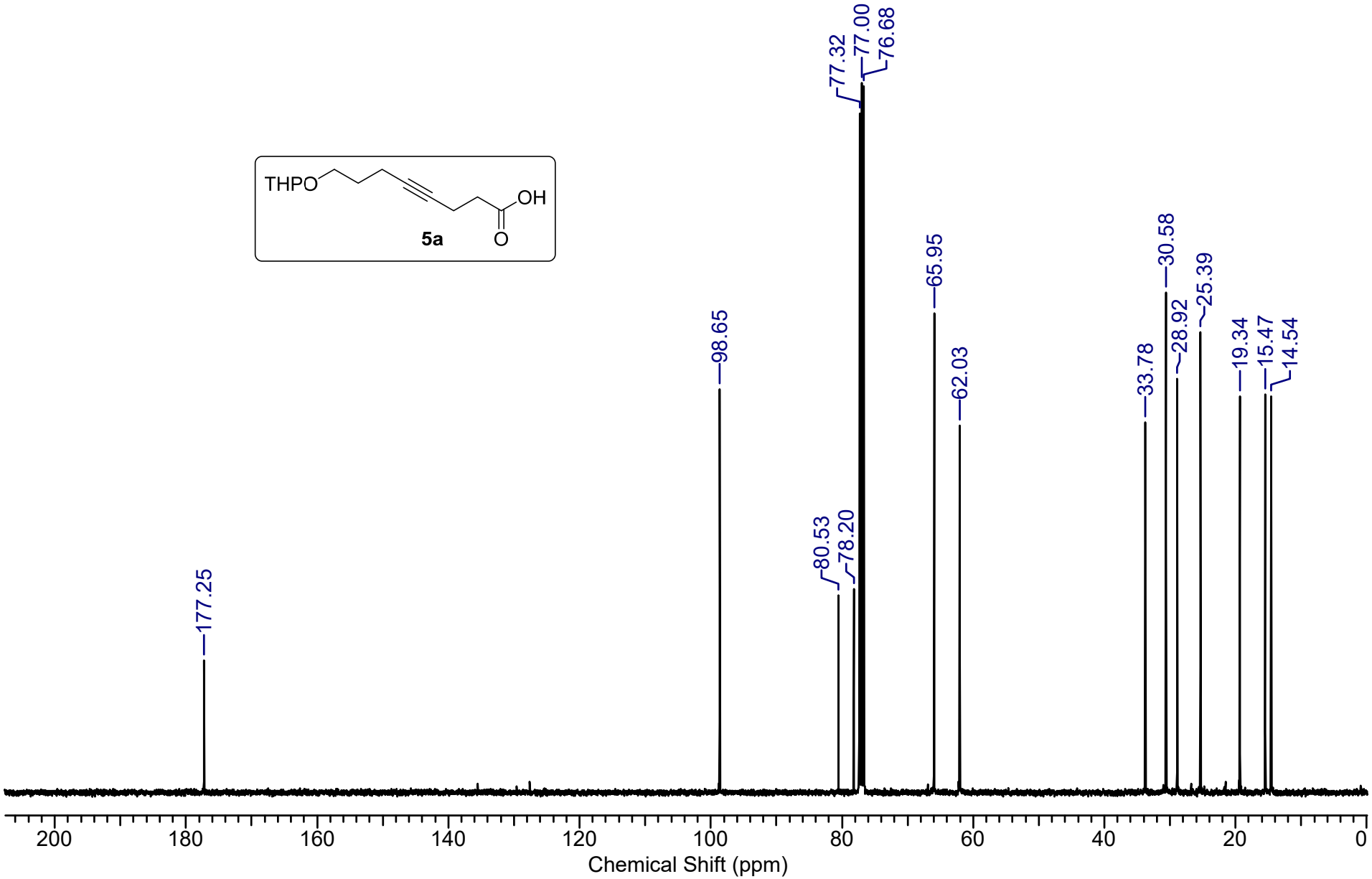

THPO
O
OH
5
77.32
77.00
76.68
78.88
77.99
79.00
78.75
78.50
78.25
78.00
Chemical Shift (ppm)
98.73
65.99
62.19
33.65
30.53
25.39
20.15
19.37
14.48
177.43
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

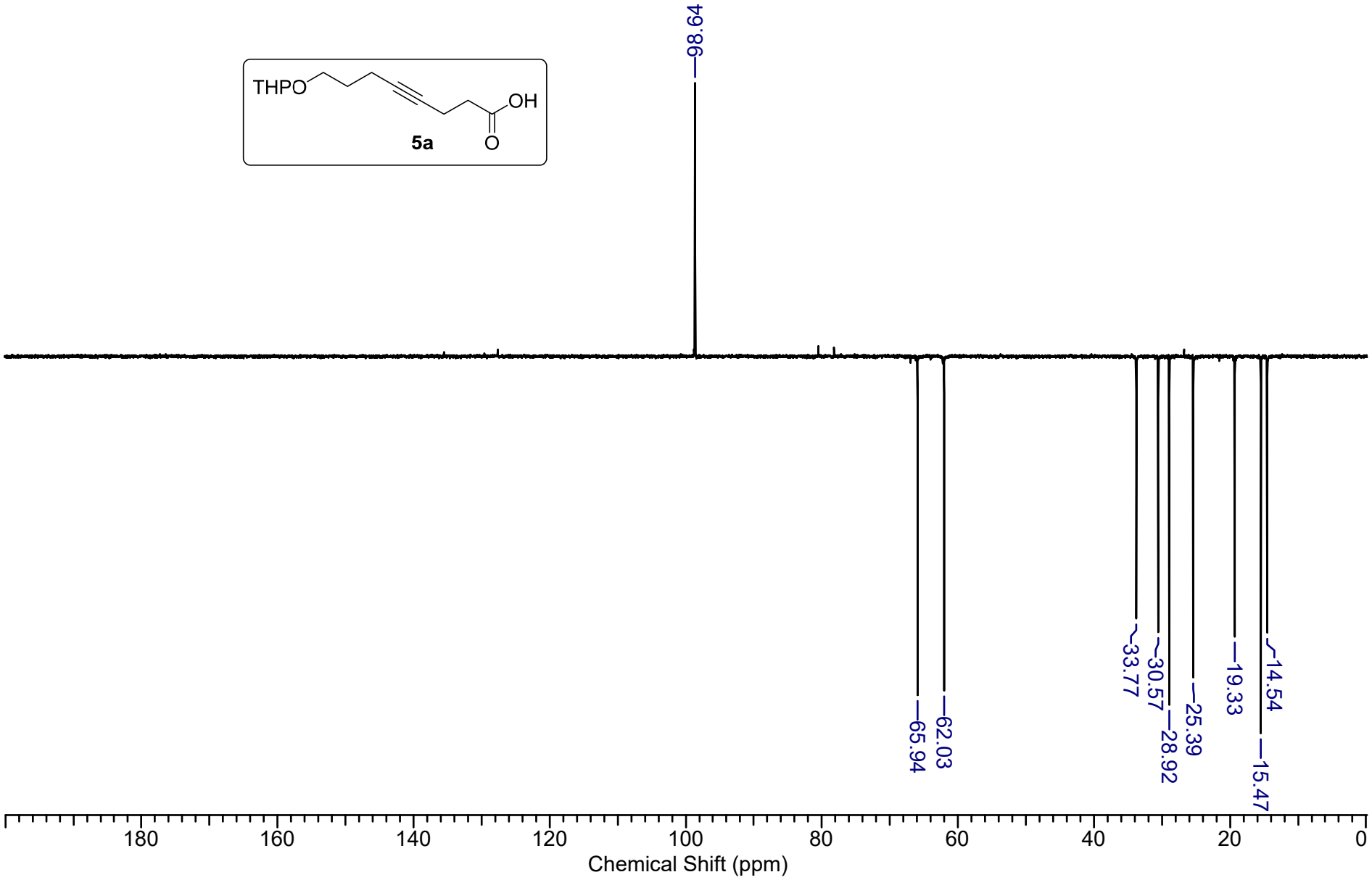

THPO
O
OH
5
98.72
65.98
62.18
33.65
30.52
25.39
20.14
19.36
14.48
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

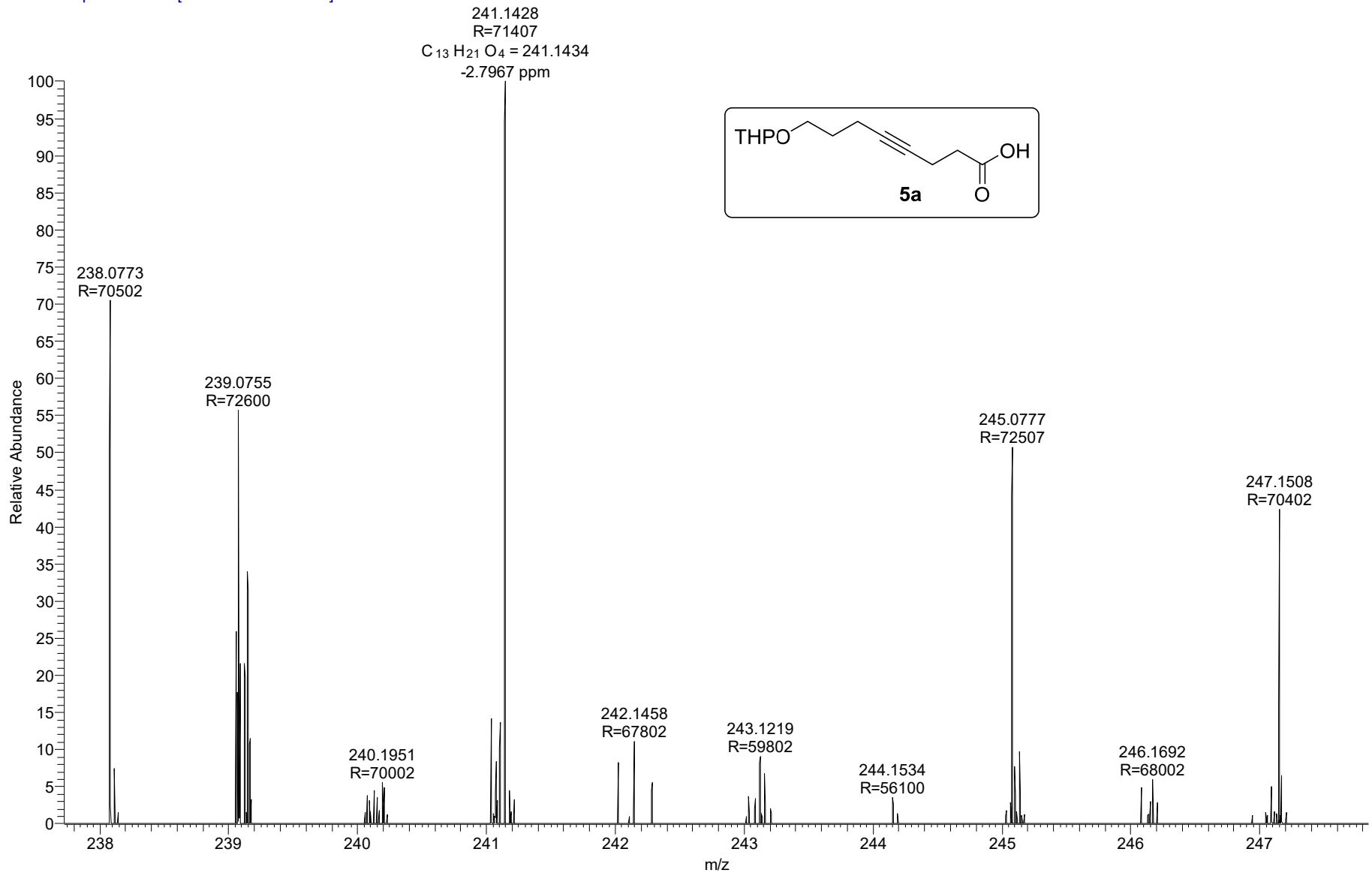
M-4 #447 RT: 2.60 AV: 1 NL: 2.41E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

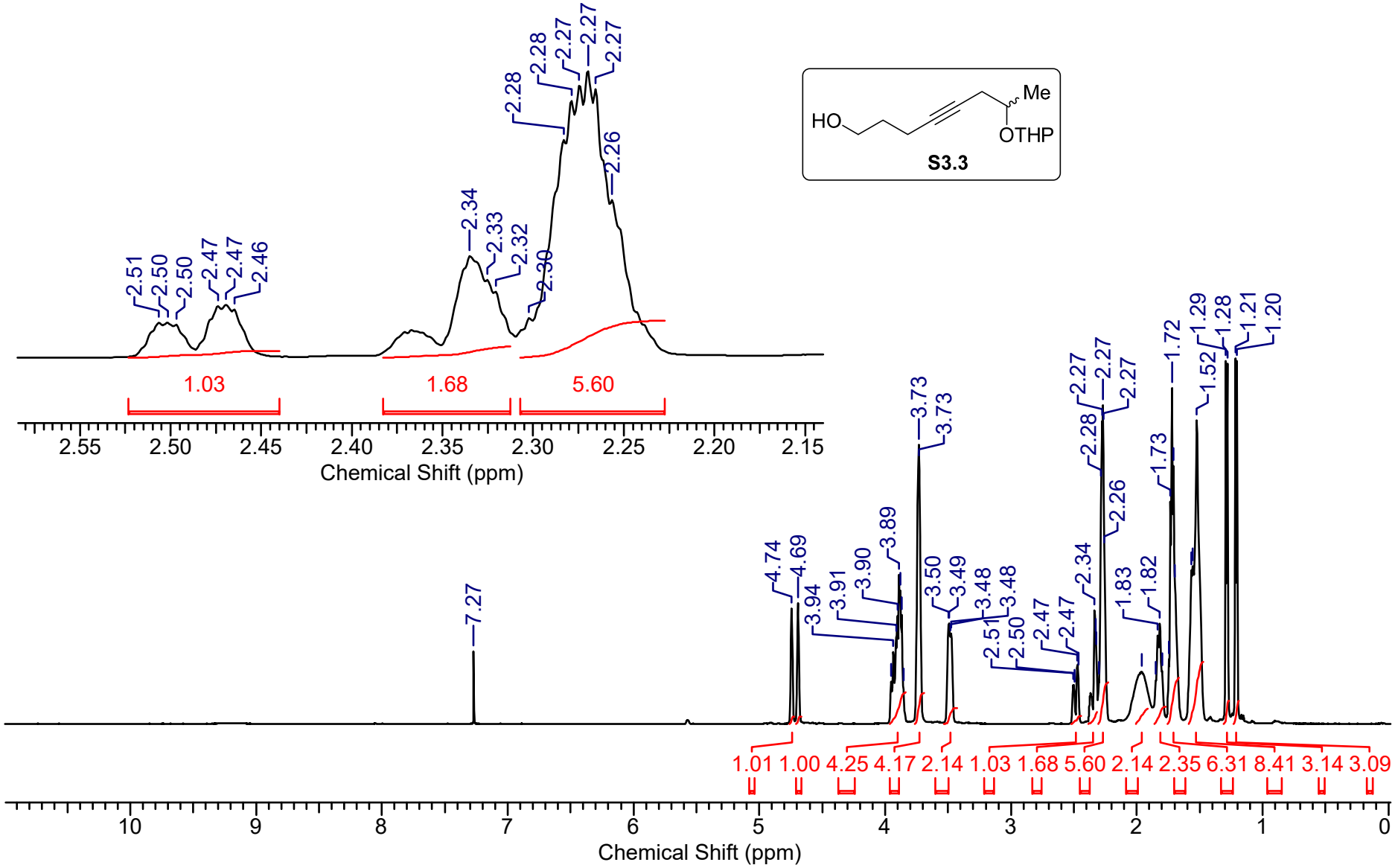


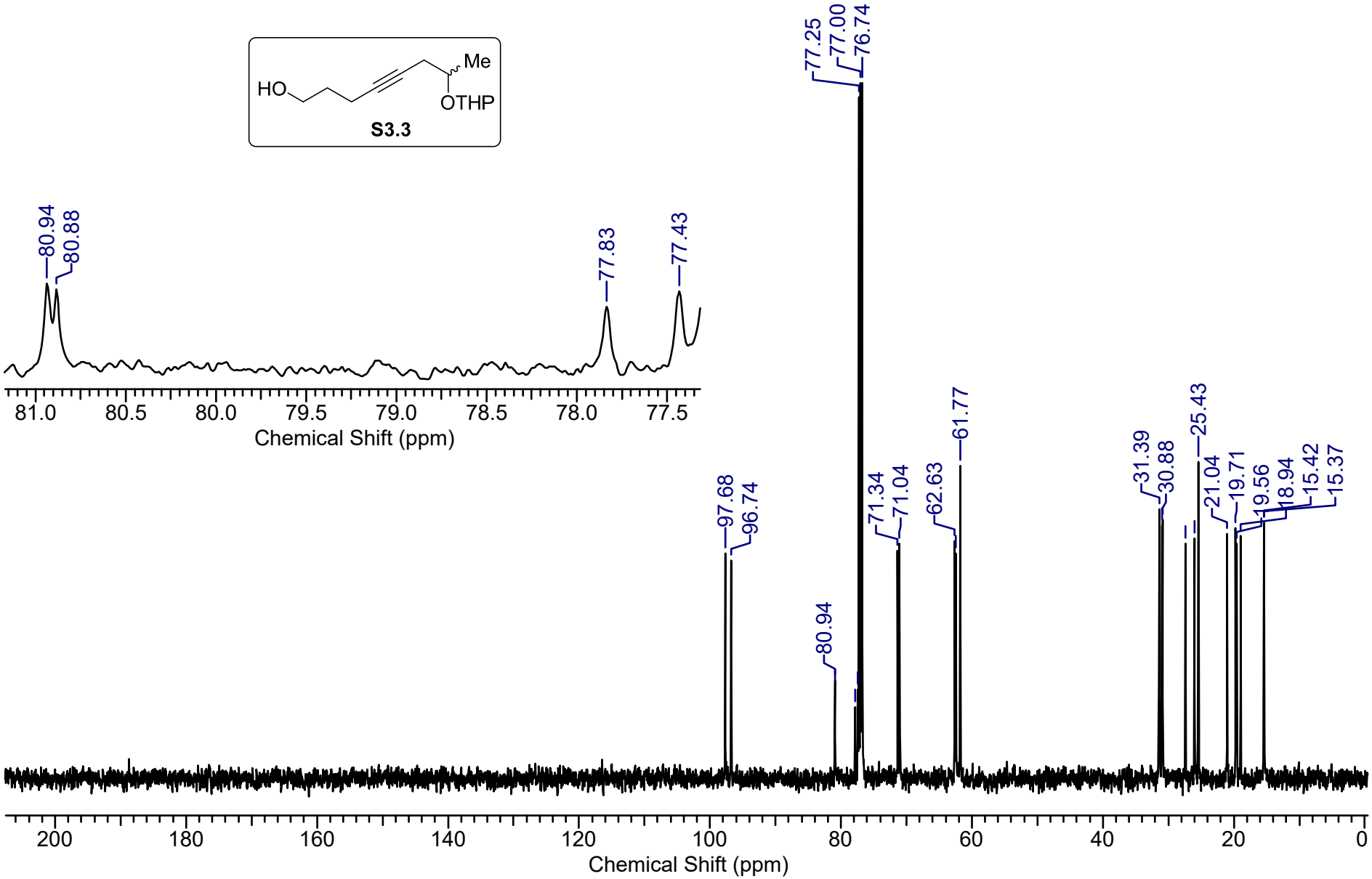

THPO
OH
O
5a
3.90
3.89
3.87
3.86
3.84
3.81
3.81
3.79
3.77
3.55
3.55
3.54
3.51
3.49
3.47
3.46
3.45
3.43
2.04
2.05
3.9
3.8
3.7
3.6
3.5
3.4
Chemical Shift (ppm)
7.27
4.62
4.61
3.90
3.89
3.87
3.81
3.79
3.54
3.47
3.45
3.43
2.57
2.56
2.55
2.54
2.53
2.46
2.26
2.25
2.24
2.23
2.22
1.83
1.79
1.77
1.75
1.74
1.55
1.54
1.53
1.52
0.99
2.04
2.05
1.99
2.03
1.97
4.07
4.05
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

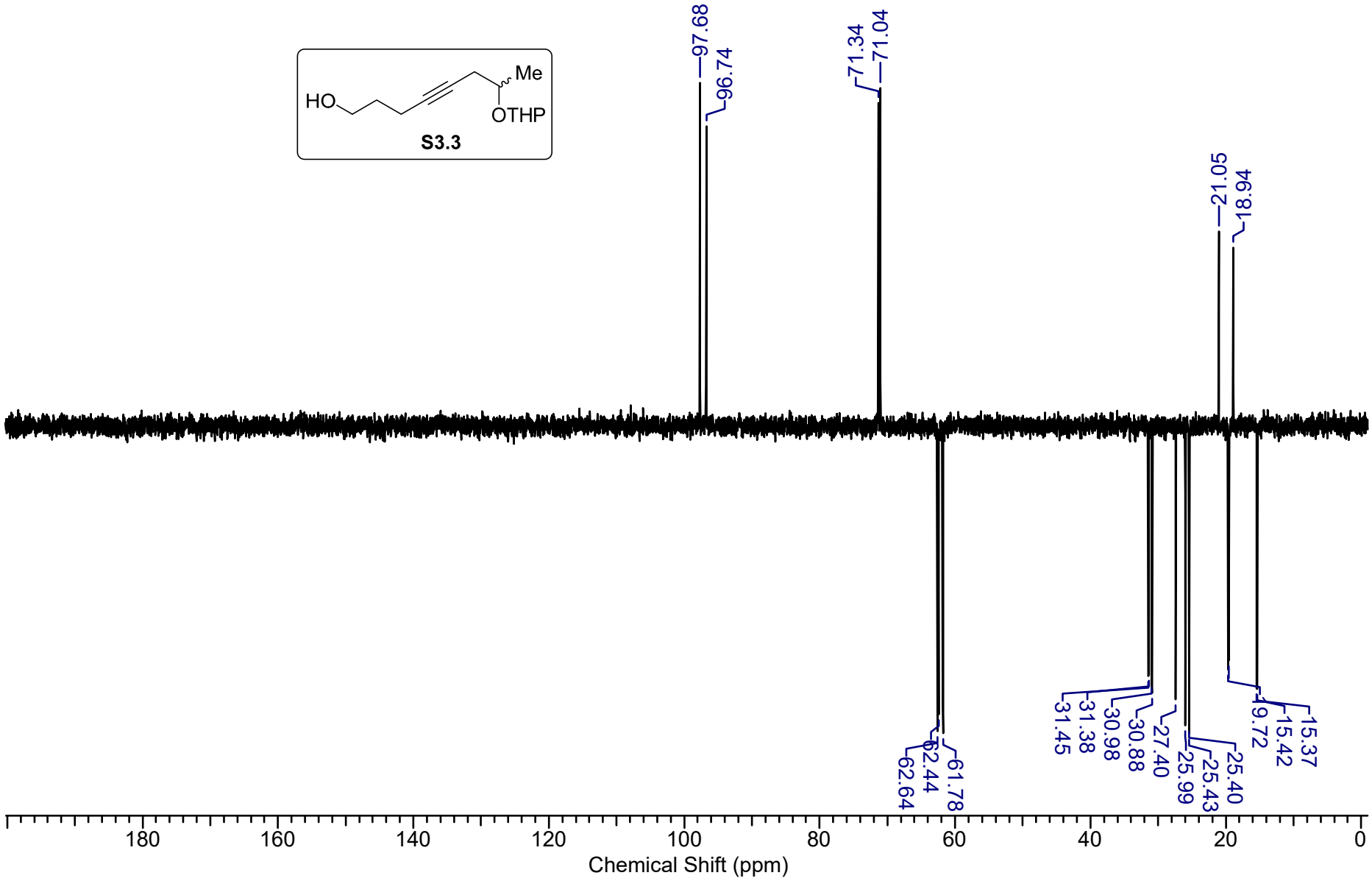

THPO
OH
O
5a
177.25
98.65
80.53
78.20
77.32
77.00
76.68
65.95
62.03
33.78
30.58
28.92
25.39
19.34
15.47
14.54
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

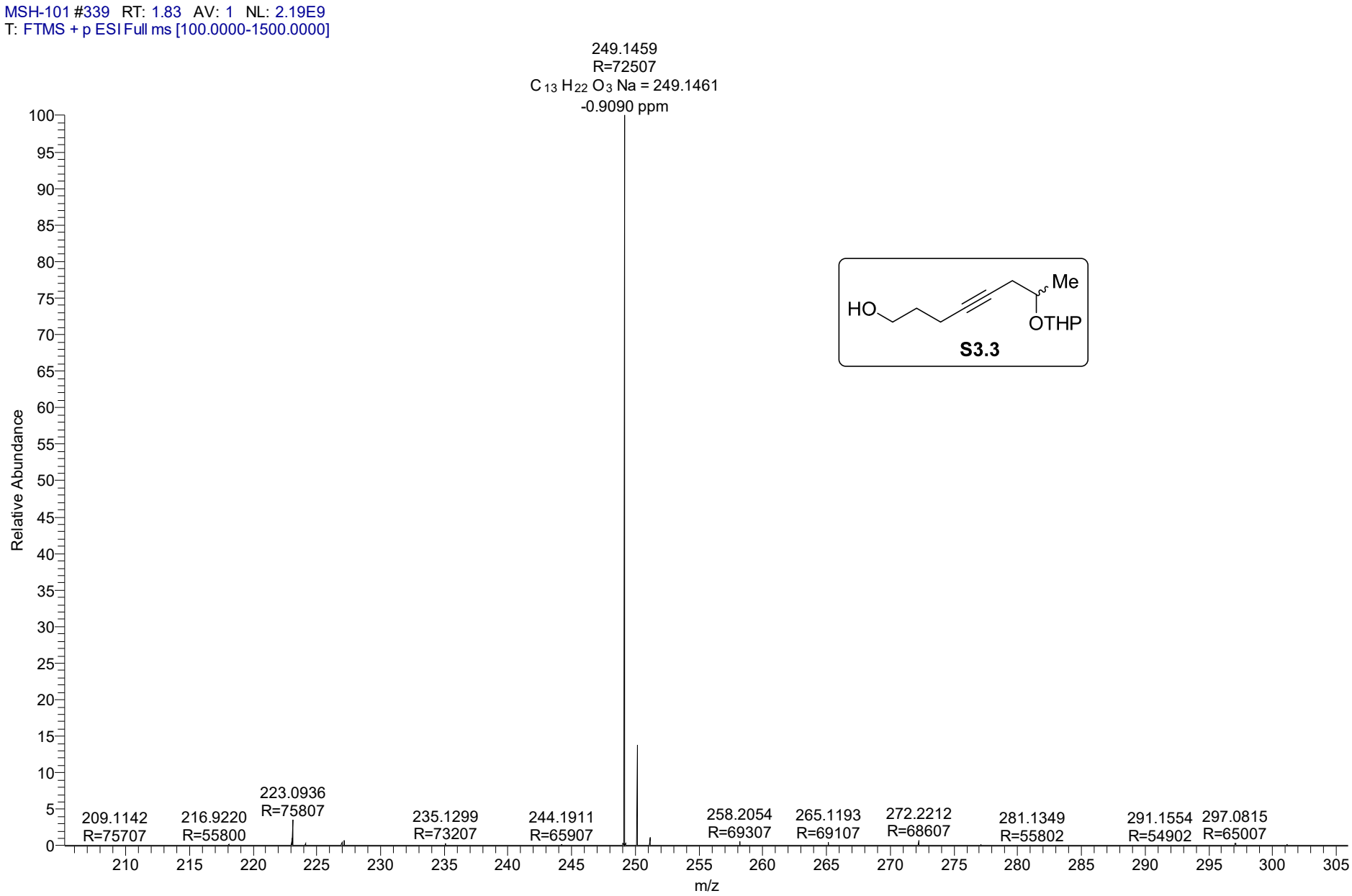

THPO
OH
O
5a
99.86
65.94
62.03
33.77
30.57
28.92
25.39
19.33
14.54
15.47
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

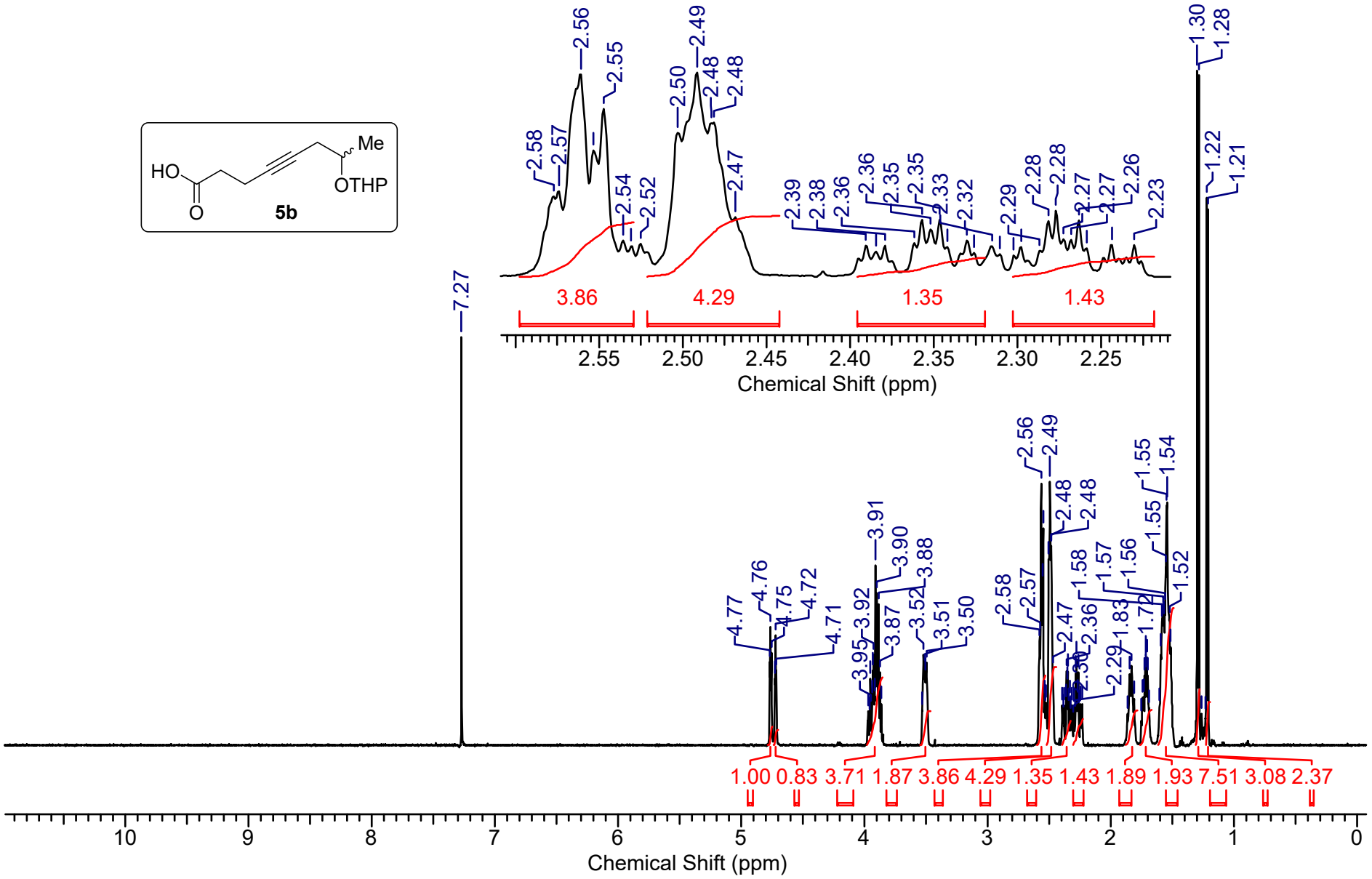

MSH-44 #308 RT: 1.67 AV: 1 NL: 2.05E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]
241.1428
R=71407
C 13 H21 O4 = 241.1434
-2.7967 ppm
THPO
OH
O
5a
238.0773
R=70502
239.0755
R=72600
240.1951
R=70002
242.1458
R=67802
243.1219
R=59802
244.1534
R=56100
245.0777
R=72507
246.1692
R=68002
247.1508
R=70402
Relative Abundance
m/z

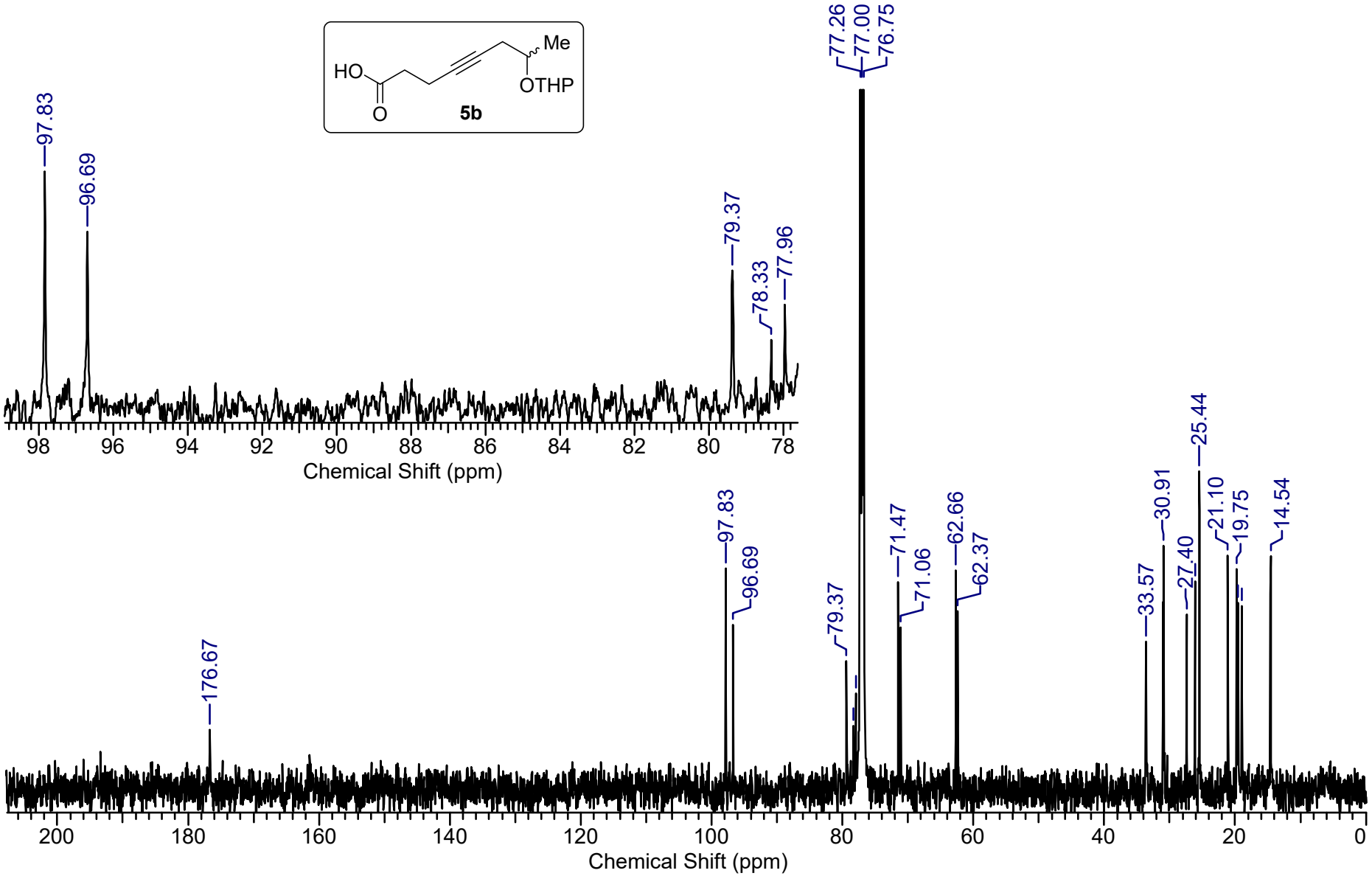

HO
Me
OTHP
S3.3
Chemical Shift (ppm)
Chemical Shift (ppm)

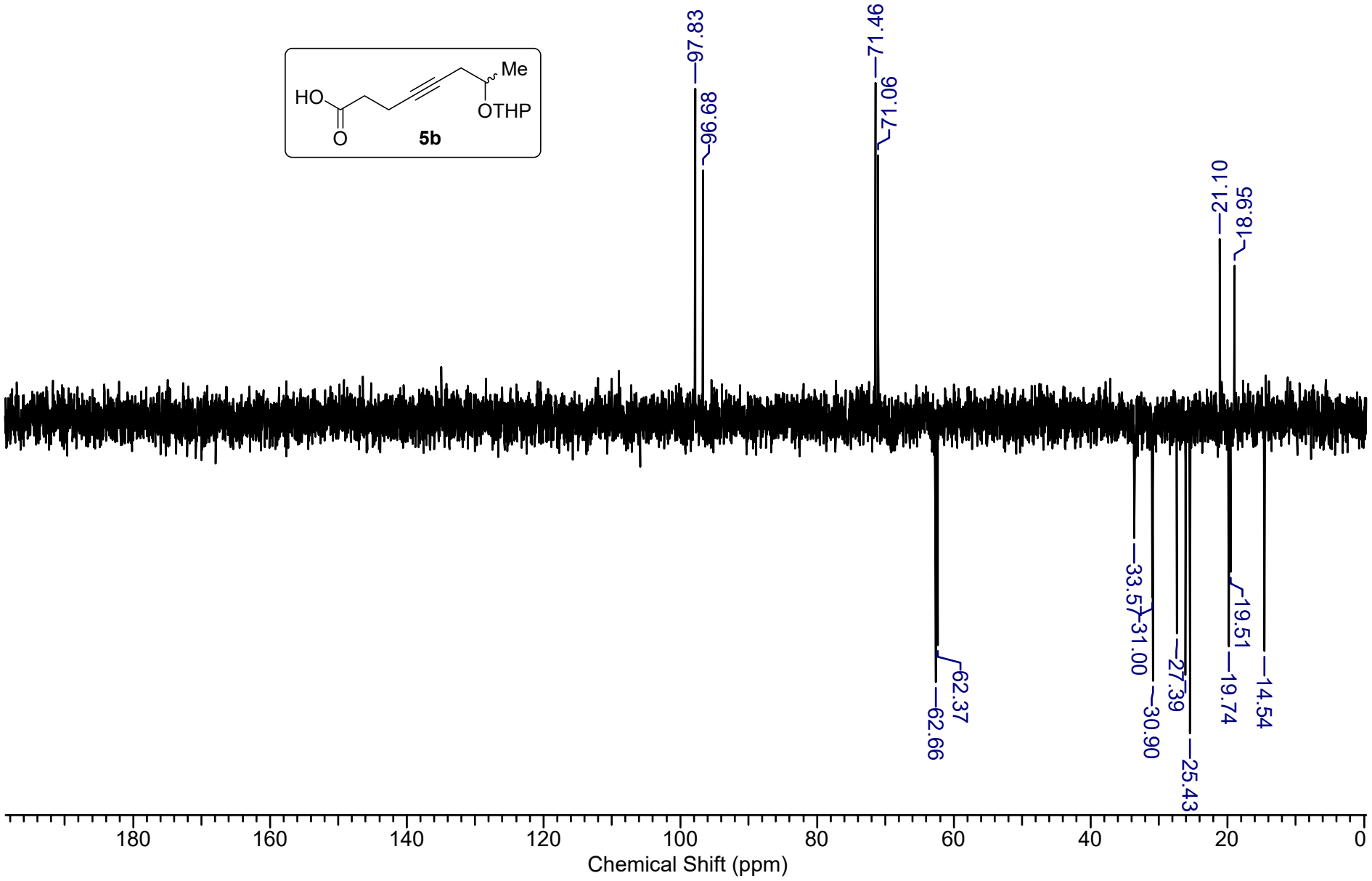

HO
Me
OTHP
S3.3
80.94
80.88
77.83
77.43
81.0
80.5
80.0
79.5
79.0
78.5
78.0
77.5
Chemical Shift (ppm)
77.25
77.00
76.74
97.68
96.74
80.94
71.34
71.04
62.63
61.77
31.39
30.88
25.43
21.04
19.71
19.56
18.94
15.42
15.37
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


HO
Me
OTHP
S3.3
97.68
96.74
71.34
71.04
21.05
18.94
62.64
62.44
61.78
31.45
31.38
30.98
30.88
27.40
25.99
25.43
25.40
19.72
15.42
15.37
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

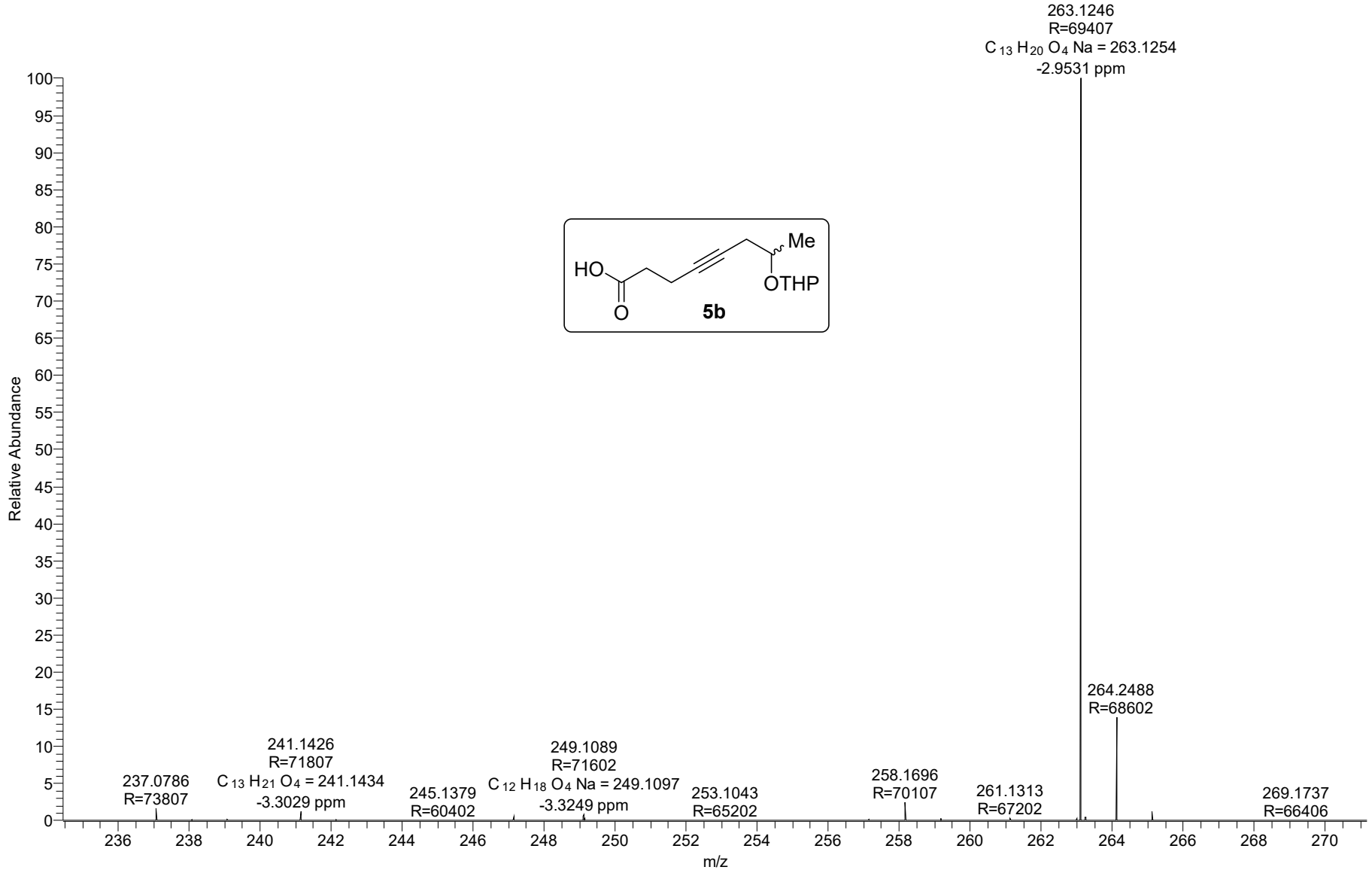

MSH-101 #339 RT: 1.83 AV: 1 NL: 2.19E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]
249.1459
R=72507
C 13 H 22 O 3 Na = 249.1461
-0.9090 ppm
HO
Me
OTHP
S3.3
Relative Abundance
m/z
209.1142
R=75707
216.9220
R=55800
223.0936
R=75807
235.1299
R=73207
244.1911
R=65907
258.2054
R=69307
265.1193
R=69107
272.2212
R=68607
281.1349
R=55802
291.1554
R=54902
297.0815
R=65007

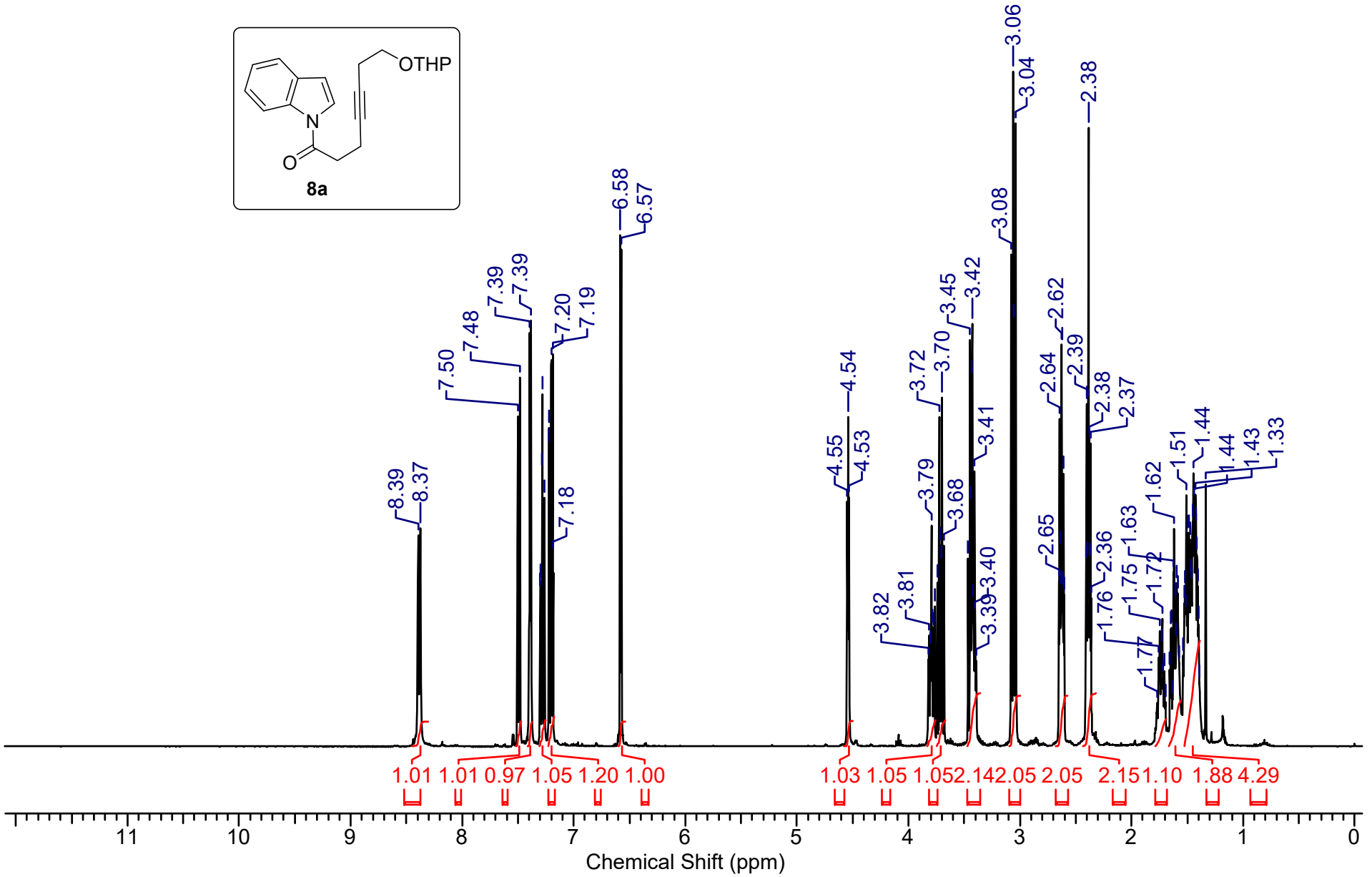

HO
O
OTHP
Me
5b
7.27
2.58 2.57 2.56 2.55 2.54 2.52 2.50 2.49 2.48 2.48 2.47 2.39 2.38 2.36 2.36 2.35 2.35 2.33 2.32 2.29 2.28 2.28 2.27 2.27 2.26 2.23
1.30 1.28 1.22 1.21
3.86 4.29 1.35 1.43
2.55 2.50 2.45 2.40 2.35 2.30 2.25
Chemical Shift (ppm)
4.77 4.76 4.75 4.72 4.71 3.95 3.92 3.91 3.90 3.88 3.87 3.52 3.51 3.50
2.58 2.57 2.56 2.49 2.48 2.48 2.47 2.36 2.30 2.29
1.83 1.72 1.58 1.57 1.56 1.55 1.55 1.54 1.52
1.00 0.83 3.71 1.87 3.86 4.29 1.35 1.43 1.89 1.93 7.51 3.08 2.37
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

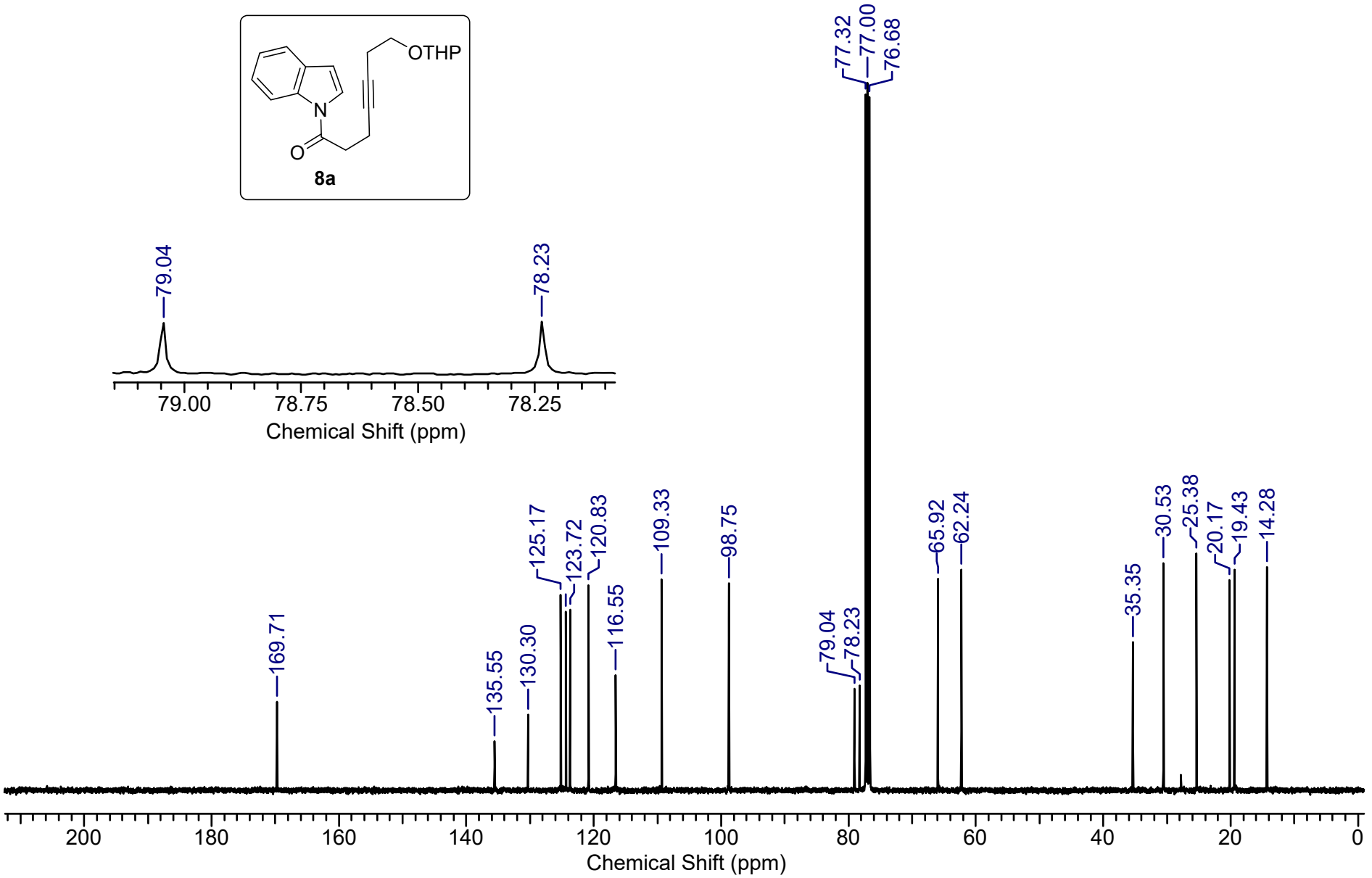

HO
O
Me
OTHP
5b
97.83
96.69
79.37
78.33
77.96
98
96
94
92
90
88
86
84
82
80
78
Chemical Shift (ppm)
77.26
77.00
76.75
97.83
96.69
79.37
71.47
71.06
62.66
62.37
33.57
30.91
27.40
25.44
21.10
19.75
14.54
176.67
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

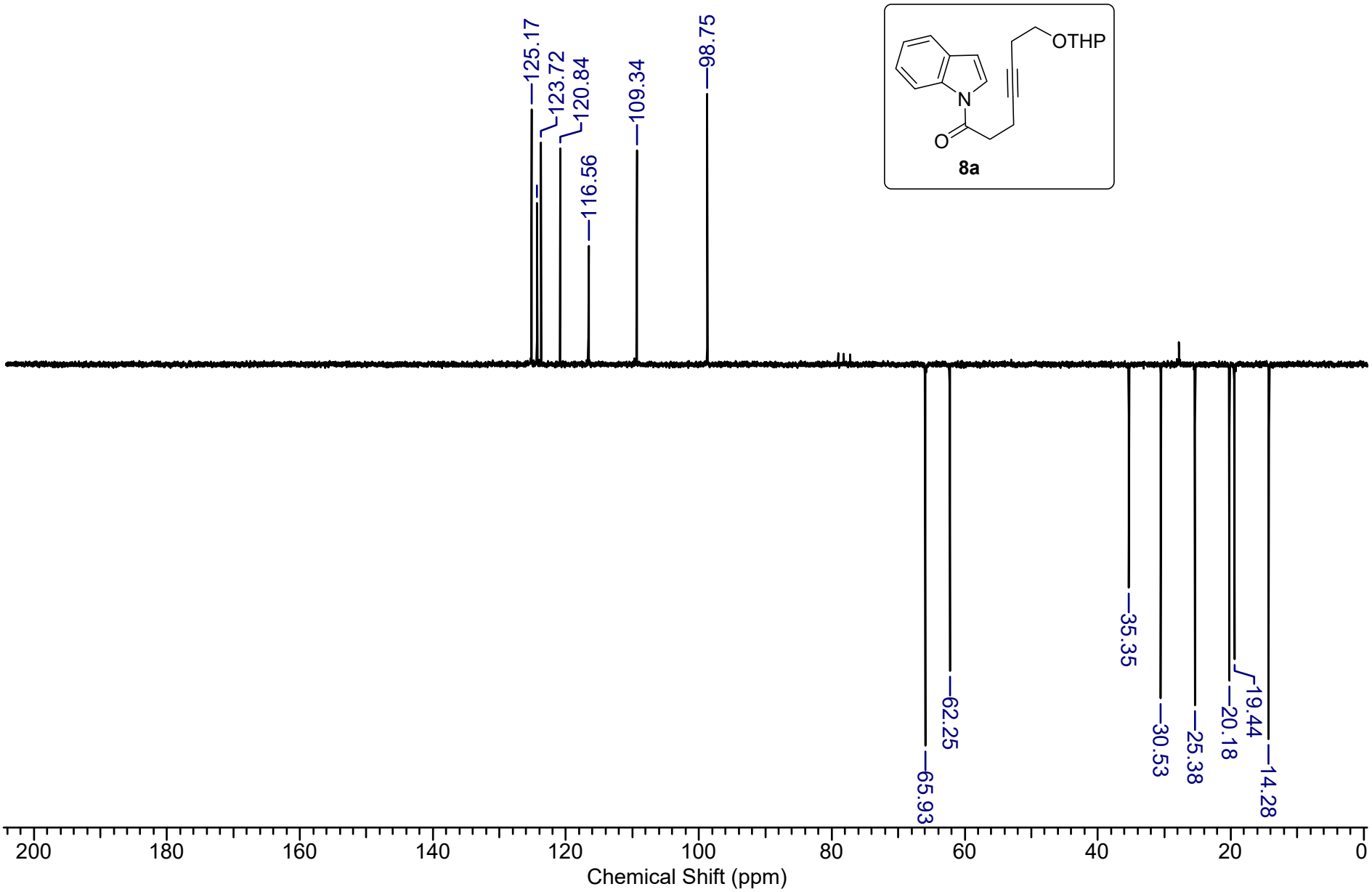

HO
O
Me
OTHP
5b
97.83
96.89
71.49
71.06
21.10
18.95
62.37
62.66
33.57
31.00
30.90
27.39
25.43
19.51
19.74
14.54
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MSH-45 #269 RT: 1.47 AV: 1 NL: 7.55E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

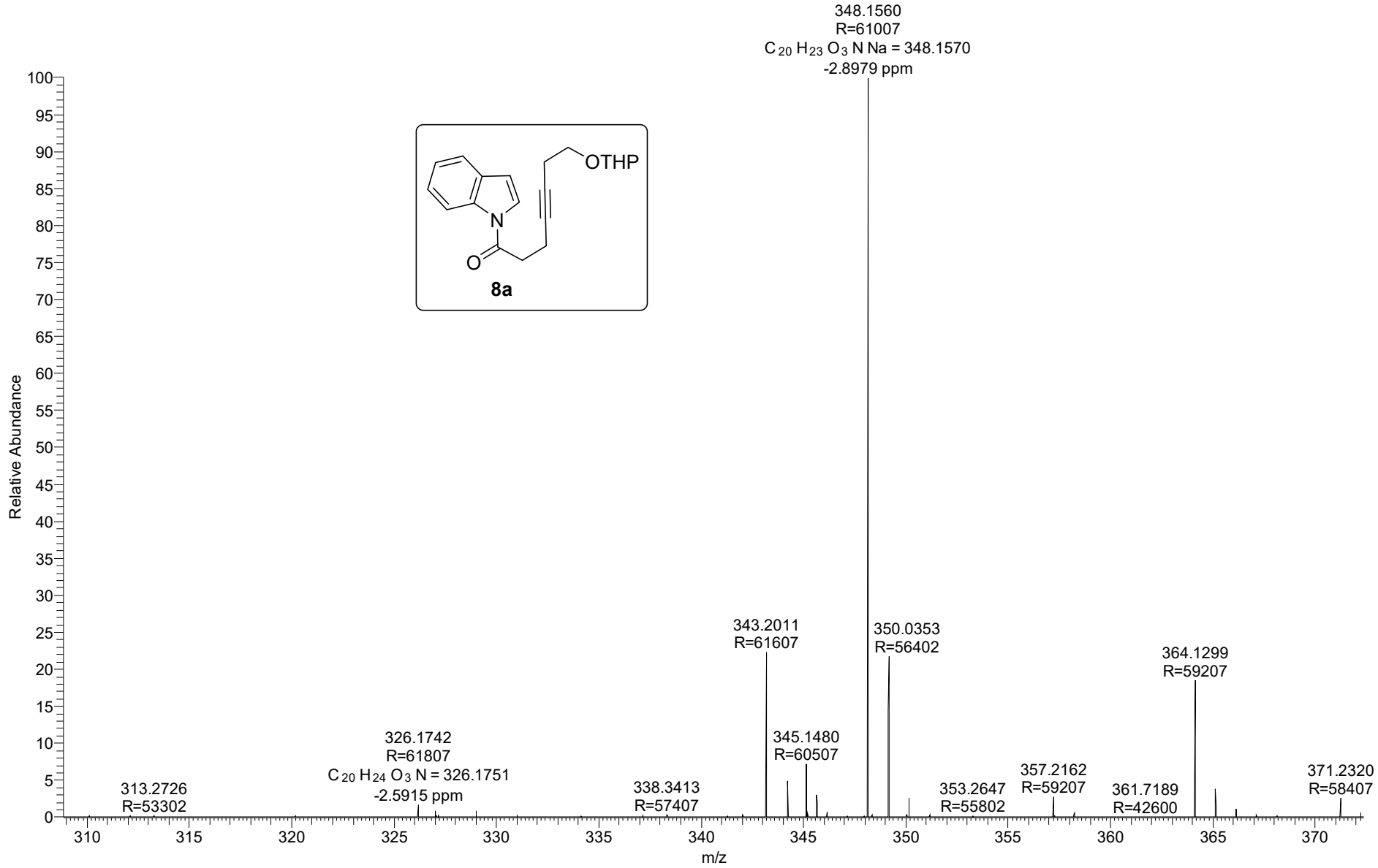


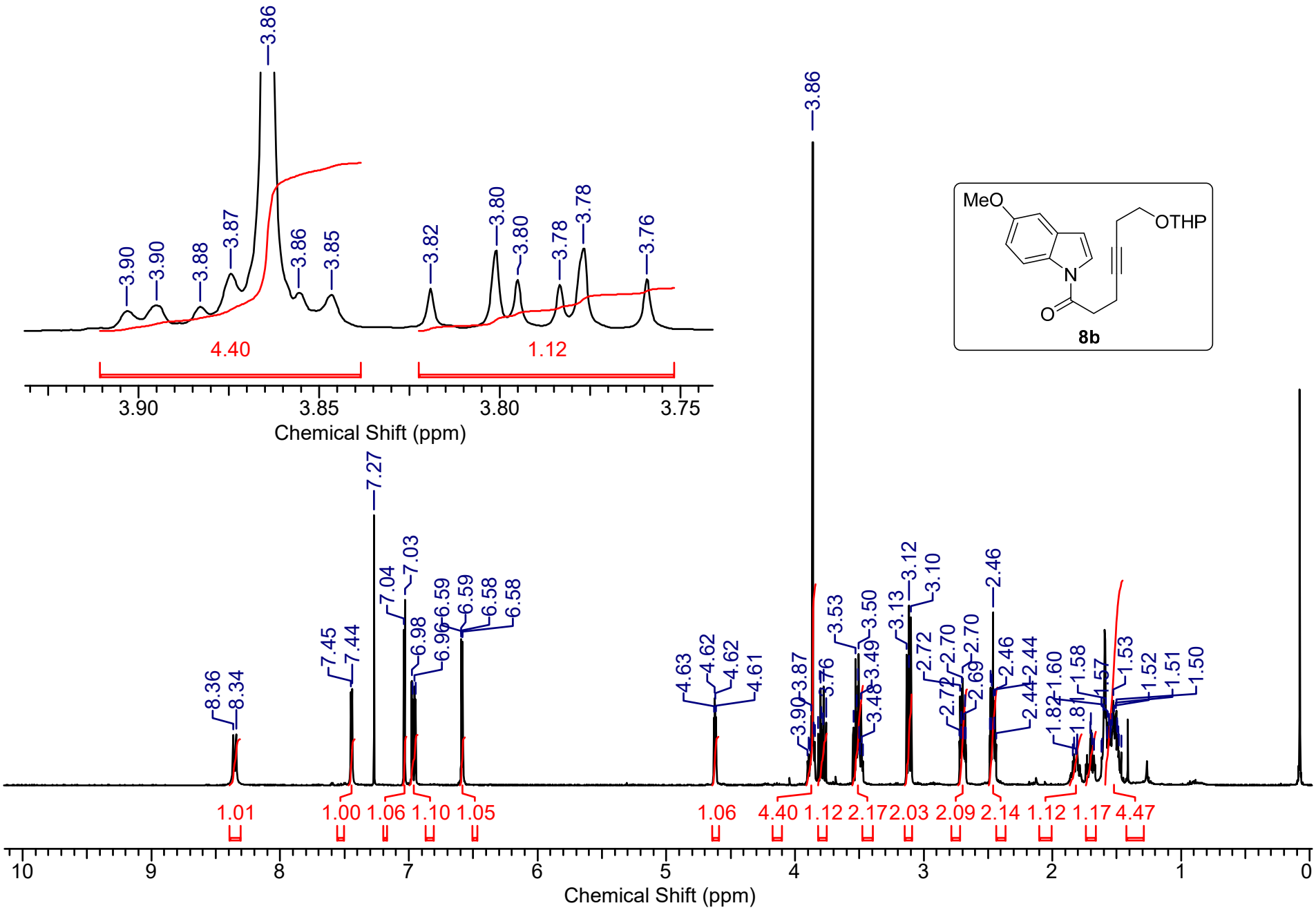

OTHP
N
O
8a
8.39
8.37
7.50
7.48
7.39
7.39
7.20
7.19
7.18
6.58
6.57
4.55
4.54
4.53
3.82
3.81
3.79
3.72
3.70
3.68
3.45
3.42
3.41
3.40
3.39
3.08
3.06
3.04
2.65
2.64
2.62
2.39
2.38
2.38
2.37
2.36
1.76
1.75
1.72
1.77
1.63
1.62
1.51
1.44
1.44
1.43
1.33
1.01 1.01 0.97 1.05 1.20 1.00
1.03 1.05 1.05 2.14 2.05 2.05 2.15 1.10 1.88 4.29
11 10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

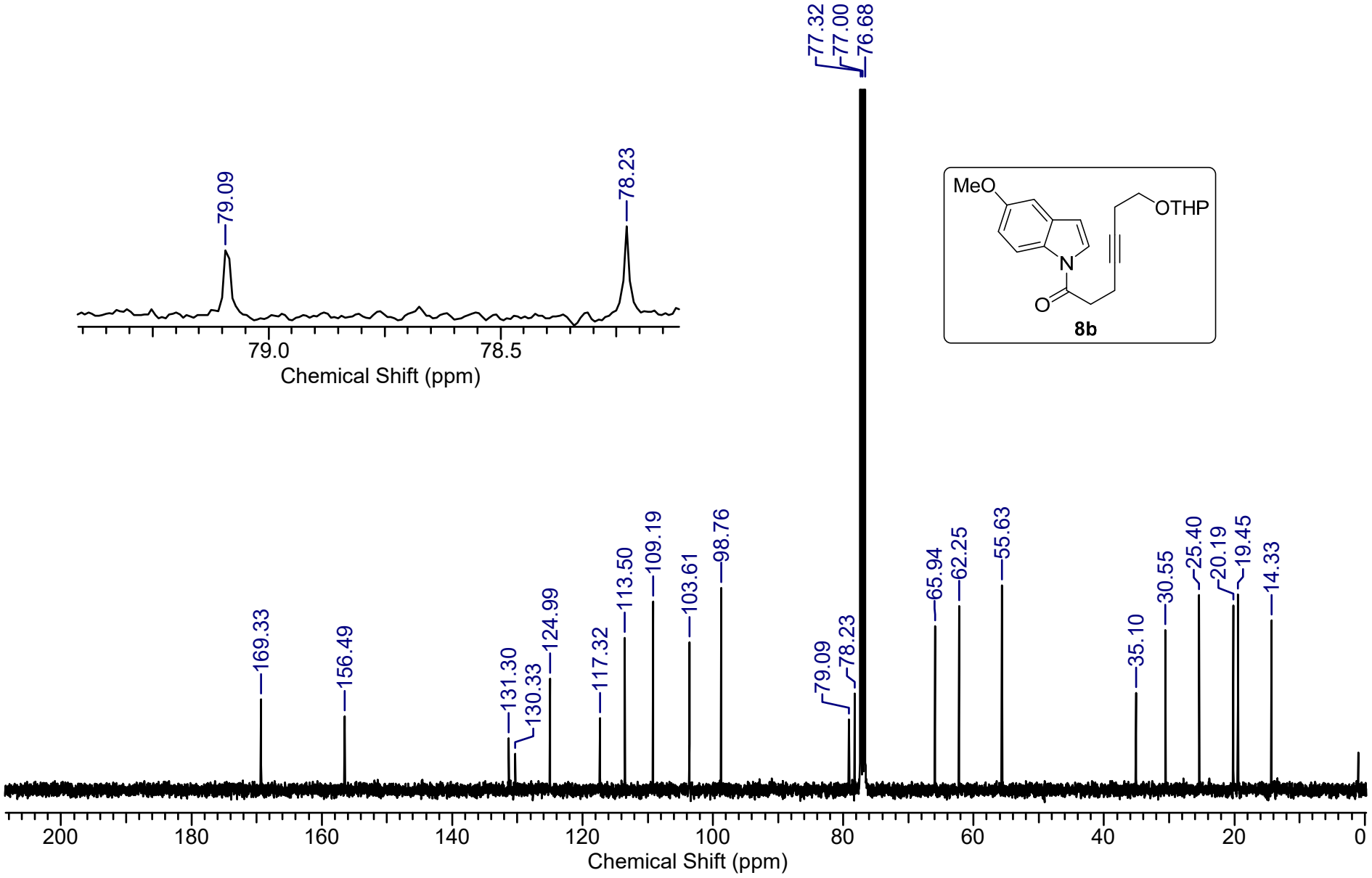

OTHP
N
O
8a
79.04
78.23
79.00
78.75
78.50
78.25
Chemical Shift (ppm)
77.32
77.00
76.68
169.71
135.55
130.30
125.17
123.72
120.83
116.55
109.33
98.75
79.04
78.23
65.92
62.24
35.35
30.53
25.38
20.17
19.43
14.28
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

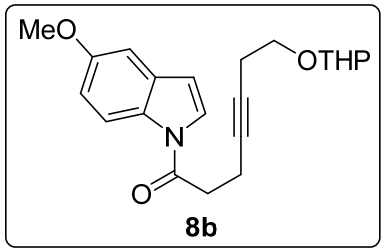

125.17
123.72
120.84
116.56
109.34
98.75
OTHP
N
O
8a
65.93
62.25
35.35
30.53
25.38
20.18
19.44
14.28
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

M-5 #498 RT: 2.92 AV: 1 NL: 9.09E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]




3.86
3.90
3.90
3.88
3.87
3.86
3.85
3.82
3.80
3.80
3.78
3.78
3.76
4.40
1.12
3.90
3.85
3.80
3.75
Chemical Shift (ppm)
MeO
OTHP
N
O
8b
7.27
7.45
7.44
7.04
7.03
6.98
6.96
6.59
6.59
6.58
6.58
8.36
8.34
4.63
4.62
4.62
4.61
3.90
3.87
3.76
3.53
3.50
3.49
3.48
3.13
3.12
3.10
2.72
2.72
2.70
2.70
2.69
2.46
2.46
2.44
2.44
1.82
1.81
1.60
1.58
1.57
1.53
1.52
1.51
1.50
1.01
1.00
1.06
1.10
1.05
1.06
4.40
1.12
2.17
2.03
2.09
2.14
1.12
1.17
4.47
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

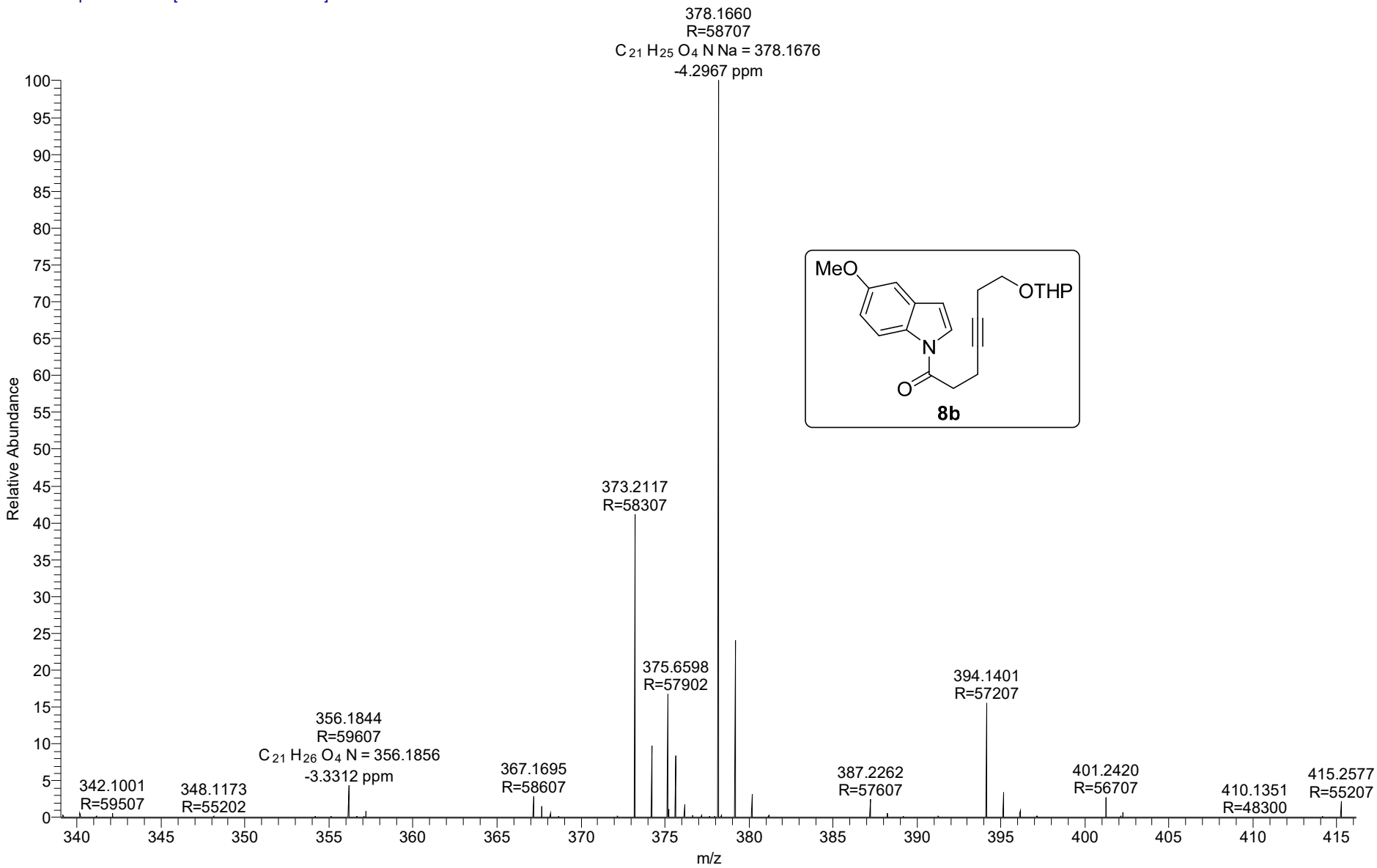

77.32
77.00
76.68
79.09
78.23
MeO
OTHP
N
O
8b
79.0
78.5
Chemical Shift (ppm)
169.33
156.49
131.30
130.33
124.99
117.32
113.50
109.19
103.61
98.76
79.09
78.23
65.94
62.25
55.63
35.10
30.55
25.40
20.19
19.45
14.33
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

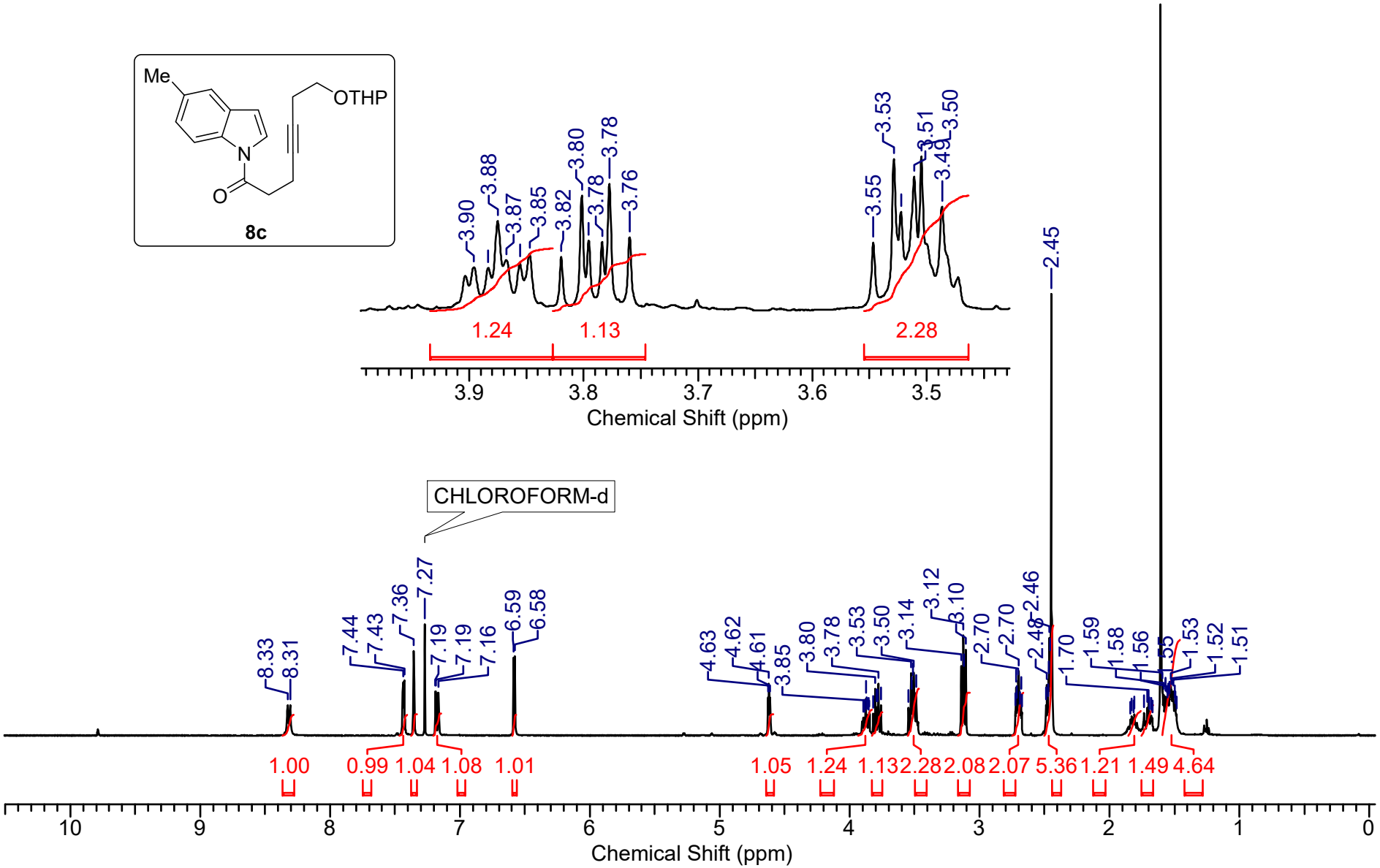

MeO
OTHP
N
O

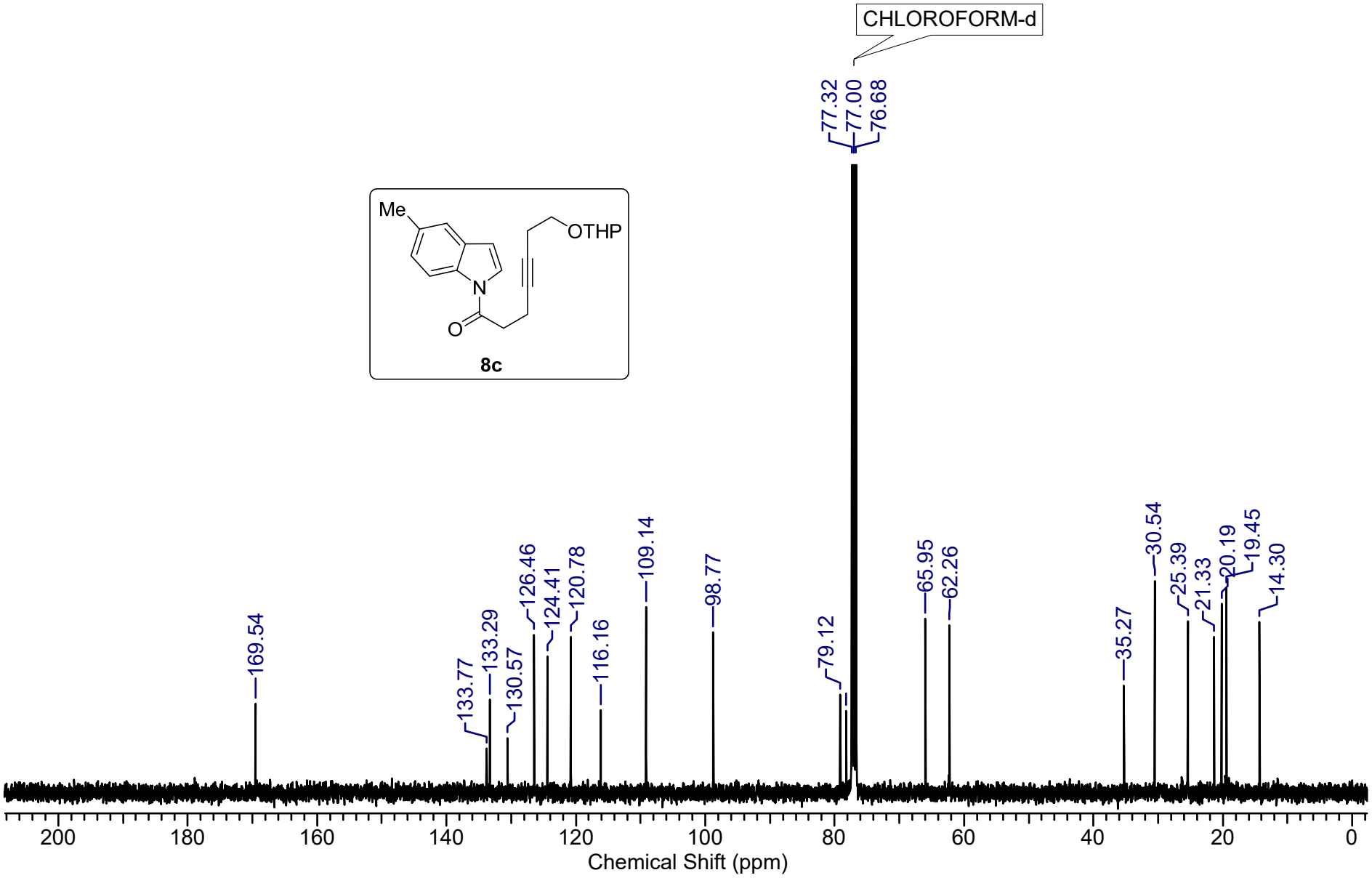
8b

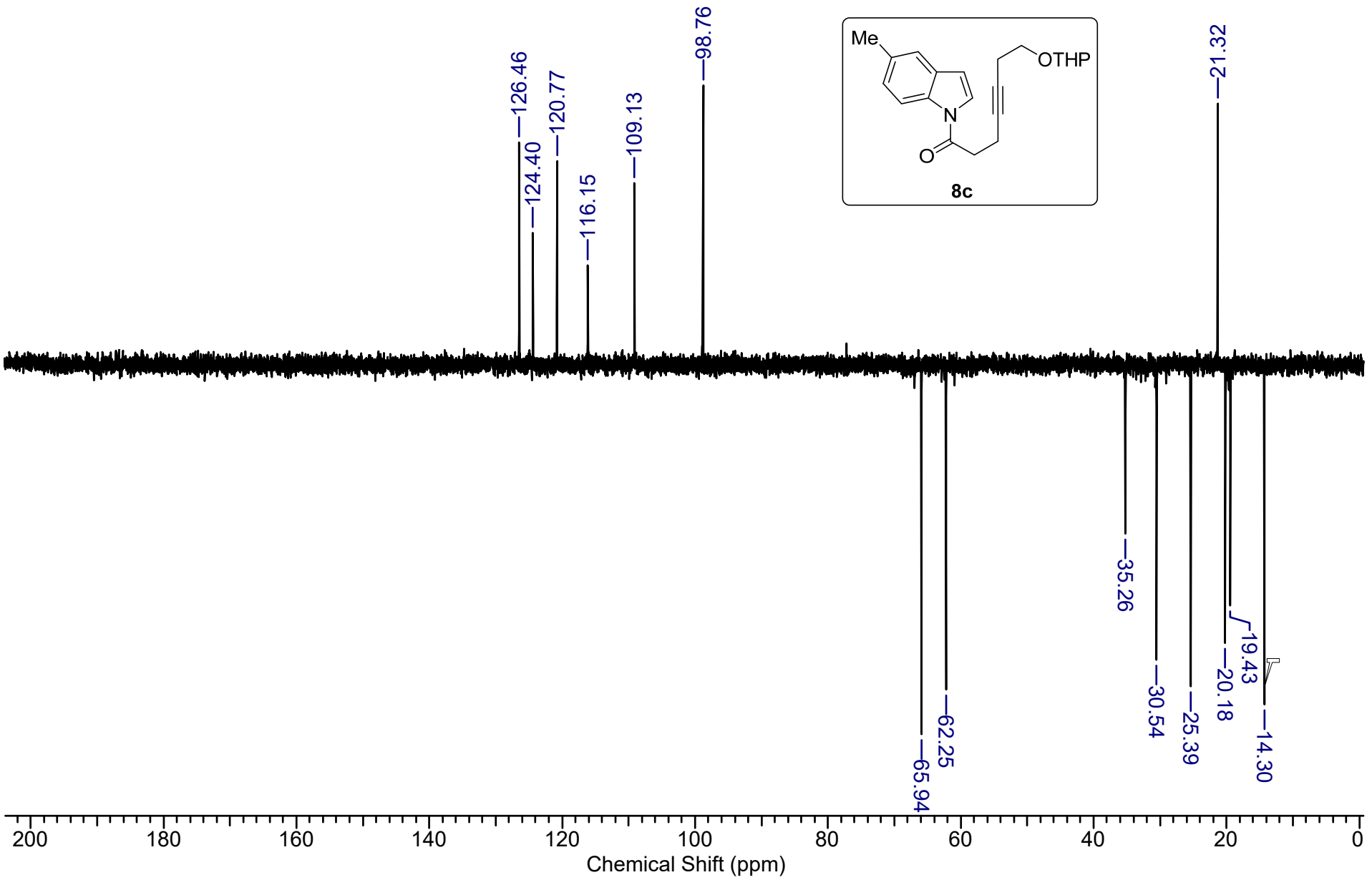
124.98
117.31
113.48
109.18
103.60
98.76
55.92
65.93
62.25
35.09
30.53
25.39
20.18
19.44
14.33
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

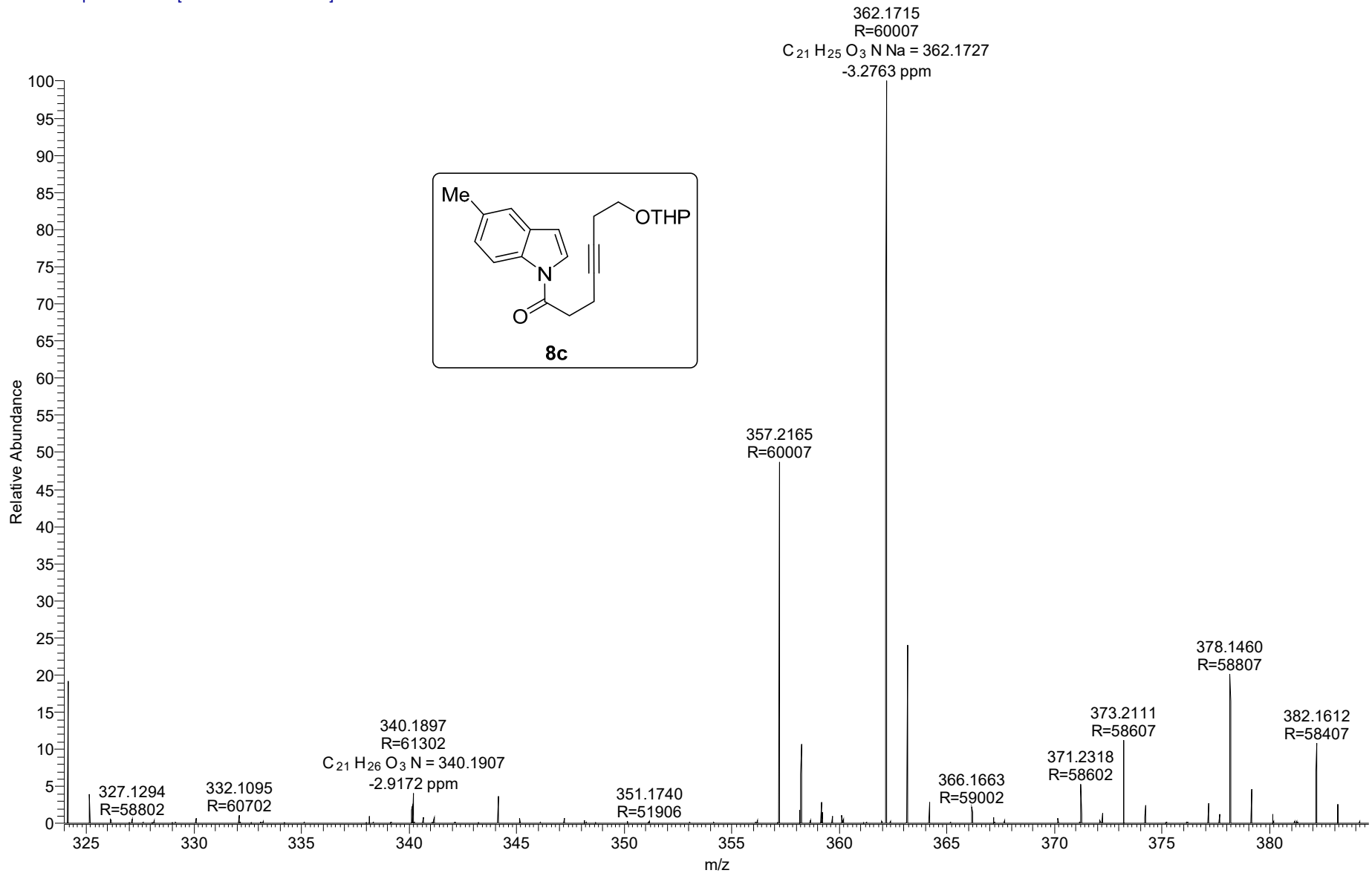
MSH-3 #298 RT: 1.62 AV: 1 NL: 4.35E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

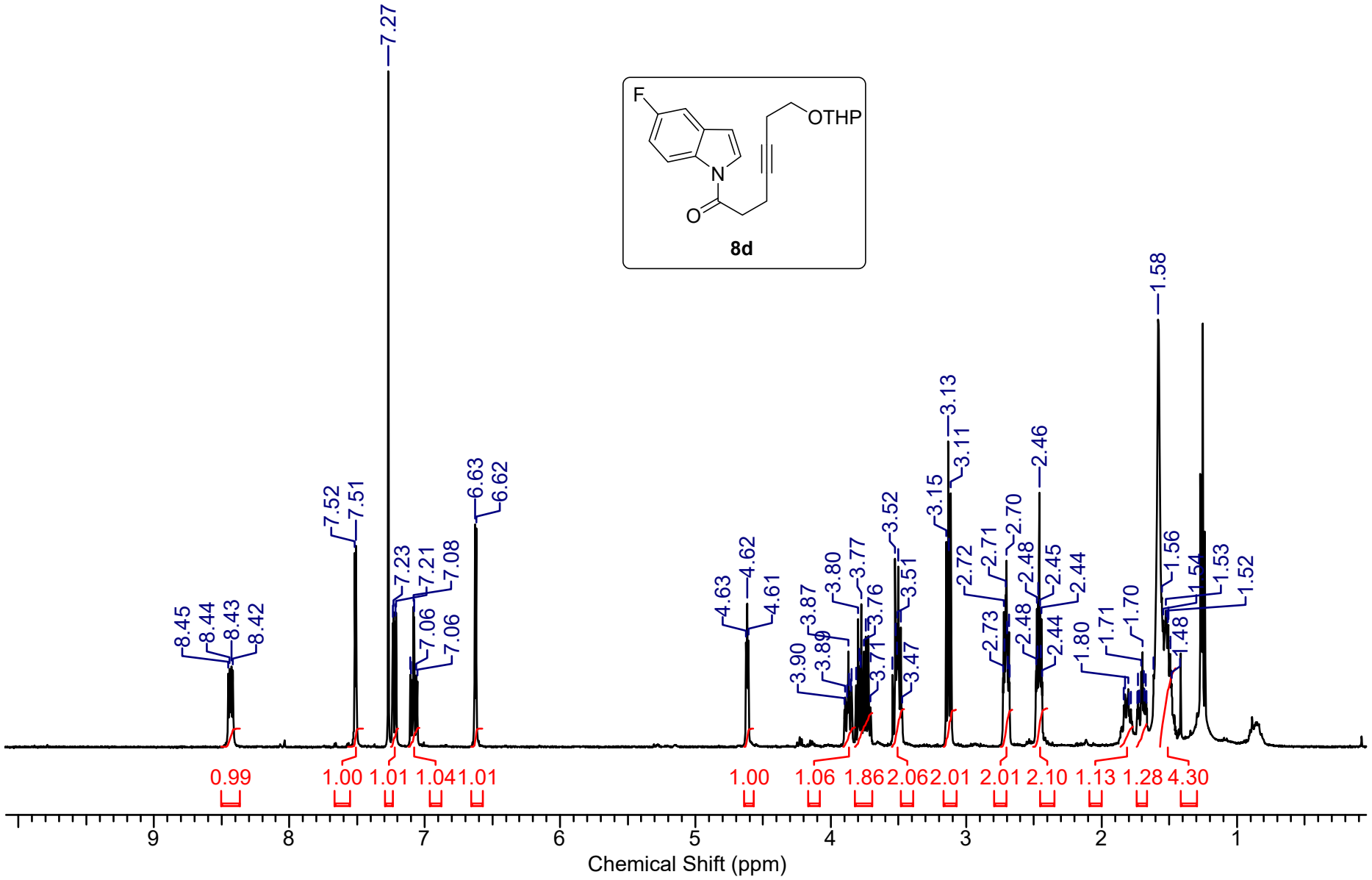


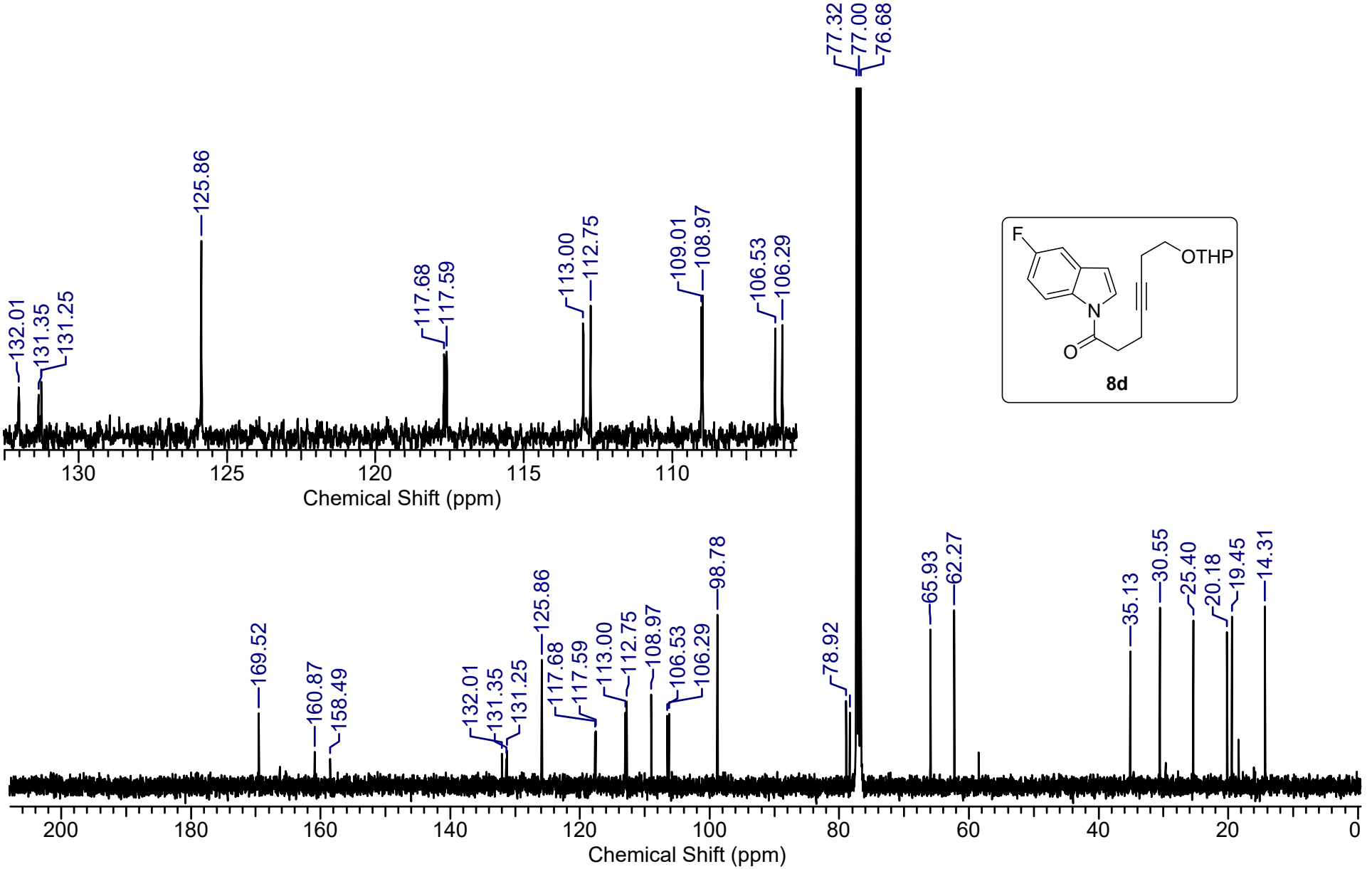

Me
OTHP
N
O
8c
3.90
3.88
3.87
3.85
3.82
3.80
3.78
3.78
3.76
3.55
3.53
3.51
3.50
3.49
2.45
1.24
1.13
2.28
3.9
3.8
3.7
3.6
3.5
Chemical Shift (ppm)
CHLOROFORM-d
8.33
8.31
7.44
7.43
7.36
7.27
7.19
7.19
7.16
6.59
6.58
4.63
4.62
4.61
3.85
3.80
3.78
3.53
3.50
3.14
3.12
3.10
2.70
2.70
2.48
2.46
1.70
1.59
1.58
1.56
1.53
1.52
1.51
1.00
0.99
1.04
1.08
1.01
1.05
1.24
1.13
2.28
2.08
2.07
5.36
1.21
1.49
4.64
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

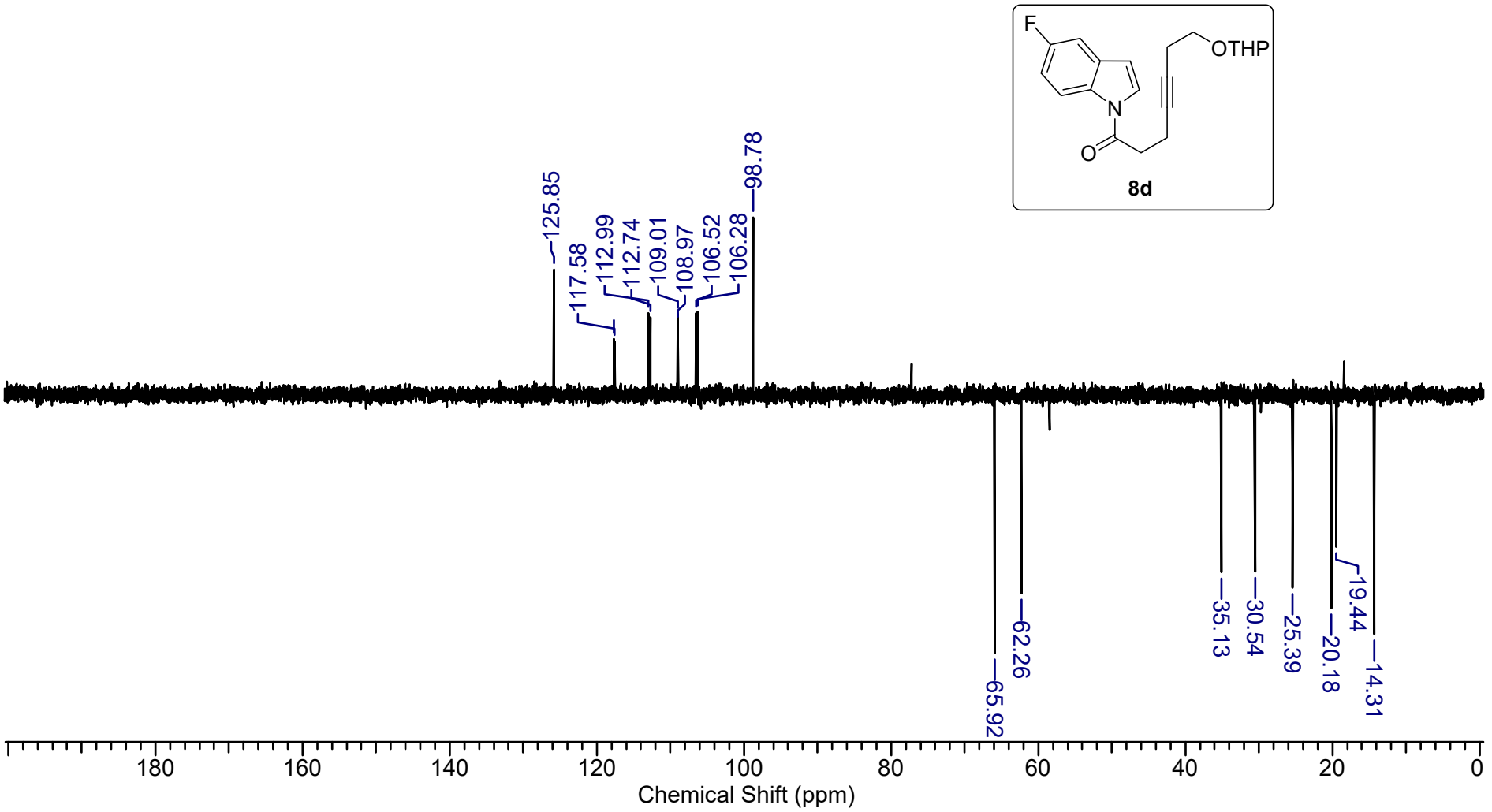

CHLOROFORM-d
77.32
77.00
76.68
Me
OTHP
N
O
8c
169.54
133.77
133.29
130.57
126.46
124.41
120.78
116.16
109.14
98.77
79.12
65.95
62.26
35.27
30.54
25.39
21.33
20.19
19.45
14.30
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

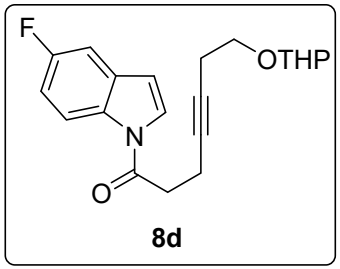

126.46
124.40
120.77
116.15
109.13
98.76
21.32
35.26
30.54
25.39
20.18
19.43
14.30
65.94
62.25
Me
OTHP
N
O
8c
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


MSH-2 #322 RT: 1.74 AV: 1 NL: 1.31E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
362.1715
R=60007
C 21 H 25 O 3 N Na = 362.1727
-3.2763 ppm
Me
OTHP
N
O
8c
357.2165
R=60007
340.1897
R=61302
C 21 H 26 O 3 N = 340.1907
-2.9172 ppm
327.1294
R=58802
332.1095
R=60702
351.1740
R=51906
366.1663
R=59002
371.2318
R=58602
373.2111
R=58607
378.1460
R=58807
382.1612
R=58407
Relative Abundance
m/z


F
OTHP
N
O
8d
7.27
8.45
8.44
8.43
8.42
7.52
7.51
7.23
7.21
7.08
7.06
7.06
6.63
6.62
4.63
4.62
4.61
3.90
3.89
3.87
3.80
3.77
3.76
3.71
3.52
3.51
3.47
3.15
3.13
3.11
2.72
2.73
2.71
2.70
2.48
2.48
2.46
2.45
2.44
2.44
1.80
1.71
1.70
1.58
1.56
1.54
1.53
1.52
1.48
0.99
1.00
1.01
1.04
1.01
1.00
1.06
1.86
2.06
2.01
2.01
2.10
1.13
1.28
4.30
9
8
7
6
5
4
3
2
1
Chemical Shift (ppm)

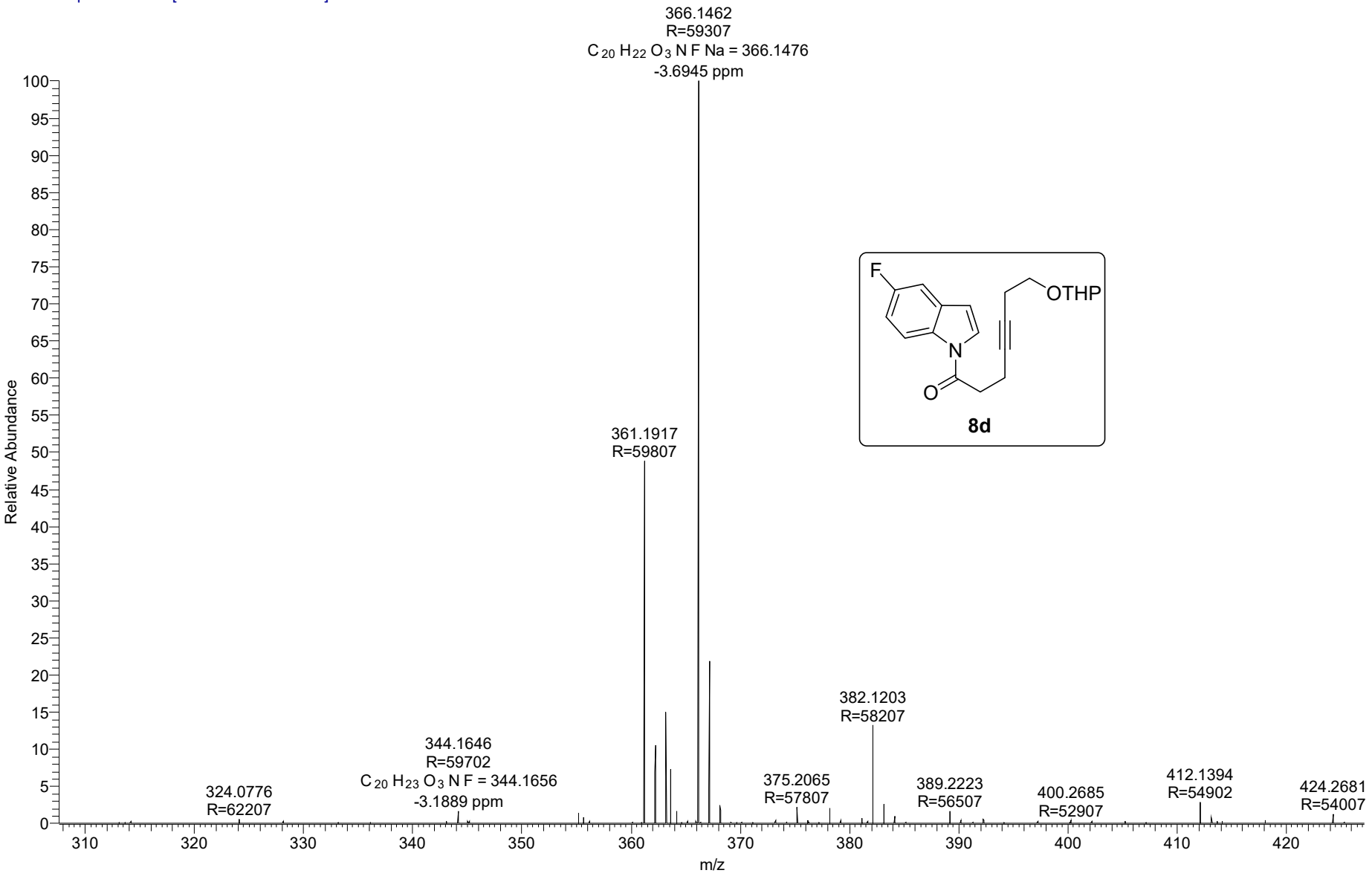

77.32
77.00
76.68
125.86
132.01
131.35
131.25
117.68
117.59
113.00
112.75
109.01
108.97
106.53
106.29
F
OTHP
N
O
8d
130
125
120
115
110
Chemical Shift (ppm)
169.52
160.87
158.49
132.01
131.35
131.25
125.86
117.68
117.59
113.00
112.75
108.97
106.53
106.29
98.78
78.92
65.93
62.27
35.13
30.55
25.40
20.18
19.45
14.31
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

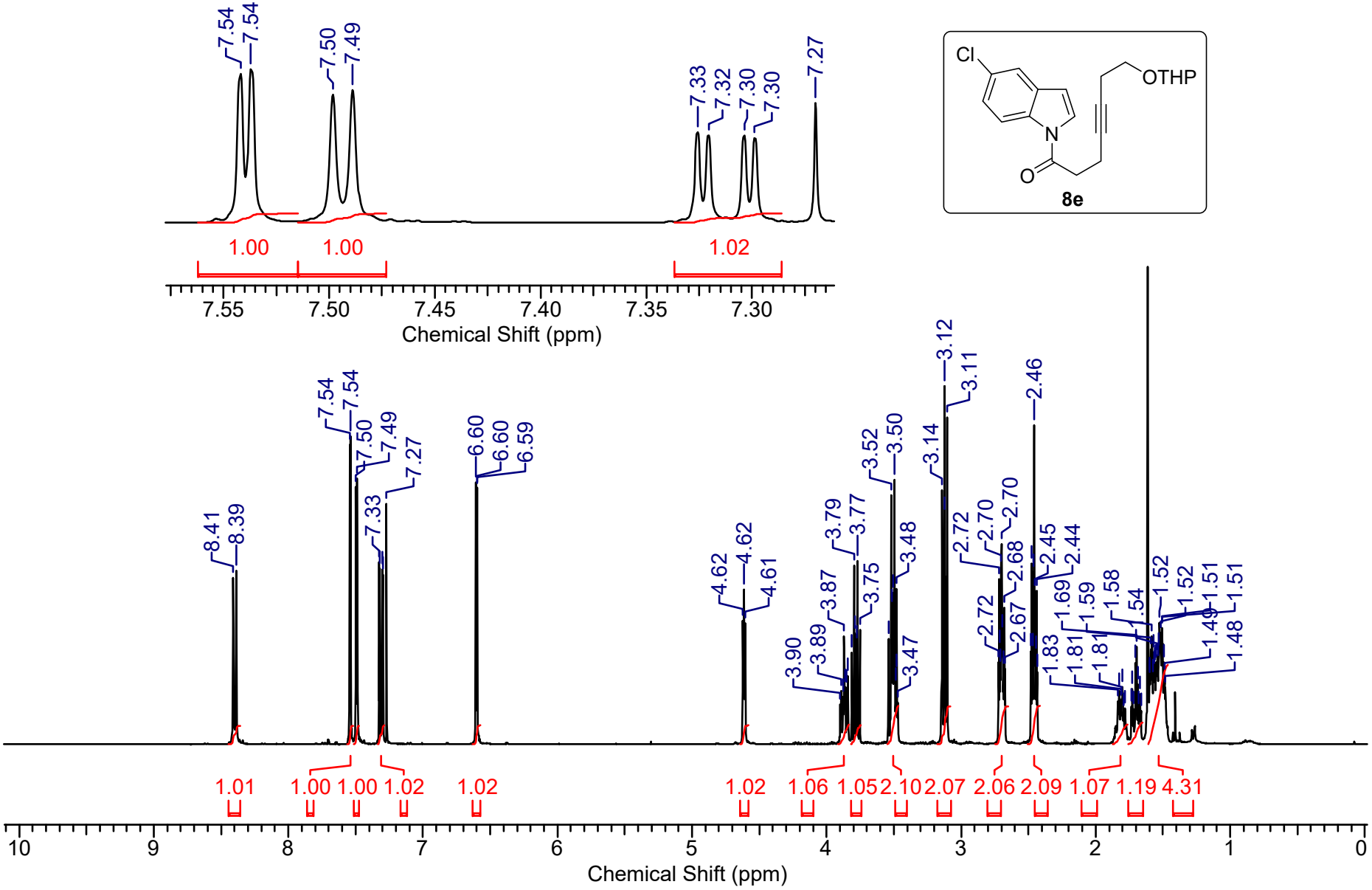

F
OTHP
N
O
8d
125.85
117.58
112.99
112.74
109.01
108.97
106.52
106.28
98.78
65.92
62.26
35.13
30.54
25.39
20.18
19.44
14.31
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

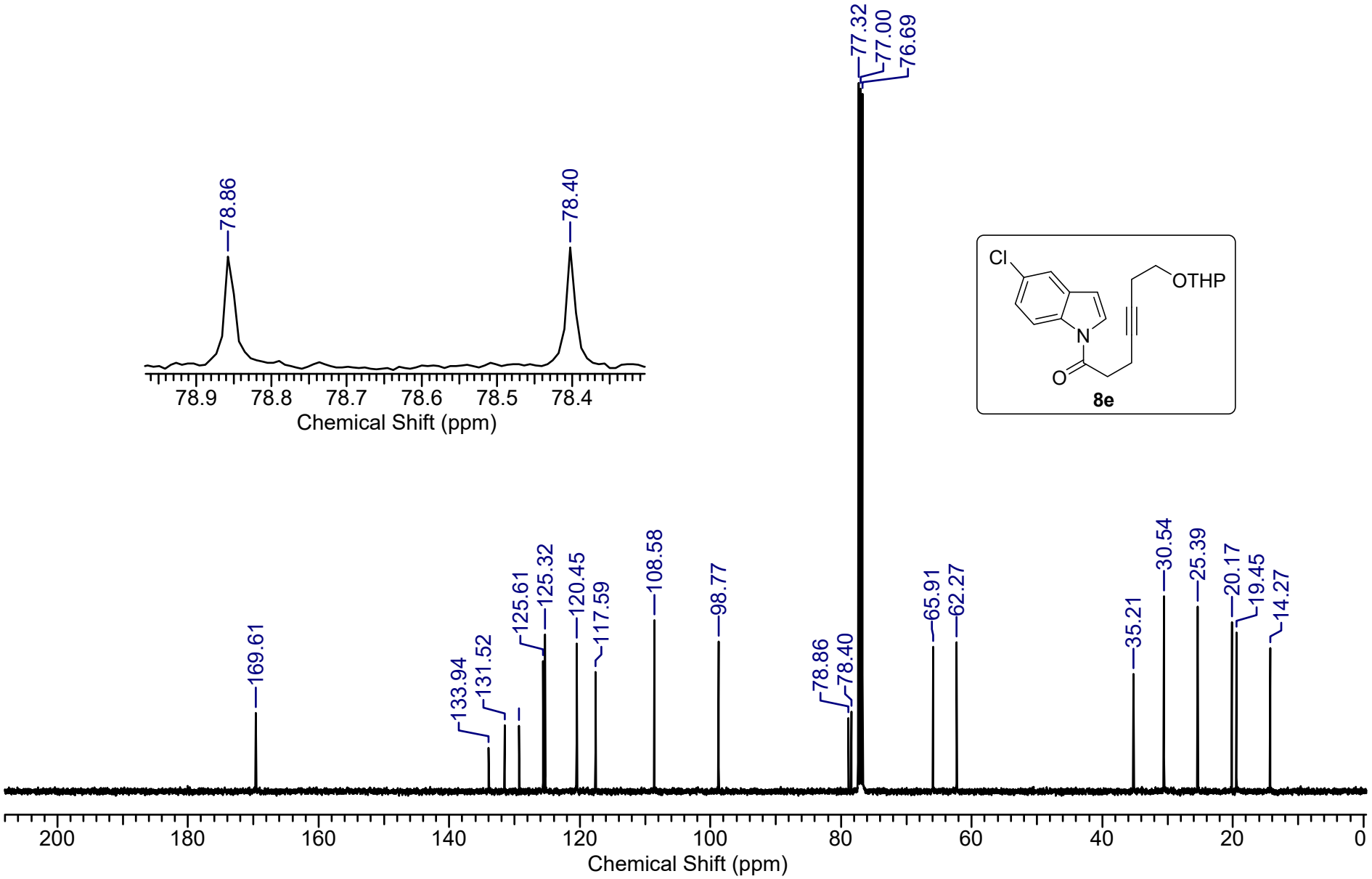


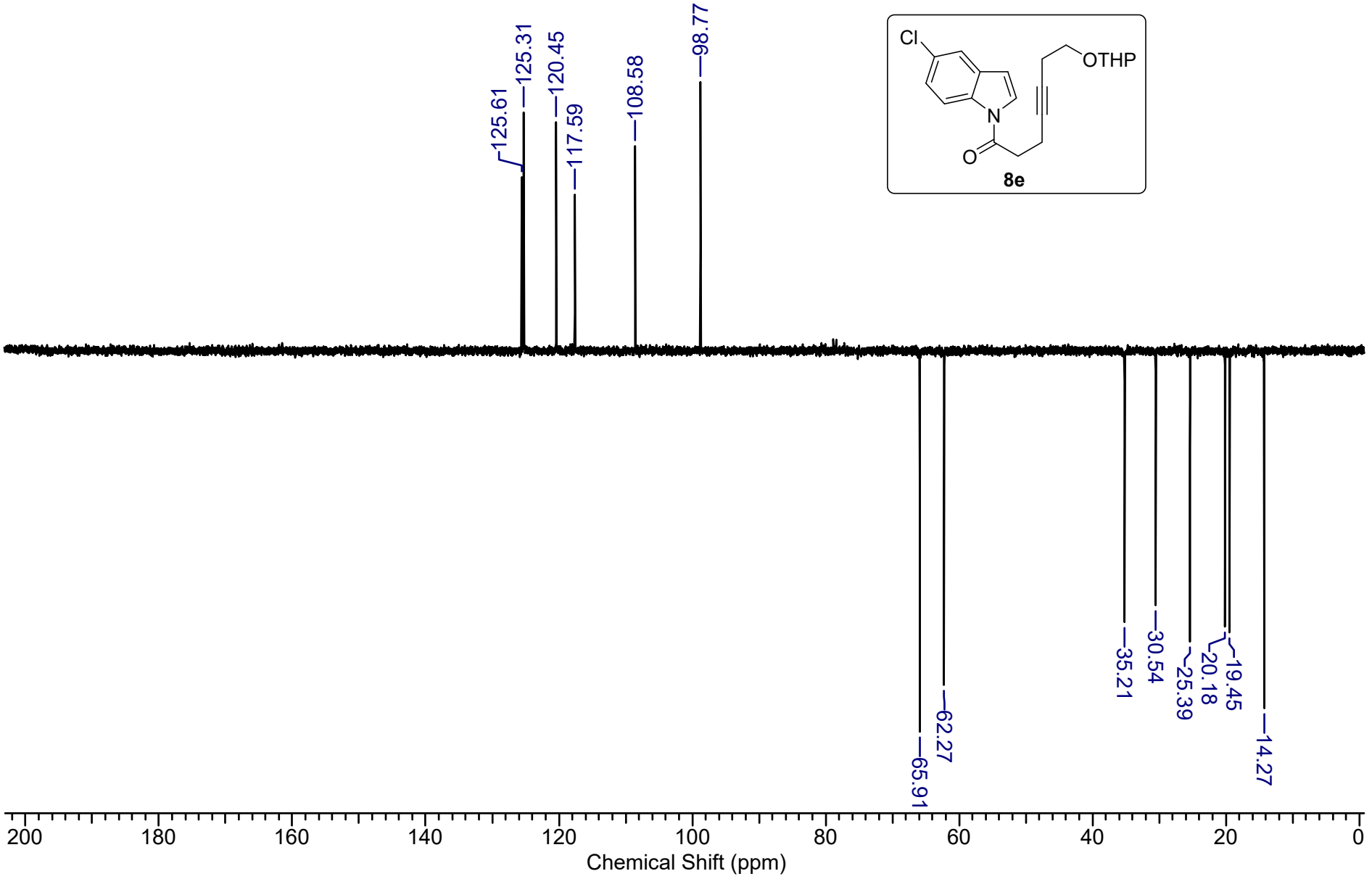
8d

MSH-4 #305 RT: 1.66 AV: 1 NL: 3.00E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

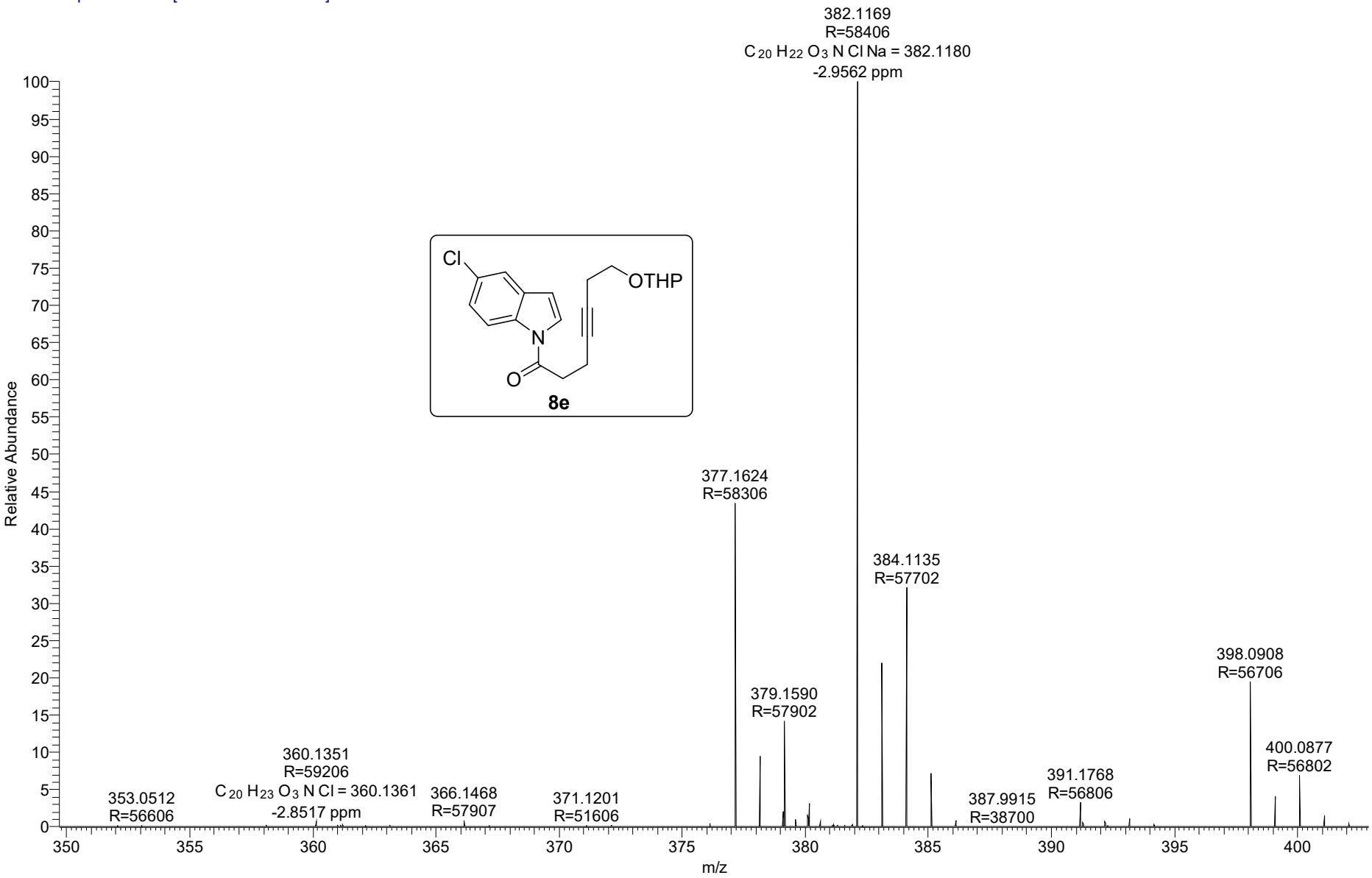


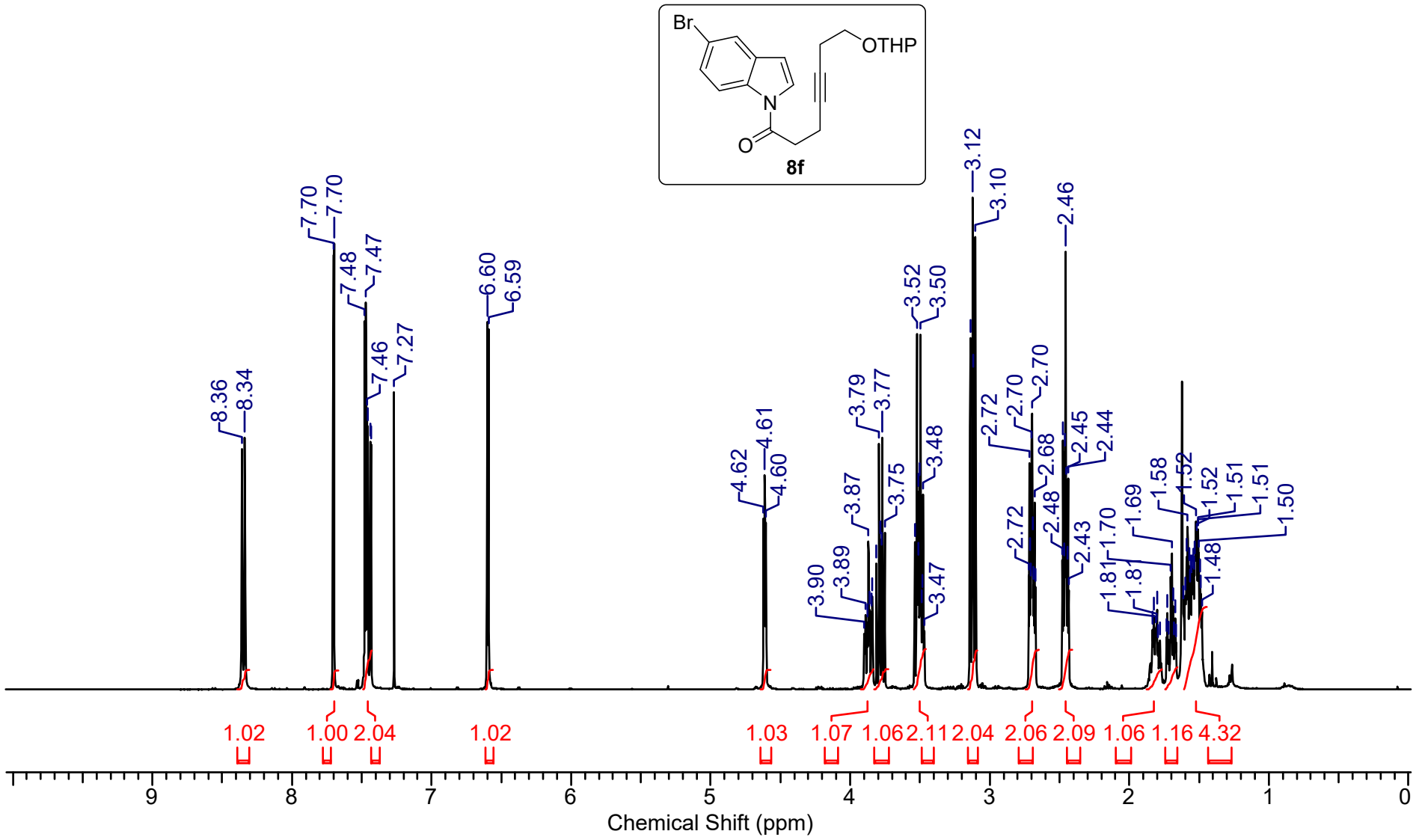

7.54
7.54
7.50
7.49
7.33
7.32
7.30
7.30
7.27
1.00
1.00
1.02
7.55
7.50
7.45
7.40
7.35
7.30
Chemical Shift (ppm)
Cl
OTHP
N
O
8e
8.41
8.39
7.54
7.54
7.50
7.49
7.33
7.27
6.60
6.60
6.59
4.62
4.62
4.61
3.90
3.89
3.87
3.79
3.77
3.75
3.52
3.50
3.48
3.47
3.14
3.12
3.11
2.72
2.72
2.70
2.70
2.68
2.67
2.46
2.45
2.44
1.83
1.81
1.81
1.69
1.59
1.58
1.54
1.52
1.52
1.51
1.49
1.48
1.51
1.01
1.00
1.00
1.02
1.02
1.02
1.06
1.05
2.10
2.07
2.06
2.09
1.07
1.19
4.31
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

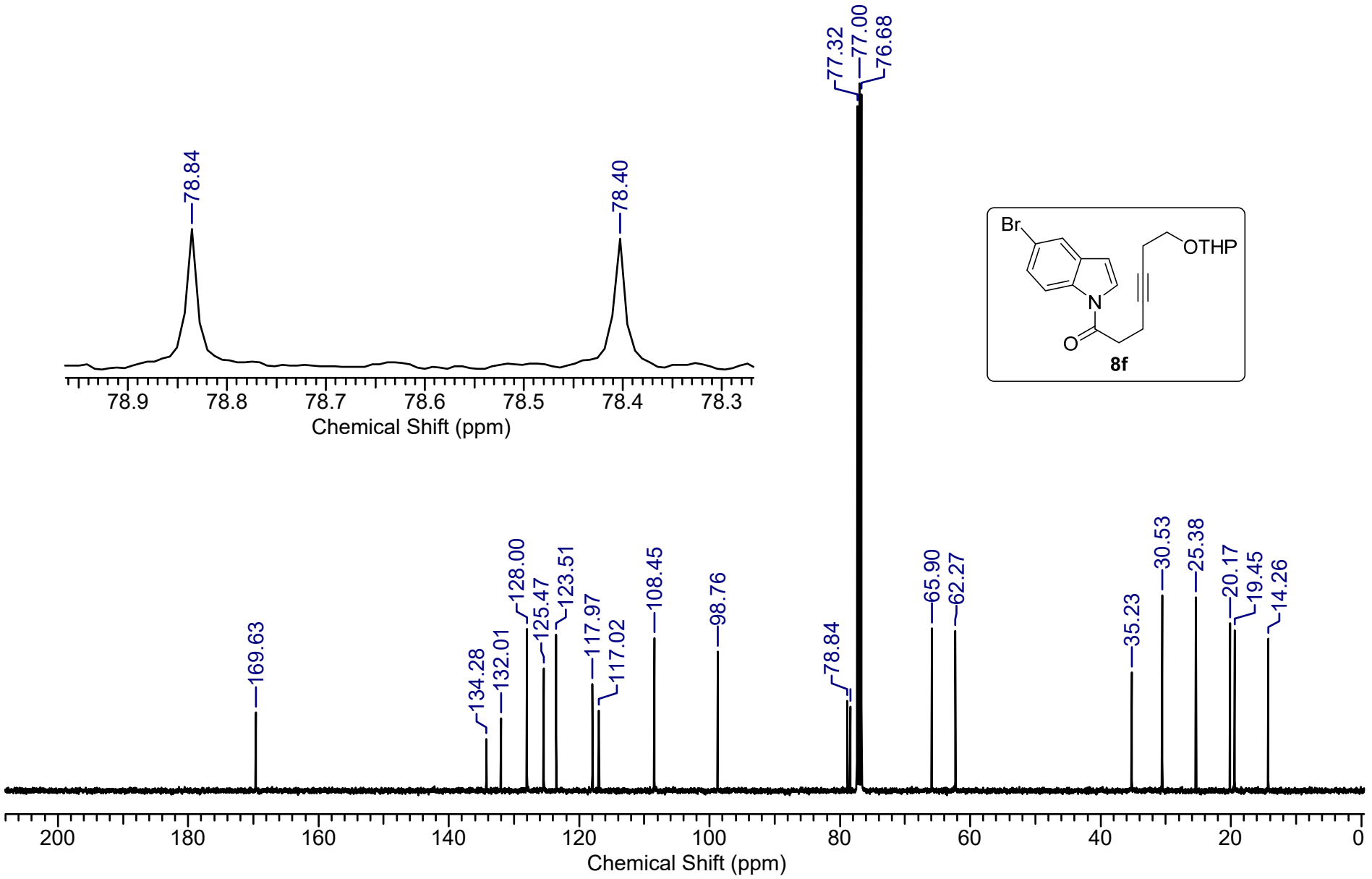

77.32
77.00
76.69
78.86
78.40
78.9
78.8
78.7
78.6
78.5
78.4
Chemical Shift (ppm)
Cl
OTHP
N
O
8e
169.61
133.94
131.52
125.61
125.32
120.45
117.59
108.58
98.77
78.86
78.40
65.91
62.27
35.21
30.54
25.39
20.17
19.45
14.27
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

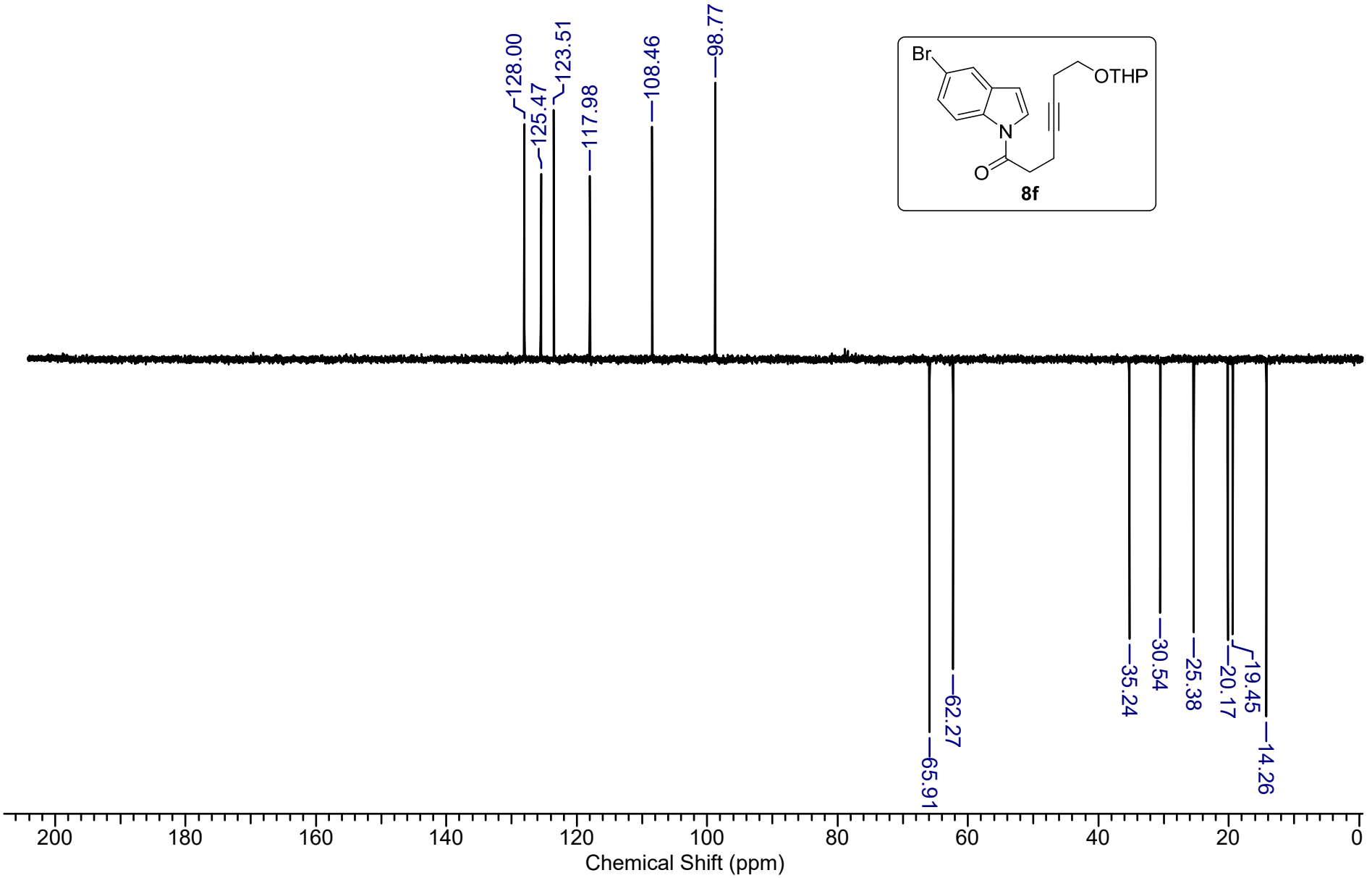

125.61
125.31
120.45
117.59
108.58
98.77
65.91
62.27
35.21
30.54
25.39
20.18
19.45
14.27
Cl
OTHP
N
O
8e
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

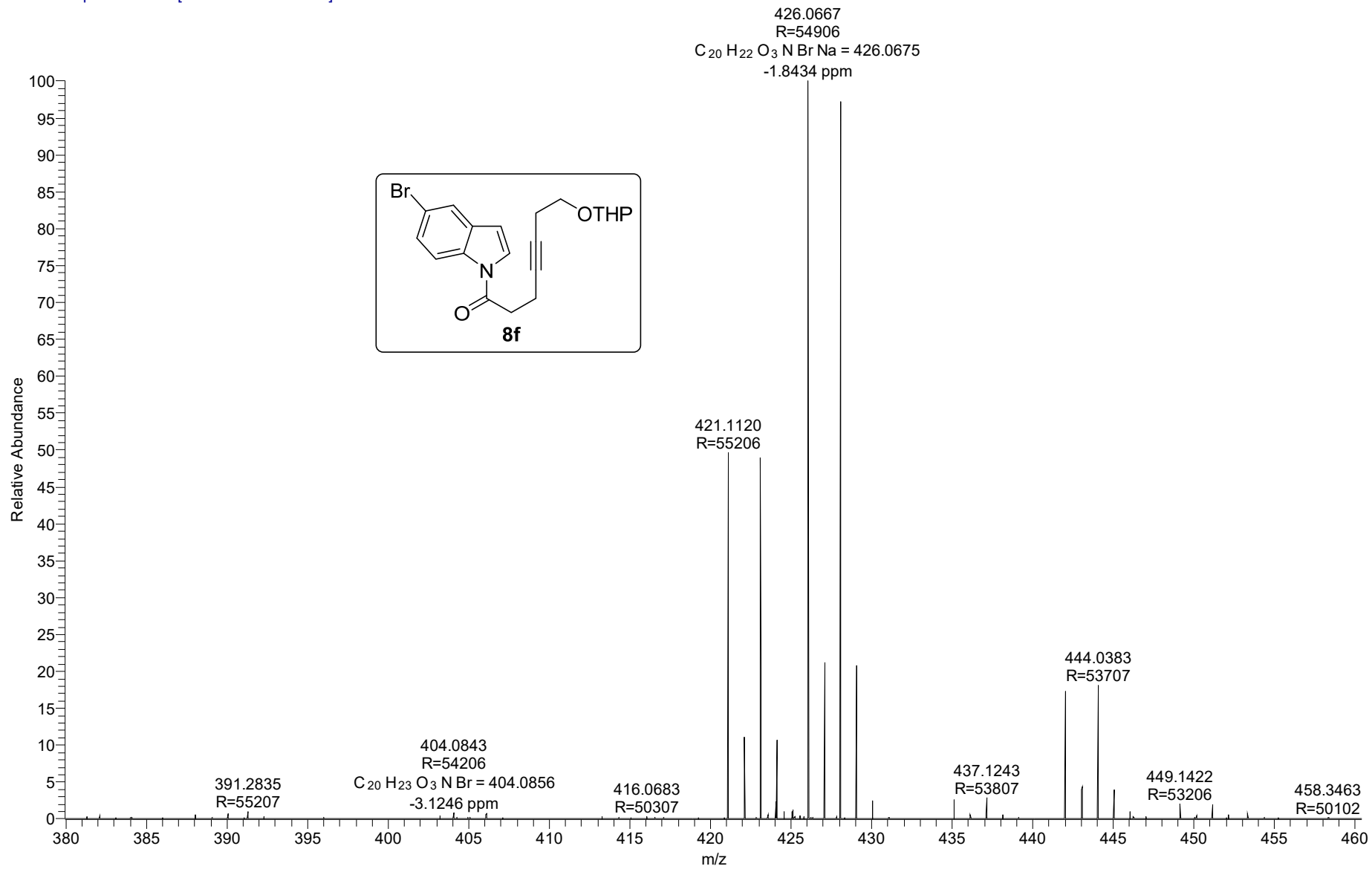
MSH-5 #342 RT: 1.86 AV: 1 NL: 1.42E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

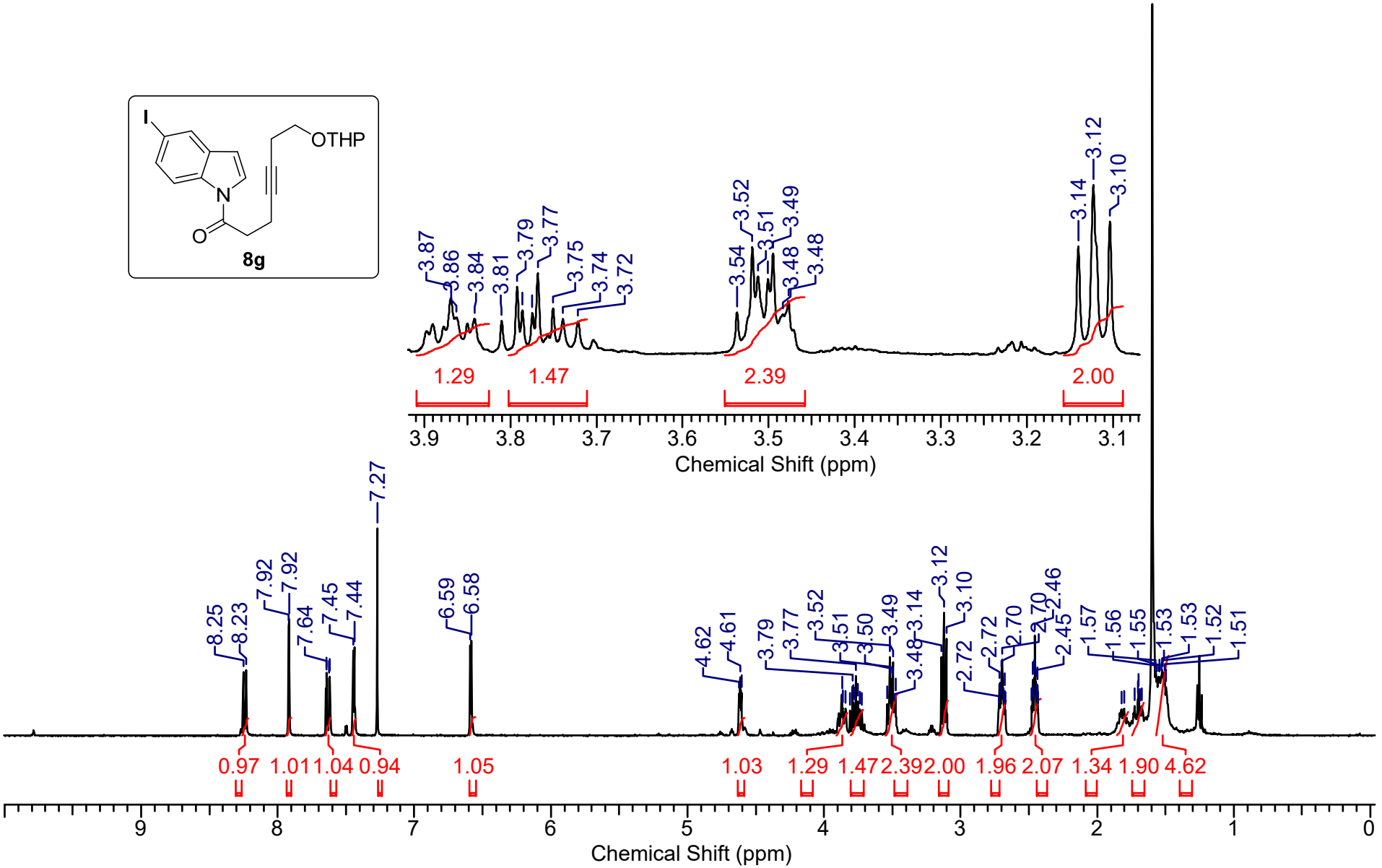


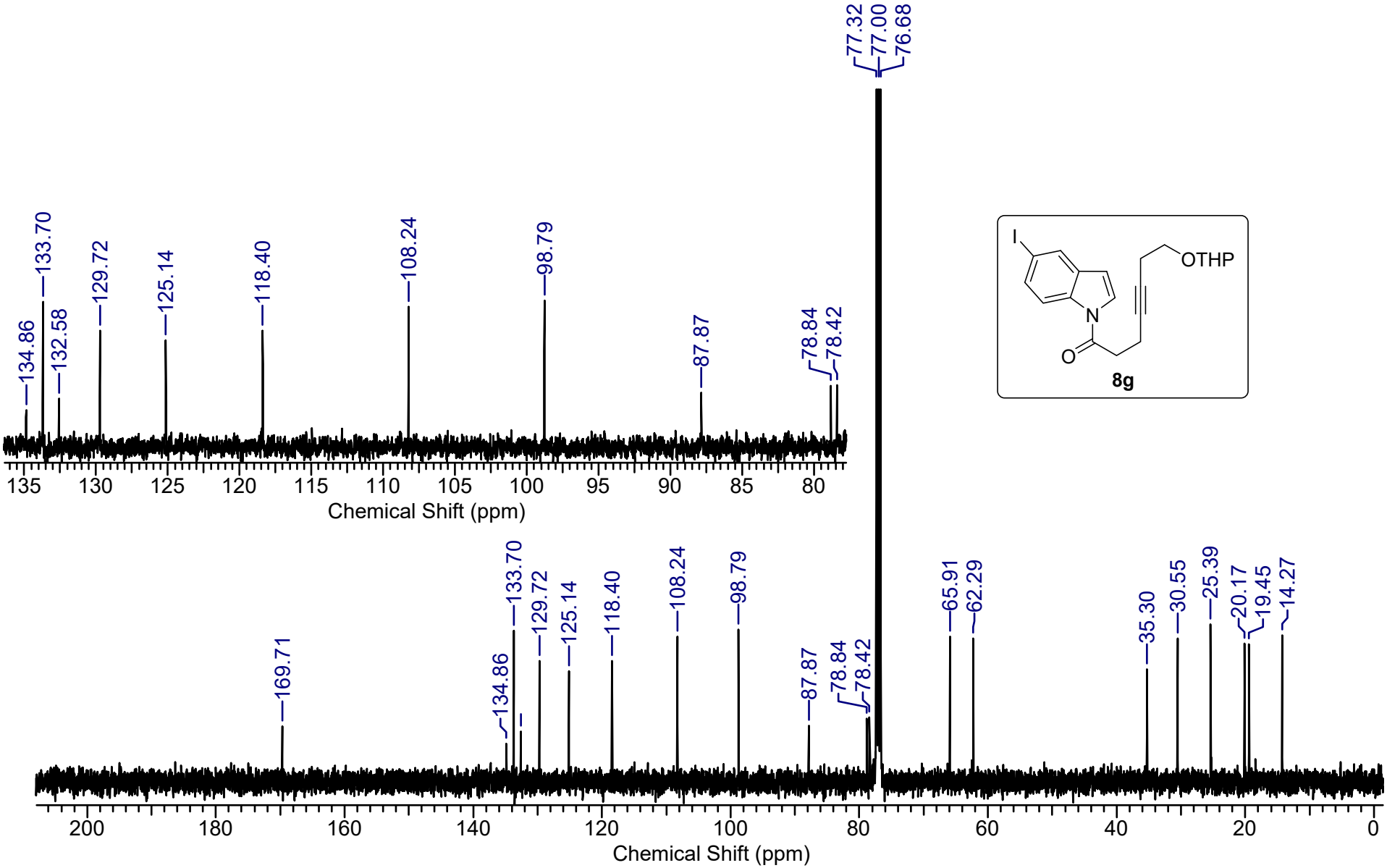

Br
OTHP
N
O
8f
8.36
8.34
7.70
7.70
7.48
7.47
7.46
7.27
6.60
6.59
4.62
4.61
4.60
3.90
3.89
3.87
3.79
3.77
3.75
3.52
3.50
3.48
3.47
3.12
3.10
2.72
2.70
2.72
2.70
2.68
2.48
2.46
2.45
2.44
2.43
1.81
1.81
1.70
1.69
1.58
1.52
1.52
1.51
1.51
1.50
1.48
1.02
1.00
2.04
1.02
1.03
1.07
1.06
2.11
2.04
2.06
2.09
1.06
1.16
4.32
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

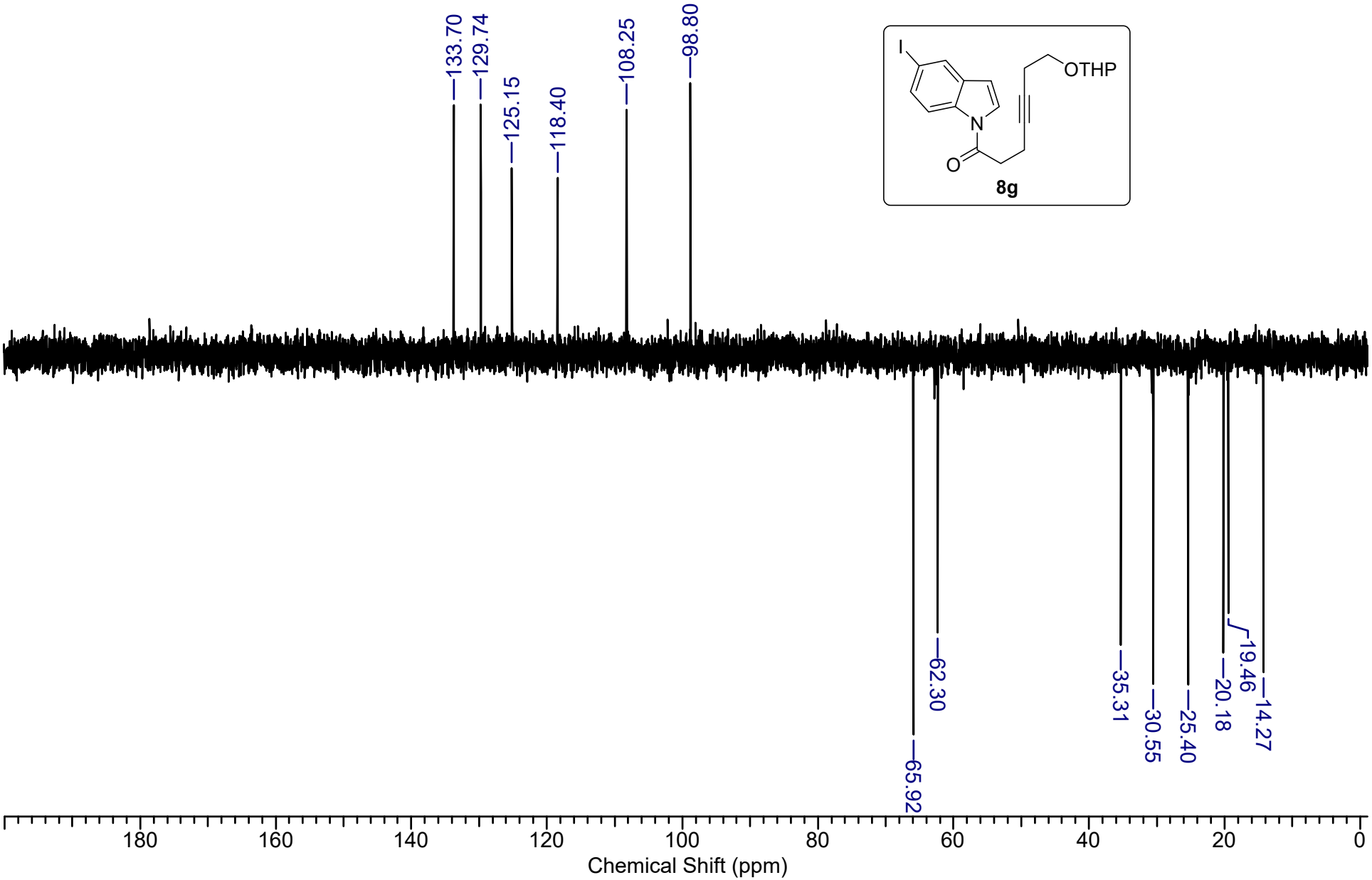

78.84
78.40
78.9
78.8
78.7
78.6
78.5
78.4
78.3
Chemical Shift (ppm)
77.32
77.00
76.68
Br
OTHP
N
O
8f
169.63
134.28
132.01
128.00
125.47
123.51
117.97
117.02
108.45
98.76
78.84
65.90
62.27
35.23
30.53
25.38
20.17
19.45
14.26
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


128.00
125.47
123.51
117.98
108.46
98.77
65.91
62.27
35.24
30.54
25.38
20.17
19.45
14.26
Br
OTHP
N
O
8f
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

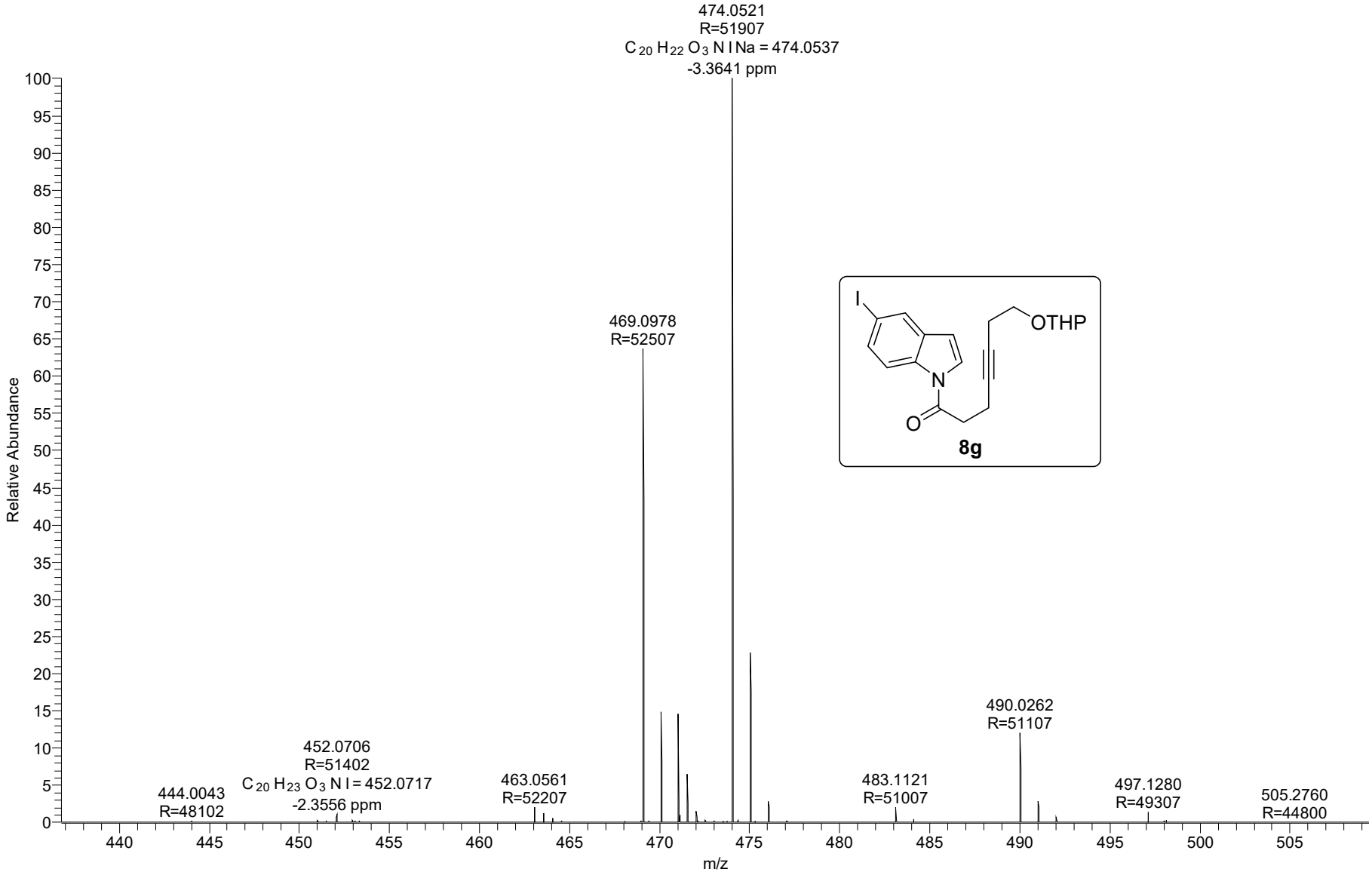

MSH-6 #378 RT: 2.05 AV: 1 NL: 9.54E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
426.0667
R=54906
C 20 H 22 O 3 N Br Na = 426.0675
-1.8434 ppm
421.1120
R=55206
444.0383
R=53707
404.0843
R=54206
C 20 H 23 O 3 N Br = 404.0856
-3.1246 ppm
391.2835
R=55207
416.0683
R=50307
437.1243
R=53807
449.1422
R=53206
458.3463
R=50102
Br
OTHP
N
O
8f
Relative Abundance
m/z

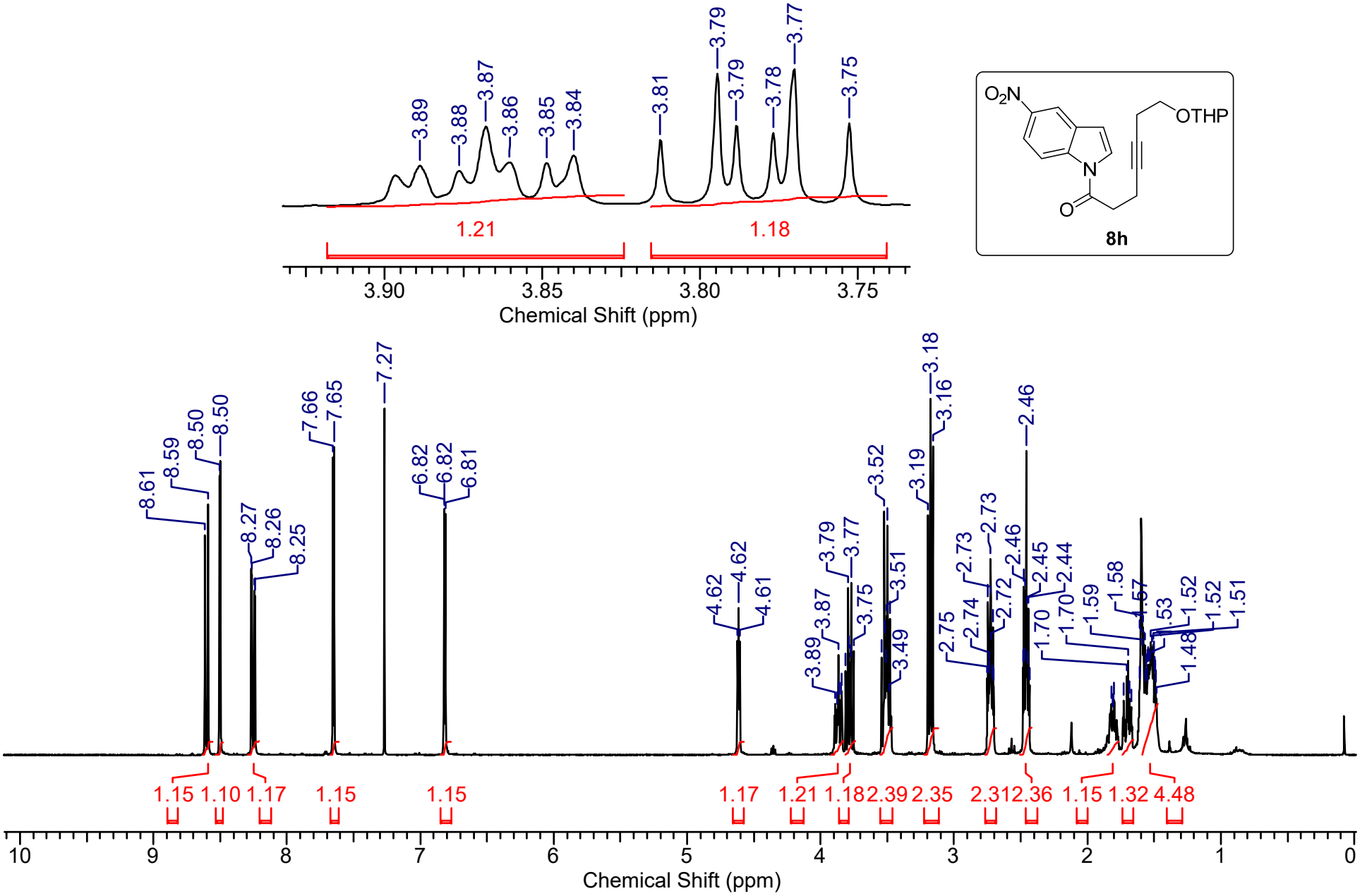

I
OTHP
N
O
8g
3.87
3.86
3.84
3.81
3.79
3.77
3.75
3.74
3.72
3.54
3.52
3.51
3.49
3.48
3.48
3.14
3.12
3.10
1.29
1.47
2.39
2.00
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
Chemical Shift (ppm)
8.25
8.23
7.92
7.92
7.64
7.45
7.44
7.27
6.59
6.58
4.62
4.61
3.79
3.77
3.52
3.51
3.50
3.49
3.48
3.14
3.12
3.10
2.72
2.72
2.70
2.70
2.46
2.45
1.57
1.56
1.55
1.53
1.53
1.52
1.51
0.97
1.01
1.04
0.94
1.05
1.03
1.29
1.47
2.39
2.00
1.96
2.07
1.34
1.90
4.62
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

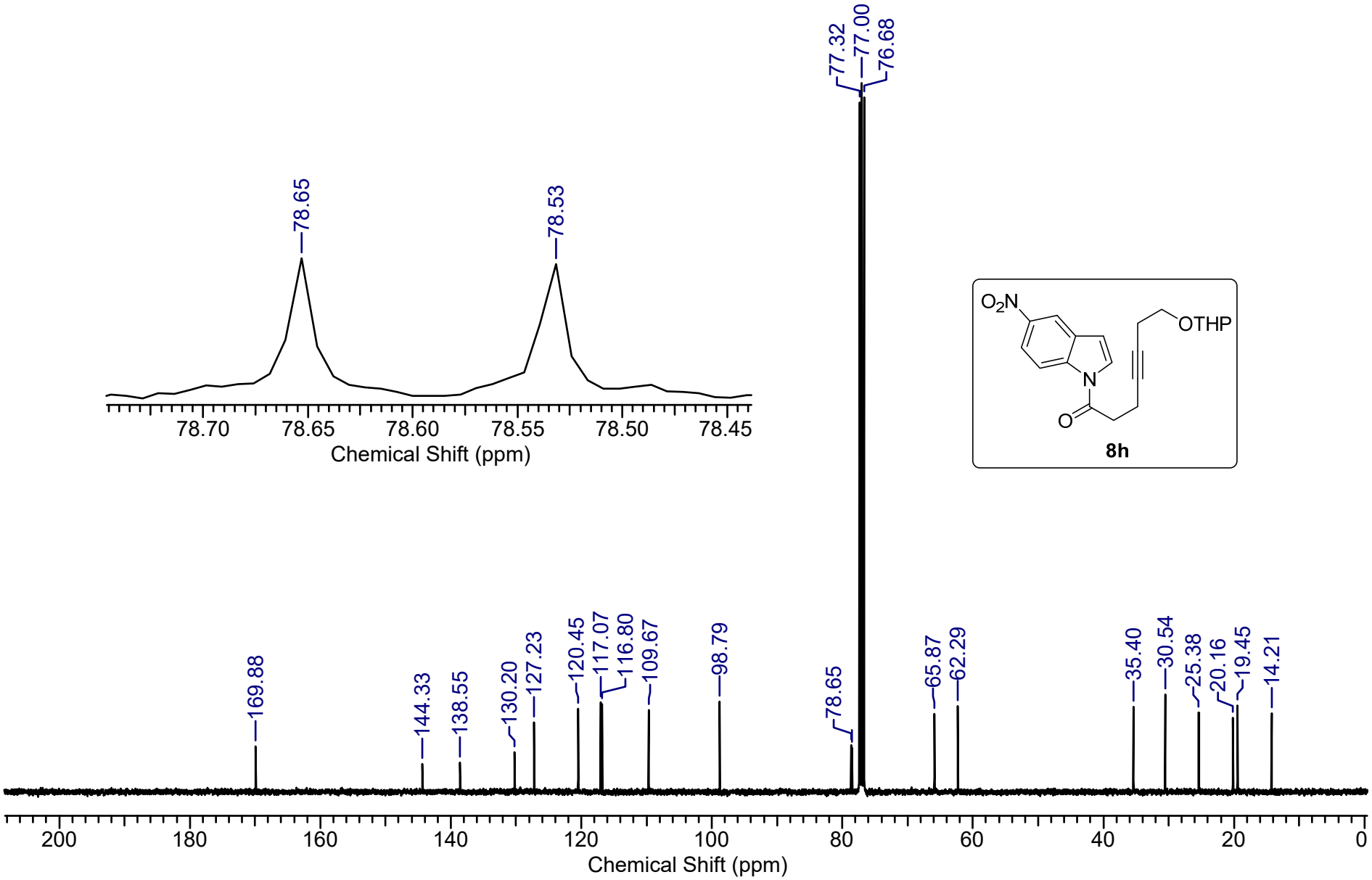

77.32
77.00
76.68
134.86
133.70
132.58
129.72
125.14
118.40
108.24
98.79
87.87
78.84
78.42
I
OTHP
N
O
8g
135
130
125
120
115
110
105
100
95
90
85
80
Chemical Shift (ppm)
169.71
134.86
133.70
129.72
125.14
118.40
108.24
98.79
87.87
78.84
78.42
65.91
62.29
35.30
30.55
25.39
20.17
19.45
14.27
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

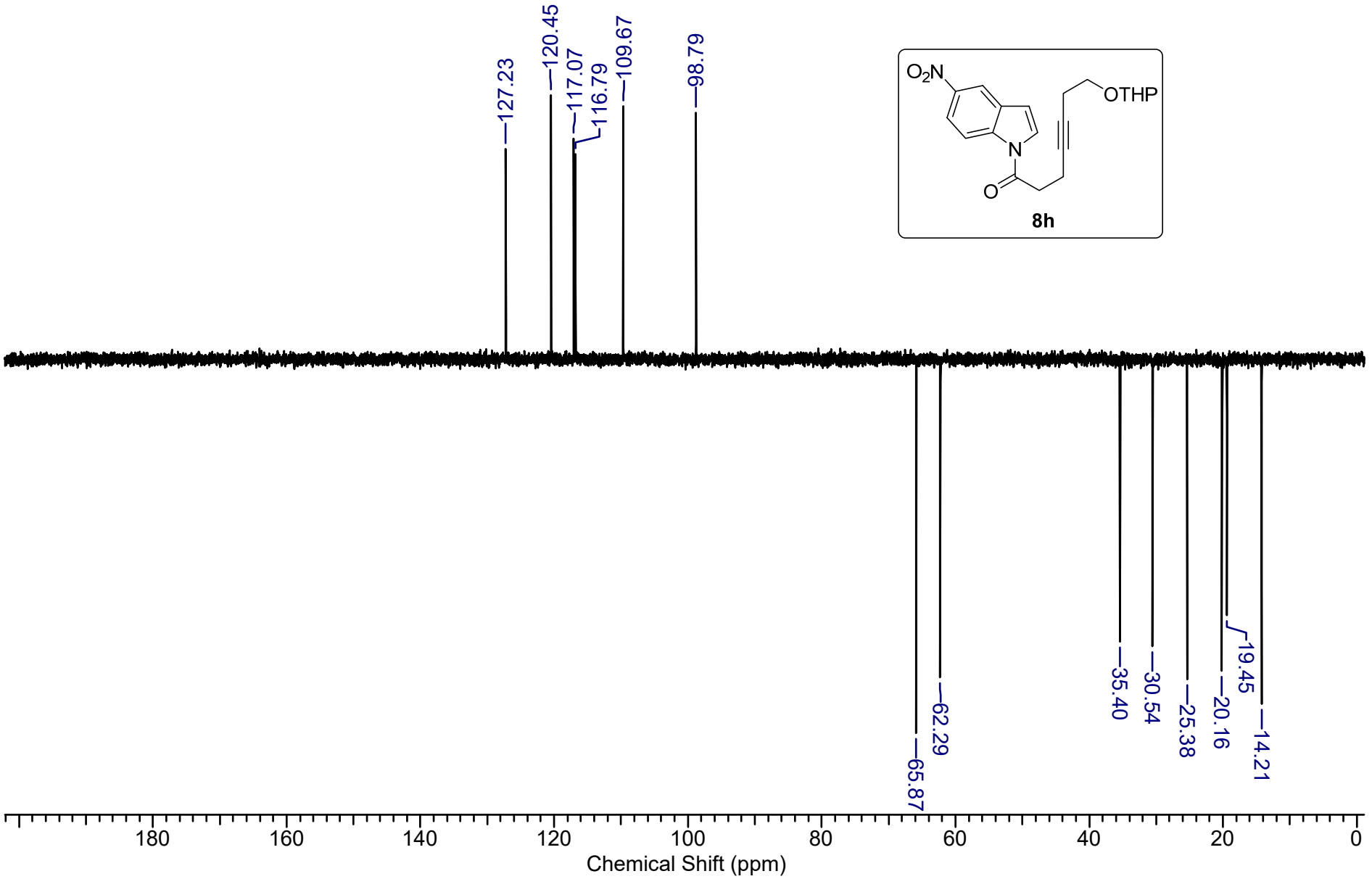

133.70
129.74
125.15
118.40
108.25
98.80
65.92
62.30
35.31
30.55
25.40
20.18
19.46
14.27
I
OTHP
N
O
8g
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MSH-7 #364 RT: 1.97 AV: 1 NL: 3.37E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

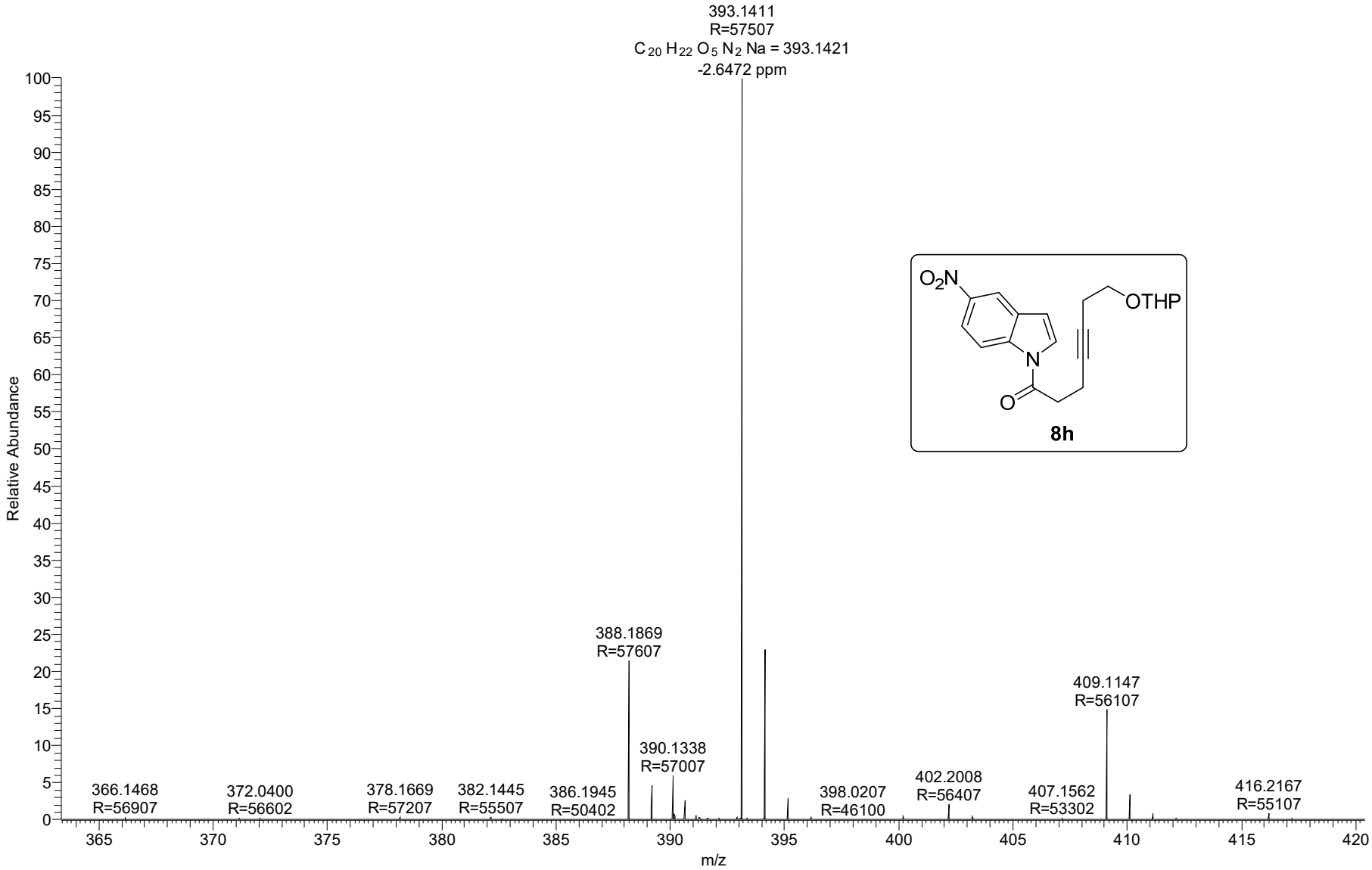


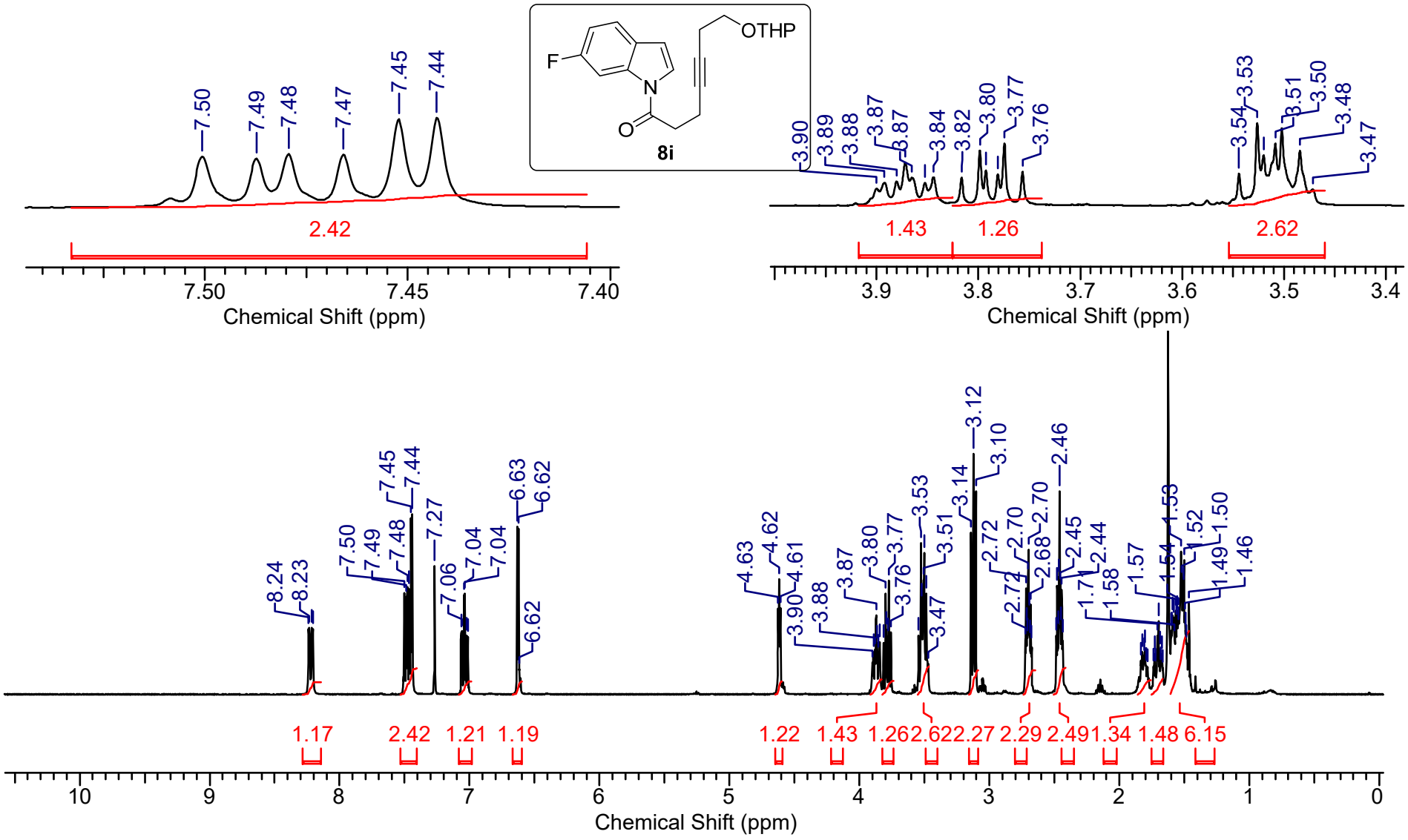

3.89
3.88
3.87
3.86
3.85
3.84
3.81
3.79
3.79
3.78
3.77
3.75
1.21
1.18
3.90
3.85
3.80
3.75
Chemical Shift (ppm)
O2N
OTHP
N
O
8h
8.61
8.59
8.50
8.50
8.27
8.26
8.25
7.66
7.65
7.27
6.82
6.82
6.81
4.62
4.62
4.61
3.89
3.87
3.79
3.77
3.75
3.52
3.51
3.49
3.19
3.18
3.16
2.75
2.74
2.73
2.72
2.73
2.46
2.46
2.45
2.44
1.70
1.59
1.58
1.53
1.52
1.52
1.51
1.48
1.15 1.10 1.17 1.15 1.15 1.17 1.21 1.18 2.39 2.35 2.31 2.36 1.15 1.32 4.48
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

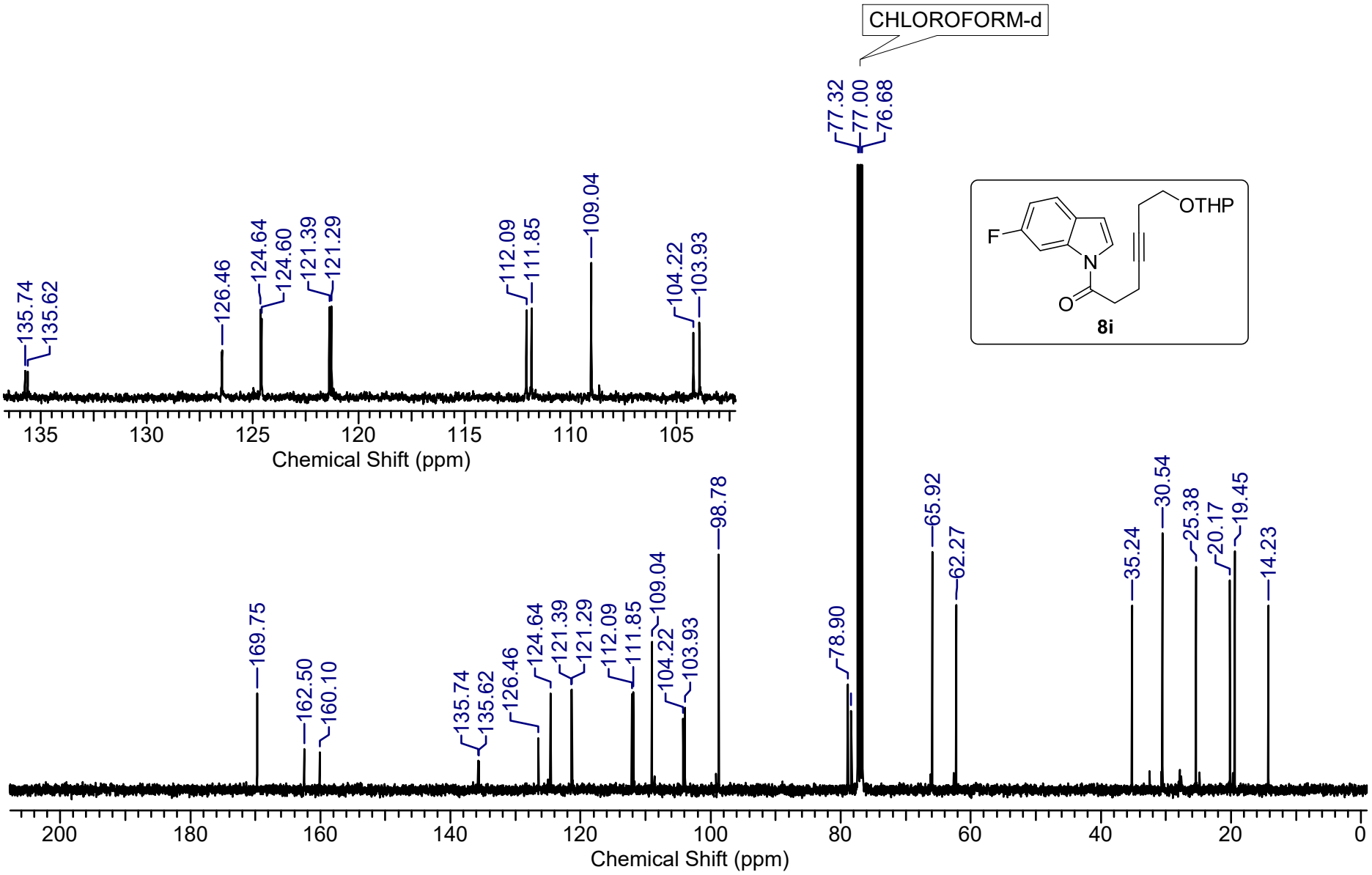

77.32
77.00
76.68
78.65
78.53
78.70
78.65
78.60
78.55
78.50
78.45
Chemical Shift (ppm)
O2N
OTHP
N
O
8h
169.88
144.33
138.55
130.20
127.23
120.45
117.07
116.80
109.67
98.79
78.65
65.87
62.29
35.40
30.54
25.38
20.16
19.45
14.21
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

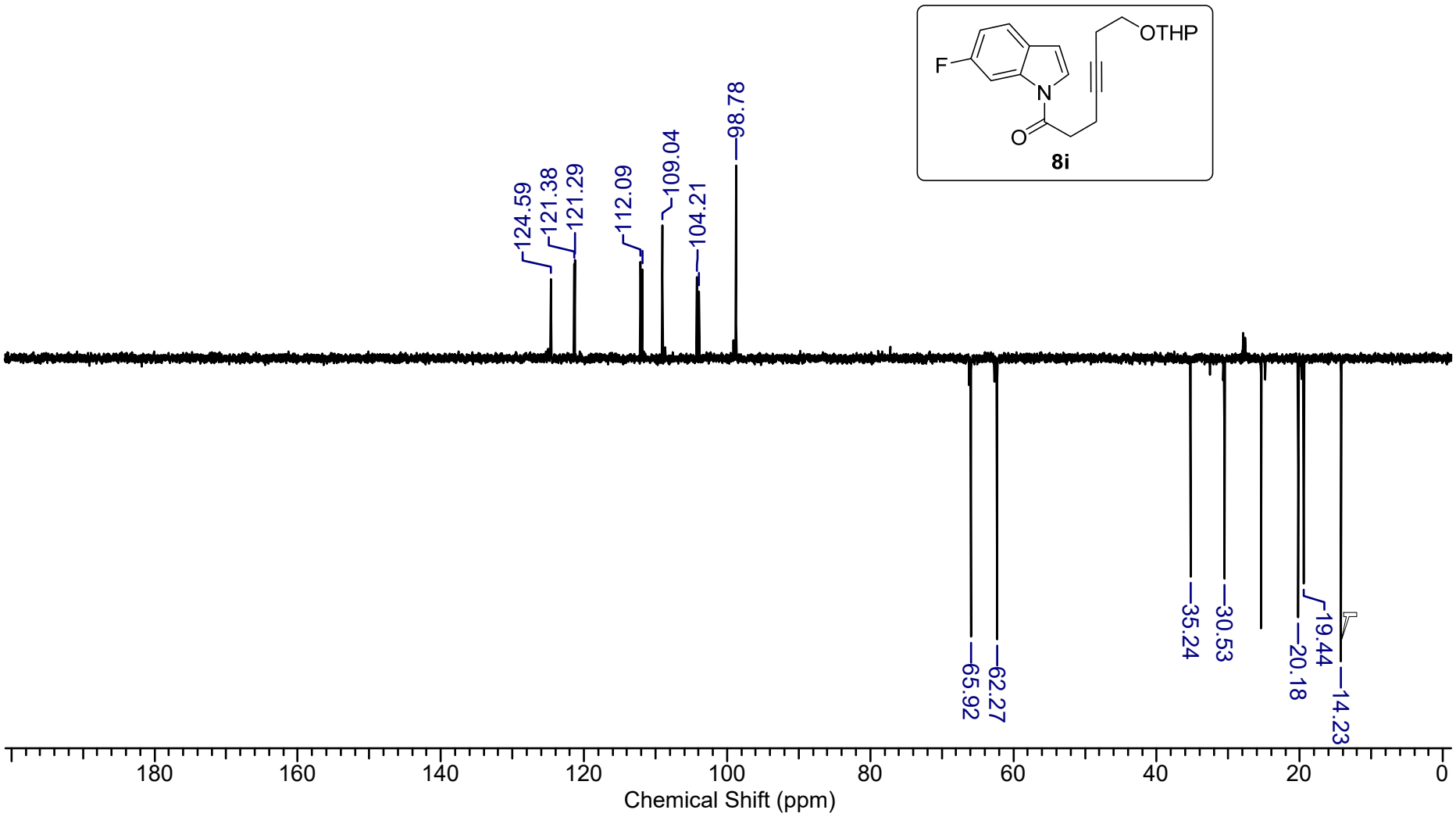

127.23
120.45
117.07
116.79
109.67
98.79
65.87
62.29
35.40
30.54
25.38
20.16
19.45
14.21
O2N
OTHP
N
O
8h
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

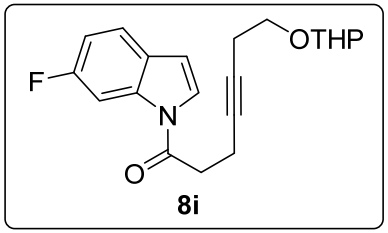
MSH-8 #302 RT: 1.64 AV: 1 NL: 3.56E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]




F
N
O
OTHP
8i
7.50
7.49
7.48
7.47
7.45
7.44
2.42
7.50
7.45
7.40
Chemical Shift (ppm)
3.90
3.89
3.88
3.87
3.87
3.84
3.82
3.80
3.77
3.76
3.54
3.53
3.51
3.50
3.48
3.47
1.43
1.26
2.62
3.9
3.8
3.7
3.6
3.5
3.4
Chemical Shift (ppm)
8.24
8.23
7.50
7.49
7.48
7.45
7.44
7.27
7.06
7.04
7.04
6.63
6.62
6.62
4.63
4.62
4.61
3.90
3.88
3.87
3.80
3.77
3.76
3.53
3.51
3.47
3.14
3.12
3.10
2.72
2.74
2.70
2.70
2.68
2.46
2.45
2.44
1.71
1.58
1.57
1.54
1.53
1.52
1.50
1.49
1.46
1.17
2.42
1.21
1.19
1.22
1.43
1.26
2.62
2.27
2.29
2.49
1.34
1.48
6.15
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

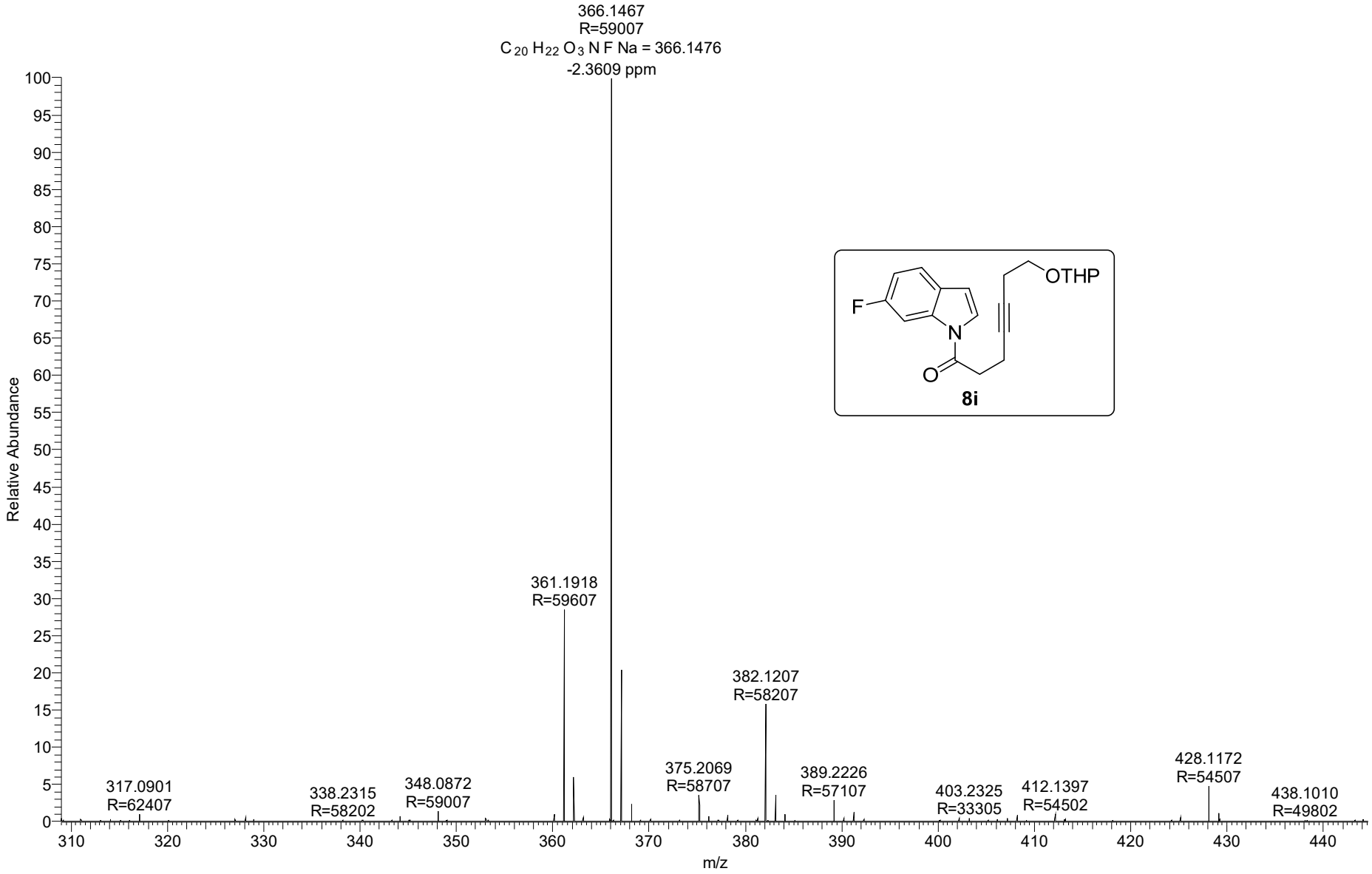

CHLOROFORM-d
77.32
77.00
76.68
OTHP
F
N
O
8i
135.74
135.62
126.46
124.64
124.60
121.39
121.29
112.09
111.85
109.04
104.22
103.93
Chemical Shift (ppm)
169.75
162.50
160.10
135.74
135.62
126.46
124.64
121.39
121.29
112.09
111.85
109.04
104.22
103.93
98.78
78.90
65.92
62.27
35.24
30.54
25.38
20.17
19.45
14.23
Chemical Shift (ppm)

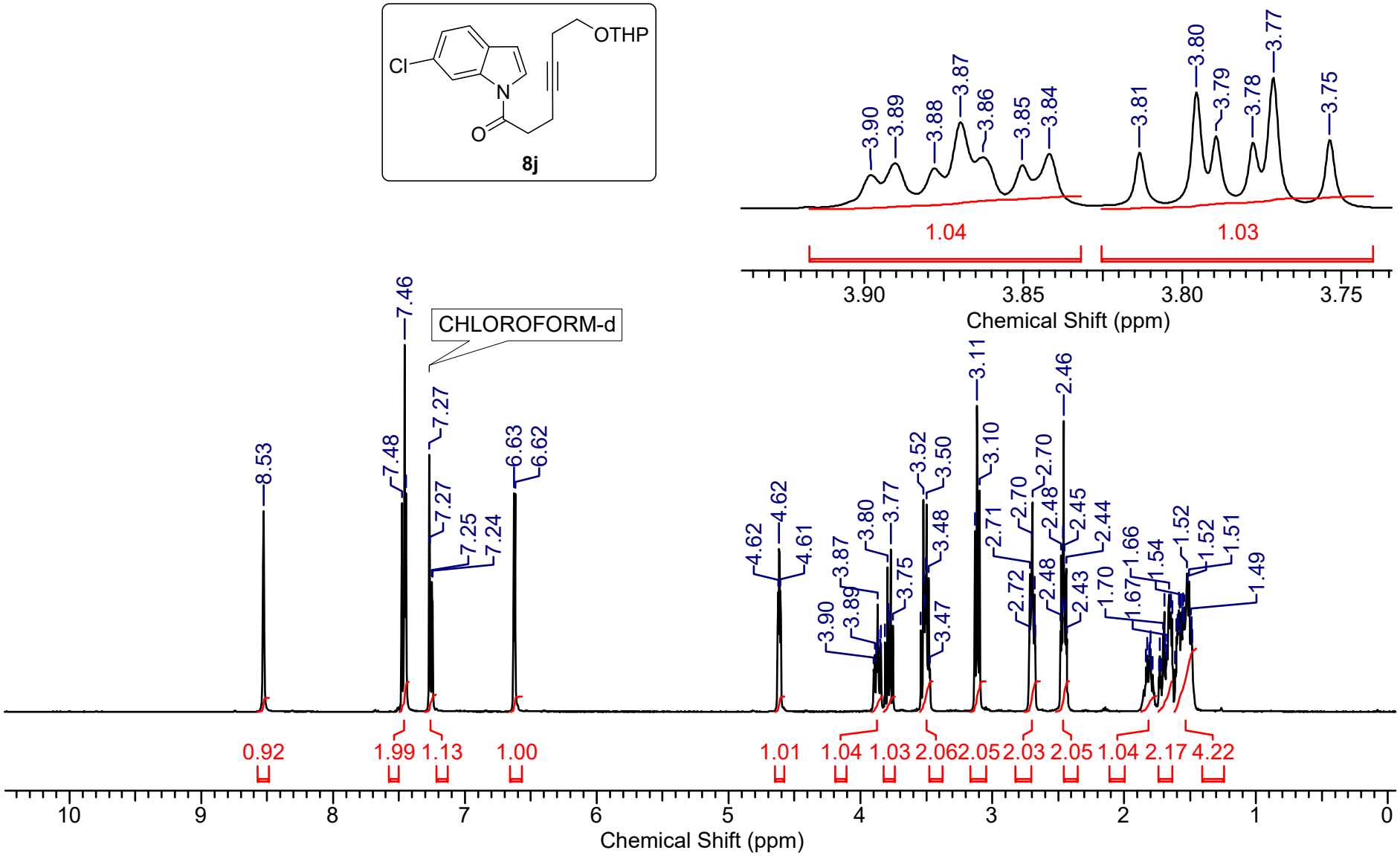

F
N
O
OTHP
8i
124.59
121.38
121.29
112.09
109.04
104.21
98.78
65.92
62.27
35.24
30.53
20.18
19.44
14.23
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

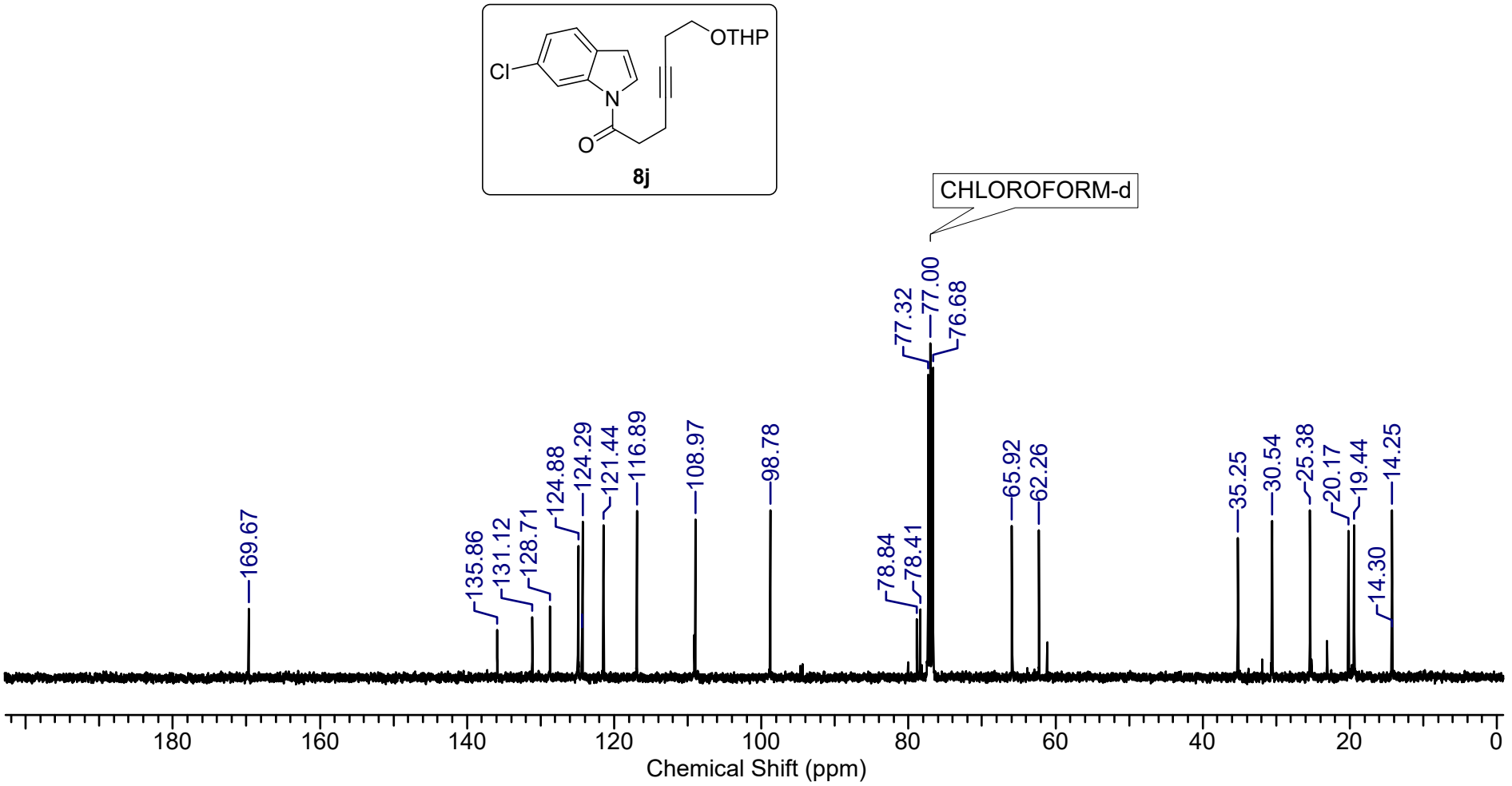


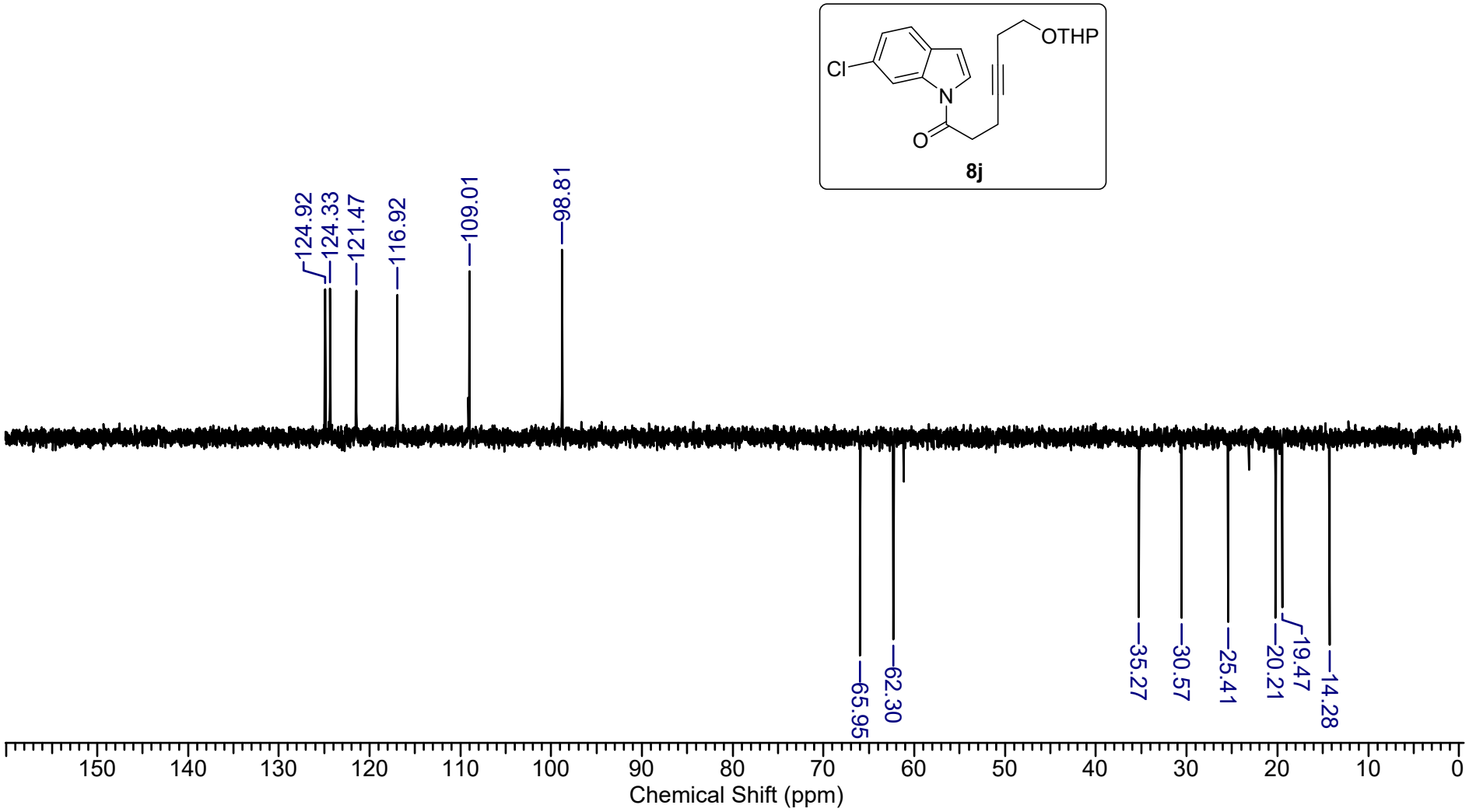
8i

MSH-4a #367 RT: 1.99 AV: 1 NL: 8.95E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

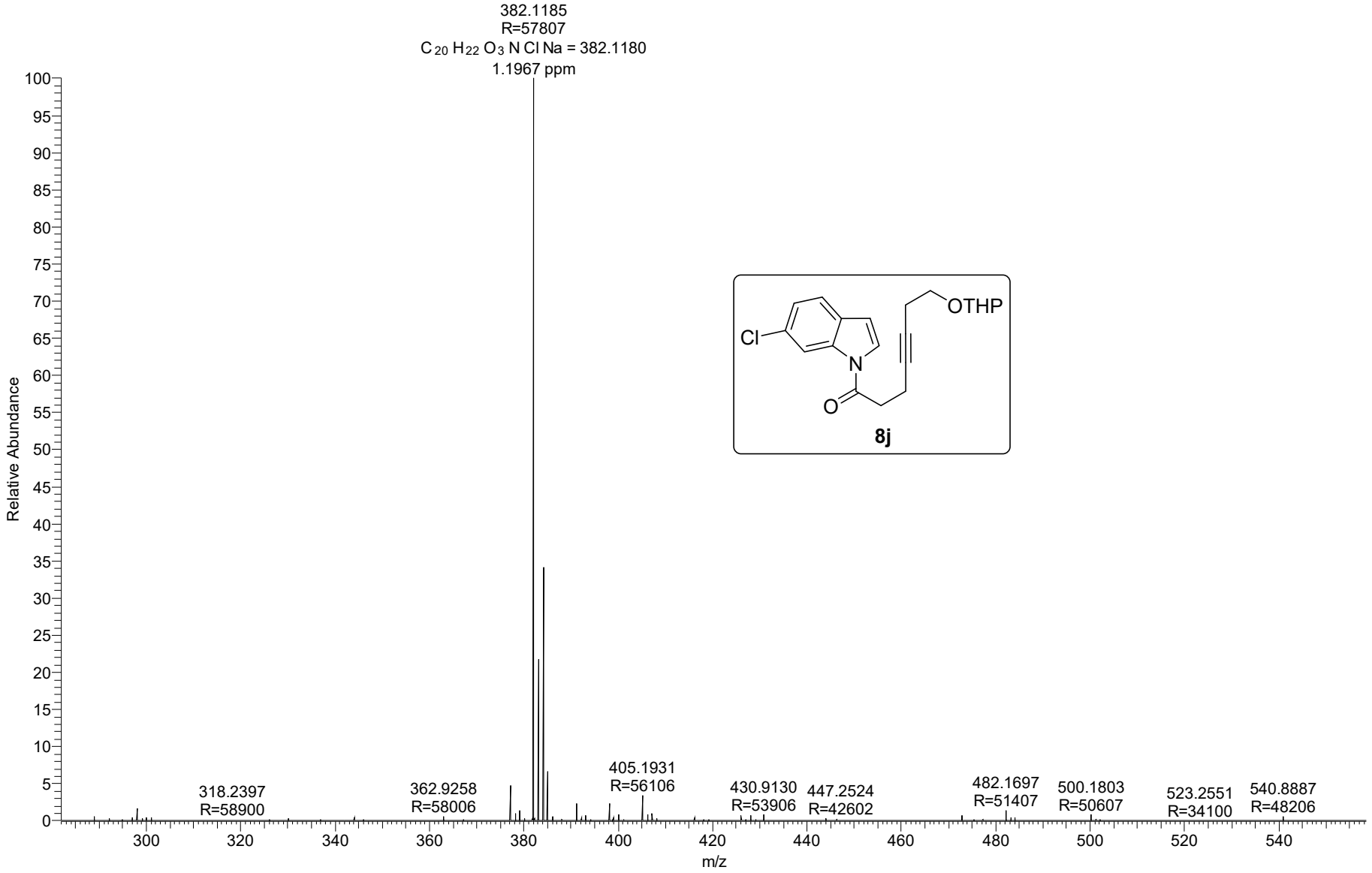


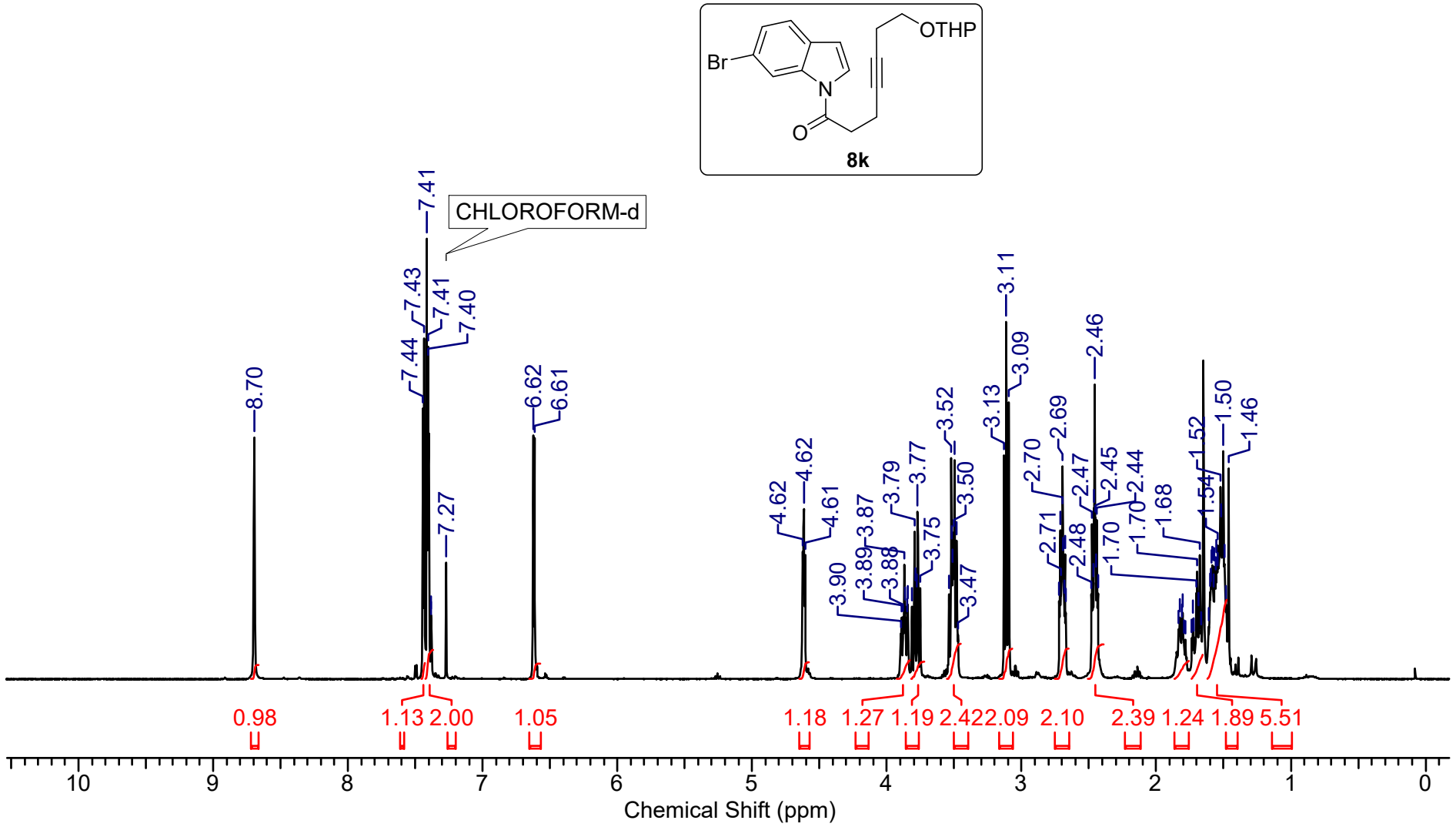

Cl
N
O
OTHP
8j
CHLOROFORM-d
Chemical Shift (ppm)
Chemical Shift (ppm)

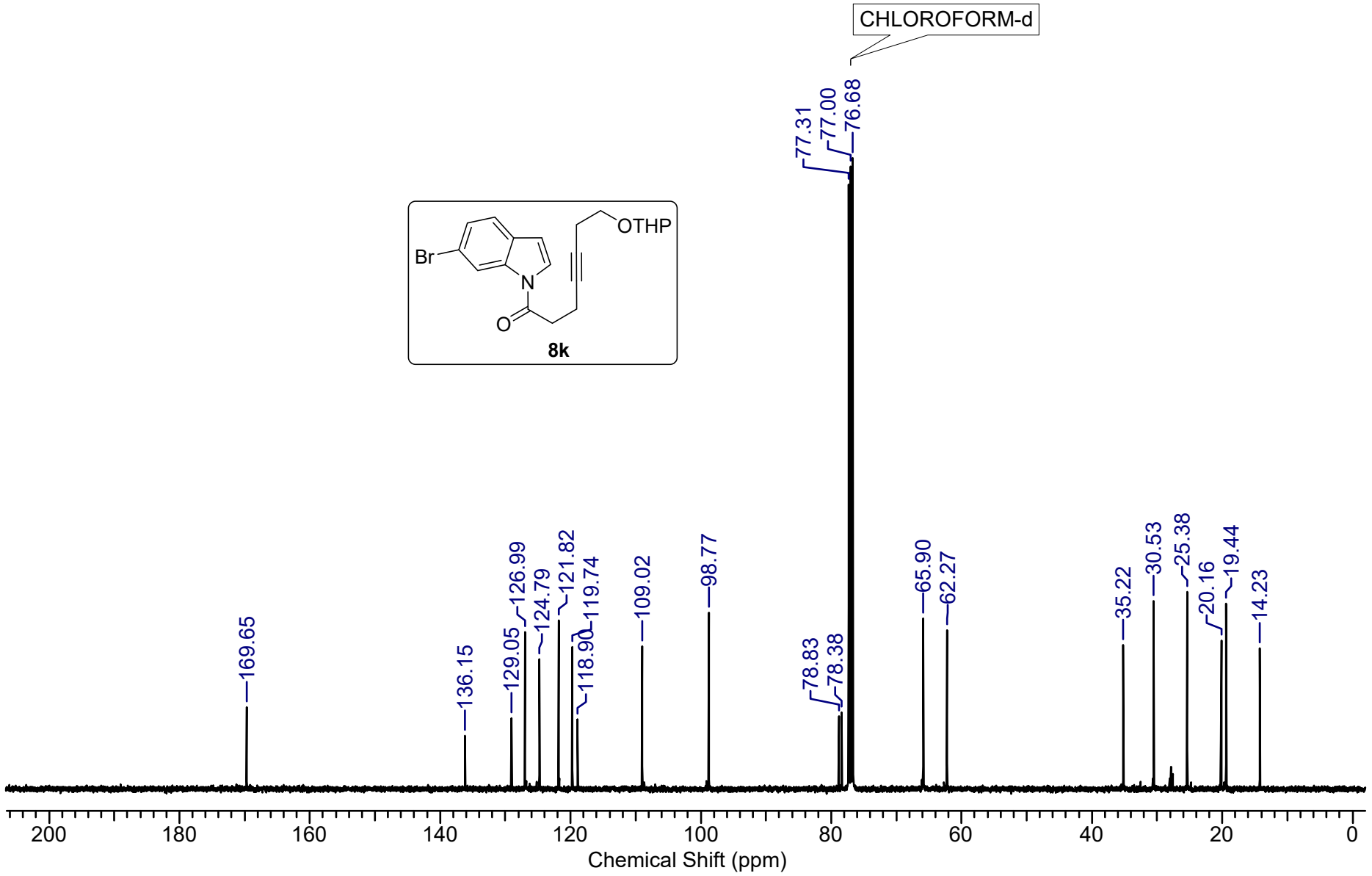

Cl
N
O
OTHP
8j
CHLOROFORM-d
169.67
135.86
131.12
128.71
124.88
124.29
121.44
116.89
108.97
98.78
78.84
78.41
77.32
77.00
76.68
65.92
62.26
35.25
30.54
25.38
20.17
19.44
14.30
14.25
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

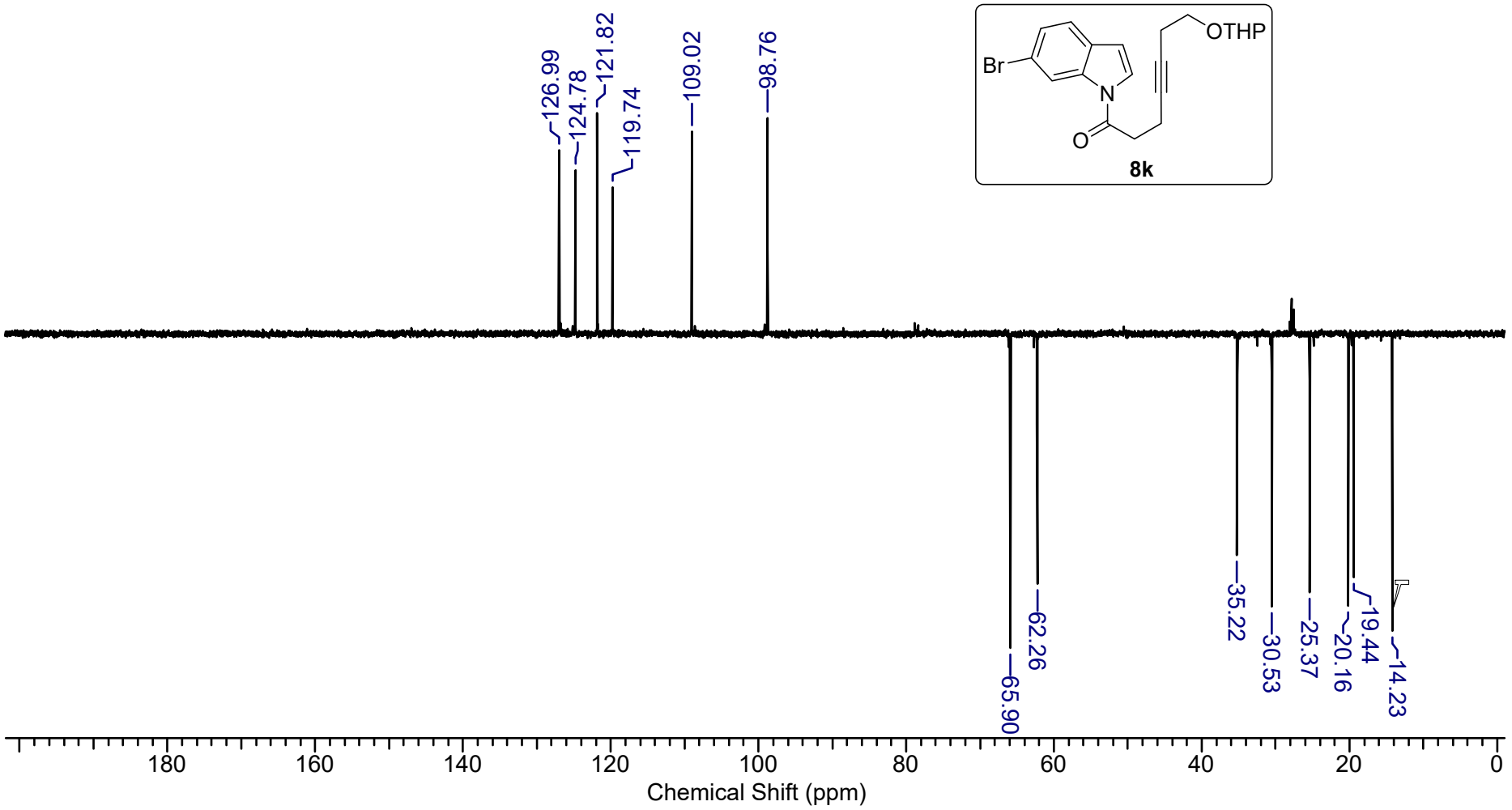

Cl
N
O
OTHP
8j
124.92
124.33
121.47
116.92
109.01
98.81
65.95
62.30
35.27
30.57
25.41
20.21
19.47
14.28
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

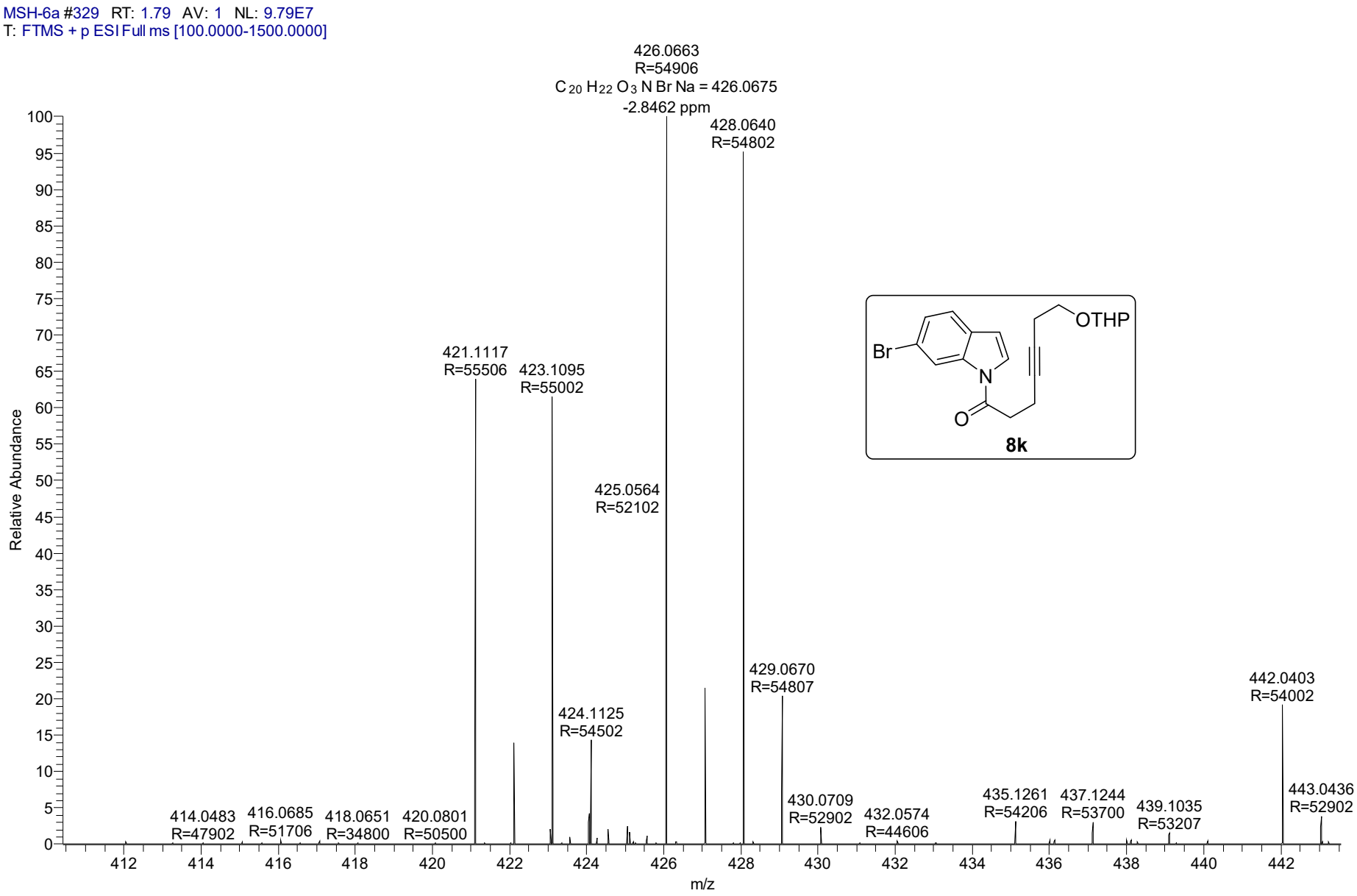
MSH-51 #337 RT: 1.83 AV: 1 NL: 6.15E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

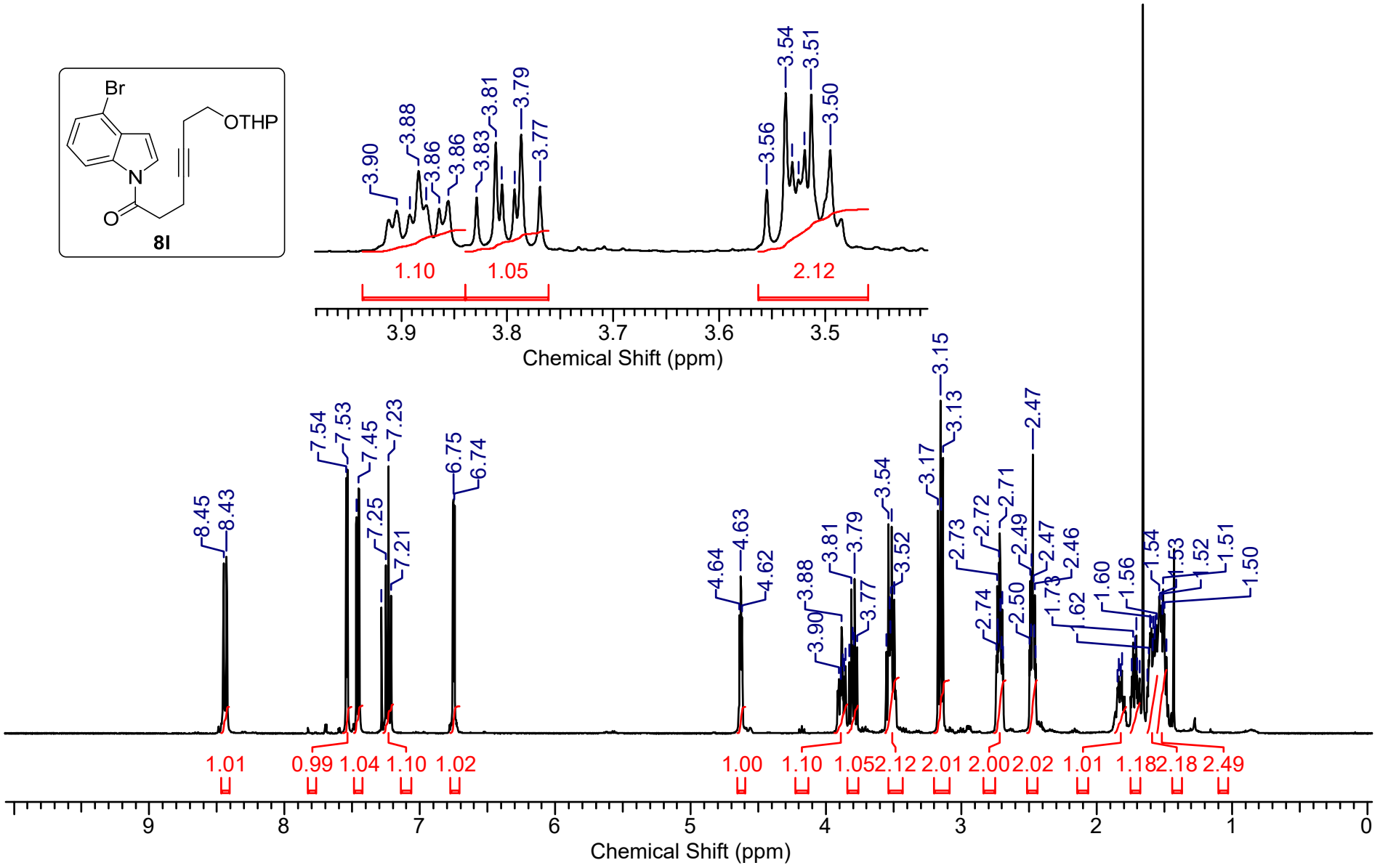


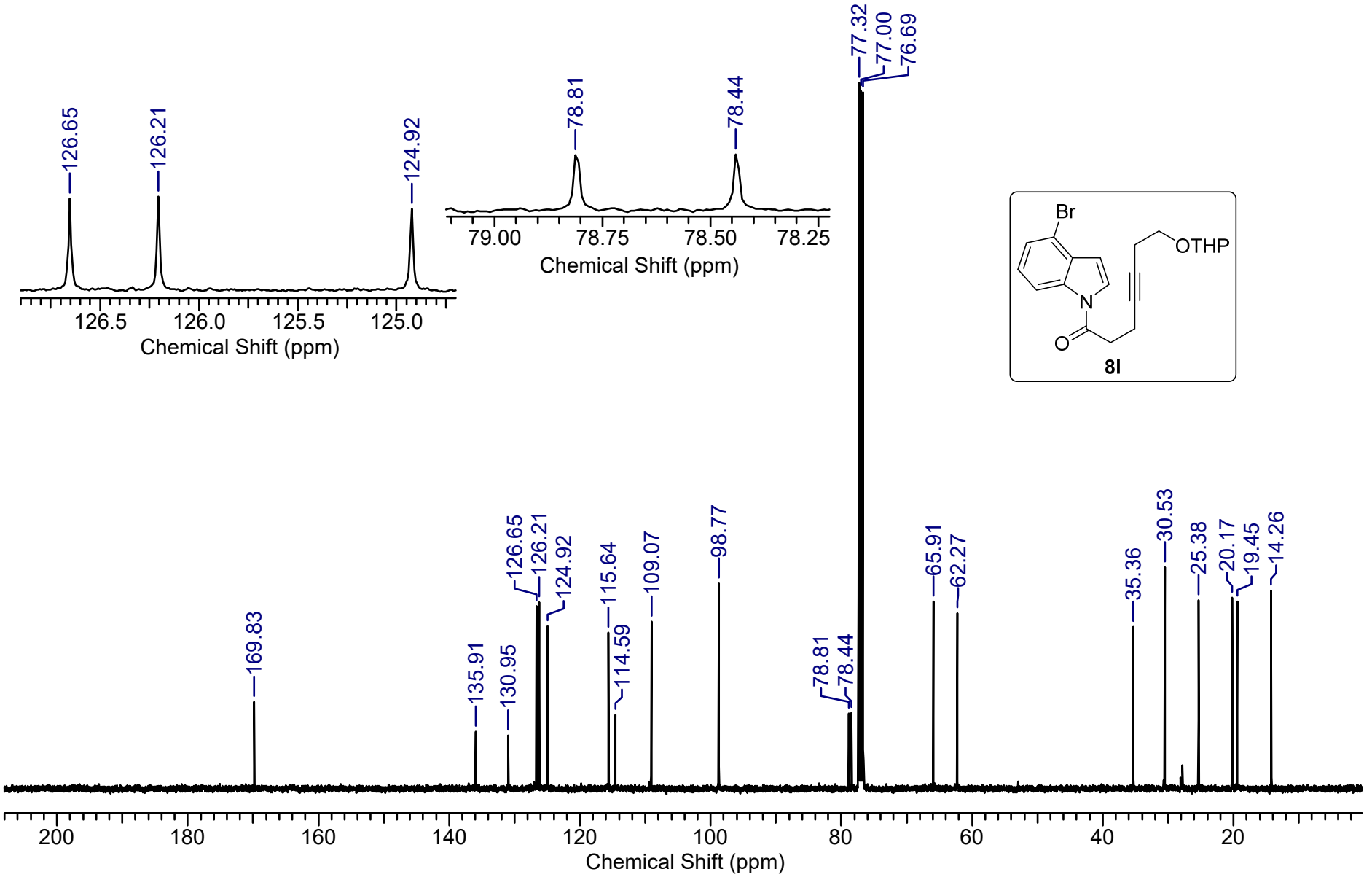

Br
N
O
OTHP
8k
CHLOROFORM-d
8.70
7.44
7.43
7.41
7.40
7.27
6.62
6.61
4.62
4.61
3.90
3.89
3.88
3.87
3.79
3.77
3.75
3.52
3.50
3.47
3.13
3.11
3.09
2.71
2.70
2.69
2.48
2.47
2.46
2.45
2.44
1.70
1.68
1.54
1.52
1.50
1.46
0.98
1.13
2.00
1.05
1.18
1.27
1.19
2.42
2.09
2.10
2.39
1.24
1.89
5.51
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

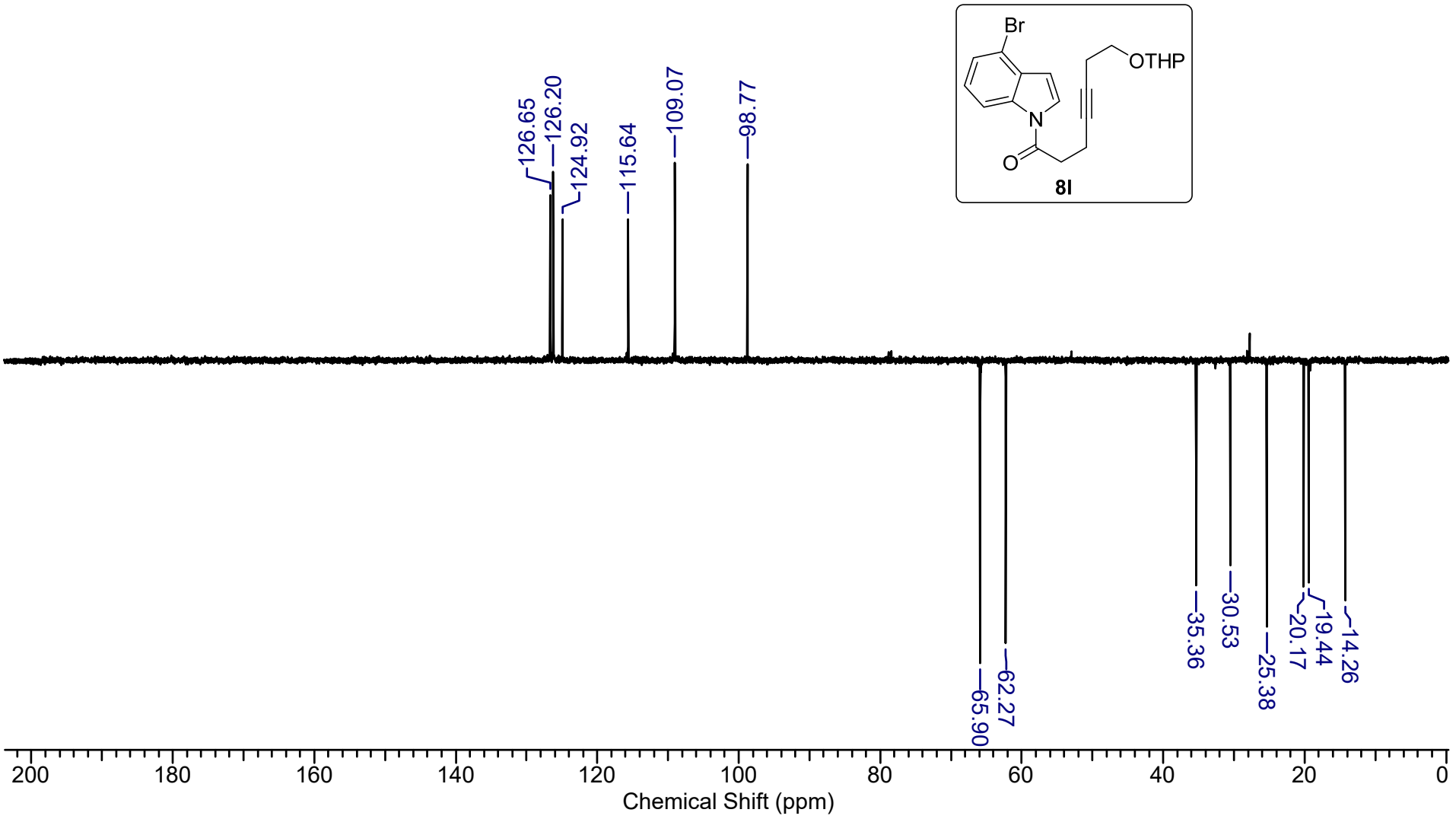

CHLOROFORM-d
77.31
77.00
76.68
OTHP
Br
N
O
8k
169.65
136.15
129.05
126.99
124.79
121.82
119.74
118.90
109.02
98.77
78.83
78.38
65.90
62.27
35.22
30.53
25.38
20.16
19.44
14.23
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


126.99
124.78
121.82
119.74
109.02
98.76
65.90
62.26
35.22
30.53
25.37
20.16
19.44
14.23
Br
N
O
OTHP
8k
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

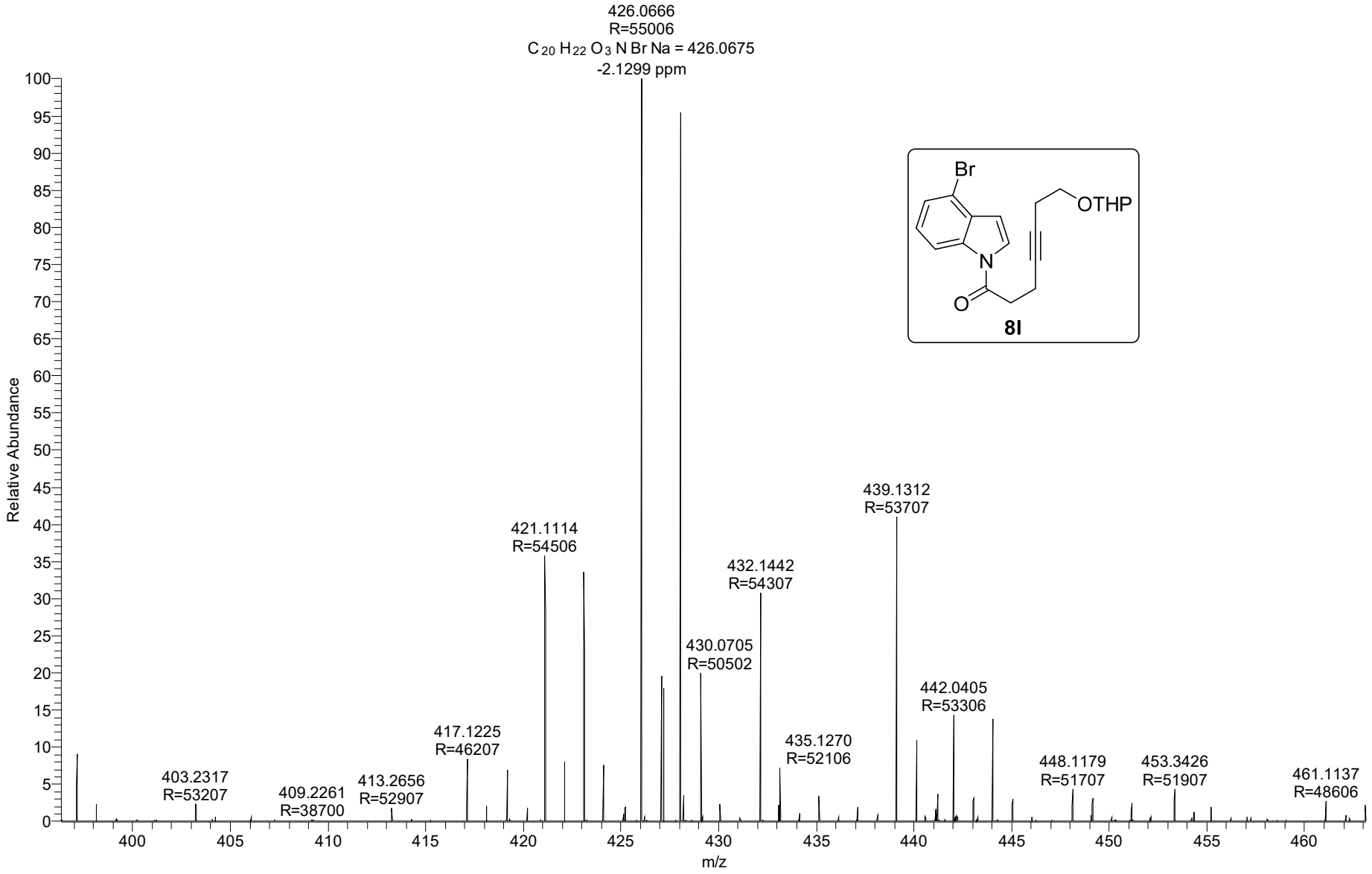

MSH-6a #329 RT: 1.79 AV: 1 NL: 9.79E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
426.0663
R=54906
C 20 H22 O3 N Br Na = 426.0675
-2.8462 ppm
428.0640
R=54802
421.1117
R=55506
423.1095
R=55002
425.0564
R=52102
424.1125
R=54502
429.0670
R=54807
442.0403
R=54002
443.0436
R=52902
414.0483
R=47902
416.0685
R=51706
418.0651
R=34800
420.0801
R=50500
430.0709
R=52902
432.0574
R=44606
435.1261
R=54206
437.1244
R=53700
439.1035
R=53207
Br
N
O
OTHP
8k
Relative Abundance
m/z

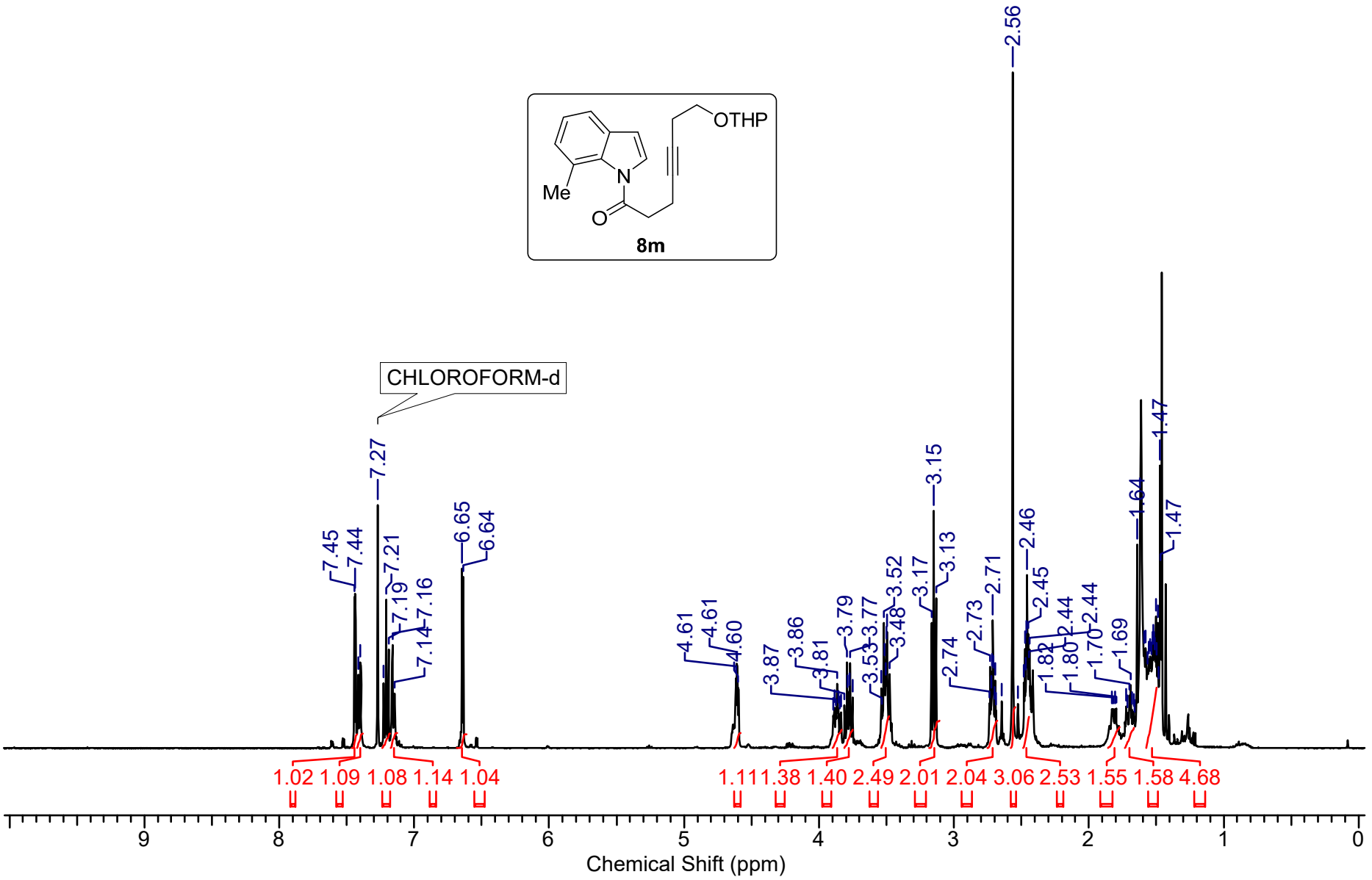

Br
OTHP
N
O
8l
3.90
3.88
3.86
3.86
3.83
3.81
3.79
3.77
3.56
3.54
3.51
3.50
1.10
1.05
2.12
3.9
3.8
3.7
3.6
3.5
Chemical Shift (ppm)
8.45
8.43
7.54
7.53
7.45
7.25
7.23
7.21
6.75
6.74
4.64
4.63
4.62
3.90
3.88
3.81
3.79
3.77
3.54
3.52
3.17
3.15
3.13
2.74
2.73
2.72
2.71
2.50
2.49
2.47
2.47
2.46
1.73
1.62
1.60
1.56
1.54
1.53
1.52
1.51
1.50
1.01
0.99
1.04
1.10
1.02
1.00
1.10
1.05
2.12
2.01
2.00
2.02
1.01
1.18
2.18
2.49
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

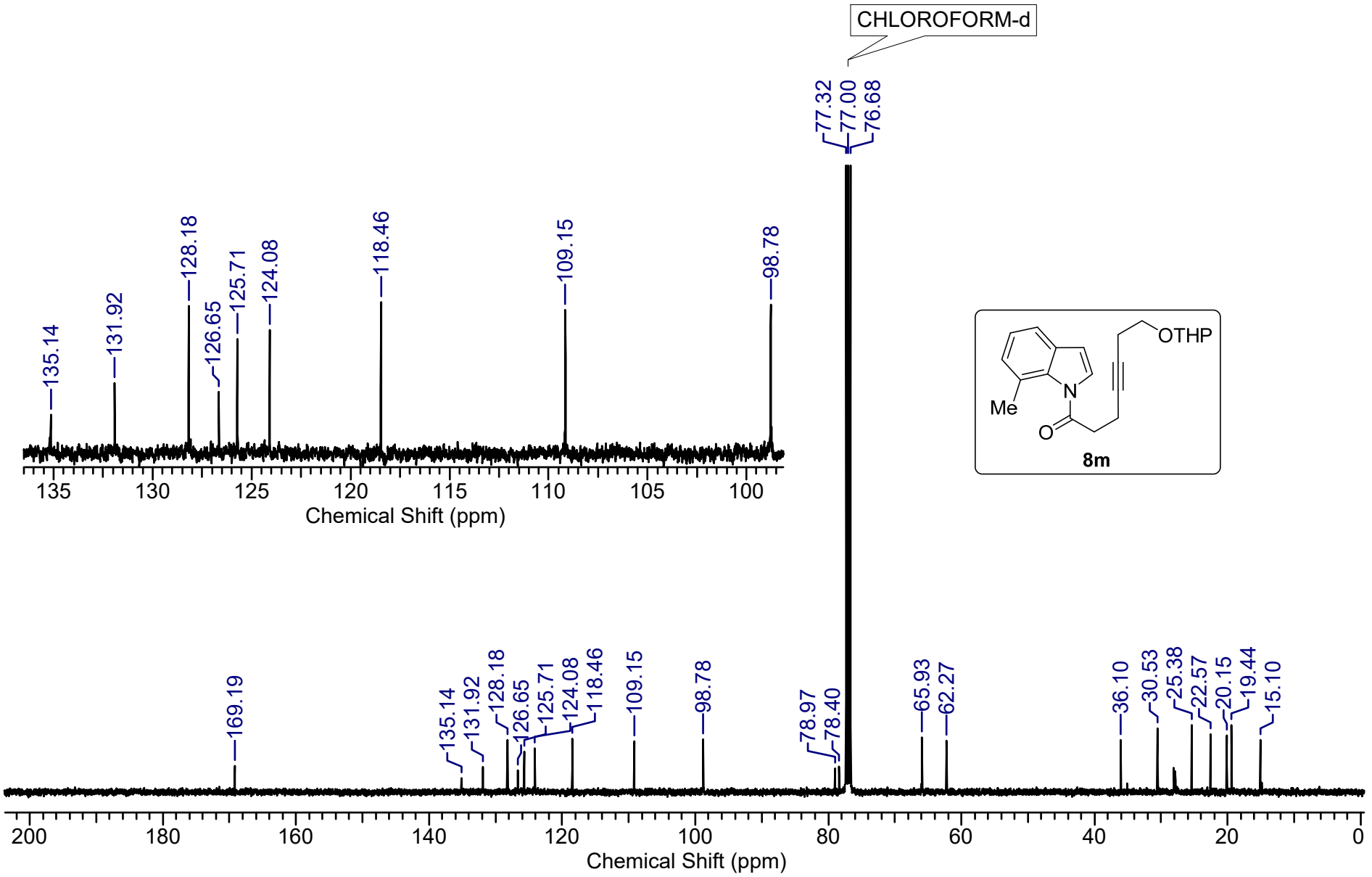

126.65
126.21
124.92
126.5
126.0
125.5
125.0
Chemical Shift (ppm)
78.81
78.44
79.00
78.75
78.50
78.25
Chemical Shift (ppm)
77.32
77.00
76.69
Br
OTHP
N
O
8l
169.83
135.91
130.95
126.65
126.21
124.92
115.64
114.59
109.07
98.77
78.81
78.44
65.91
62.27
35.36
30.53
25.38
20.17
19.45
14.26
200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)

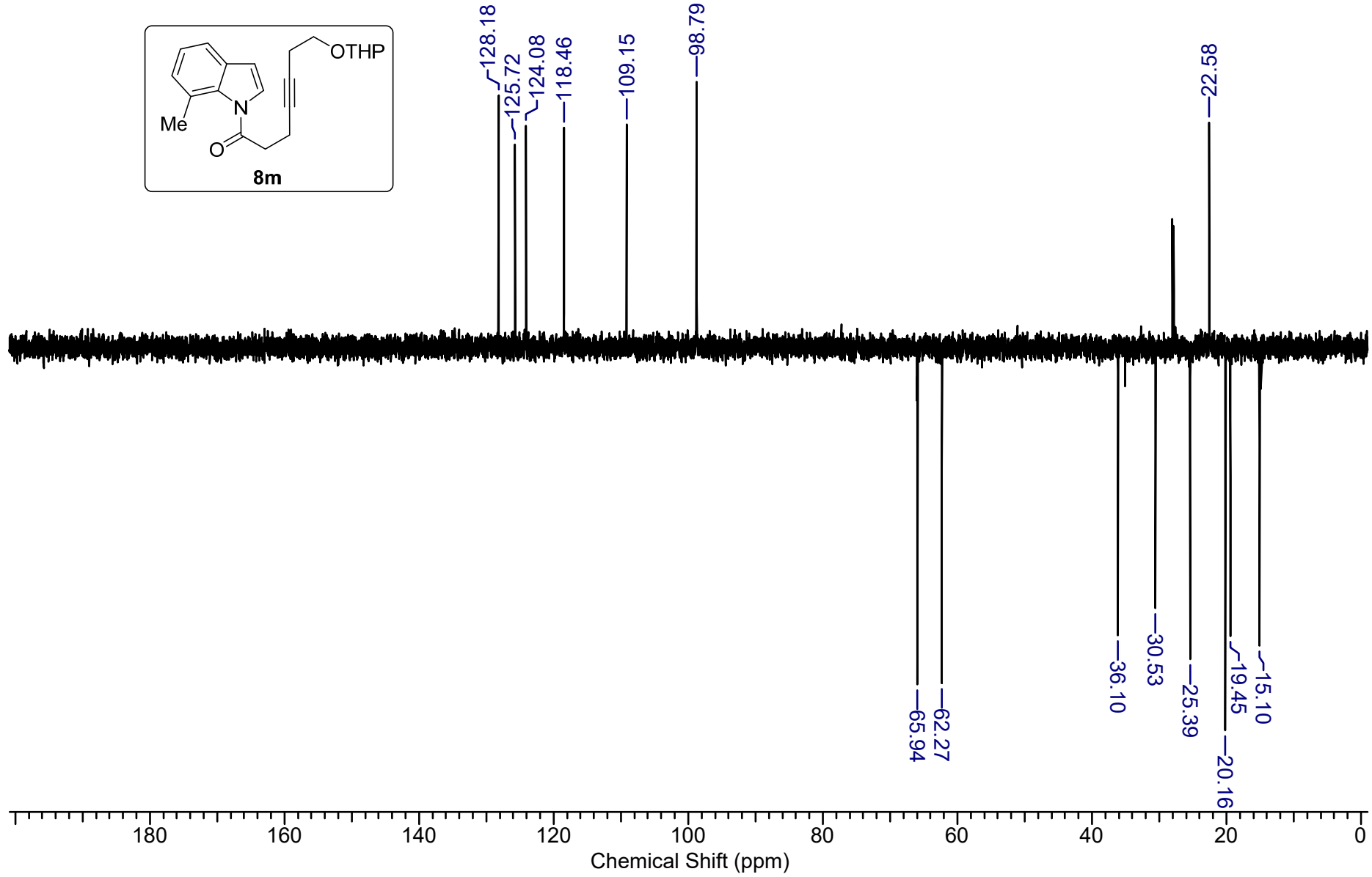

Br
OTHP
N
O
8l
126.65
126.20
124.92
115.64
109.07
98.77
65.90
62.27
35.36
30.53
25.38
20.17
19.44
14.26
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

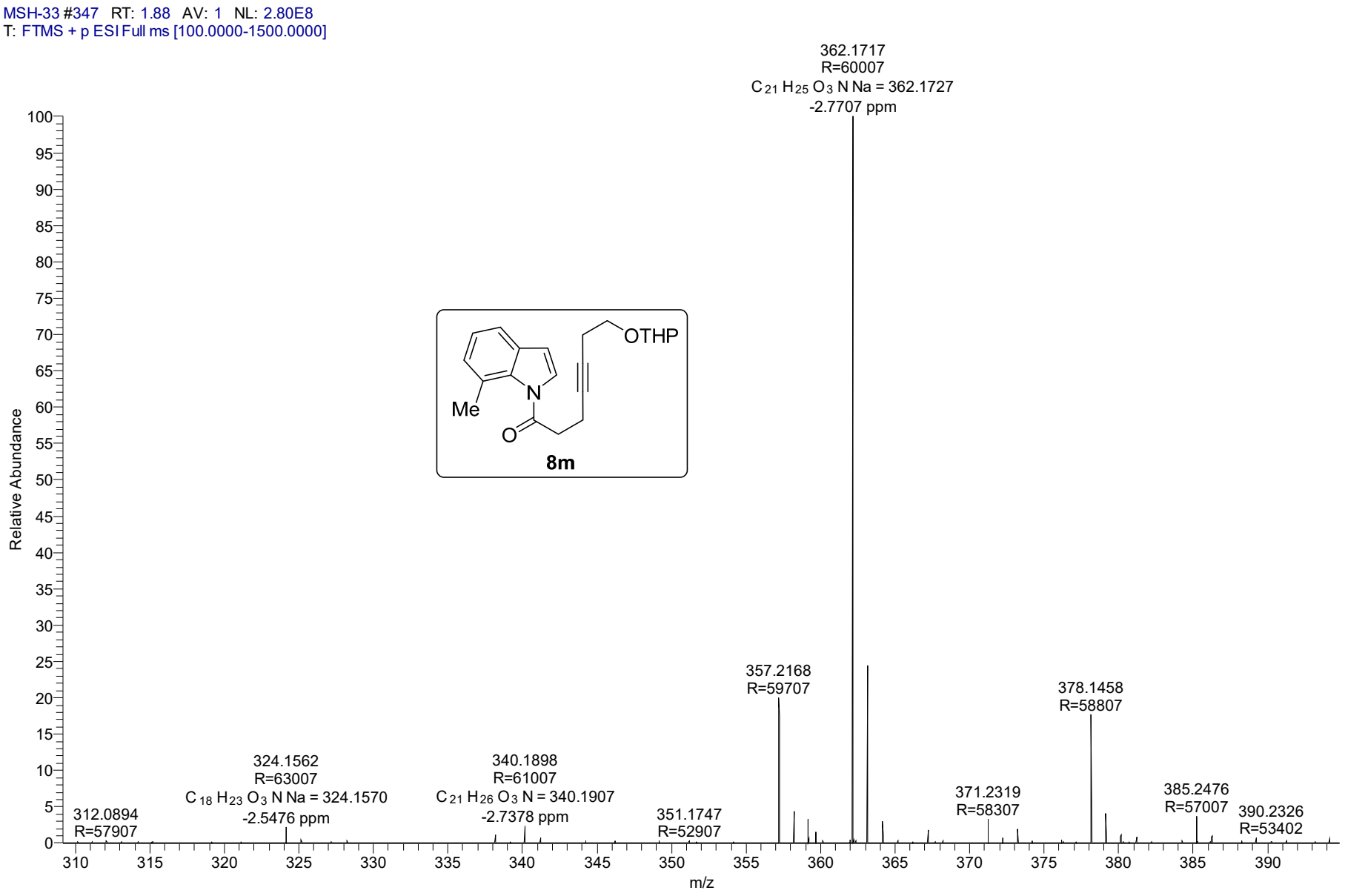
MSH-11 #352 RT: 1.90 AV: 1 NL: 2.06E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

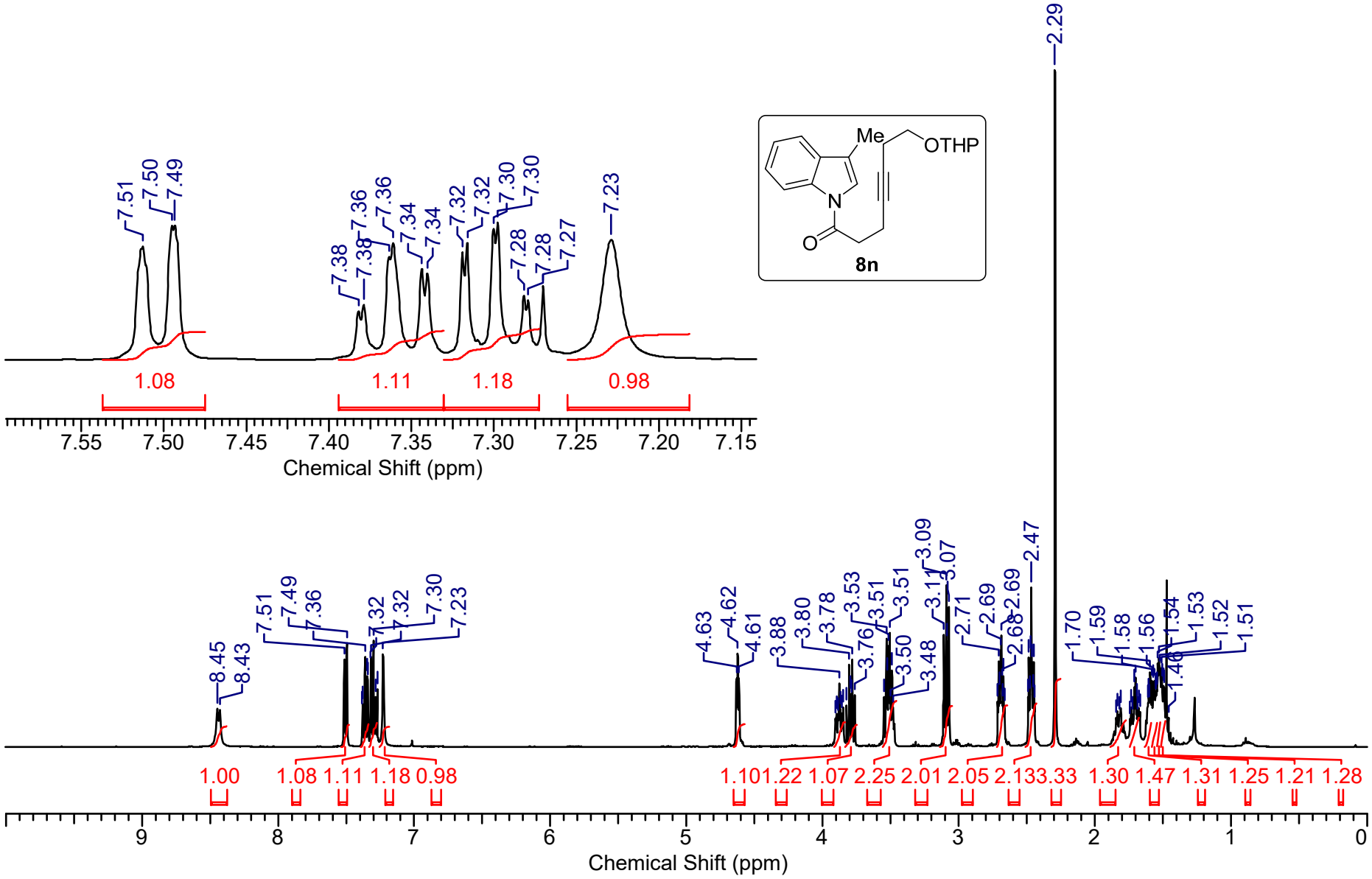


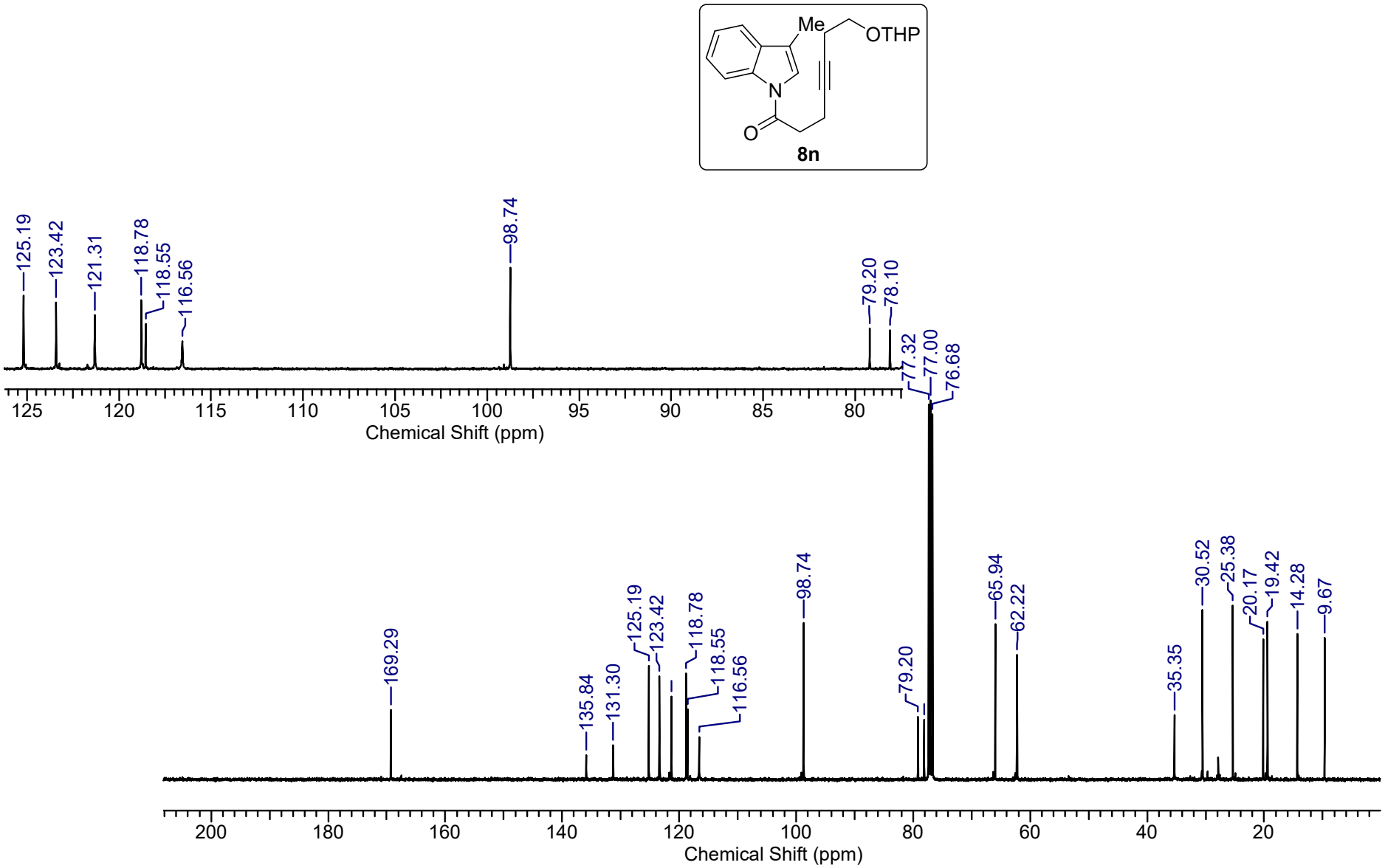

OTHP
Me
N
O
8m
CHLOROFORM-d
2.56
7.27
7.45
7.44
7.21
7.19
7.16
7.14
6.65
6.64
4.61
4.61
4.60
3.87
3.86
3.81
3.79
3.77
3.53
3.52
3.48
3.17
3.15
3.13
2.74
2.73
2.71
2.46
2.45
2.44
2.44
1.82
1.80
1.70
1.69
1.64
1.47
1.47
1.02 1.09 1.08 1.14 1.04
1.11 1.38 1.40 2.49 2.01 2.04 3.06 2.53 1.55 1.58 4.68
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

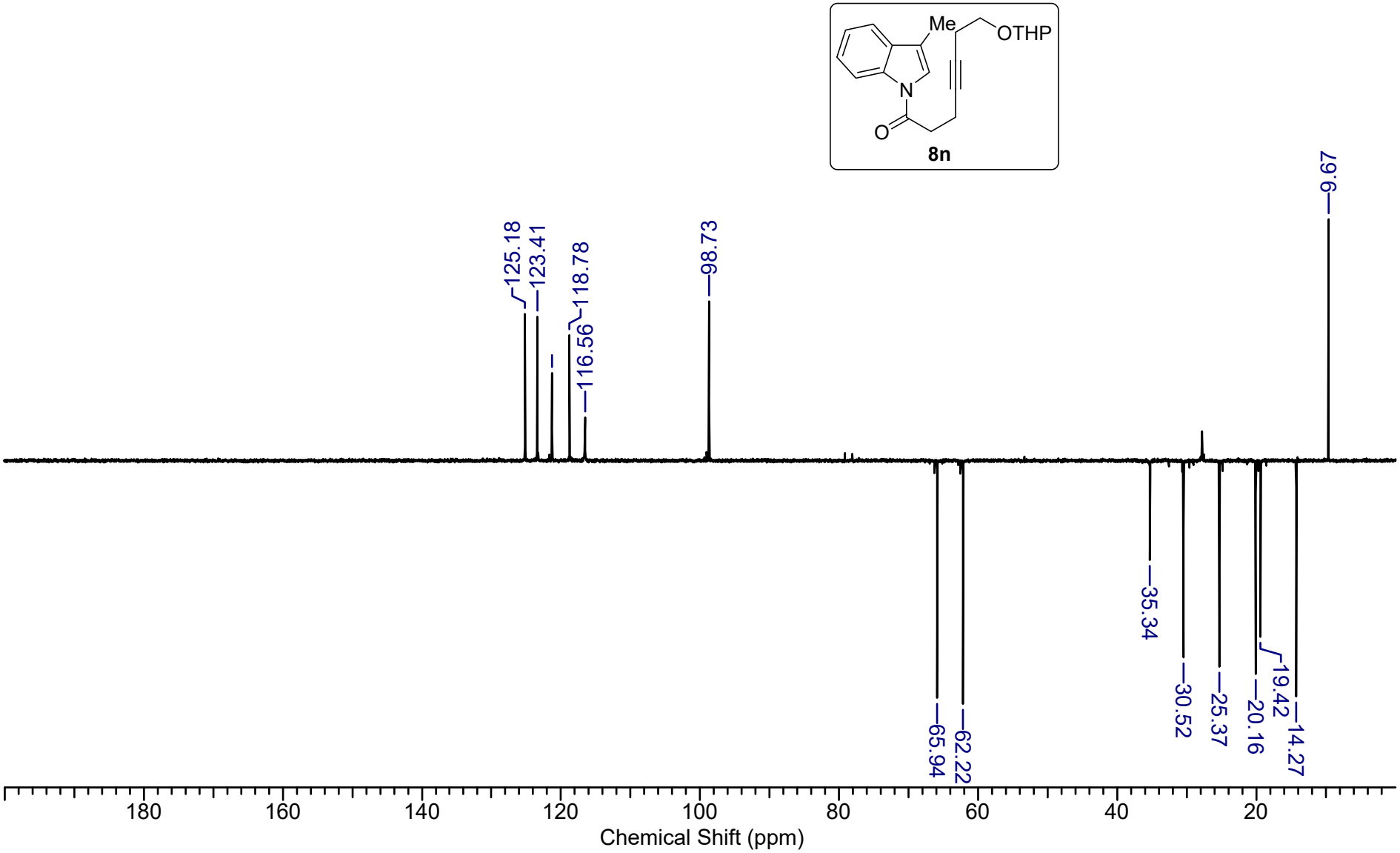

CHLOROFORM-d
77.32
77.00
76.68
135.14
131.92
128.18
126.65
125.71
124.08
118.46
109.15
98.78
169.19
78.97
78.40
65.93
62.27
36.10
30.53
25.38
22.57
20.15
19.44
15.10
OTHP
N
Me
O
8m
Chemical Shift (ppm)
Chemical Shift (ppm)


OTHP
N
Me
O
8m
128.18
125.72
124.08
118.46
109.15
98.79
22.58
65.94
62.27
36.10
30.53
25.39
20.16
19.45
15.10
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

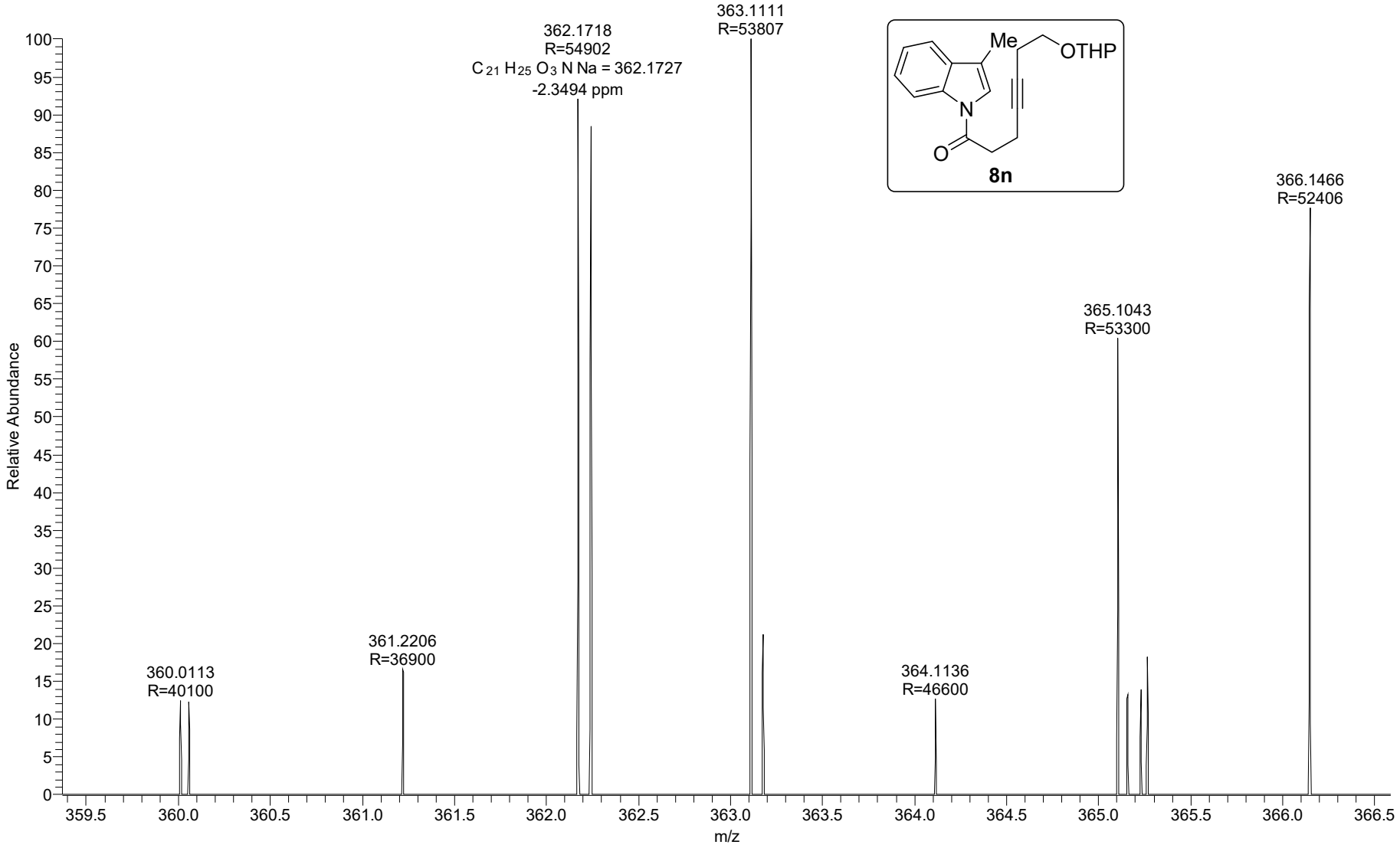

MSH-33 #347 RT: 1.88 AV: 1 NL: 2.80E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
362.1717
R=60007
C21 H25 O3 N Na = 362.1727
-2.7707 ppm
OTHP
Me
N
O
8m
Relative Abundance
m/z
357.2168
R=59707
378.1458
R=58807
324.1562
R=63007
C18 H23 O3 N Na = 324.1570
-2.5476 ppm
340.1898
R=61007
C21 H26 O3 N = 340.1907
-2.7378 ppm
312.0894
R=57907
351.1747
R=52907
371.2319
R=58307
385.2476
R=57007
390.2326
R=53402

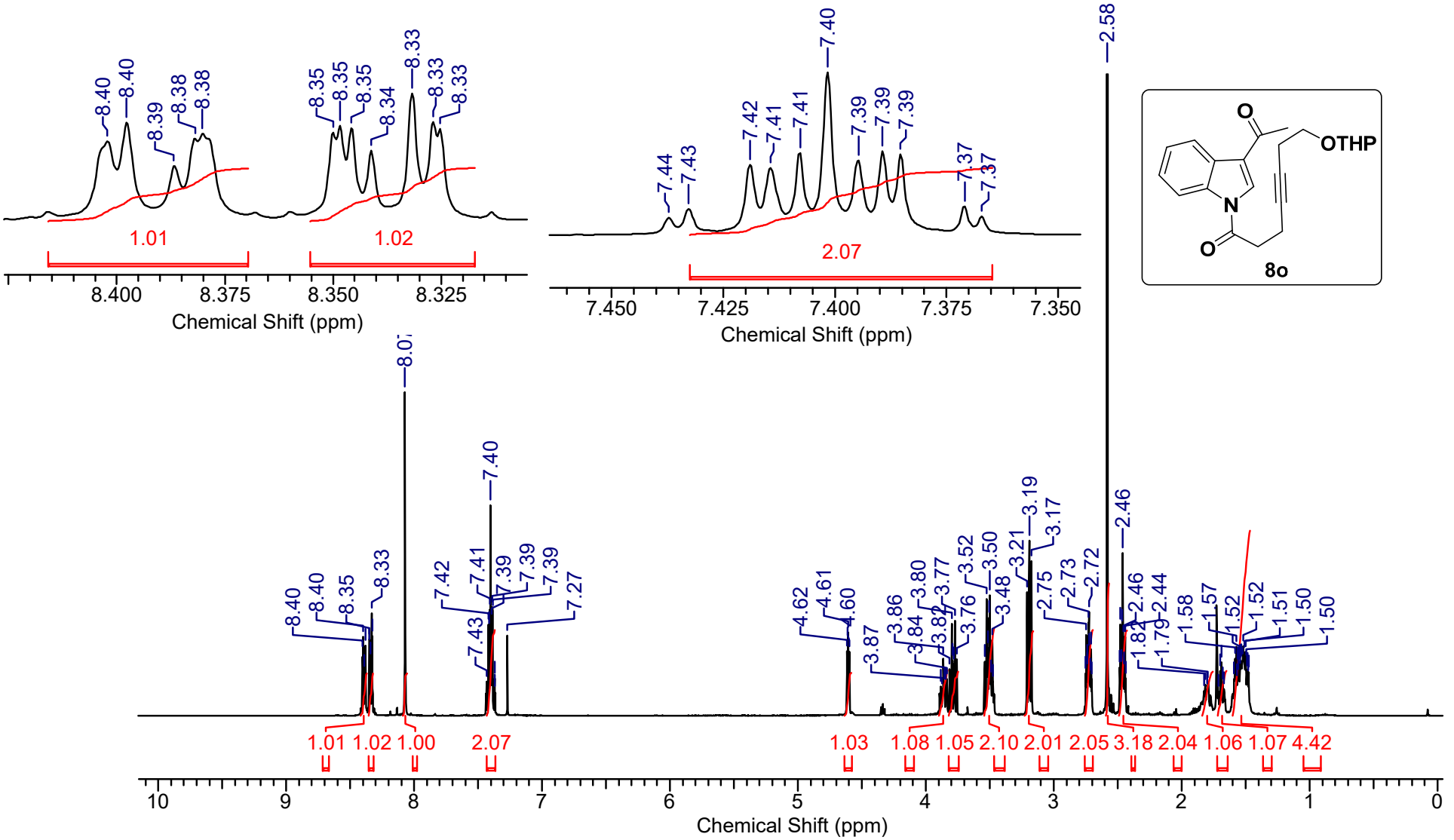

Me
OTHP
N
O
8n
Chemical Shift (ppm)
Chemical Shift (ppm)

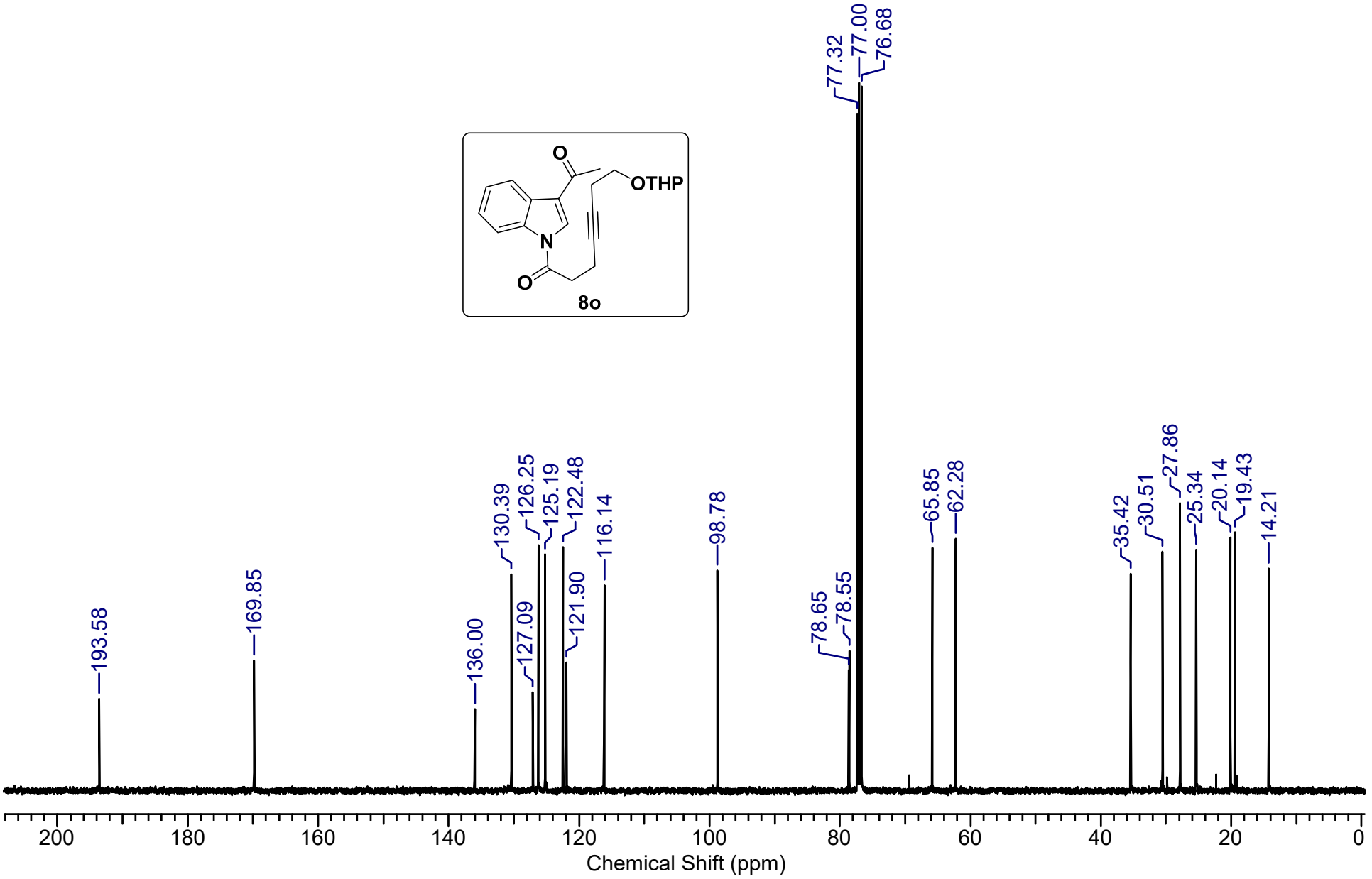

Me
OTHP
N
O
8n
125.19
123.42
121.31
118.78
118.55
116.56
98.74
79.20
78.10
77.32
77.00
76.68
Chemical Shift (ppm)
169.29
135.84
131.30
125.19
123.42
118.78
118.55
116.56
98.74
79.20
65.94
62.22
35.35
30.52
25.38
20.17
19.42
14.28
9.67
Chemical Shift (ppm)

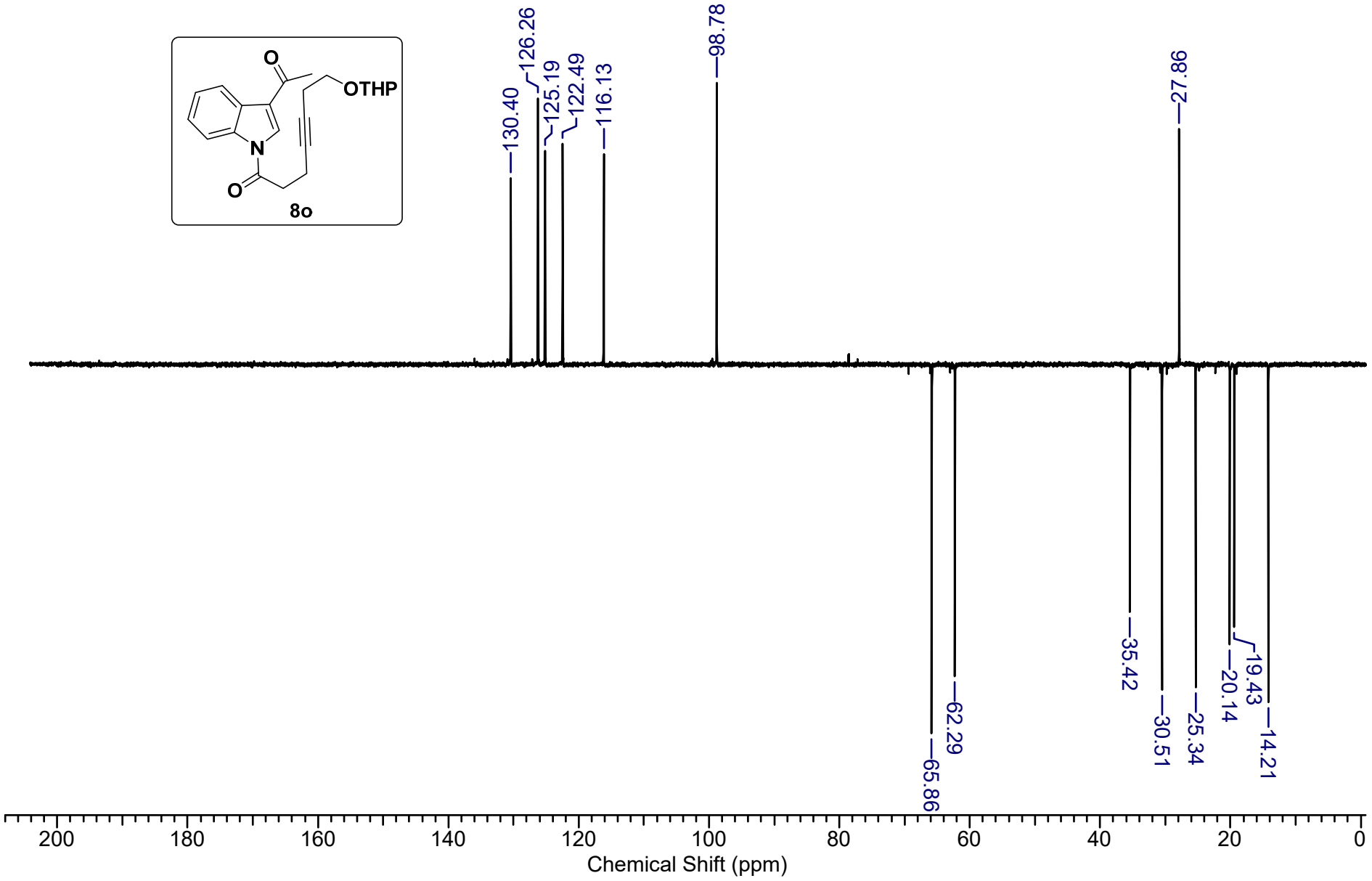

Me
OTHP
N
O
8n
9.67
125.18
123.41
118.78
116.56
98.73
65.94
62.22
35.34
30.52
25.37
20.16
19.42
14.27
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)

MSH-10 #330 RT: 1.79 AV: 1 NL: 1.90E5
T: FTMS + p ESI Full ms [100.0000-1500.0000]

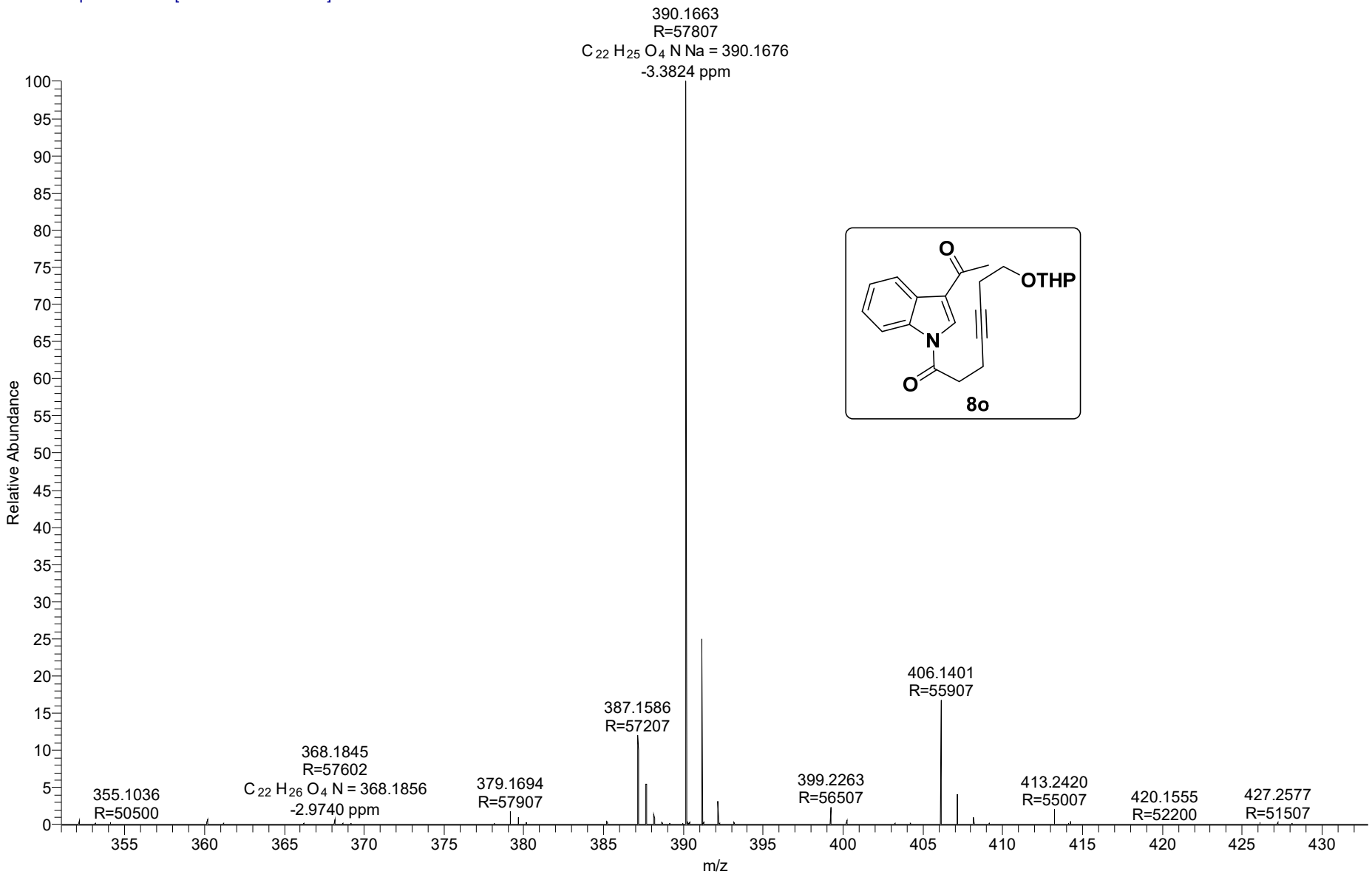


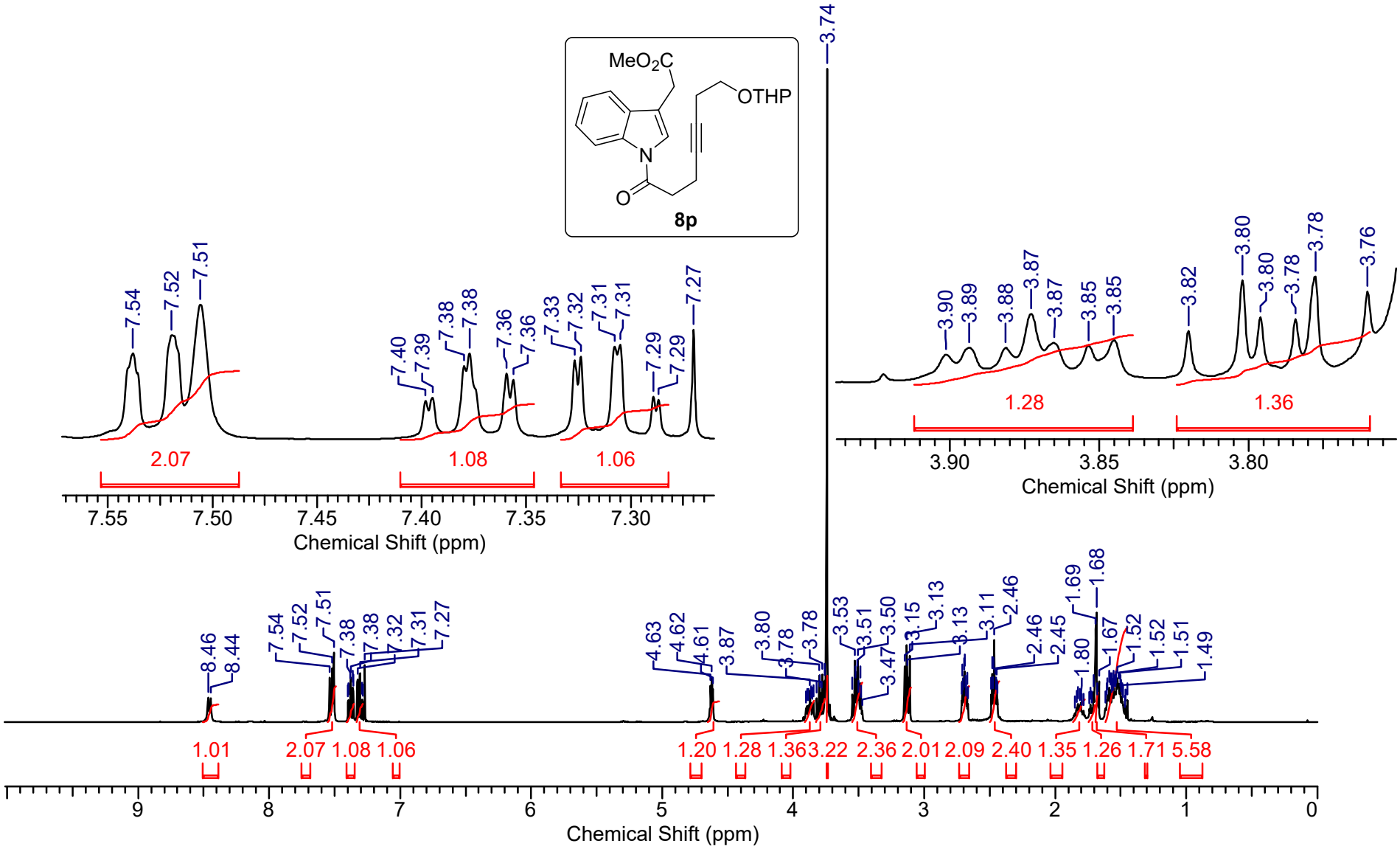

8.40
8.40
8.39
8.38
8.38
8.35
8.35
8.35
8.34
8.33
8.33
8.33
1.01
1.02
8.400
8.375
8.350
8.325
Chemical Shift (ppm)
7.44
7.43
7.42
7.41
7.40
7.39
7.39
7.39
7.37
7.37
2.07
7.450
7.425
7.400
7.375
7.350
Chemical Shift (ppm)
O
OTHP
N
O
8o
8.40
8.40
8.35
8.33
7.42
7.41
7.40
7.39
7.39
7.43
7.27
4.62
4.61
4.60
3.87
3.86
3.84
3.82
3.80
3.77
3.76
3.52
3.50
3.48
3.21
3.19
3.17
2.75
2.73
2.72
2.58
2.46
2.46
2.44
1.82
1.79
1.58
1.57
1.52
1.52
1.51
1.50
1.50
1.01 1.02 1.00 2.07 1.03 1.08 1.05 2.10 2.01 2.05 3.18 2.04 1.06 1.07 4.42
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

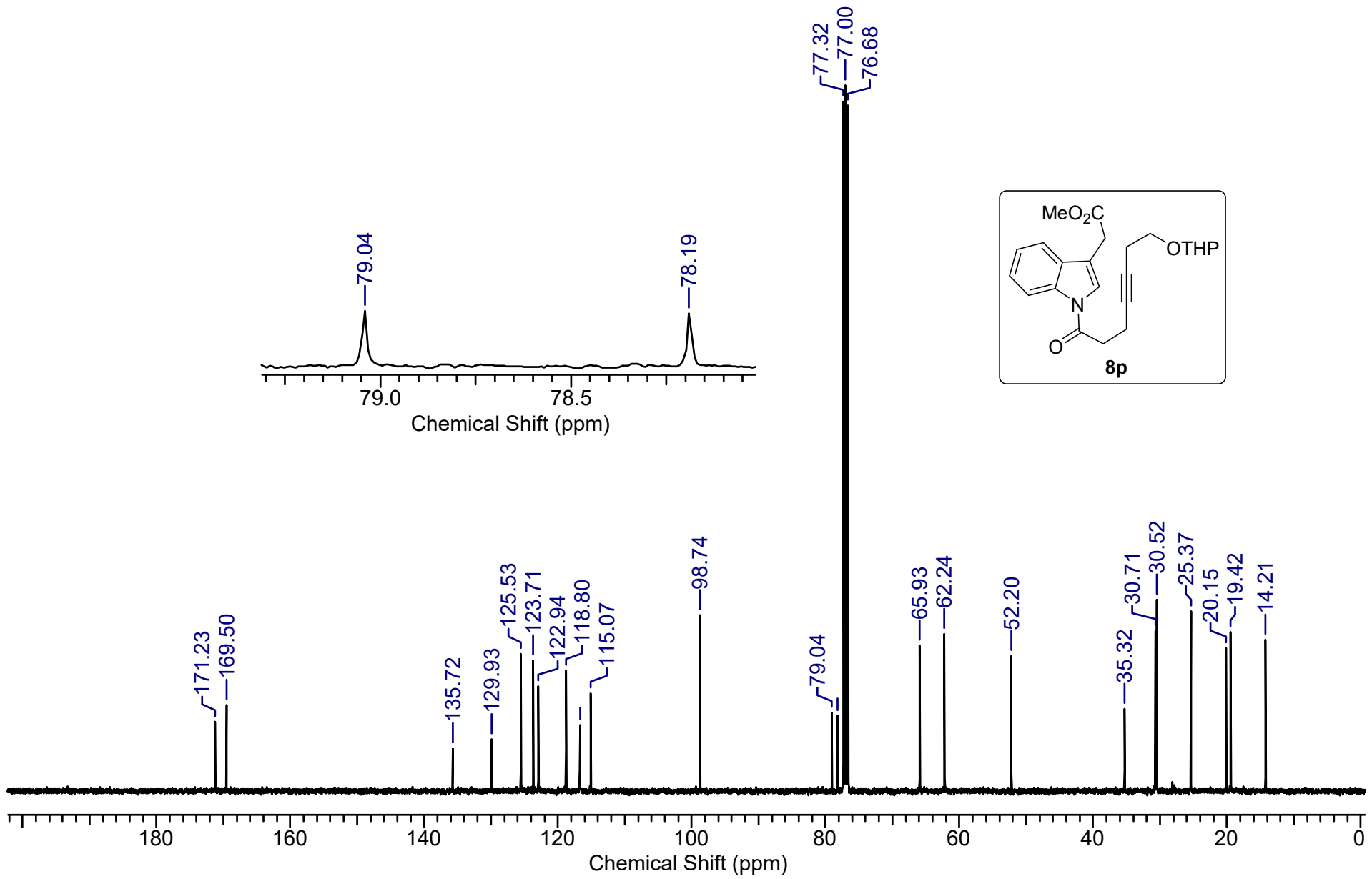

O
OTHP
N
O
8o
193.58
169.85
136.00
130.39
127.09
126.25
125.19
122.48
121.90
116.14
98.78
78.65
78.55
77.32
77.00
76.68
65.85
62.28
35.42
30.51
27.86
25.34
20.14
19.43
14.21
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

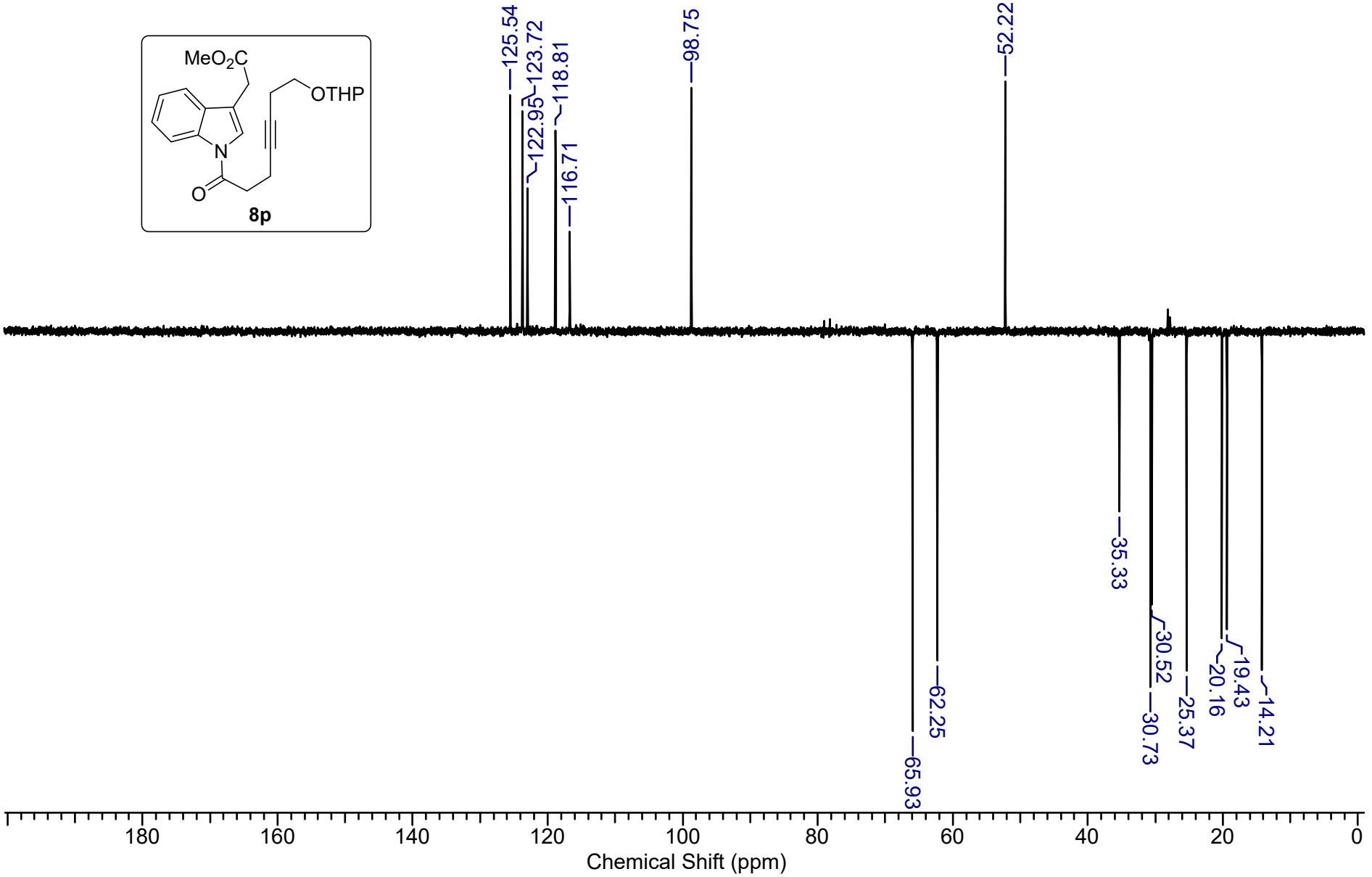

O
OTHP
N
O
8o
130.40
126.26
125.19
122.49
116.13
98.78
27.86
65.86
62.29
35.42
30.51
25.34
20.14
19.43
14.21
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MSH-9 #311 RT: 1.69 AV: 1 NL: 2.61E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

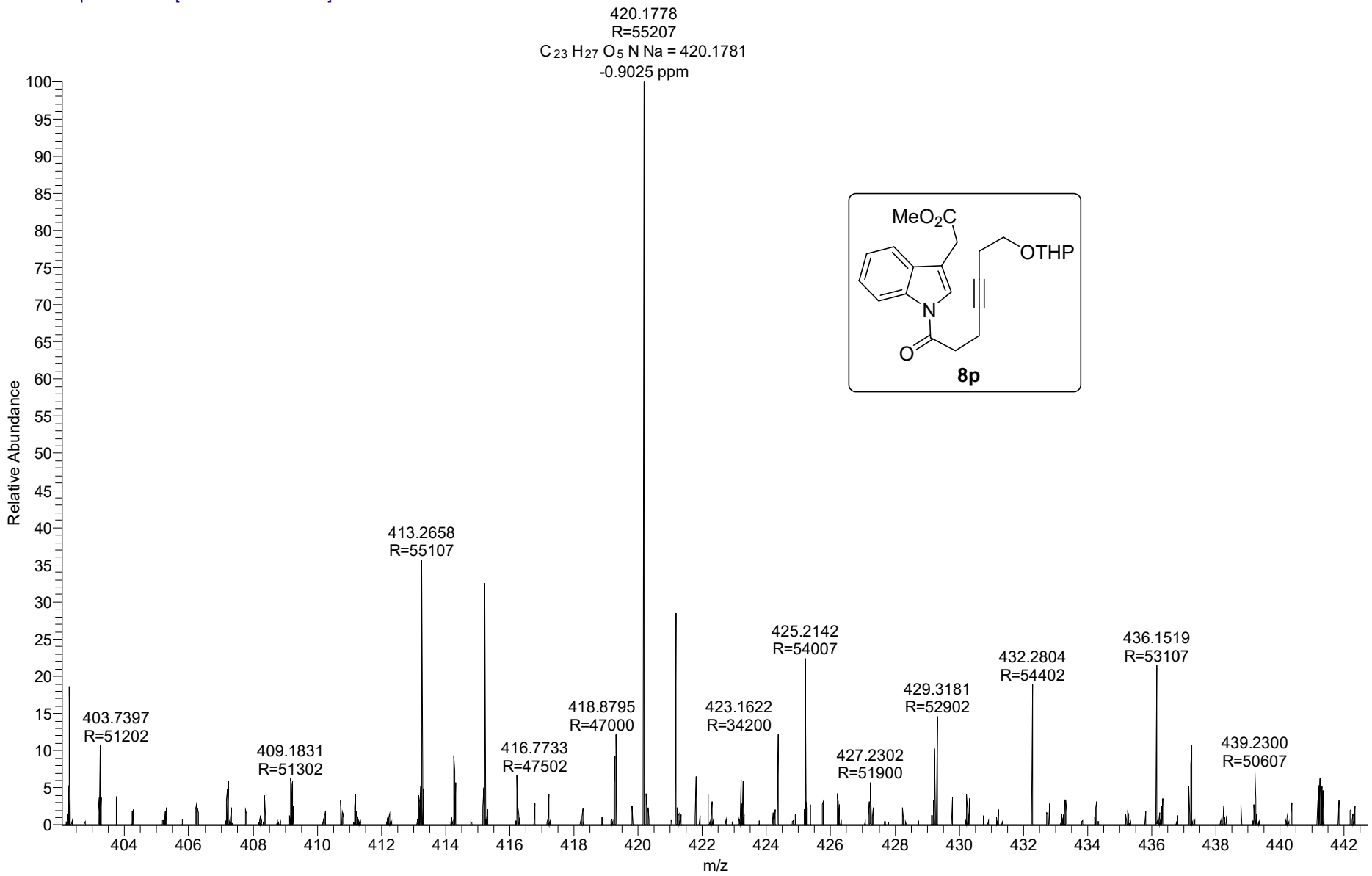


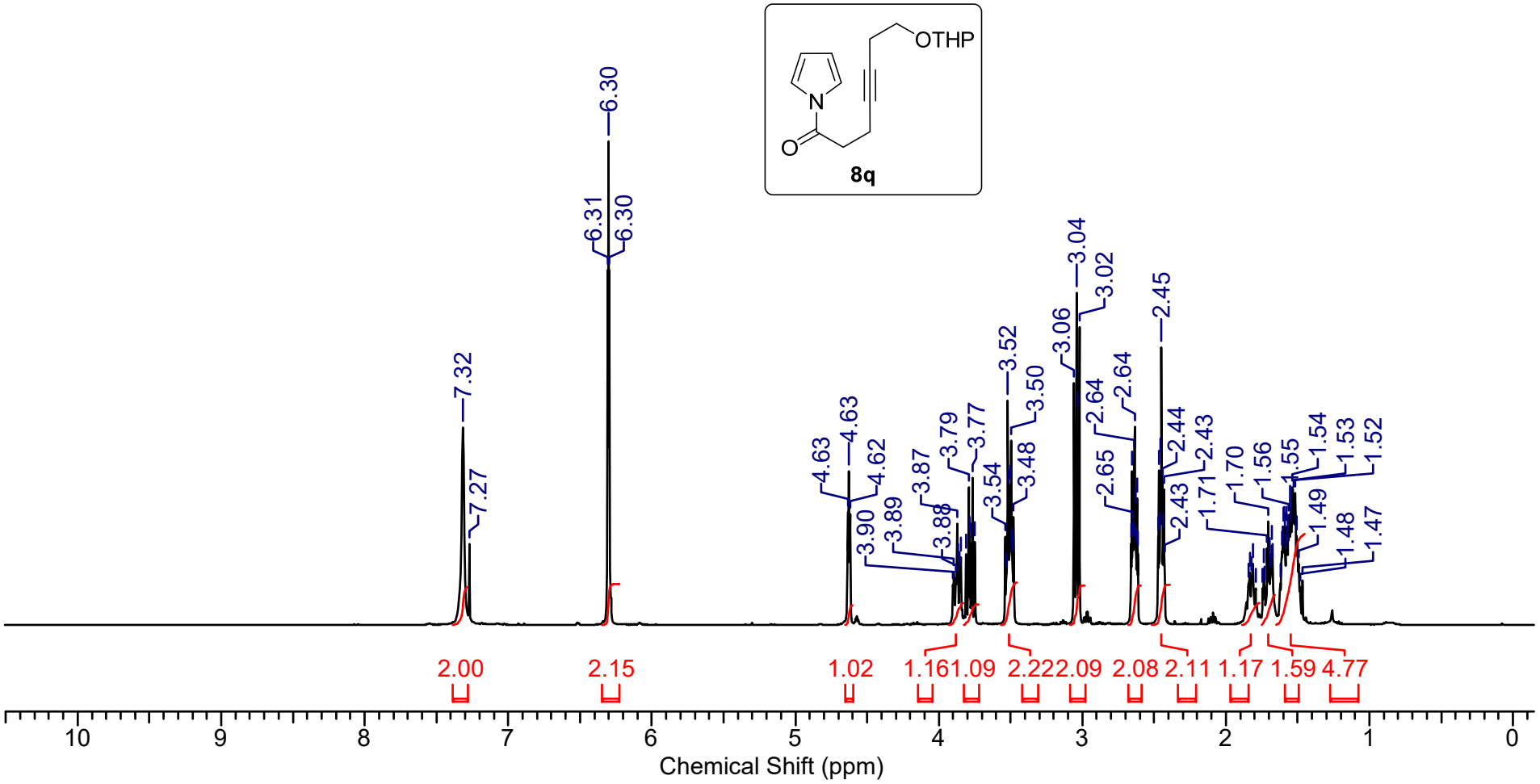

MeO2C
OTHP
N
O
8p
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

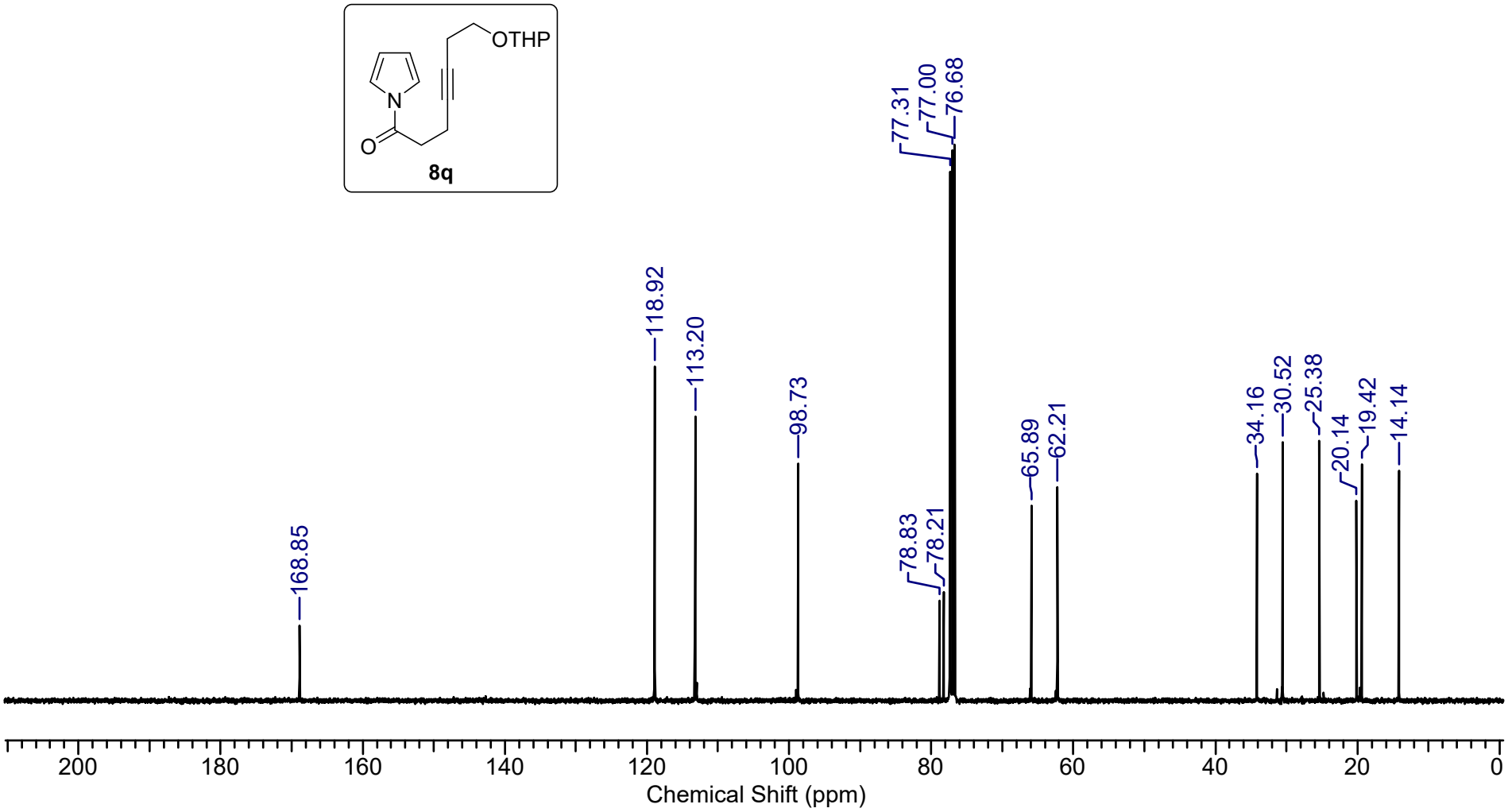

77.32
77.00
76.68
79.04
78.19
79.0
78.5
Chemical Shift (ppm)
MeO2C
OTHP
N
O
8p
171.23
169.50
135.72
129.93
125.53
123.71
122.94
118.80
115.07
98.74
79.04
65.93
62.24
52.20
35.32
30.71
30.52
25.37
20.15
19.42
14.21
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

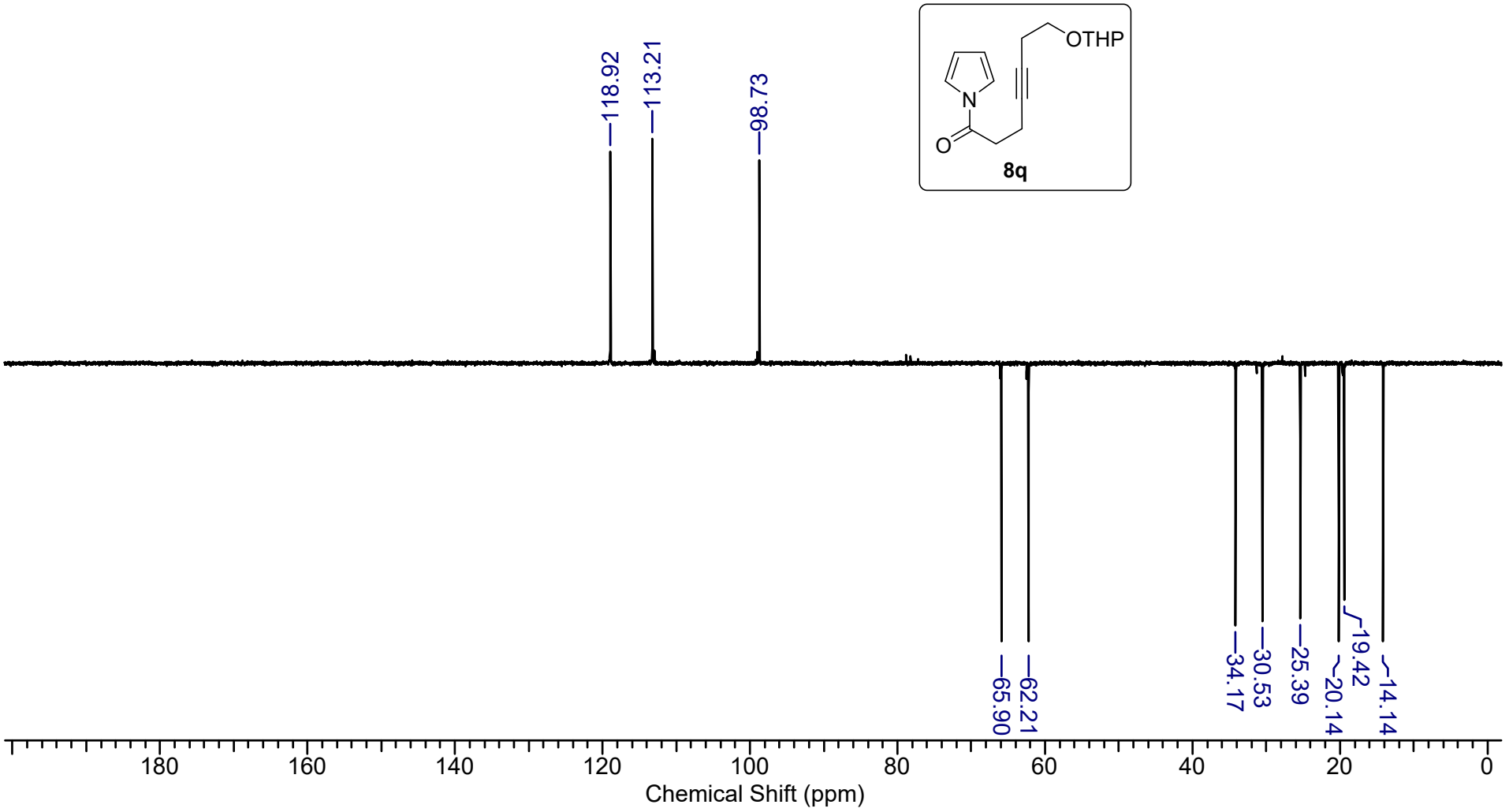

MeO2C
OTHP
N
O
8p
125.54
123.72
122.95
118.81
116.71
98.75
52.22
65.93
62.25
35.33
30.73
30.52
25.37
20.16
19.43
14.21
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MSH-47 #437 RT: 2.37 AV: 1 NL: 3.32E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]

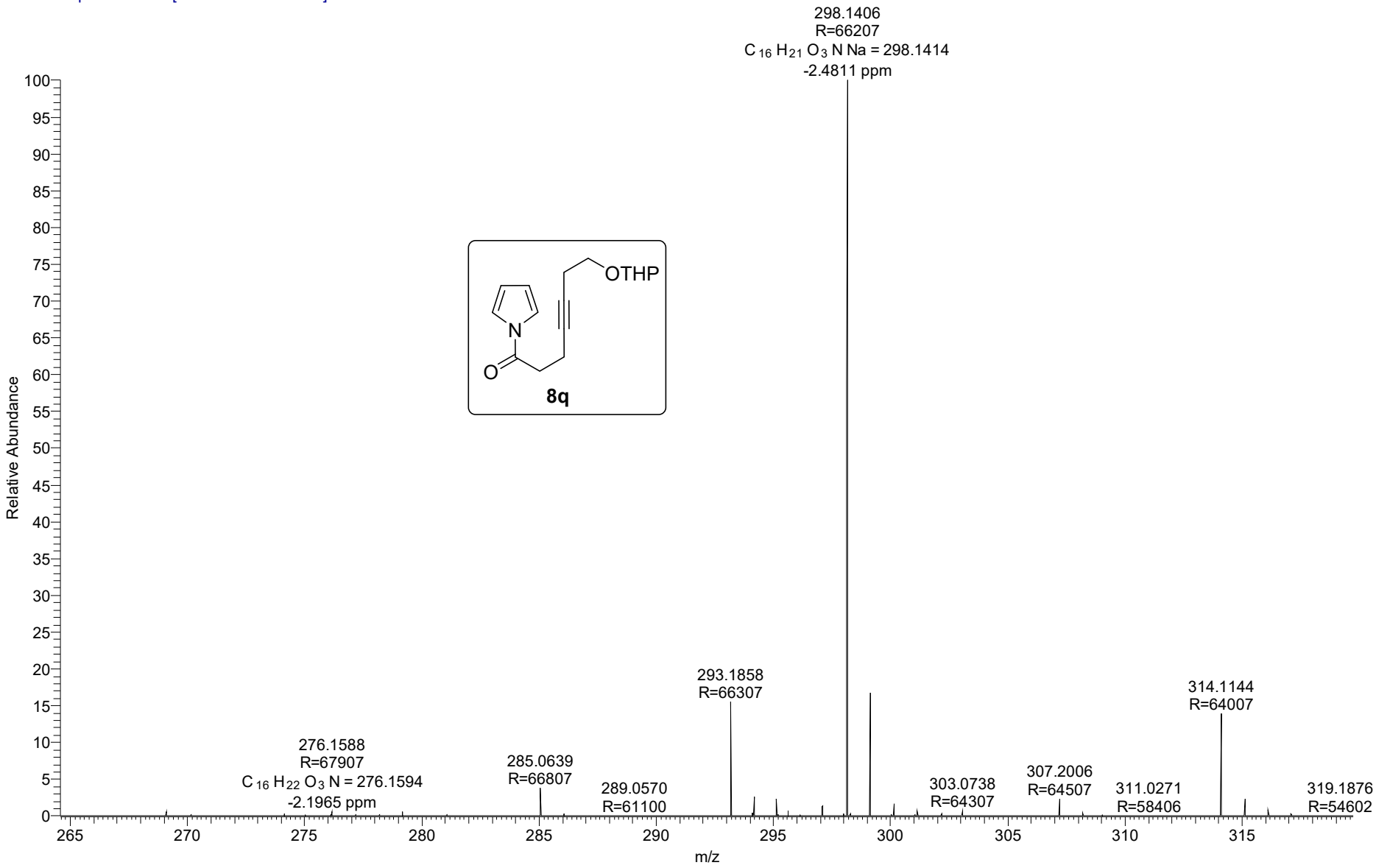


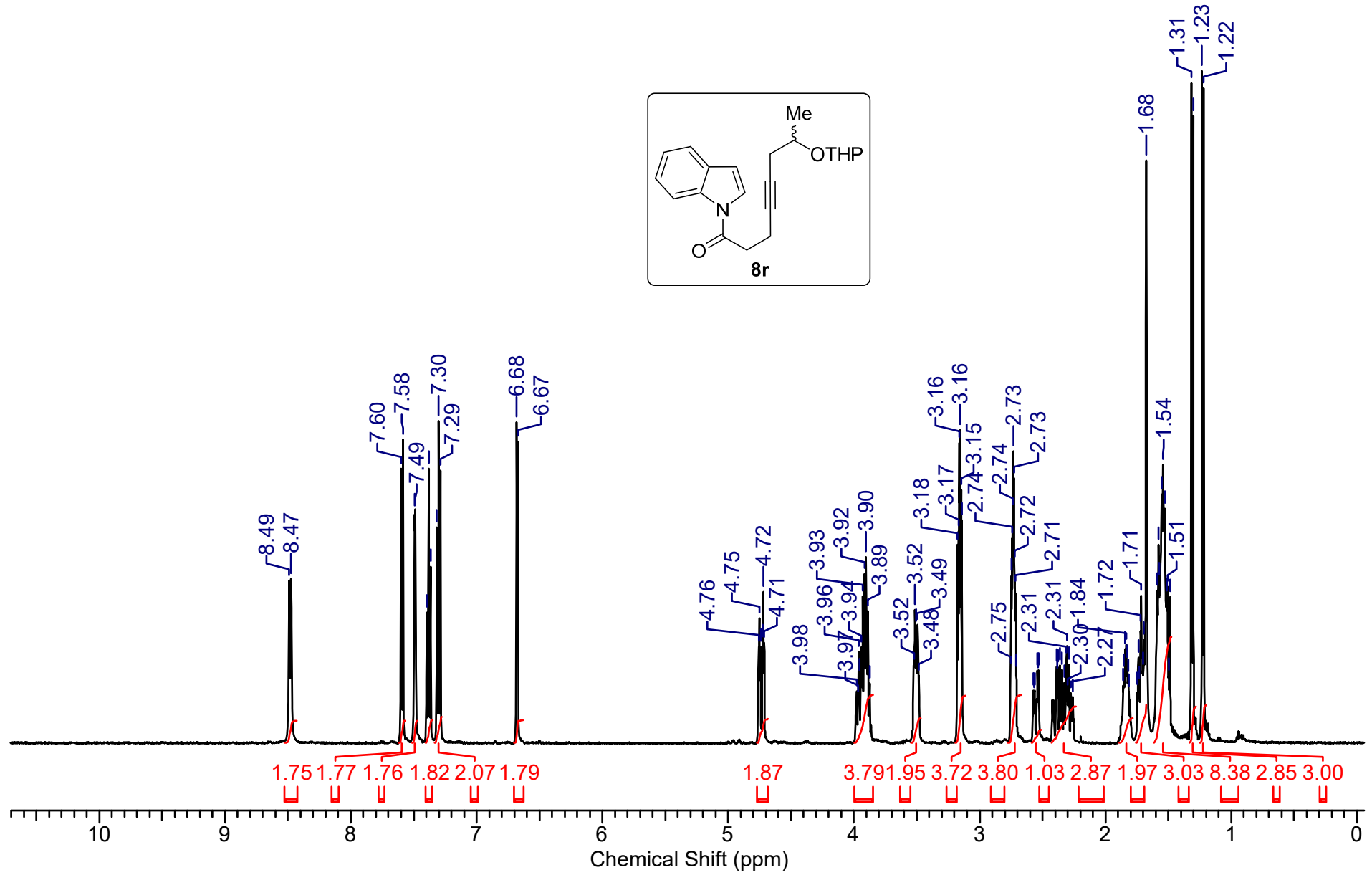

OTHP
N
O
8q
7.32
7.27
6.31
6.30
6.30
4.63
4.63
4.62
3.90
3.89
3.88
3.87
3.79
3.77
3.54
3.52
3.50
3.48
3.06
3.04
3.02
2.64
2.65
2.64
2.45
2.44
2.43
2.43
1.71
1.70
1.56
1.55
1.54
1.53
1.52
1.49
1.48
1.47
2.00
2.15
1.02
1.16
1.09
2.22
2.09
2.08
2.11
1.17
1.59
4.77
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

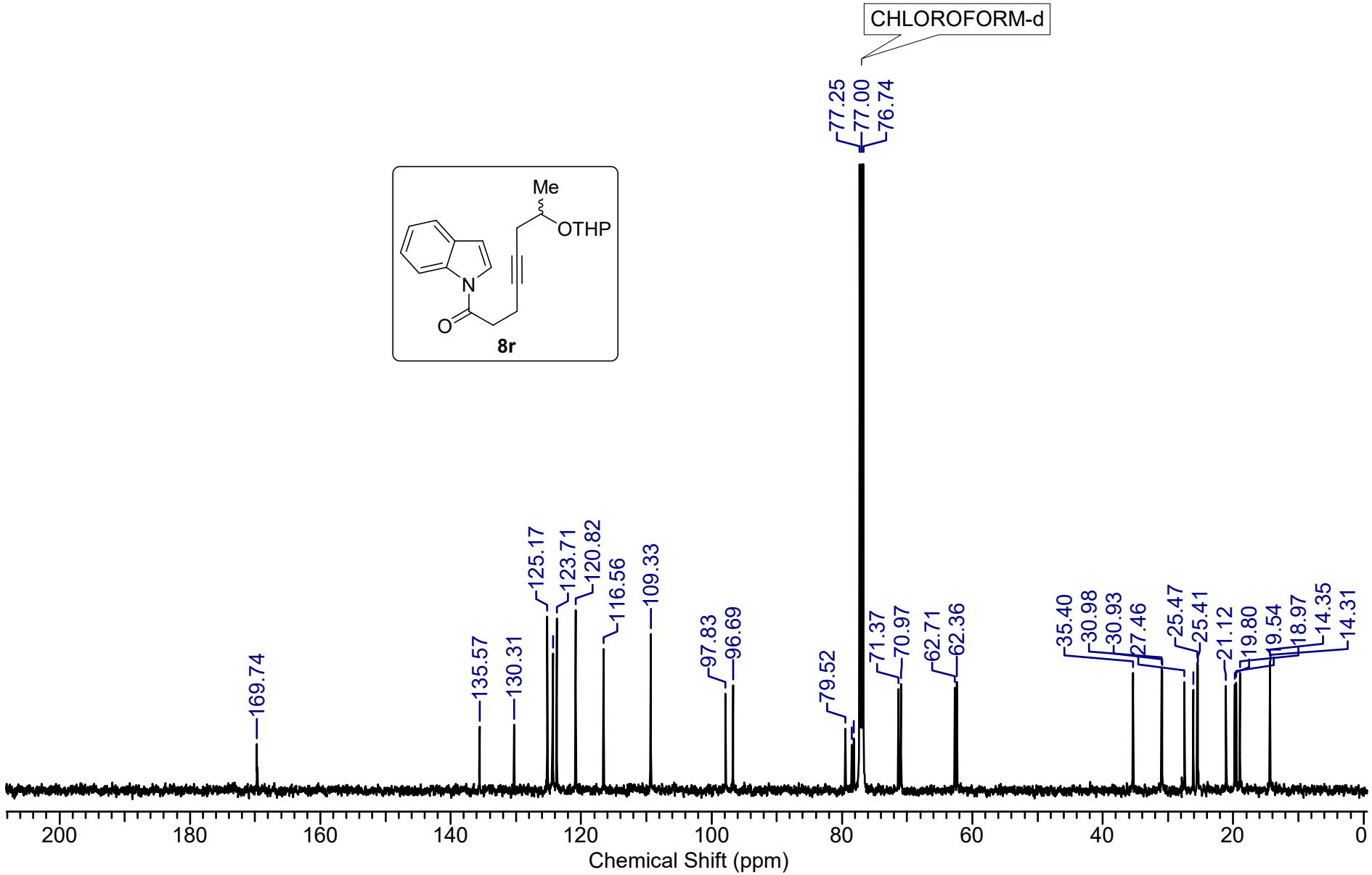

OTHP
N
O
8q
168.85
118.92
113.20
98.73
78.83
78.21
77.31
77.00
76.68
65.89
62.21
34.16
30.52
25.38
20.14
19.42
14.14
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

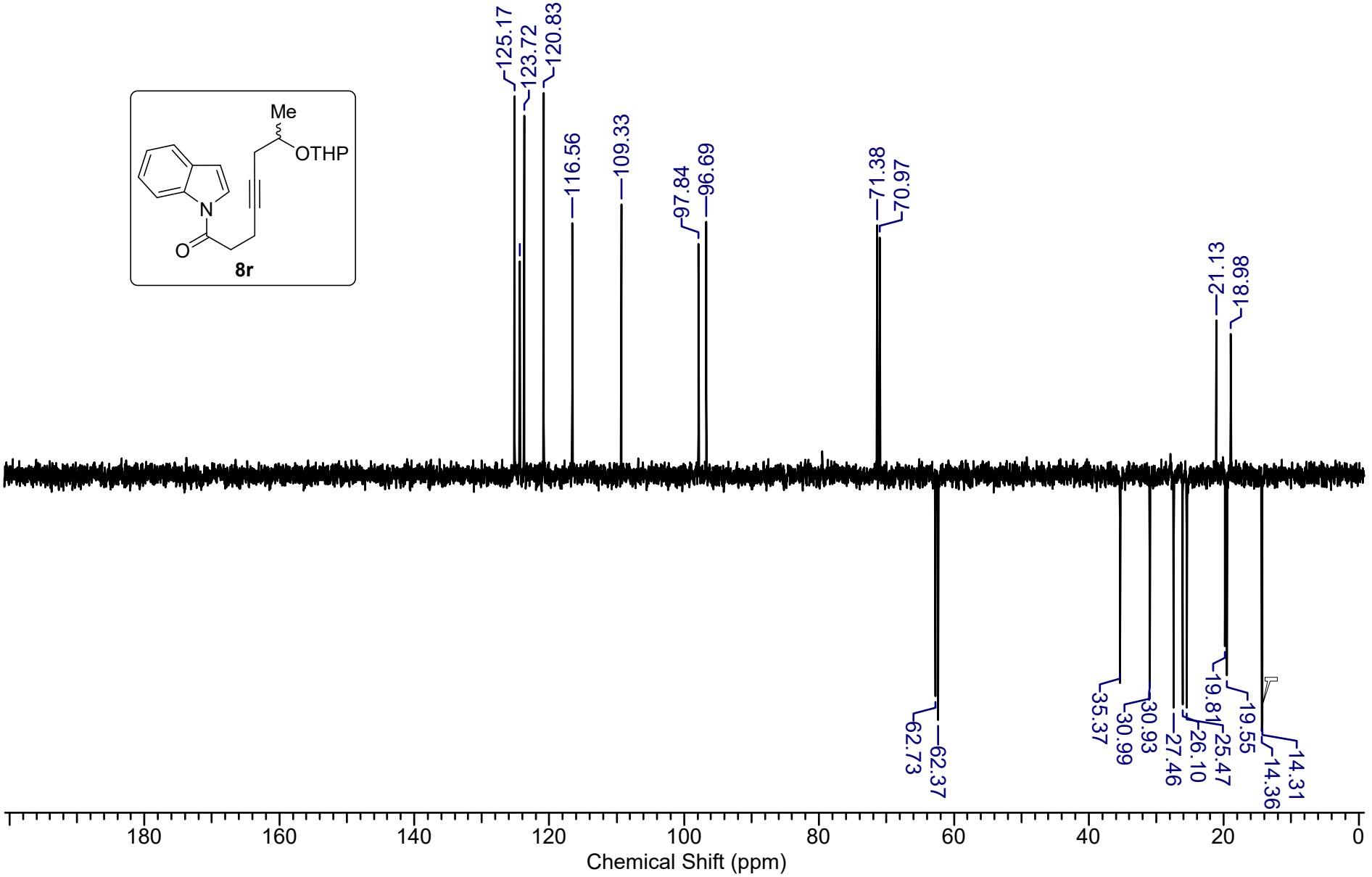

118.92
113.21
98.73
OTHP
N
O
8q
65.90
62.21
34.17
30.53
25.39
20.14
19.42
14.14
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

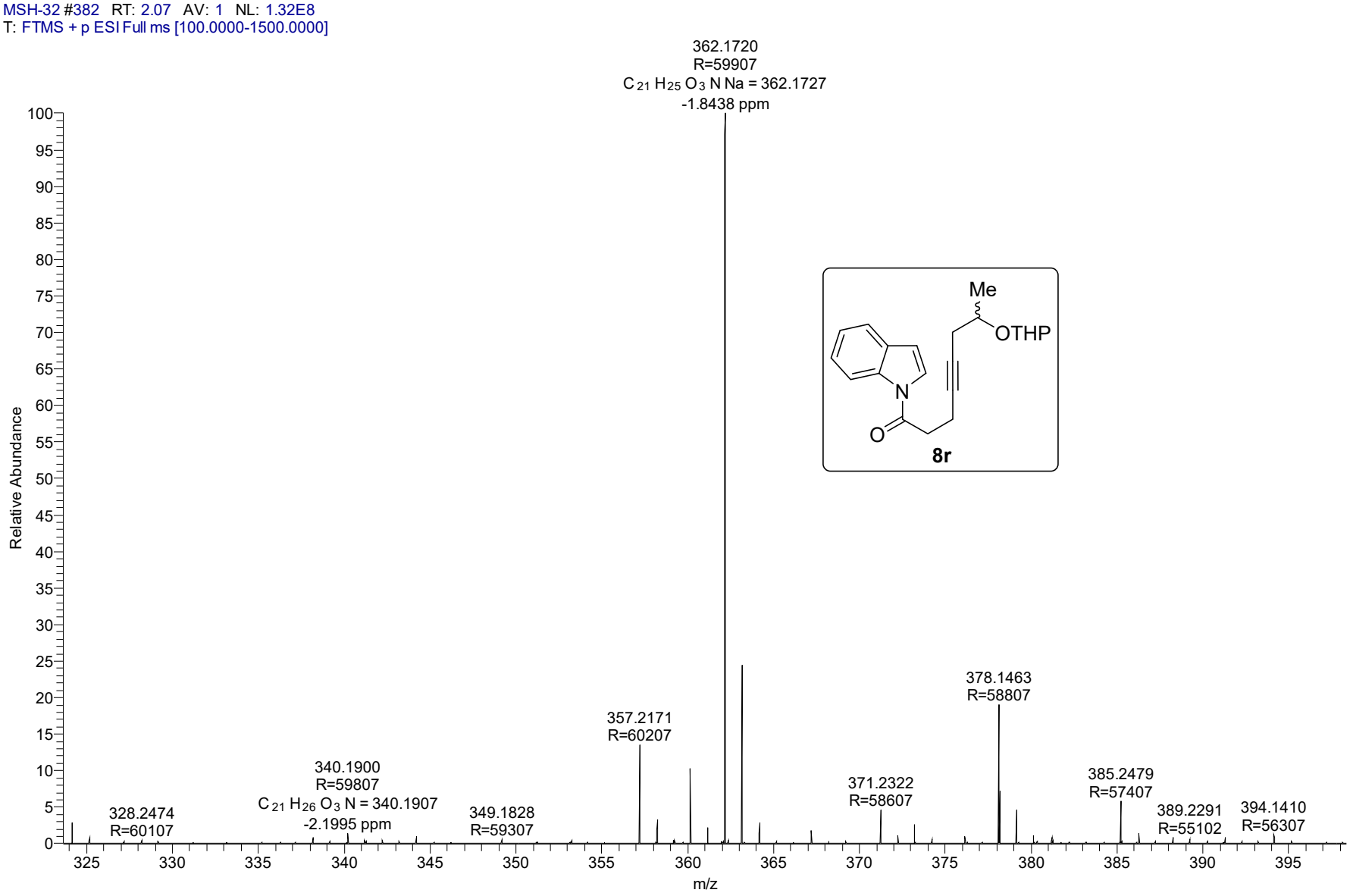
MSH-16 #330 RT: 1.78 AV: 1 NL: 2.69E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

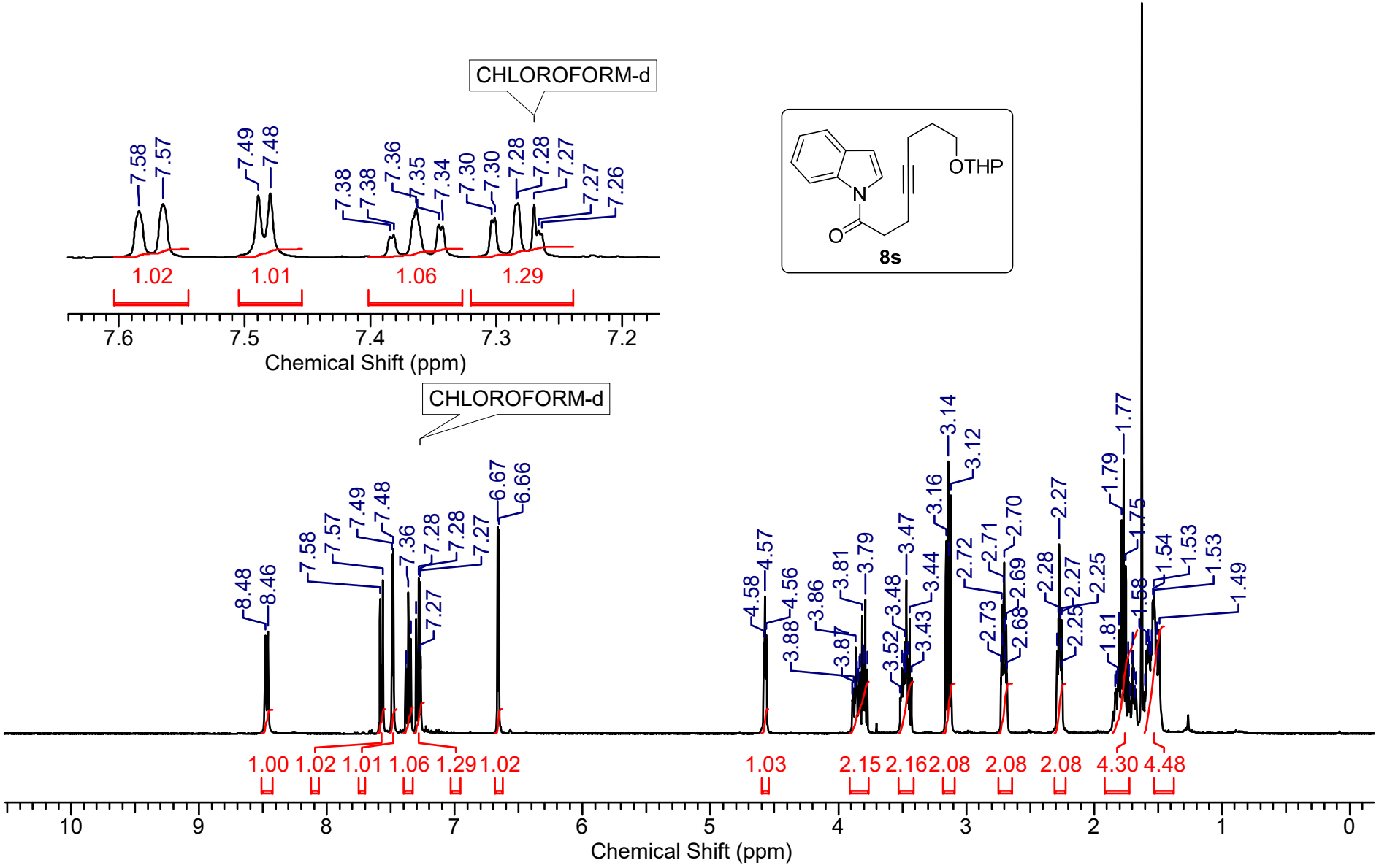


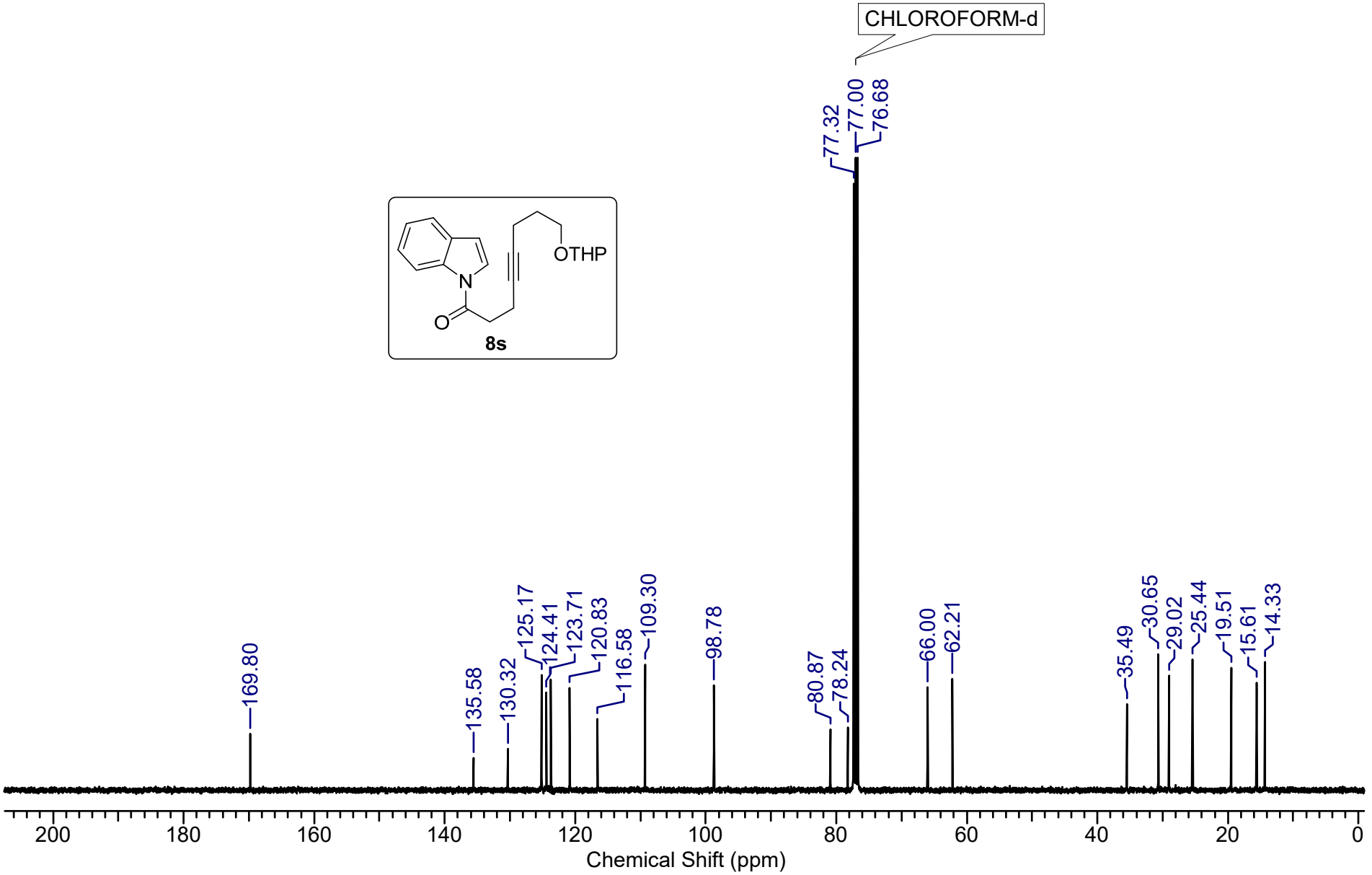

Me
OTHP
N
O
8r
8.49
8.47
7.60
7.58
7.49
7.30
7.29
6.68
6.67
4.76
4.75
4.72
4.71
3.98
3.96
3.94
3.93
3.92
3.90
3.89
3.52
3.52
3.49
3.48
3.18
3.17
3.16
3.16
3.15
2.74
2.74
2.75
2.73
2.73
2.72
2.72
2.71
2.31
2.31
2.30
1.84
2.27
1.72
1.71
1.68
1.54
1.51
1.31
1.23
1.22
1.75 1.77 1.76 1.82 2.07 1.79
1.87
3.79 1.95 3.72 3.80 1.03 2.87 1.97 3.03 8.38 2.85 3.00
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

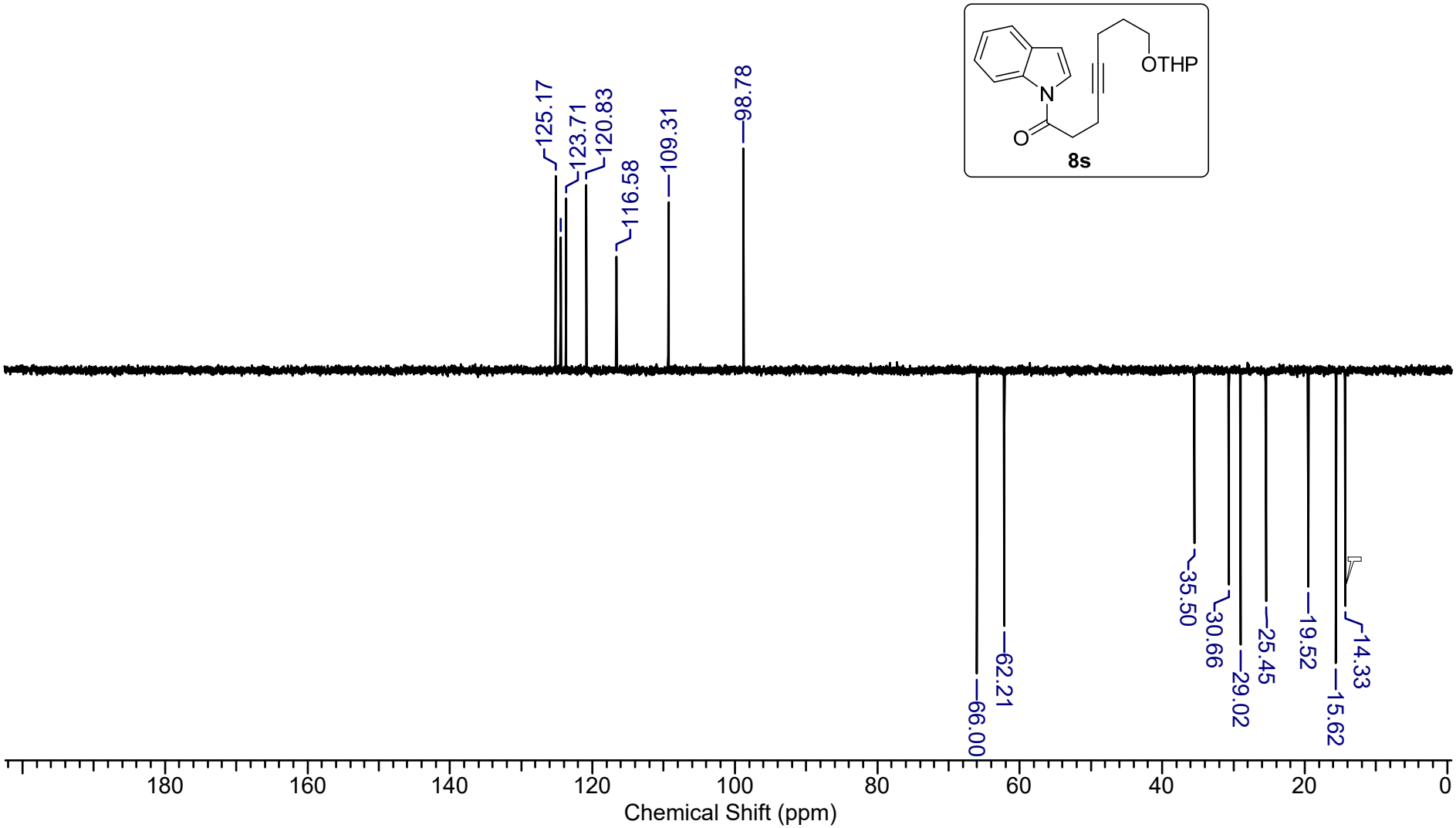

CHLOROFORM-d
77.25
77.00
76.74
Me
OTHP
N
O
8r
169.74
135.57
130.31
125.17
123.71
120.82
116.56
109.33
97.83
96.69
79.52
71.37
70.97
62.71
62.36
35.40
30.98
30.93
27.46
25.47
25.41
21.12
19.80
19.54
18.97
14.35
14.31
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

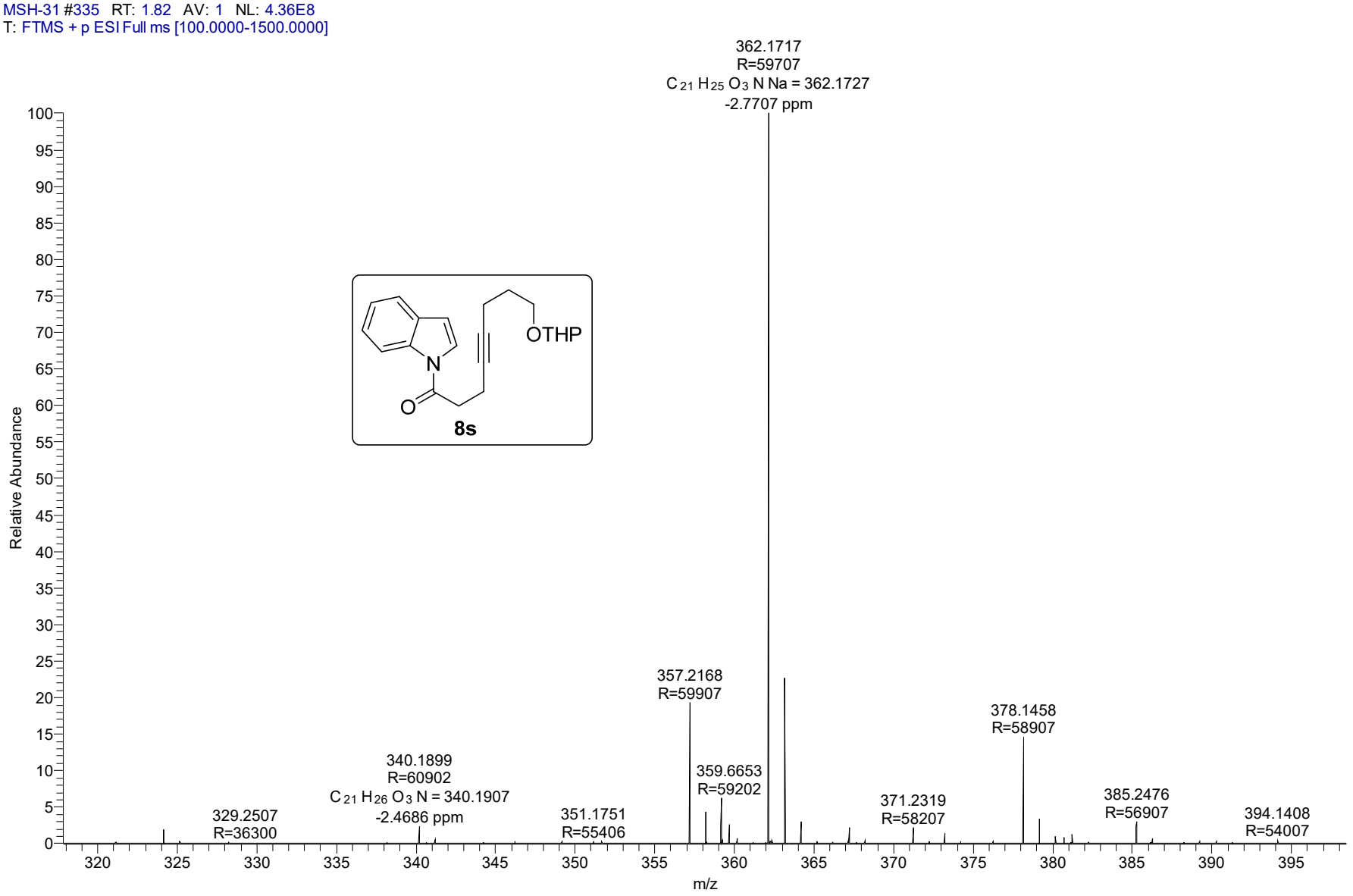

Me
OTHP
N
O
8r
125.17
123.72
120.83
116.56
109.33
97.84
96.69
71.38
70.97
31.13
30.98
62.73
62.37
35.37
30.99
30.93
27.46
26.10
25.47
19.81
19.55
14.31
14.36
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

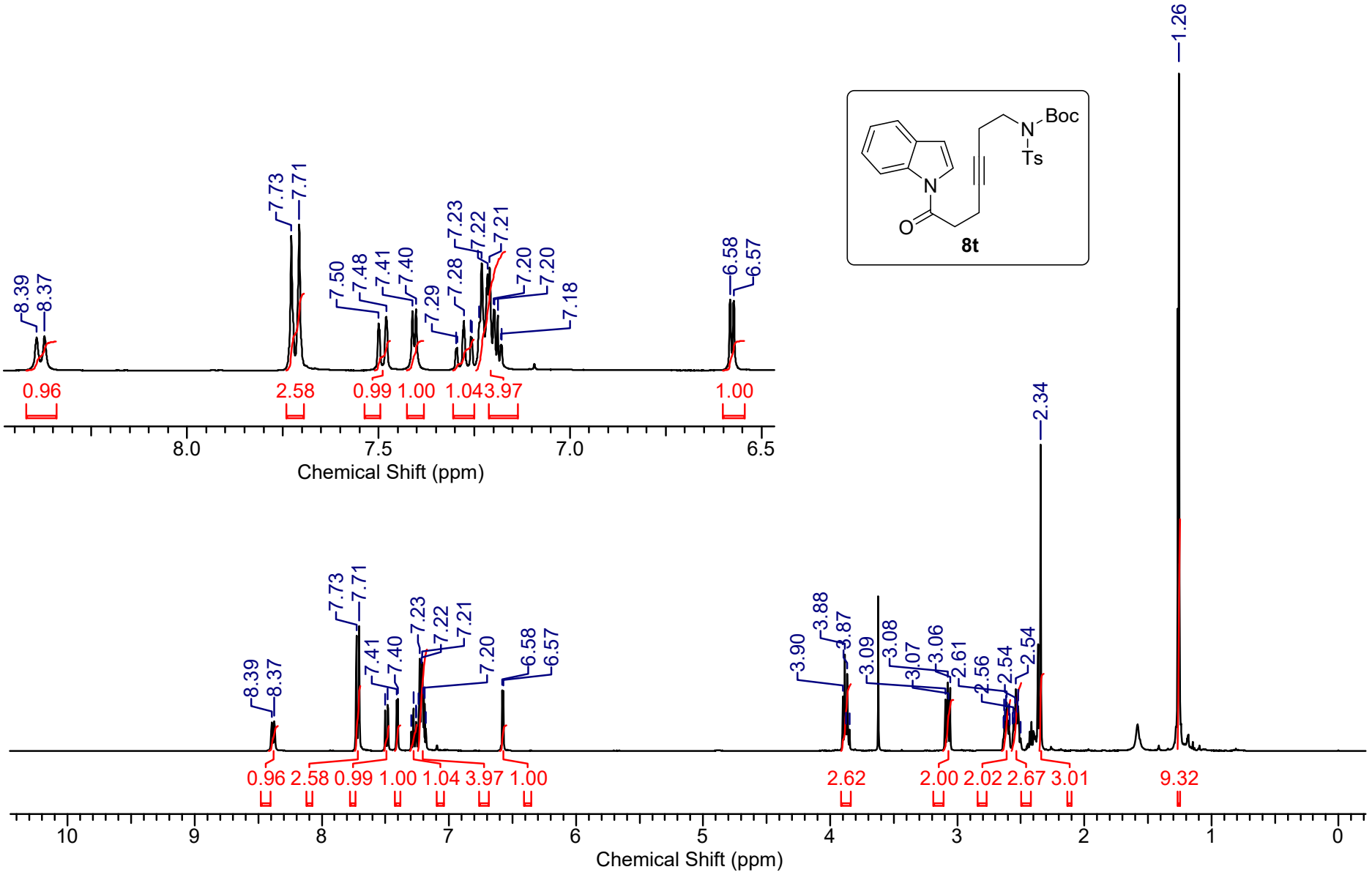

MSH-32 #382 RT: 2.07 AV: 1 NL: 1.32E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
362.1720
R=59907
C 21 H 25 O 3 N Na = 362.1727
-1.8438 ppm
Me
OTHP
N
O
8r
Relative Abundance
m/z
328.2474
R=60107
340.1900
R=59807
C 21 H 26 O 3 N = 340.1907
-2.1995 ppm
349.1828
R=59307
357.2171
R=60207
371.2322
R=58607
378.1463
R=58807
385.2479
R=57407
389.2291
R=55102
394.1410
R=56307

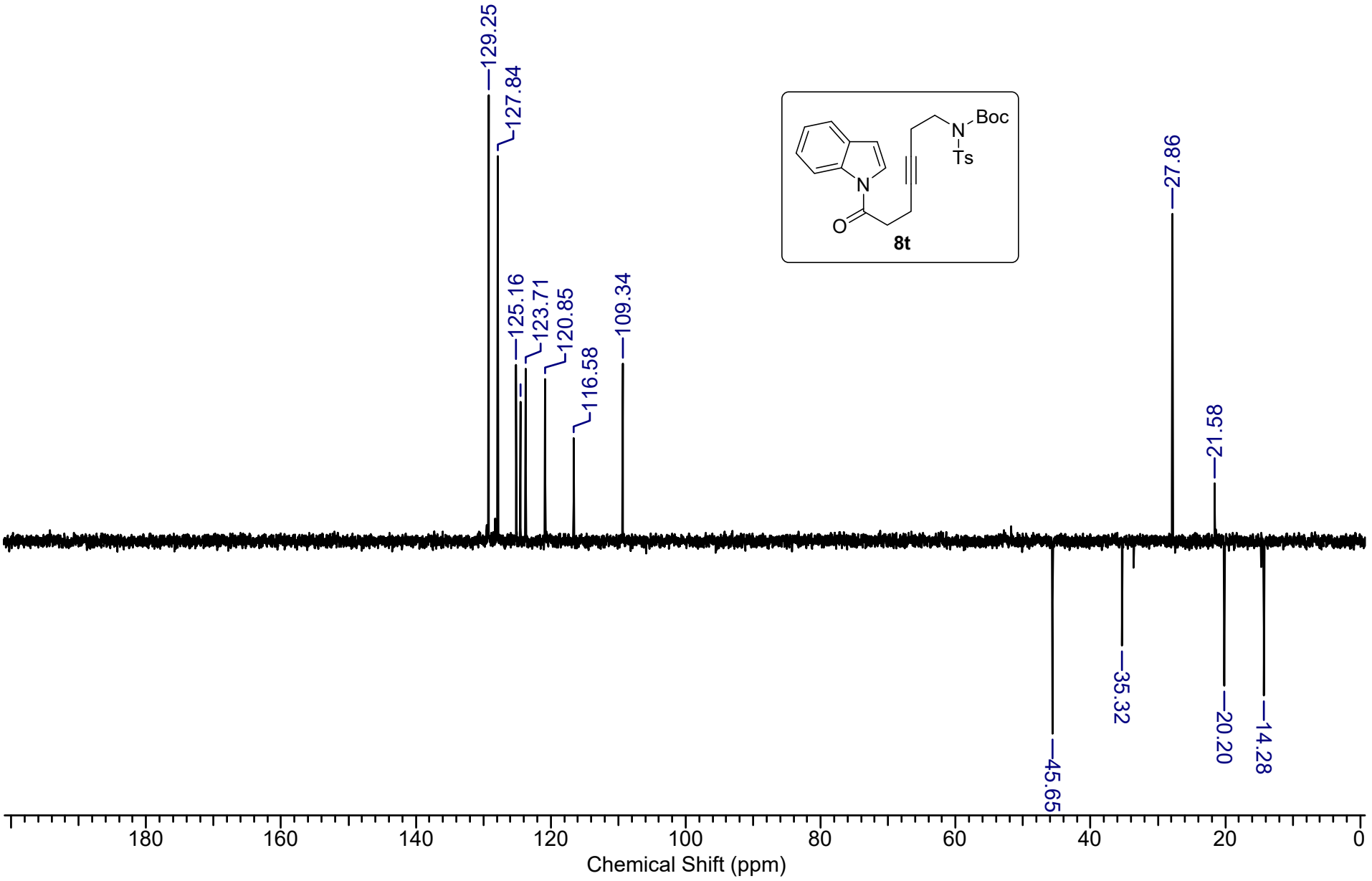

CHLOROFORM-d
7.58
7.57
7.49
7.48
7.38
7.38
7.36
7.35
7.34
7.30
7.30
7.28
7.28
7.27
7.27
7.26
1.02
1.01
1.06
1.29
7.6
7.5
7.4
7.3
7.2
Chemical Shift (ppm)
OTHP
N
O
8s
CHLOROFORM-d
8.48
8.46
7.58
7.57
7.49
7.48
7.36
7.28
7.28
7.27
7.27
6.67
6.66
4.58
4.57
4.56
3.88
3.86
3.87
3.81
3.79
3.52
3.48
3.47
3.44
3.43
3.16
3.14
3.12
2.73
2.72
2.71
2.70
2.69
2.68
2.28
2.27
2.25
2.25
1.81
1.79
1.77
1.75
1.58
1.54
1.53
1.53
1.49
1.00
1.02
1.01
1.06
1.29
1.02
1.03
2.15
2.16
2.08
2.08
2.08
4.30
4.48
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

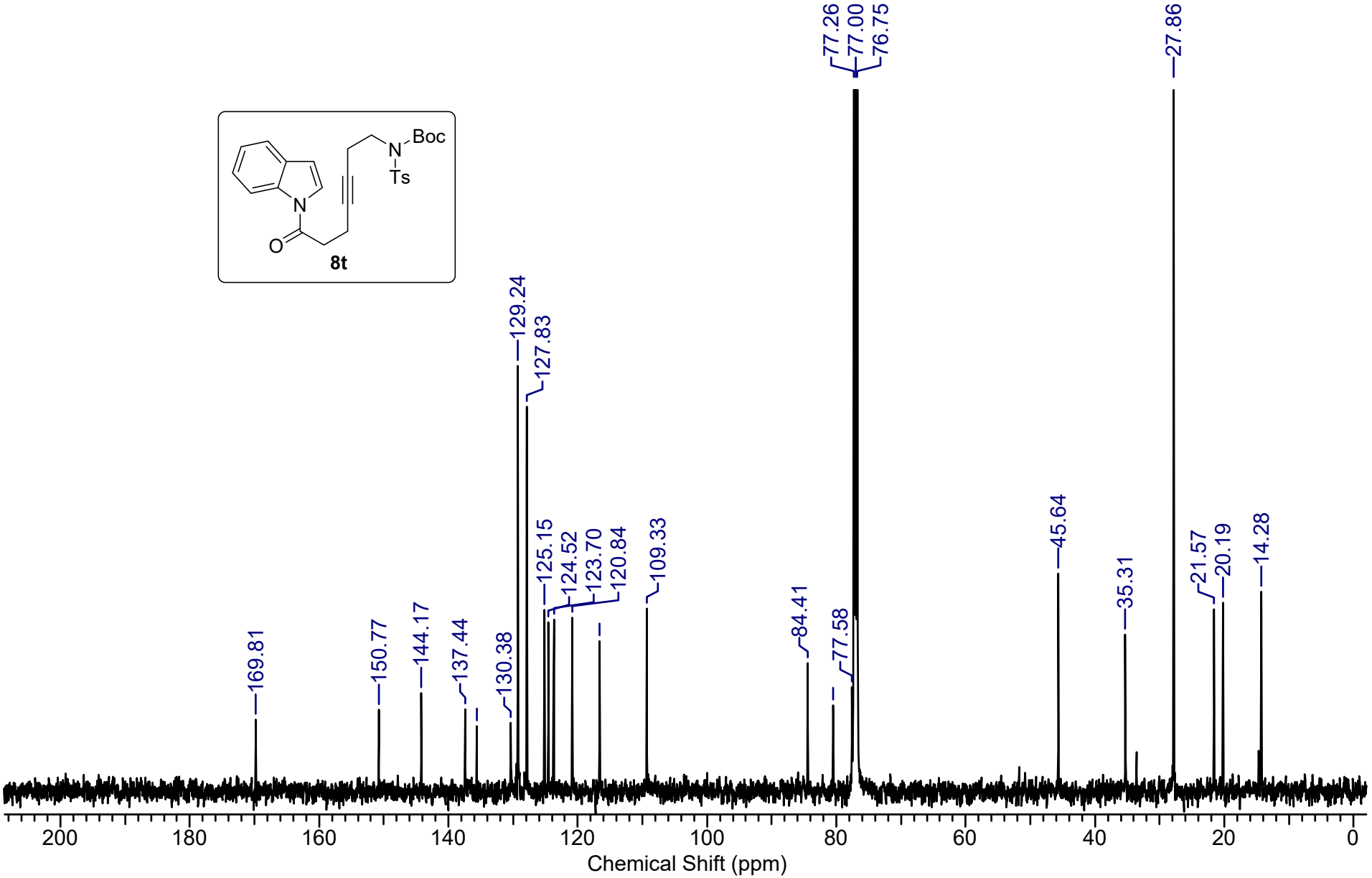

CHLOROFORM-d
77.32
77.00
76.68
OTHP
N
O
8s
169.80
135.58
130.32
125.17
124.41
123.71
120.83
116.58
109.30
98.78
80.87
78.24
66.00
62.21
35.49
30.65
29.02
25.44
19.51
15.61
14.33
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


OTHP
N
O
8s
125.17
123.71
120.83
116.58
109.31
98.78
66.00
62.21
35.50
30.66
29.02
25.45
19.52
15.62
14.33
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

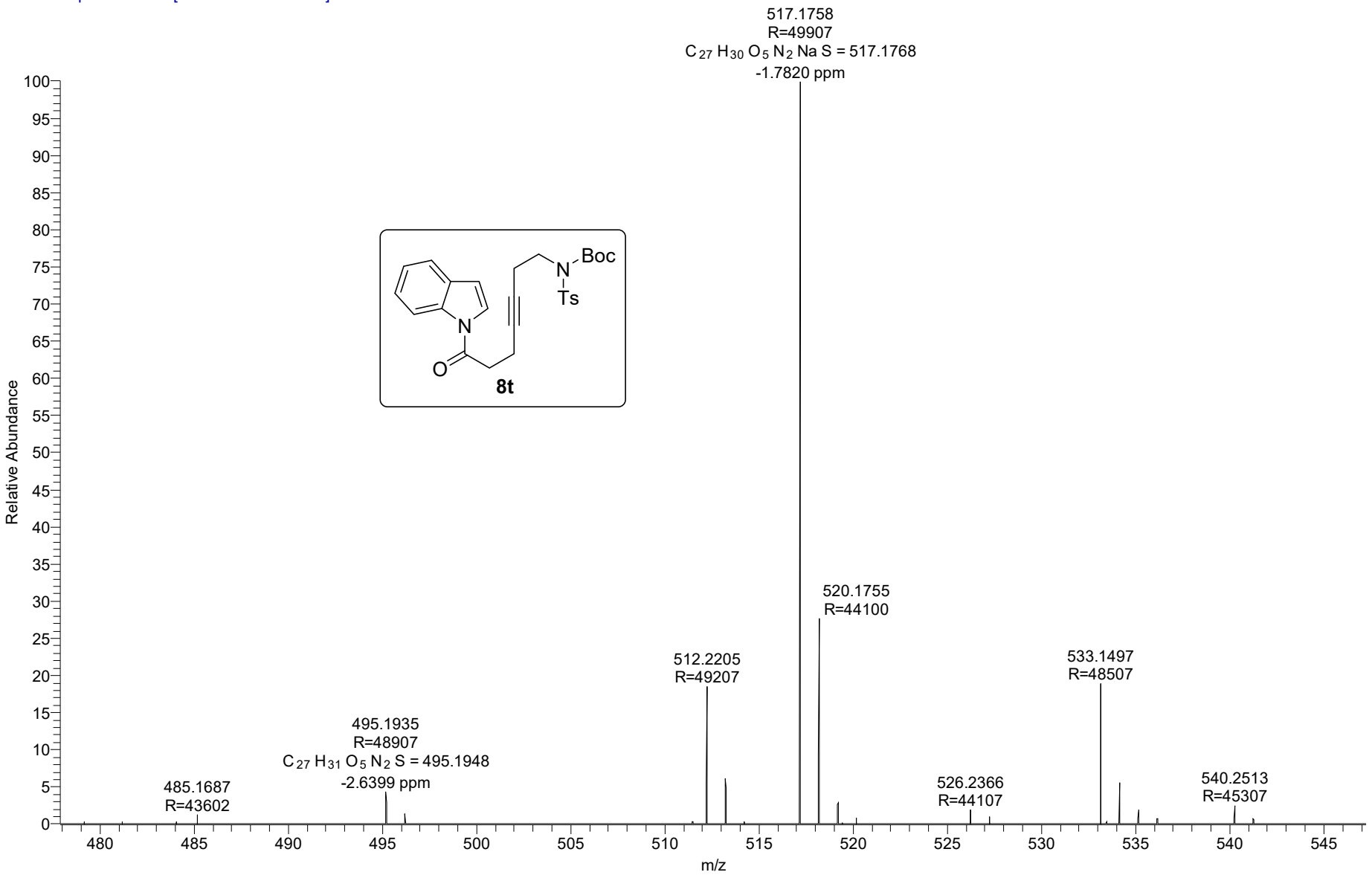

MSH-31 #335 RT: 1.82 AV: 1 NL: 4.36E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
362.1717
R=59707
C 21 H25 O3 N Na = 362.1727
-2.7707 ppm
OTHP
N
O
8s
Relative Abundance
m/z
357.2168
R=59907
378.1458
R=58907
340.1899
R=60902
C 21 H26 O3 N = 340.1907
-2.4686 ppm
359.6653
R=59202
329.2507
R=36300
351.1751
R=55406
371.2319
R=58207
385.2476
R=56907
394.1408
R=54007

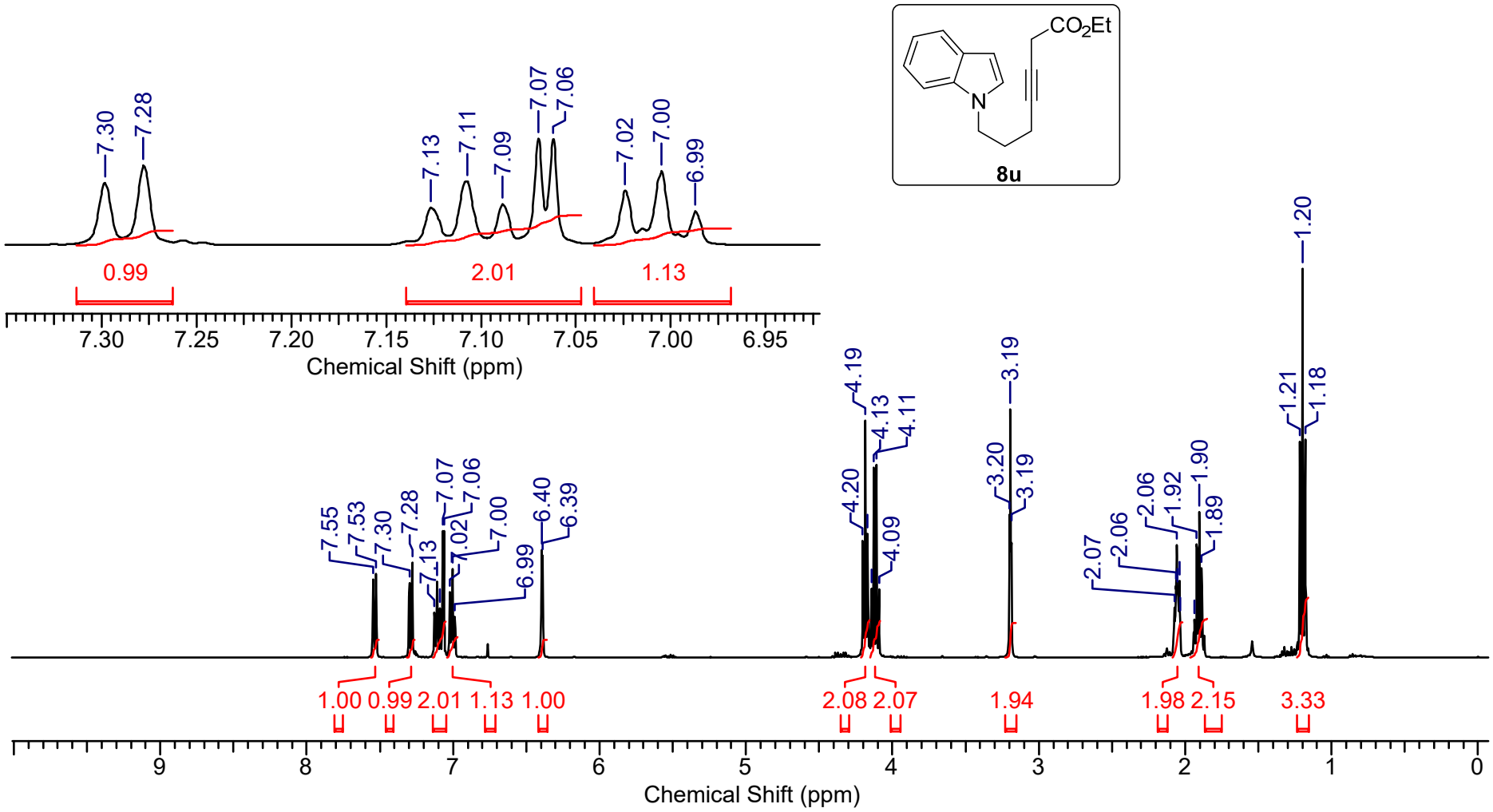

8.39
8.37
7.73
7.71
7.50
7.48
7.41
7.40
7.29
7.28
7.23
7.22
7.21
7.20
7.20
7.18
6.58
6.57
0.96
2.58
0.99
1.00
1.04
3.97
1.00
8.0
7.5
7.0
6.5
Chemical Shift (ppm)
Boc
N
Ts
N
O
8t
1.26
2.34
3.90
3.88
3.87
3.09
3.08
3.07
3.06
2.61
2.56
2.54
2.54
0.96
2.58
0.99
1.00
1.04
3.97
1.00
2.62
2.00
2.02
2.67
3.01
9.32
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

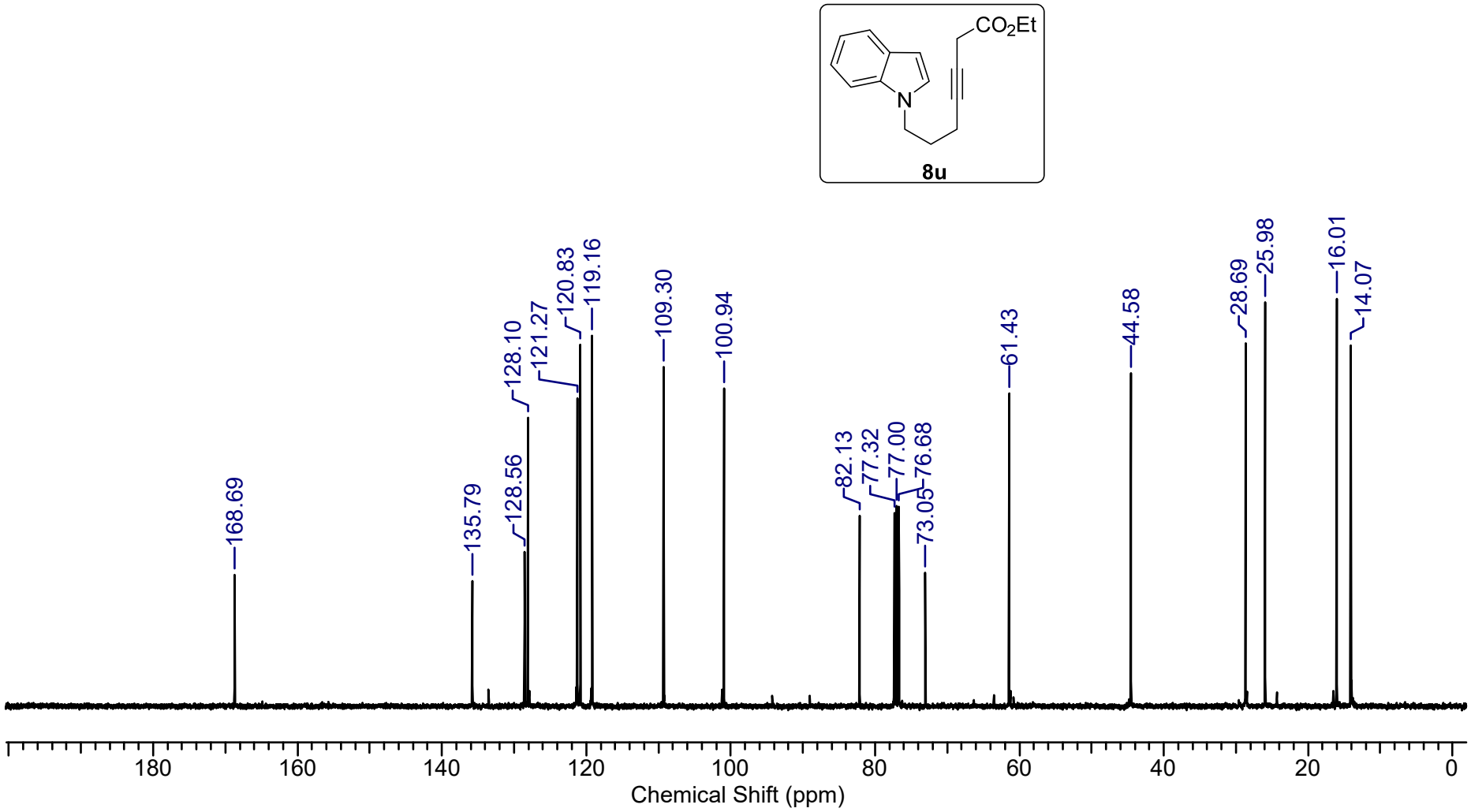

129.25
127.84
125.16
123.71
120.85
116.58
109.34
27.86
21.58
45.65
35.32
20.20
14.28
Boc
N
Ts
N
O
8t
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

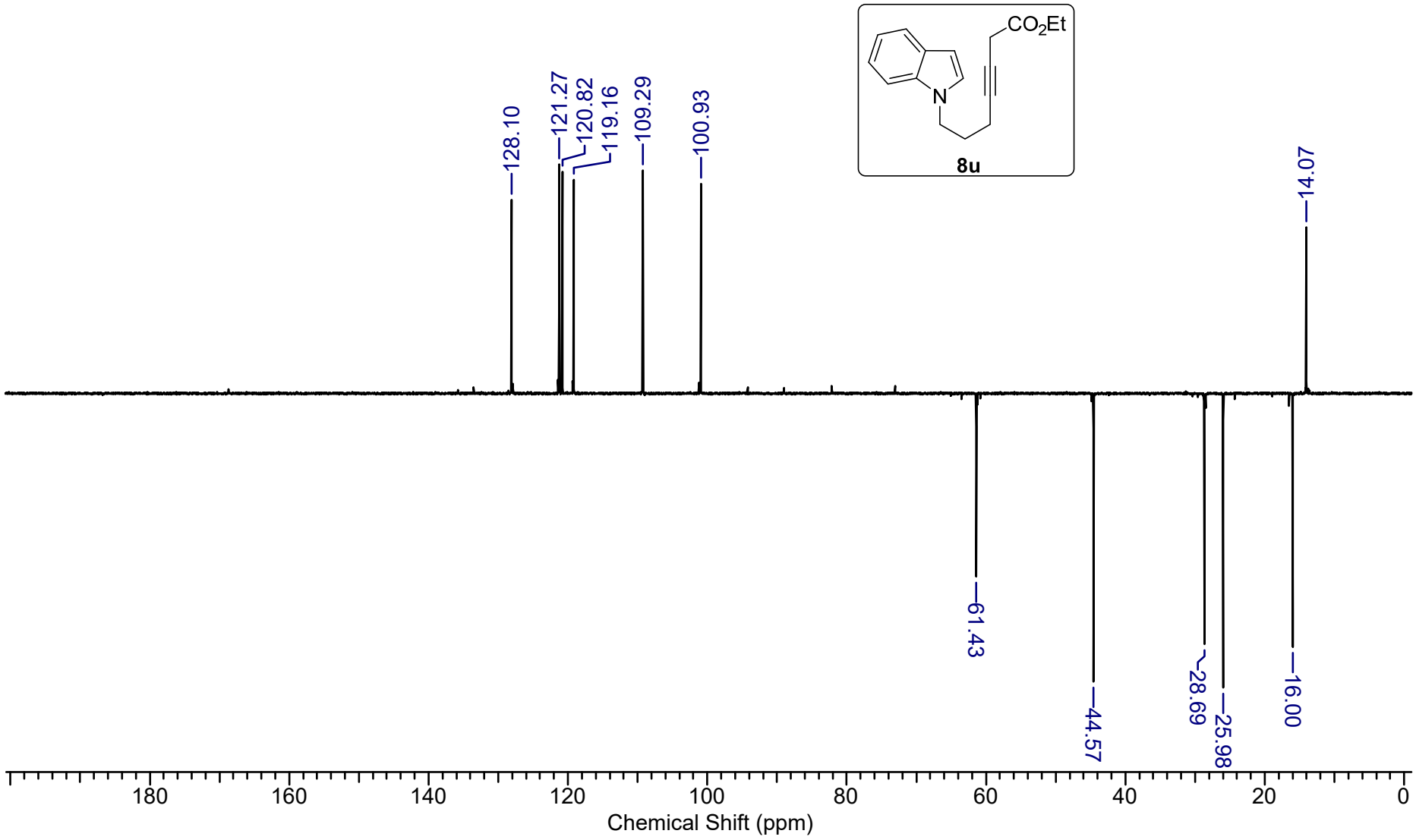

Boc
N
Ts
N
O
8t
77.26
77.00
76.75
27.86
129.24
127.83
125.15
124.52
123.70
120.84
109.33
84.41
77.58
45.64
35.31
21.57
20.19
14.28
169.81
150.77
144.17
137.44
130.38
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

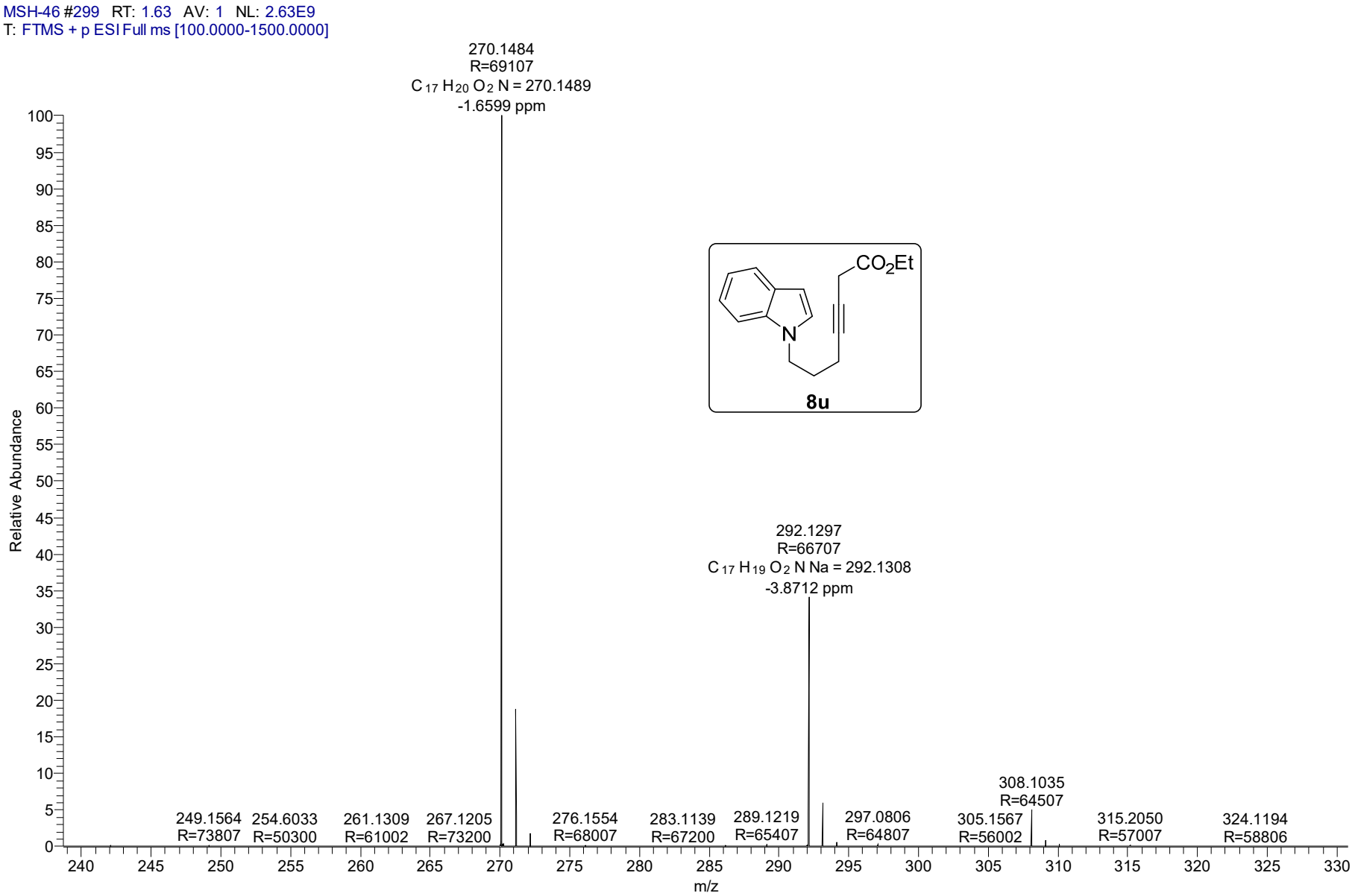
MSH-12 #303 RT: 1.64 AV: 1 NL: 1.45E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

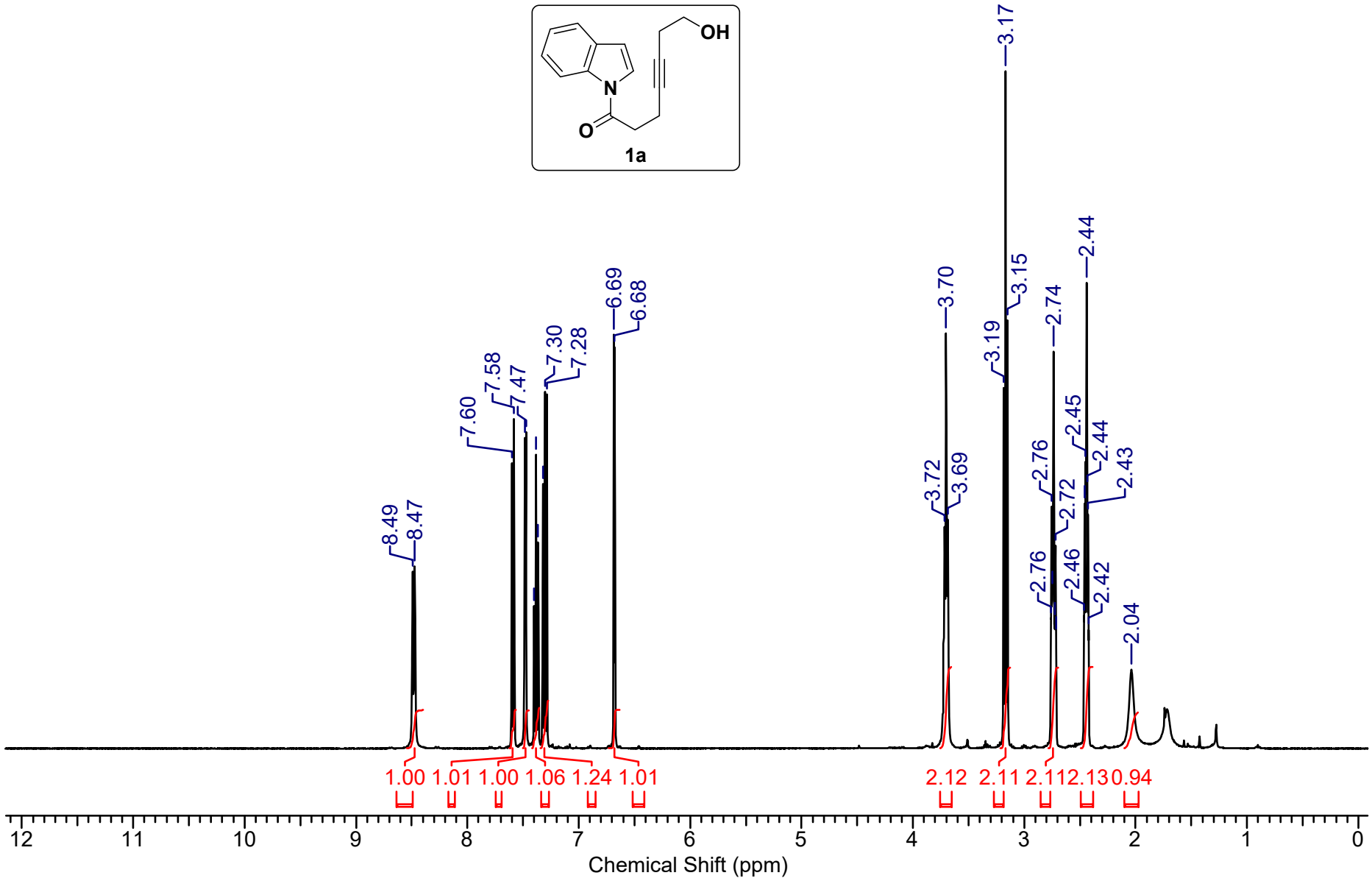


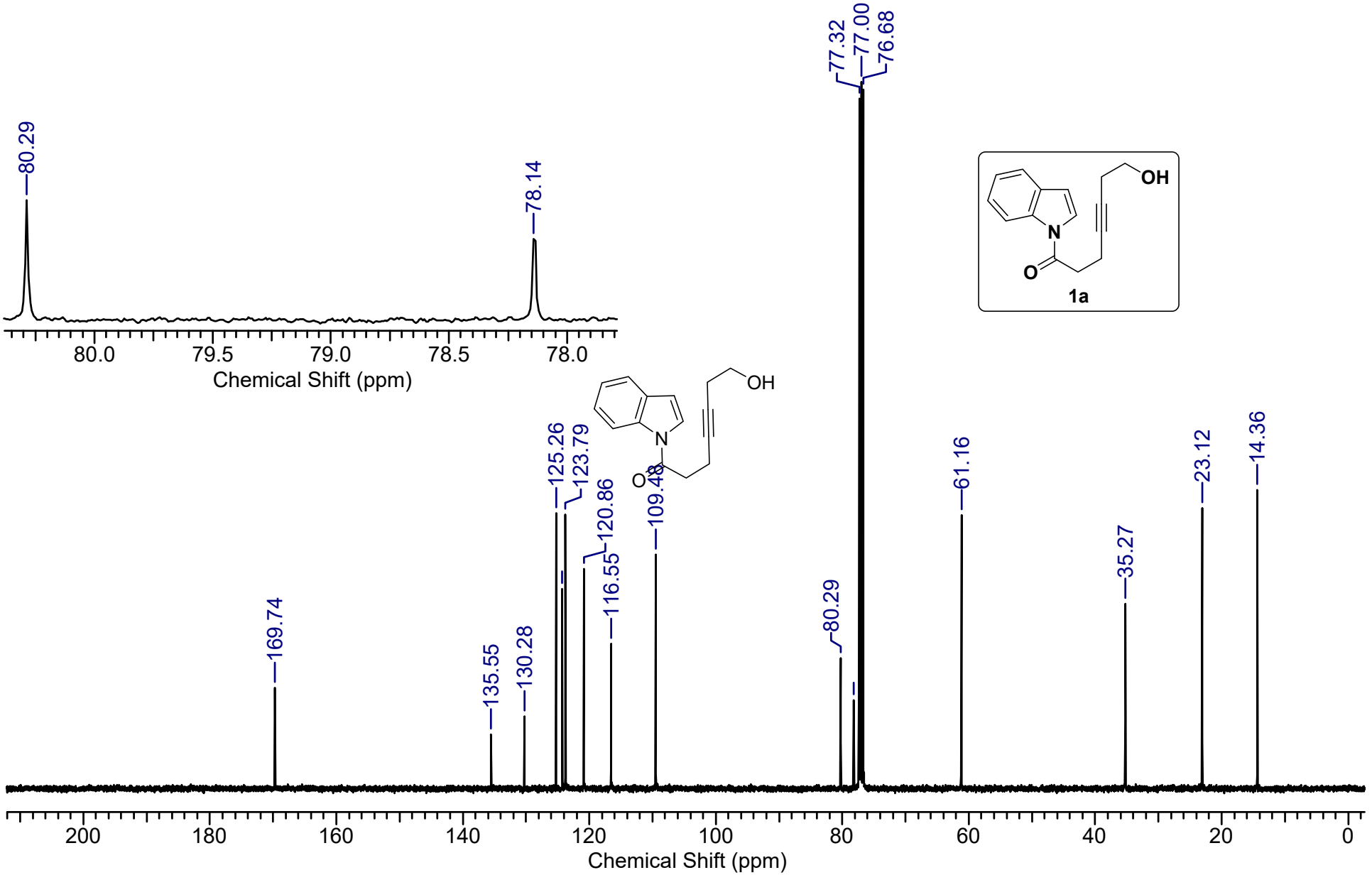

CO2Et
N
8u
7.30
7.28
7.13
7.11
7.09
7.07
7.06
7.02
7.00
6.99
0.99
2.01
1.13
7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95
Chemical Shift (ppm)
7.55
7.53
7.30
7.28
7.13
7.07
7.06
7.02
7.00
6.99
6.40
6.39
4.19
4.13
4.11
4.20
4.09
3.20
3.19
3.19
2.07
2.06
2.06
1.92
1.90
1.89
1.21
1.18
1.20
1.00 0.99 2.01 1.13 1.00
2.08 2.07
1.94
1.98 2.15
3.33
9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

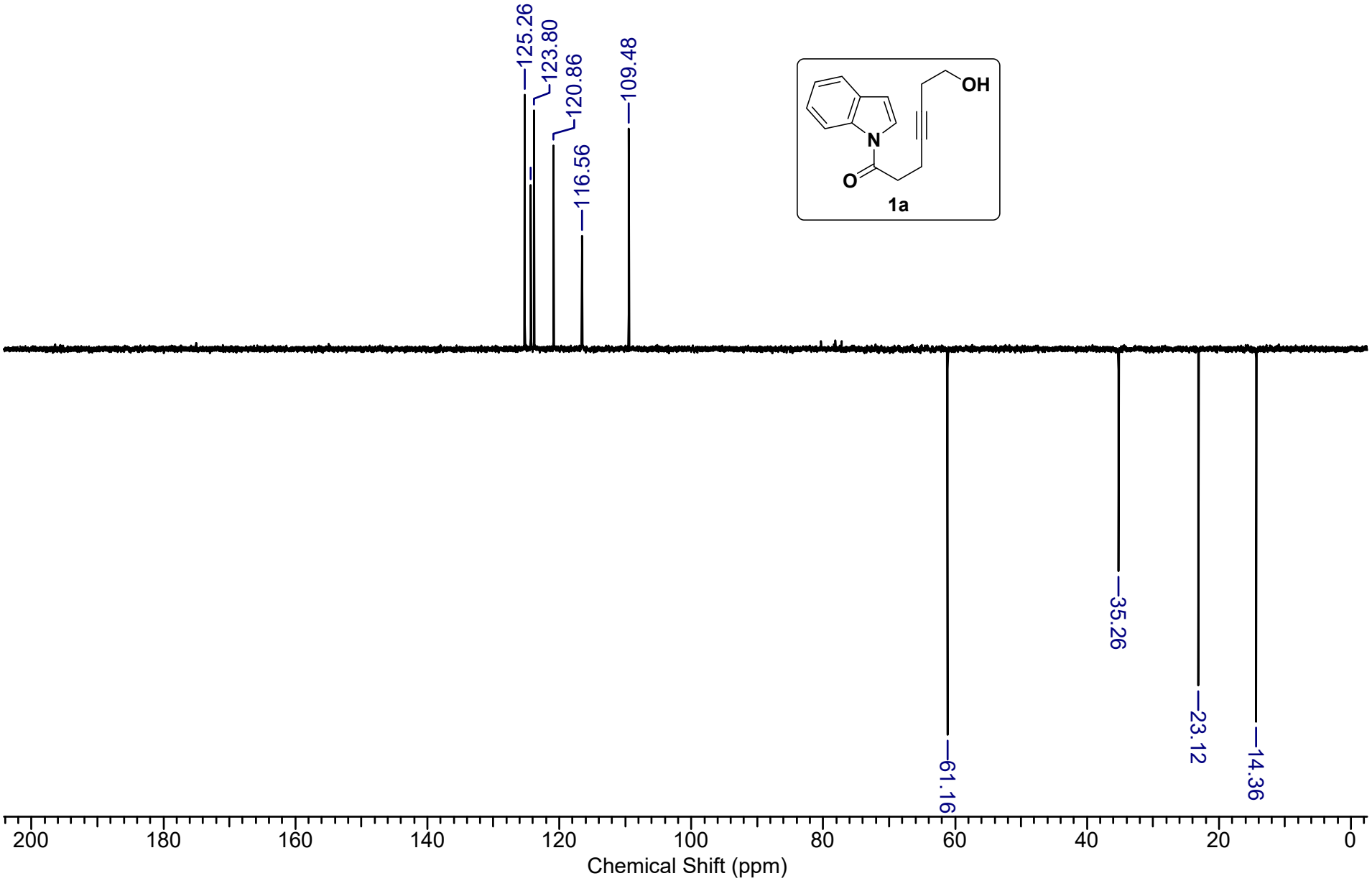

CO2Et
N
8u
168.69
135.79
128.56
128.10
121.27
120.83
119.16
109.30
100.94
82.13
77.32
77.00
76.68
73.05
61.43
44.58
28.69
25.98
16.01
14.07
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

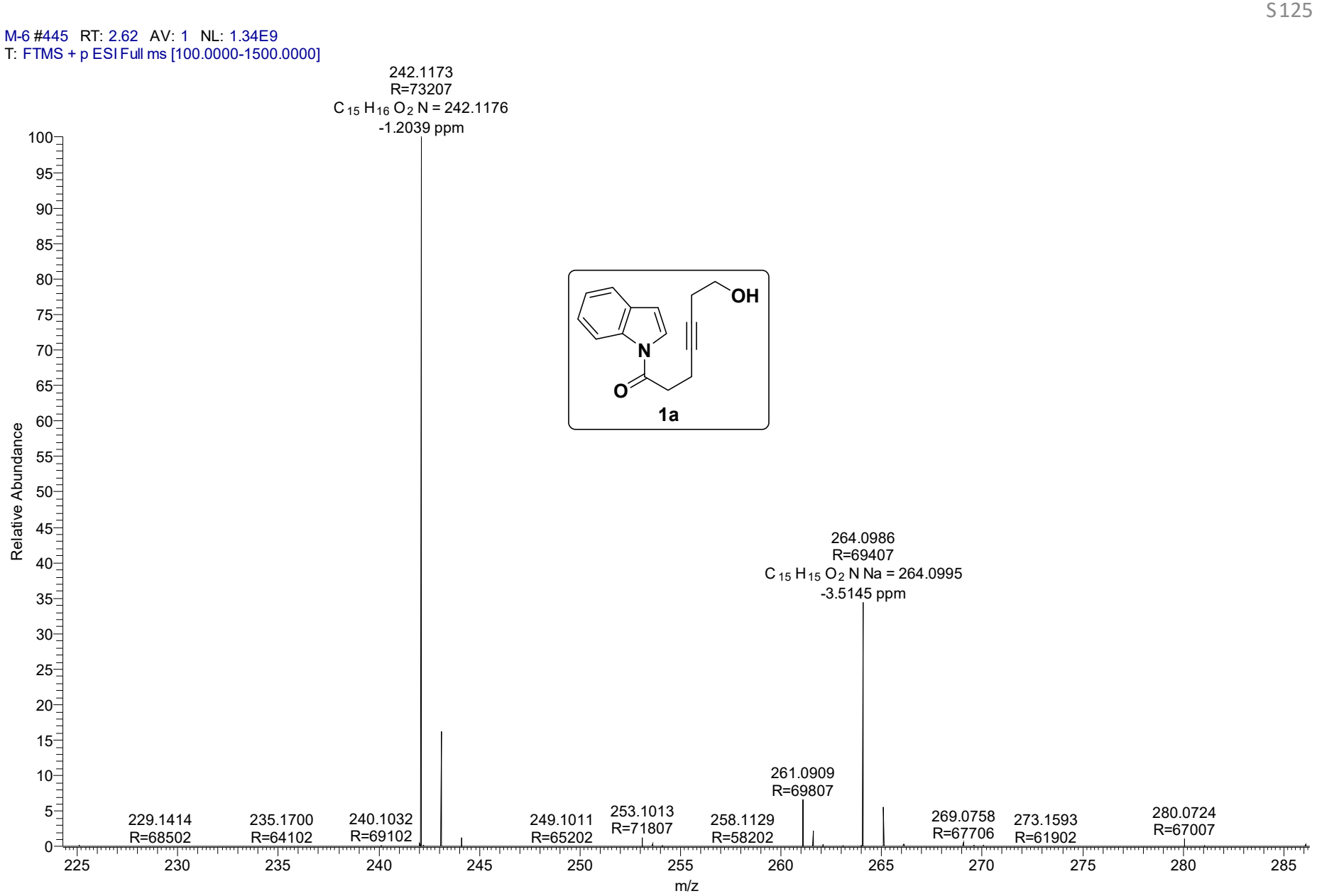

CO2Et
N
8u
128.10
121.27
120.82
119.16
109.29
100.93
14.07
61.43
44.57
28.69
25.98
16.00
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

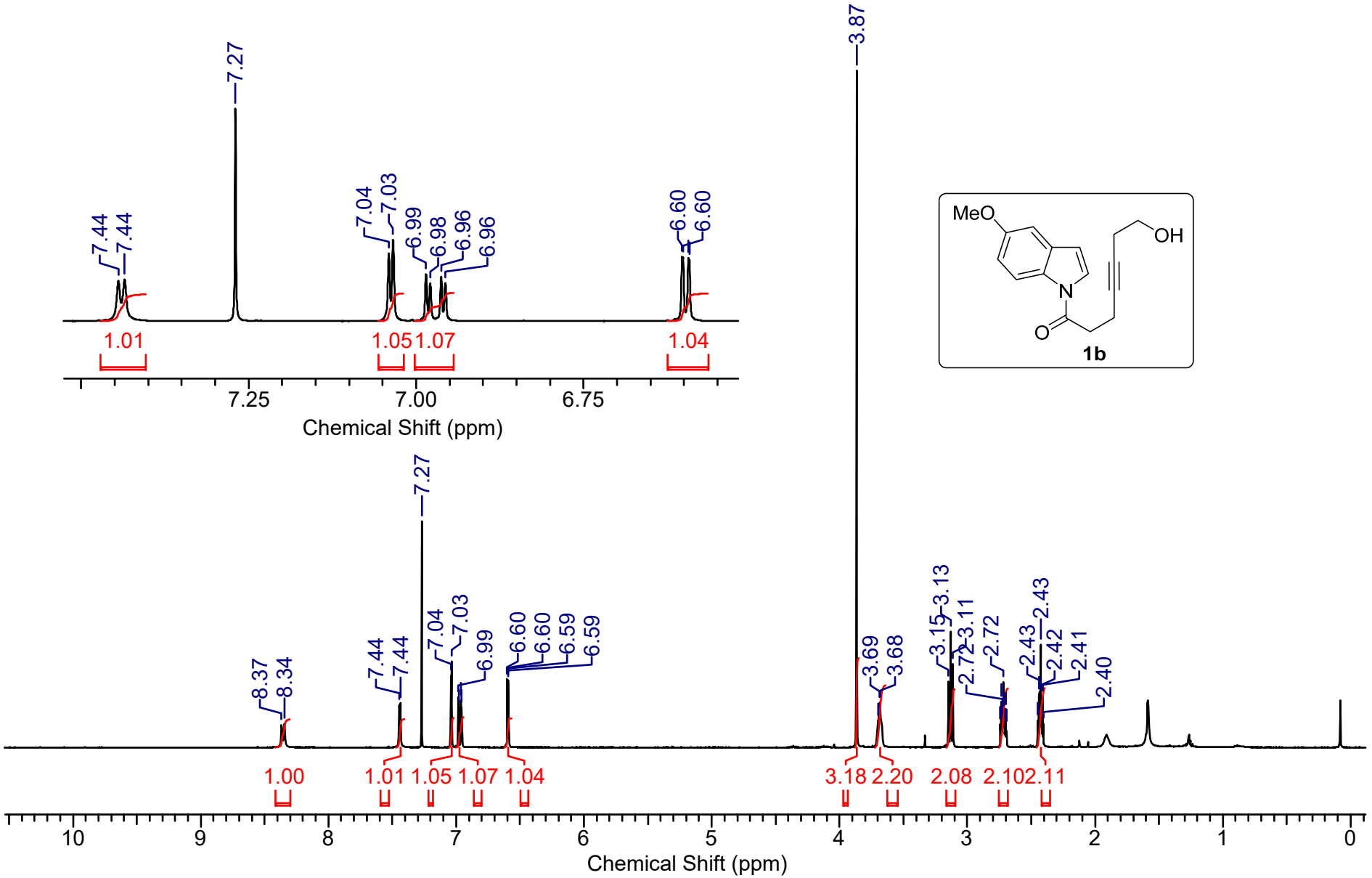

MSH-46 #299 RT: 1.63 AV: 1 NL: 2.63E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]
270.1484
R=69107
C 17 H 20 O 2 N = 270.1489
-1.6599 ppm
CO2Et
N
8u
292.1297
R=66707
C 17 H 19 O 2 N Na = 292.1308
-3.8712 ppm
249.1564 R=73807
254.6033 R=50300
261.1309 R=61002
267.1205 R=73200
276.1554 R=68007
283.1139 R=67200
289.1219 R=65407
297.0806 R=64807
305.1567 R=56002
308.1035 R=64507
315.2050 R=57007
324.1194 R=58806
Relative Abundance
m/z

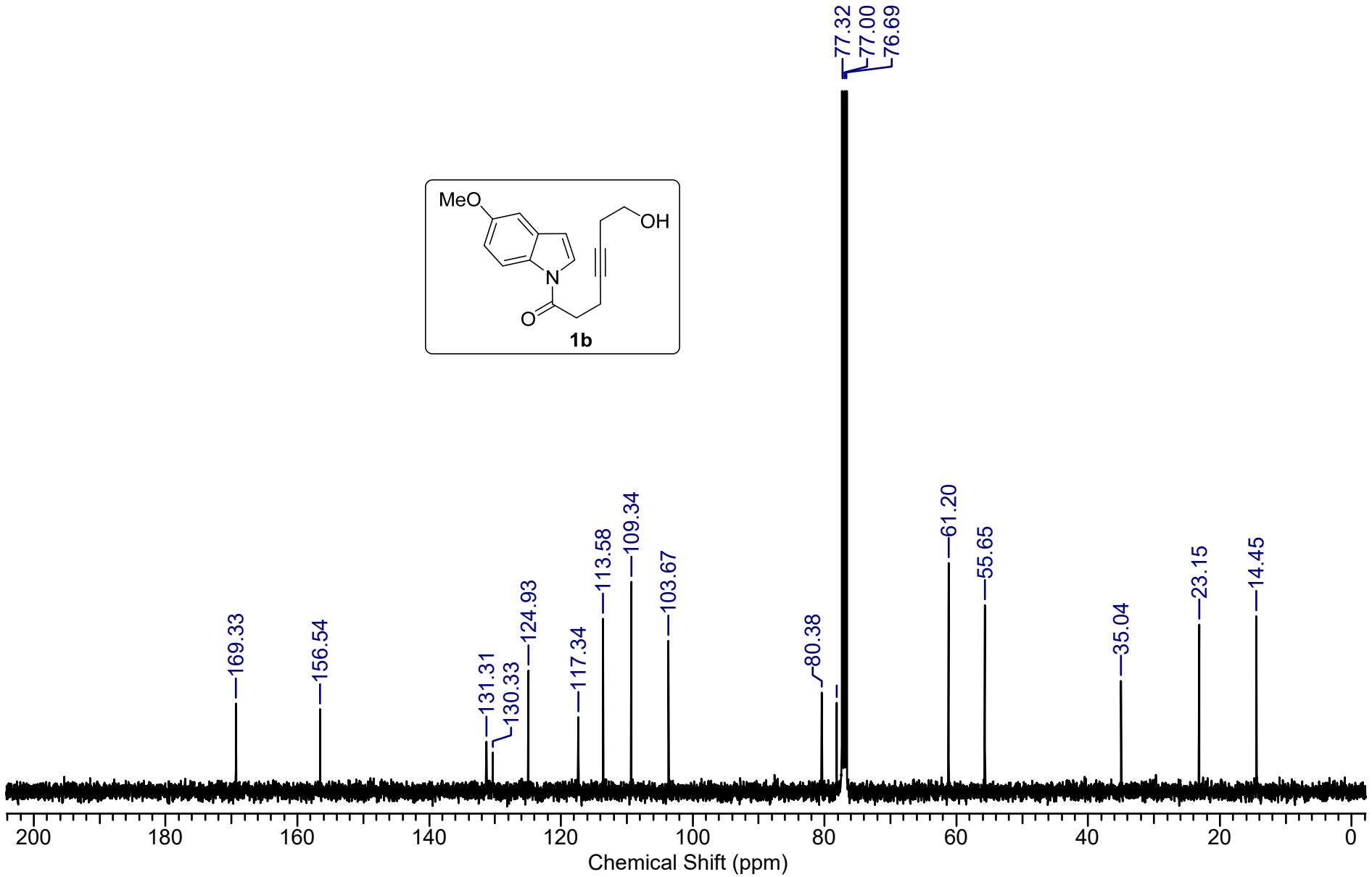

OH
N
O
1a
8.49
8.47
7.60
7.58
7.47
7.30
7.28
6.69
6.68
3.72
3.70
3.69
3.19
3.17
3.15
2.76
2.74
2.72
2.46
2.45
2.44
2.43
2.42
2.04
1.00 1.01 1.00 1.06 1.24 1.01
2.12 2.11 2.11 2.13 0.94
12 11 10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

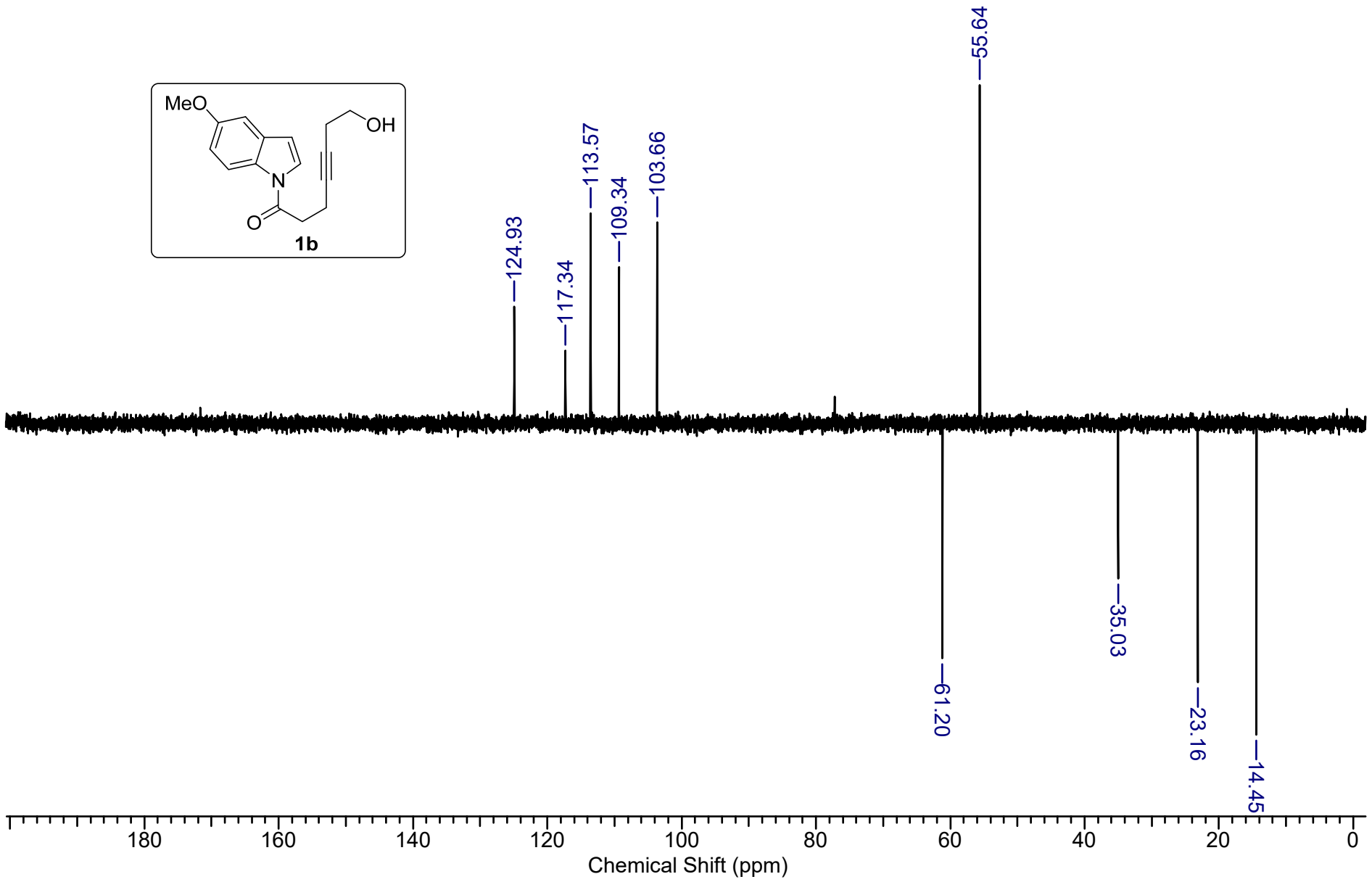

80.29
78.14
80.0
79.5
79.0
78.5
78.0
Chemical Shift (ppm)
77.32
77.00
76.68
OH
N
O
1a
125.26
123.79
120.86
116.55
109.48
61.16
35.27
23.12
14.36
169.74
135.55
130.28
80.29
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


125.26
123.80
120.86
116.56
109.48
OH
N
O
1a
35.26
23.12
14.36
61.16
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

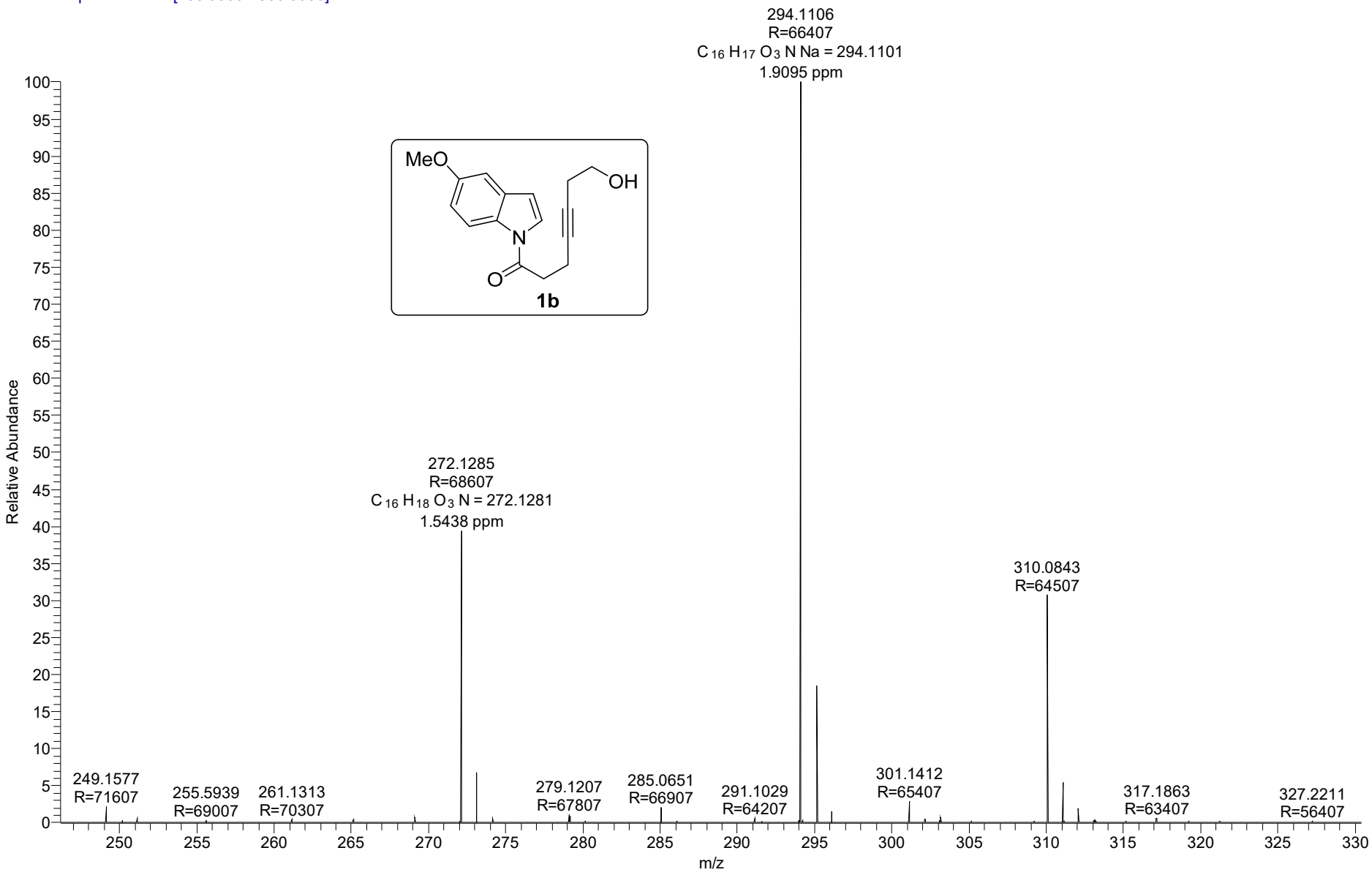

M-6 #445 RT: 2.62 AV: 1 NL: 1.34E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]
242.1173
R=73207
C 15 H 16 O 2 N = 242.1176
-1.2039 ppm
OH
N
O
1a
264.0986
R=69407
C 15 H 15 O 2 N Na = 264.0995
-3.5145 ppm
261.0909
R=69807
229.1414
R=68502
235.1700
R=64102
240.1032
R=69102
249.1011
R=65202
253.1013
R=71807
258.1129
R=58202
269.0758
R=67706
273.1593
R=61902
280.0724
R=67007
Relative Abundance
m/z

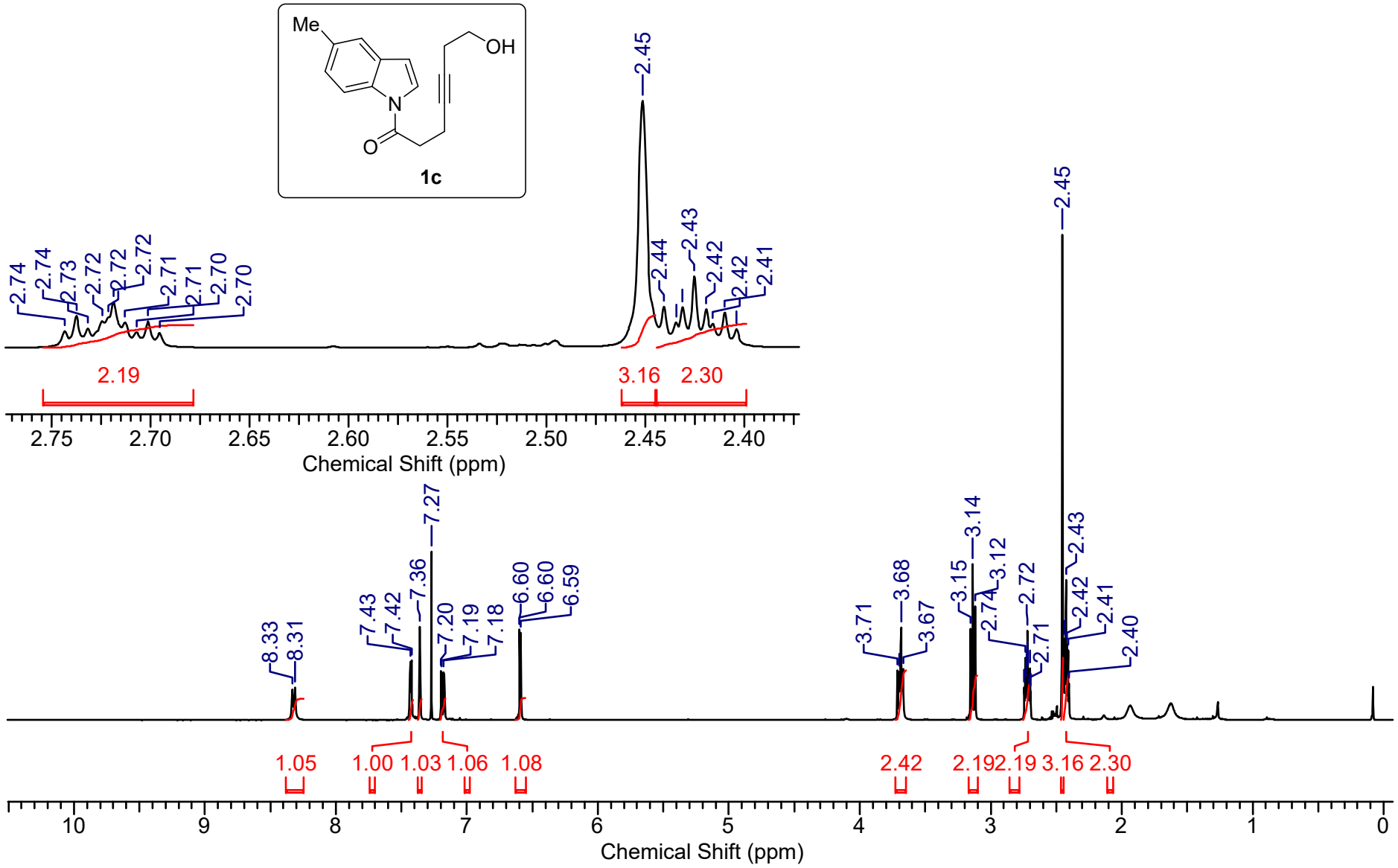

7.27
7.44
7.44
7.04
7.03
6.99
6.98
6.96
6.96
6.60
6.60
1.01
1.05
1.07
1.04
7.25
7.00
6.75
Chemical Shift (ppm)
3.87
MeO
OH
N
O
1b
8.37
8.34
7.27
7.44
7.44
7.04
7.03
6.99
6.60
6.60
6.59
6.59
3.69
3.68
3.15
3.13
3.11
2.72
2.72
2.43
2.43
2.42
2.41
2.40
1.00
1.01
1.05
1.07
1.04
3.18
2.20
2.08
2.10
2.11
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

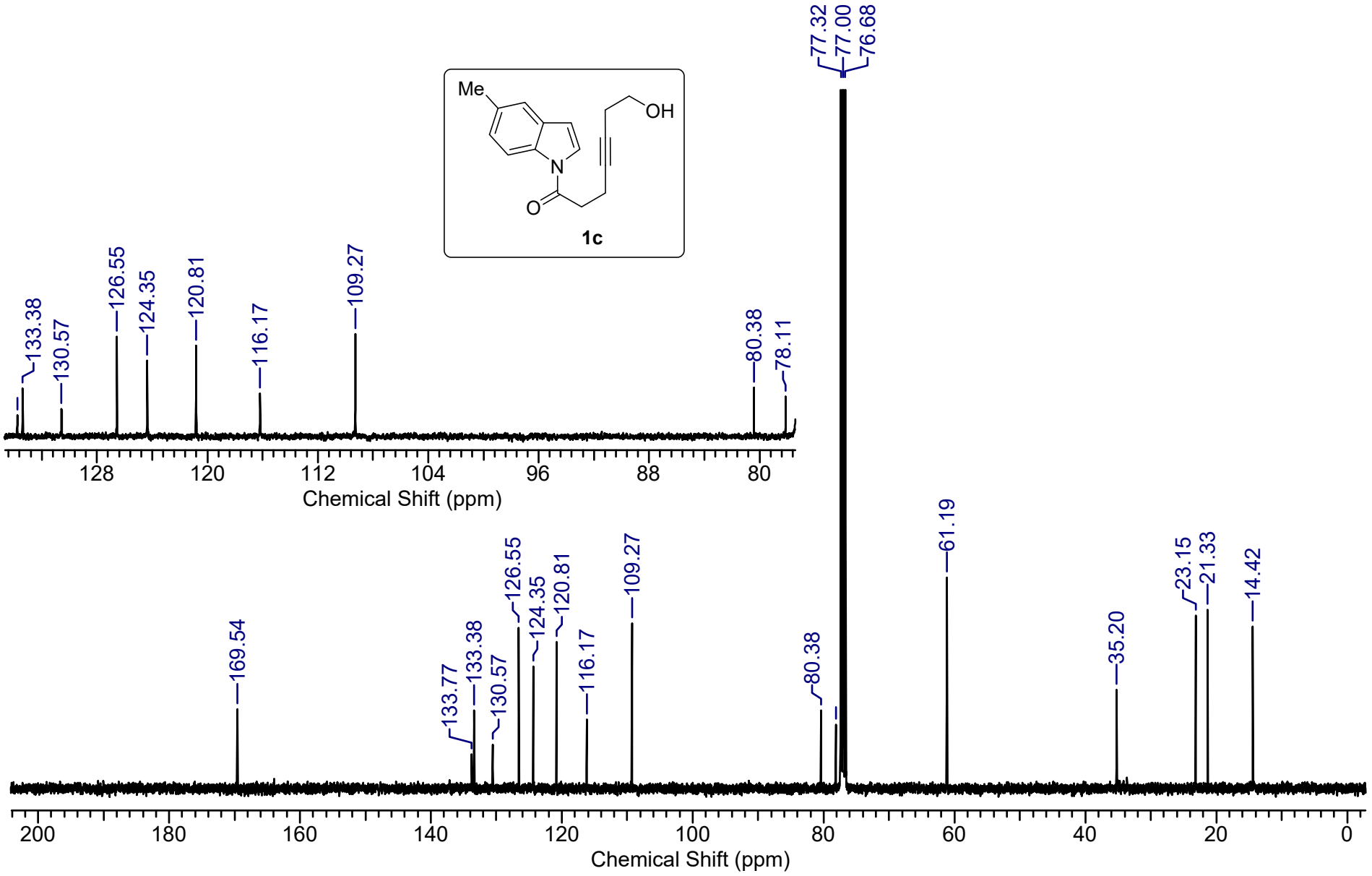

MeO
OH
N
O
1b
77.32
77.00
76.69
169.33
156.54
131.31
130.33
124.93
117.34
113.58
109.34
103.67
80.38
61.20
55.65
35.04
23.15
14.45
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

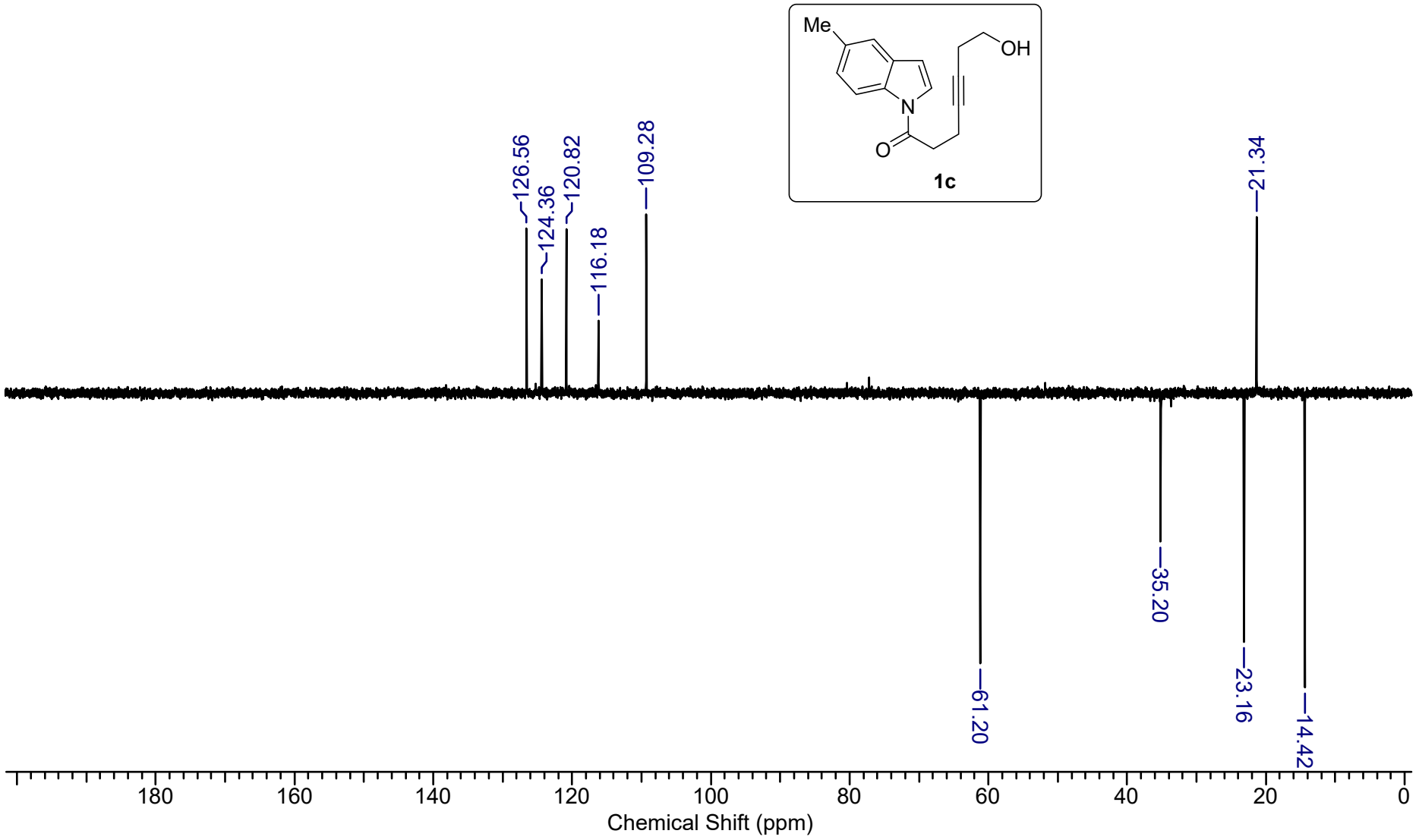

MeO
OH
N
O
1b
124.93
117.34
113.57
109.34
103.66
55.64
61.20
35.03
23.16
14.45
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-3 #263 RT: 1.41 AV: 1 NL: 3.55E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

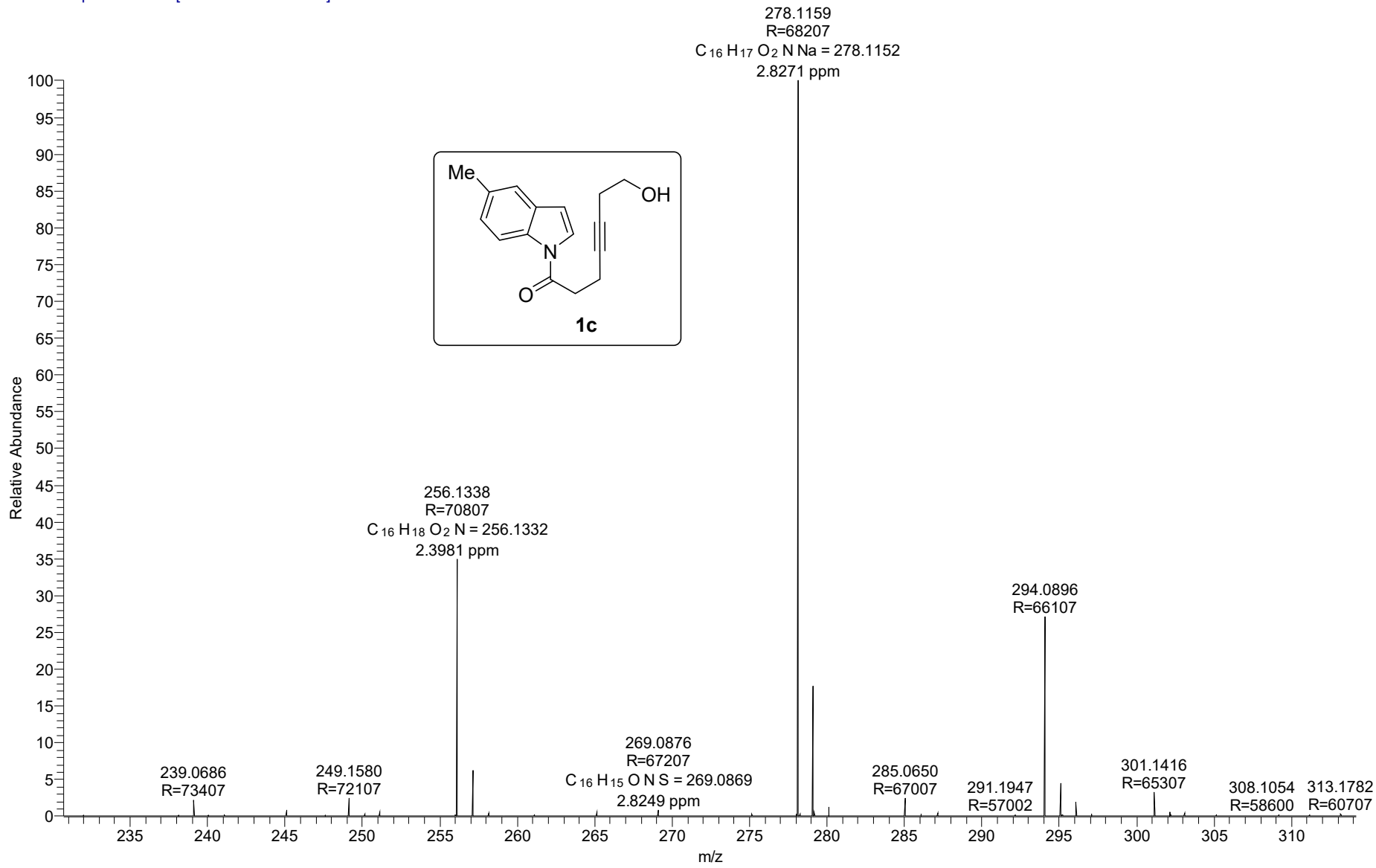


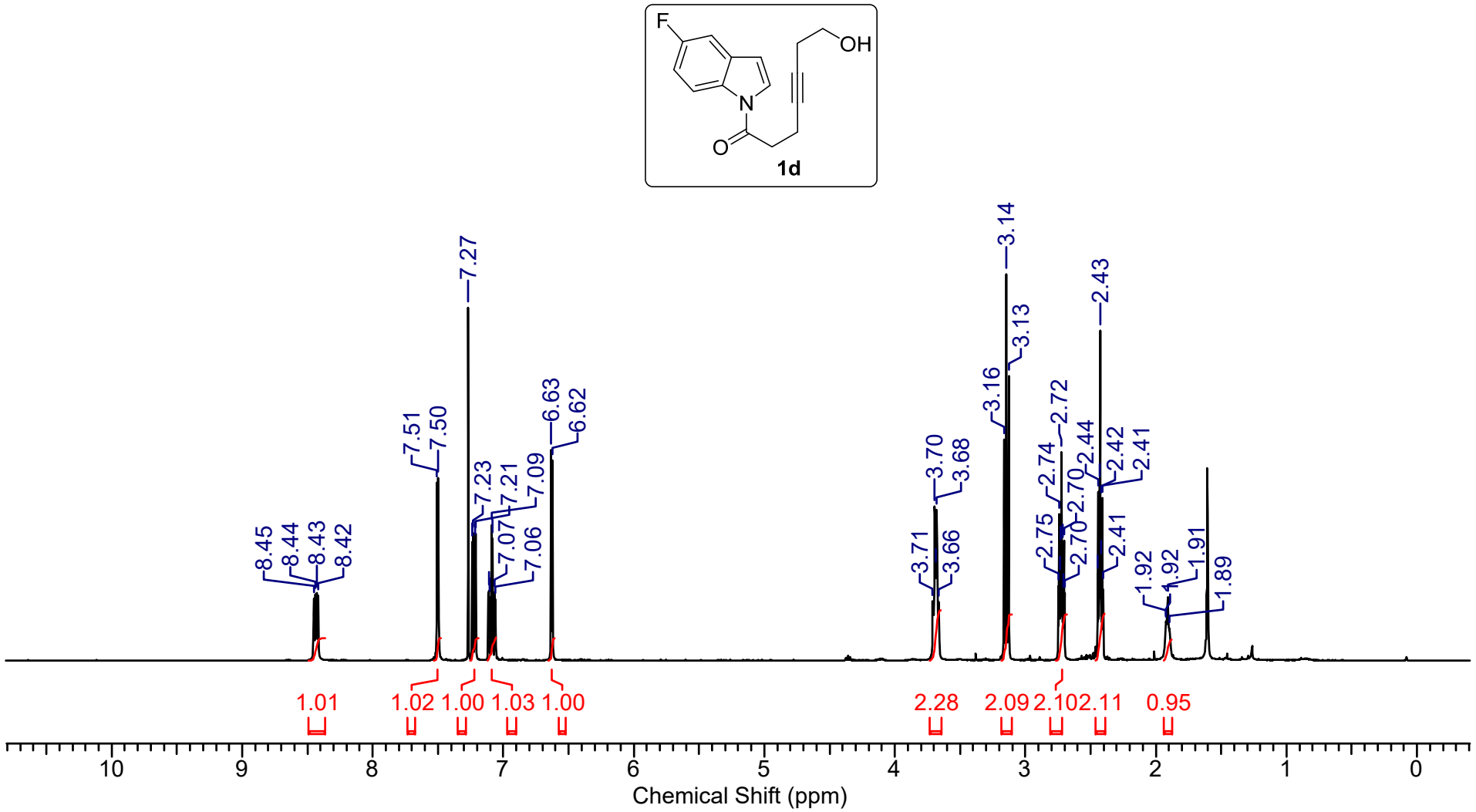

Me
OH
N
O
1c
2.74
2.74
2.73
2.72
2.72
2.72
2.71
2.71
2.70
2.70
2.45
2.44
2.43
2.42
2.42
2.41
2.19
3.16
2.30
2.75
2.70
2.65
2.60
2.55
2.50
2.45
2.40
Chemical Shift (ppm)
8.33
8.31
7.43
7.42
7.36
7.27
7.20
7.19
7.18
6.60
6.60
6.59
3.71
3.68
3.67
3.15
3.14
3.12
2.74
2.72
2.71
2.45
2.43
2.42
2.41
2.40
1.05
1.00
1.03
1.06
1.08
2.42
2.19
2.19
3.16
2.30
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

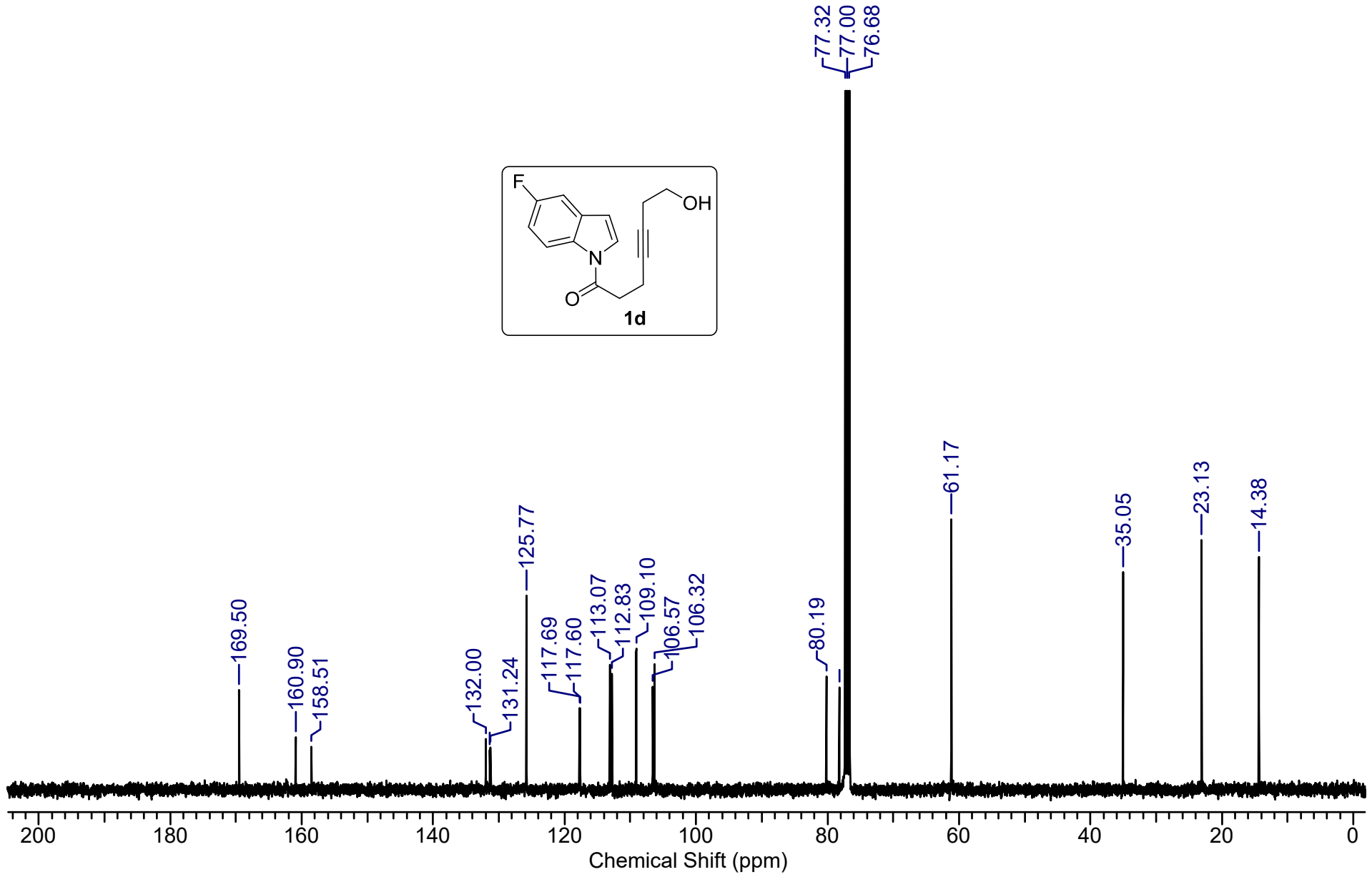

Me
OH
N
O
1c
77.32
77.00
76.68
133.38
130.57
126.55
124.35
120.81
116.17
109.27
80.38
78.11
128
120
112
104
96
88
80
Chemical Shift (ppm)
169.54
133.77
133.38
130.57
126.55
124.35
120.81
116.17
109.27
80.38
61.19
35.20
23.15
21.33
14.42
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

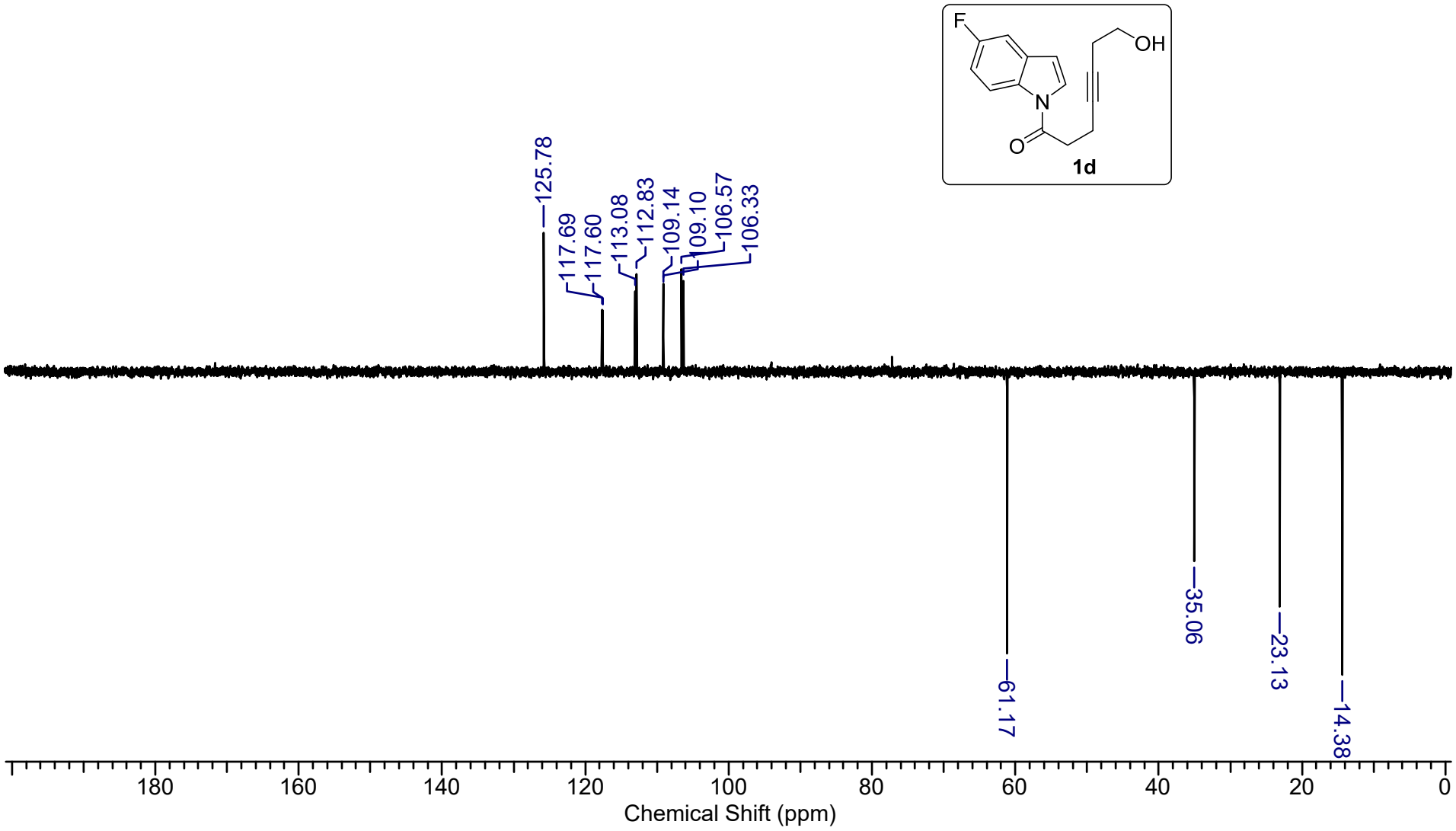

Me
OH
N
O
1c
126.56
124.36
120.82
116.18
109.28
71.34
61.20
35.20
23.16
14.42
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

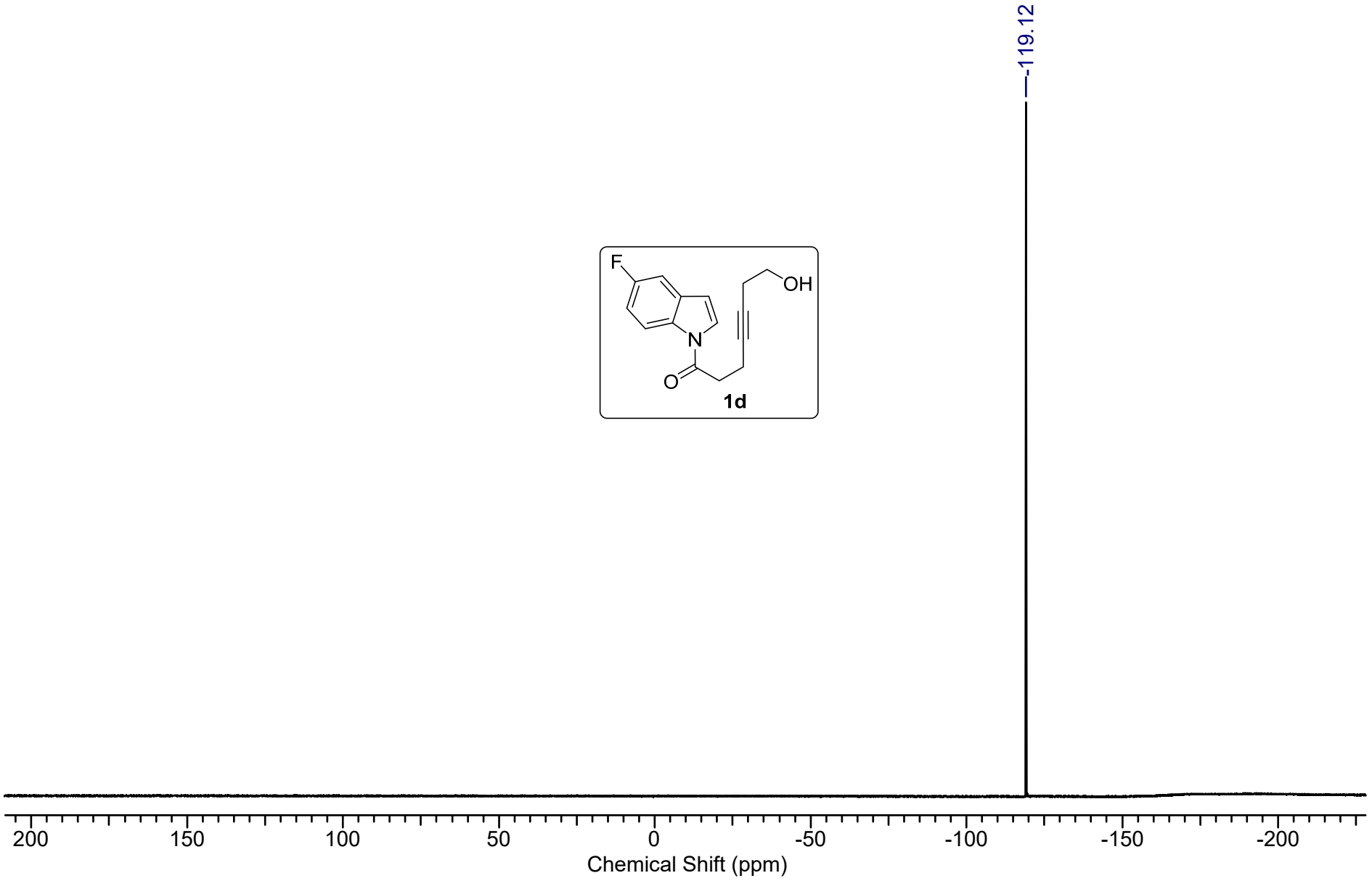
MS-2 #286 RT: 1.53 AV: 1 NL: 2.78E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

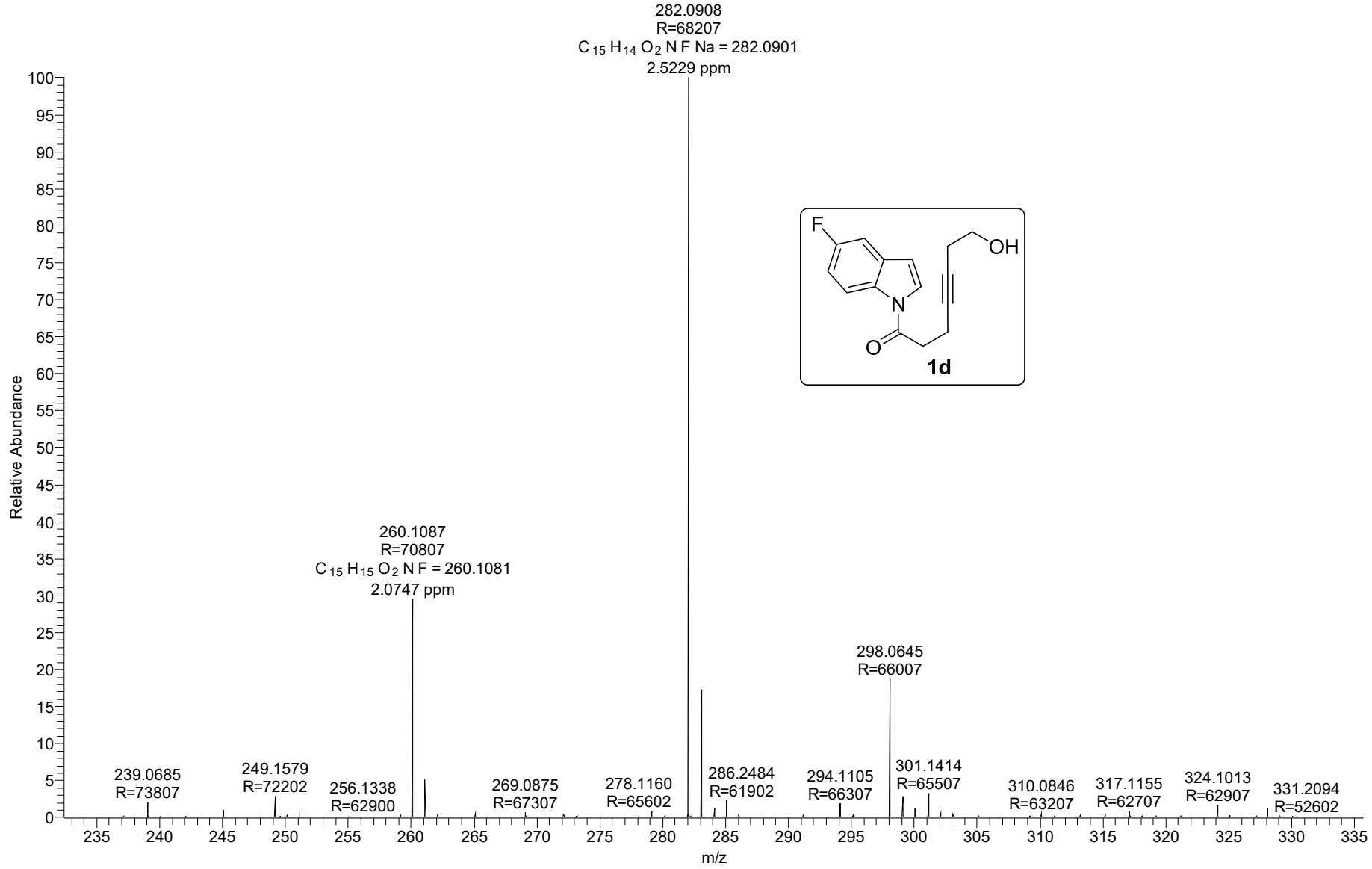


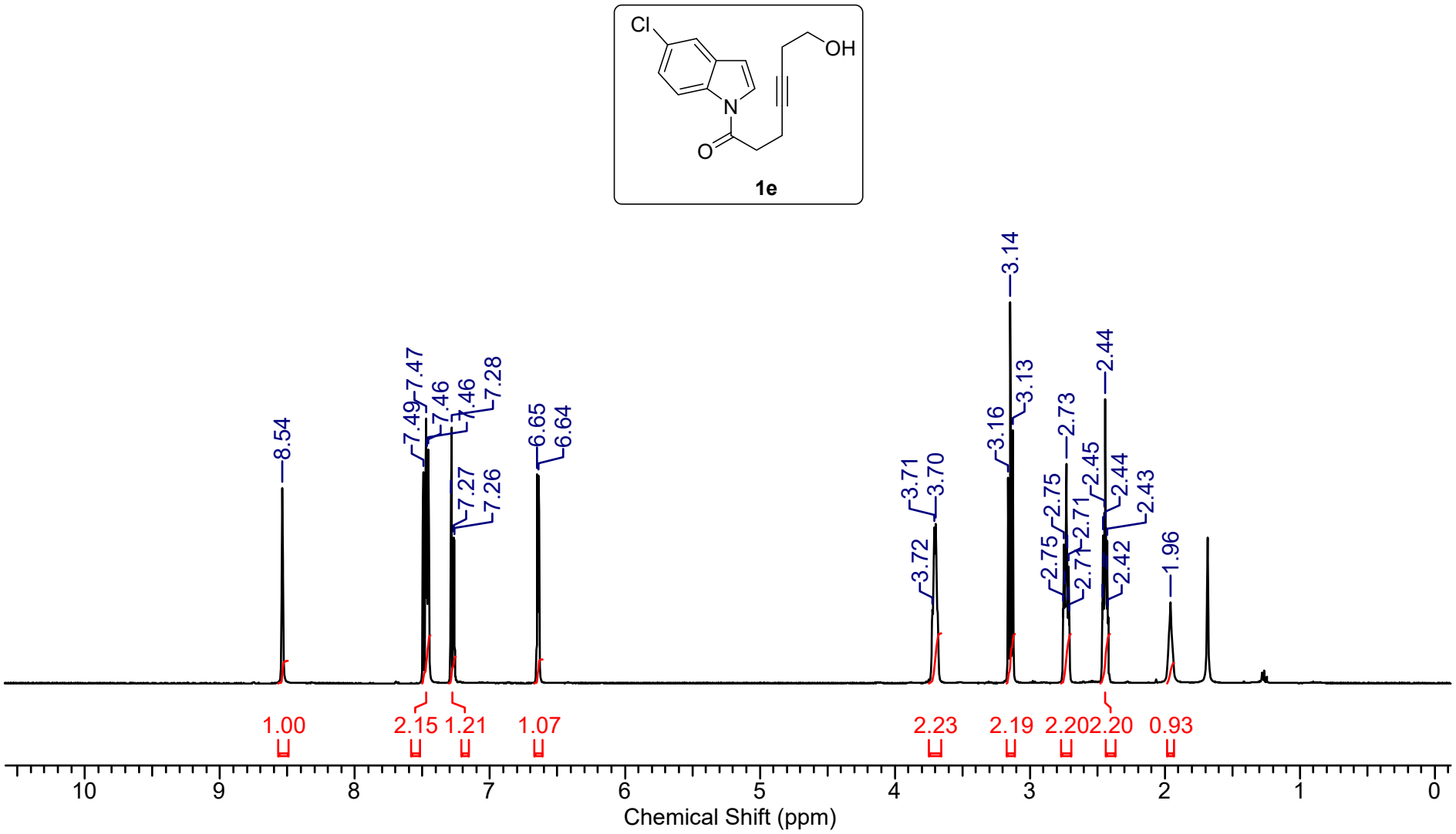

F
OH
N
O
1d
8.45
8.44
8.43
8.42
7.51
7.50
7.27
7.23
7.21
7.09
7.07
7.06
6.63
6.62
3.71
3.70
3.68
3.66
3.16
3.14
3.13
2.75
2.74
2.72
2.70
2.70
2.44
2.43
2.42
2.41
2.41
1.92
1.92
1.91
1.89
1.01
1.02
1.00
1.03
1.00
2.28
2.09
2.10
2.11
0.95
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

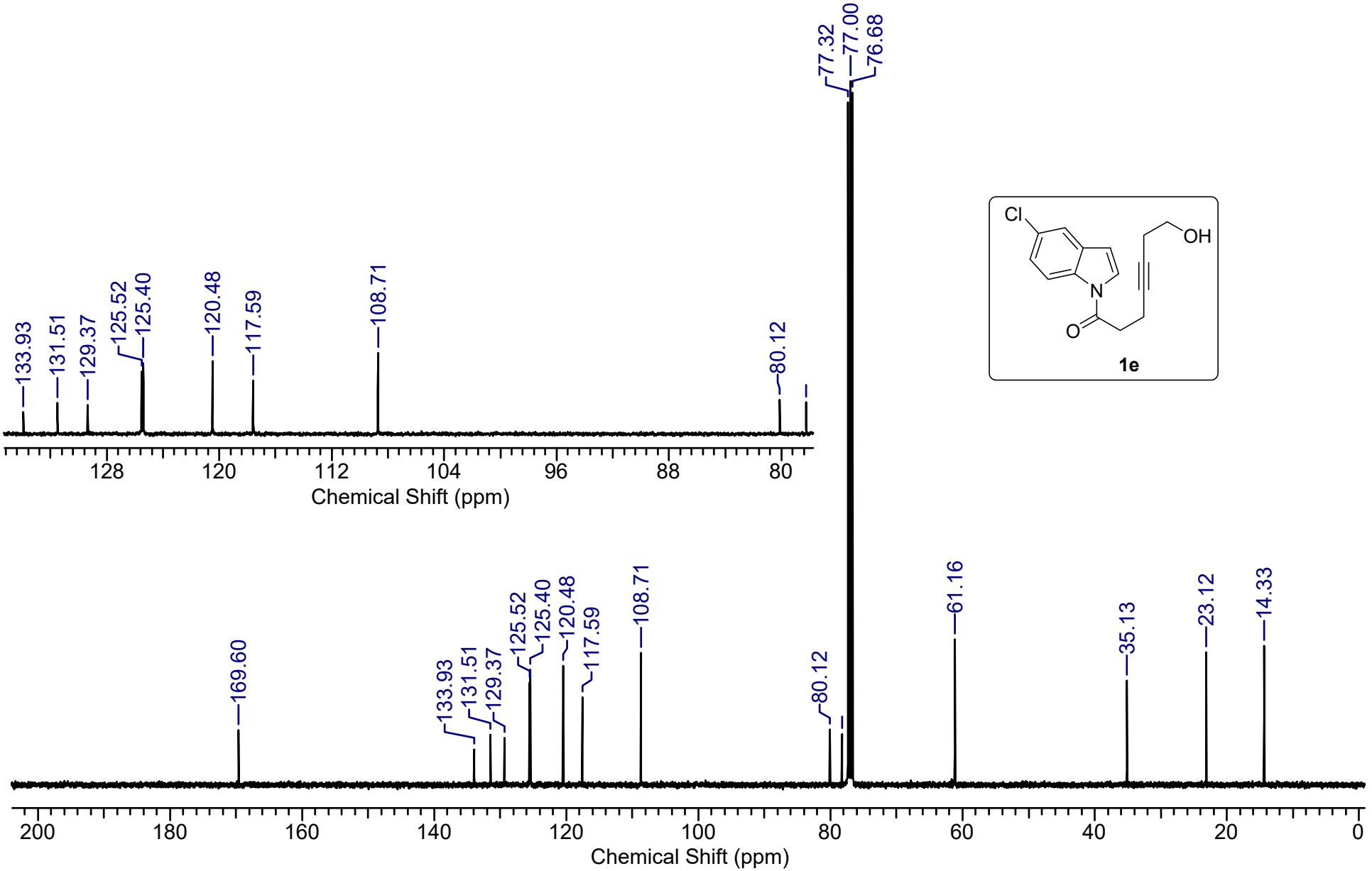

77.32
77.00
76.68
F
OH
N
O
1d
169.50
160.90
158.51
132.00
131.24
125.77
117.69
117.60
113.07
112.83
109.10
106.57
106.32
80.19
61.17
35.05
23.13
14.38
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

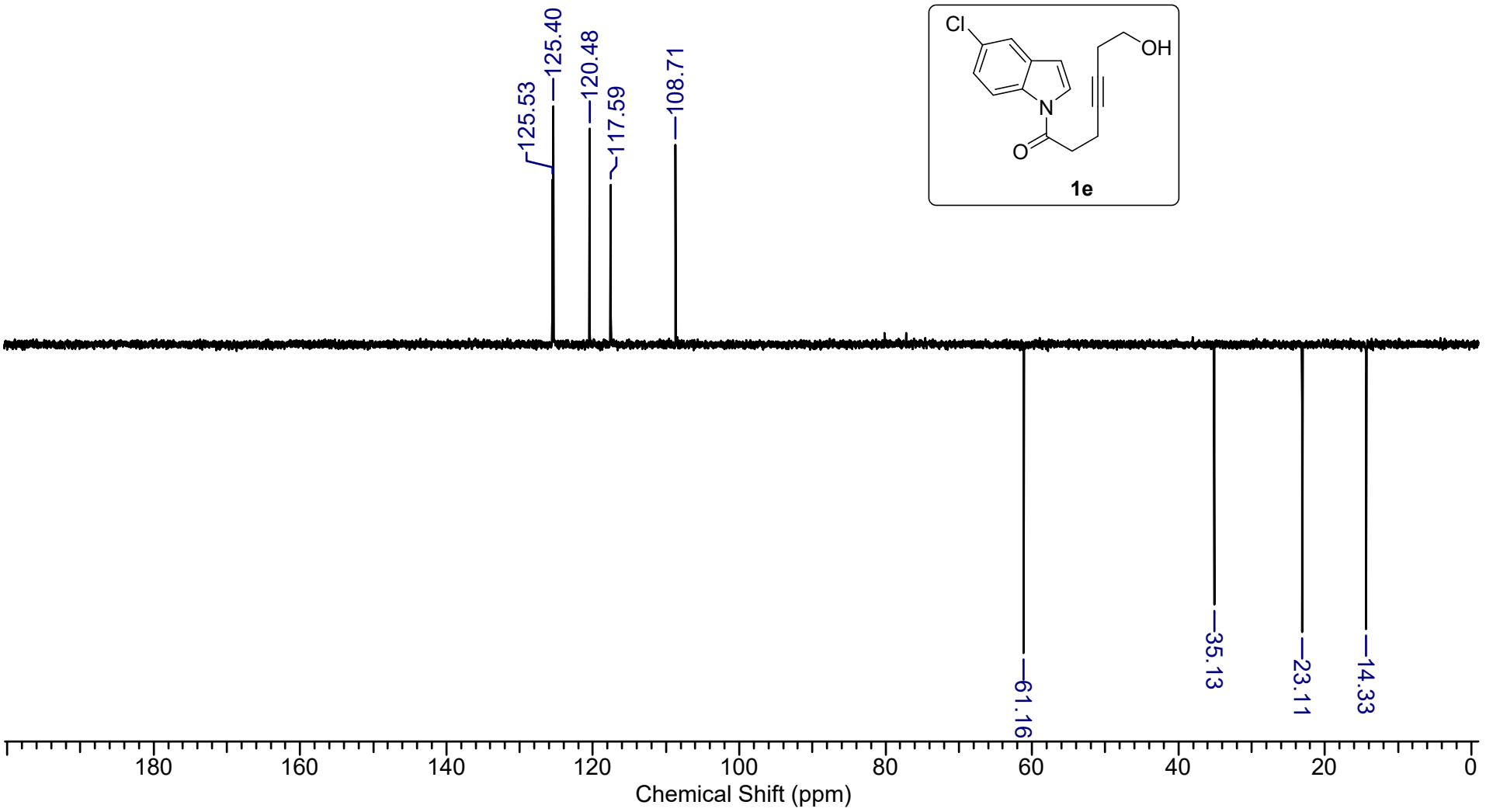

F
OH
N
O
1d
125.78
117.69
117.60
113.08
112.83
109.14
109.10
106.57
106.33
61.17
35.06
23.13
14.38
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

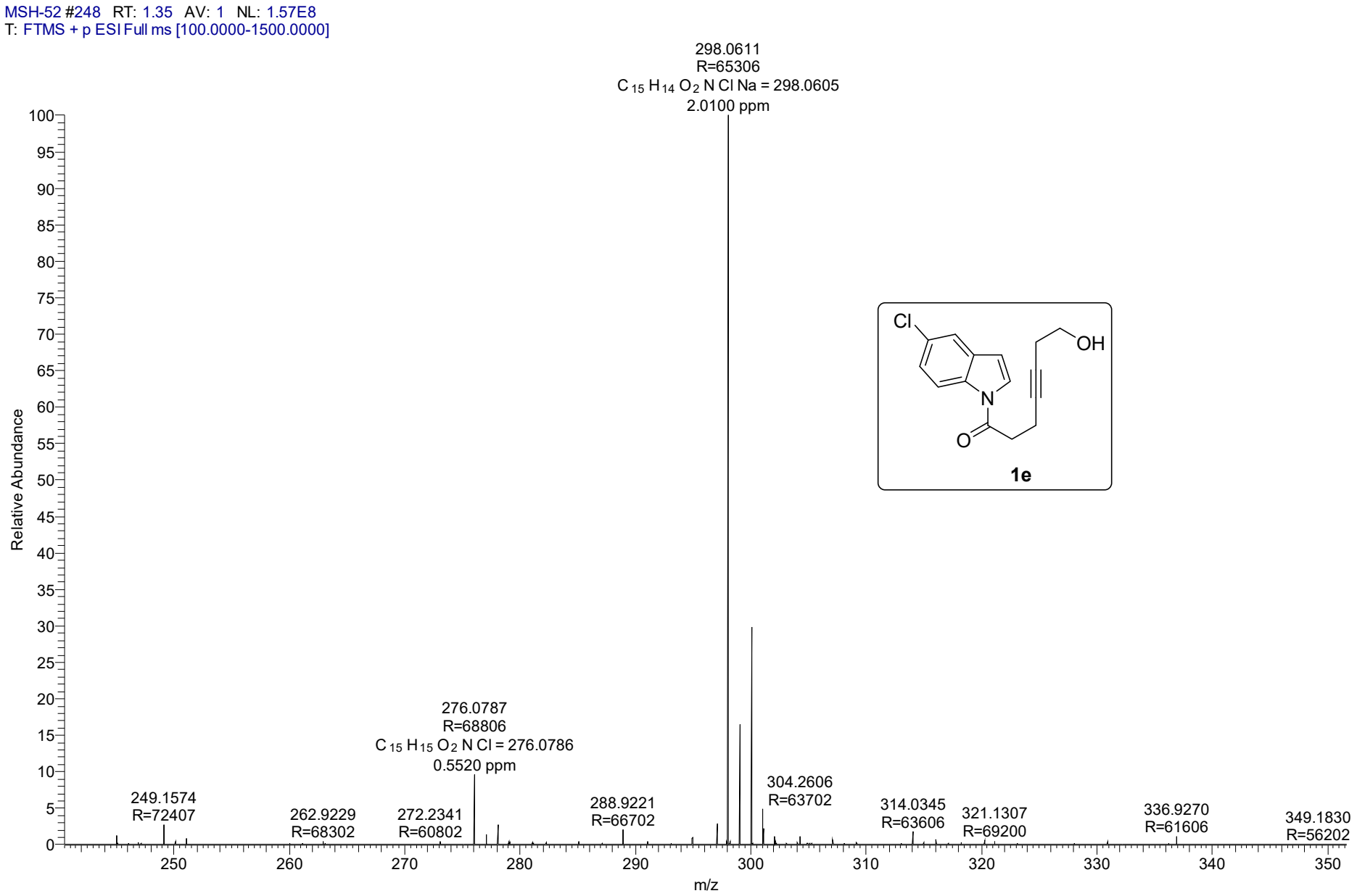

-119.12
F
OH
N
O
1d
200
150
100
50
0
-50
-100
-150
-200
Chemical Shift (ppm)

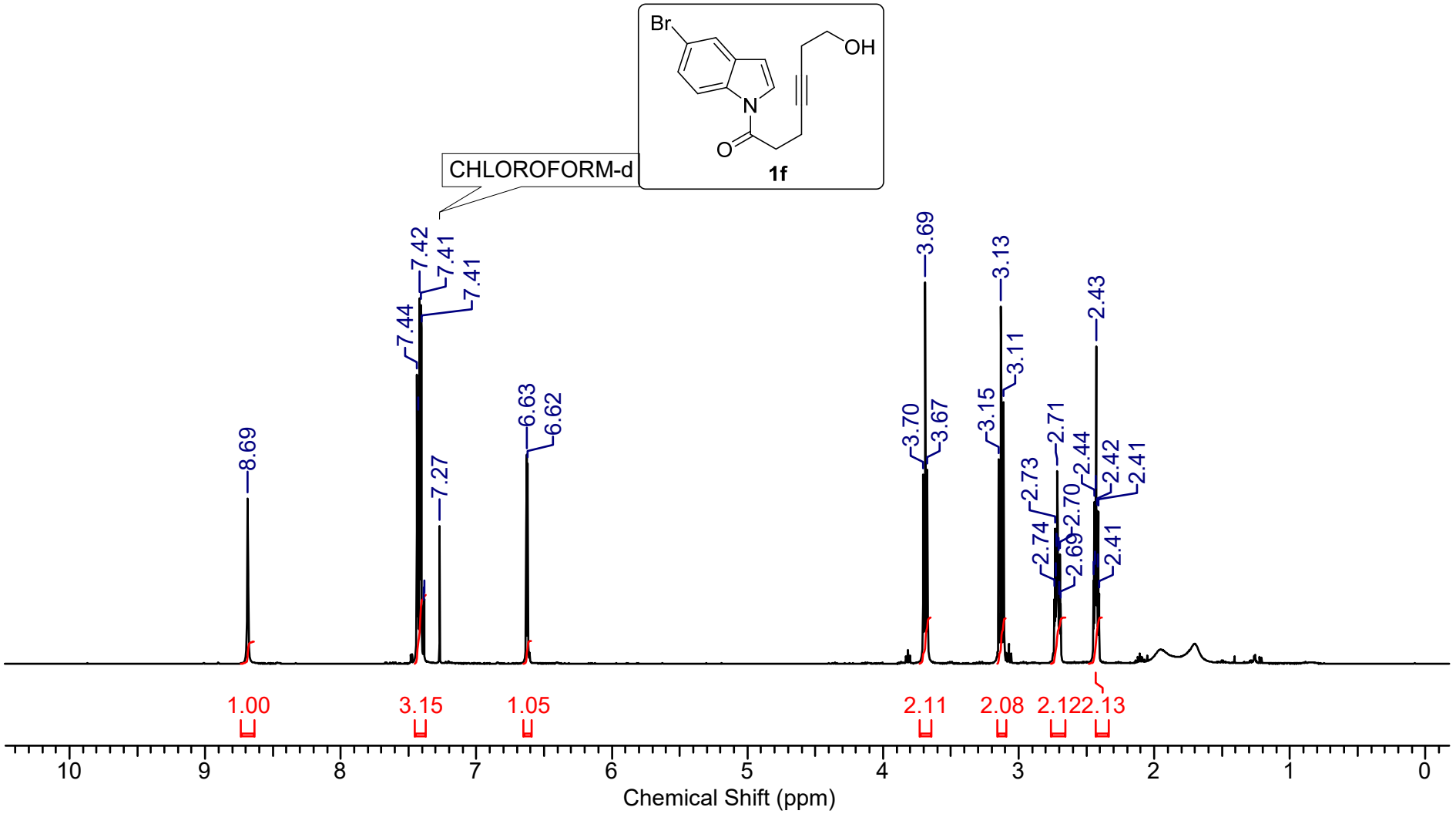

MS-4 #266 RT: 1.42 AV: 1 NL: 2.80E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
282.0908
R=68207
C 15 H 14 O 2 N F Na = 282.0901
2.5229 ppm
260.1087
R=70807
C 15 H 15 O 2 N F = 260.1081
2.0747 ppm
298.0645
R=66007
239.0685
R=73807
249.1579
R=72202
256.1338
R=62900
269.0875
R=67307
278.1160
R=65602
286.2484
R=61902
294.1105
R=66307
301.1414
R=65507
310.0846
R=63207
317.1155
R=62707
324.1013
R=62907
331.2094
R=52602
F
OH
N
O
1d
Relative Abundance
m/z

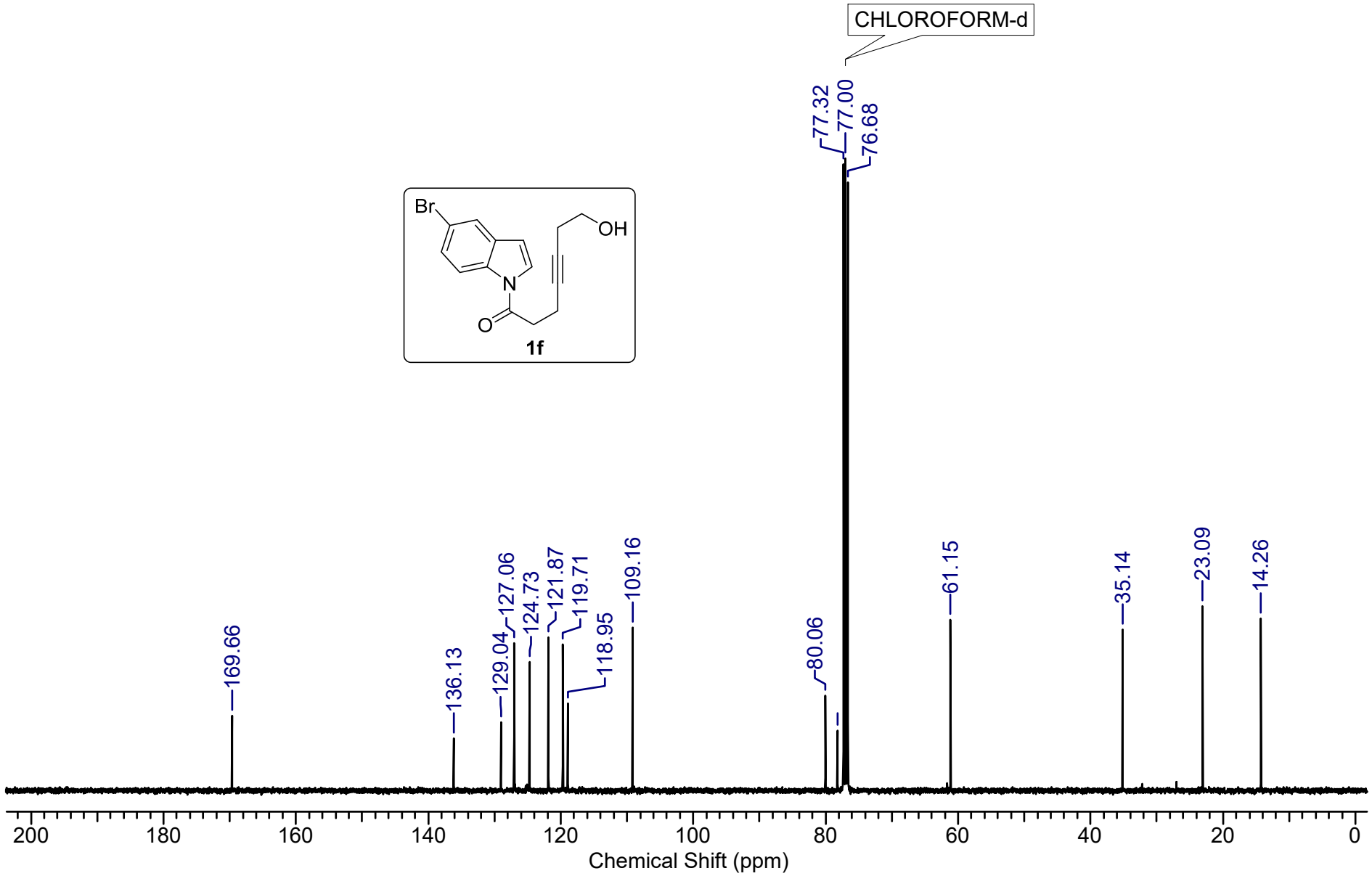

Cl
OH
N
O
1e
8.54
7.49
7.47
7.46
7.46
7.28
7.27
7.26
6.65
6.64
3.71
3.70
3.72
3.16
3.14
3.13
2.75
2.73
2.71
2.45
2.44
2.43
2.75
2.71
2.42
2.44
1.96
1.00
2.15
1.21
1.07
2.23
2.19
2.20
2.20
0.93
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

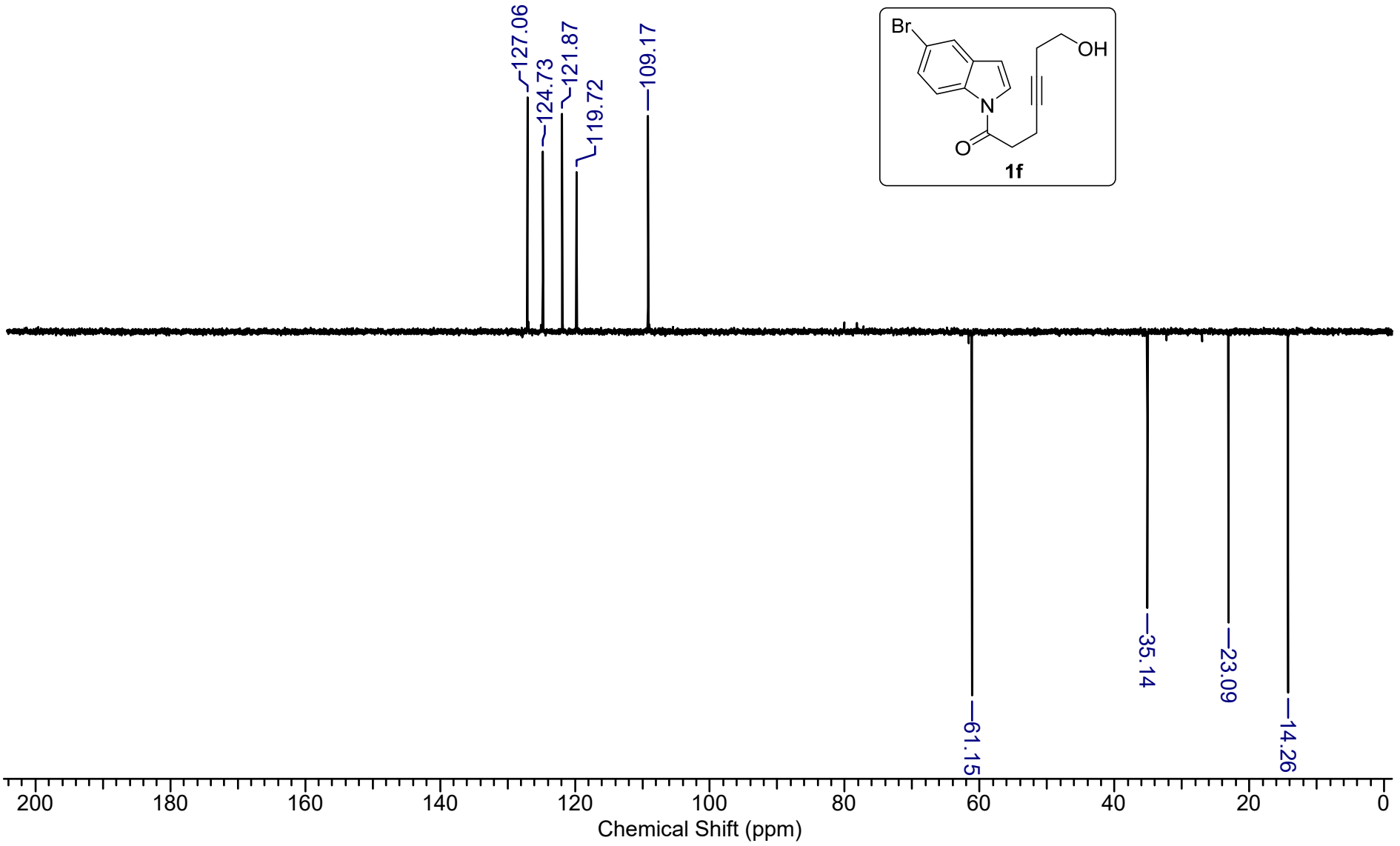

77.32
77.00
76.68
133.93
131.51
129.37
125.52
125.40
120.48
117.59
108.71
80.12
Cl
OH
N
O
1e
128
120
112
104
96
88
80
Chemical Shift (ppm)
169.60
133.93
131.51
129.37
125.52
125.40
120.48
117.59
108.71
80.12
61.16
35.13
23.12
14.33
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


125.53
125.40
120.48
117.59
108.71
61.16
35.13
23.11
14.33
Cl
OH
N
O
1e
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

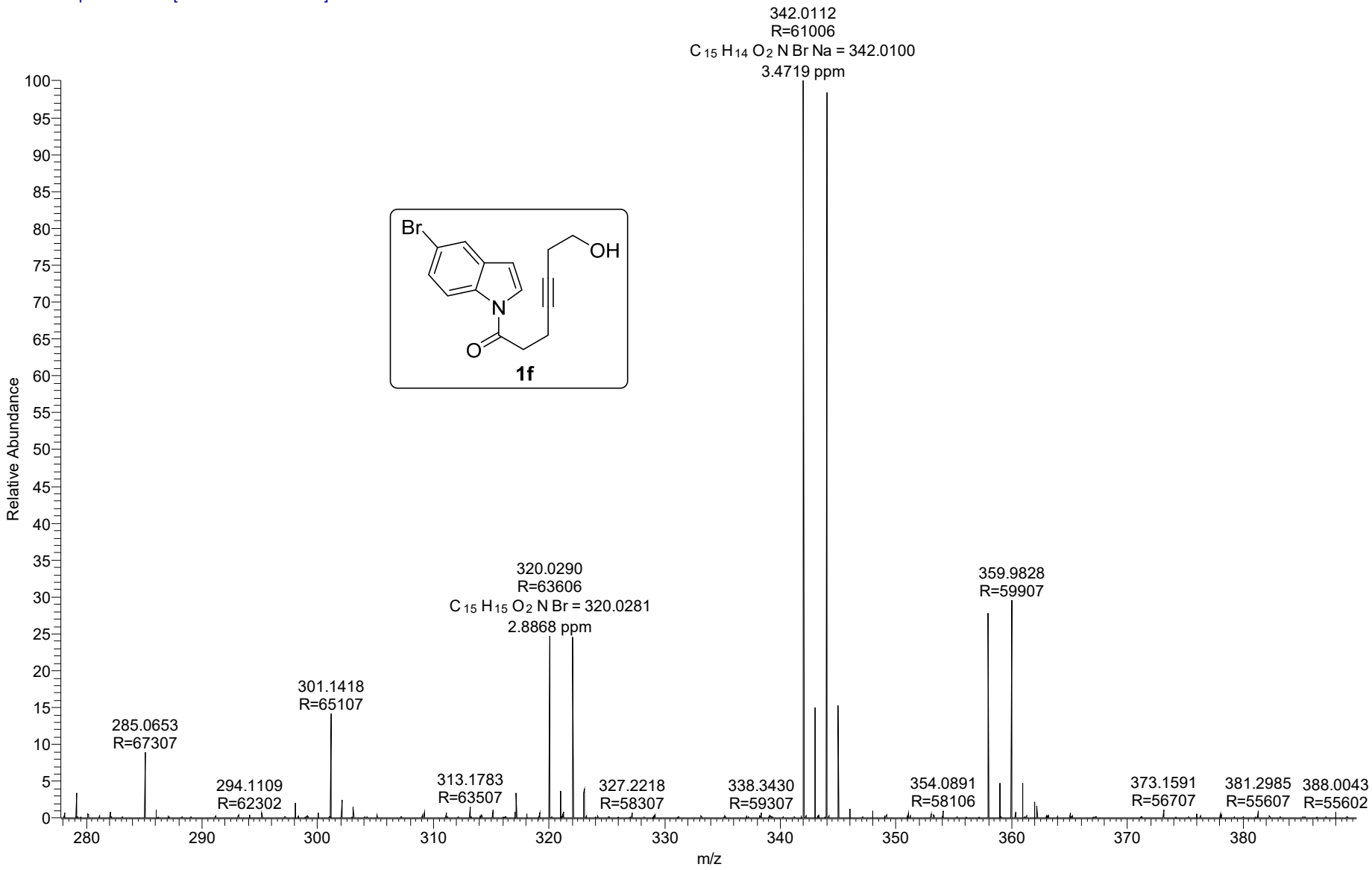

MSH-52 #248 RT: 1.35 AV: 1 NL: 1.57E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
298.0611
R=65306
C 15 H 14 O 2 N Cl Na = 298.0605
2.0100 ppm
276.0787
R=68806
C 15 H 15 O 2 N Cl = 276.0786
0.5520 ppm
249.1574
R=72407
262.9229
R=68302
272.2341
R=60802
288.9221
R=66702
304.2606
R=63702
314.0345
R=63606
321.1307
R=69200
336.9270
R=61606
349.1830
R=56202
Relative Abundance
m/z
Cl
OH
N
O
1e

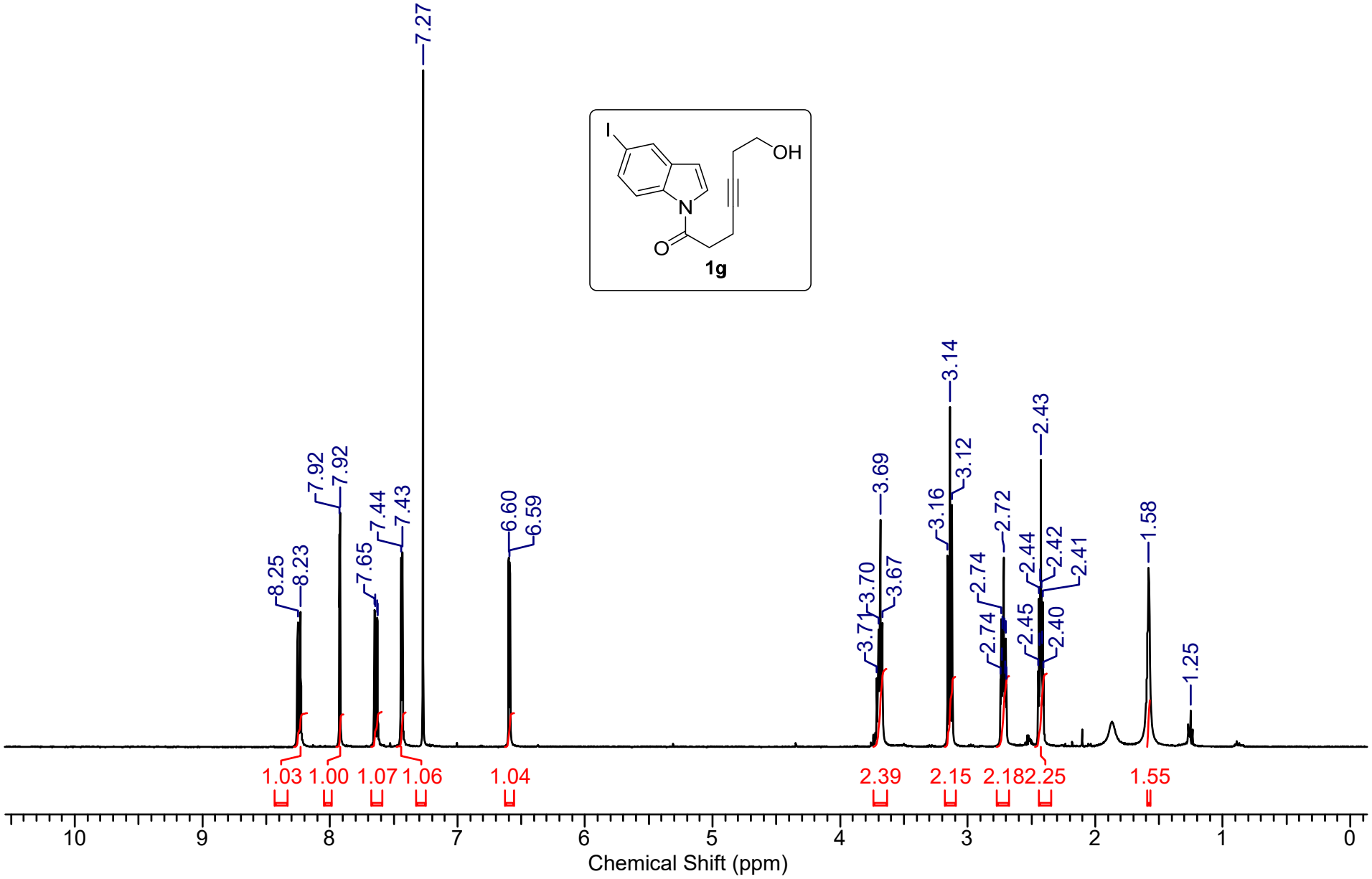

Br
OH
N
O
1f
CHLOROFORM-d
8.69
7.44
7.42
7.41
7.41
7.27
6.63
6.62
3.70
3.69
3.67
3.15
3.13
3.11
2.74
2.73
2.71
2.70
2.69
2.44
2.43
2.42
2.41
2.41
1.00
3.15
1.05
2.11
2.08
2.12
2.13
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

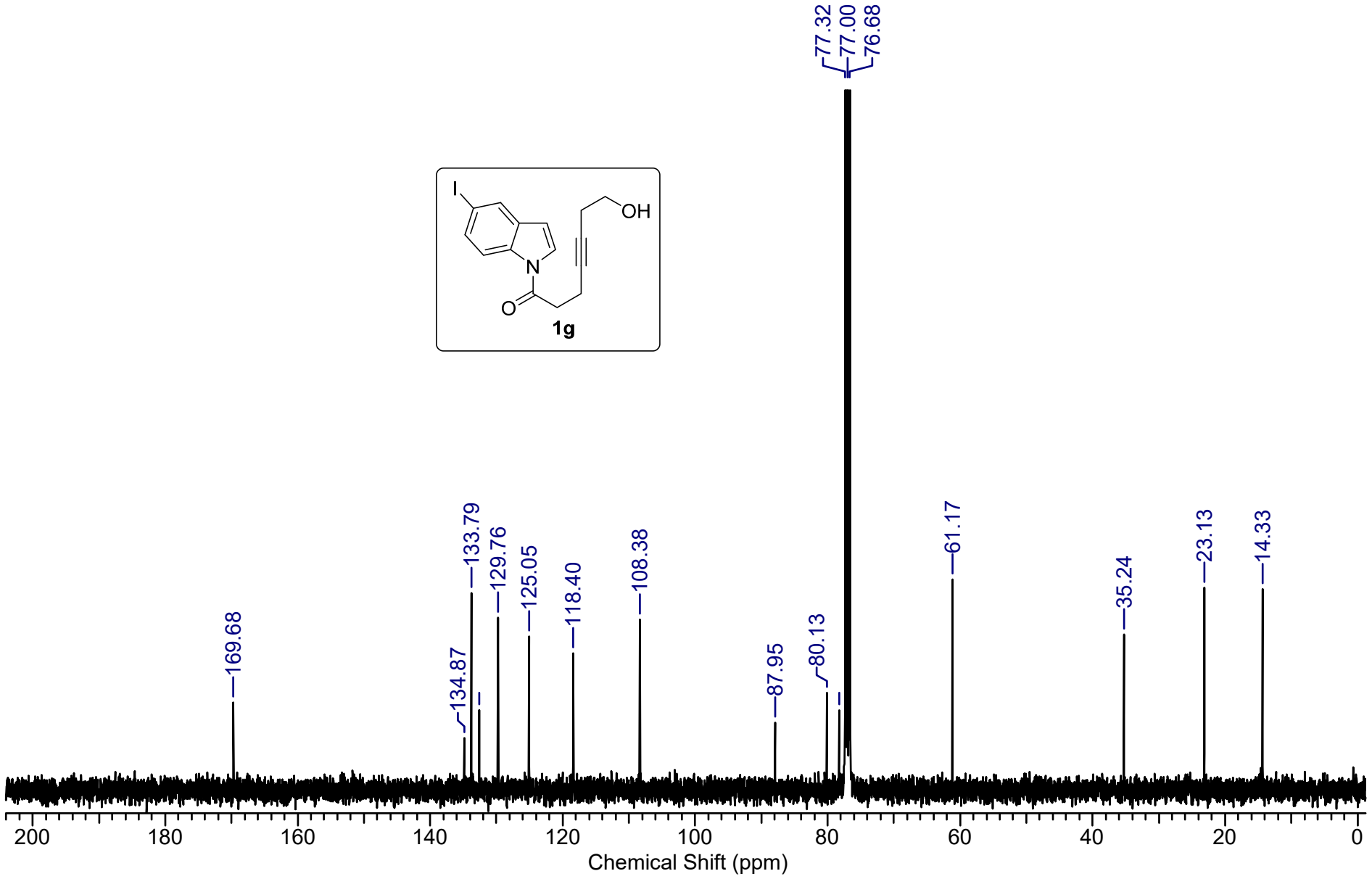

CHLOROFORM-d
77.32
77.00
76.68
Br
OH
N
O
1f
169.66
136.13
129.04
127.06
124.73
121.87
119.71
118.95
109.16
80.06
61.15
35.14
23.09
14.26
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

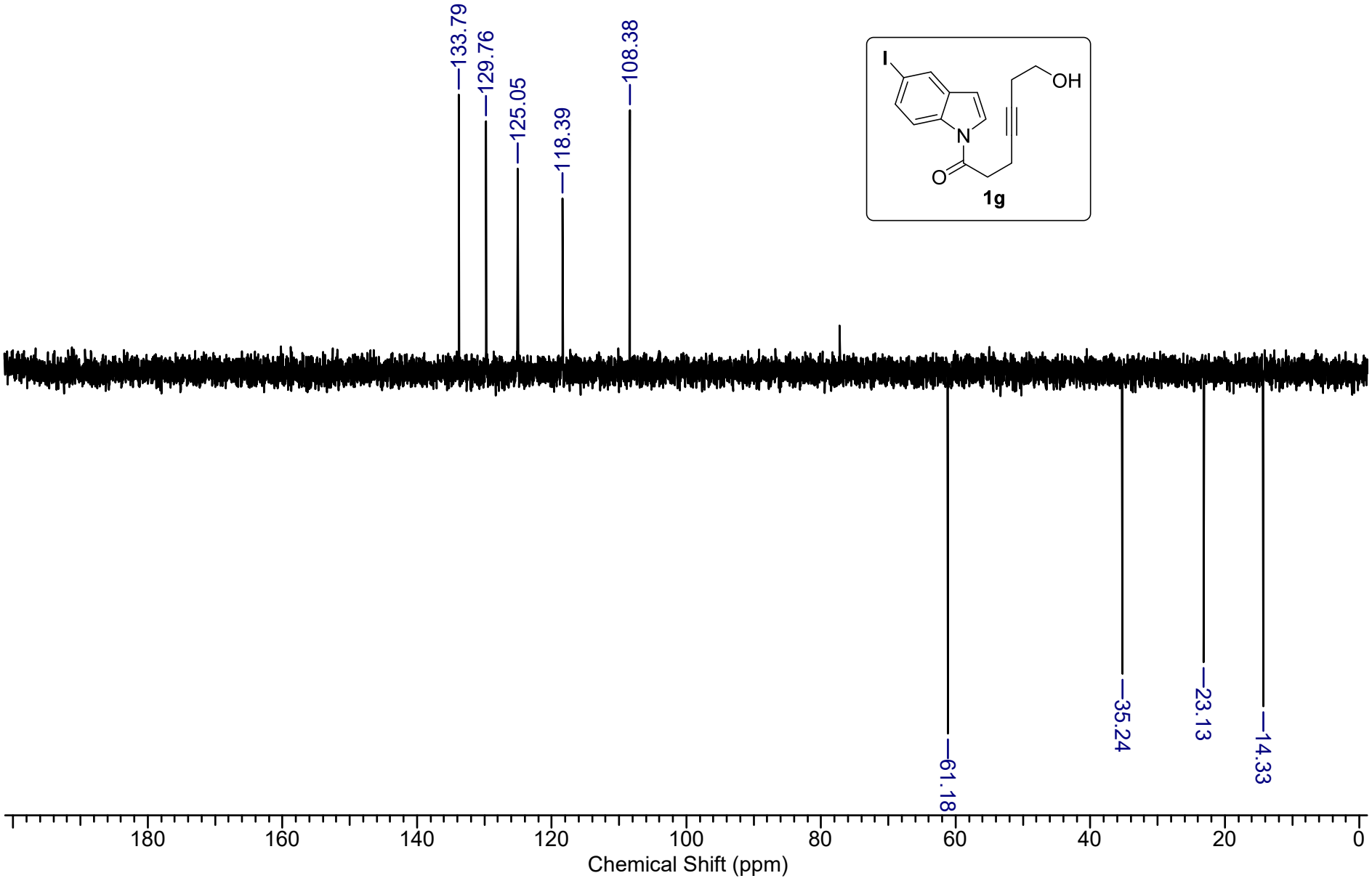

127.06
124.73
121.87
119.72
109.17
61.15
35.14
23.09
14.26
Br
OH
N
O
1f
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

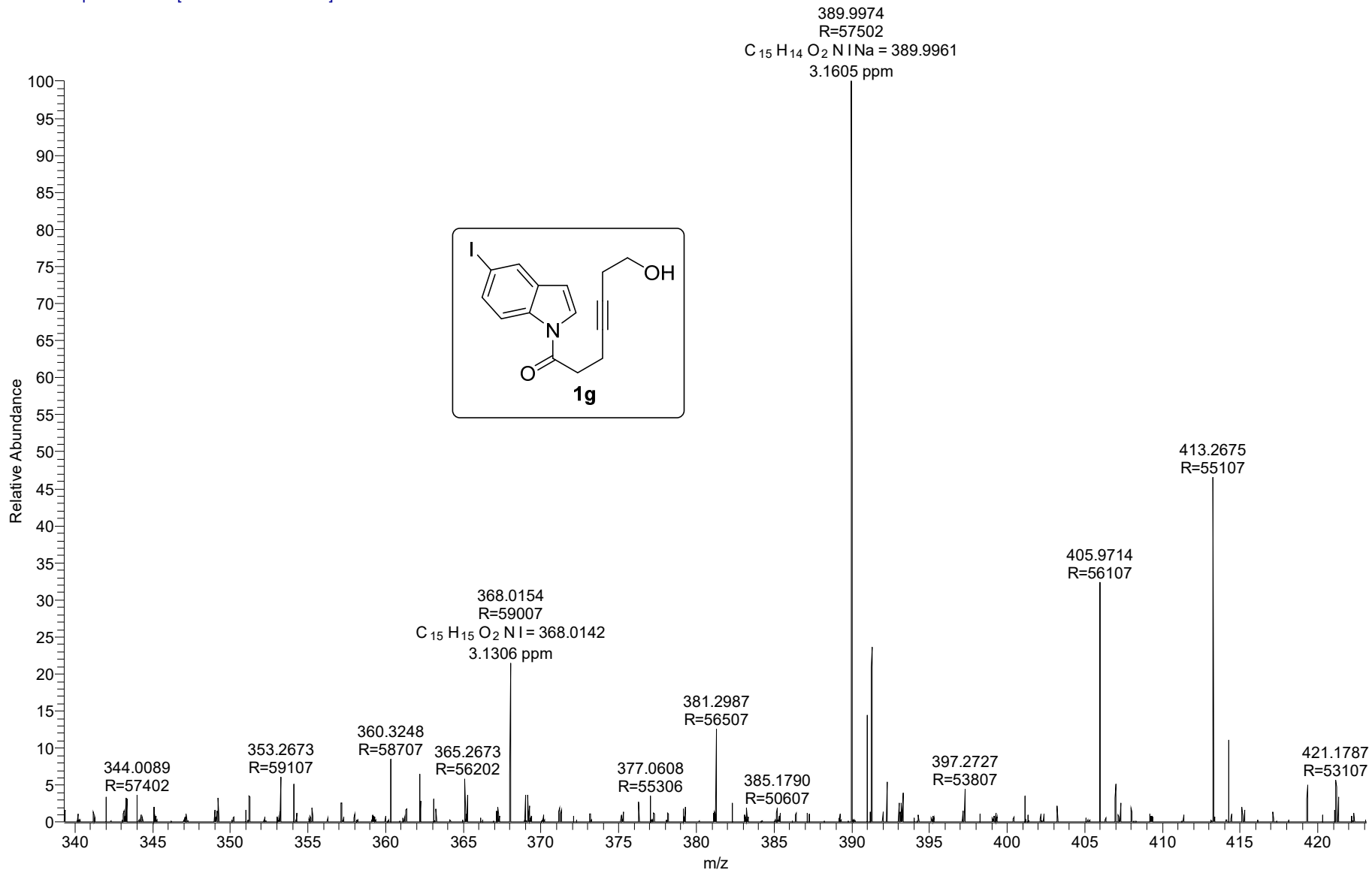
MS-6 #282 RT: 1.51 AV: 1 NL: 7.17E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

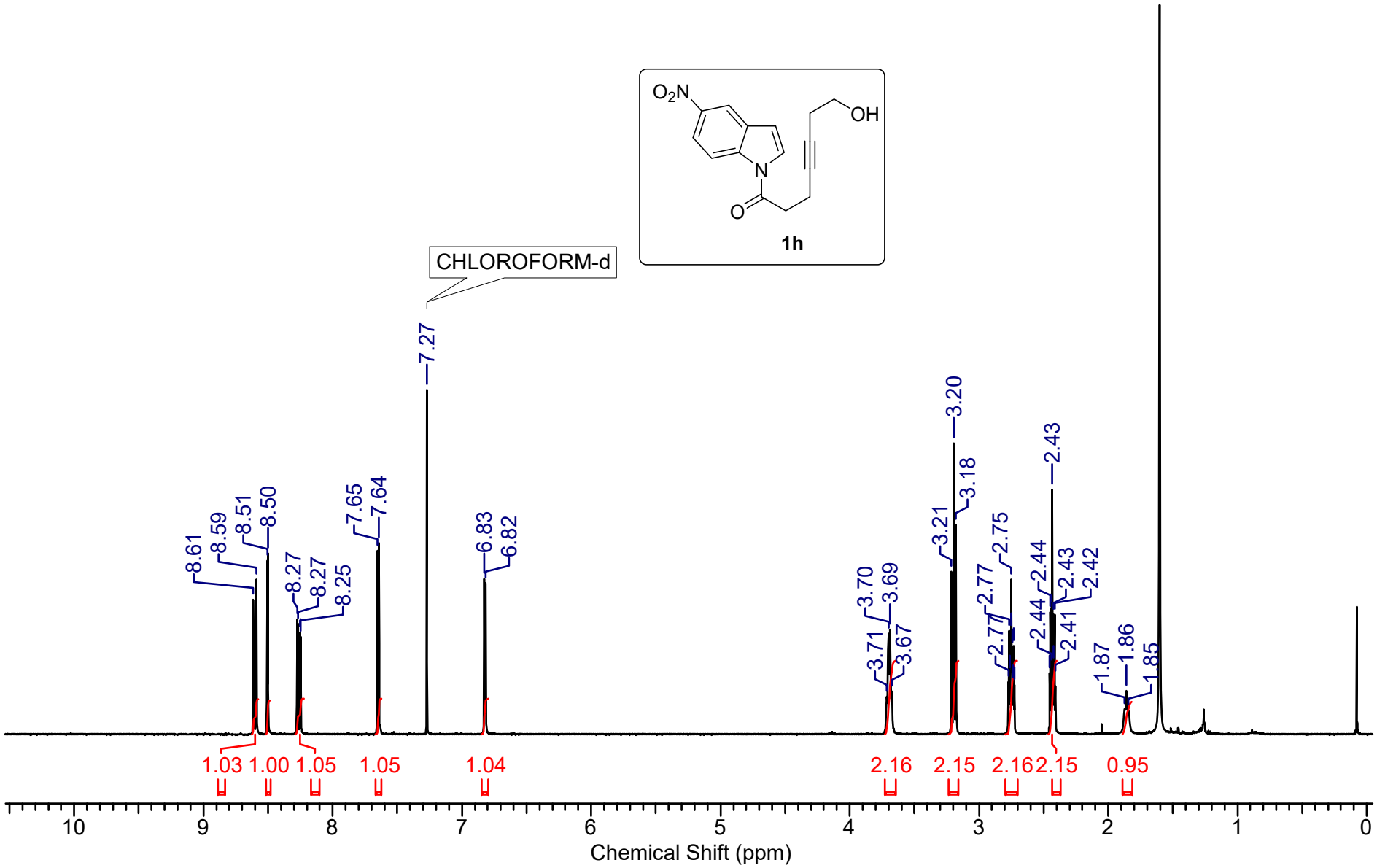


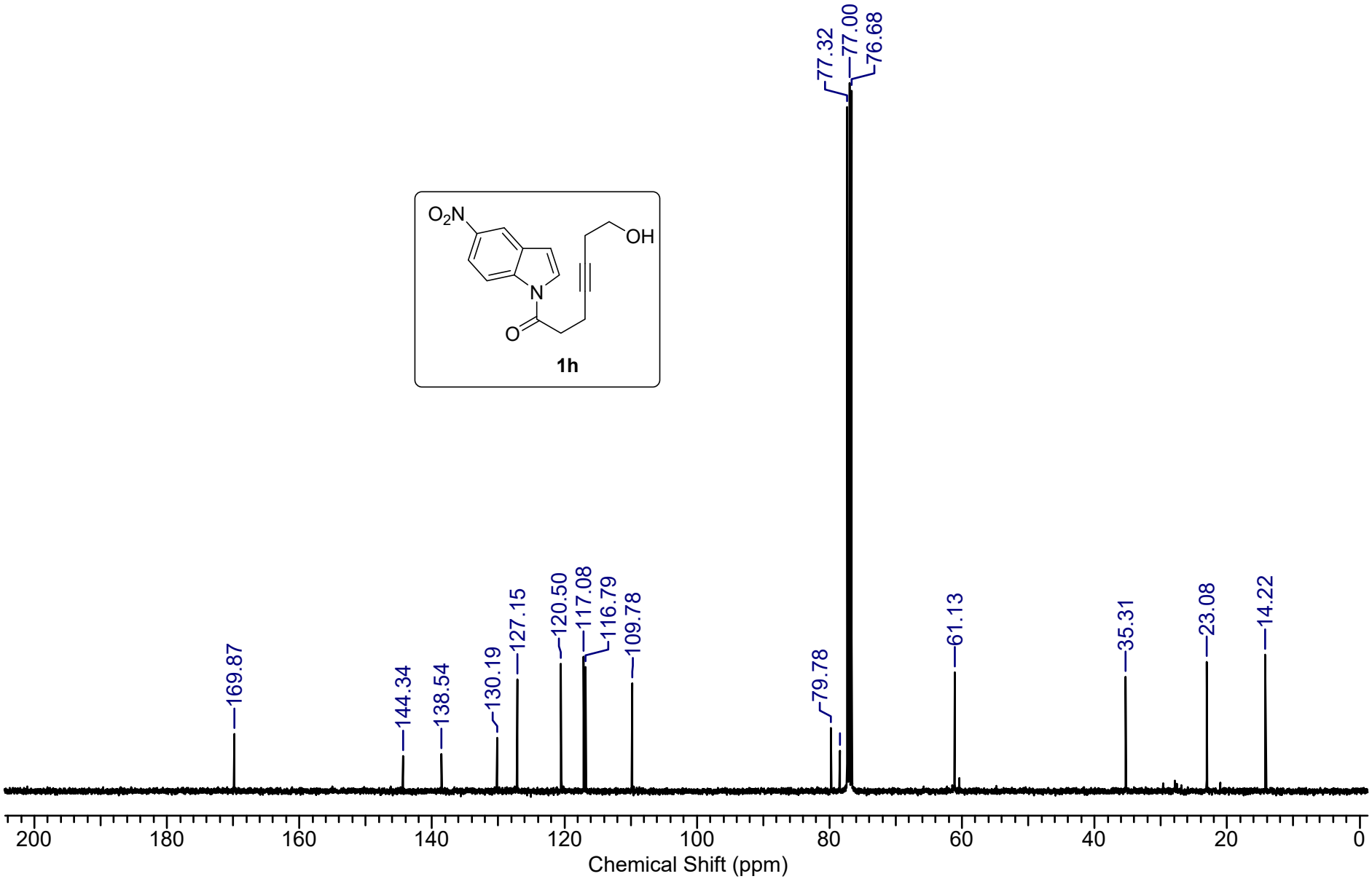

I
OH
N
O
1g
7.27
8.25
8.23
7.92
7.92
7.65
7.44
7.43
6.60
6.59
3.69
3.71
3.70
3.67
3.16
3.14
3.12
2.74
2.74
2.72
2.45
2.44
2.43
2.42
2.41
2.40
1.58
1.25
1.03
1.00
1.07
1.06
1.04
2.39
2.15
2.18
2.25
1.55
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

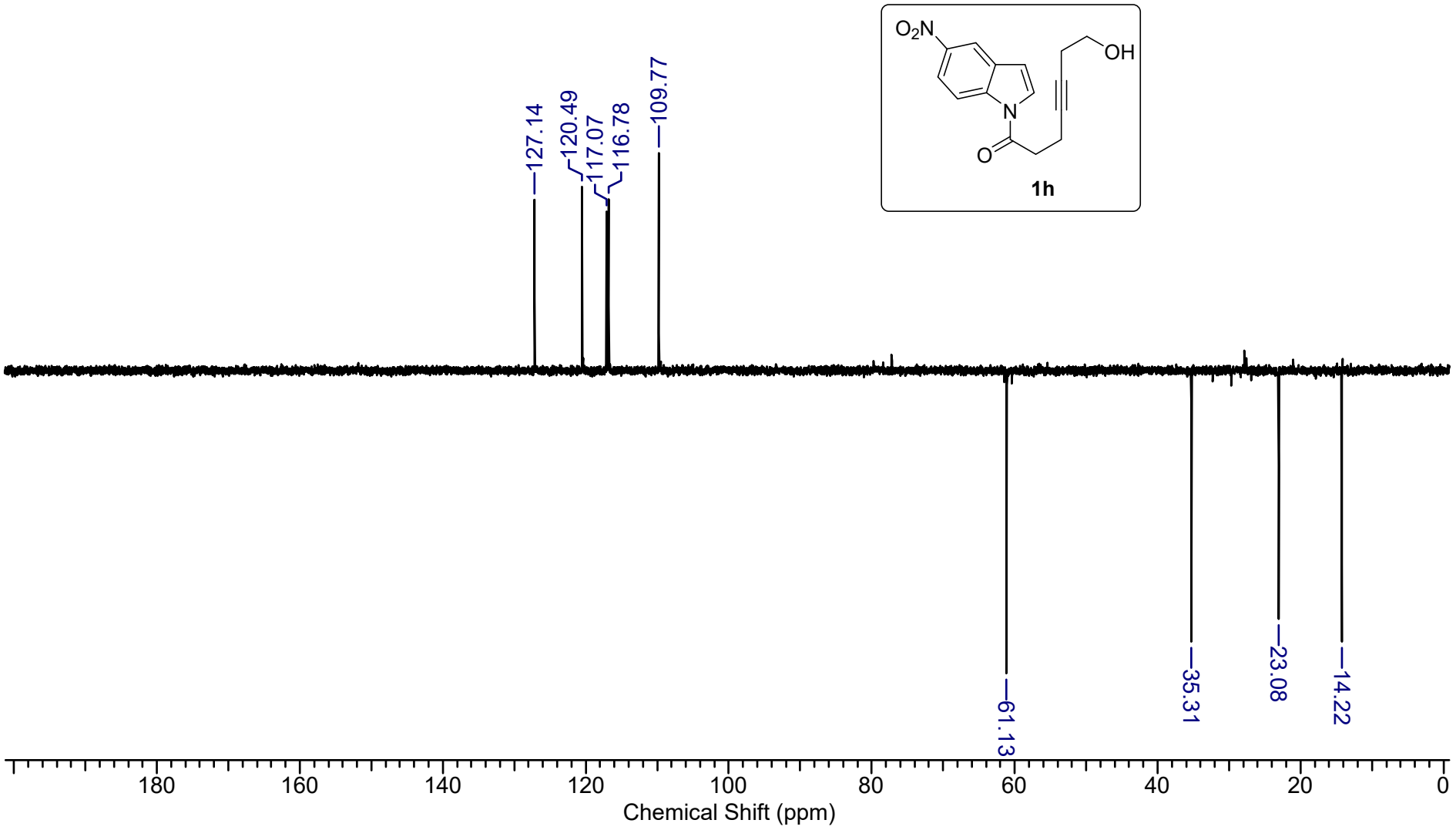

77.32
77.00
76.68
I
OH
N
O
1g
169.68
134.87
133.79
129.76
125.05
118.40
108.38
87.95
80.13
61.17
35.24
23.13
14.33
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

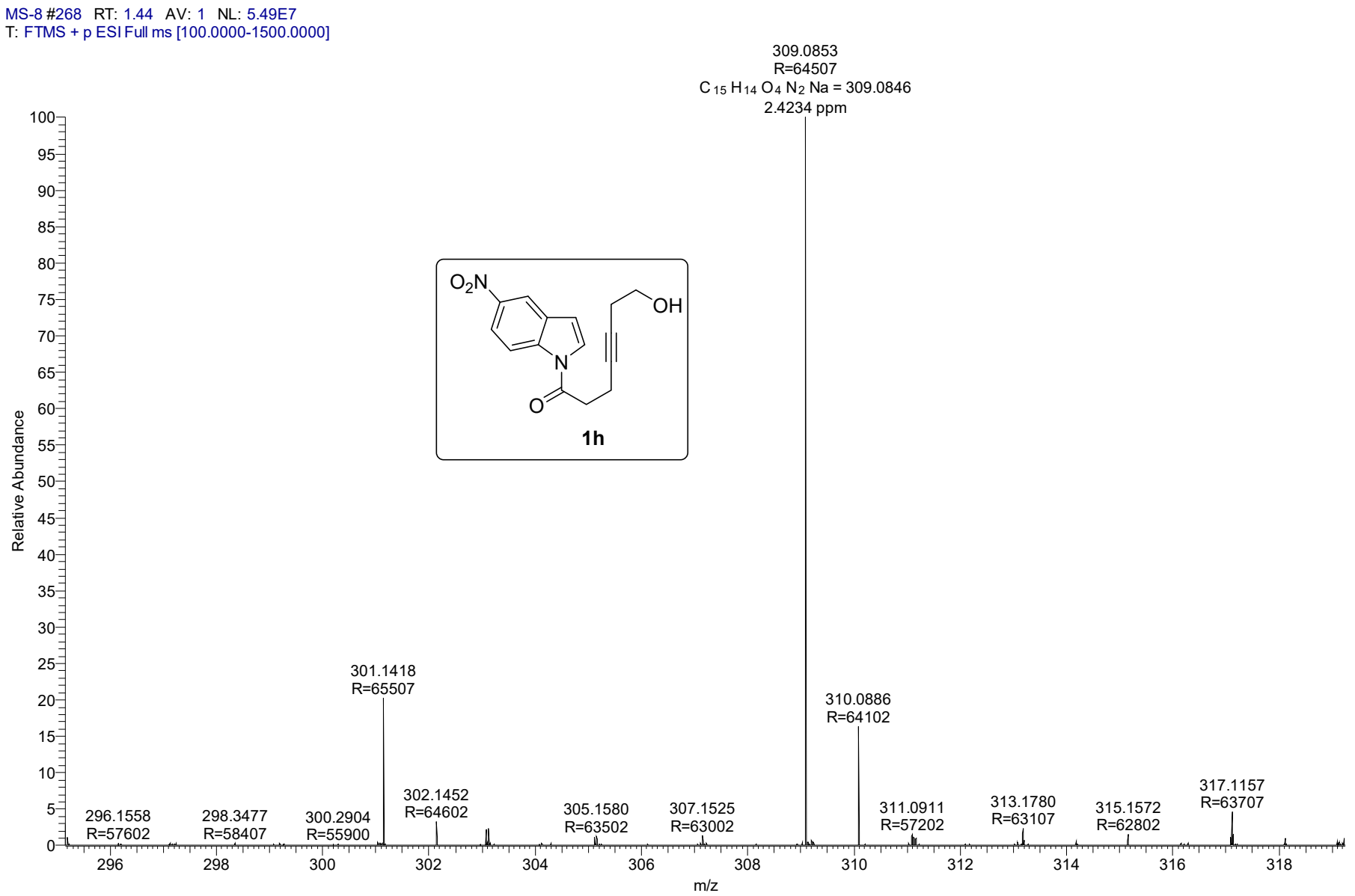

133.79
129.76
125.05
118.39
108.38
61.18
35.24
23.13
14.33
I
OH
N
O
1g
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

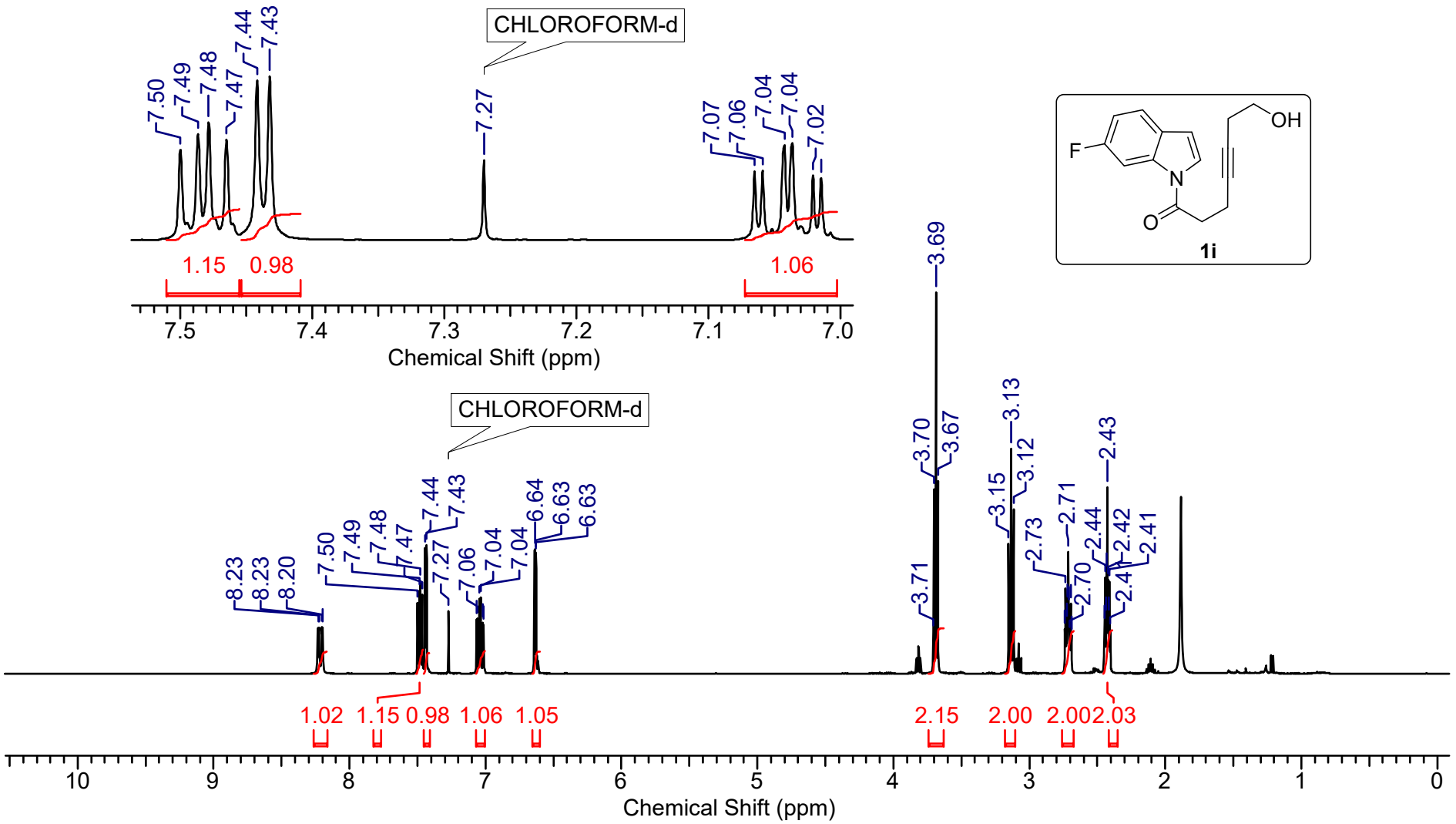

MS-7 #305 RT: 1.63 AV: 1 NL: 9.82E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]
389.9974
R=57502
C 15 H 14 O 2 N I Na = 389.9961
3.1605 ppm
I
OH
N
O
1g
413.2675
R=55107
405.9714
R=56107
368.0154
R=59007
C 15 H 15 O 2 N I = 368.0142
3.1306 ppm
381.2987
R=56507
360.3248
R=58707
353.2673
R=59107
365.2673
R=56202
344.0089
R=57402
377.0608
R=55306
385.1790
R=50607
397.2727
R=53807
421.1787
R=53107
Relative Abundance
m/z

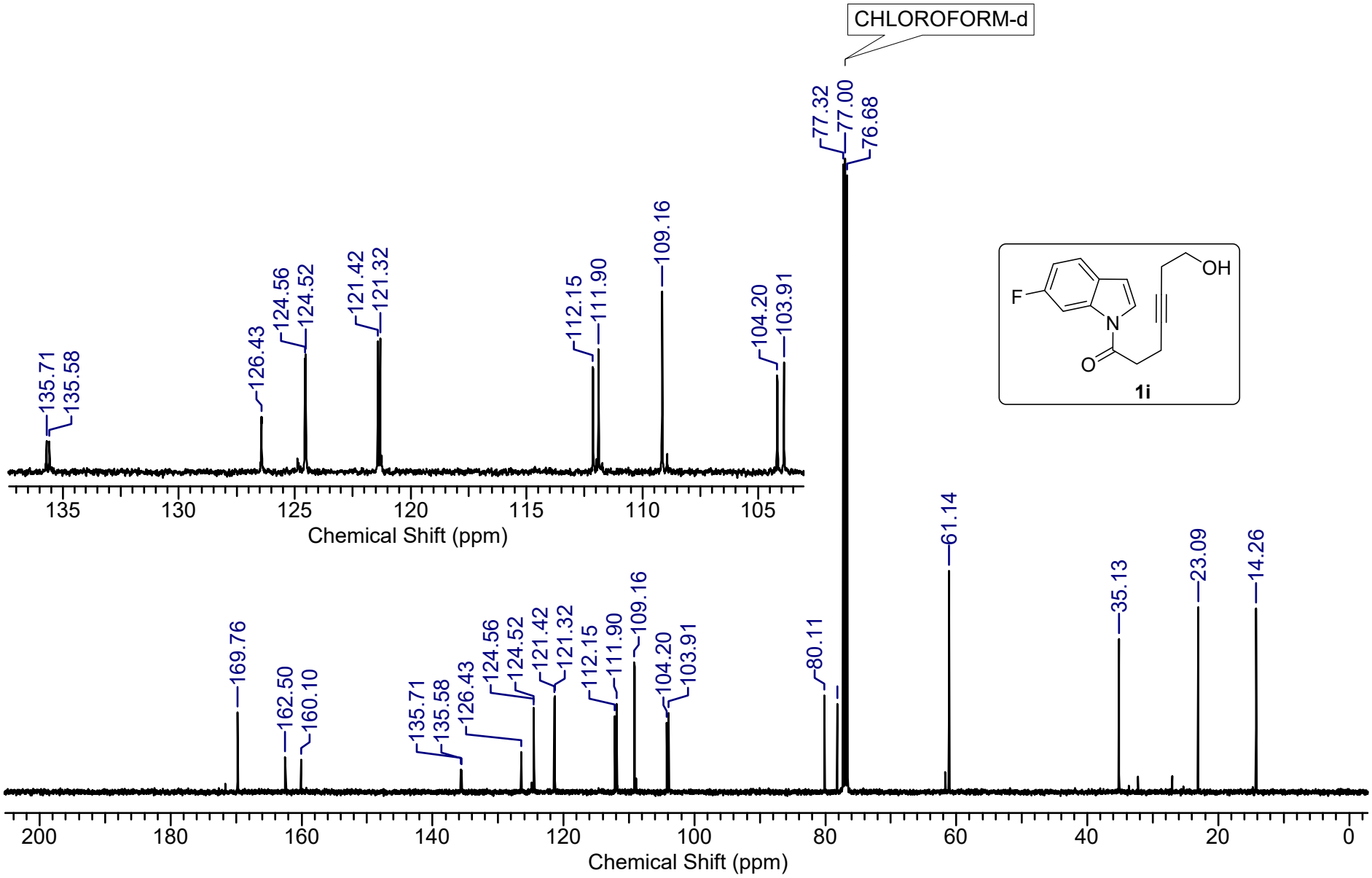

O2N
OH
N
O
1h
CHLOROFORM-d
8.61
8.59
8.51
8.50
8.27
8.27
8.25
7.65
7.64
7.27
6.83
6.82
3.70
3.69
3.71
3.67
3.21
3.20
3.18
2.77
2.75
2.77
2.44
2.44
2.43
2.43
2.42
2.41
1.87
1.86
1.85
1.03
1.00
1.05
1.05
1.04
2.16
2.15
2.16
2.15
0.95
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

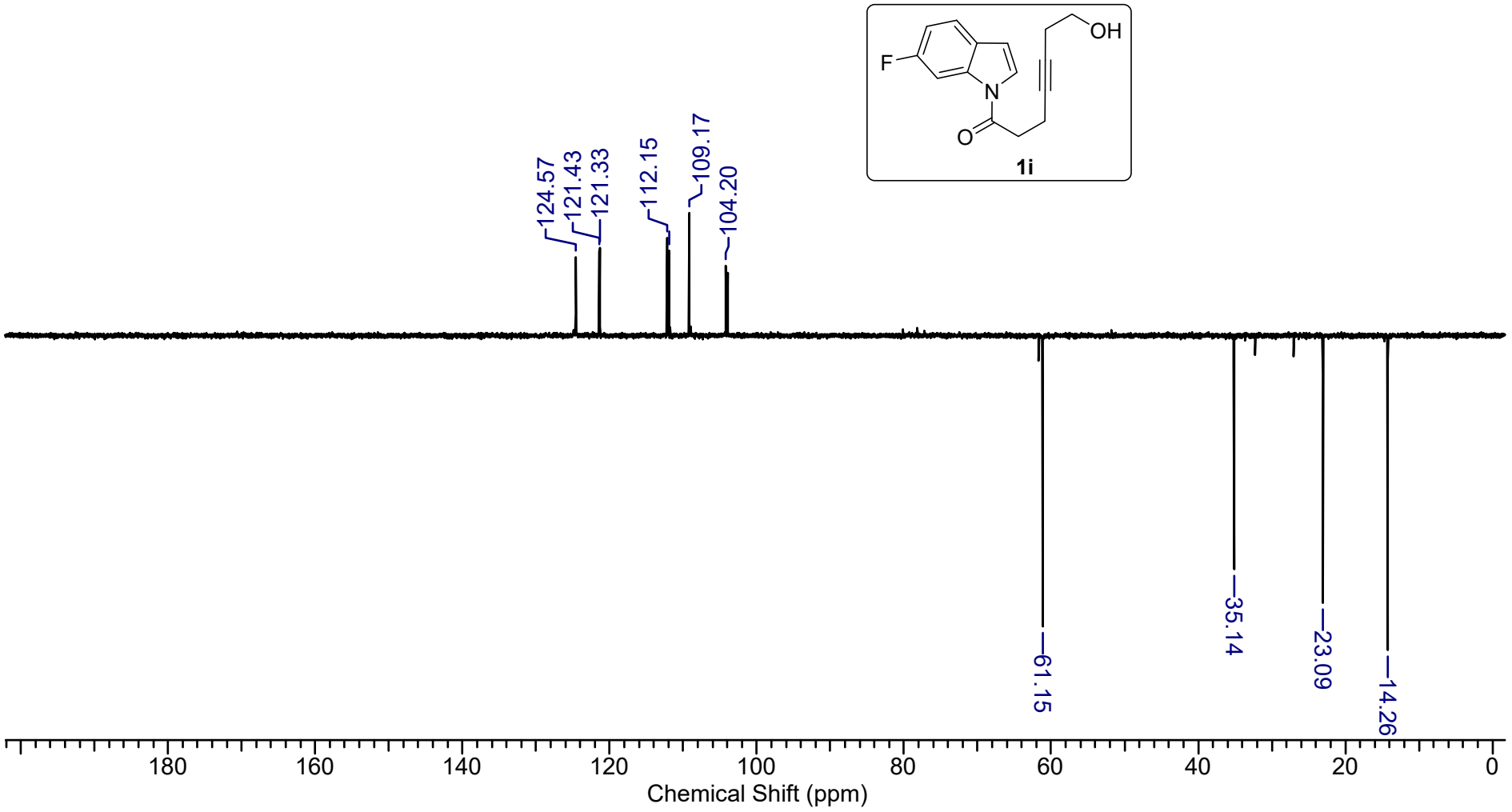

O2N
OH
N
O
1h
77.32
77.00
76.68
169.87
144.34
138.54
130.19
127.15
120.50
117.08
116.79
109.78
79.78
61.13
35.31
23.08
14.22
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

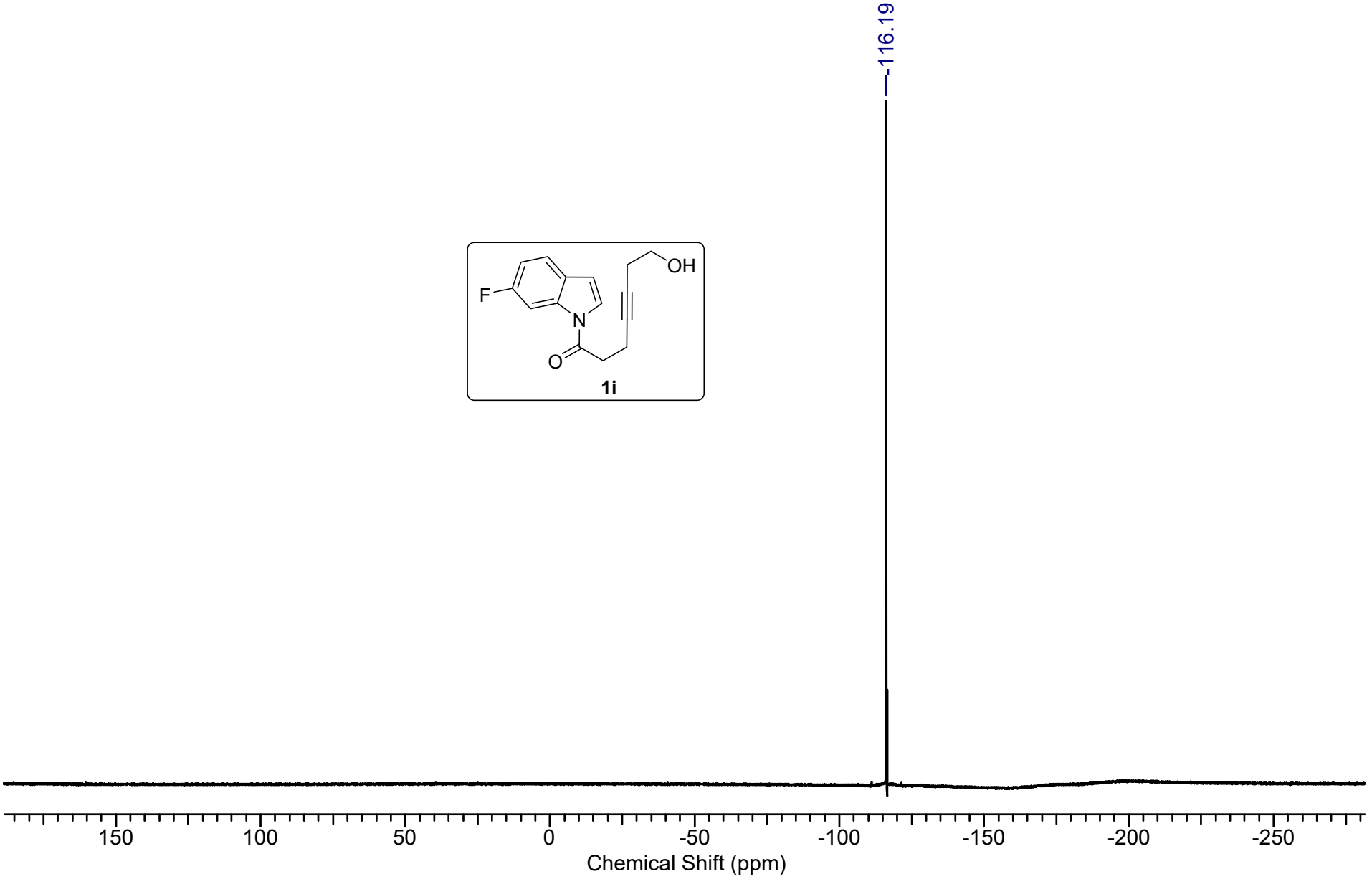

127.14
120.49
117.07
116.78
109.77
61.13
35.31
23.08
14.22
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)
O2N
OH
N
O
1h

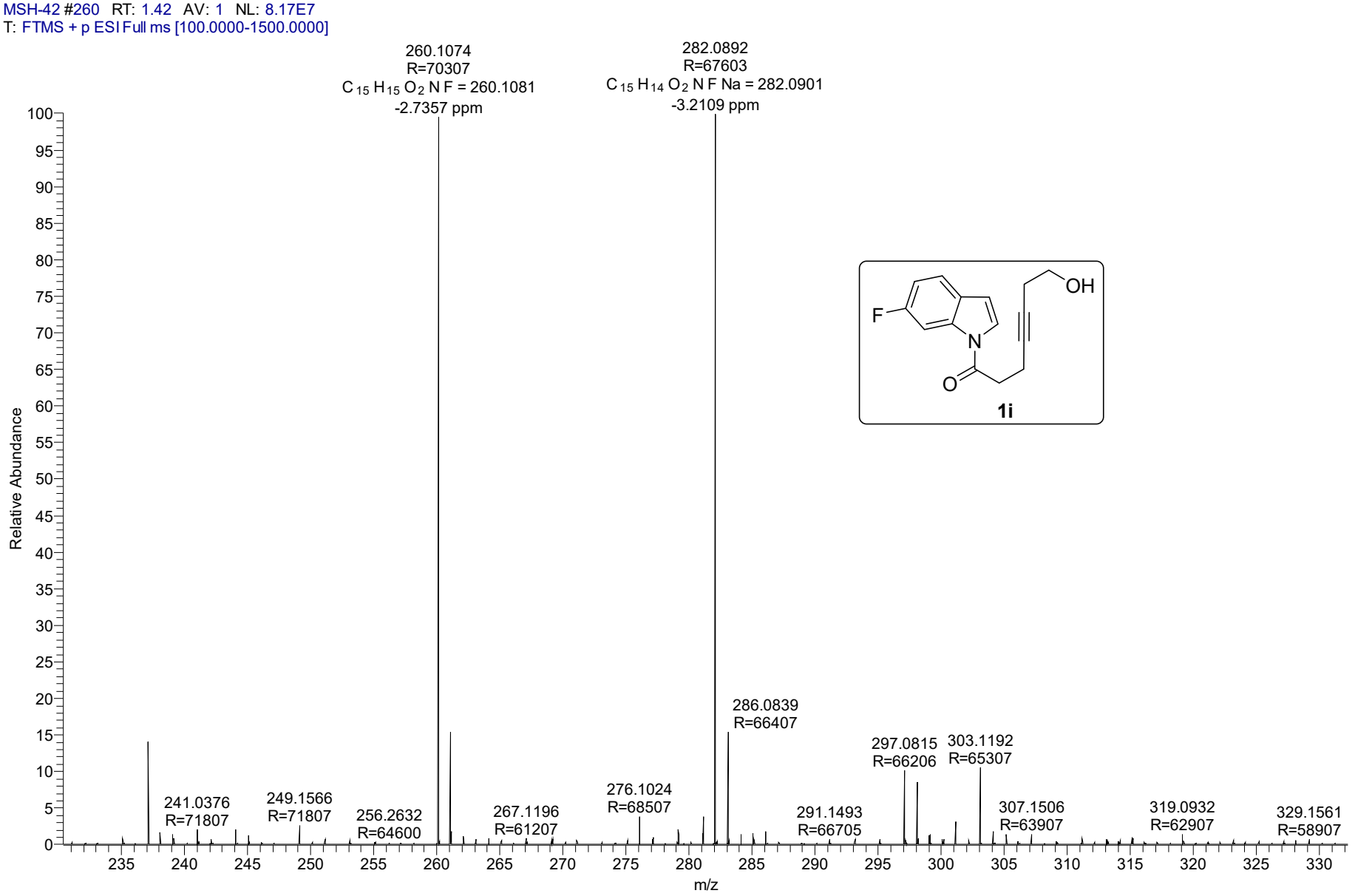

MS-8 #268 RT: 1.44 AV: 1 NL: 5.49E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
309.0853
R=64507
C 15 H 14 O 4 N 2 Na = 309.0846
2.4234 ppm
O2N
OH
N
O
1h
Relative Abundance
m/z
301.1418
R=65507
310.0886
R=64102
296.1558
R=57602
298.3477
R=58407
300.2904
R=55900
302.1452
R=64602
305.1580
R=63502
307.1525
R=63002
311.0911
R=57202
313.1780
R=63107
315.1572
R=62802
317.1157
R=63707

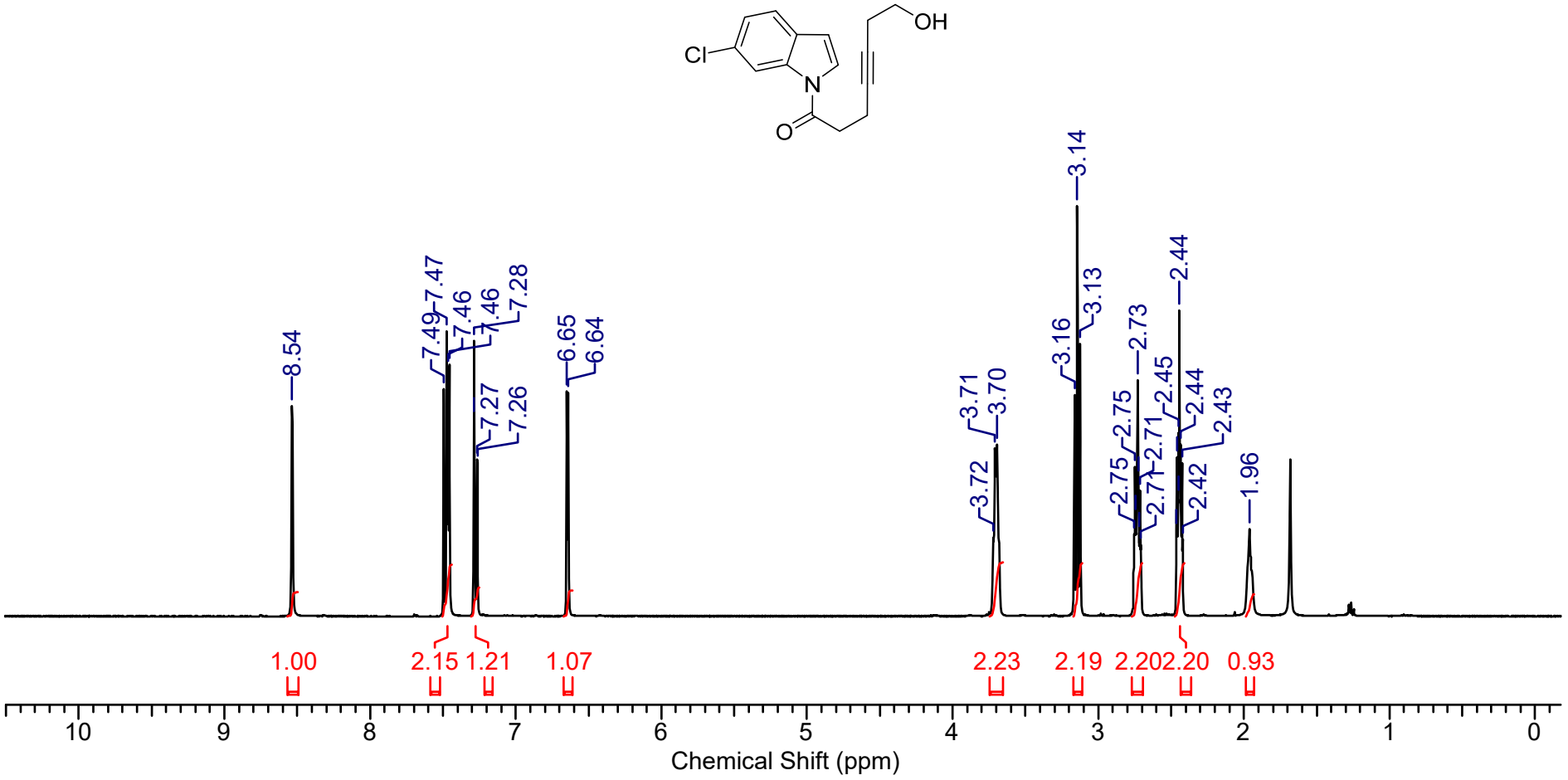

CHLOROFORM-d
7.50
7.49
7.48
7.47
7.44
7.43
7.27
7.07
7.06
7.04
7.04
7.02
1.15
0.98
1.06
7.5
7.4
7.3
7.2
7.1
7.0
Chemical Shift (ppm)
F
N
O
OH
1i
CHLOROFORM-d
8.23
8.23
8.20
7.50
7.49
7.48
7.47
7.44
7.43
7.27
7.06
7.04
7.04
6.64
6.63
6.63
3.69
3.70
3.67
3.71
3.15
3.13
3.12
2.73
2.71
2.44
2.43
2.42
2.41
2.70
1.02
1.15
0.98
1.06
1.05
2.15
2.00
2.00
2.03
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

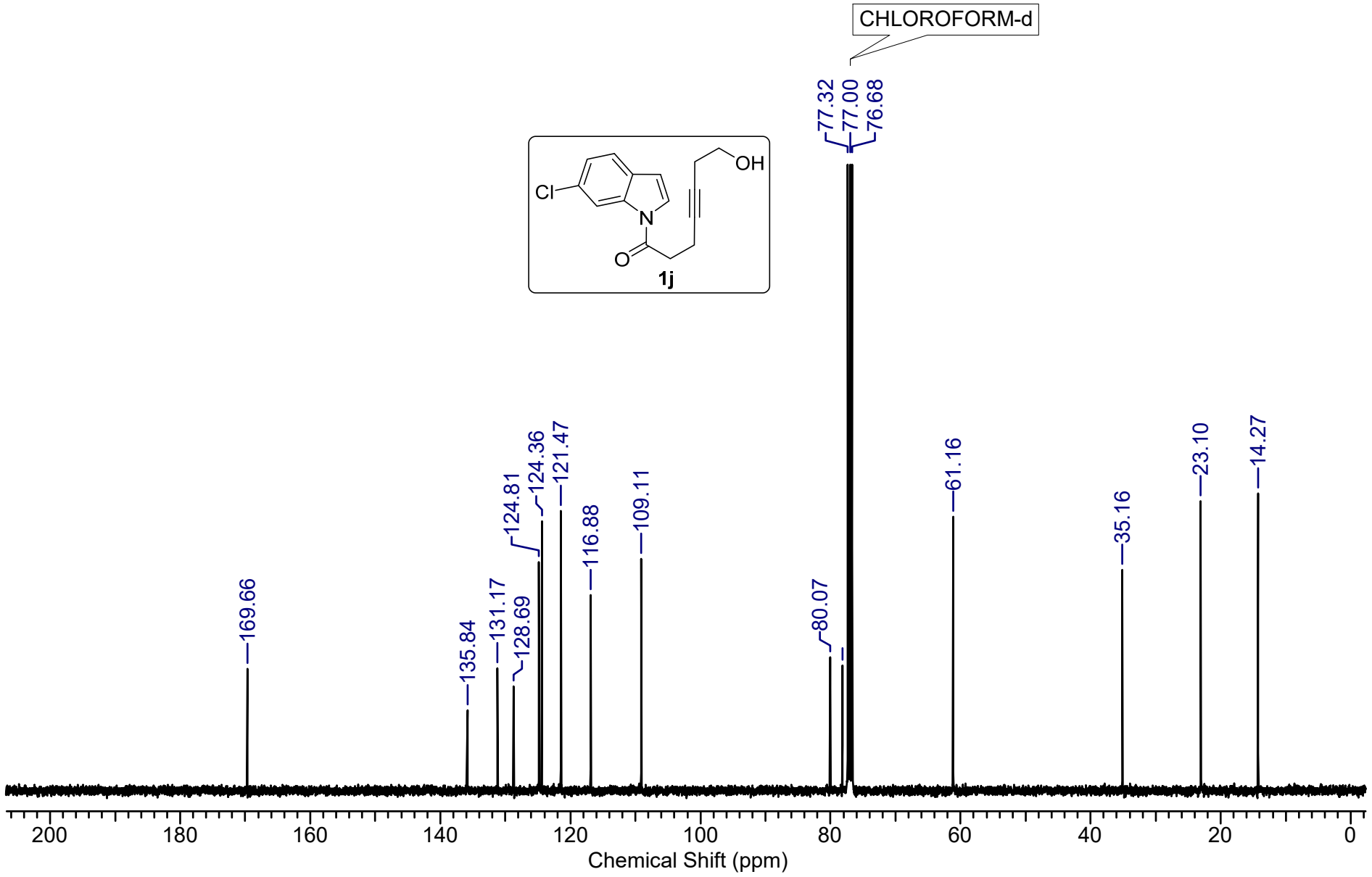

CHLOROFORM-d
77.32
77.00
76.68
135.71
135.58
126.43
124.56
124.52
121.42
121.32
112.15
111.90
109.16
104.20
103.91
61.14
35.13
23.09
14.26
80.11
169.76
162.50
160.10
F
OH
N
O
1i
Chemical Shift (ppm)
Chemical Shift (ppm)

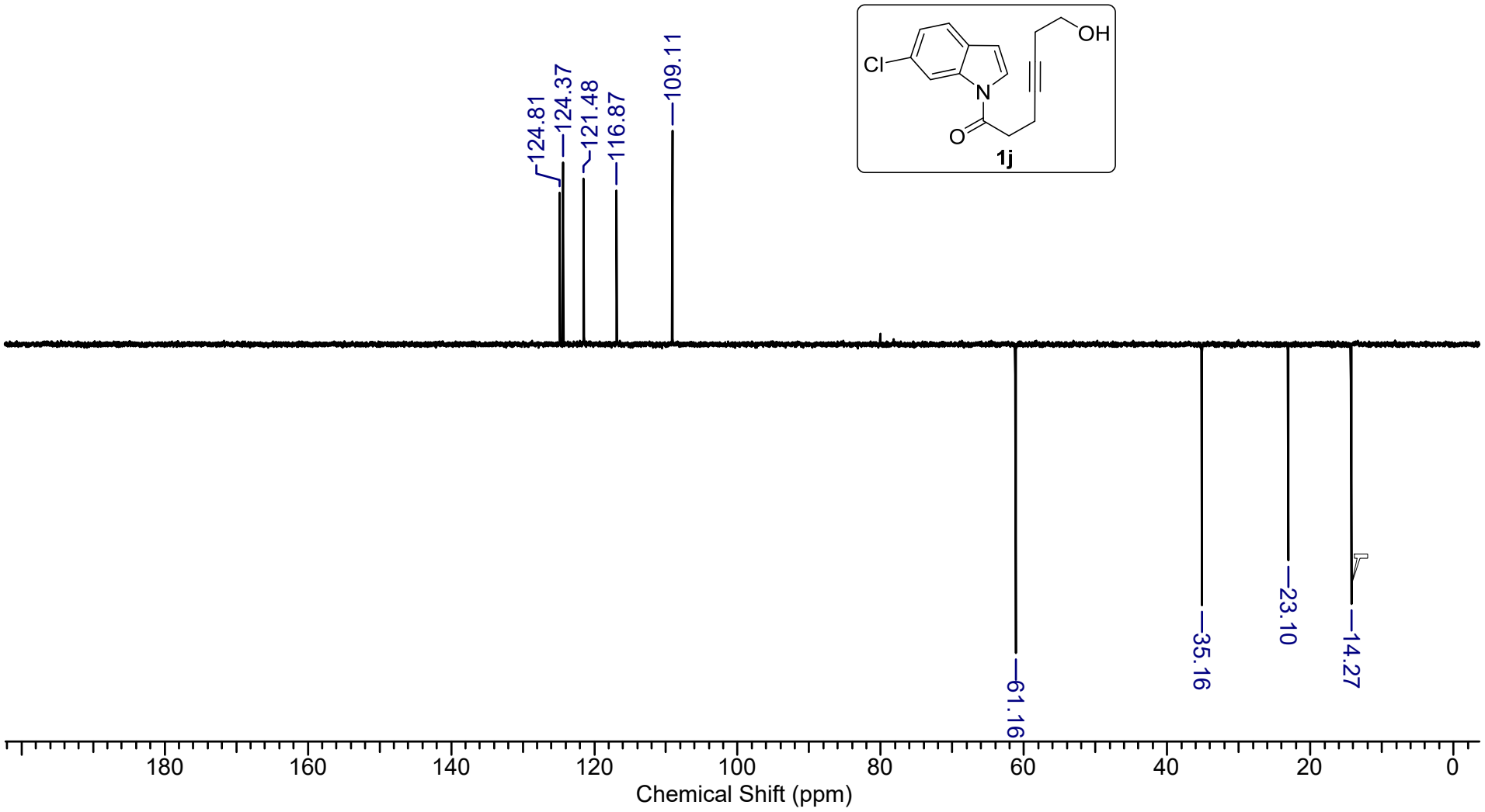

F
N
O
OH
1i
124.57
121.43
121.33
112.15
109.17
104.20
61.15
35.14
23.09
14.26
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

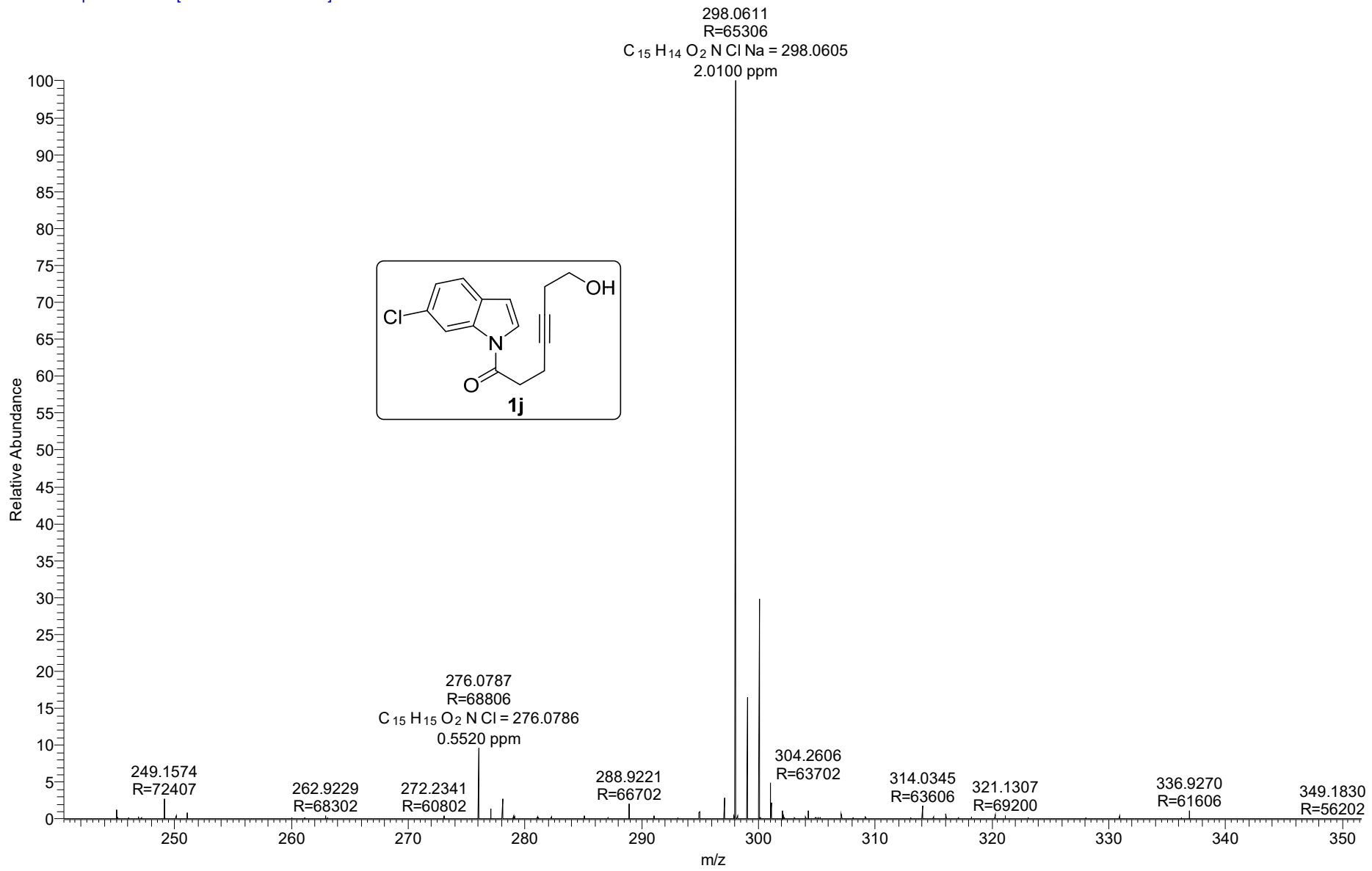

-116.19
F
N
O
OH
1i
150
100
50
0
-50
-100
-150
-200
-250
Chemical Shift (ppm)

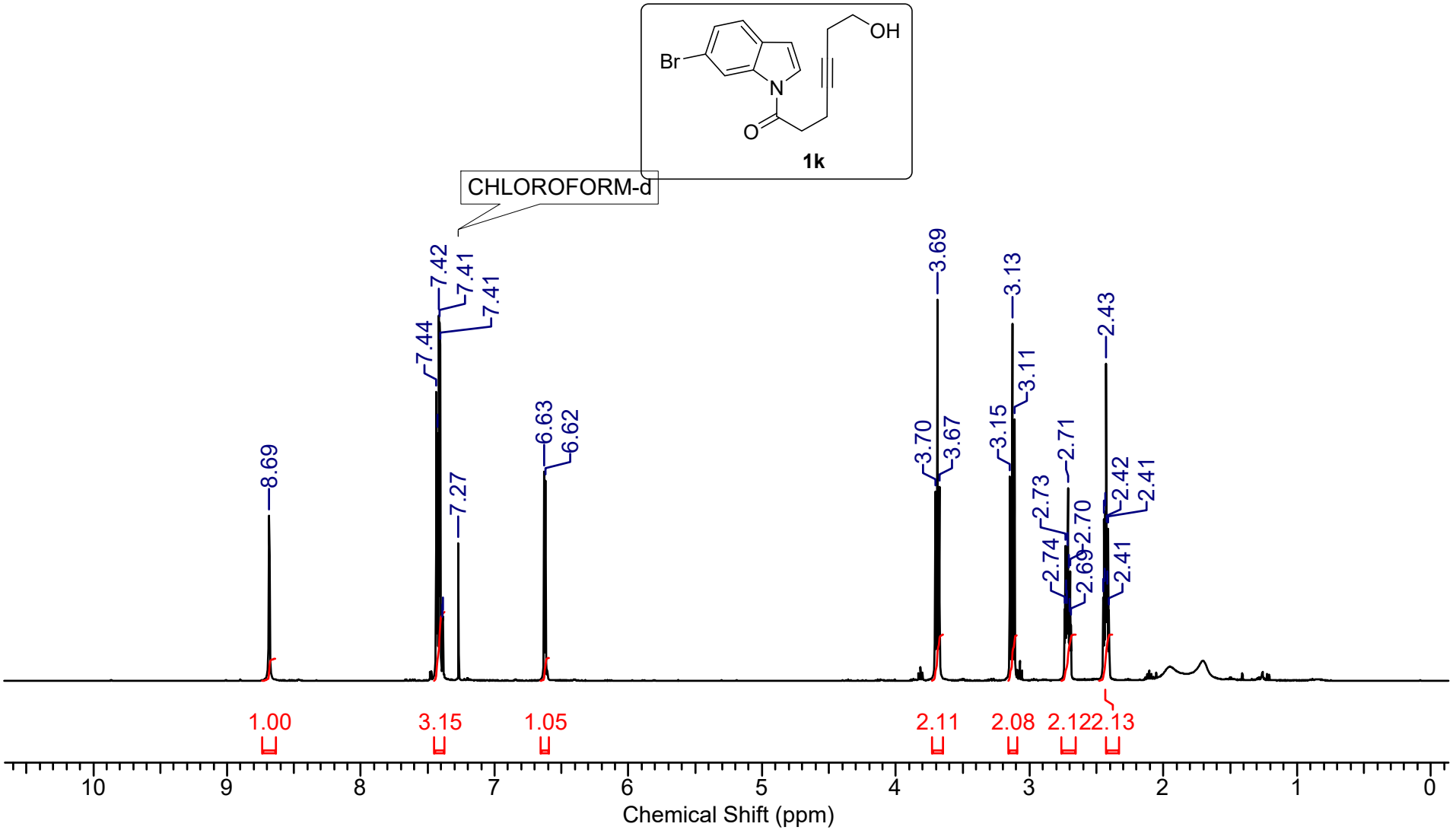

MSH-42 #260 RT: 1.42 AV: 1 NL: 8.17E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
260.1074
R=70307
C 15 H 15 O 2 N F = 260.1081
-2.7357 ppm
282.0892
R=67603
C 15 H 14 O 2 N F Na = 282.0901
-3.2109 ppm
OH
F
N
O
1i
Relative Abundance
m/z
241.0376
R=71807
249.1566
R=71807
256.2632
R=64600
267.1196
R=61207
276.1024
R=68507
286.0839
R=66407
291.1493
R=66705
297.0815
R=66206
303.1192
R=65307
307.1506
R=63907
319.0932
R=62907
329.1561
R=58907

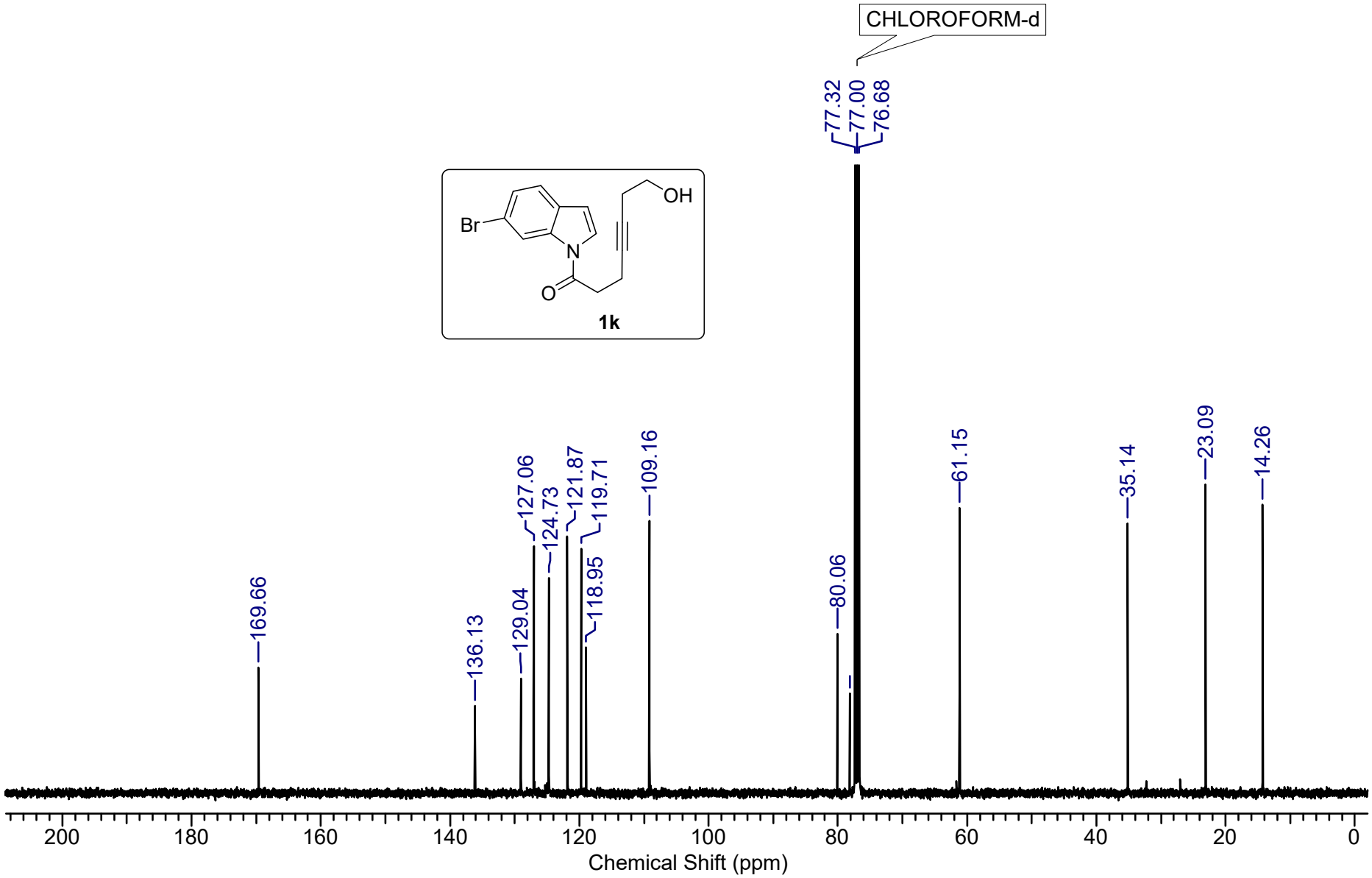

Cl
N
O
OH
8.54
7.49
7.47
7.46
7.46
7.28
7.27
7.26
6.65
6.64
3.72
3.71
3.70
3.16
3.14
3.13
2.75
2.73
2.71
2.71
2.45
2.44
2.44
2.43
2.42
1.96
1.00
2.15
1.21
1.07
2.23
2.19
2.20
2.20
0.93
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

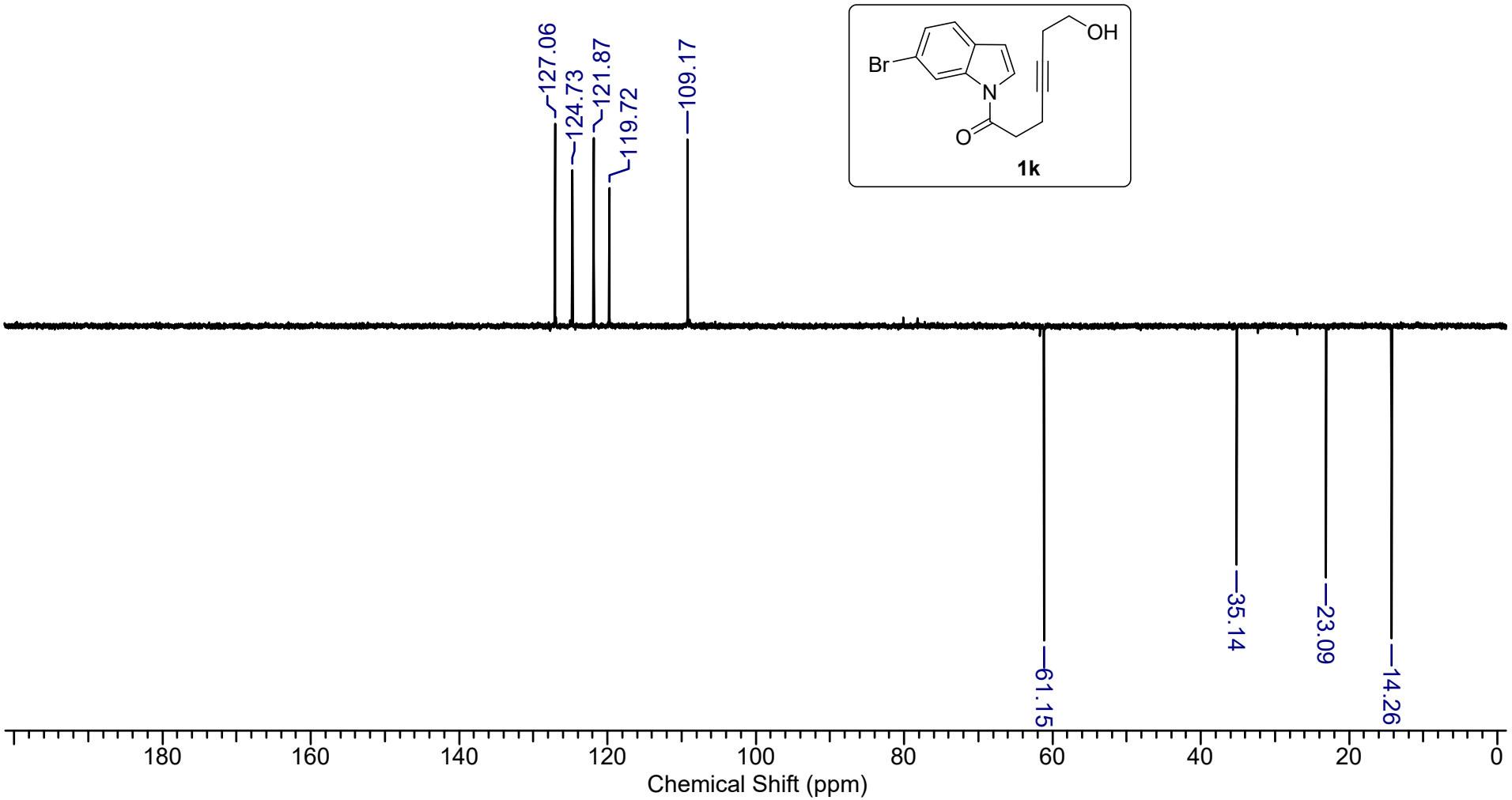

CHLOROFORM-d
77.32
77.00
76.68
Cl
N
O
OH
1j
169.66
135.84
131.17
128.69
124.81
124.36
121.47
116.88
109.11
80.07
61.16
35.16
23.10
14.27
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

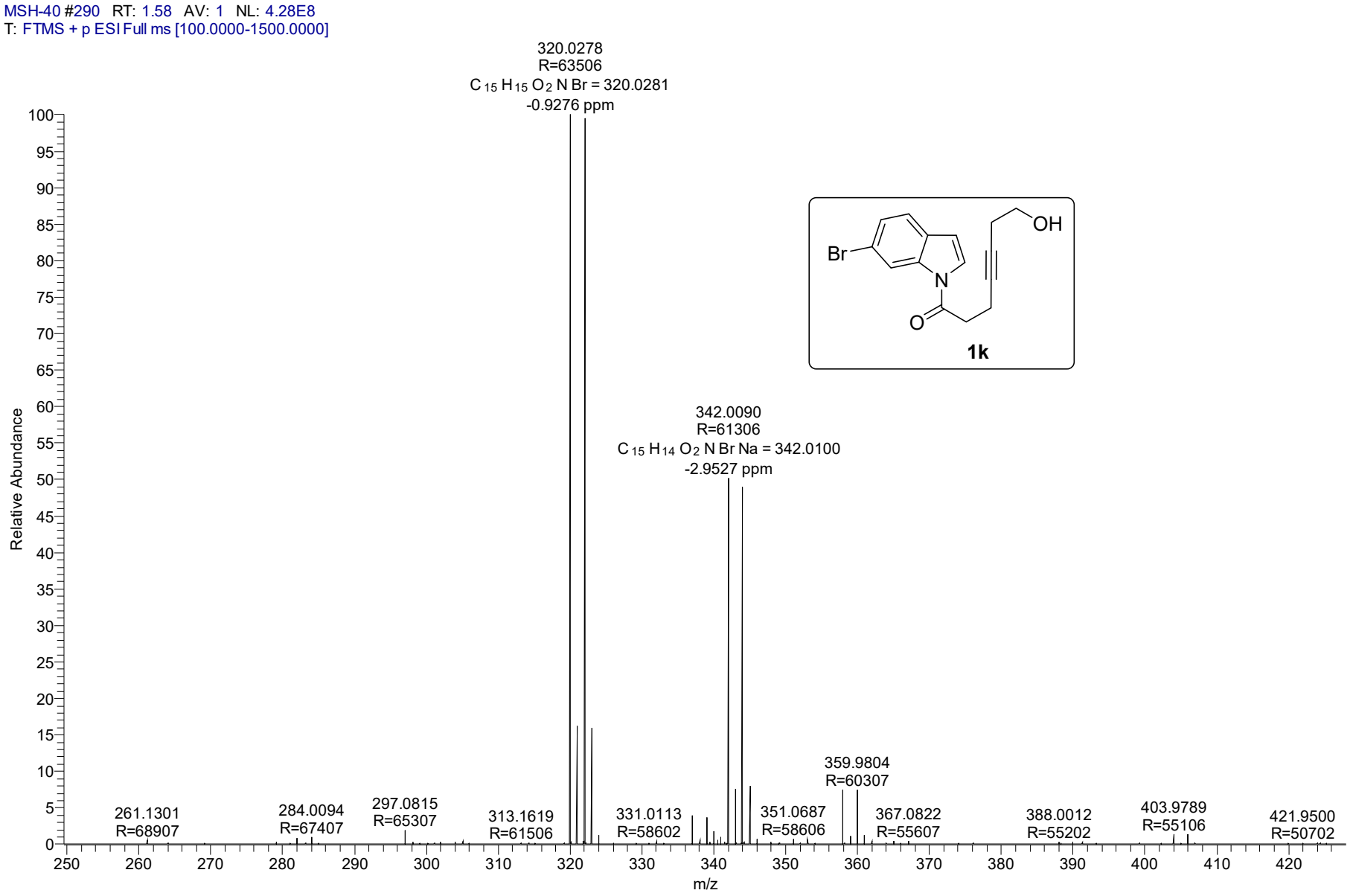

124.81
124.37
121.48
116.87
109.11
61.16
35.16
23.10
14.27
Cl
N
O
OH
1j
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

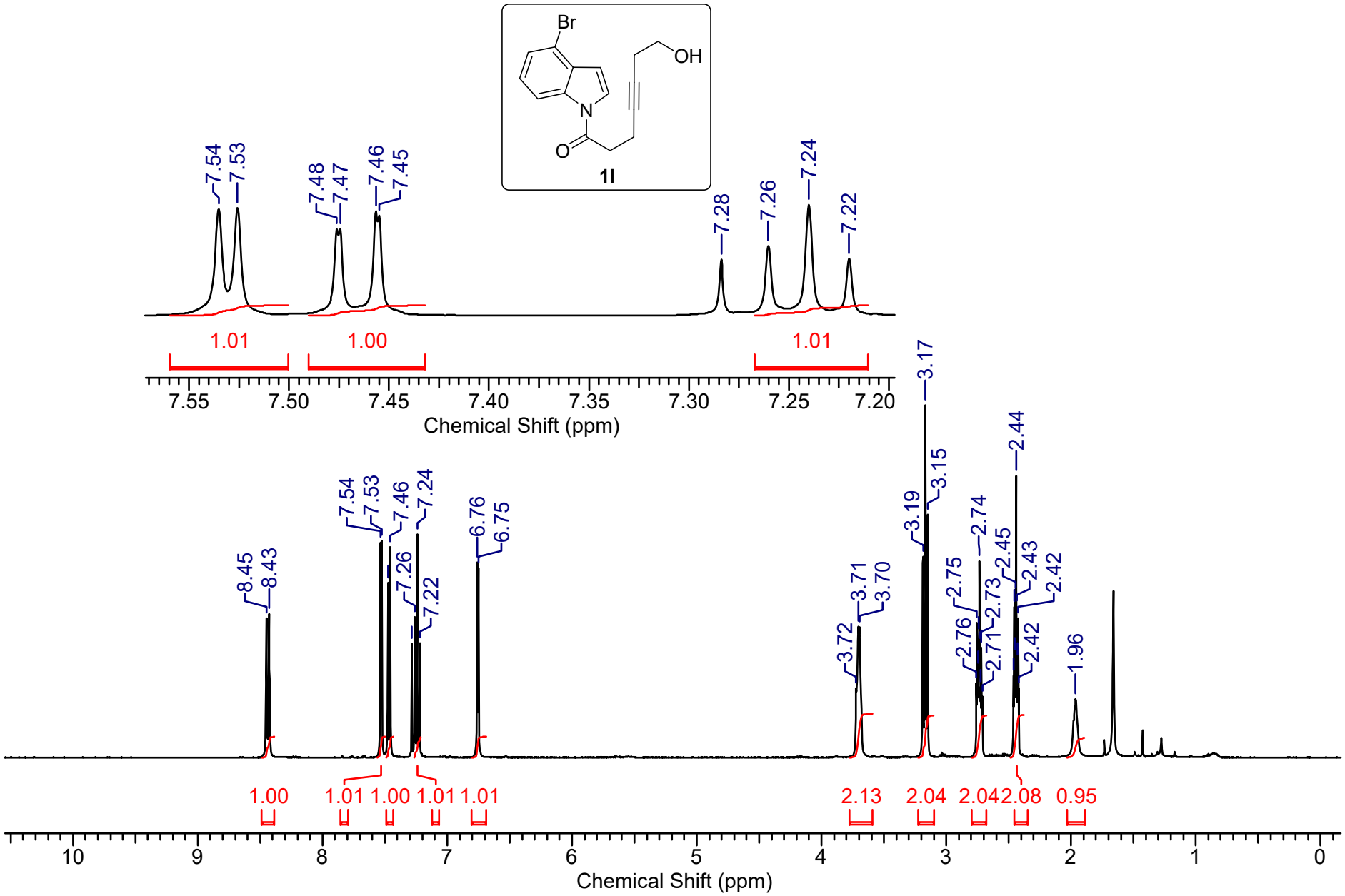

MSH-52 #248 RT: 1.35 AV: 1 NL: 1.57E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
298.0611
R=65306
C 15 H 14 O 2 N Cl Na = 298.0605
2.0100 ppm
Cl
N
O
OH
1j
276.0787
R=68806
C 15 H 15 O 2 N Cl = 276.0786
0.5520 ppm
249.1574
R=72407
262.9229
R=68302
272.2341
R=60802
288.9221
R=66702
304.2606
R=63702
314.0345
R=63606
321.1307
R=69200
336.9270
R=61606
349.1830
R=56202
Relative Abundance
m/z

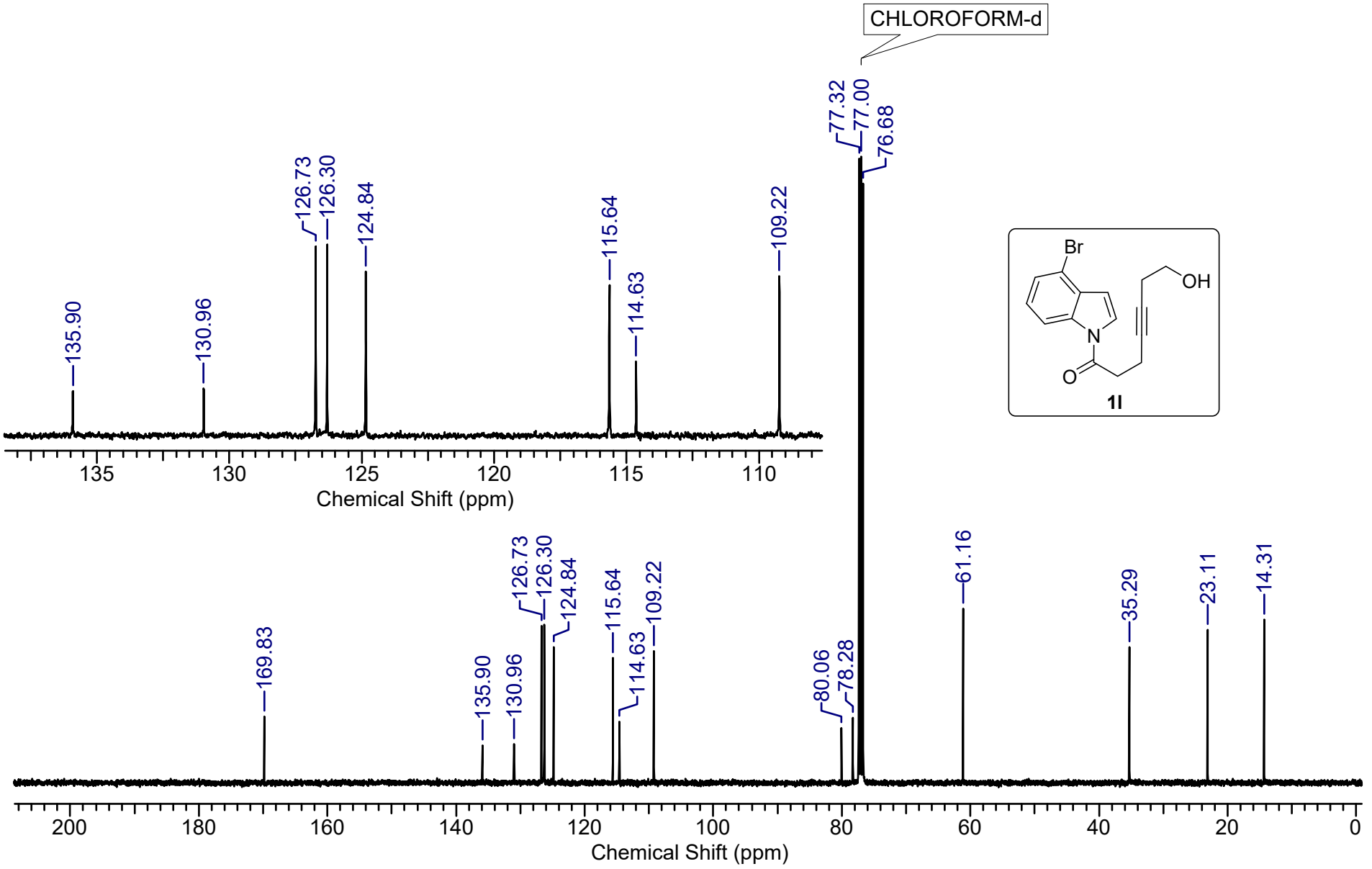

Br
N
O
OH
1k
CHLOROFORM-d
8.69
7.44
7.42
7.41
7.41
7.27
6.63
6.62
3.70
3.69
3.67
3.15
3.13
3.11
2.74
2.73
2.71
2.70
2.69
2.43
2.42
2.41
2.41
1.00
3.15
1.05
2.11
2.08
2.12
2.13
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

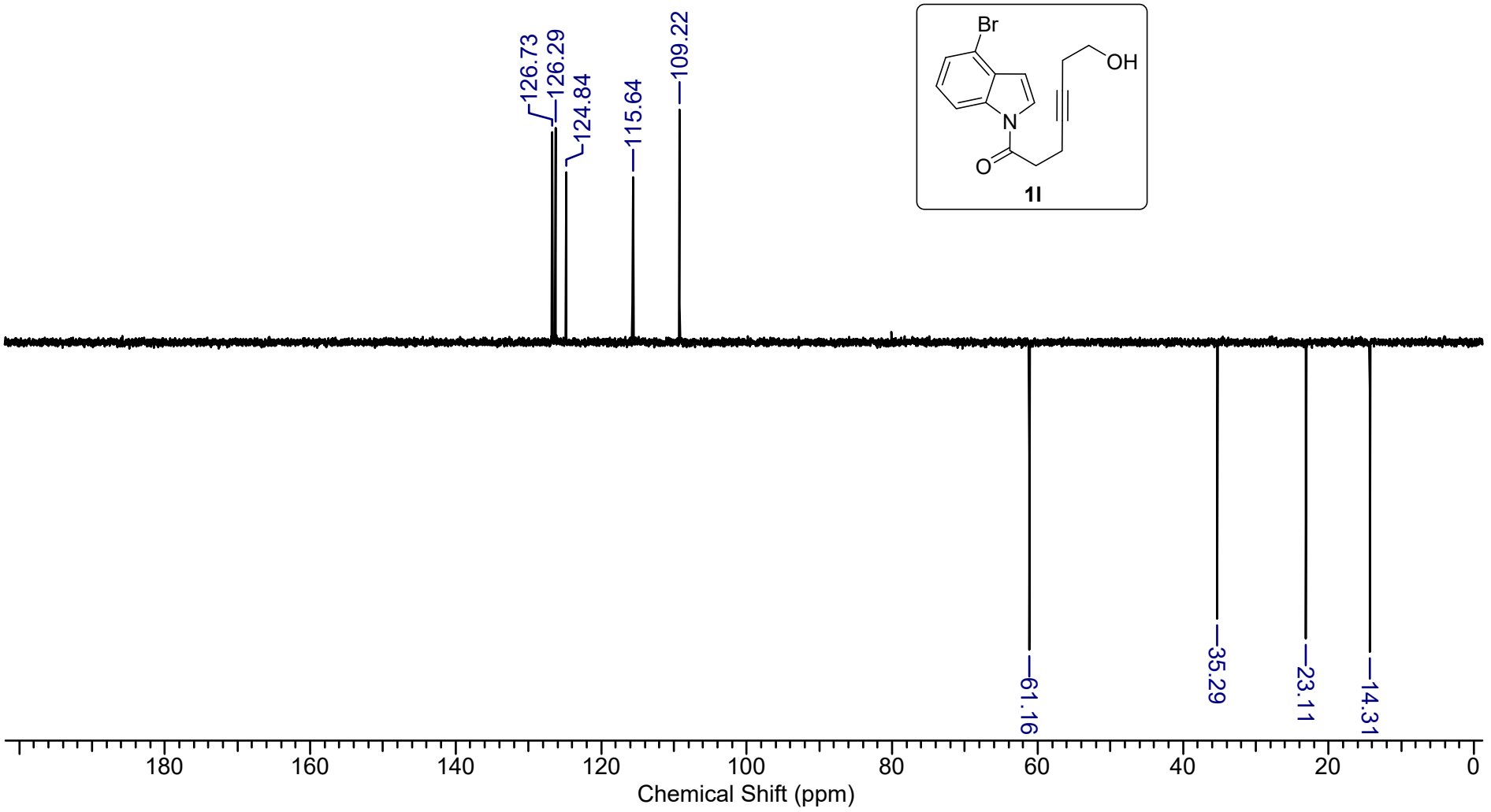

CHLOROFORM-d
77.32
77.00
76.68
Br
N
O
OH
1k
169.66
136.13
129.04
127.06
124.73
121.87
119.71
118.95
109.16
80.06
61.15
35.14
23.09
14.26
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


127.06
124.73
121.87
119.72
109.17
Br
N
O
OH
1k
61.15
35.14
23.09
14.26
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

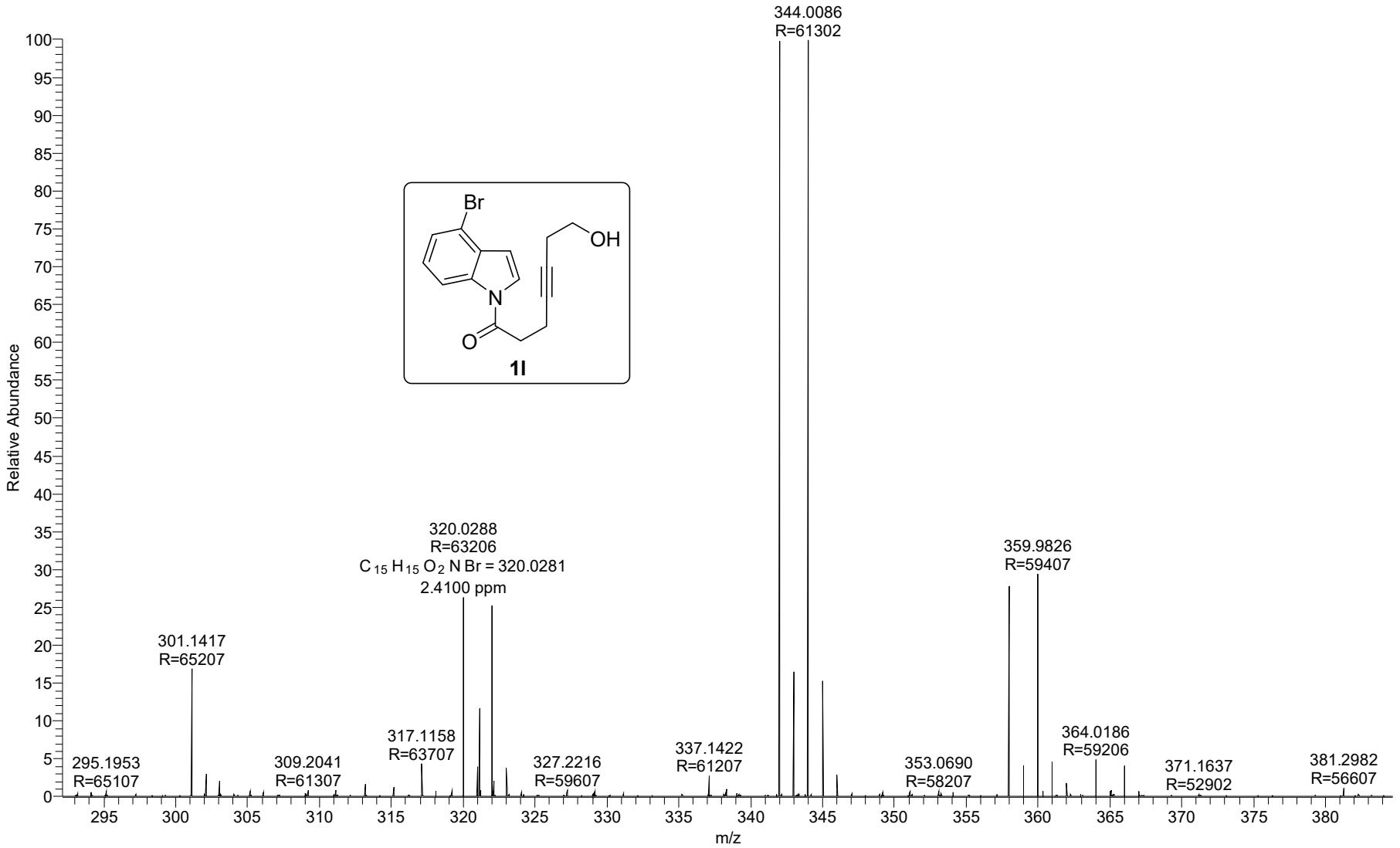

MSH-40 #290 RT: 1.58 AV: 1 NL: 4.28E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
320.0278
R=63506
C 15 H 15 O 2 N Br = 320.0281
-0.9276 ppm
342.0090
R=61306
C 15 H 14 O 2 N Br Na = 342.0100
-2.9527 ppm
261.1301
R=68907
284.0094
R=67407
297.0815
R=65307
313.1619
R=61506
331.0113
R=58602
351.0687
R=58606
359.9804
R=60307
367.0822
R=55607
388.0012
R=55202
403.9789
R=55106
421.9500
R=50702
Br
N
O
OH
1k
Relative Abundance
m/z

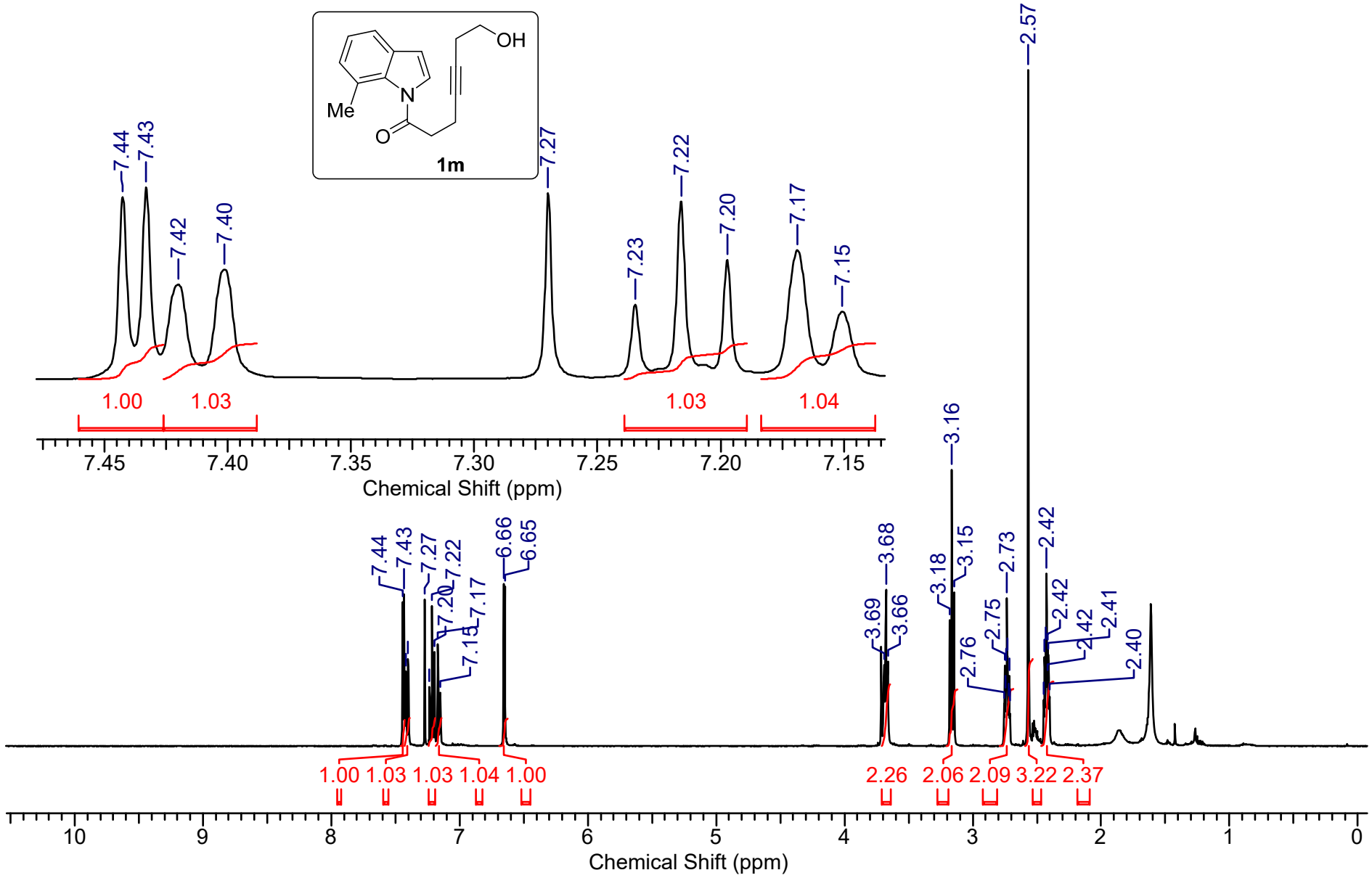

Br
OH
N
O
1l
7.54
7.53
7.48
7.47
7.46
7.45
7.28
7.26
7.24
7.22
1.01
1.00
1.01
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
Chemical Shift (ppm)
8.45
8.43
7.54
7.53
7.46
7.24
7.26
7.22
6.76
6.75
3.72
3.71
3.70
3.19
3.17
3.15
2.76
2.75
2.74
2.73
2.71
2.45
2.44
2.43
2.42
2.42
1.96
1.00
1.01
1.00
1.01
1.01
2.13
2.04
2.04
2.08
0.95
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

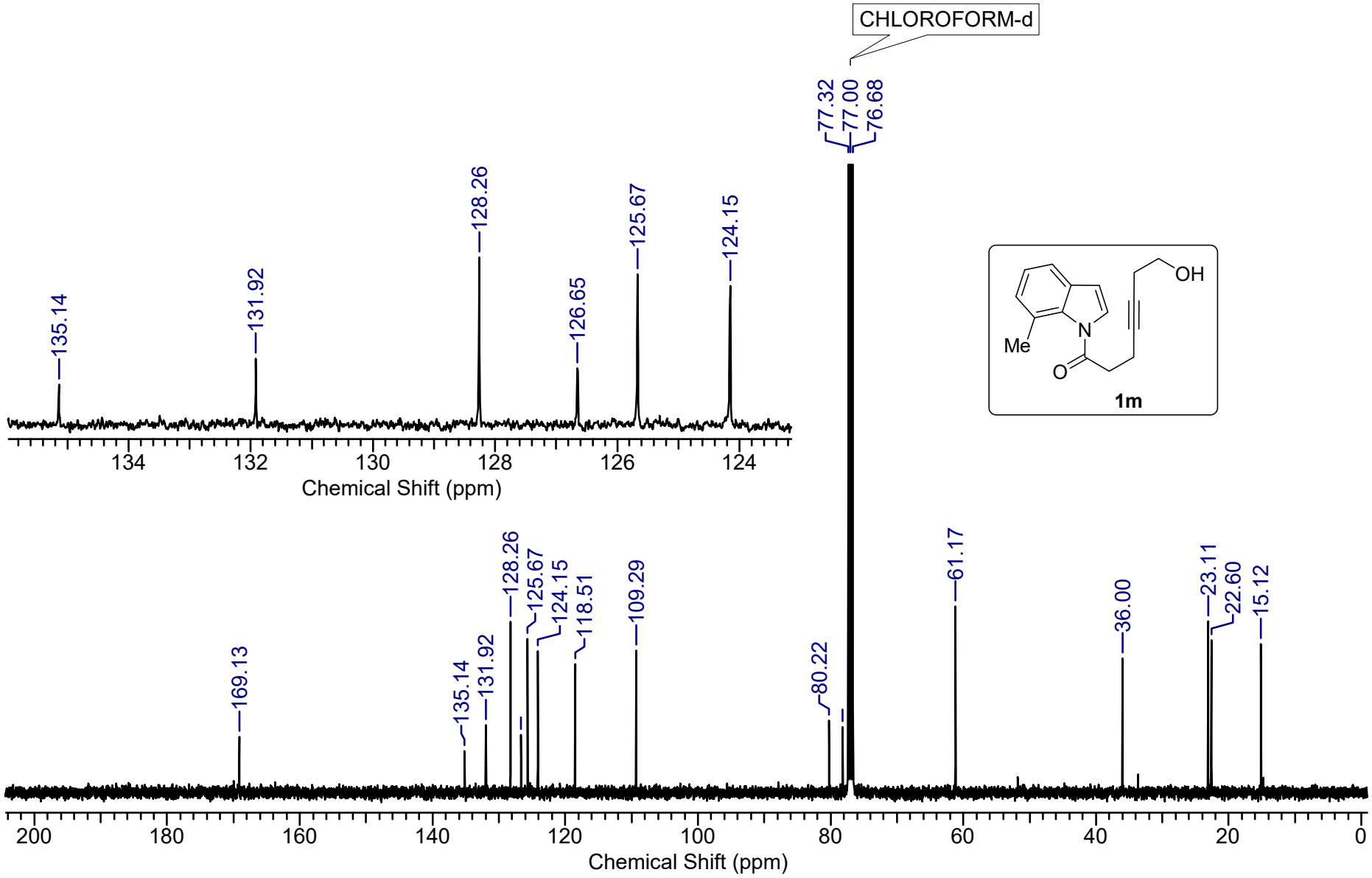

CHLOROFORM-d
77.32
77.00
76.68
126.73
126.30
124.84
115.64
114.63
109.22
135.90
130.96
Br
OH
N
O
1l
135
130
125
120
115
110
Chemical Shift (ppm)
169.83
135.90
130.96
126.73
126.30
124.84
115.64
114.63
109.22
80.06
78.28
61.16
35.29
23.11
14.31
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

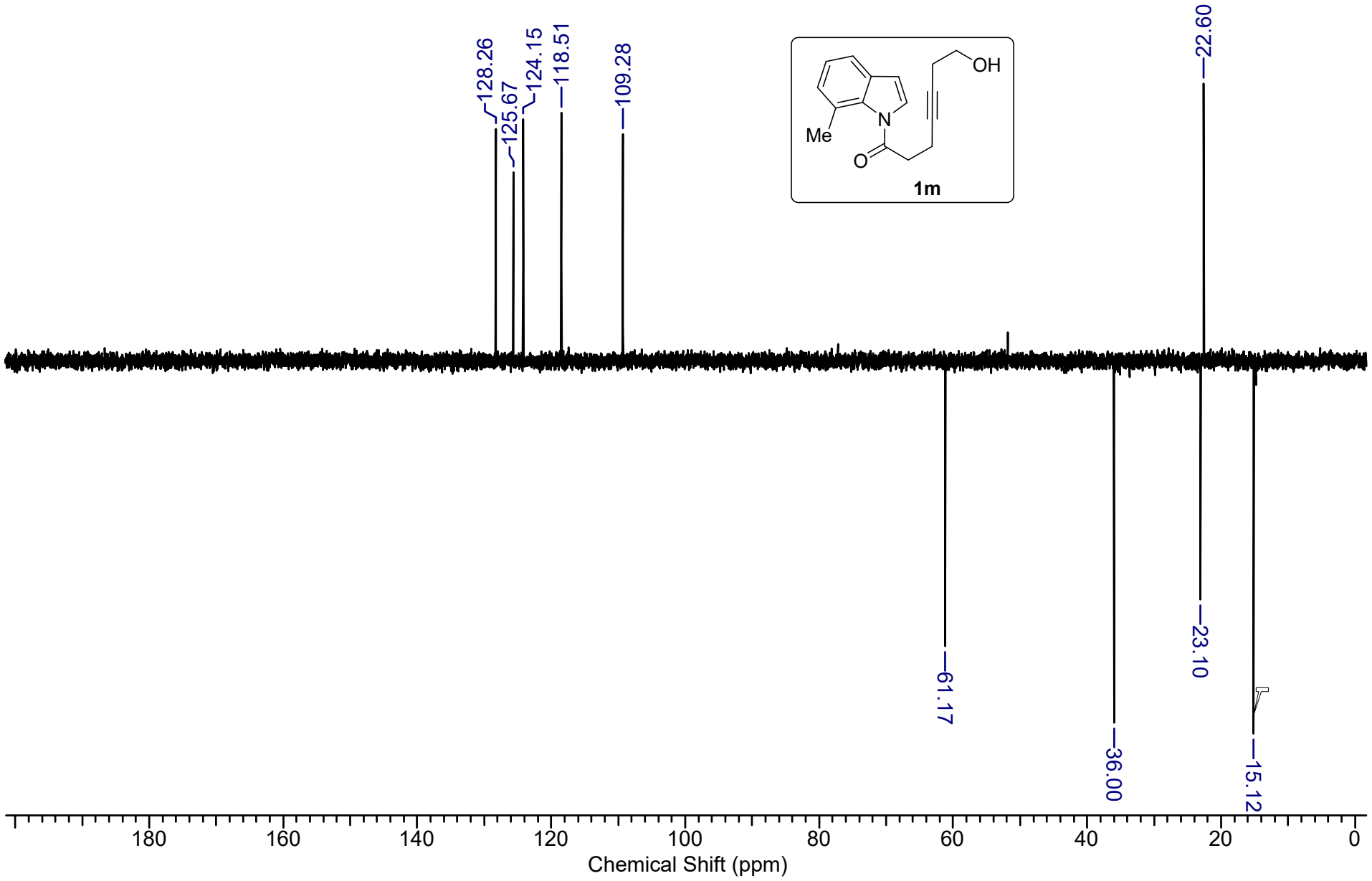

126.73
126.29
124.84
115.64
109.22
Br
OH
N
O
1l
61.16
35.29
23.11
14.31
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

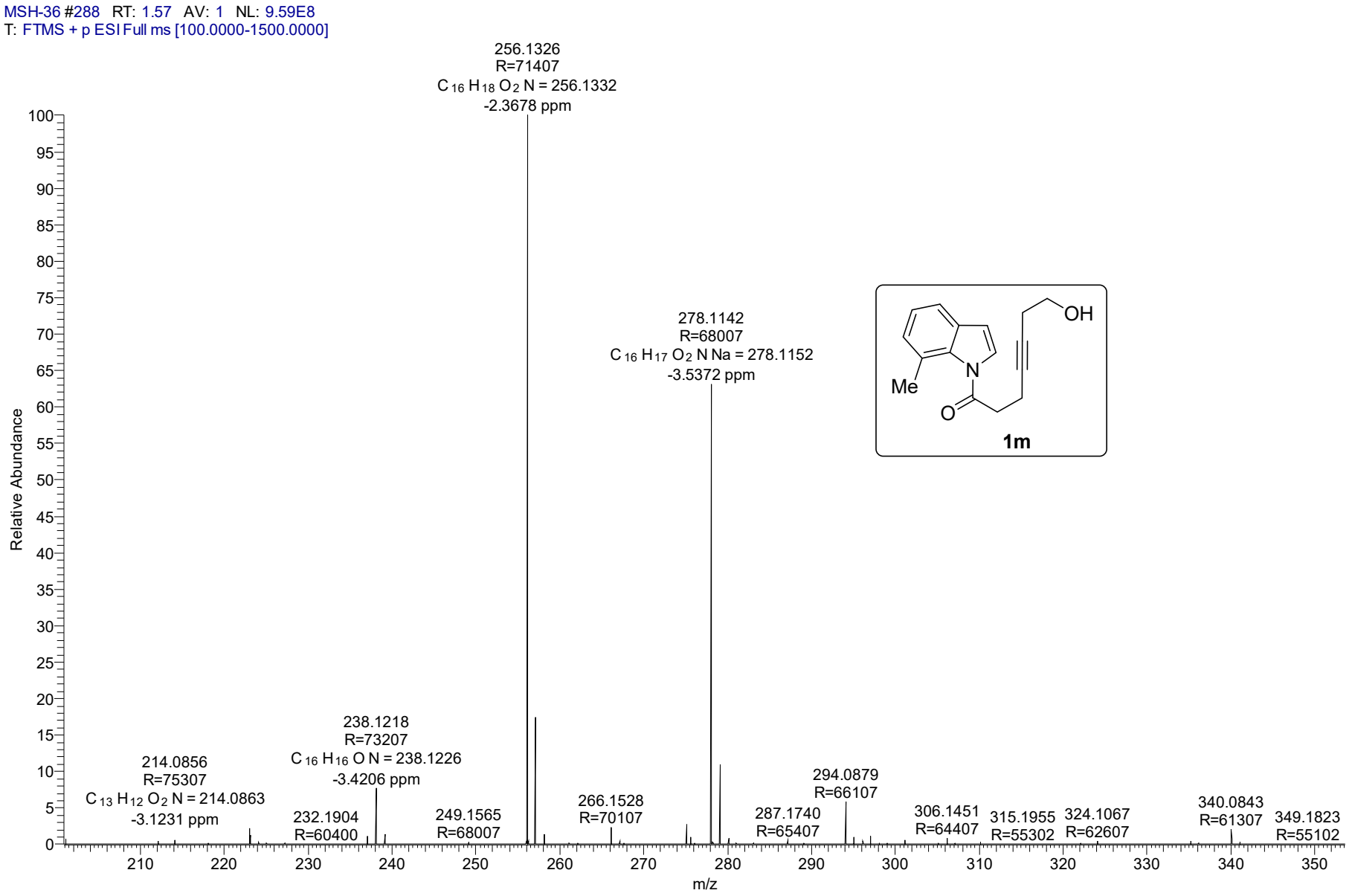
MS-11 #297 RT: 1.59 AV: 1 NL: 6.04E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

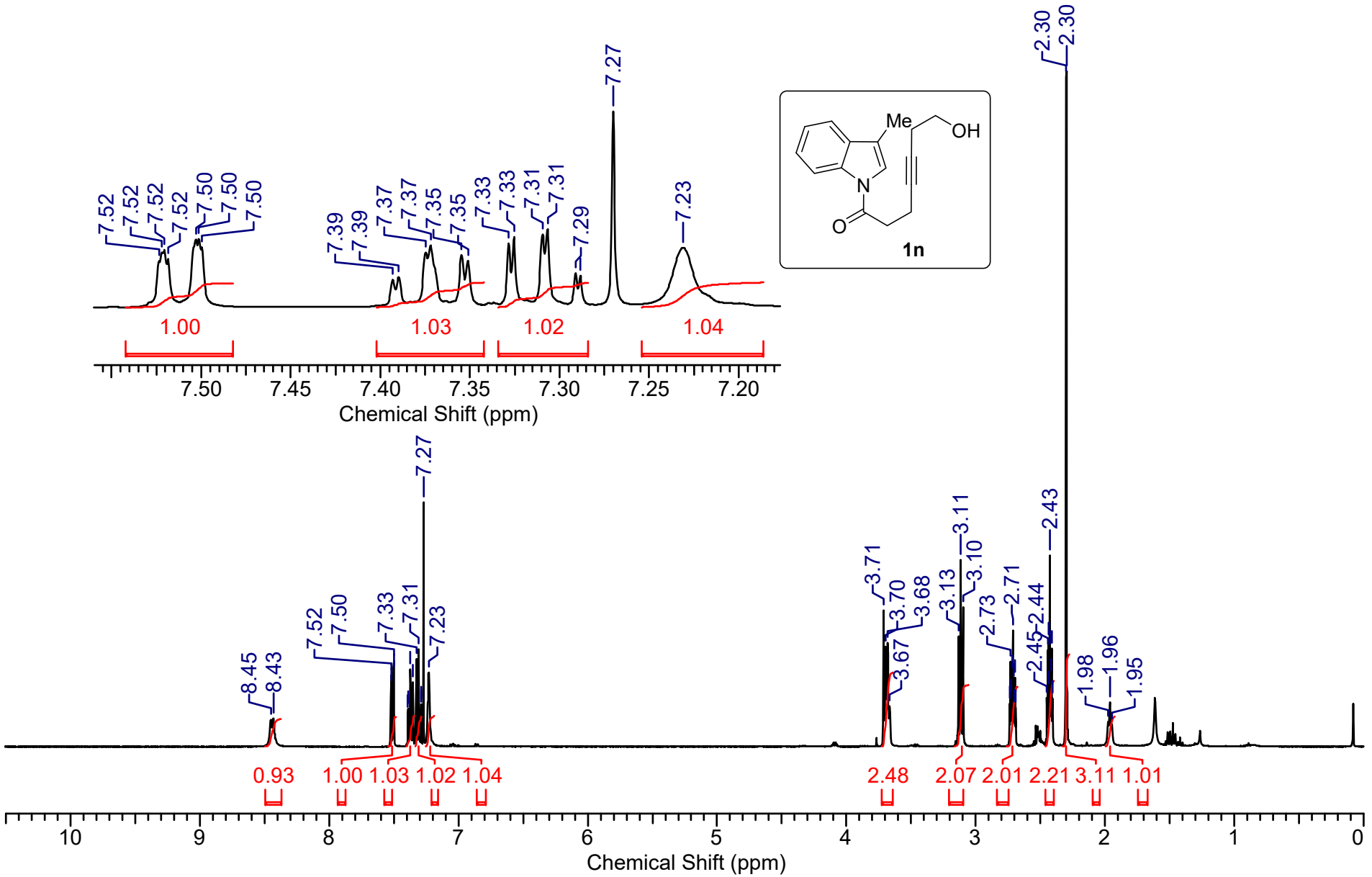


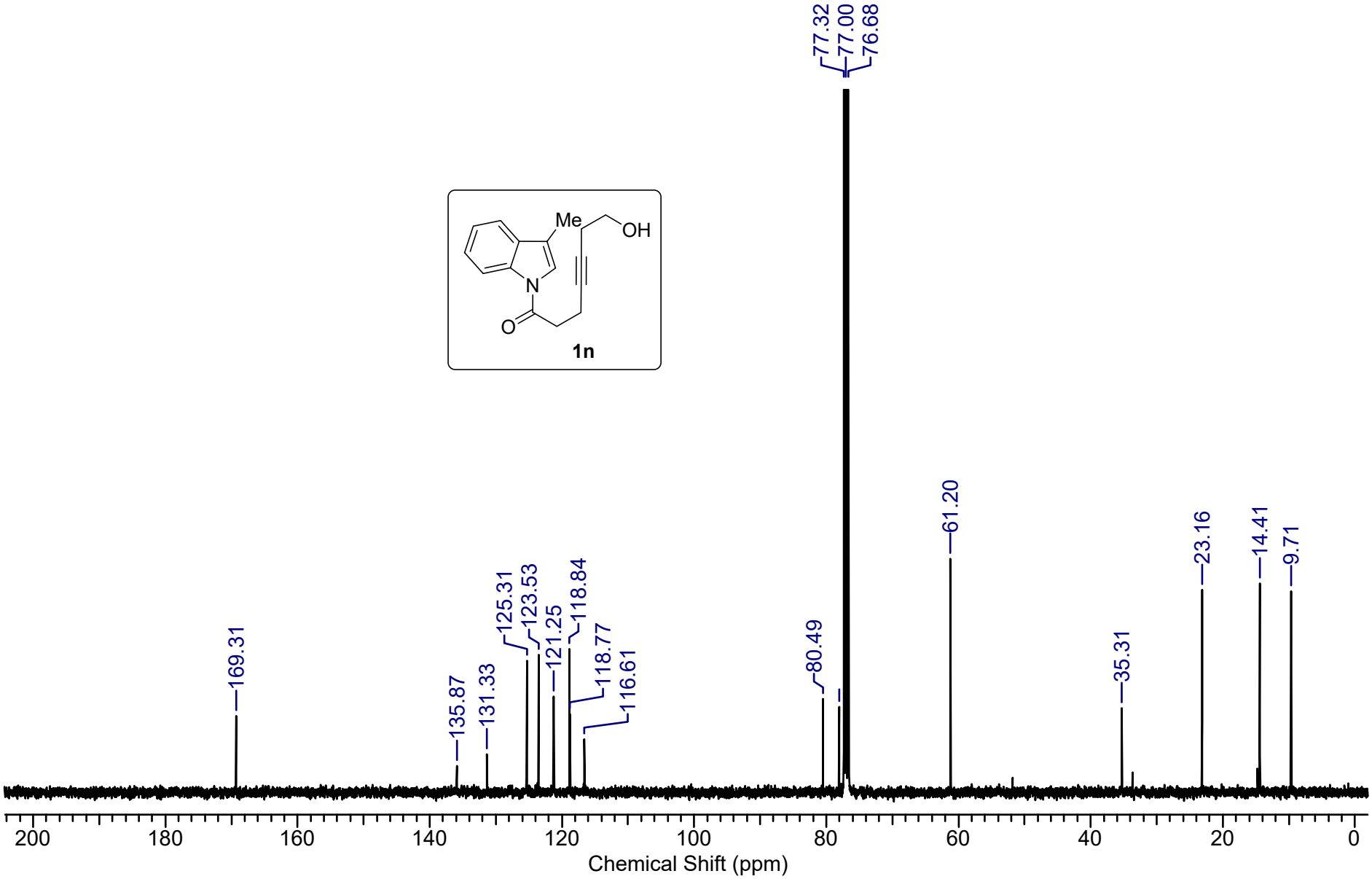

OH
Me
N
O
1m
7.44
7.43
7.42
7.40
7.27
7.23
7.22
7.20
7.17
7.15
2.57
3.16
1.00
1.03
1.03
1.04
7.45
7.40
7.35
7.30
7.25
7.20
7.15
Chemical Shift (ppm)
6.66
6.65
3.68
3.69
3.66
3.18
3.15
2.73
2.75
2.76
2.42
2.42
2.42
2.41
2.40
1.00
1.03
1.03
1.04
1.00
2.26
2.06
2.09
3.22
2.37
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

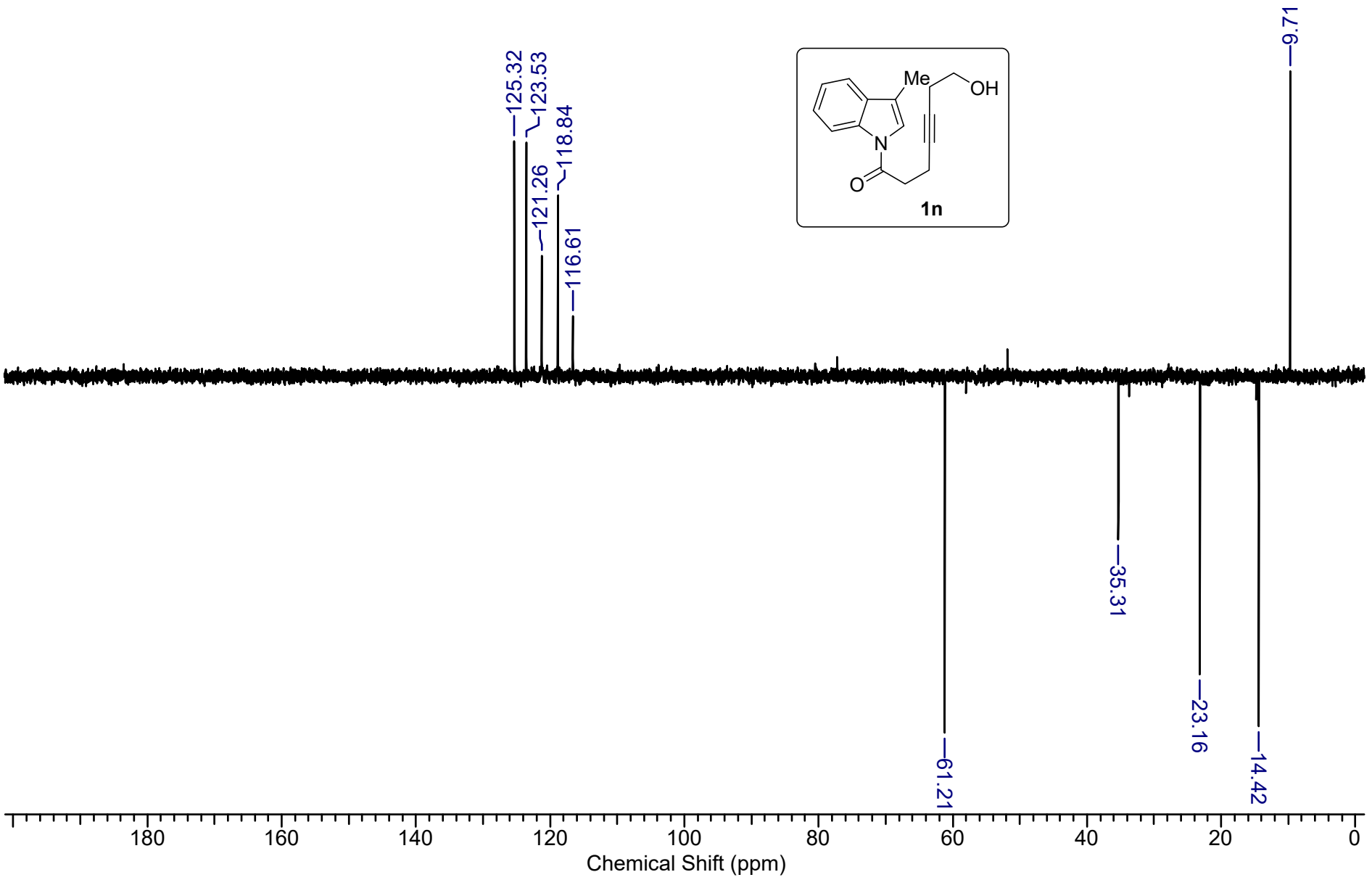

CHLOROFORM-d
77.32
77.00
76.68
135.14
131.92
128.26
126.65
125.67
124.15
134
132
130
128
126
124
Chemical Shift (ppm)
OH
N
Me
O
1m
169.13
135.14
131.92
128.26
125.67
124.15
118.51
109.29
80.22
61.17
36.00
23.11
22.60
15.12
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


128.26
125.67
124.15
118.51
109.28
61.17
36.00
23.10
15.12
OH
N
Me
O
1m
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

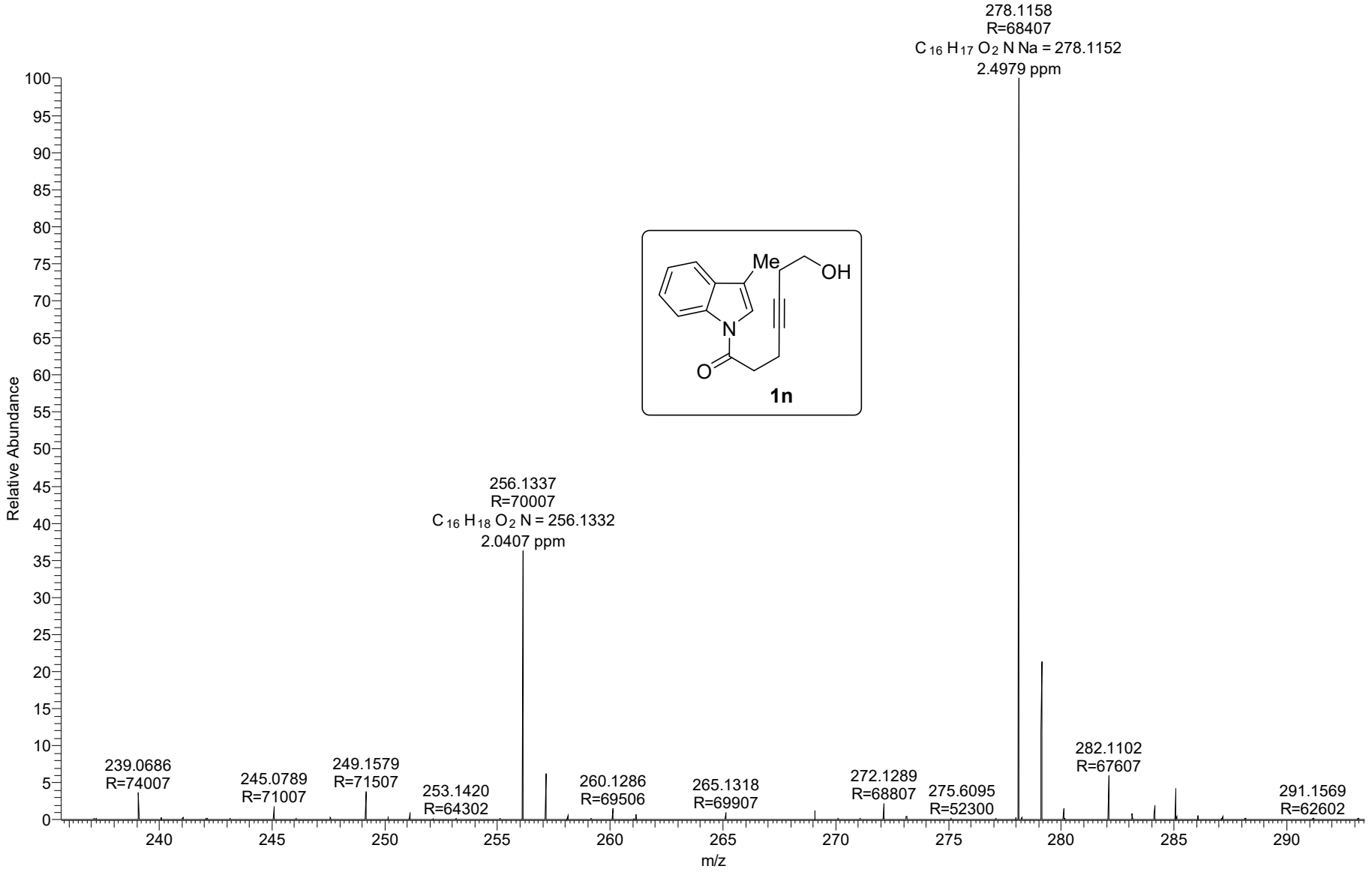

MSH-36 #288 RT: 1.57 AV: 1 NL: 9.59E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
256.1326
R=71407
C 16 H 18 O 2 N = 256.1332
-2.3678 ppm
278.1142
R=68007
C 16 H 17 O 2 N Na = 278.1152
-3.5372 ppm
214.0856
R=75307
C 13 H 12 O 2 N = 214.0863
-3.1231 ppm
238.1218
R=73207
C 16 H 16 O N = 238.1226
-3.4206 ppm
232.1904
R=60400
249.1565
R=68007
266.1528
R=70107
287.1740
R=65407
294.0879
R=66107
306.1451
R=64407
315.1955
R=55302
324.1067
R=62607
340.0843
R=61307
349.1823
R=55102
OH
N
Me
O
1m
Relative Abundance
m/z

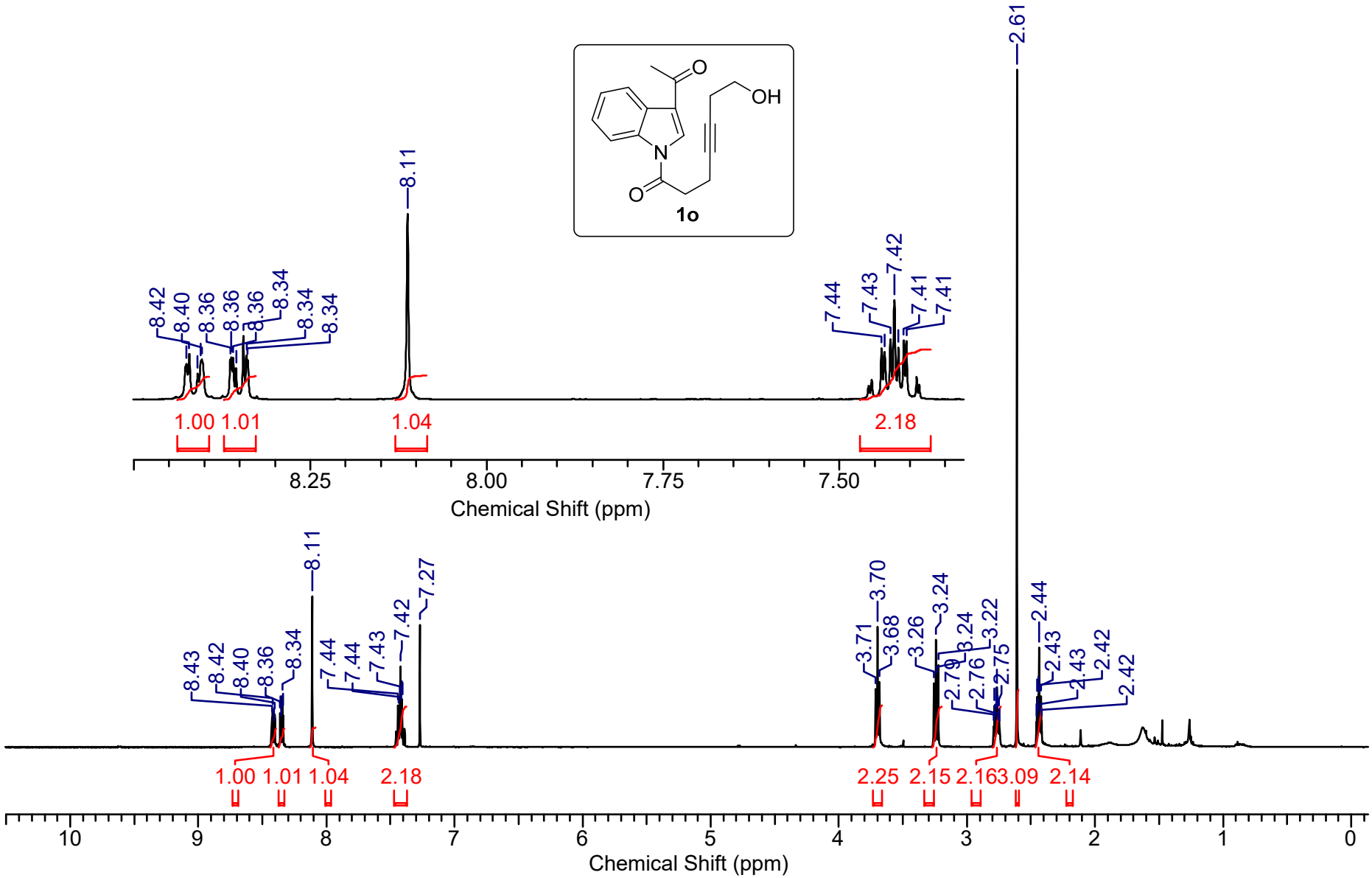

Me
OH
N
O
1n
Chemical Shift (ppm)
Chemical Shift (ppm)

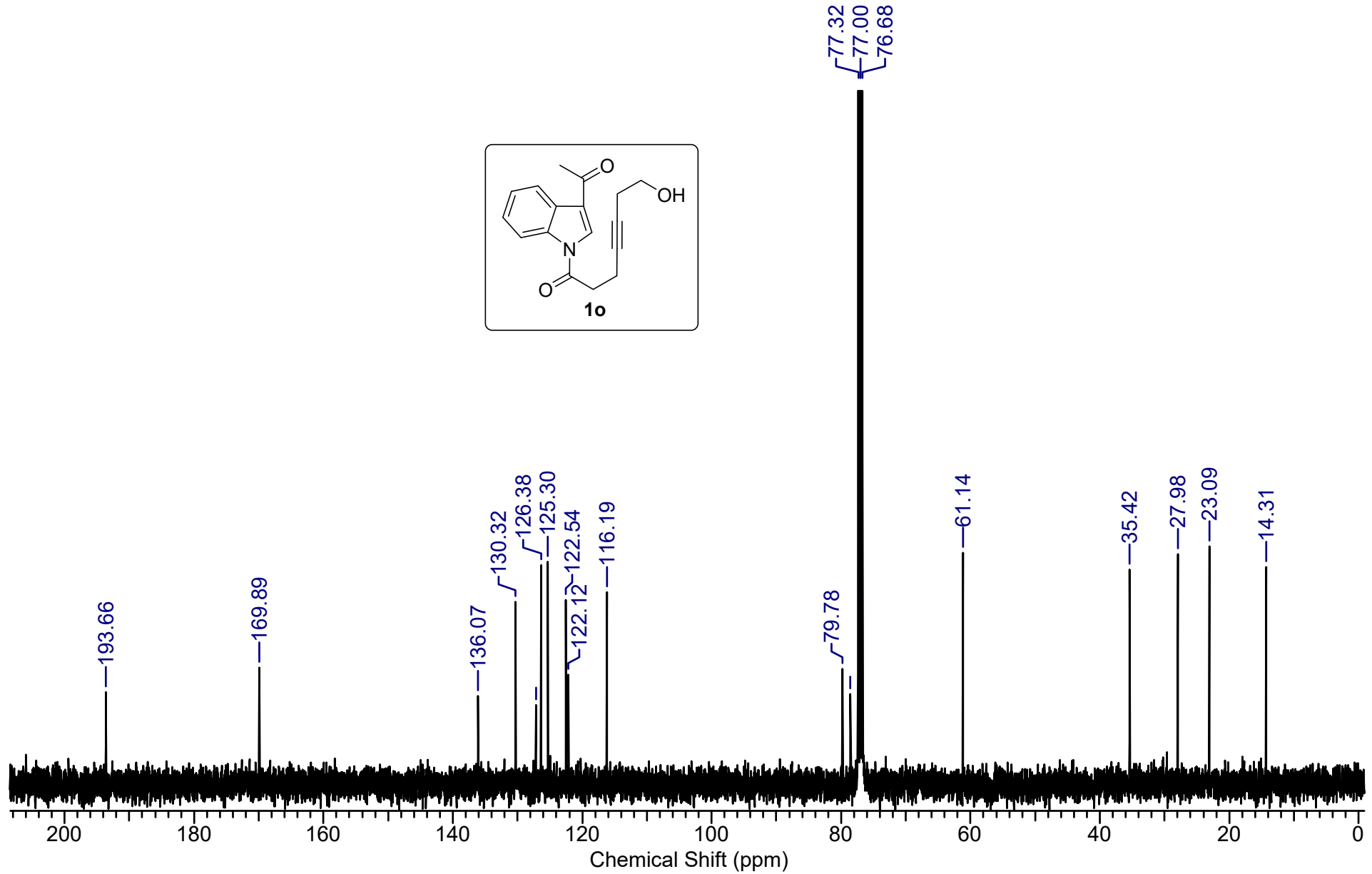

77.32
77.00
76.68
Me
OH
N
O
1n
169.31
135.87
131.33
125.31
123.53
121.25
118.84
118.77
116.61
80.49
61.20
35.31
23.16
14.41
9.71
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

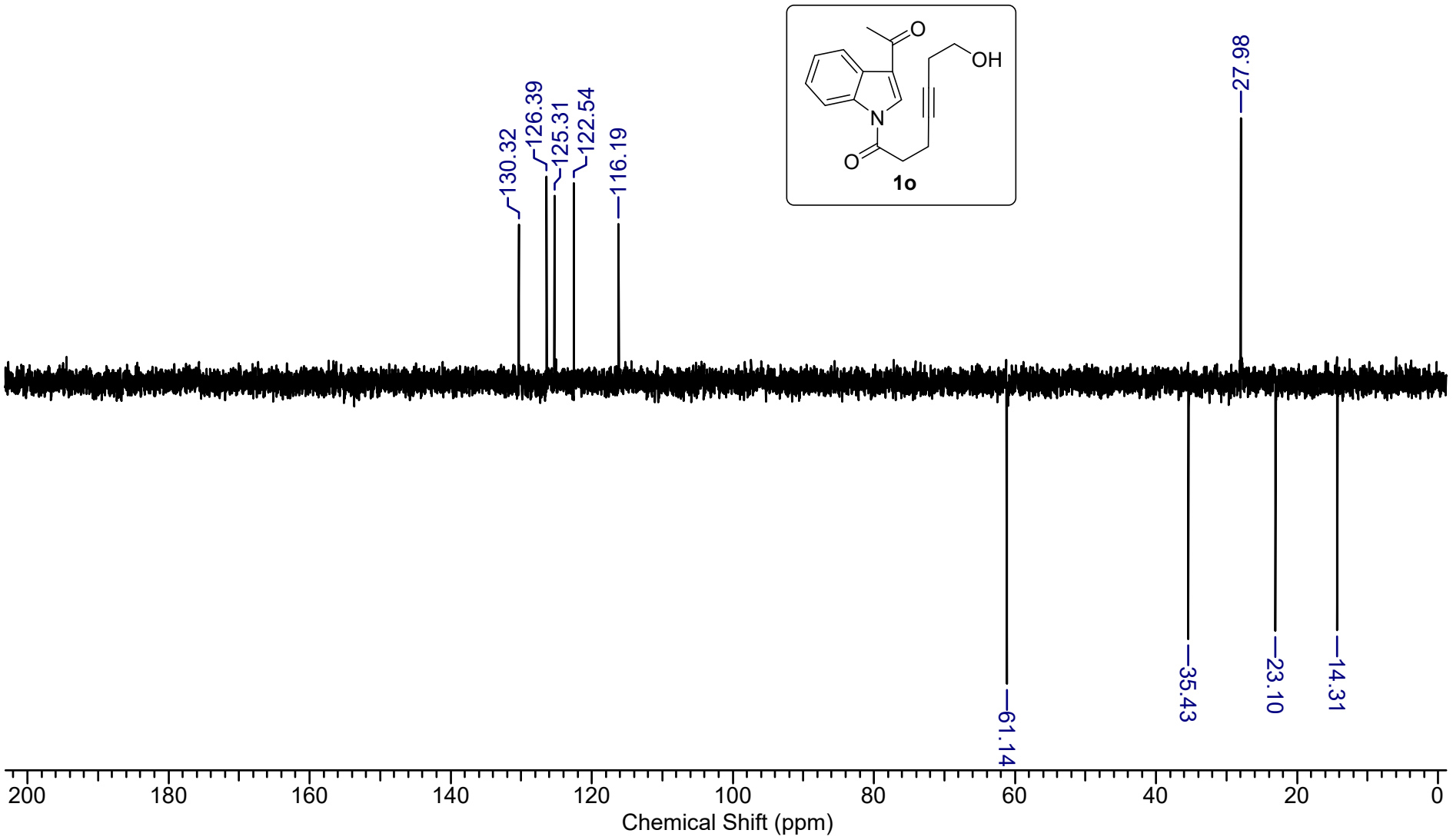

125.32
123.53
121.26
118.84
116.61
Me
OH
N
O
1n
17.71
35.31
23.16
14.42
61.21
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-10 #275 RT: 1.47 AV: 1 NL: 1.83E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

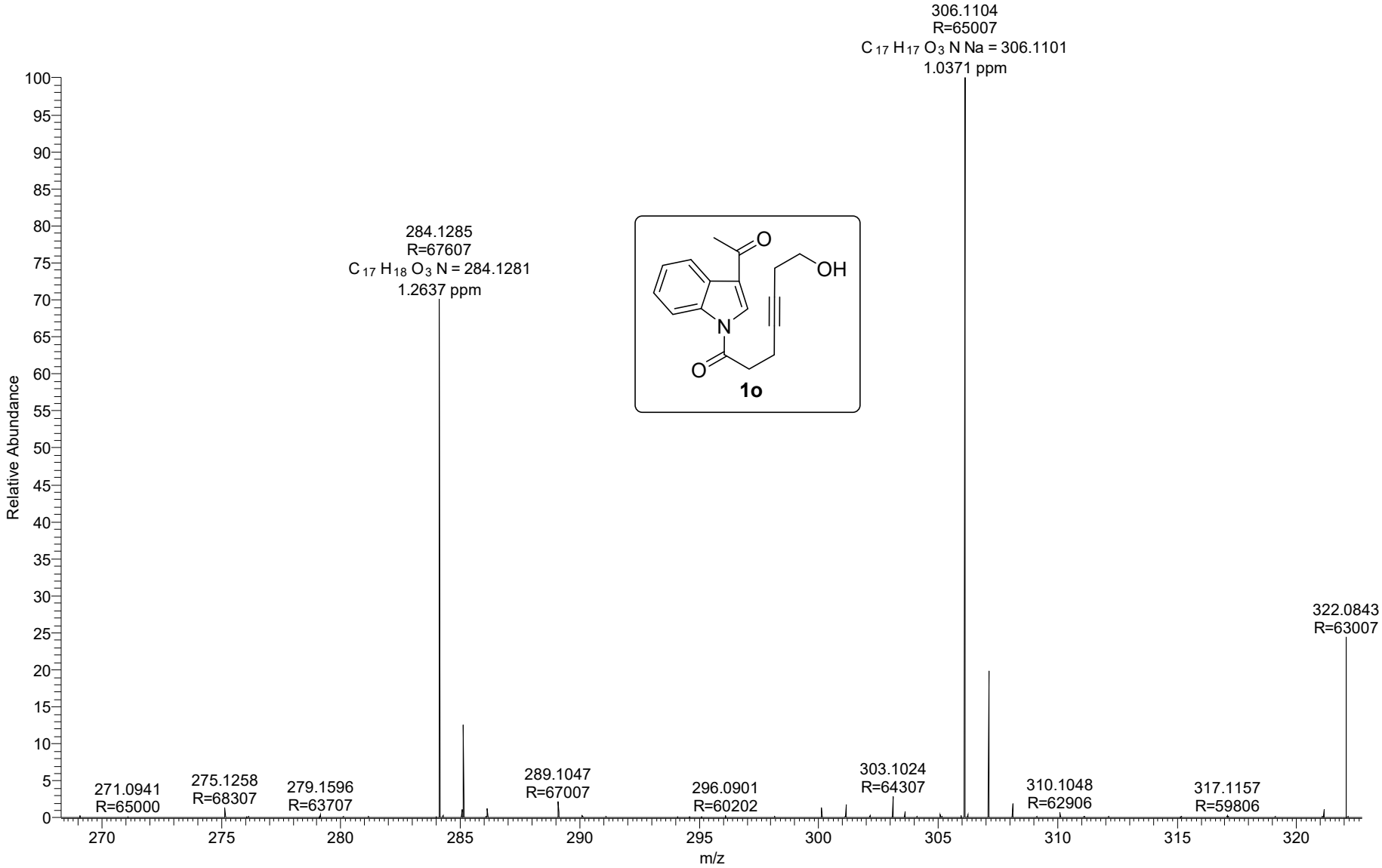


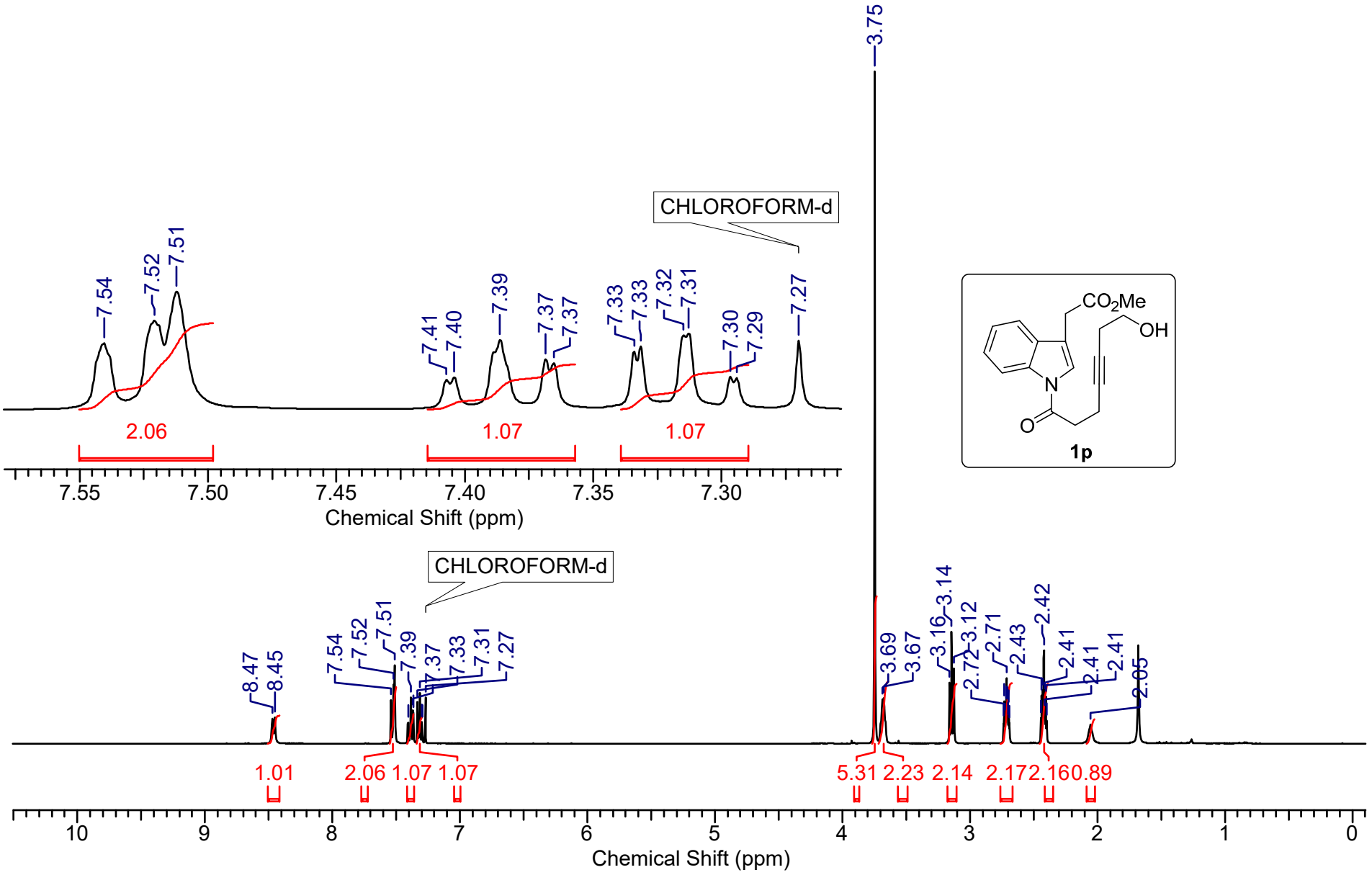

O
OH
N
O
1o
8.42
8.40
8.36
8.36
8.36
8.34
8.34
8.34
8.11
7.44
7.43
7.42
7.41
7.41
1.00 1.01
1.04
2.18
8.25
8.00
7.75
7.50
Chemical Shift (ppm)
2.61
8.43
8.42
8.40
8.36
8.34
8.11
7.44
7.44
7.43
7.42
7.27
3.71
3.70
3.68
3.26
3.24
3.24
3.22
2.79
2.76
2.75
2.44
2.43
2.43
2.42
2.42
1.00 1.01 1.04
2.18
2.25 2.15 2.16 3.09 2.14
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

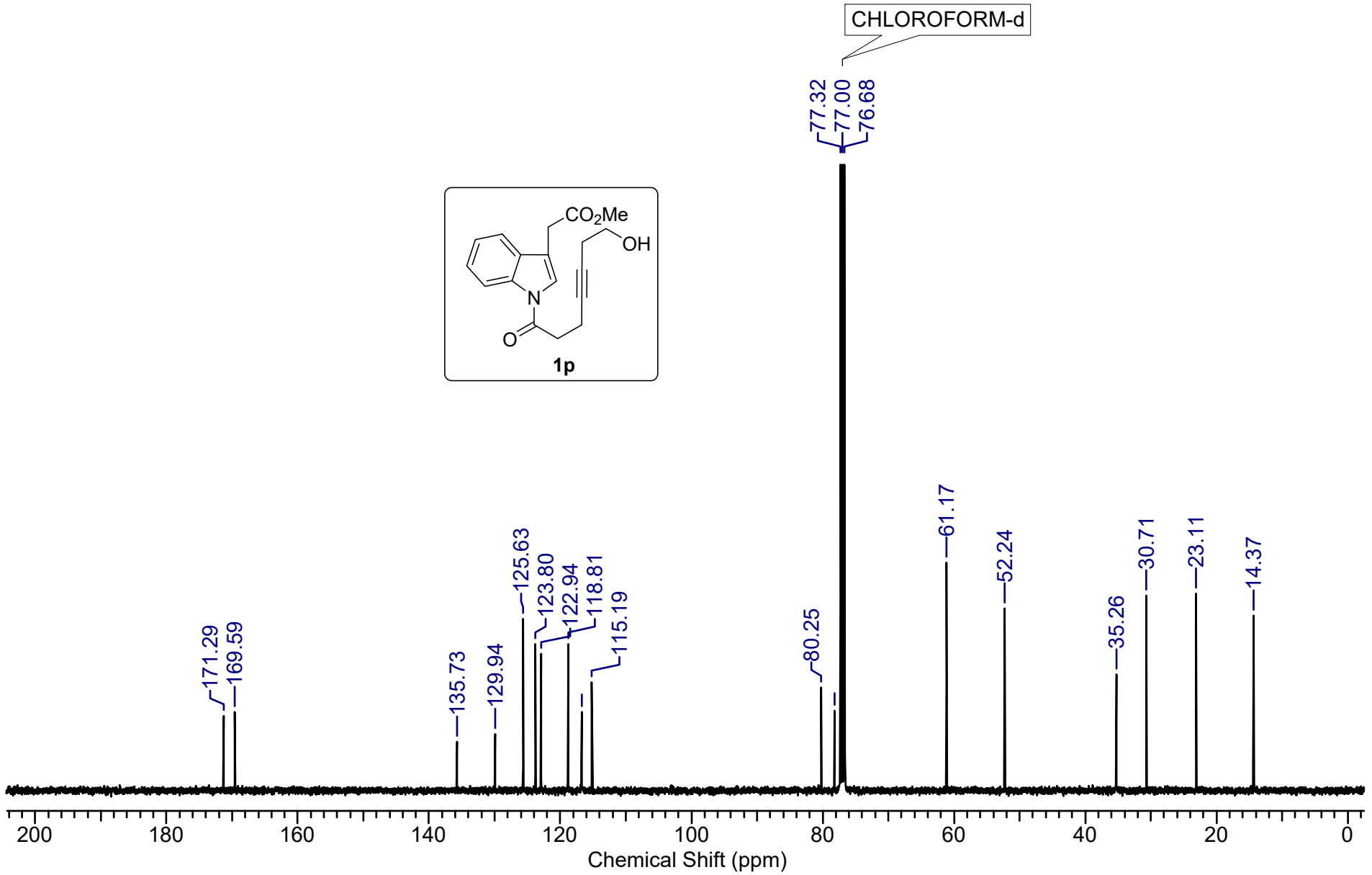

77.32
77.00
76.68
193.66
169.89
136.07
130.32
126.38
125.30
122.54
122.12
116.19
79.78
61.14
35.42
27.98
23.09
14.31
O
OH
N
O
1o
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

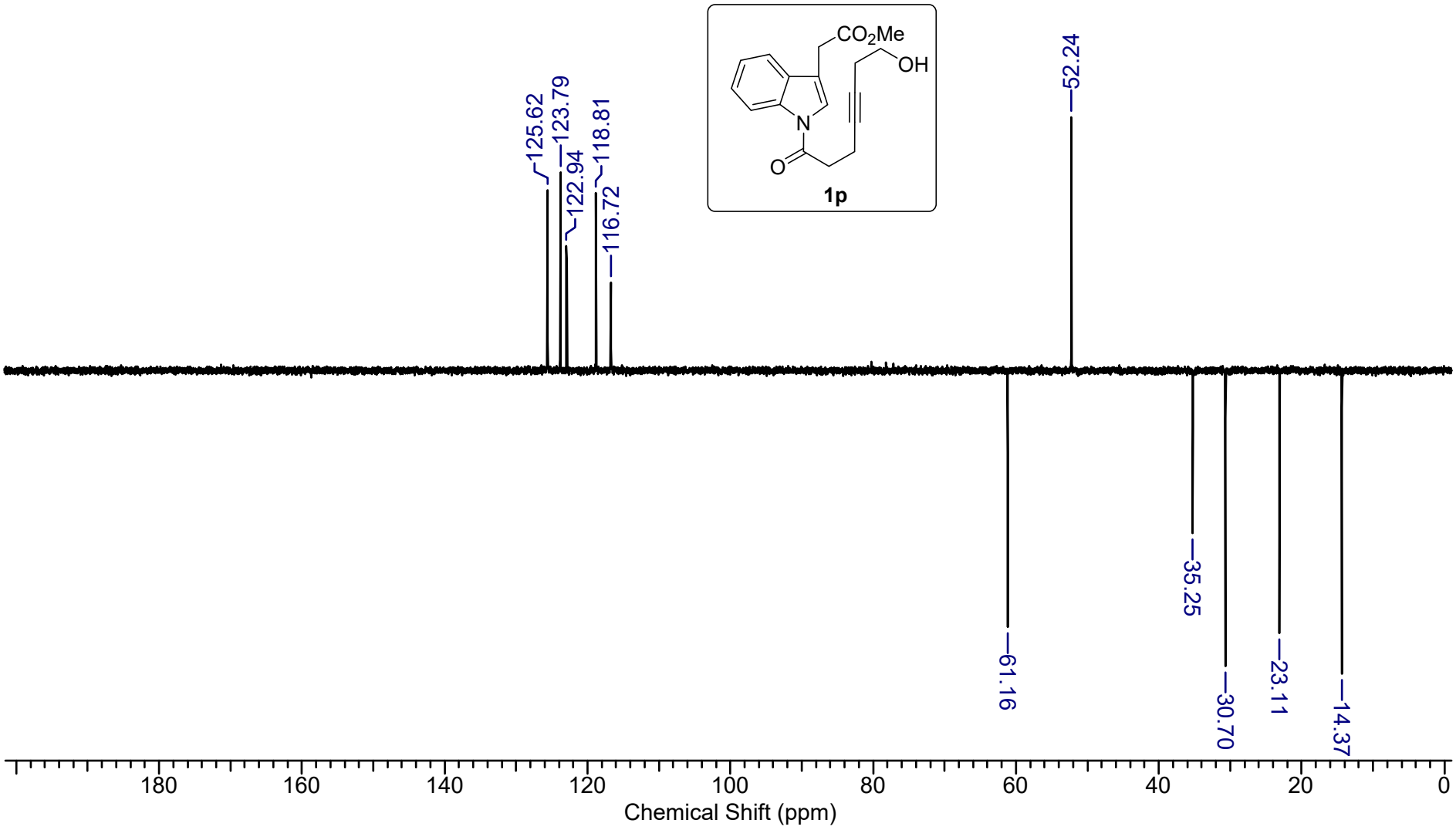

130.32
126.39
125.31
122.54
116.19
27.98
61.14
35.43
23.10
14.31
O
OH
N
O
1o
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-9 #268 RT: 1.43 AV: 1 NL: 3.62E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

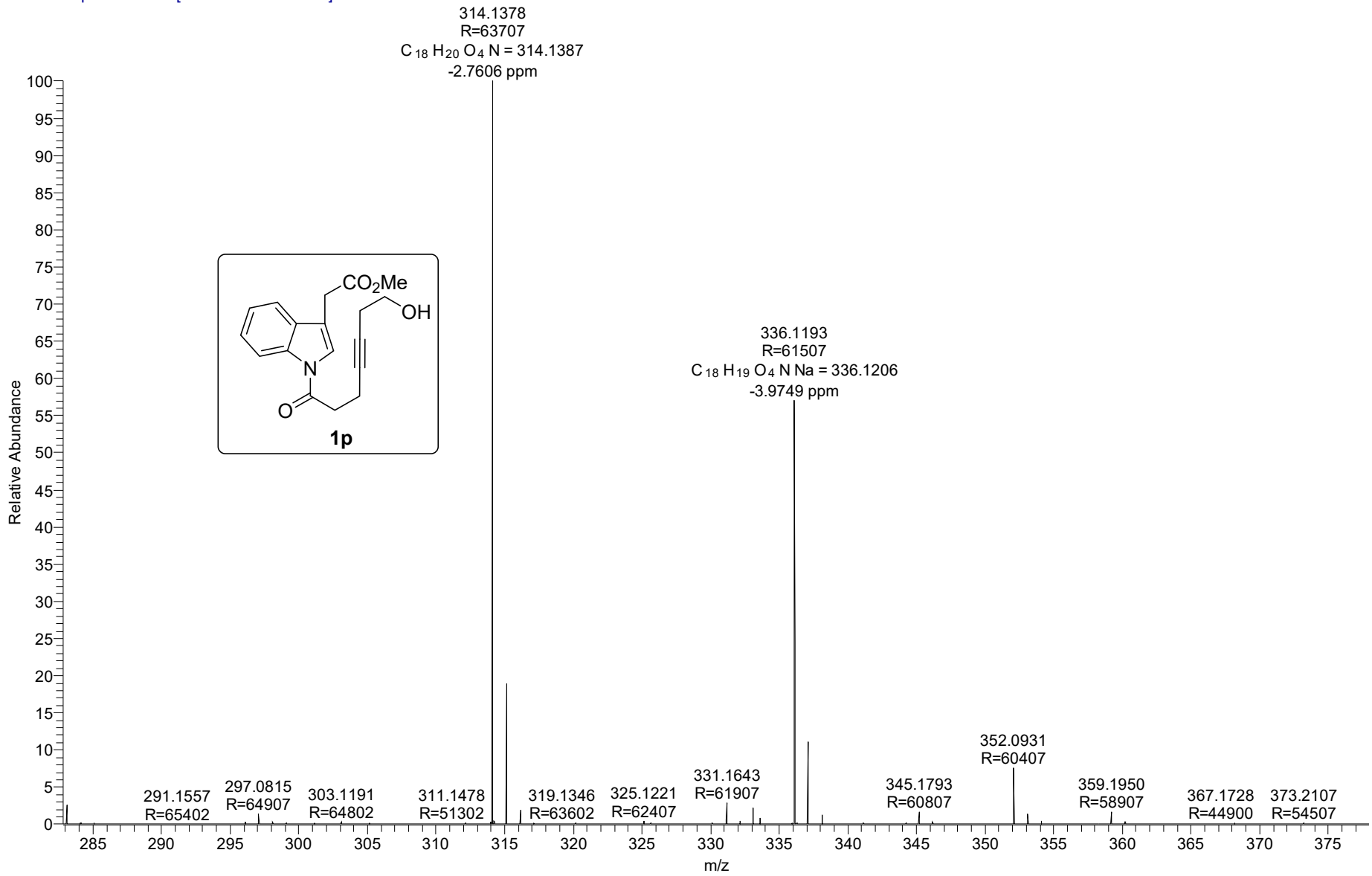


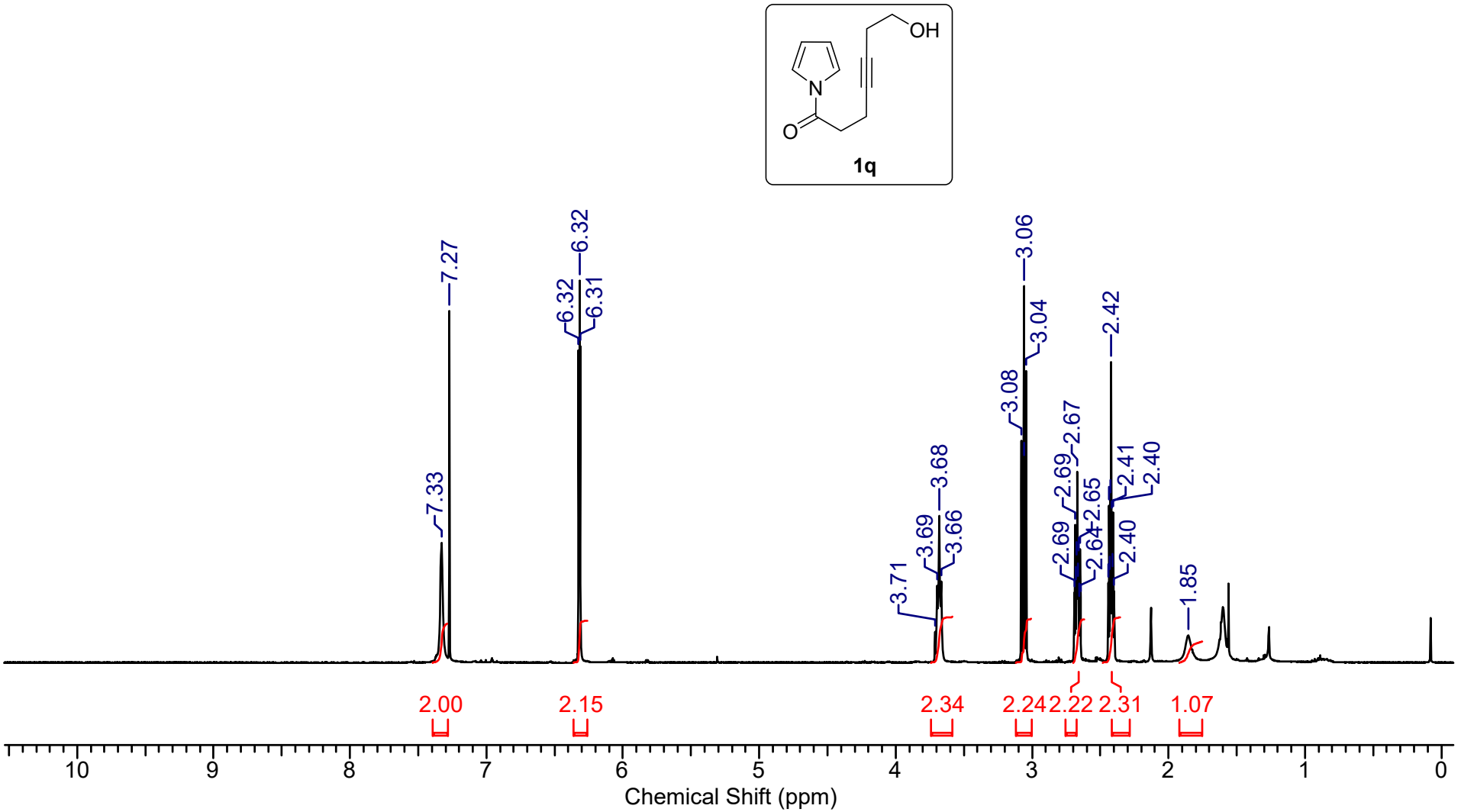

CHLOROFORM-d
7.54
7.52
7.51
7.41
7.40
7.39
7.37
7.37
7.33
7.33
7.32
7.31
7.30
7.29
7.27
2.06
1.07
1.07
7.55
7.50
7.45
7.40
7.35
7.30
Chemical Shift (ppm)
CO2Me
OH
N
O
1p
CHLOROFORM-d
8.47
8.45
7.54
7.52
7.51
7.39
7.37
7.33
7.31
7.27
3.75
3.69
3.67
3.16
3.14
2.72
3.12
2.71
2.43
2.42
2.41
2.41
2.41
2.05
1.01
2.06
1.07
1.07
5.31
2.23
2.14
2.17
2.16
0.89
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

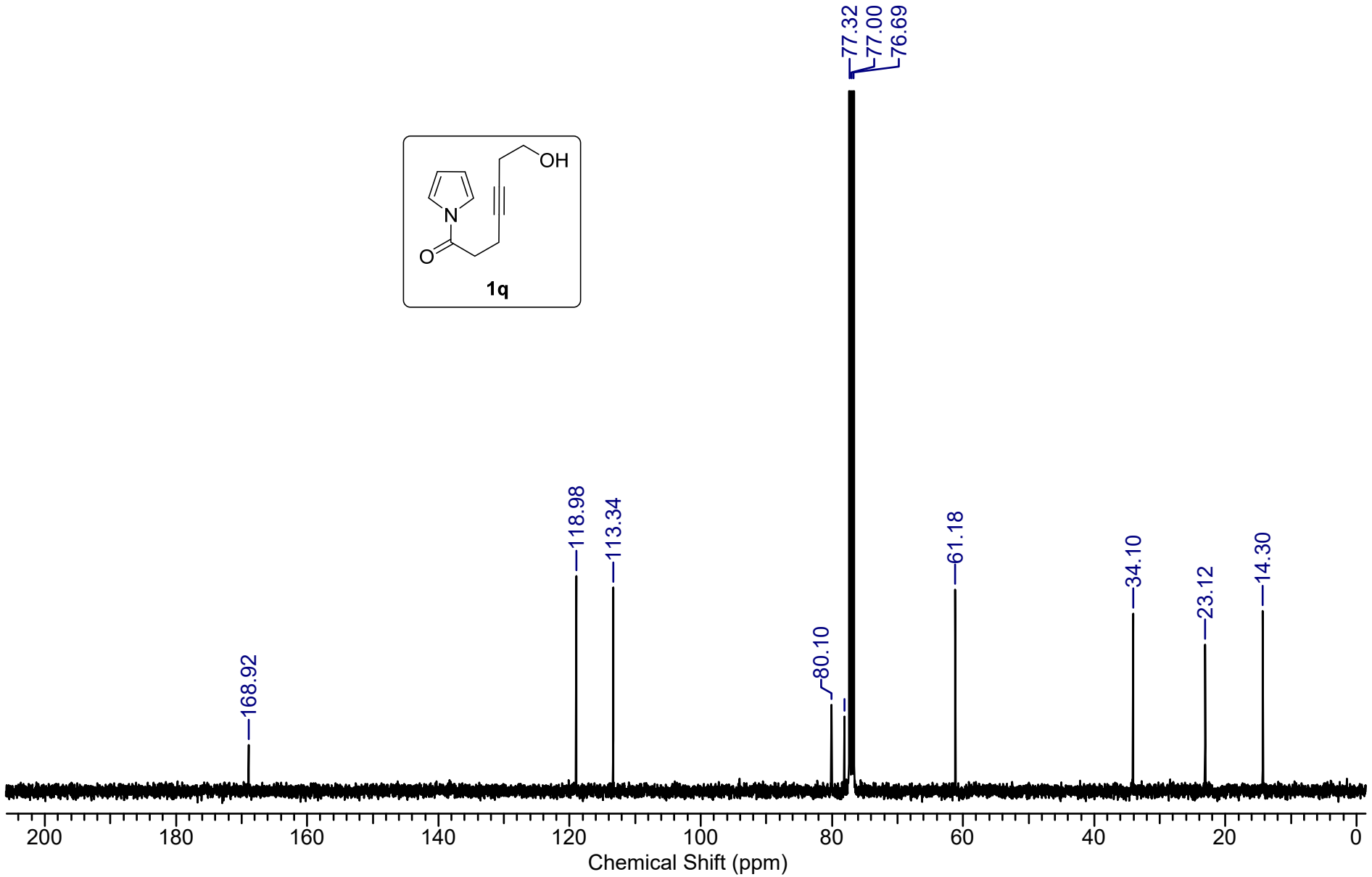

CHLOROFORM-d
77.32
77.00
76.68
CO2Me
OH
N
O
1p
171.29
169.59
135.73
129.94
125.63
123.80
122.94
118.81
115.19
80.25
61.17
52.24
35.26
30.71
23.11
14.37
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

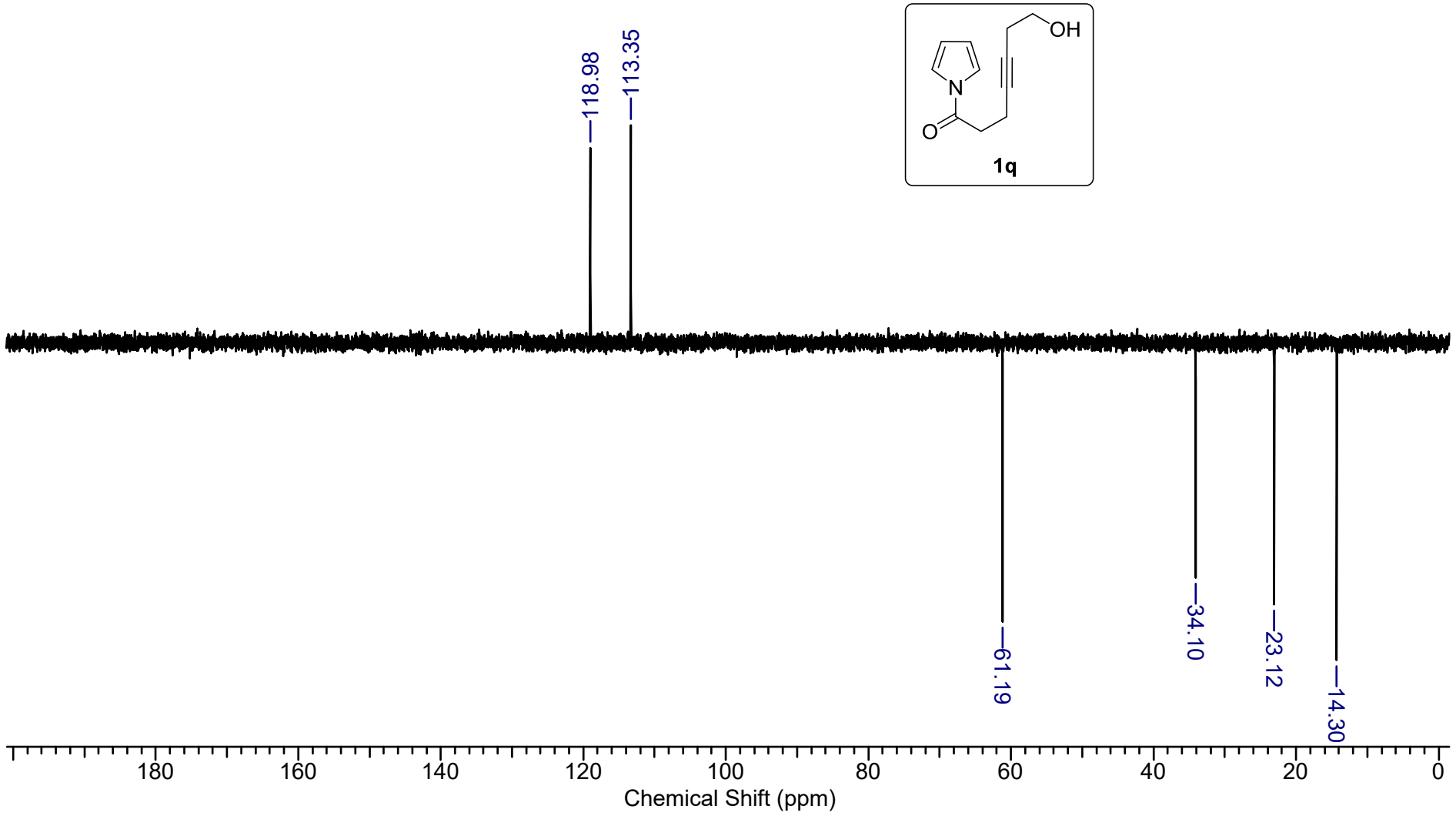

CO2Me
OH
N
O
1p
125.62
123.79
122.94
118.81
116.72
52.24
61.16
35.25
30.70
23.11
14.37
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

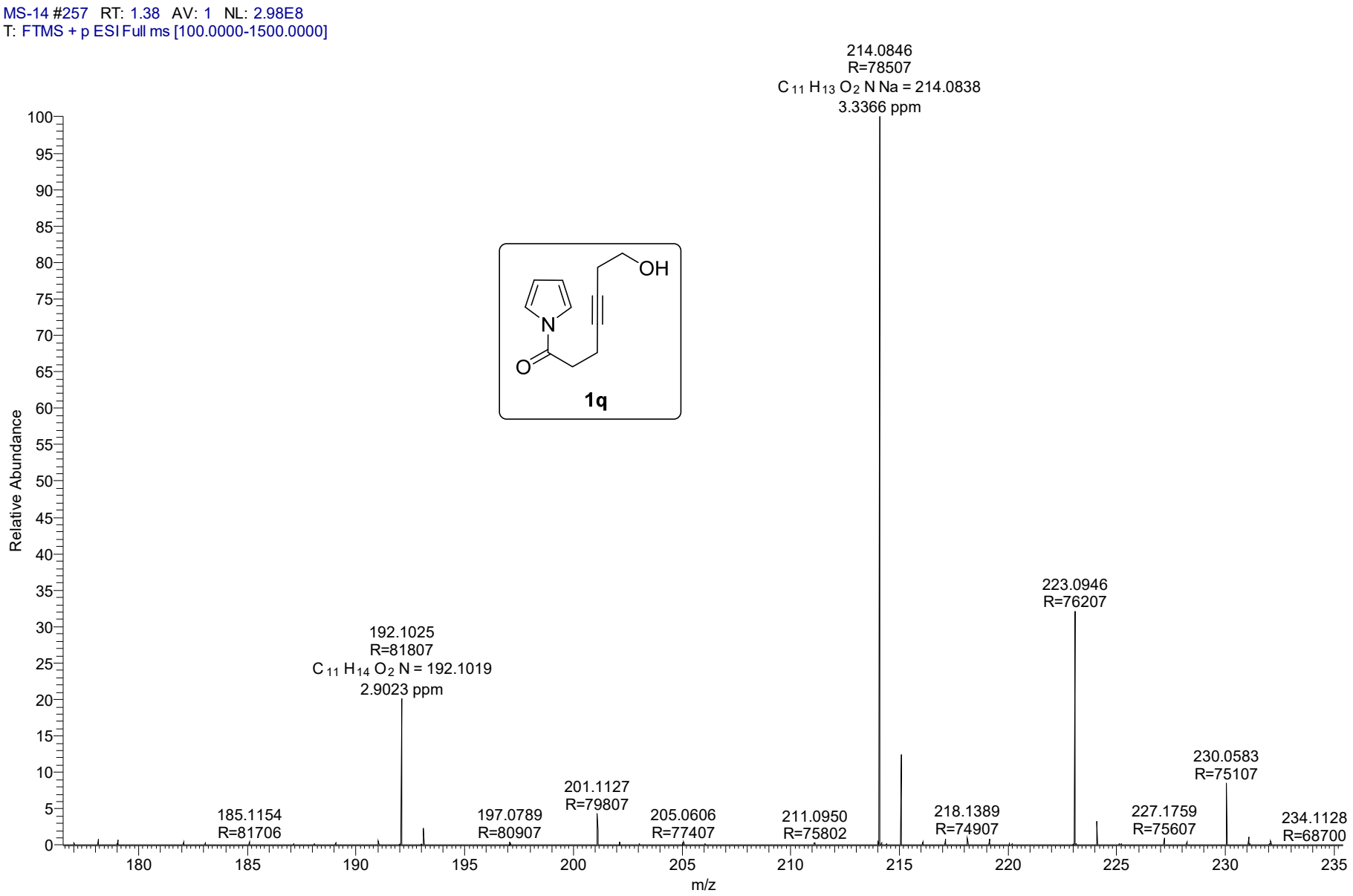
MSH-39 #267 RT: 1.46 AV: 1 NL: 5.41E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

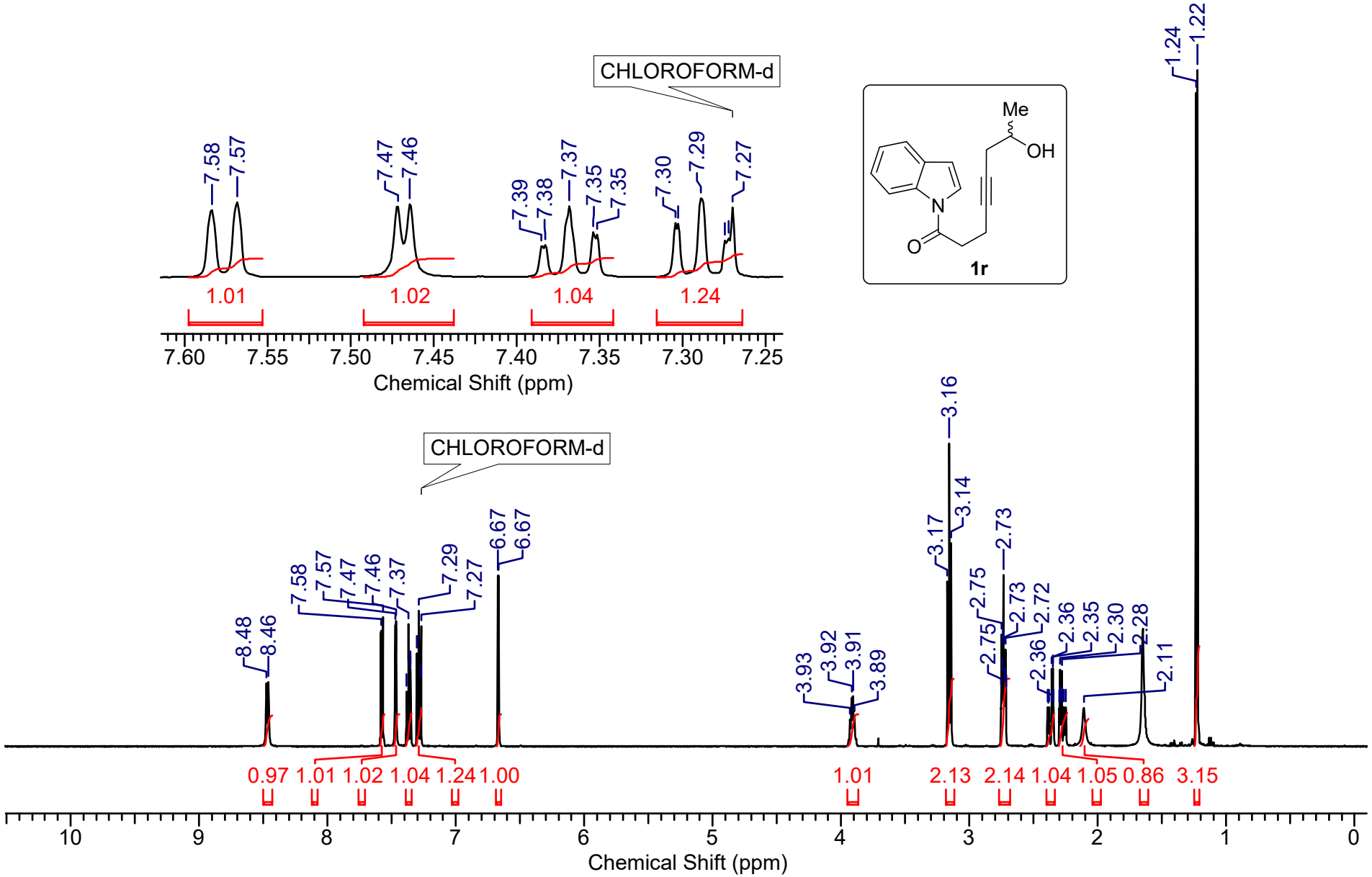


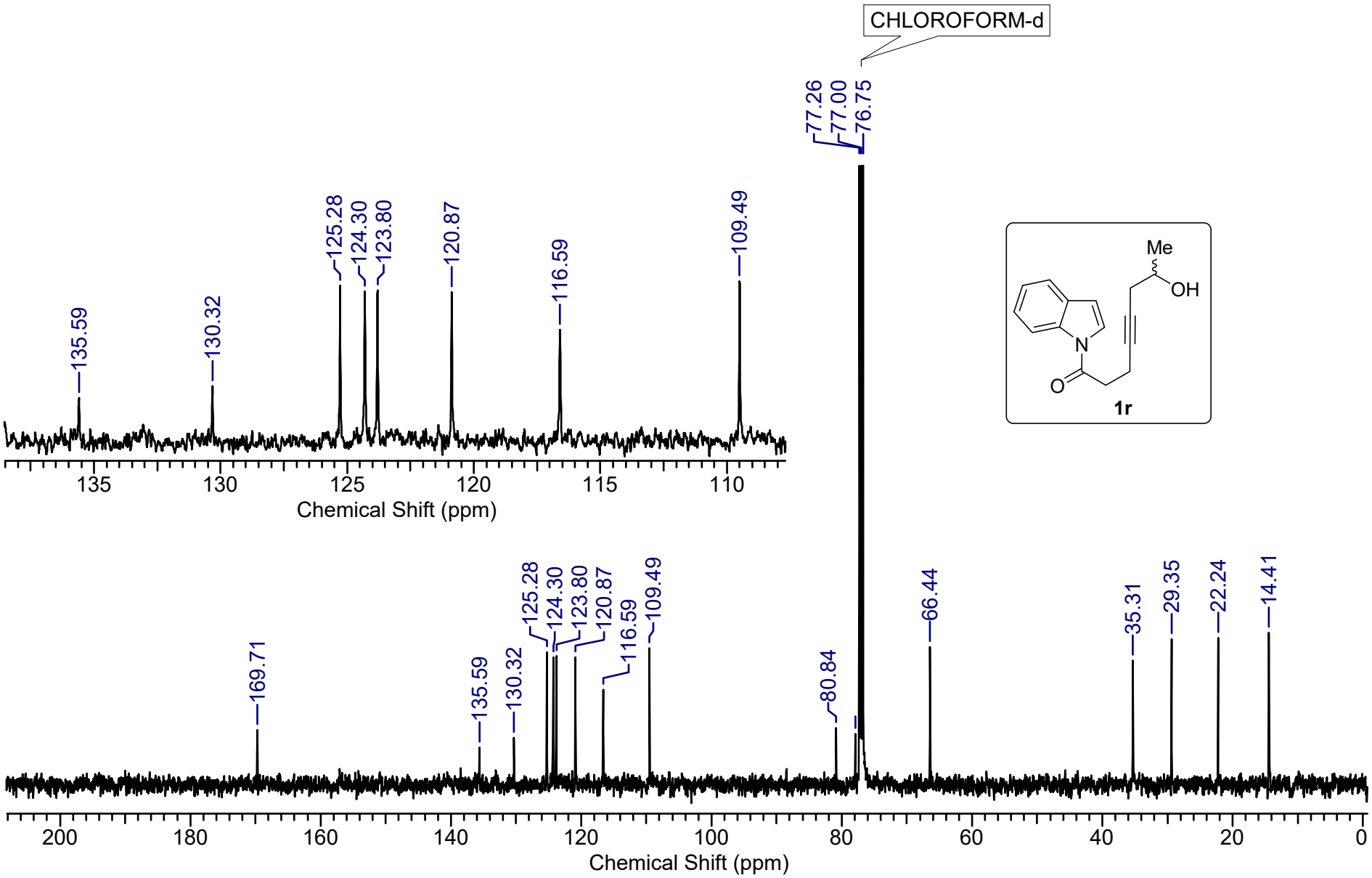

OH
N
O
1q
7.27
6.32
6.32
6.31
7.33
3.71
3.69
3.68
3.66
3.08
3.06
3.04
2.69
2.67
2.65
2.64
2.42
2.41
2.40
2.40
1.85
2.00
2.15
2.34
2.24
2.22
2.31
1.07
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

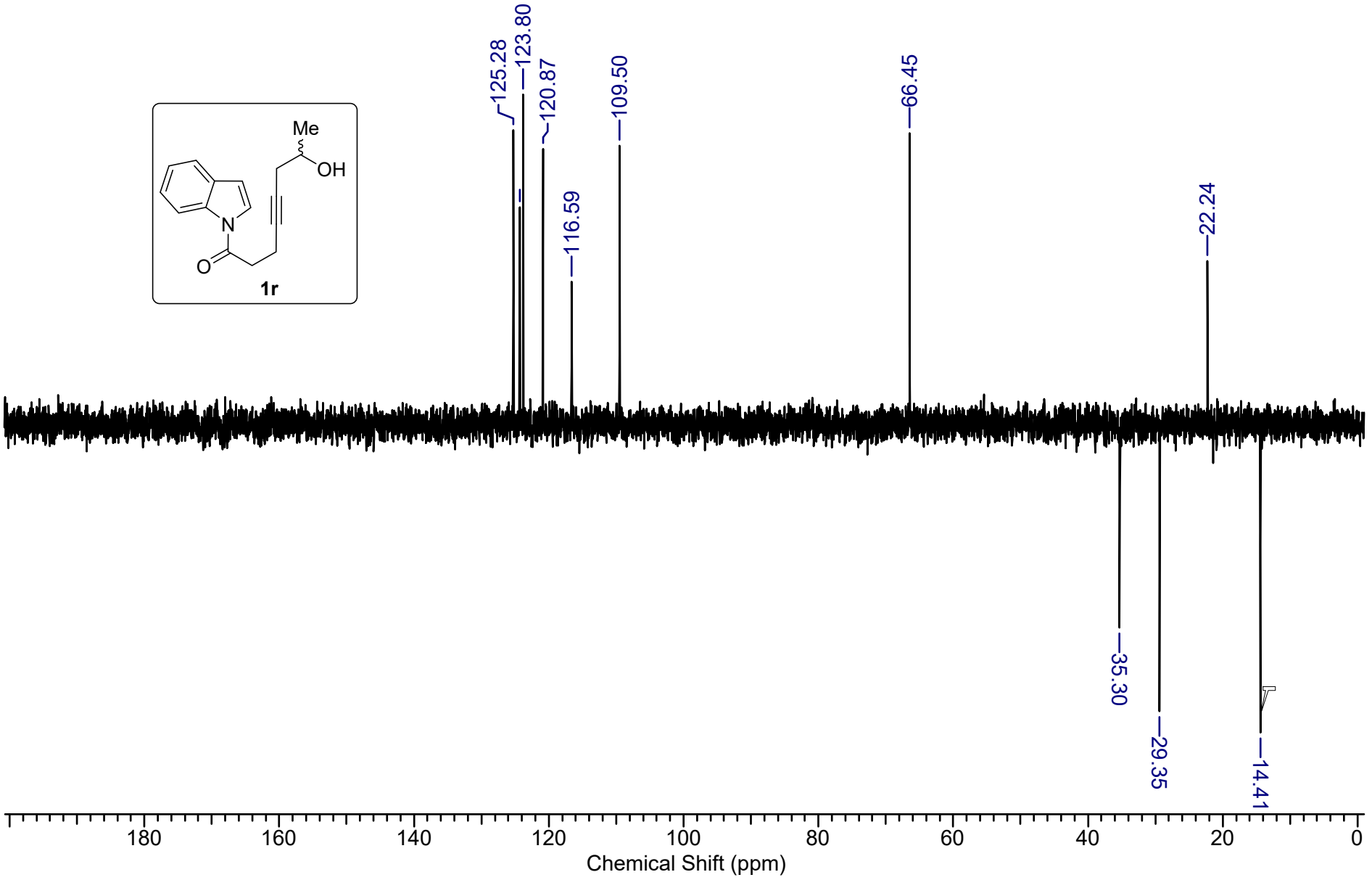

77.32
77.00
76.69
OH
N
O
1q
168.92
118.98
113.34
80.10
61.18
34.10
23.12
14.30
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

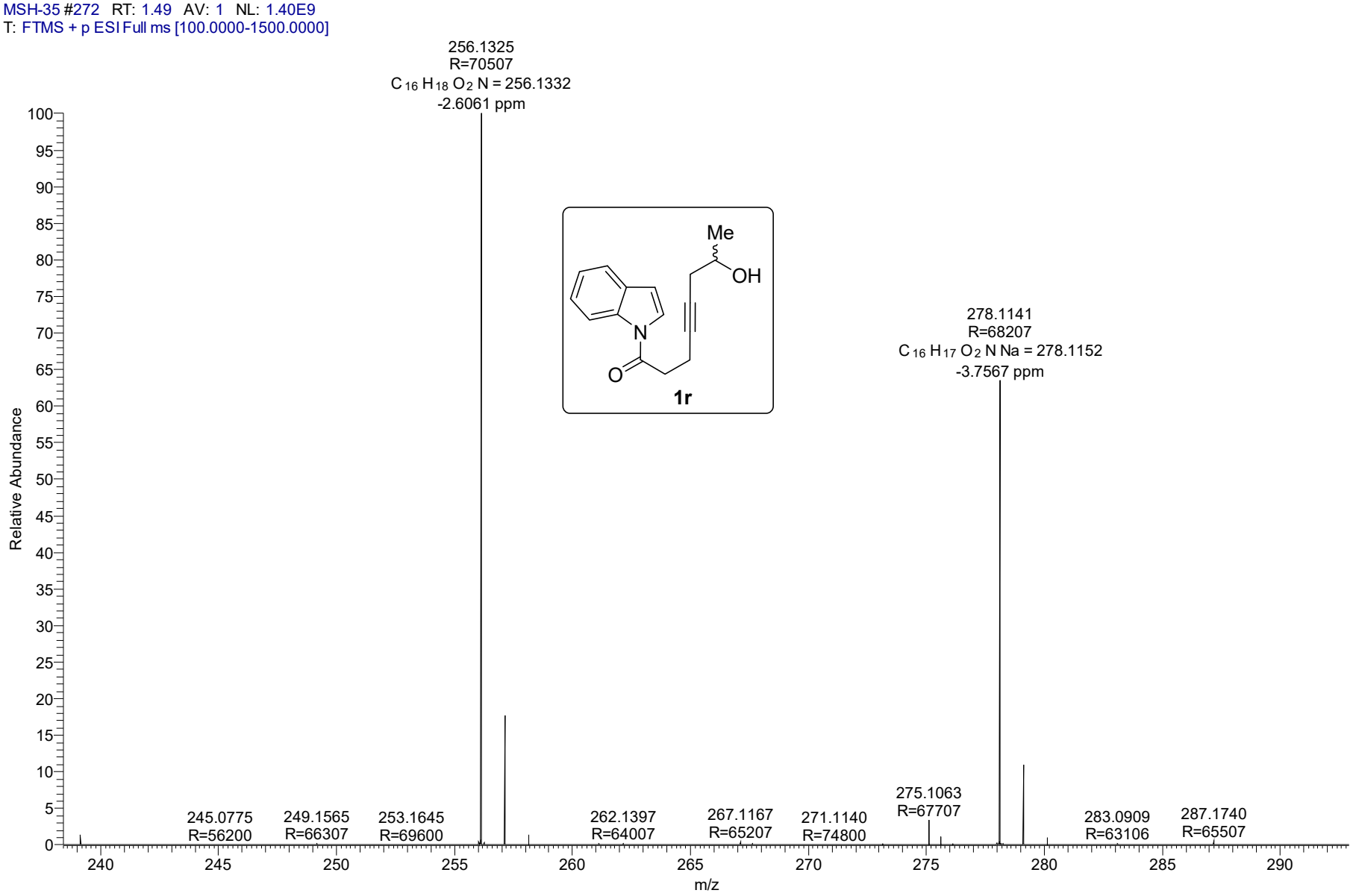

118.98
113.35
OH
N
O
1q
61.19
34.10
23.12
14.30
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

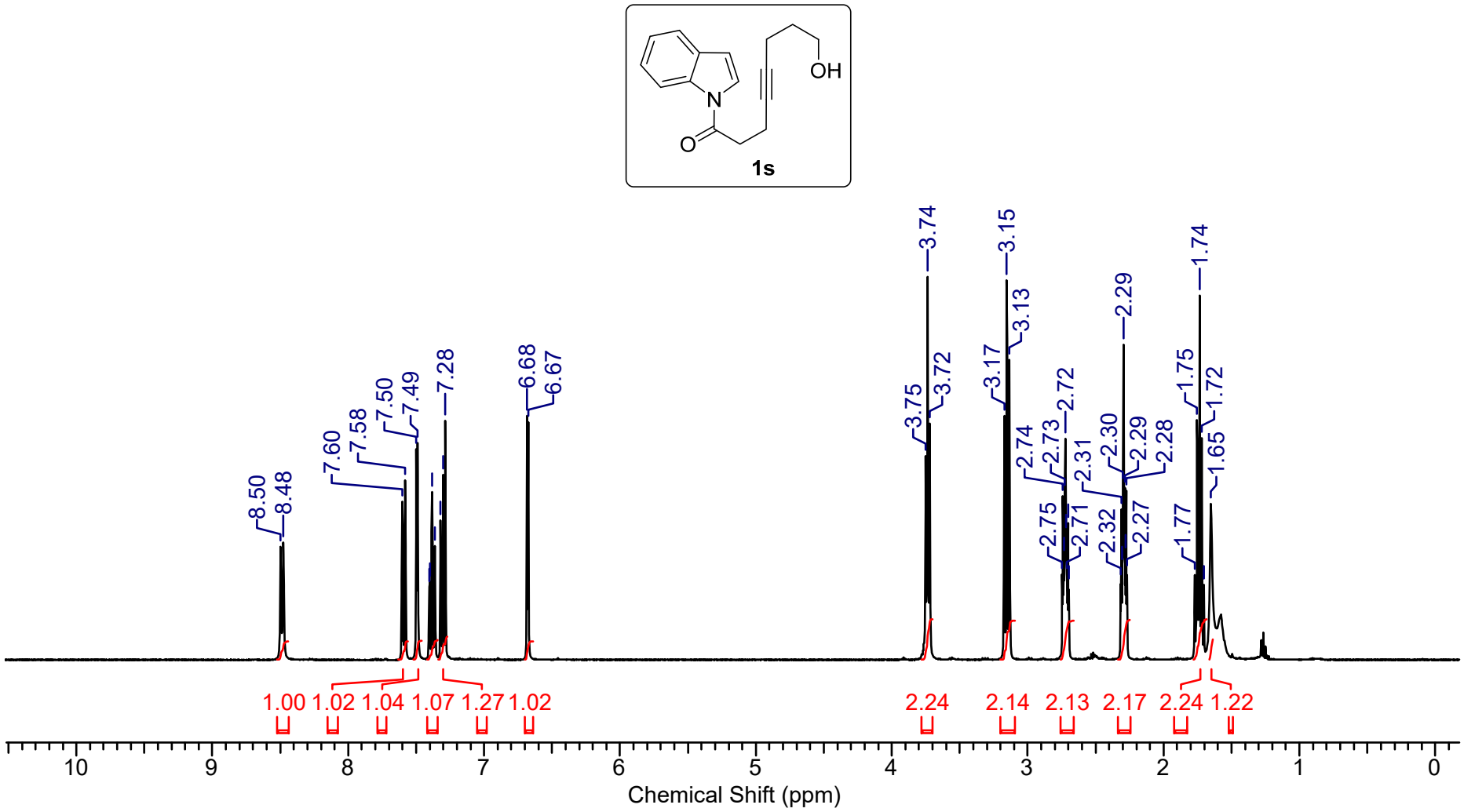

MS-14 #257 RT: 1.38 AV: 1 NL: 2.98E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
214.0846
R=78507
C 11 H 13 O 2 N Na = 214.0838
3.3366 ppm
OH
N
O
1q
Relative Abundance
m/z
192.1025
R=81807
C 11 H 14 O 2 N = 192.1019
2.9023 ppm
223.0946
R=76207
230.0583
R=75107
185.1154
R=81706
197.0789
R=80907
201.1127
R=79807
205.0606
R=77407
211.0950
R=75802
218.1389
R=74907
227.1759
R=75607
234.1128
R=68700

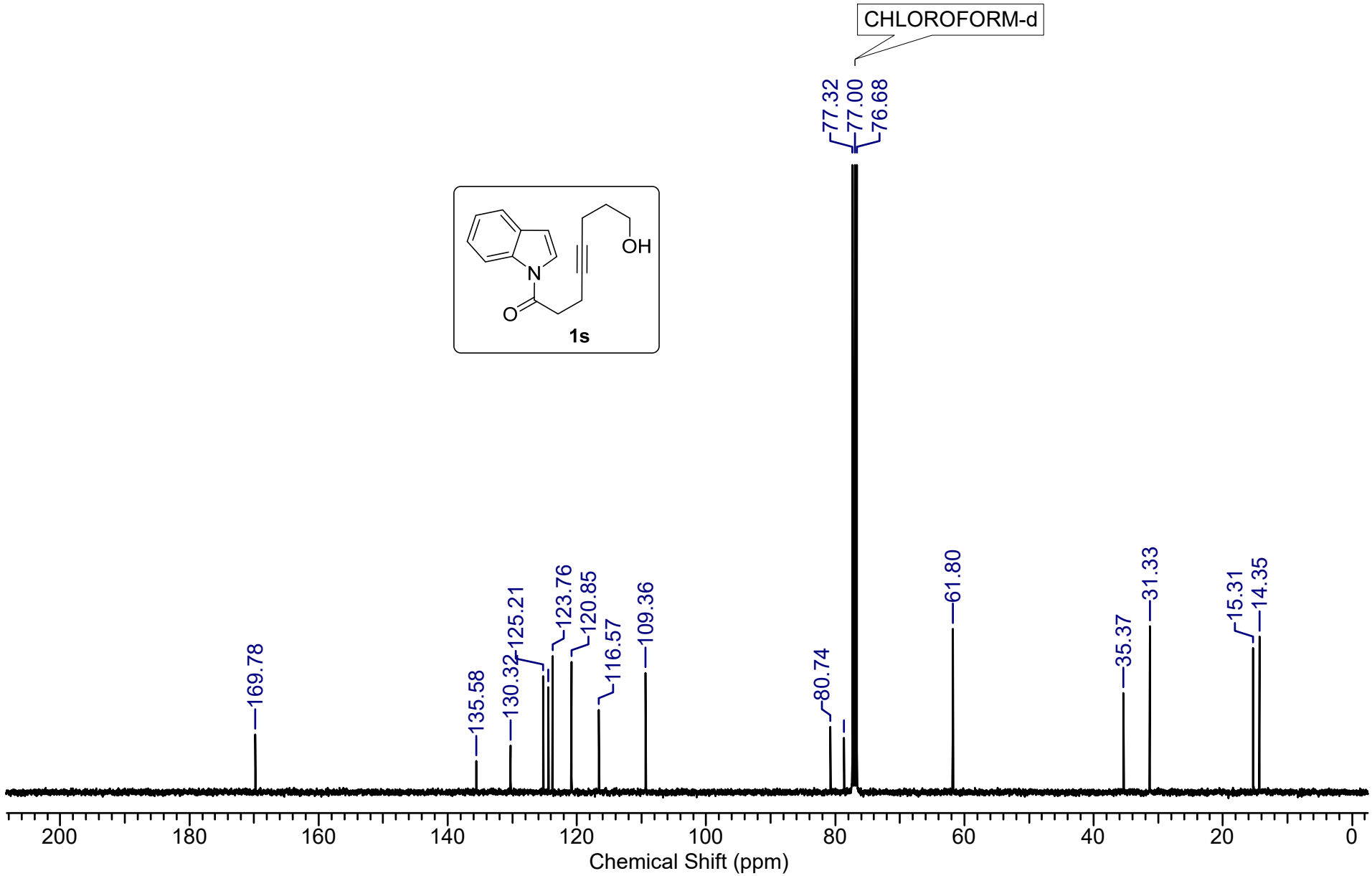

CHLOROFORM-d
7.58
7.57
7.47
7.46
7.39
7.38
7.37
7.35
7.35
7.30
7.29
7.27
1.01
1.02
1.04
1.24
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
Me
OH
N
O
1r
1.24
1.22
CHLOROFORM-d
3.16
3.14
3.17
2.73
2.75
2.73
2.72
2.36
2.35
2.30
2.28
2.11
2.75
2.36
3.93
3.92
3.91
3.89
8.48
8.46
7.58
7.57
7.47
7.46
7.37
7.29
7.27
6.67
6.67
0.97
1.01
1.02
1.04
1.24
1.00
1.01
2.13
2.14
1.04
1.05
0.86
3.15
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

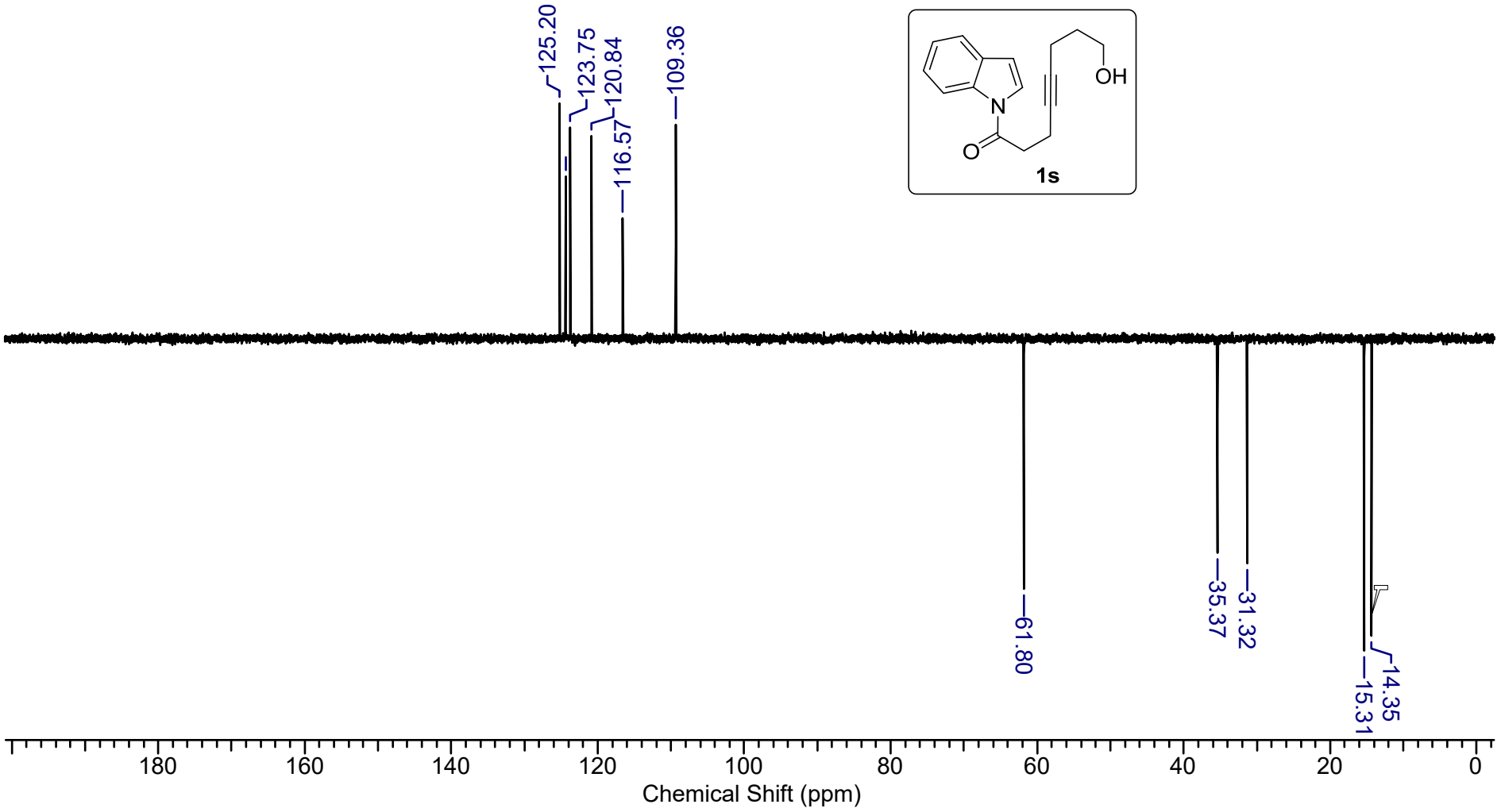

CHLOROFORM-d
77.26
77.00
76.75
125.28
124.30
123.80
120.87
116.59
109.49
135.59
130.32
Me
OH
N
O
1r
135
130
125
120
115
110
Chemical Shift (ppm)
169.71
135.59
130.32
125.28
124.30
123.80
120.87
116.59
109.49
80.84
66.44
35.31
29.35
22.24
14.41
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

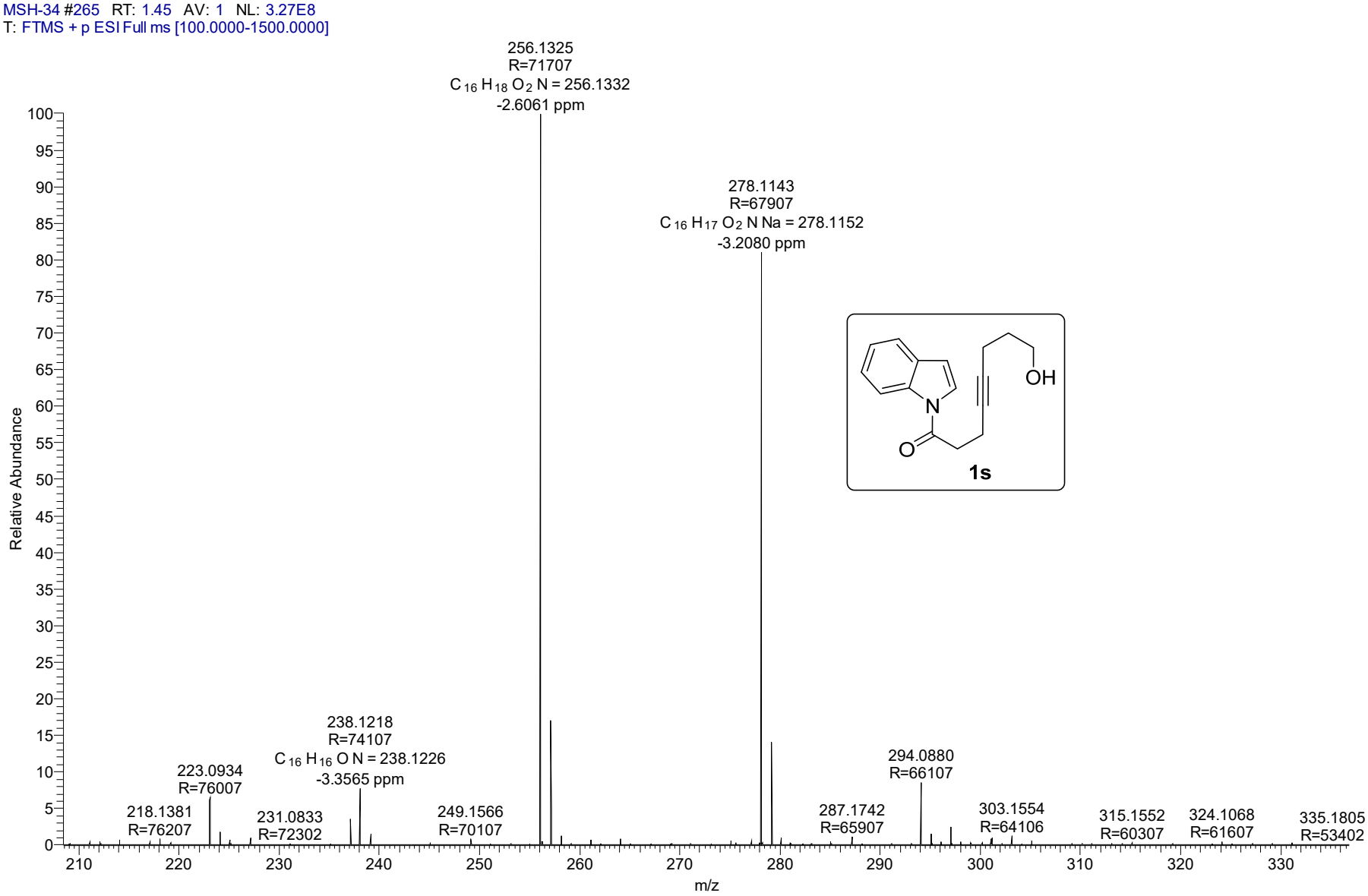

Me
OH
N
O
1r
125.28
123.80
120.87
116.59
109.50
66.45
22.24
35.30
29.35
14.41
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

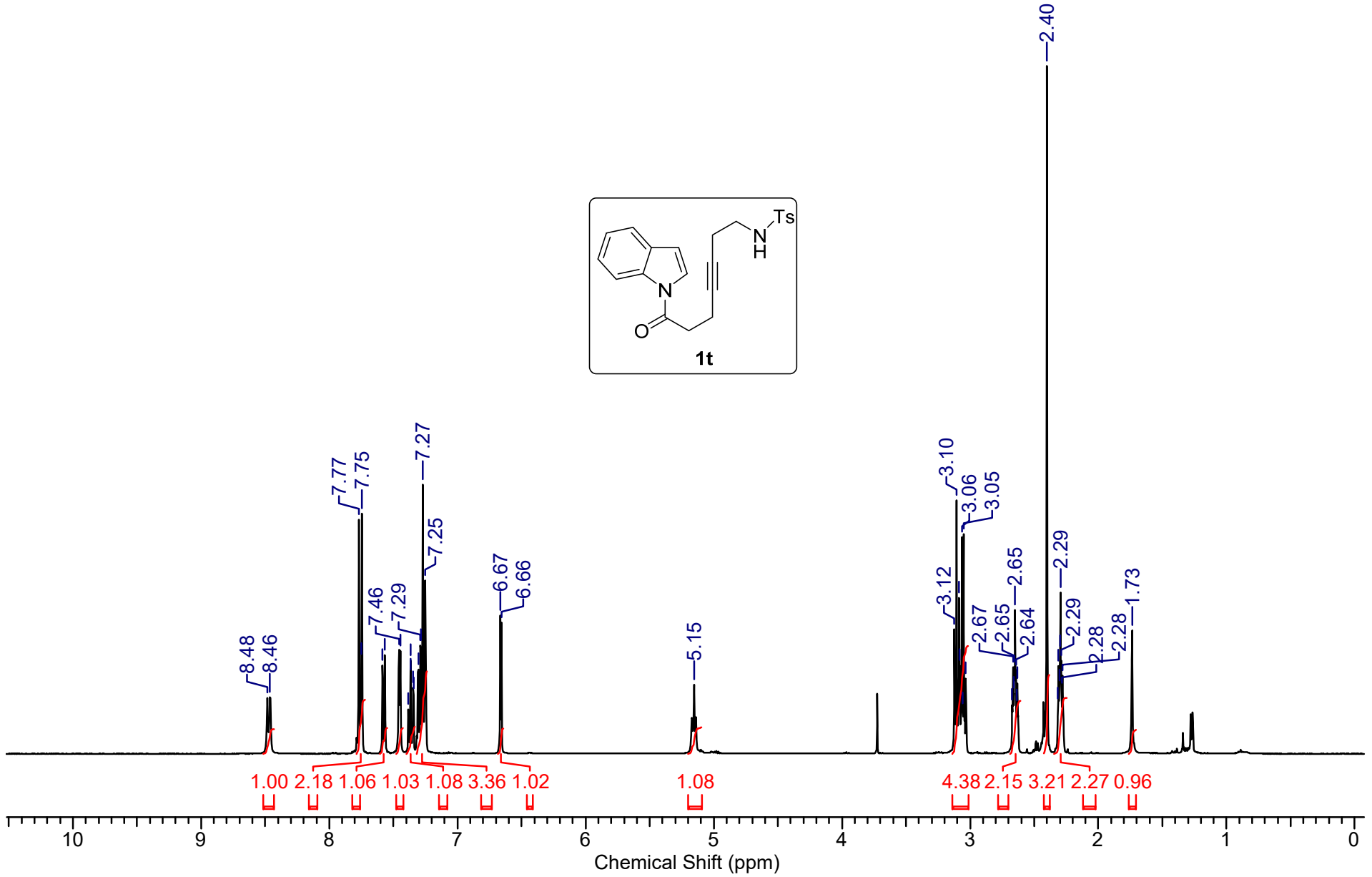

MSH-35 #272 RT: 1.49 AV: 1 NL: 1.40E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]
256.1325
R=70507
C 16 H 18 O 2 N = 256.1332
-2.6061 ppm
278.1141
R=68207
C 16 H 17 O 2 N Na = 278.1152
-3.7567 ppm
Me
OH
N
O
1r
245.0775
R=56200
249.1565
R=66307
253.1645
R=69600
262.1397
R=64007
267.1167
R=65207
271.1140
R=74800
275.1063
R=67707
283.0909
R=63106
287.1740
R=65507
Relative Abundance
m/z

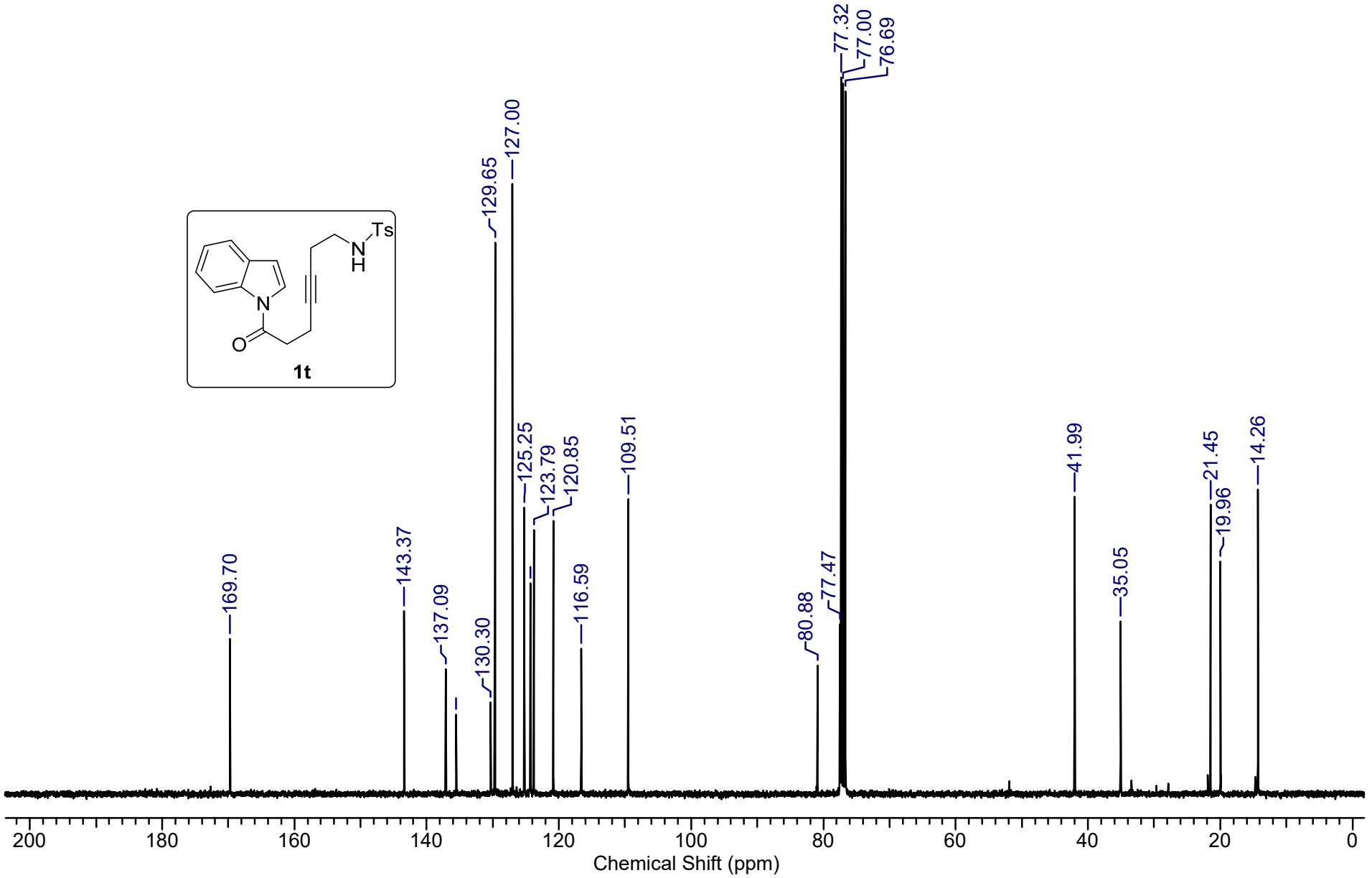

OH
N
O
1s
8.50
8.48
7.60
7.58
7.50
7.49
7.28
6.68
6.67
3.75
3.74
3.72
3.17
3.15
3.13
2.74
2.73
2.72
2.31
2.30
2.29
2.28
2.75
2.71
2.32
2.27
2.29
1.77
1.75
1.74
1.72
1.65
1.00 1.02 1.04 1.07 1.27 1.02
2.24 2.14 2.13 2.17 2.24 1.22
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

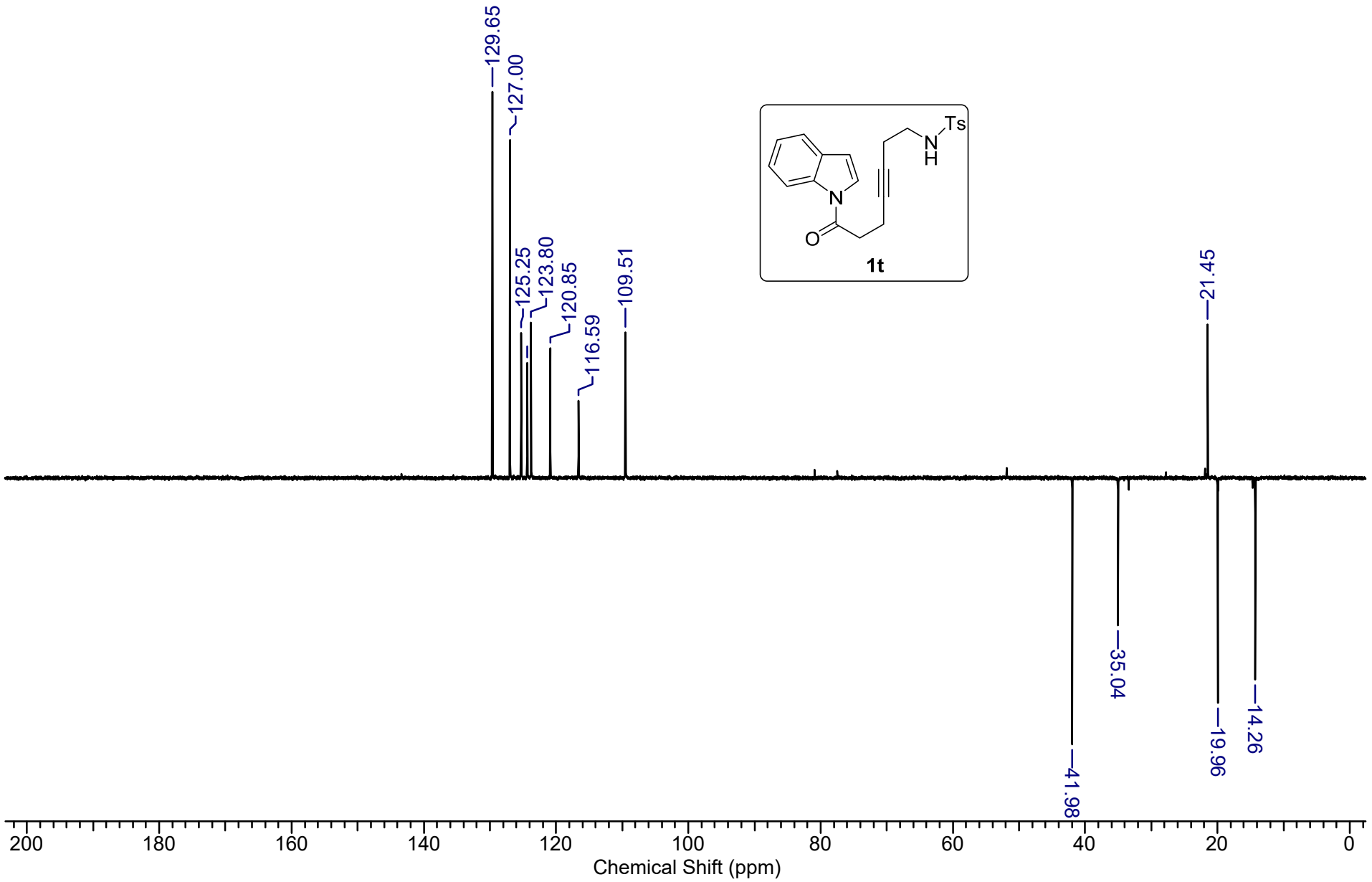

CHLOROFORM-d
77.32
77.00
76.68
OH
N
O
1s
169.78
135.58
130.32
125.21
123.76
120.85
116.57
109.36
80.74
61.80
35.37
31.33
15.31
14.35
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


125.20
123.75
120.84
116.57
109.36
61.80
35.37
31.32
15.31
14.35
OH
N
O
1s
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

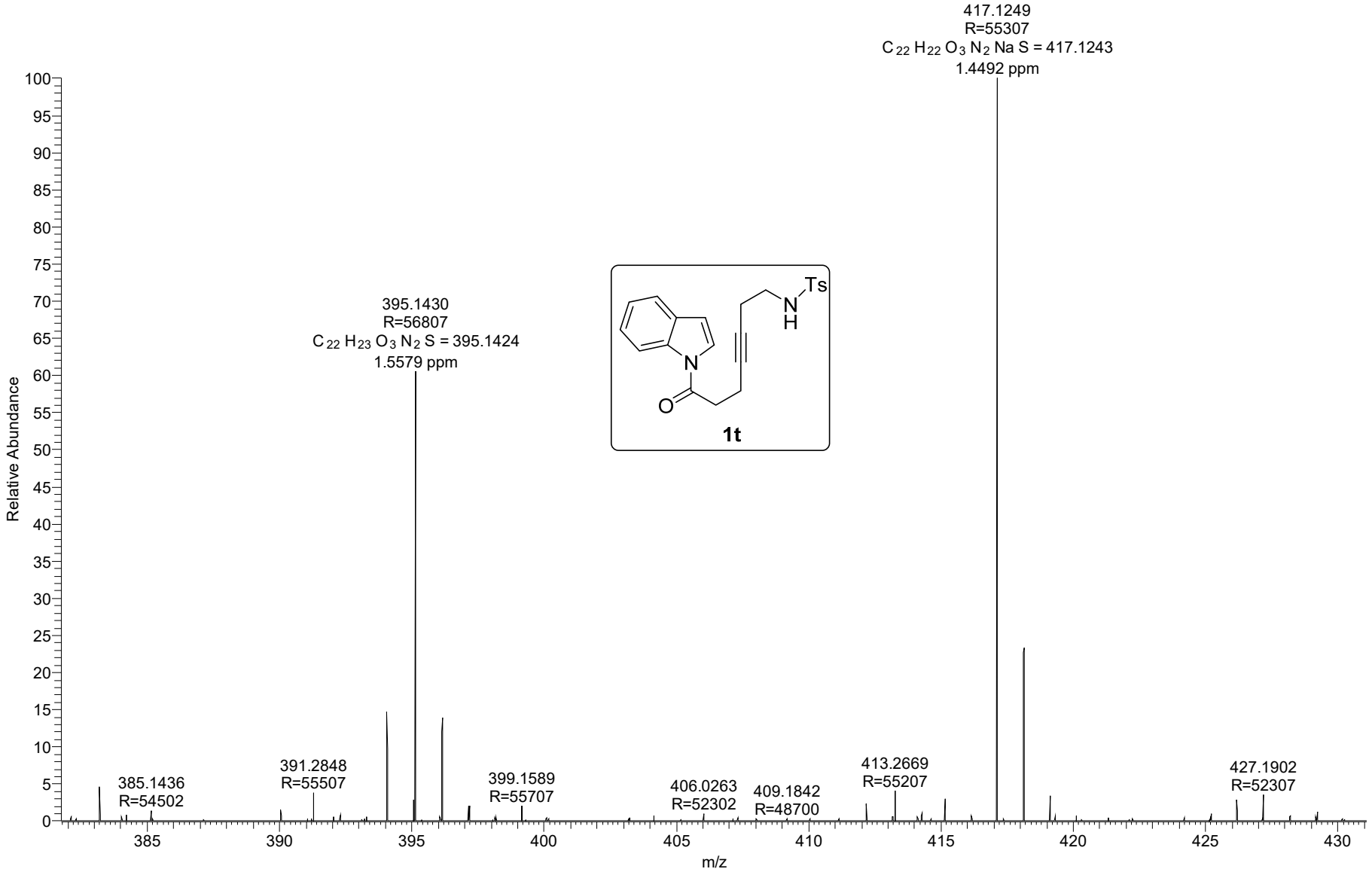

MSH-34 #265 RT: 1.45 AV: 1 NL: 3.27E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
256.1325
R=71707
C 16 H 18 O 2 N = 256.1332
-2.6061 ppm
278.1143
R=67907
C 16 H 17 O 2 N Na = 278.1152
-3.2080 ppm
238.1218
R=74107
C 16 H 16 O N = 238.1226
-3.3565 ppm
223.0934
R=76007
218.1381
R=76207
231.0833
R=72302
249.1566
R=70107
287.1742
R=65907
294.0880
R=66107
303.1554
R=64106
315.1552
R=60307
324.1068
R=61607
335.1805
R=53402
OH
N
O
1s
Relative Abundance
m/z

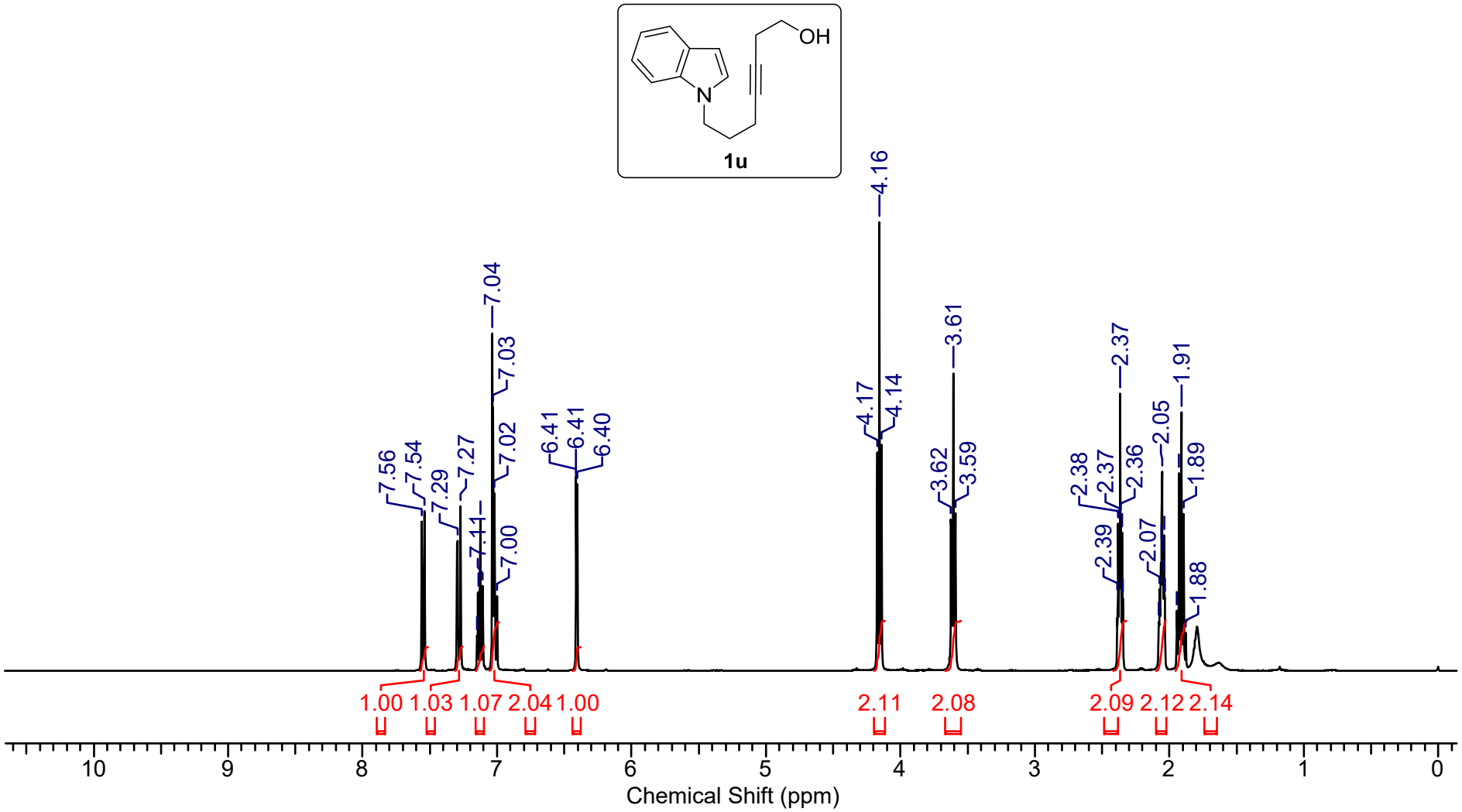

2.40
Ts
N
H
N
O
1t
8.48
8.46
7.77
7.75
7.46
7.29
7.27
7.25
6.67
6.66
5.15
3.10
3.06
3.05
3.12
2.67
2.65
2.64
2.65
2.29
2.29
2.28
2.28
1.73
1.00 2.18 1.06 1.03 1.08 3.36 1.02
1.08
4.38 2.15 3.21 2.27 0.96
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

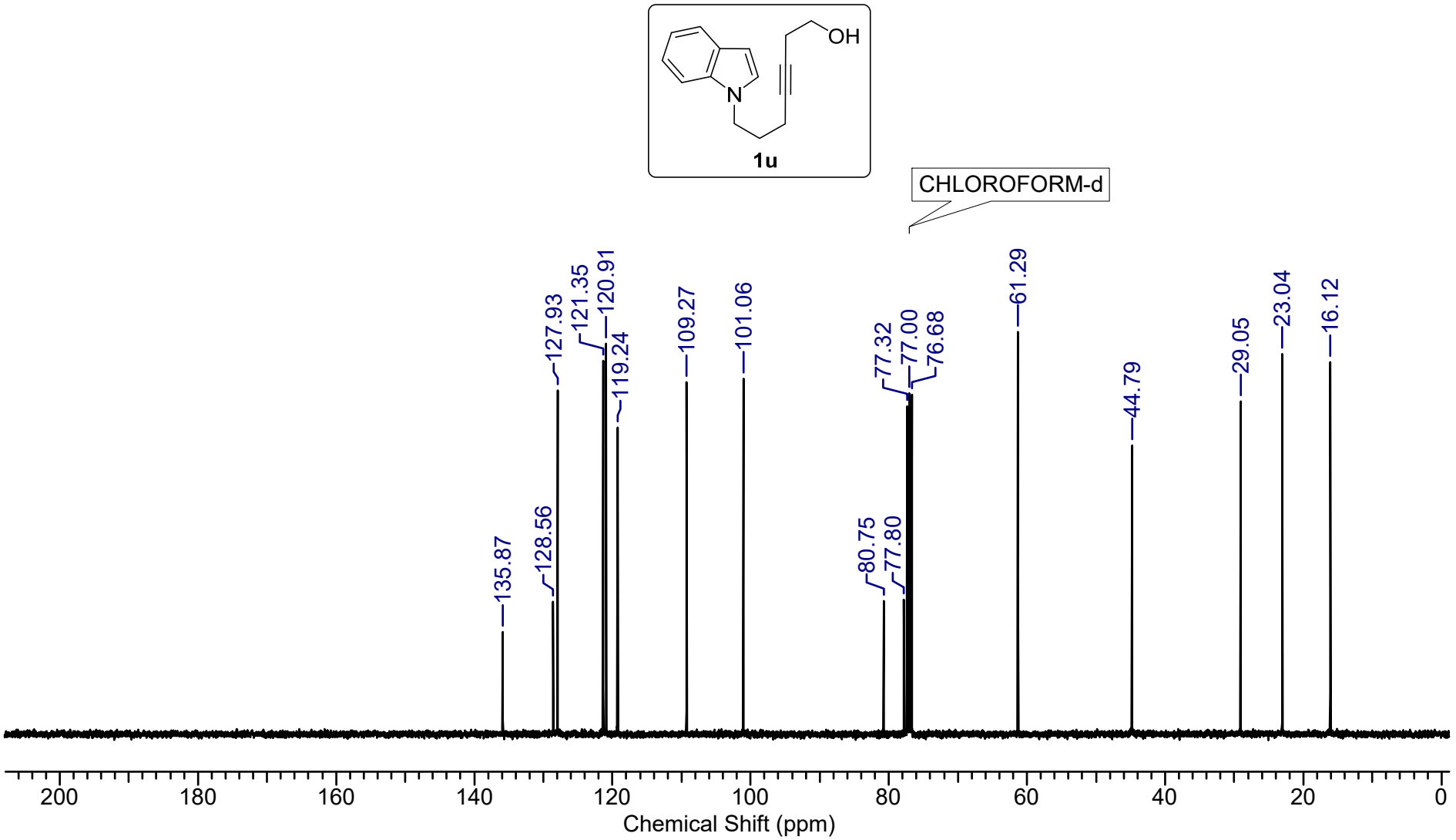

Ts
N
H
N
O
1t
169.70
143.37
137.09
130.30
129.65
127.00
125.25
123.79
120.85
116.59
109.51
80.88
77.47
77.32
77.00
76.69
41.99
35.05
21.45
19.96
14.26
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

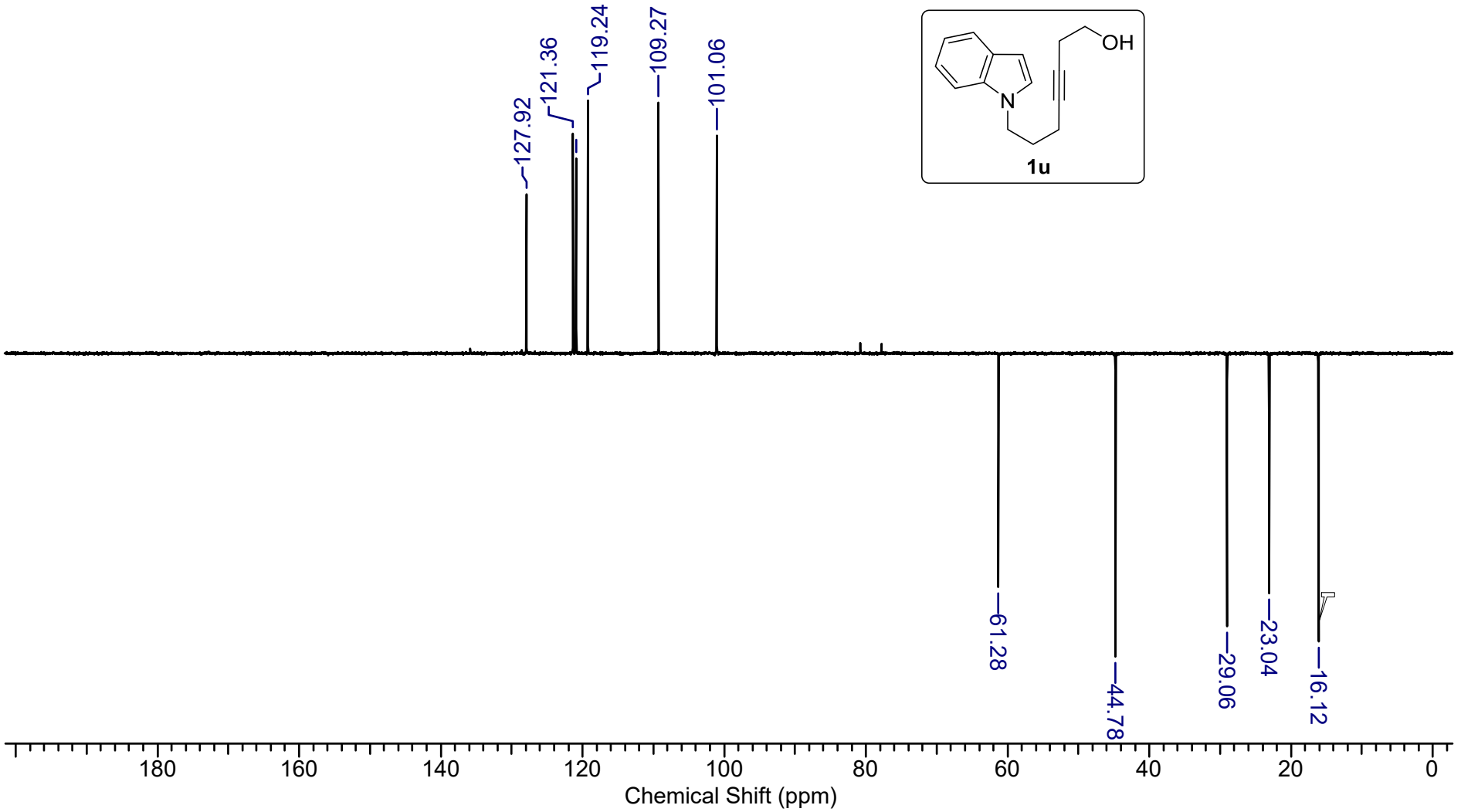

129.65
127.00
125.25
123.80
120.85
116.59
109.51
21.45
41.98
35.04
19.96
14.26
Ts
N
H
N
O
1t
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

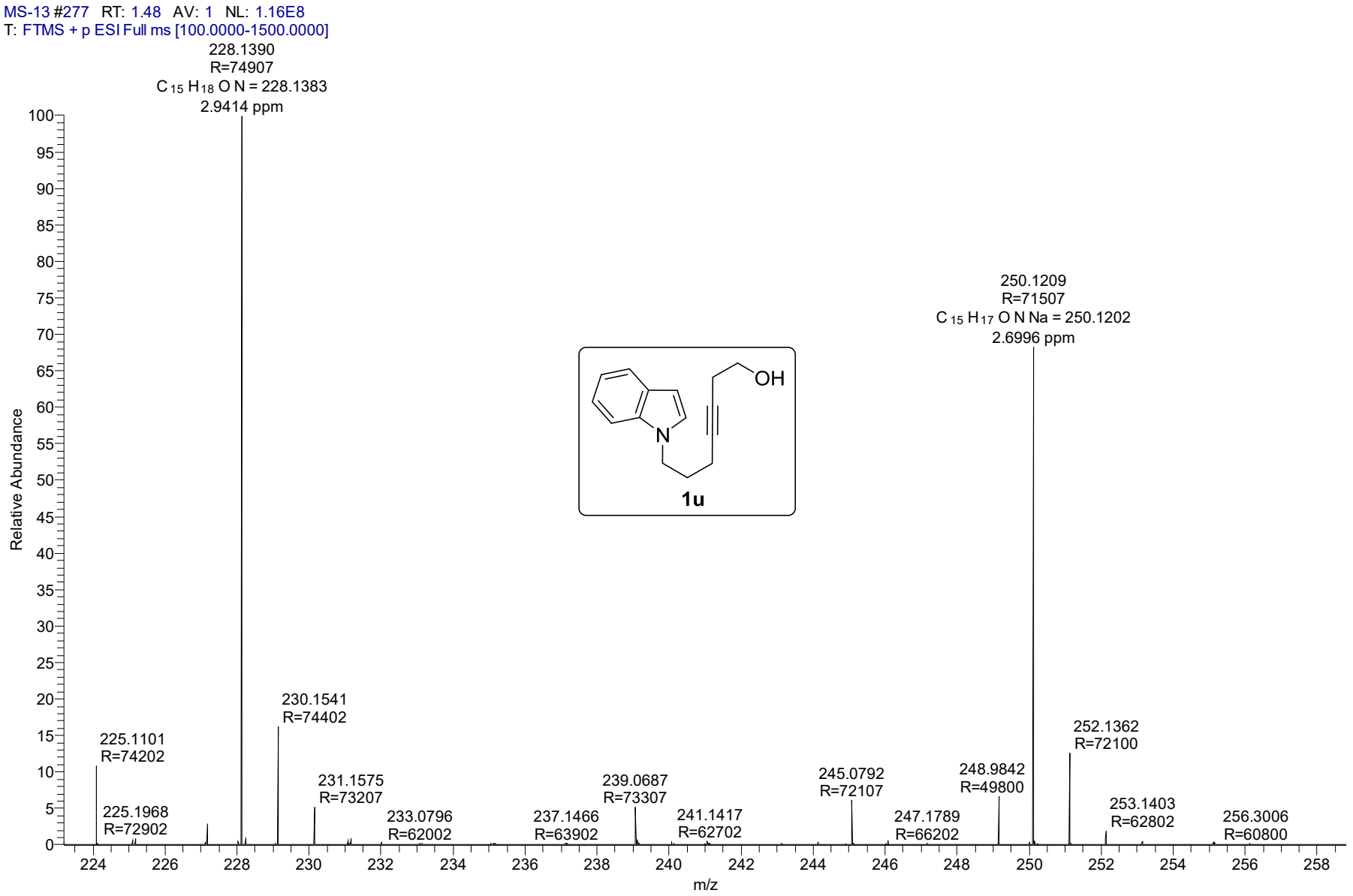
MS-12 #266 RT: 1.42 AV: 1 NL: 5.96E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

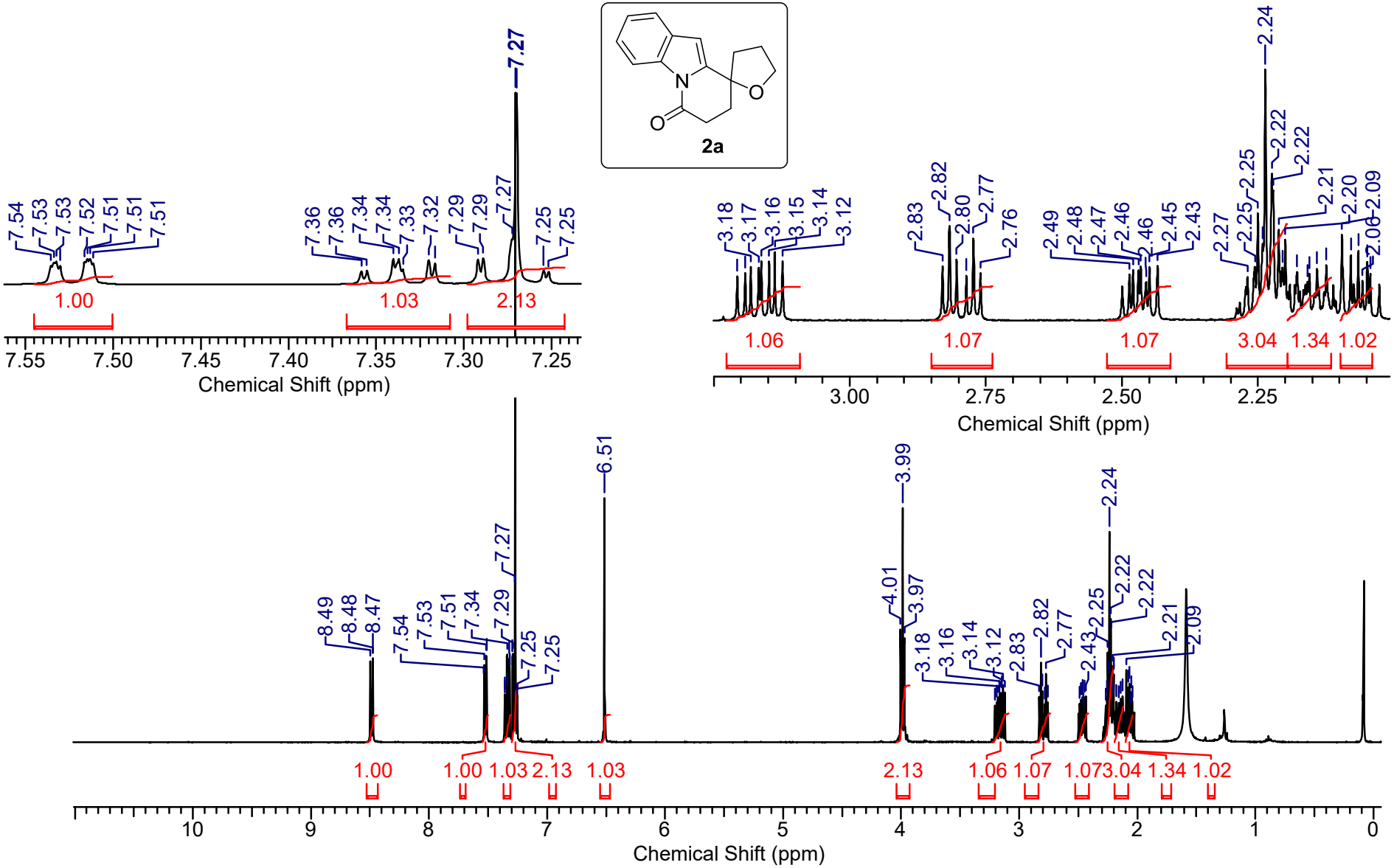


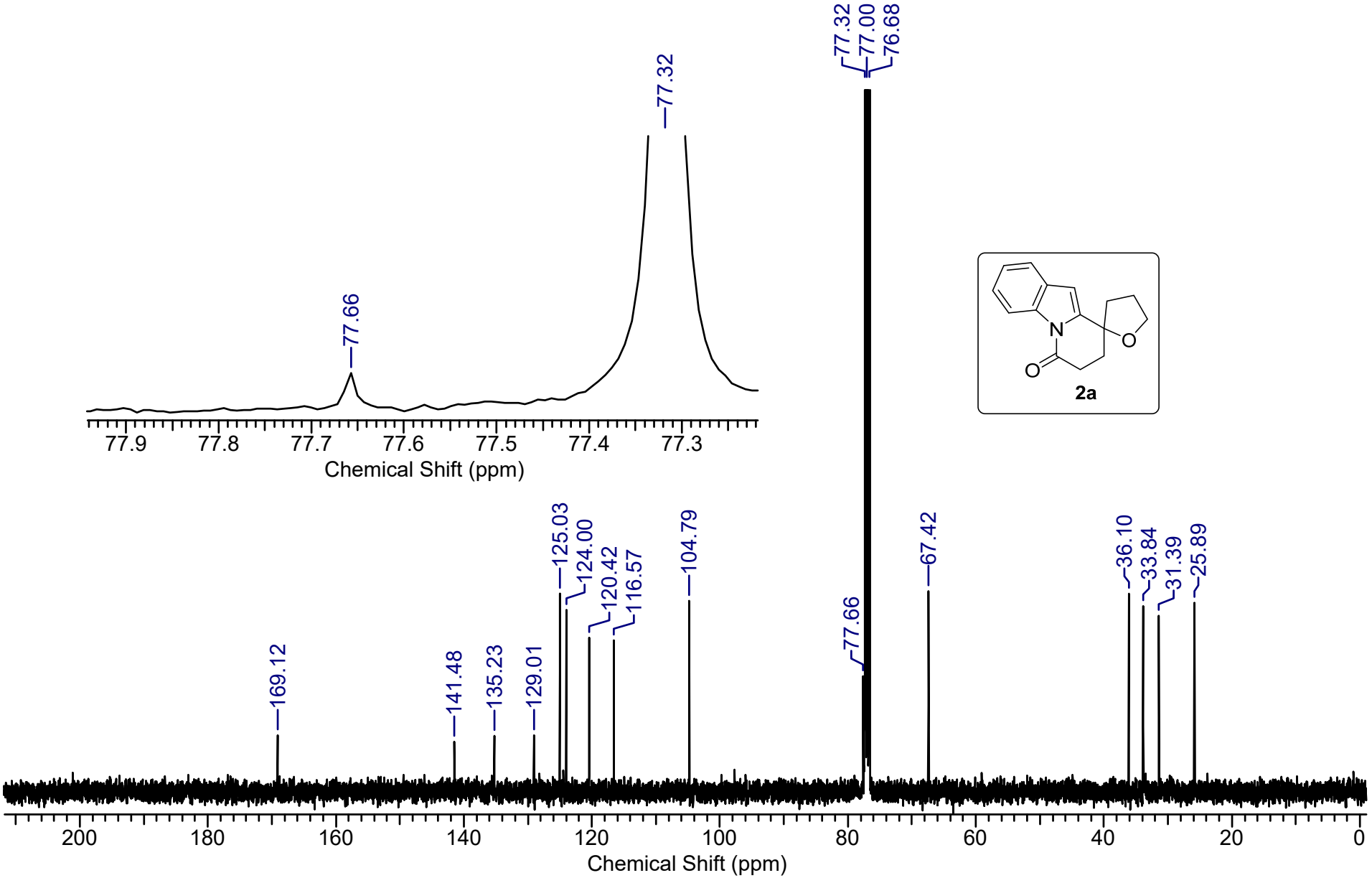

OH
N
1u
7.56
7.54
7.29
7.27
7.11
7.00
7.04
7.03
7.02
6.41
6.41
6.40
4.16
4.17
4.14
3.61
3.62
3.59
2.38
2.37
2.36
2.39
2.37
2.07
2.05
1.91
1.89
1.88
1.00 1.03 1.07 2.04 1.00
2.11
2.08
2.09 2.12 2.14
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

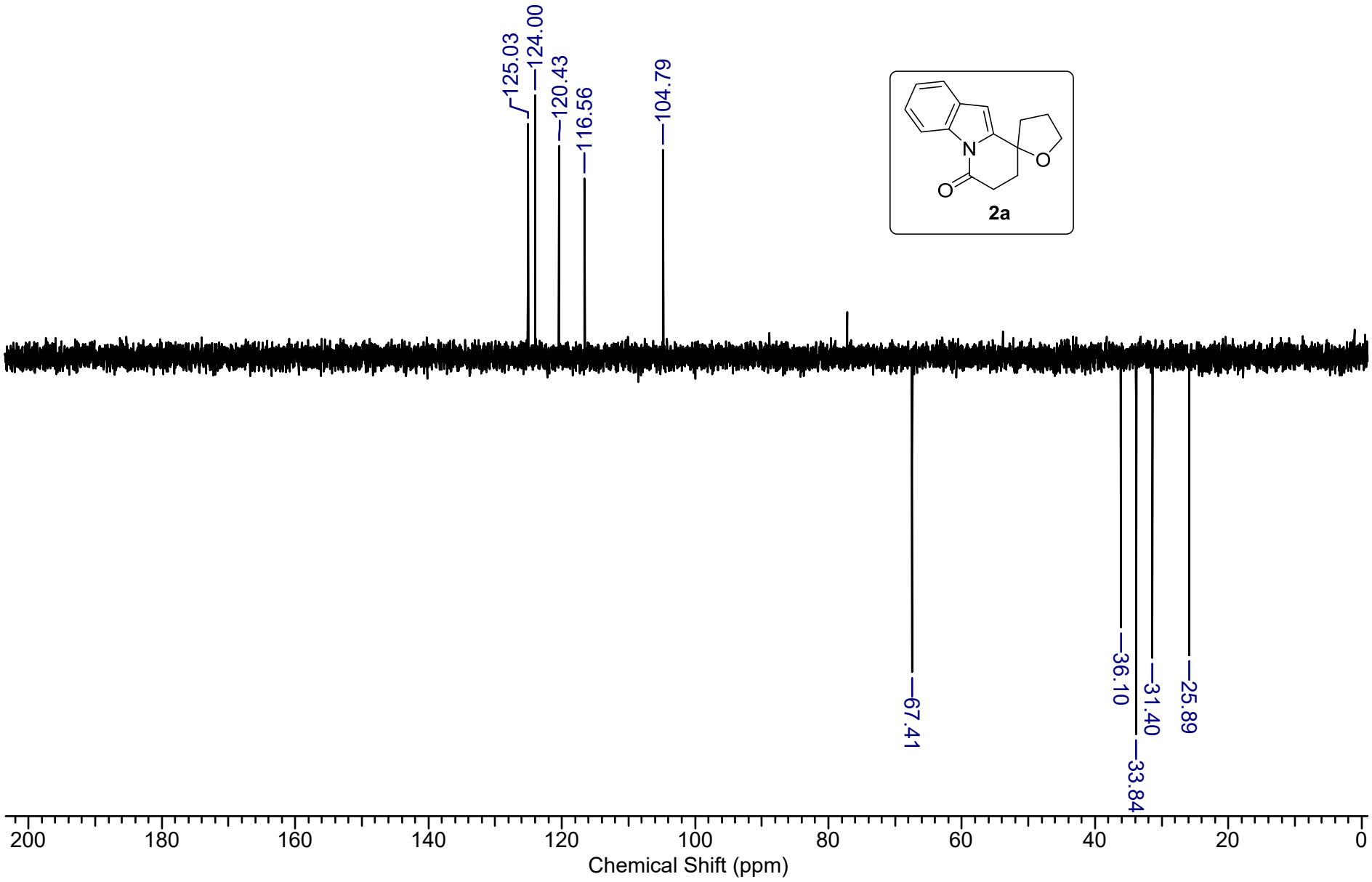

OH
N
1u
CHLOROFORM-d
135.87
128.56
127.93
121.35
120.91
119.24
109.27
101.06
80.75
77.80
77.32
77.00
76.68
61.29
44.79
29.05
23.04
16.12
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


127.92
121.36
119.24
109.27
101.06
61.28
44.78
29.06
23.04
16.12
OH
N
1u
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

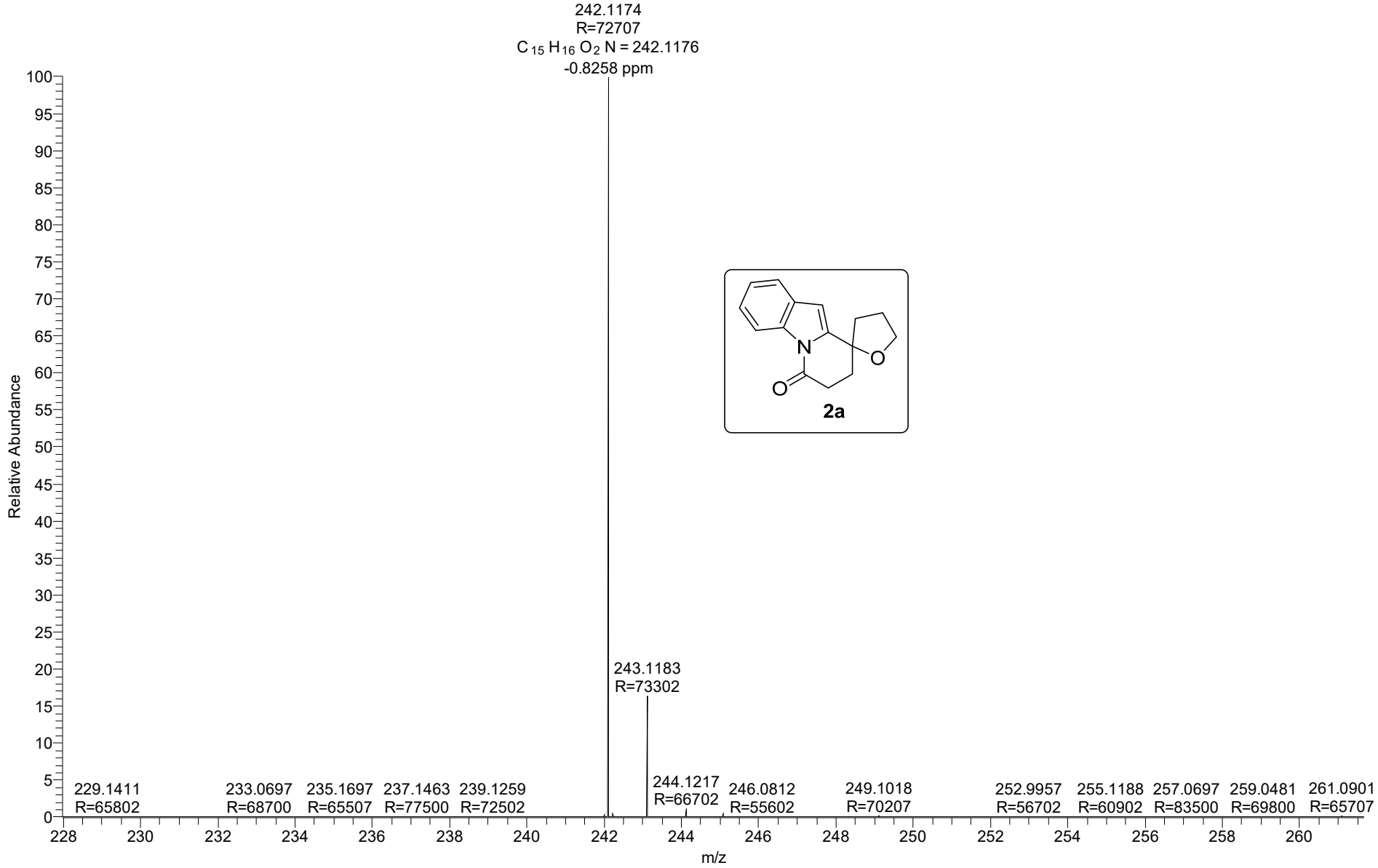

MS-13 #277 RT: 1.48 AV: 1 NL: 1.16E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
228.1390
R=74907
C 15 H 18 O N = 228.1383
2.9414 ppm
250.1209
R=71507
C 15 H 17 O N Na = 250.1202
2.6996 ppm
OH
N
1u
Relative Abundance
m/z
225.1101
R=74202
225.1968
R=72902
230.1541
R=74402
231.1575
R=73207
233.0796
R=62002
237.1466
R=63902
239.0687
R=73307
241.1417
R=62702
245.0792
R=72107
247.1789
R=66202
248.9842
R=49800
252.1362
R=72100
253.1403
R=62802
256.3006
R=60800

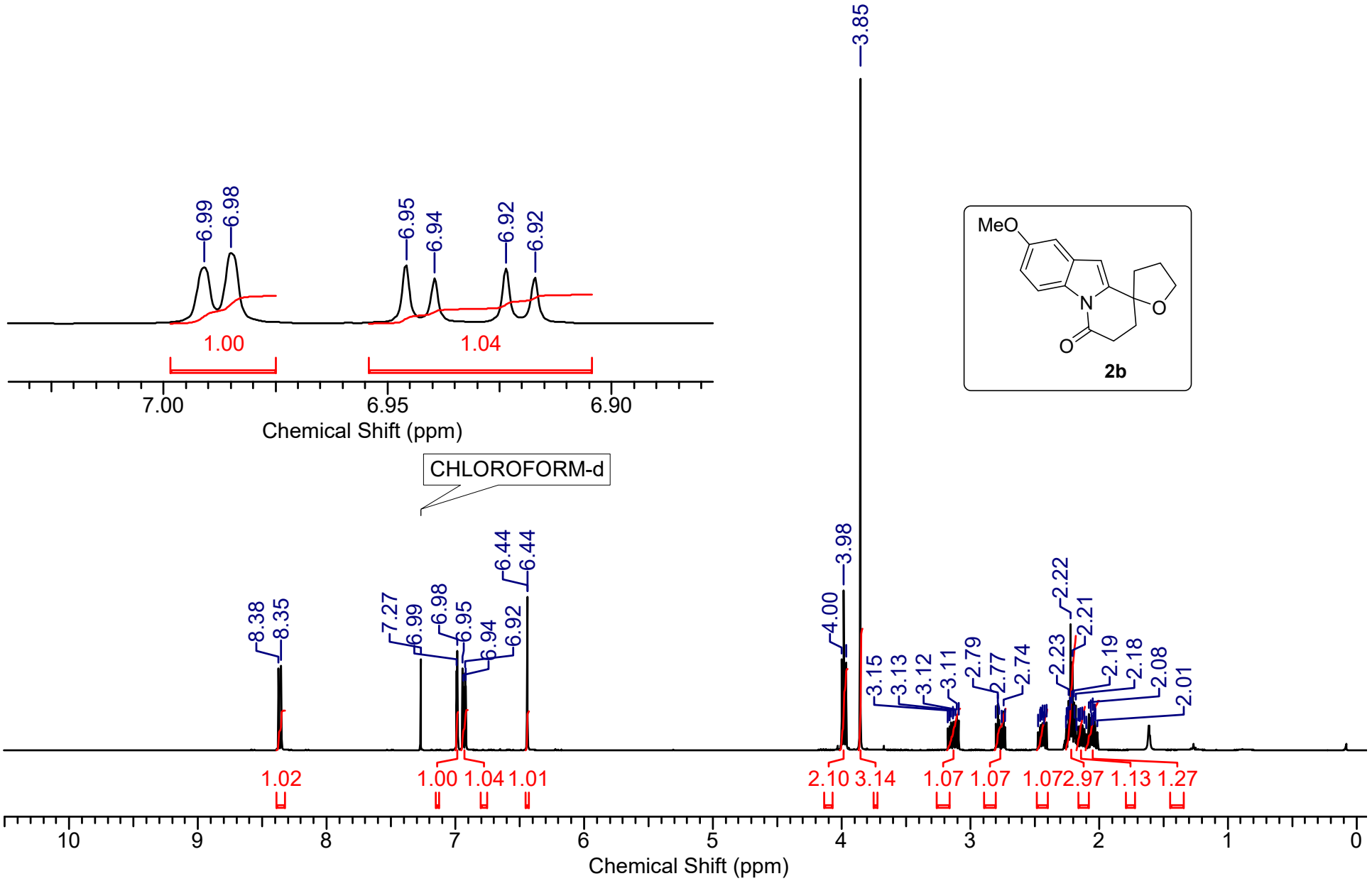

2a
7.54
7.53
7.53
7.52
7.51
7.51
7.51
7.36
7.36
7.34
7.34
7.33
7.32
7.29
7.29
7.27
7.25
7.25
1.00
1.03
2.13
7.55
7.50
7.45
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
3.18
3.17
3.16
3.15
3.14
3.12
2.83
2.82
2.80
2.77
2.76
2.49
2.48
2.47
2.46
2.46
2.45
2.43
2.27
2.25
2.25
2.24
2.22
2.22
2.21
2.20
2.09
2.06
1.06
1.07
1.07
3.04
1.34
1.02
3.00
2.75
2.50
2.25
Chemical Shift (ppm)
8.49
8.48
8.47
7.54
7.53
7.51
7.34
7.29
7.27
7.25
7.25
6.51
4.01
3.99
3.97
3.18
3.16
3.14
3.12
2.83
2.82
2.77
2.43
2.25
2.24
2.22
2.22
2.21
2.09
1.00
1.00
1.03
2.13
1.03
2.13
1.06
1.07
1.07
3.04
1.34
1.02
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

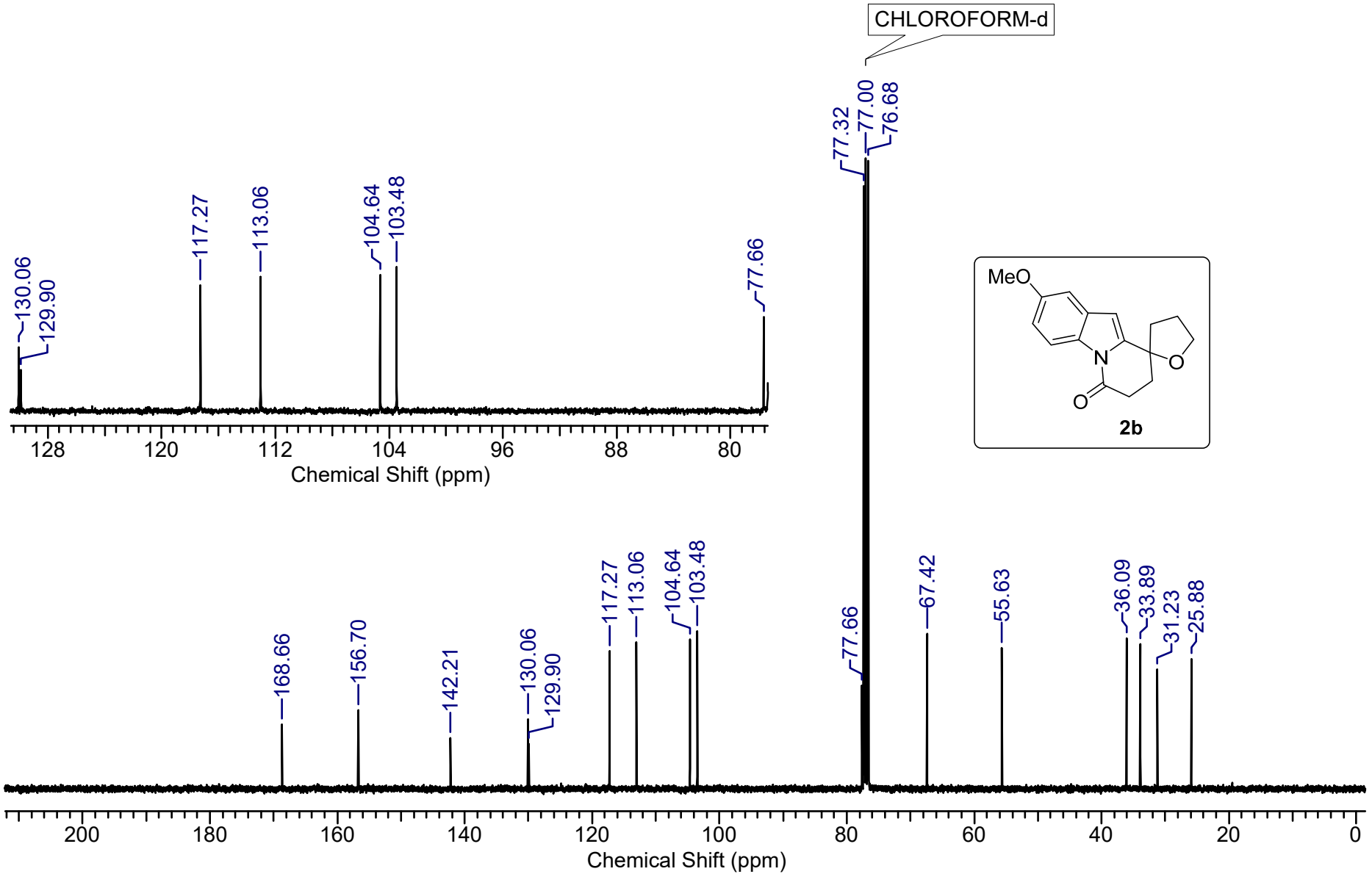

77.32
77.00
76.68
77.66
77.9
77.8
77.7
77.6
77.5
77.4
77.3
Chemical Shift (ppm)
N
O
O
2a
169.12
141.48
135.23
129.01
125.03
124.00
120.42
116.57
104.79
77.66
67.42
36.10
33.84
31.39
25.89
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

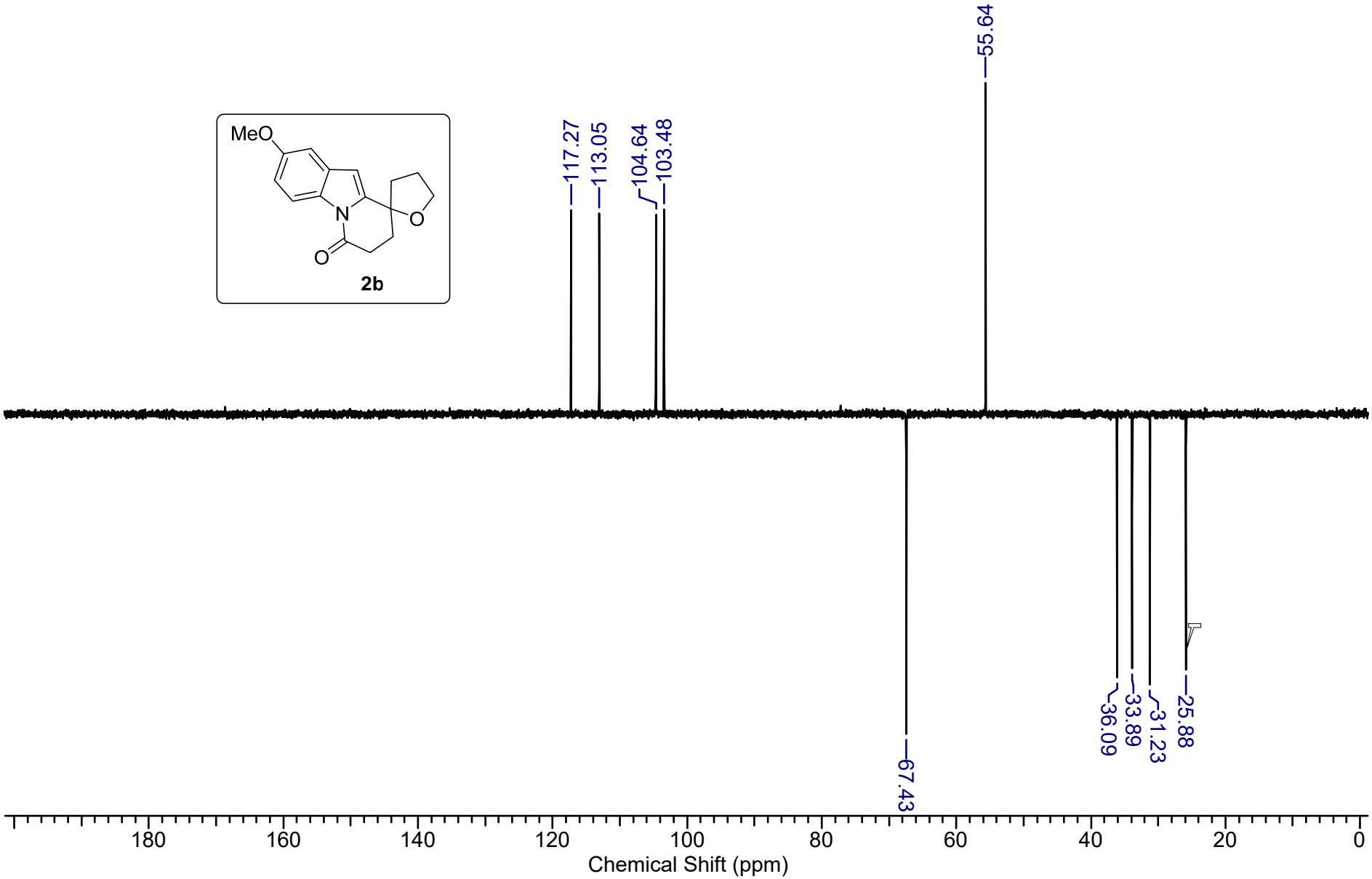

125.03
124.00
120.43
116.56
104.79
N
O
O
2a
67.41
36.10
33.84
31.40
25.89
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

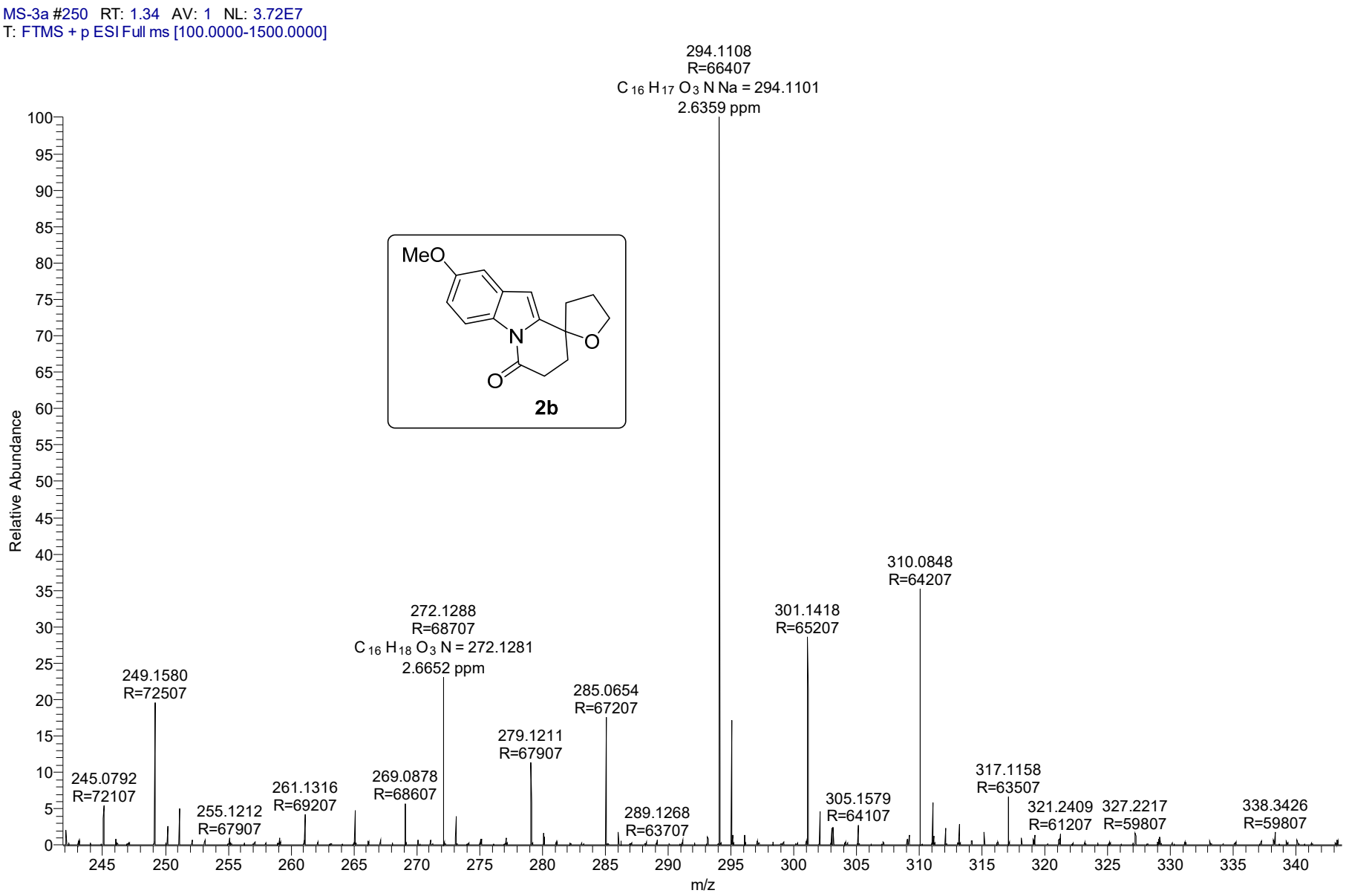
M-8 #457 RT: 2.75 AV: 1 NL: 4.62E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

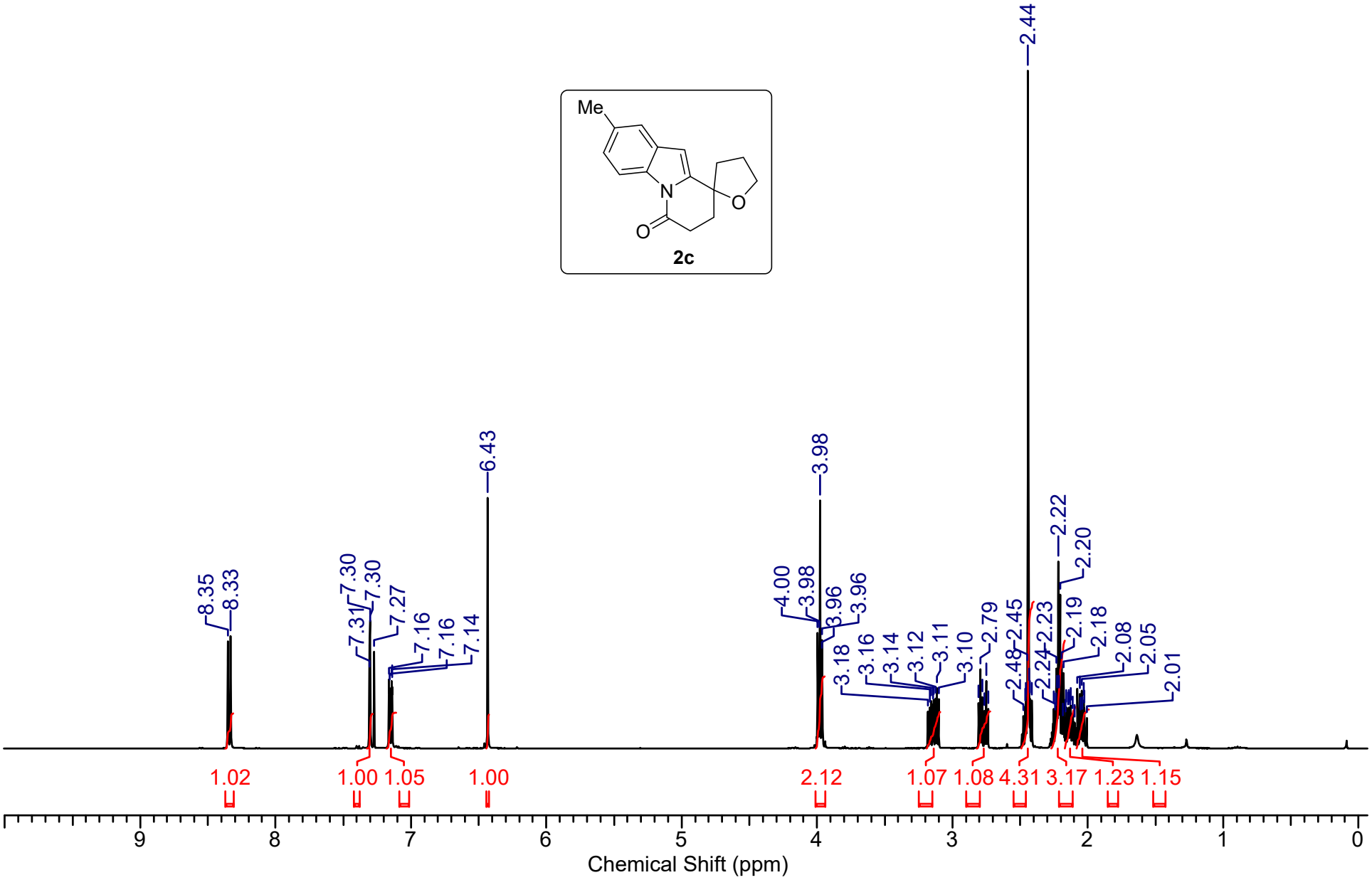


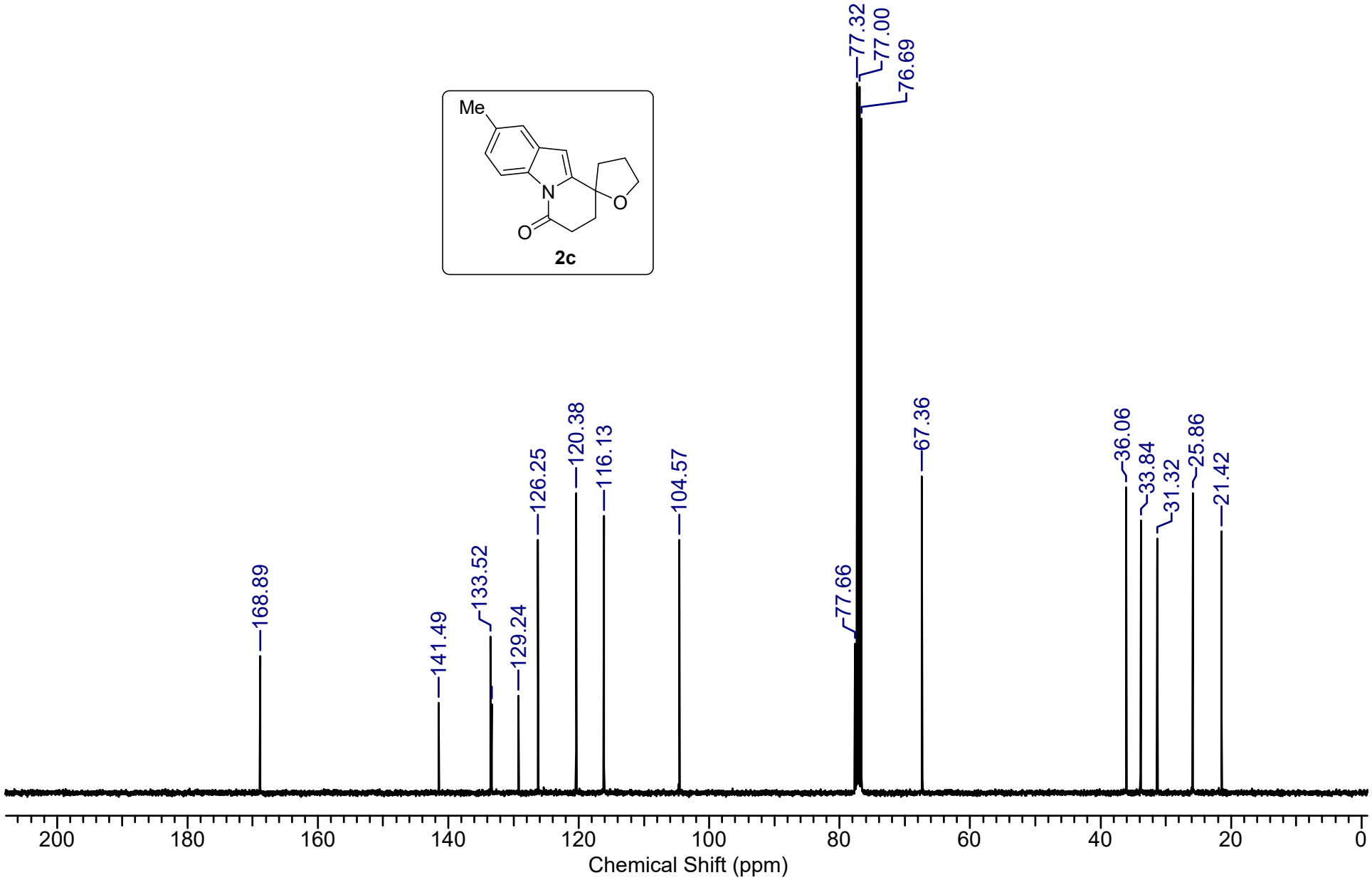

6.99
6.98
6.95
6.94
6.92
6.92
1.00
1.04
7.00
6.95
6.90
Chemical Shift (ppm)
MeO
N
O
O
2b
CHLOROFORM-d
3.85
8.38
8.35
7.27
6.99
6.98
6.95
6.94
6.92
6.44
6.44
3.98
4.00
3.15
3.13
3.12
3.11
2.79
2.77
2.74
2.23
2.22
2.21
2.19
2.18
2.08
2.01
1.02
1.00
1.04
1.01
2.10
3.14
1.07
1.07
1.07
2.97
1.13
1.27
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

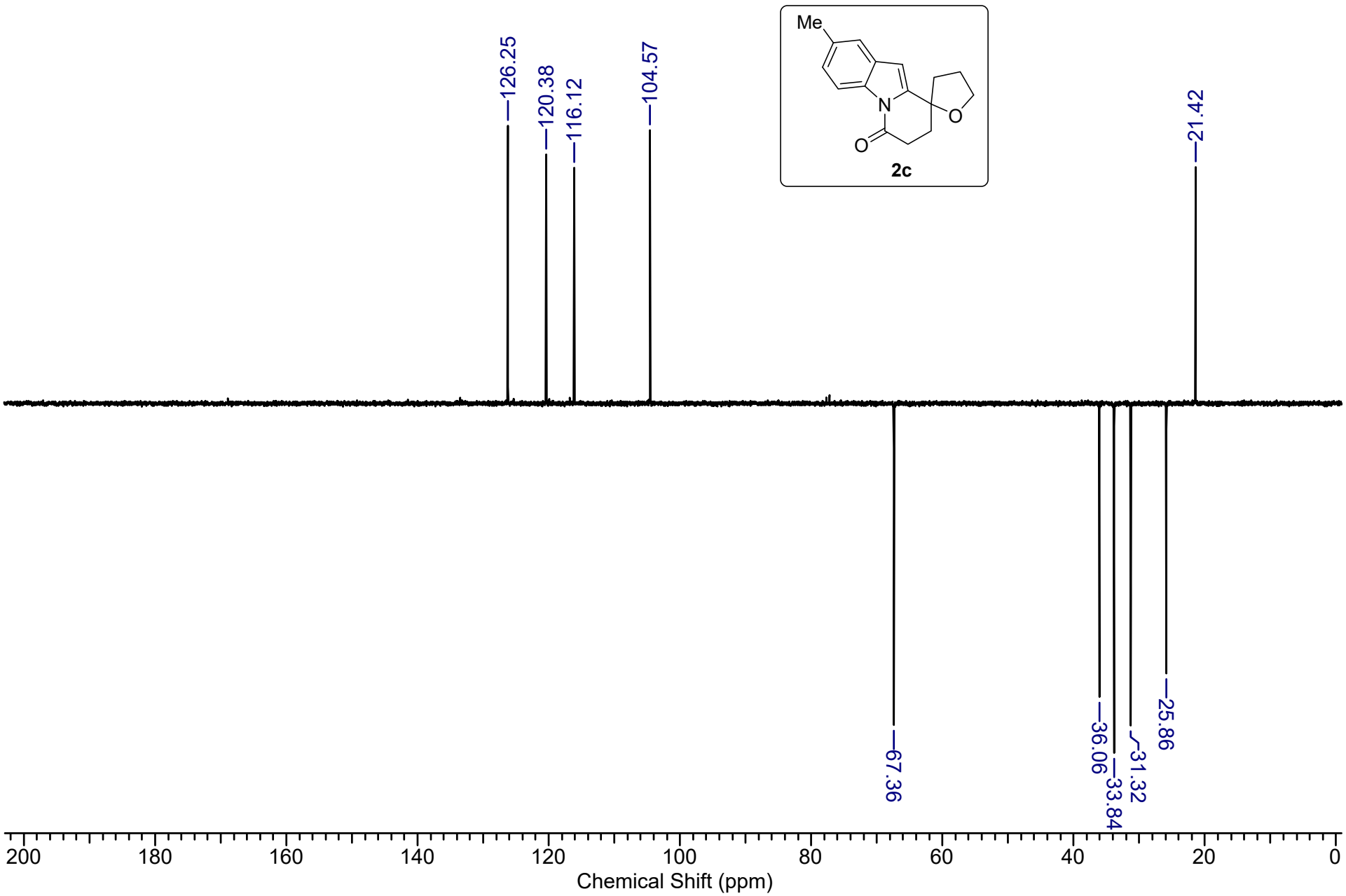

CHLOROFORM-d
77.32
77.00
76.68
130.06
129.90
117.27
113.06
104.64
103.48
77.66
MeO
N
O
O
2b
168.66
156.70
142.21
130.06
129.90
117.27
113.06
104.64
103.48
77.66
67.42
55.63
36.09
33.89
31.23
25.88
128
120
112
104
96
88
80
Chemical Shift (ppm)
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


MeO
N
O
O
2b
117.27
113.05
104.64
103.48
55.94
67.43
36.09
33.89
31.23
25.88
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

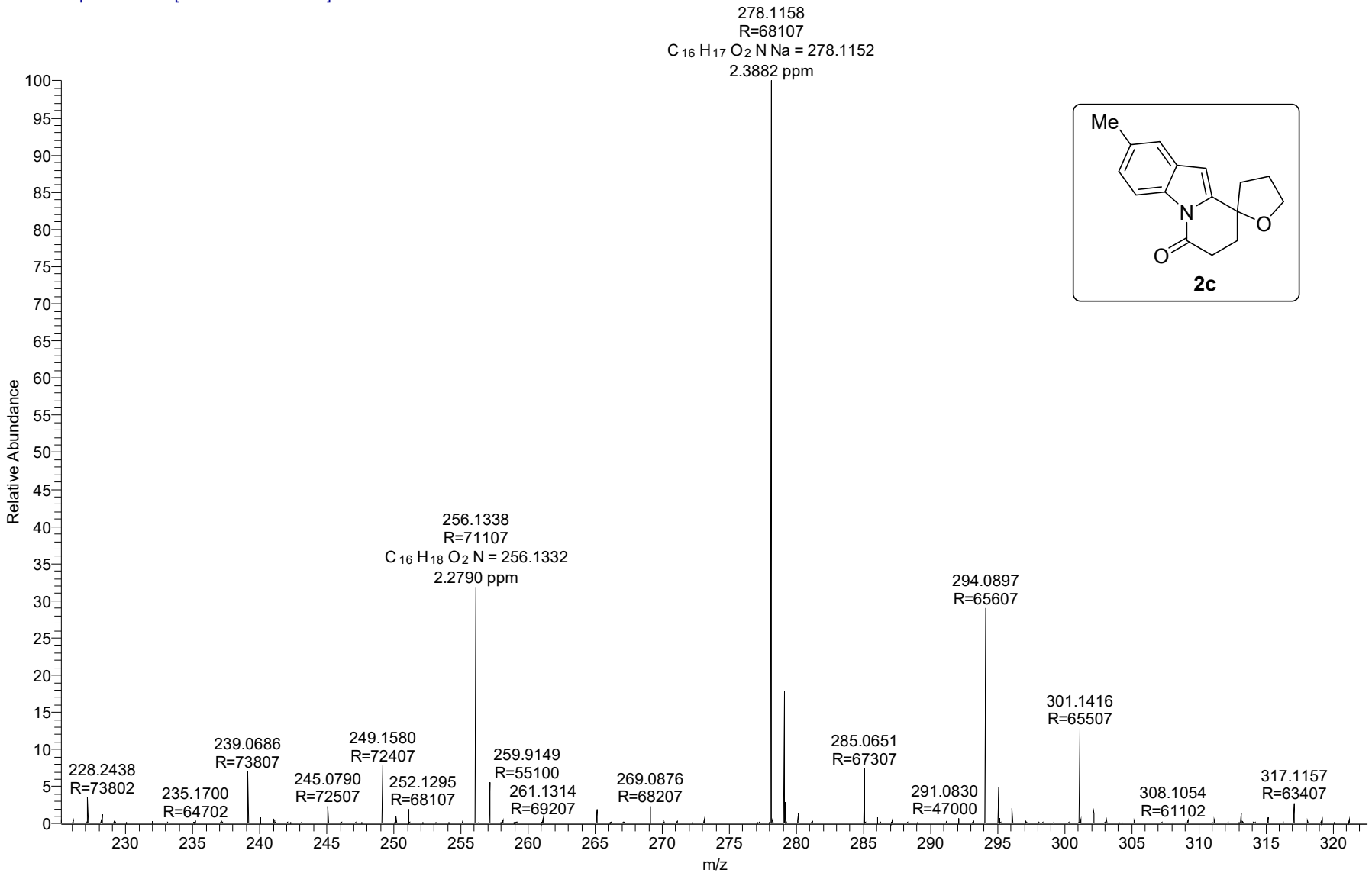

MS-3a #250 RT: 1.34 AV: 1 NL: 3.72E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
294.1108
R=66407
C 16 H 17 O 3 N Na = 294.1101
2.6359 ppm
MeO
N
O
O
2b
Relative Abundance
m/z
272.1288
R=68707
C 16 H 18 O 3 N = 272.1281
2.6652 ppm
310.0848
R=64207
301.1418
R=65207
249.1580
R=72507
285.0654
R=67207
279.1211
R=67907
245.0792
R=72107
269.0878
R=68607
261.1316
R=69207
255.1212
R=67907
289.1268
R=63707
305.1579
R=64107
317.1158
R=63507
321.2409
R=61207
327.2217
R=59807
338.3426
R=59807

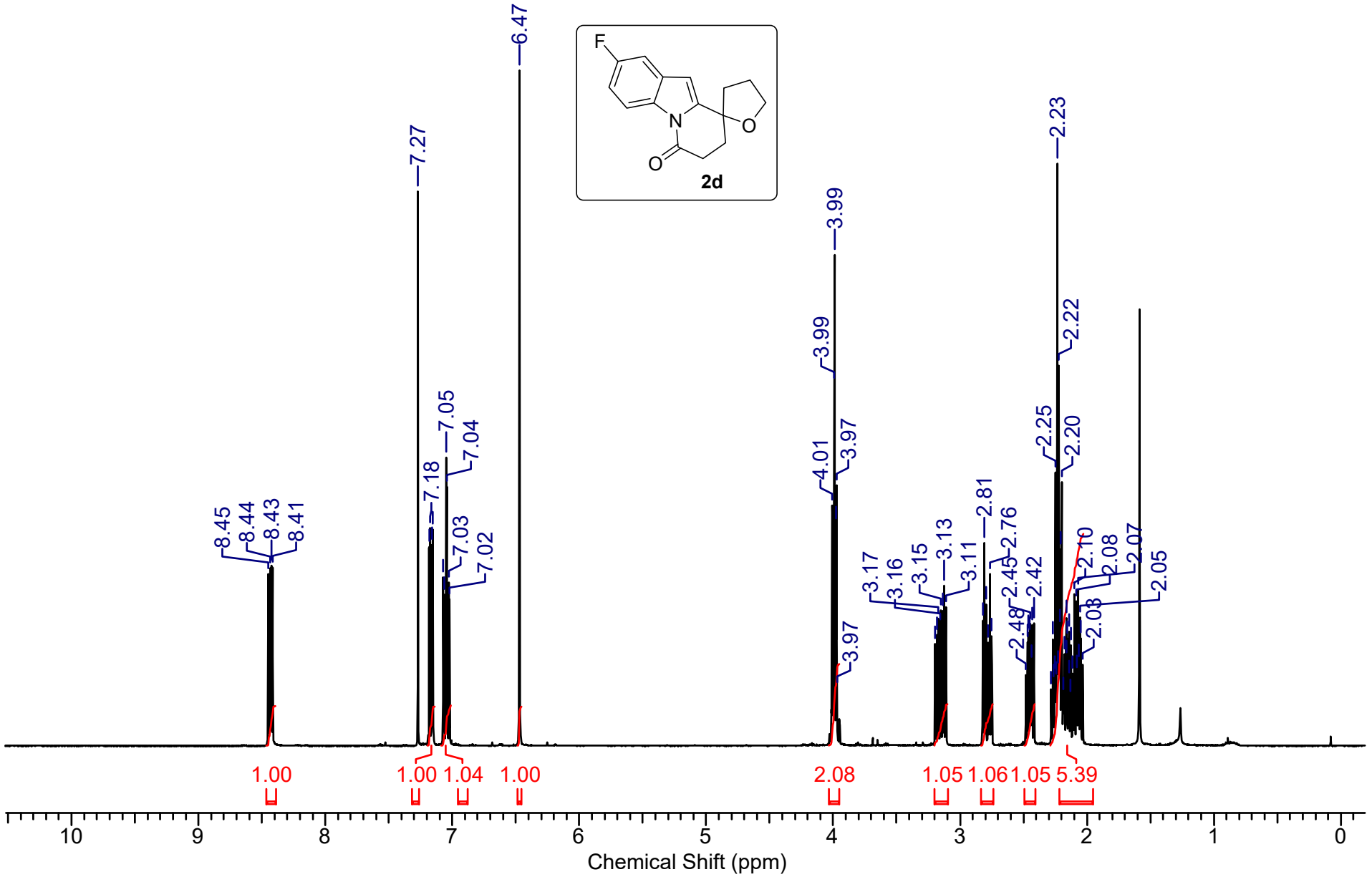

Me
N
O
O
2c
8.35
8.33
7.30
7.30
7.31
7.27
7.16
7.16
7.14
6.43
4.00
3.98
3.98
3.96
3.96
3.18
3.16
3.14
3.12
3.11
3.10
2.79
2.48
2.45
2.44
2.24
2.23
2.22
2.20
2.19
2.18
2.08
2.05
2.01
1.02
1.00
1.05
1.00
2.12
1.07
1.08
4.31
3.17
1.23
1.15
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

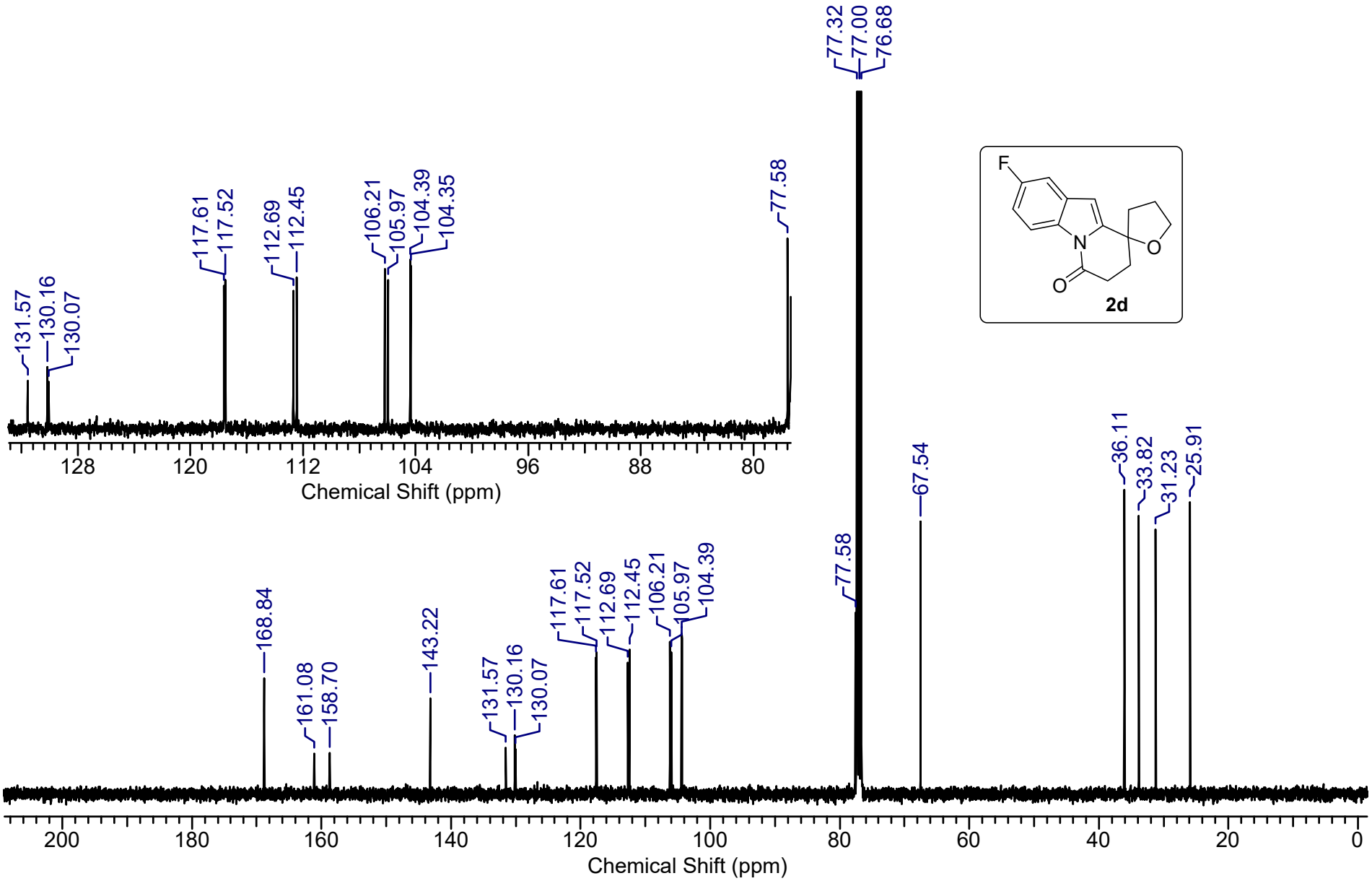

Me
N
O
O
2c
77.32
77.00
76.69
168.89
141.49
133.52
129.24
126.25
120.38
116.13
104.57
77.66
67.36
36.06
33.84
31.32
25.86
21.42
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

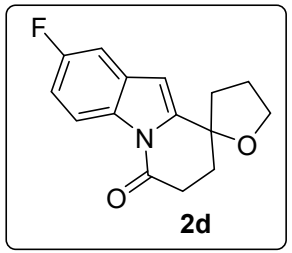

Me
N
O
O
2c
126.25
120.38
116.12
104.57
21.42
67.36
36.06
33.84
31.32
25.86
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-2a #302 RT: 1.61 AV: 1 NL: 9.27E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

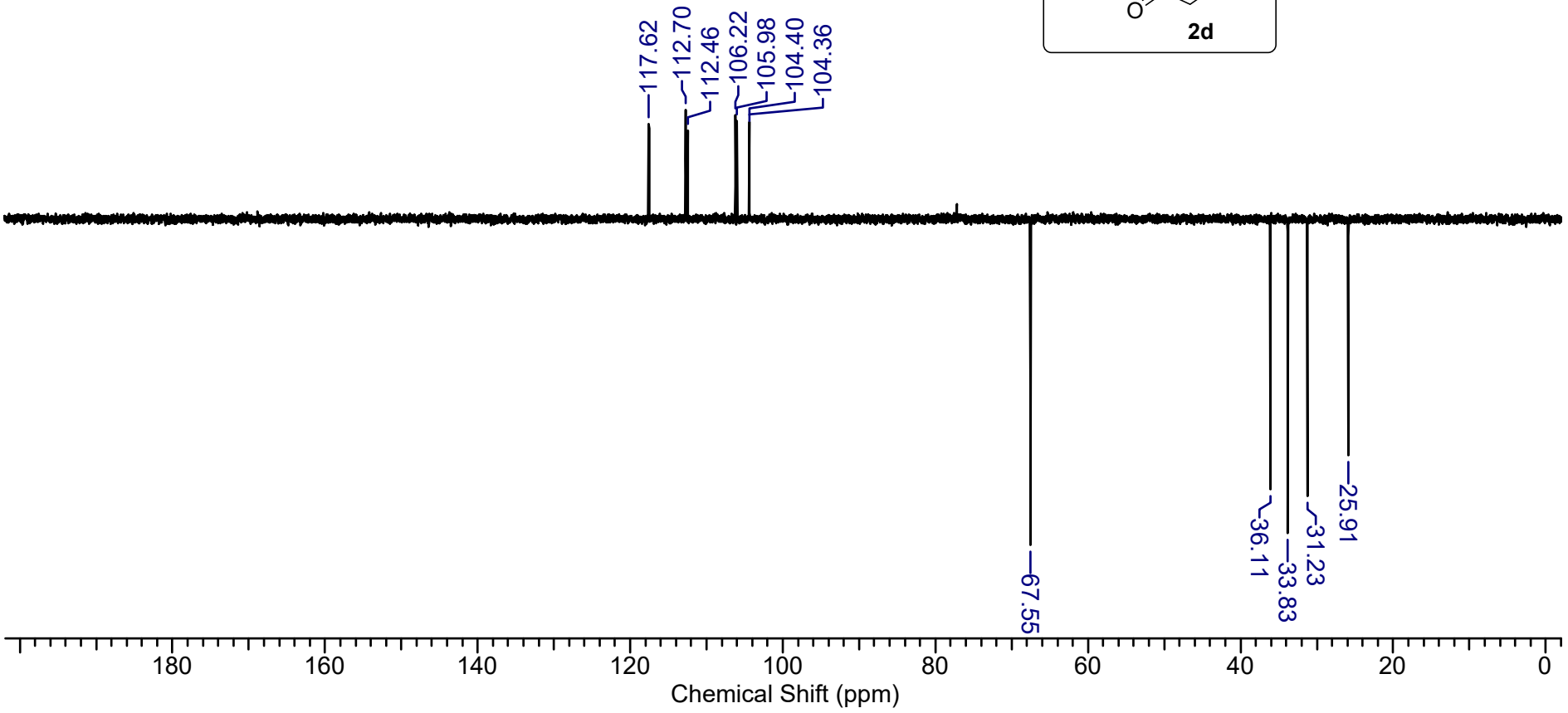


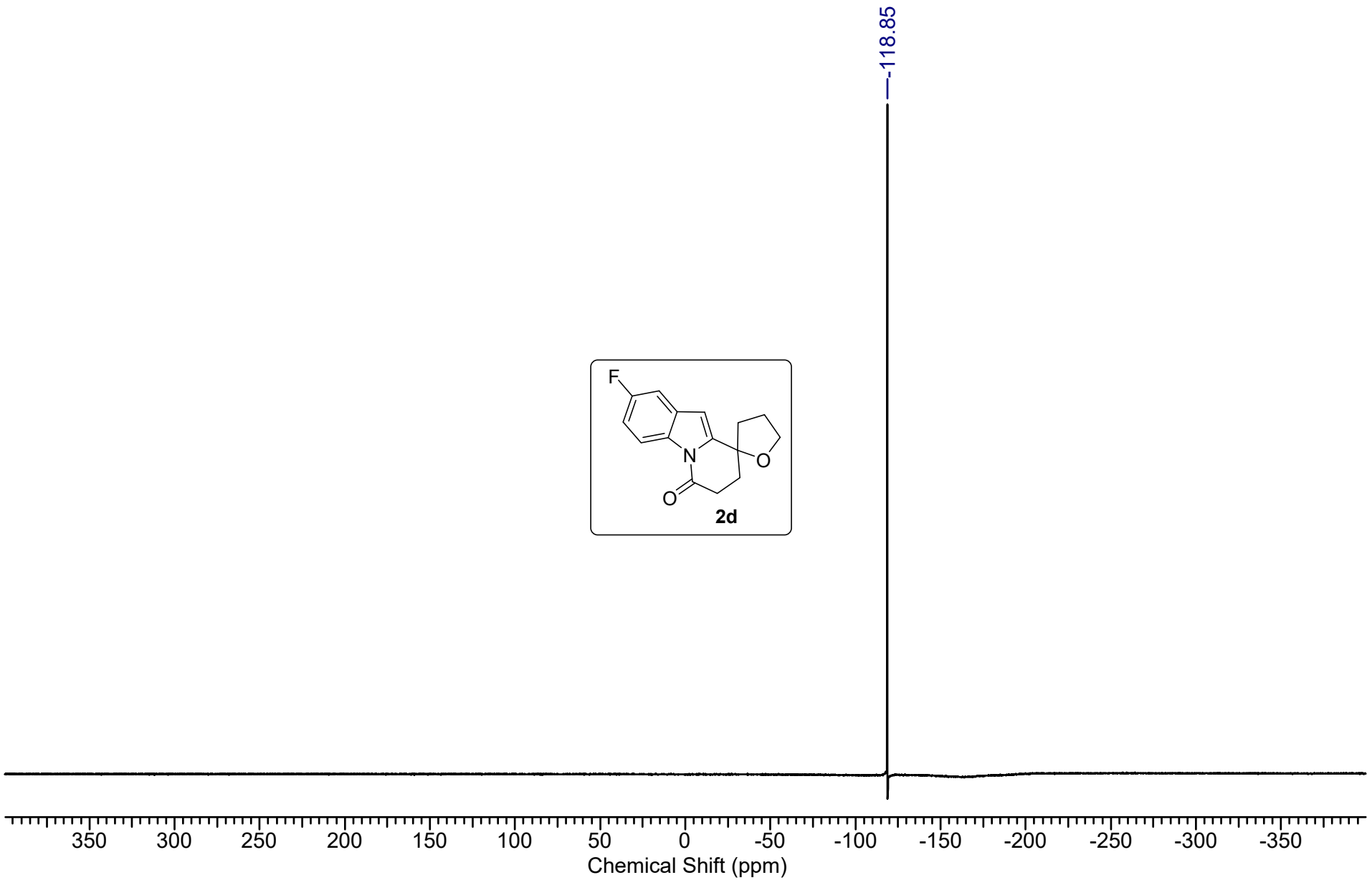

F
N
O
O
2d
8.45
8.44
8.43
8.41
7.27
7.18
7.05
7.04
7.03
7.02
6.47
4.01
3.99
3.99
3.97
3.97
3.17
3.16
3.15
3.13
3.11
2.81
2.76
2.49
2.45
2.42
2.25
2.23
2.22
2.20
2.10
2.08
2.08
2.07
2.05
1.00
1.00
1.04
1.00
2.08
1.05
1.06
1.05
5.39
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


77.32
77.00
76.68
F
N
O
O
2d
131.57
130.16
130.07
117.61
117.52
112.69
112.45
106.21
105.97
104.39
104.35
77.58
128
120
112
104
96
88
80
Chemical Shift (ppm)
168.84
161.08
158.70
143.22
131.57
130.16
130.07
117.61
117.52
112.69
112.45
106.21
105.97
104.39
77.58
67.54
36.11
33.82
31.23
25.91
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

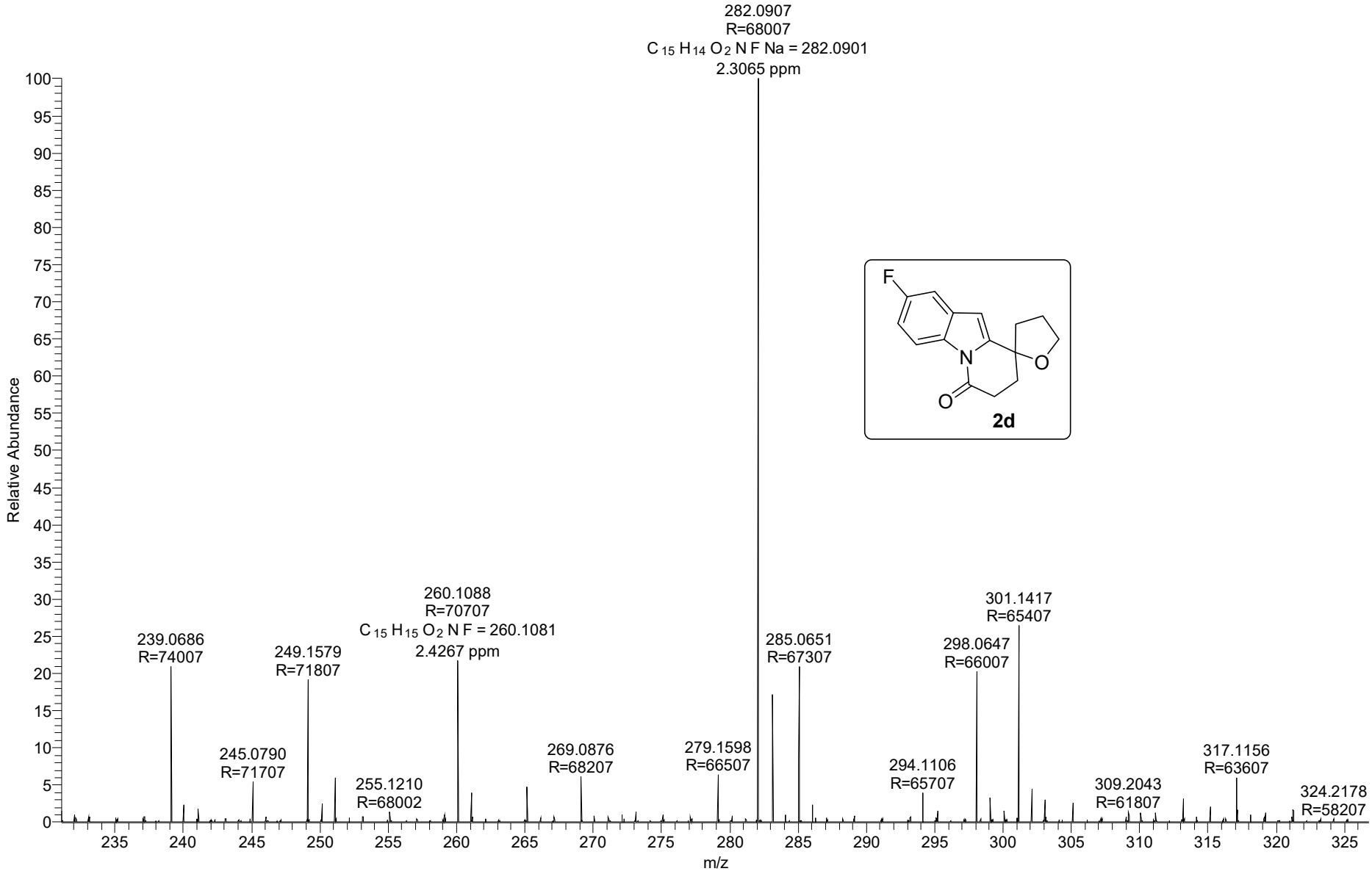

F
N
O
O

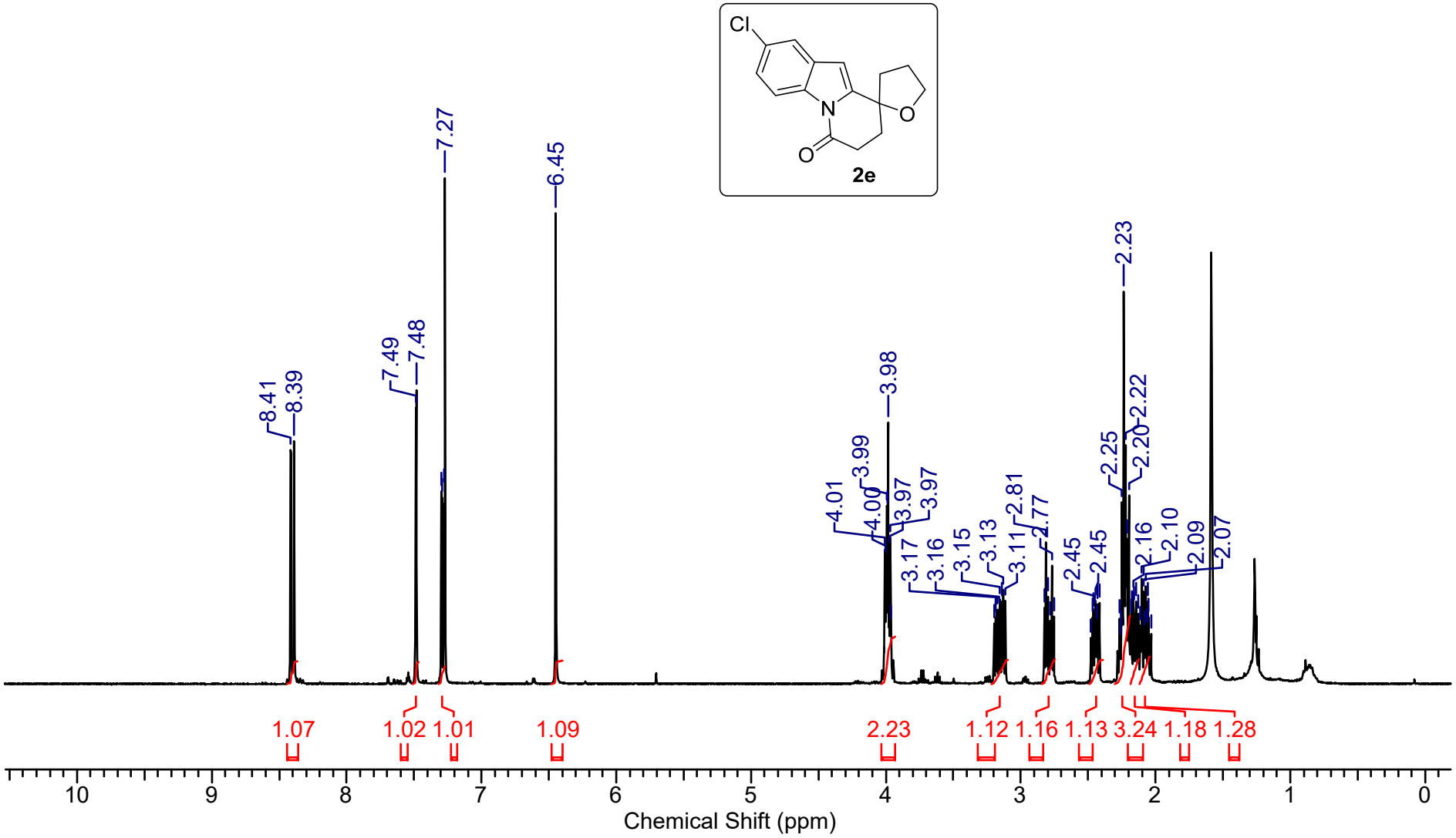
2d

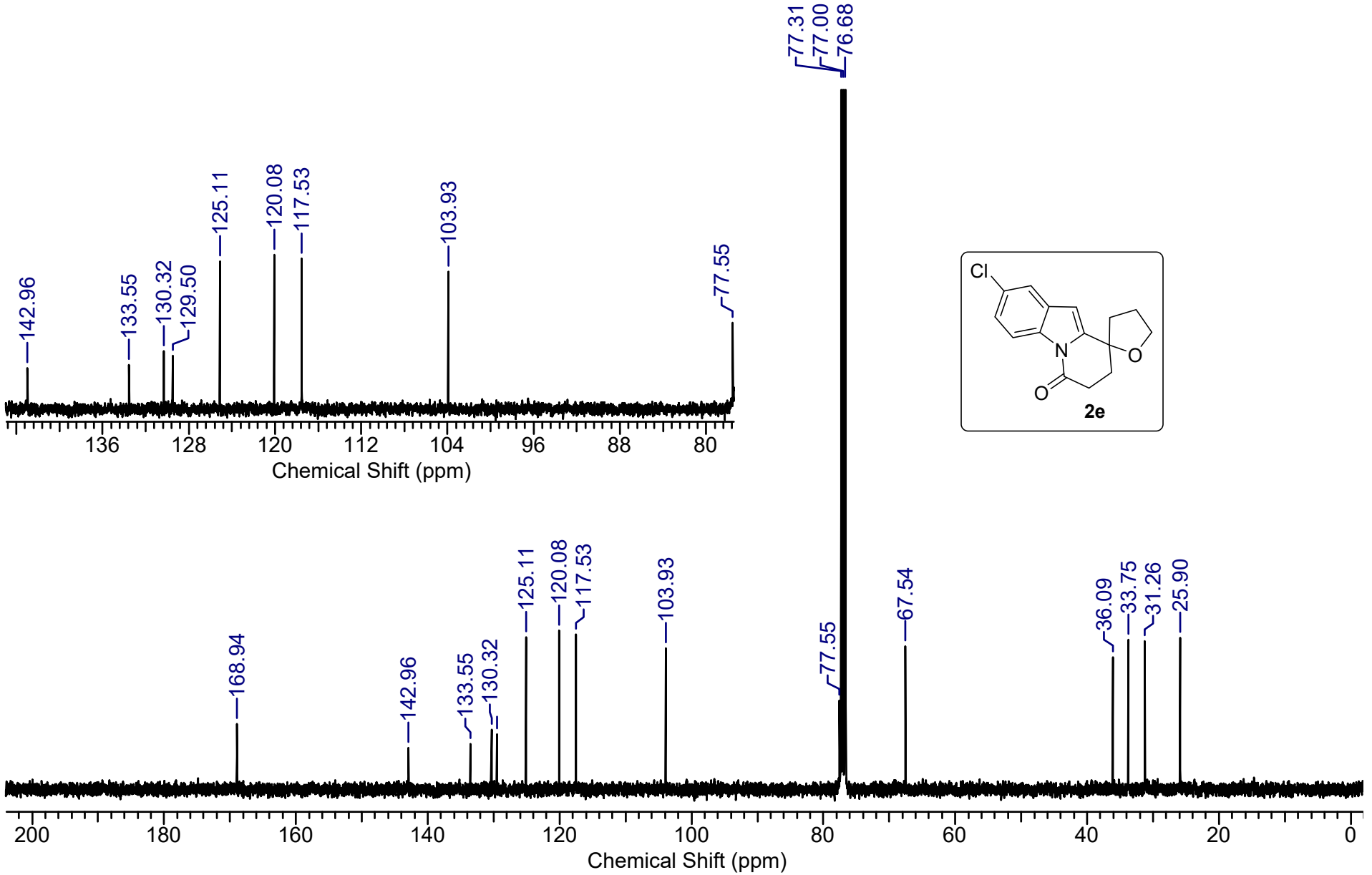

117.62
112.70
112.46
106.22
105.98
104.40
104.36
67.55
36.11
33.83
31.23
25.91
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

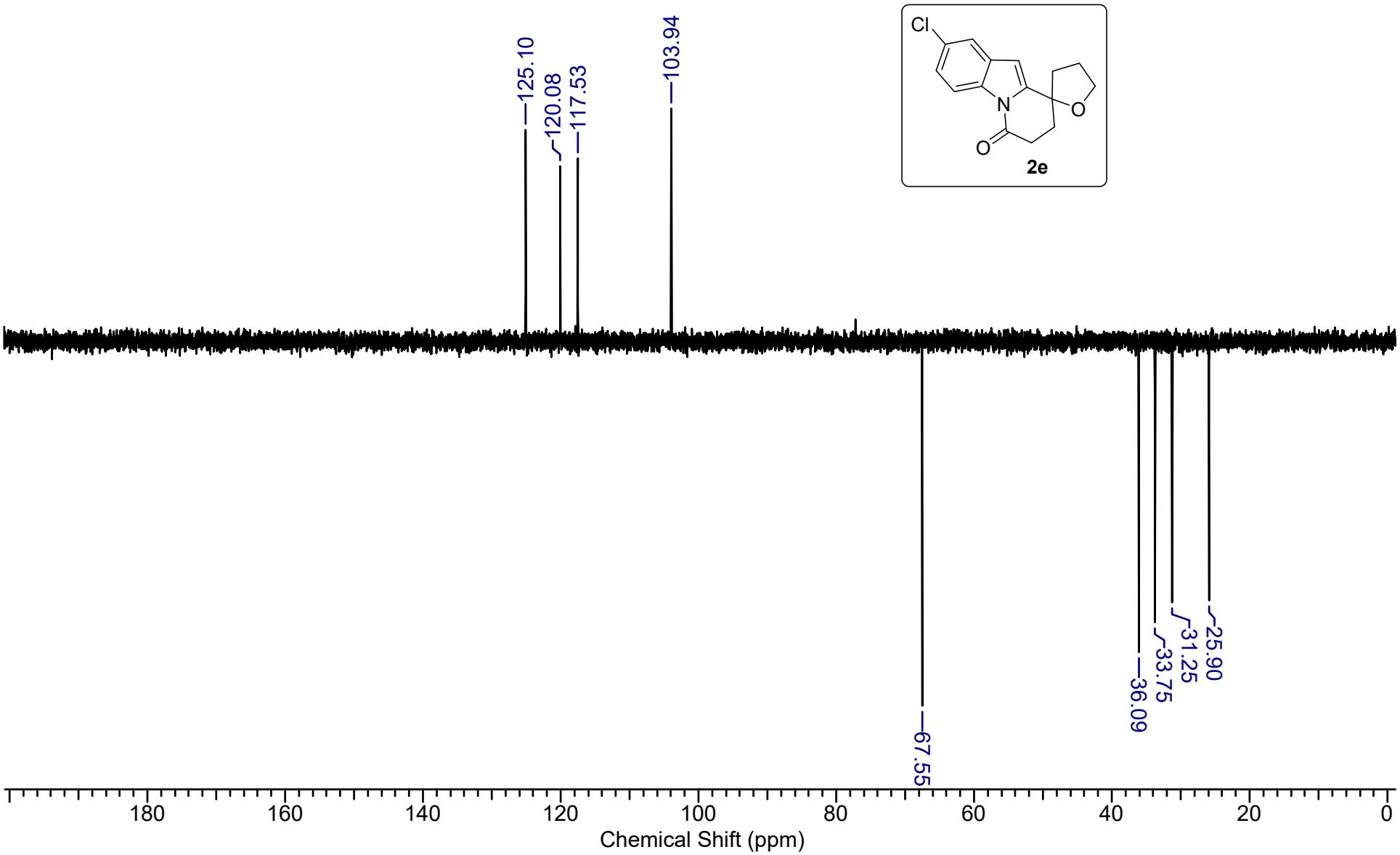

-118.85
F
N
O
O
2d
350
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
Chemical Shift (ppm)

MS-4a #253 RT: 1.35 AV: 1 NL: 3.65E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

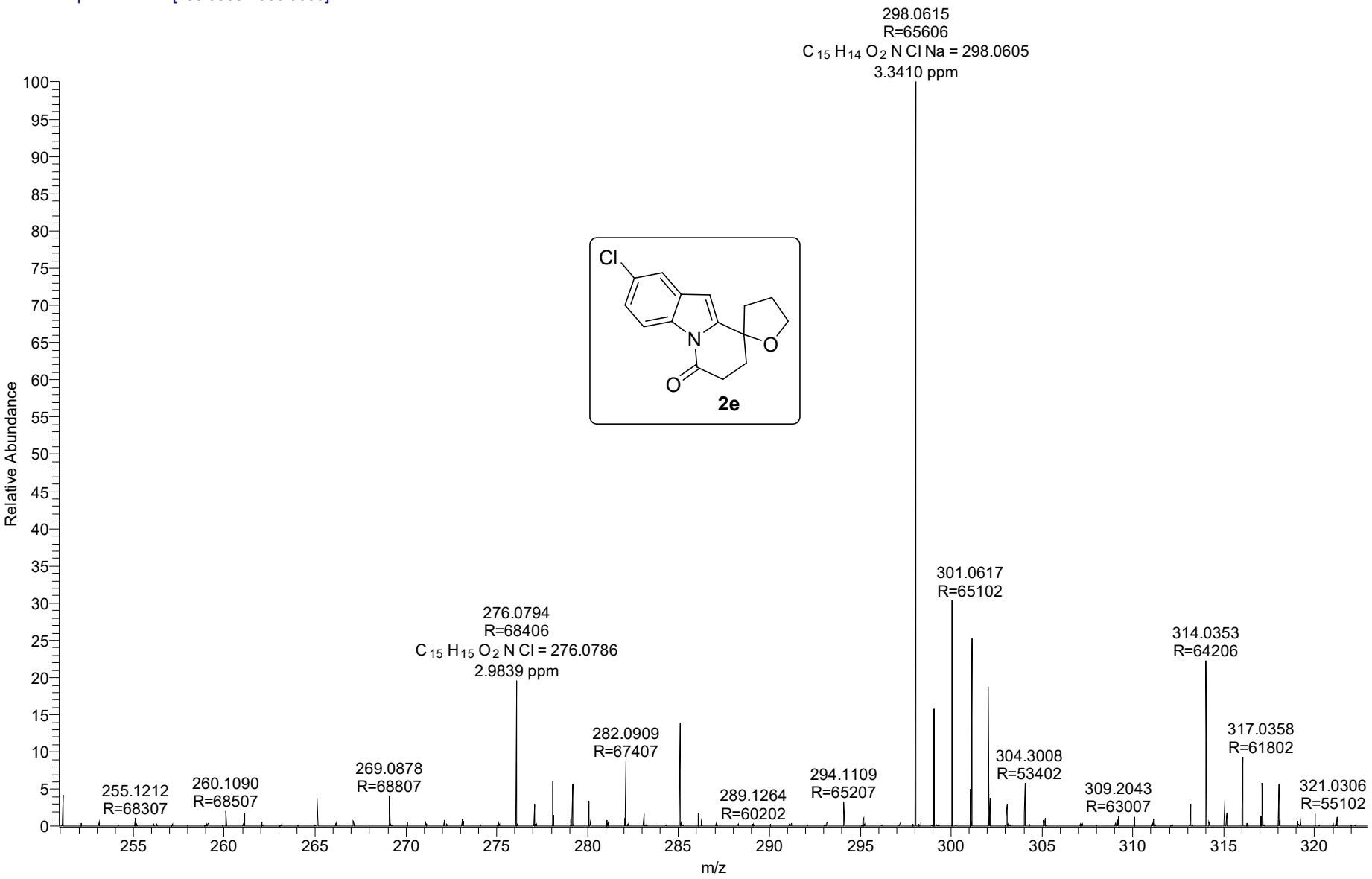


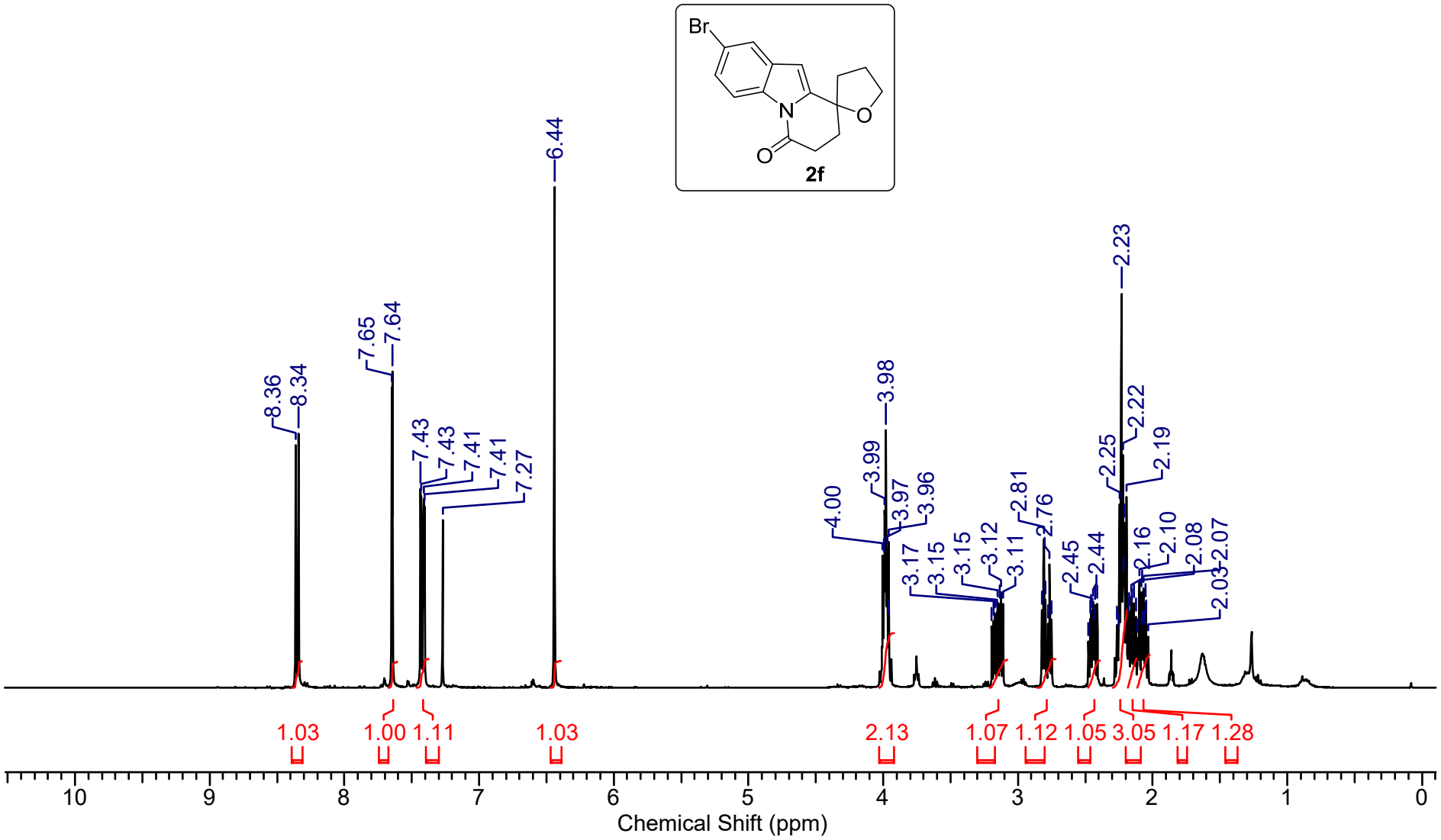

Cl
N
O
O
2e
8.41
8.39
7.49
7.48
7.27
6.45
4.01
4.00
3.99
3.98
3.97
3.97
3.17
3.16
3.15
3.13
3.11
2.81
2.77
2.45
2.45
2.25
2.23
2.22
2.20
2.16
2.10
2.09
2.07
1.07
1.02
1.01
1.09
2.23
1.12
1.16
1.13
3.24
1.18
1.28
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

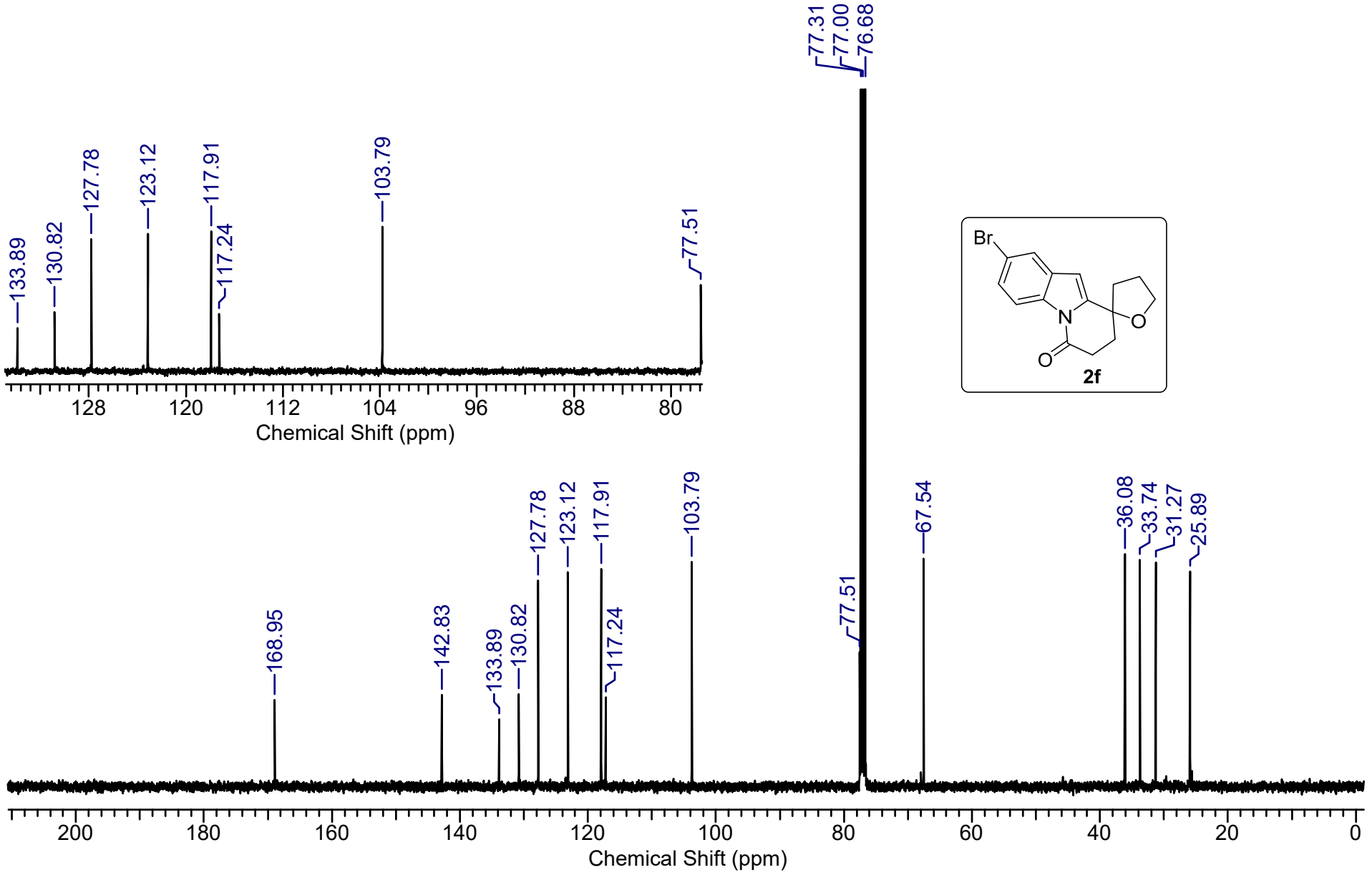

77.31
77.00
76.68
142.96
133.55
130.32
129.50
125.11
120.08
117.53
103.93
77.55
Cl
N
O
O
2e
136
128
120
112
104
96
88
80
Chemical Shift (ppm)
168.94
142.96
133.55
130.32
125.11
120.08
117.53
103.93
77.55
67.54
36.09
33.75
31.26
25.90
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

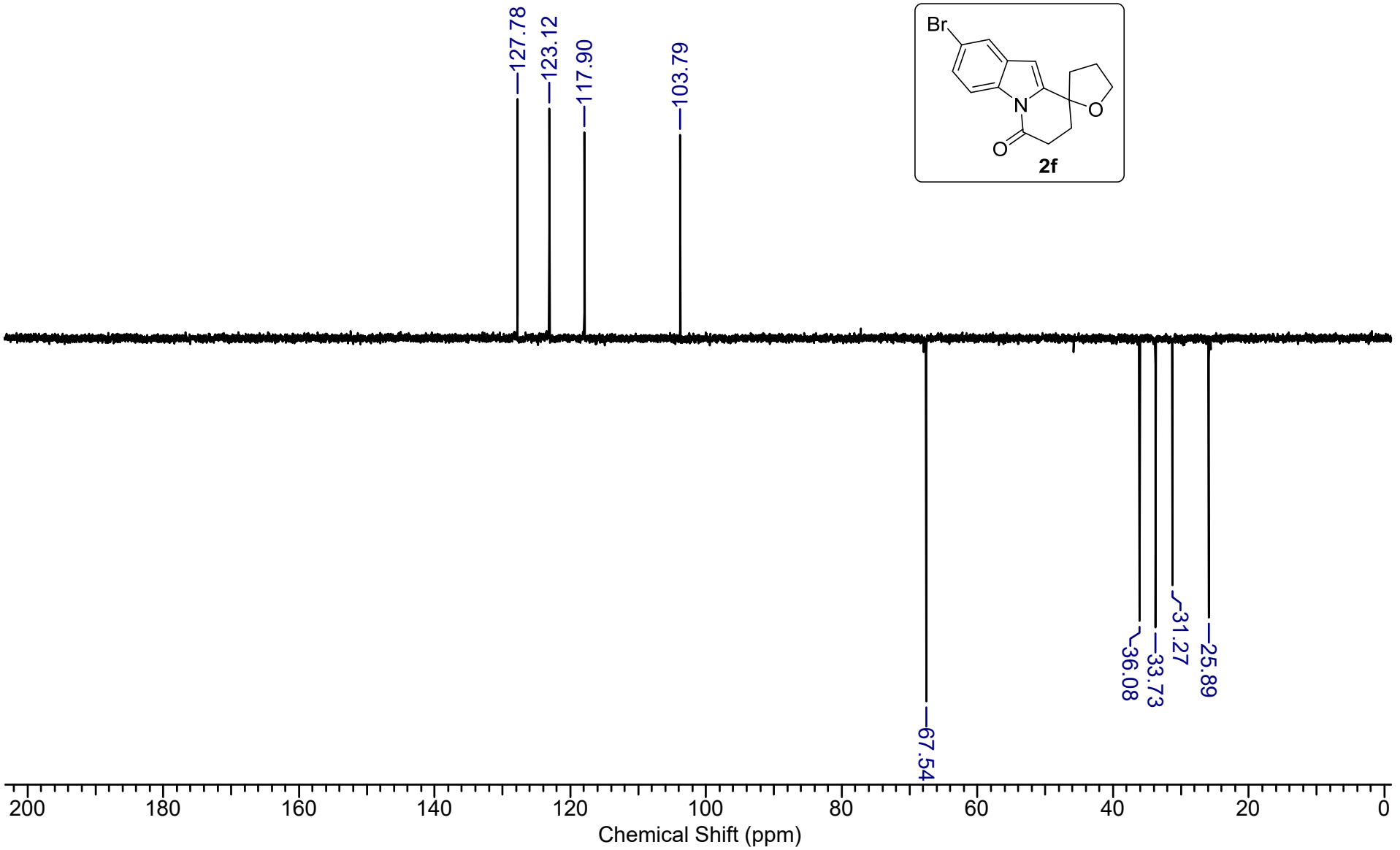

125.10
120.08
117.53
103.94
67.55
36.09
33.75
31.25
25.90
Cl
N
O
O
2e
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-5a #274 RT: 1.47 AV: 1 NL: 4.56E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

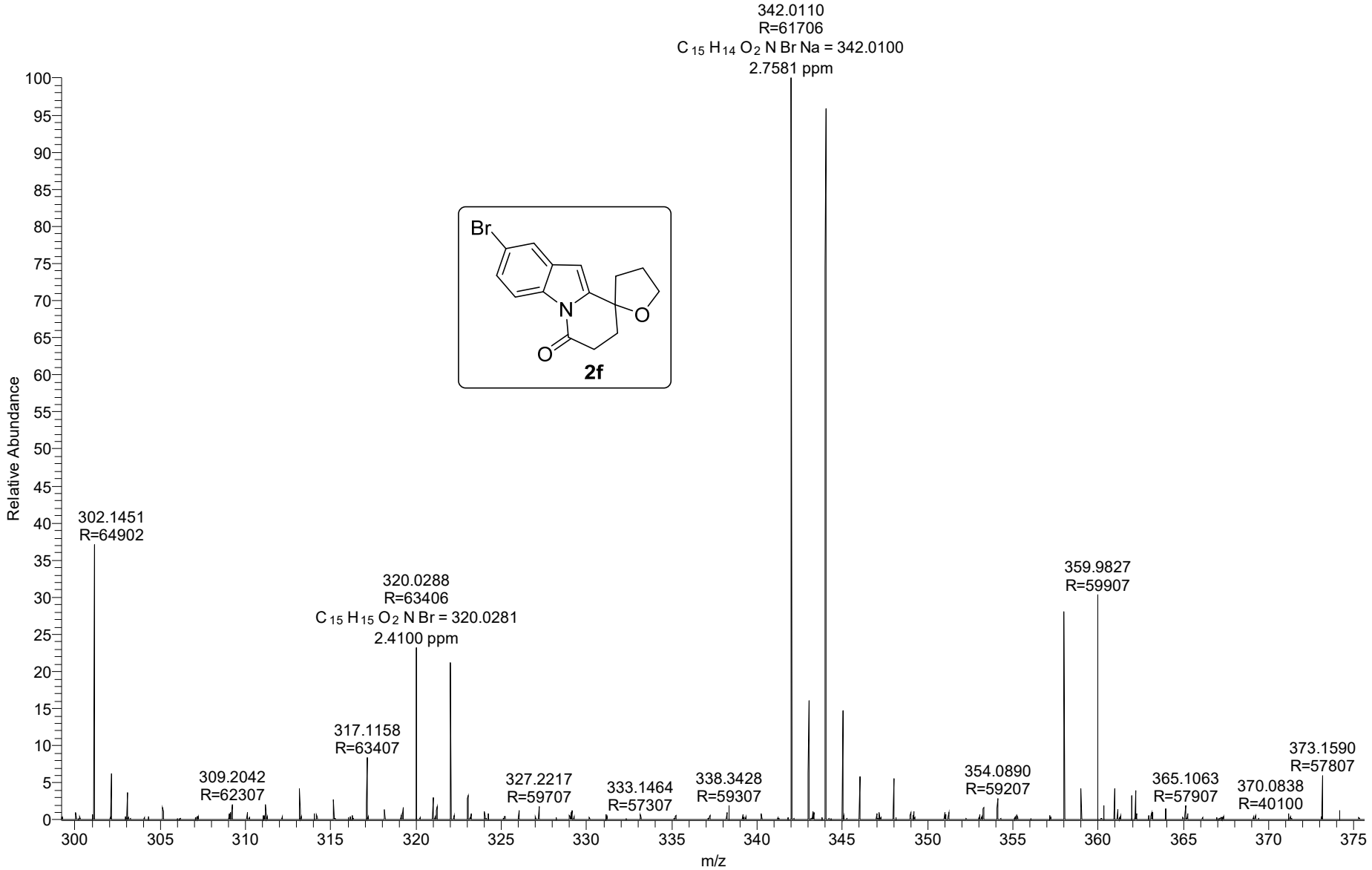


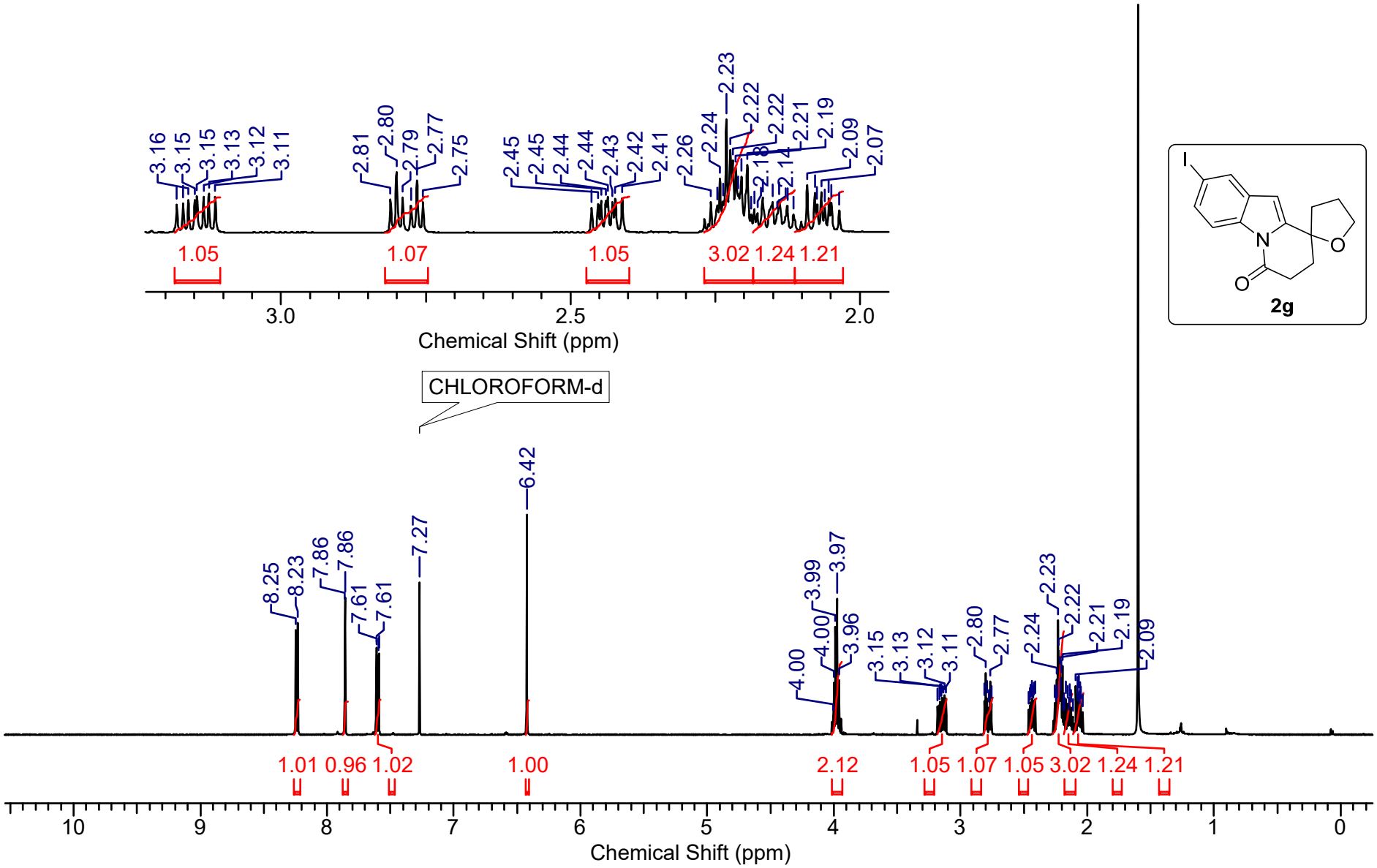

Br
N
O
O
2f
8.36
8.34
7.65
7.64
7.43
7.43
7.41
7.41
7.27
6.44
4.00
3.99
3.98
3.97
3.96
3.17
3.15
3.15
3.12
3.11
2.81
2.76
2.45
2.44
2.25
2.23
2.22
2.19
2.16
2.10
2.08
2.07
2.03
1.03
1.00
1.11
1.03
2.13
1.07
1.12
1.05
3.05
1.17
1.28
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

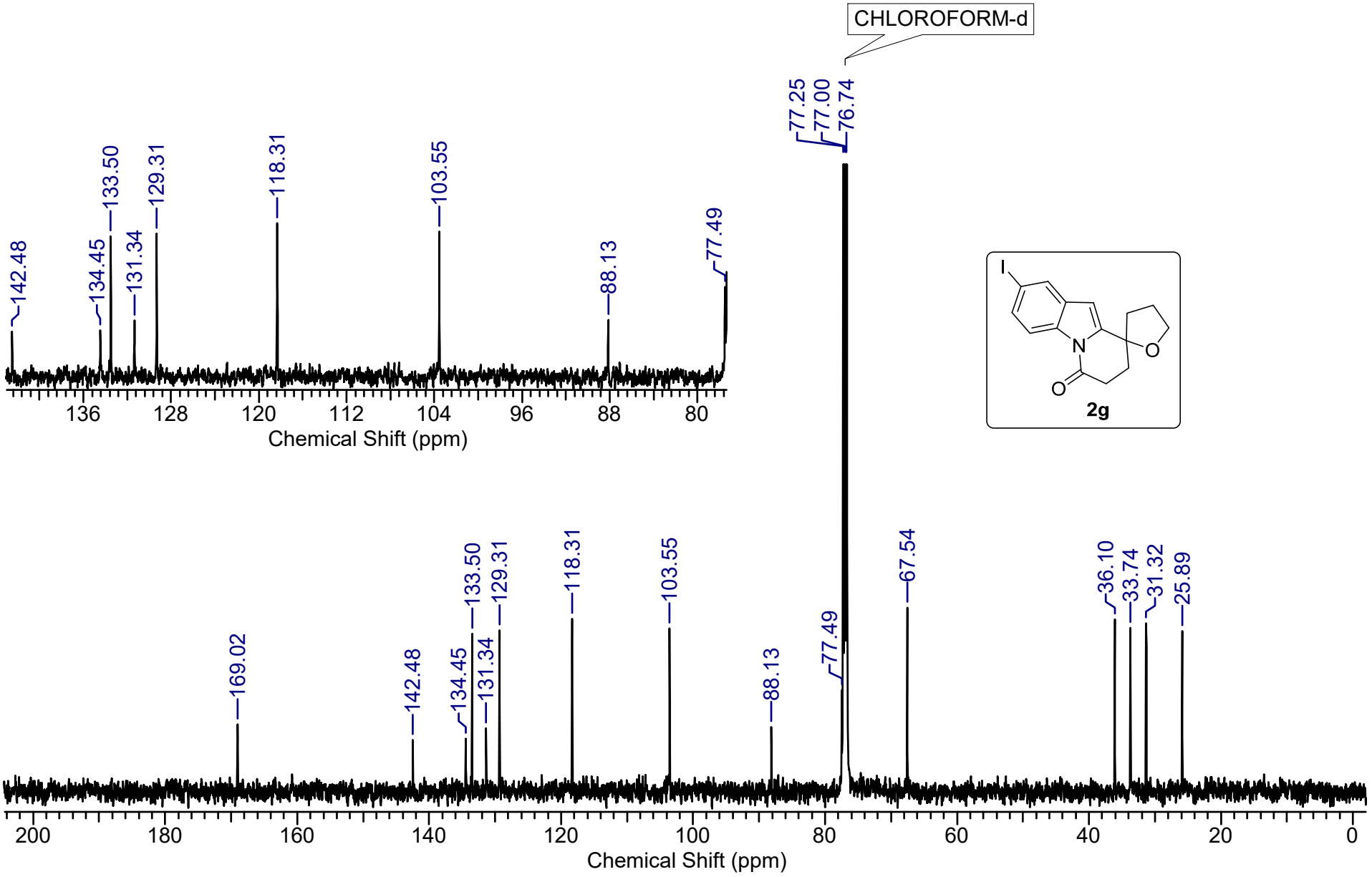

77.31
77.00
76.68
133.89
130.82
127.78
123.12
117.91
117.24
103.79
77.51
Br
N
O
O
2f
128
120
112
104
96
88
80
Chemical Shift (ppm)
168.95
142.83
133.89
130.82
127.78
123.12
117.91
117.24
103.79
77.51
67.54
36.08
33.74
31.27
25.89
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

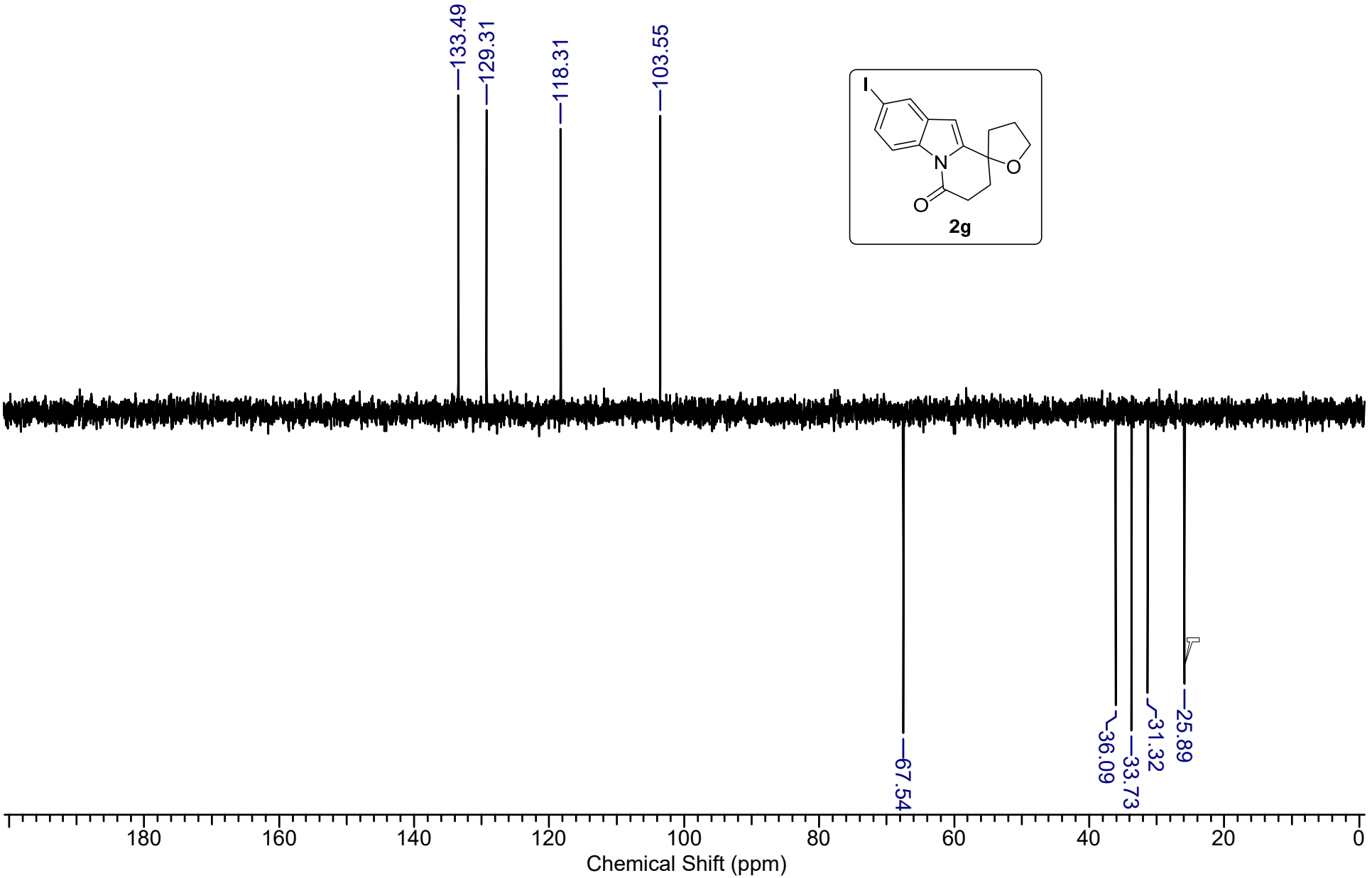

127.78
123.12
117.90
103.79
Br
N
O
O
2f
67.54
36.08
33.73
31.27
25.89
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-6a #275 RT: 1.47 AV: 1 NL: 2.85E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

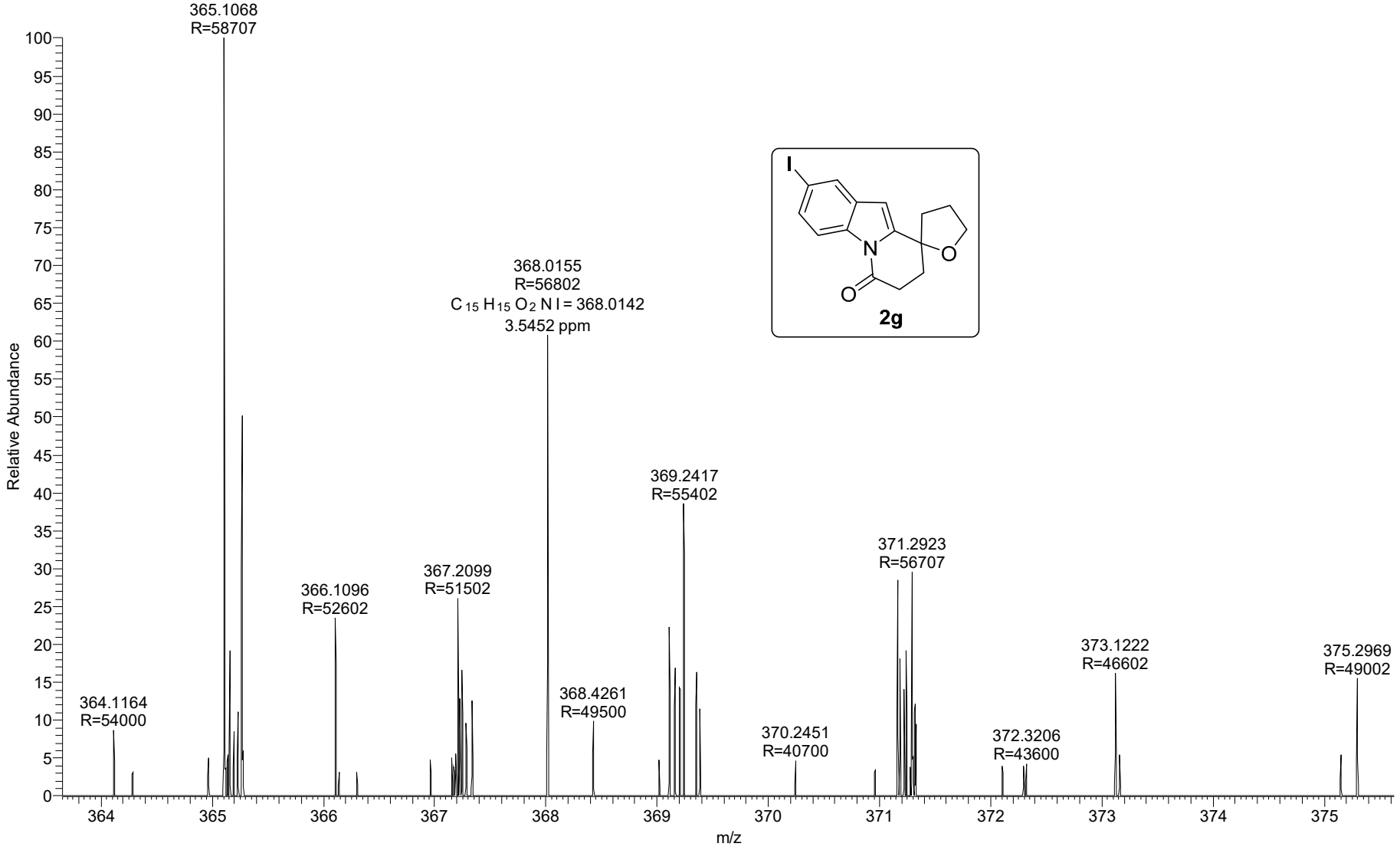


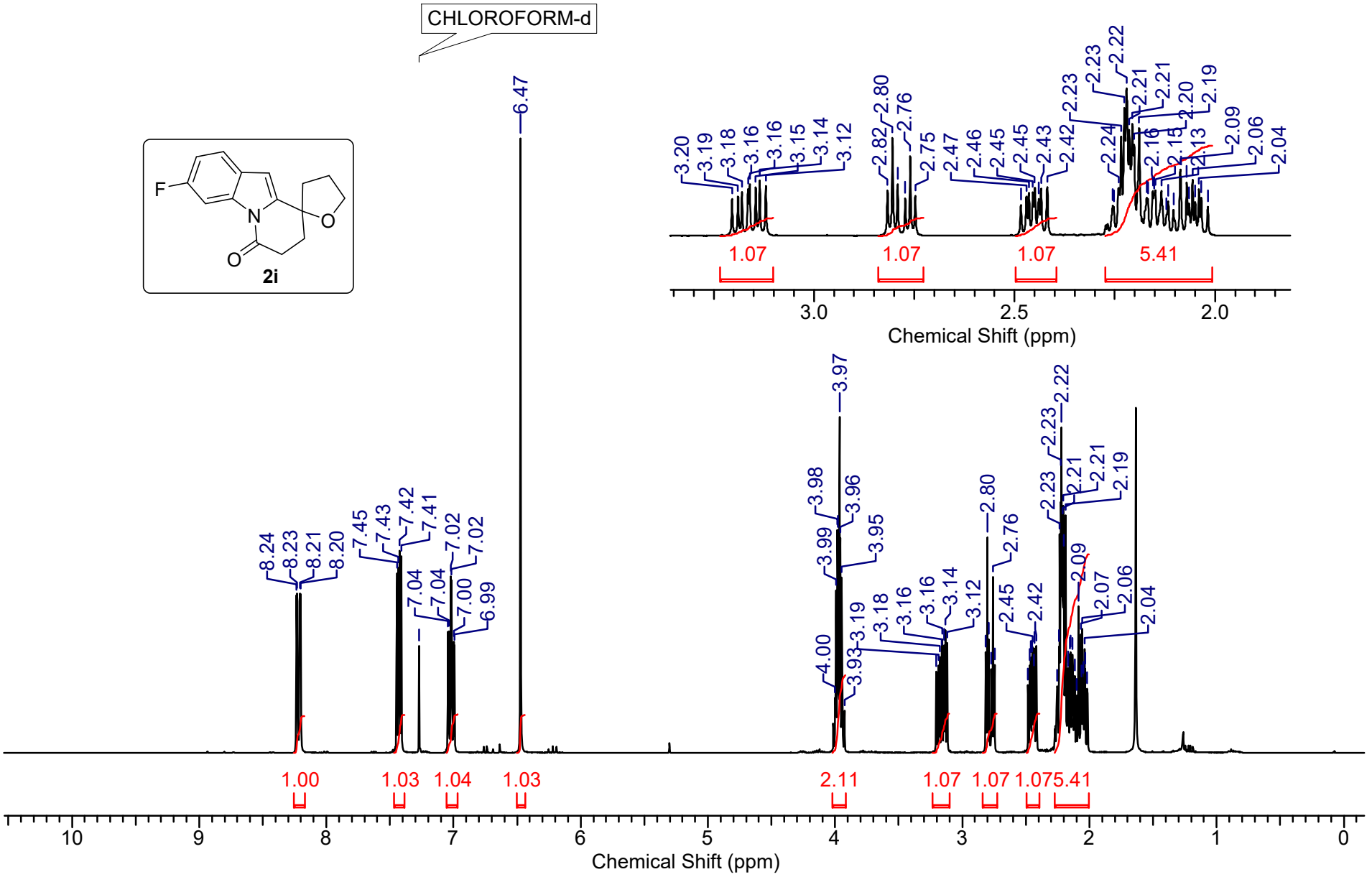

3.16
3.15
3.15
3.13
3.12
3.11
2.81
2.80
2.79
2.77
2.75
2.45
2.45
2.44
2.44
2.43
2.42
2.41
2.26
2.24
2.23
2.22
2.22
2.21
2.19
2.18
2.14
2.09
2.07
1.05
1.07
1.05
3.02 1.24 1.21
3.0
2.5
2.0
Chemical Shift (ppm)
I
N
O
O
2g
CHLOROFORM-d
8.25
8.23
7.86
7.86
7.61
7.61
7.27
6.42
4.00
3.99
4.00
3.97
3.96
3.15
3.13
3.12
3.11
2.80
2.77
2.24
2.23
2.22
2.21
2.19
2.09
1.01 0.96 1.02
1.00
2.12
1.05 1.07 1.05 3.02 1.24 1.21
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

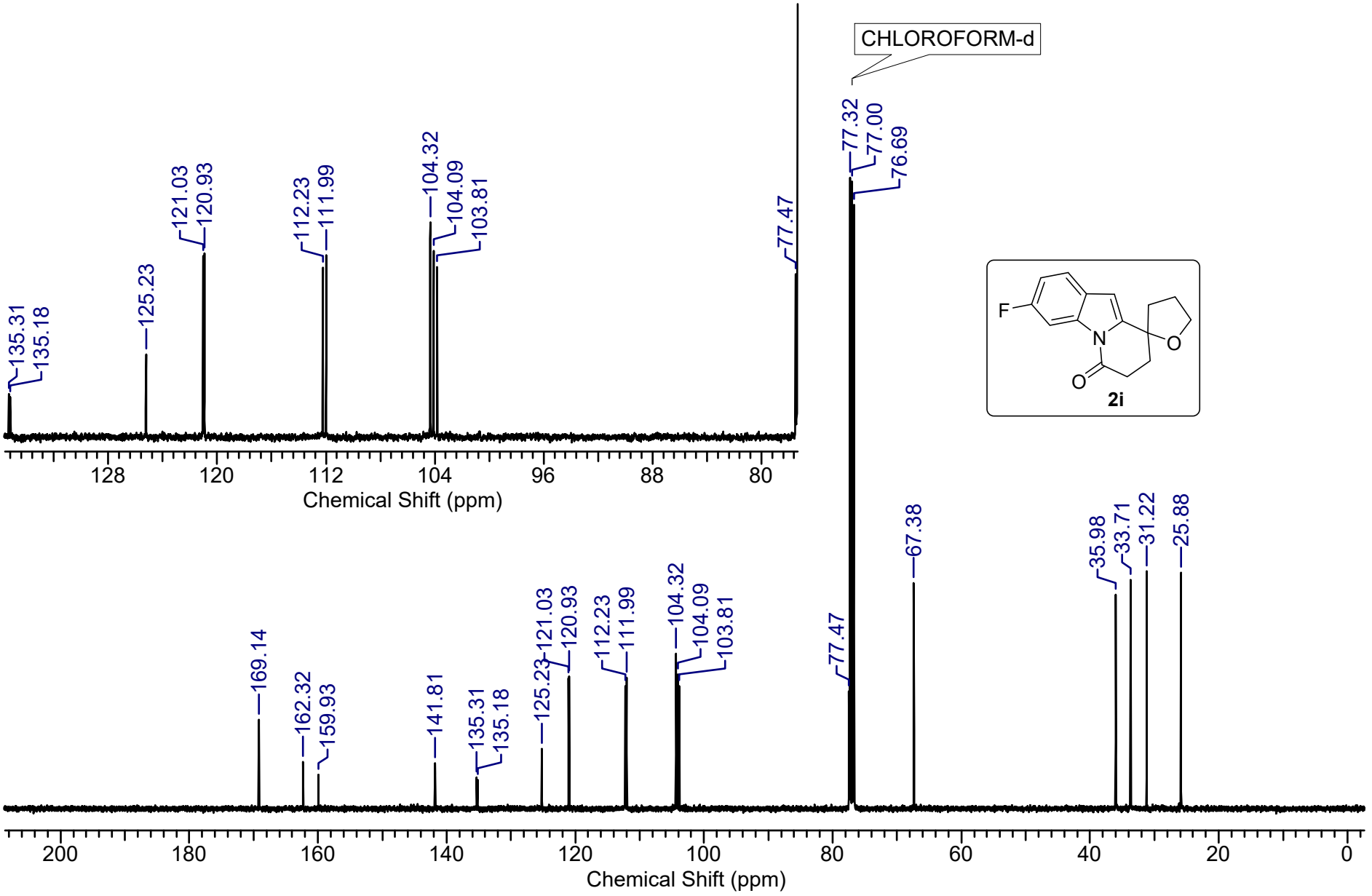

CHLOROFORM-d
77.25
77.00
76.74
142.48
134.45
133.50
131.34
129.31
118.31
103.55
88.13
77.49
136
128
120
112
104
96
88
80
Chemical Shift (ppm)
I
N
O
O
2g
169.02
142.48
134.45
133.50
131.34
129.31
118.31
103.55
88.13
77.49
67.54
36.10
33.74
31.32
25.89
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

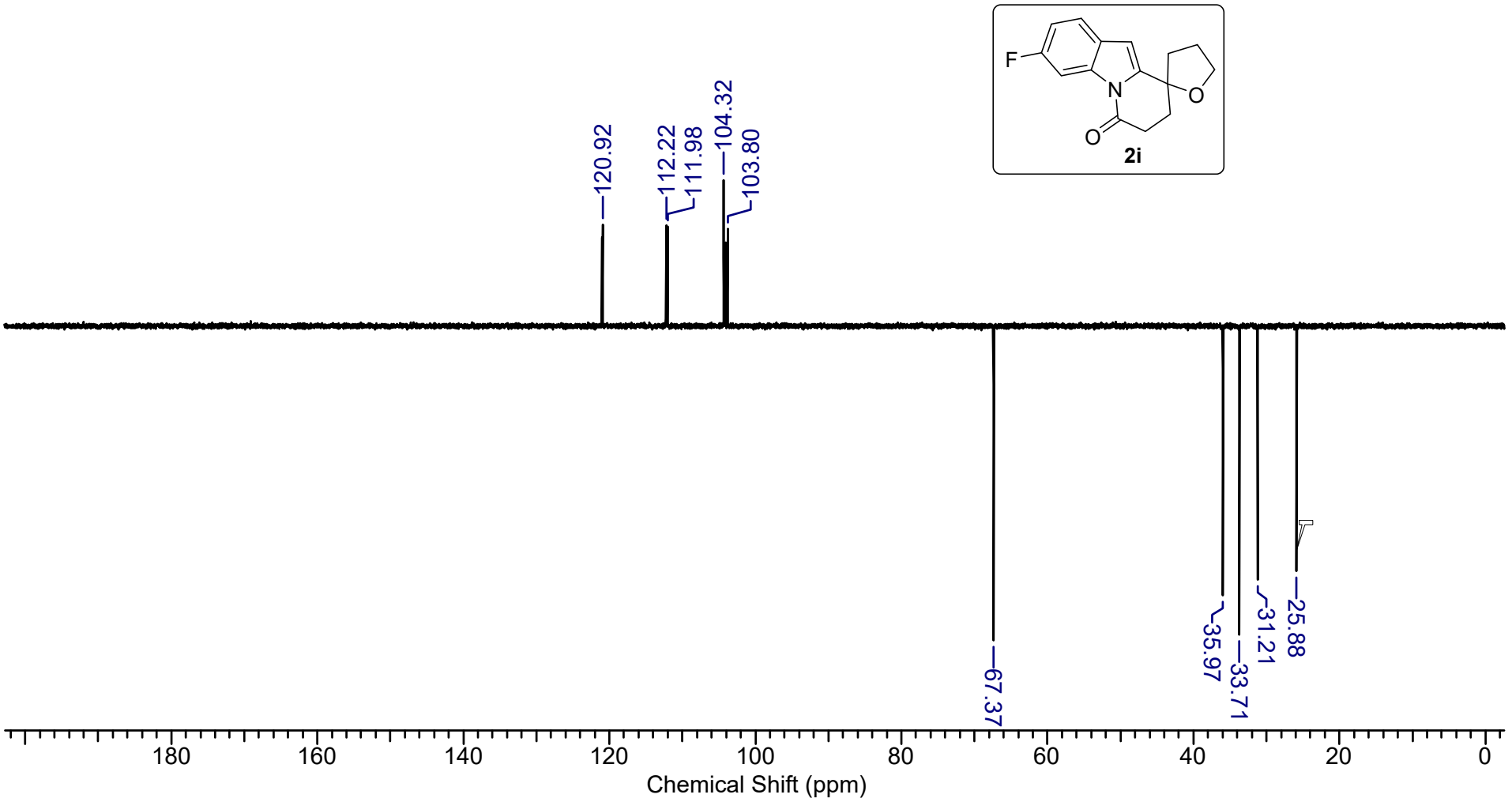

133.49
129.31
118.31
103.55
I
N
O
O
2g
67.54
36.09
33.73
31.32
25.89
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

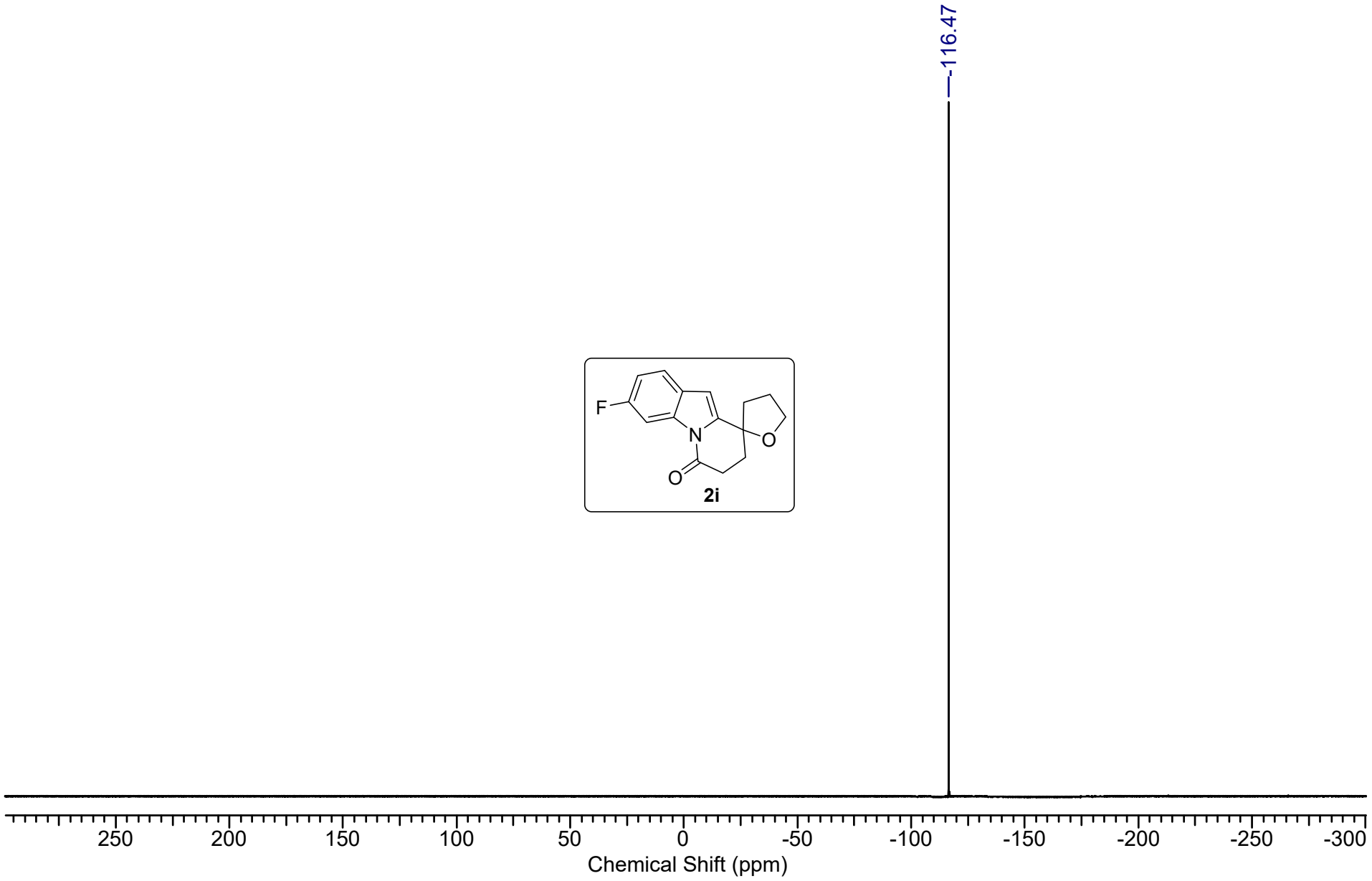
MS-7a #327 RT: 1.75 AV: 1 NL: 5.81E5
T: FTMS + p ESI Full ms [100.0000-1500.0000]



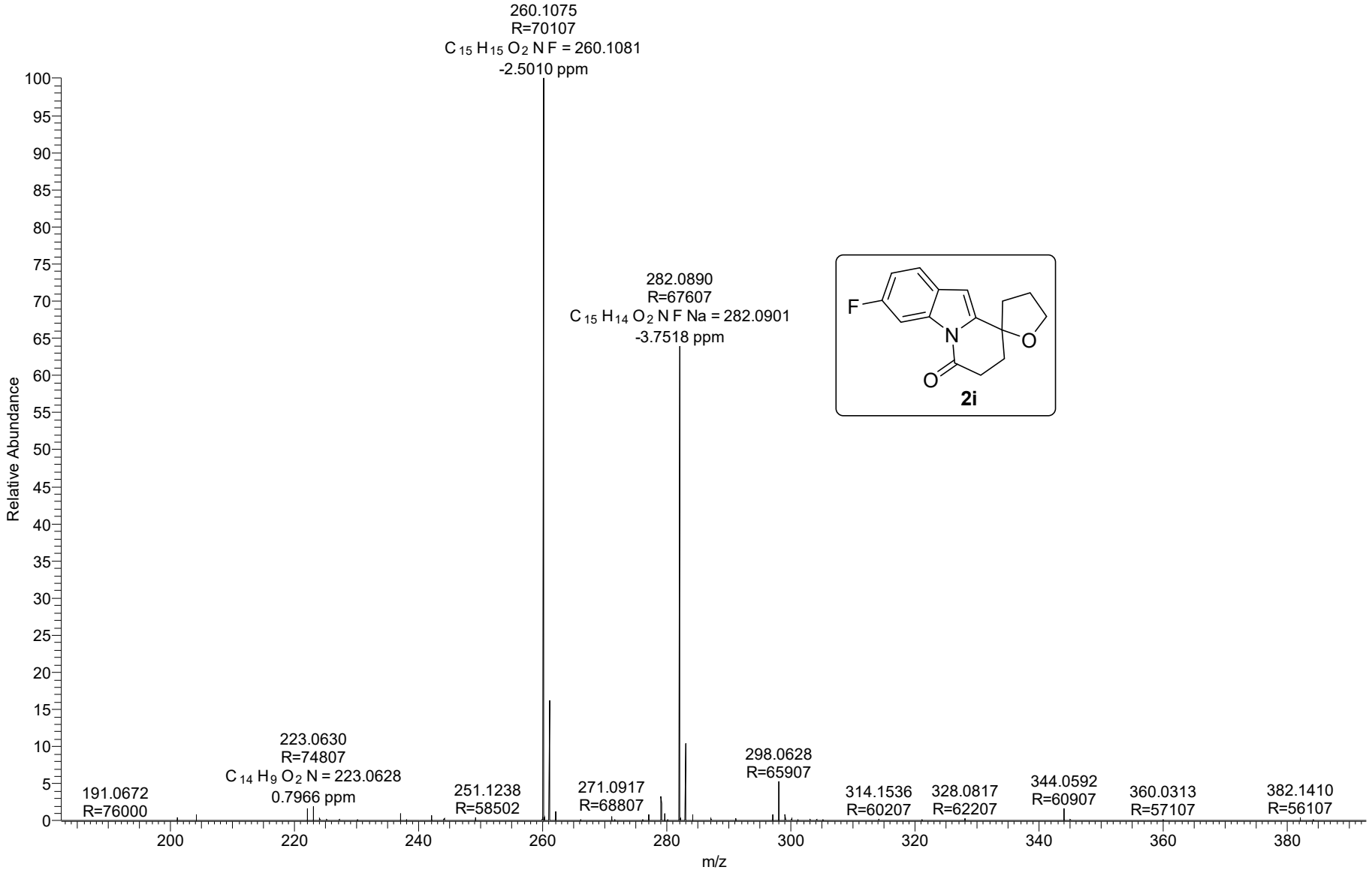

CHLOROFORM-d
F
N
O
O
2i
6.47
8.24
8.23
8.21
8.20
7.45
7.43
7.42
7.41
7.04
7.04
7.02
7.02
7.00
6.99
3.97
3.98
3.96
3.99
3.95
3.19
3.18
3.16
3.16
3.14
3.12
4.00
3.93
2.80
2.76
2.45
2.42
2.23
2.22
2.23
2.21
2.21
2.19
2.09
2.07
2.06
2.04
1.00
1.03
1.04
1.03
2.11
1.07
1.07
1.07
5.41
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
3.20
3.19
3.18
3.16
3.16
3.15
3.14
3.12
2.82
2.80
2.76
2.75
2.47
2.46
2.45
2.45
2.43
2.42
2.24
2.23
2.22
2.21
2.21
2.20
2.19
2.16
2.13
2.09
2.06
2.04
1.07
1.07
1.07
5.41
3.0
2.5
2.0
Chemical Shift (ppm)

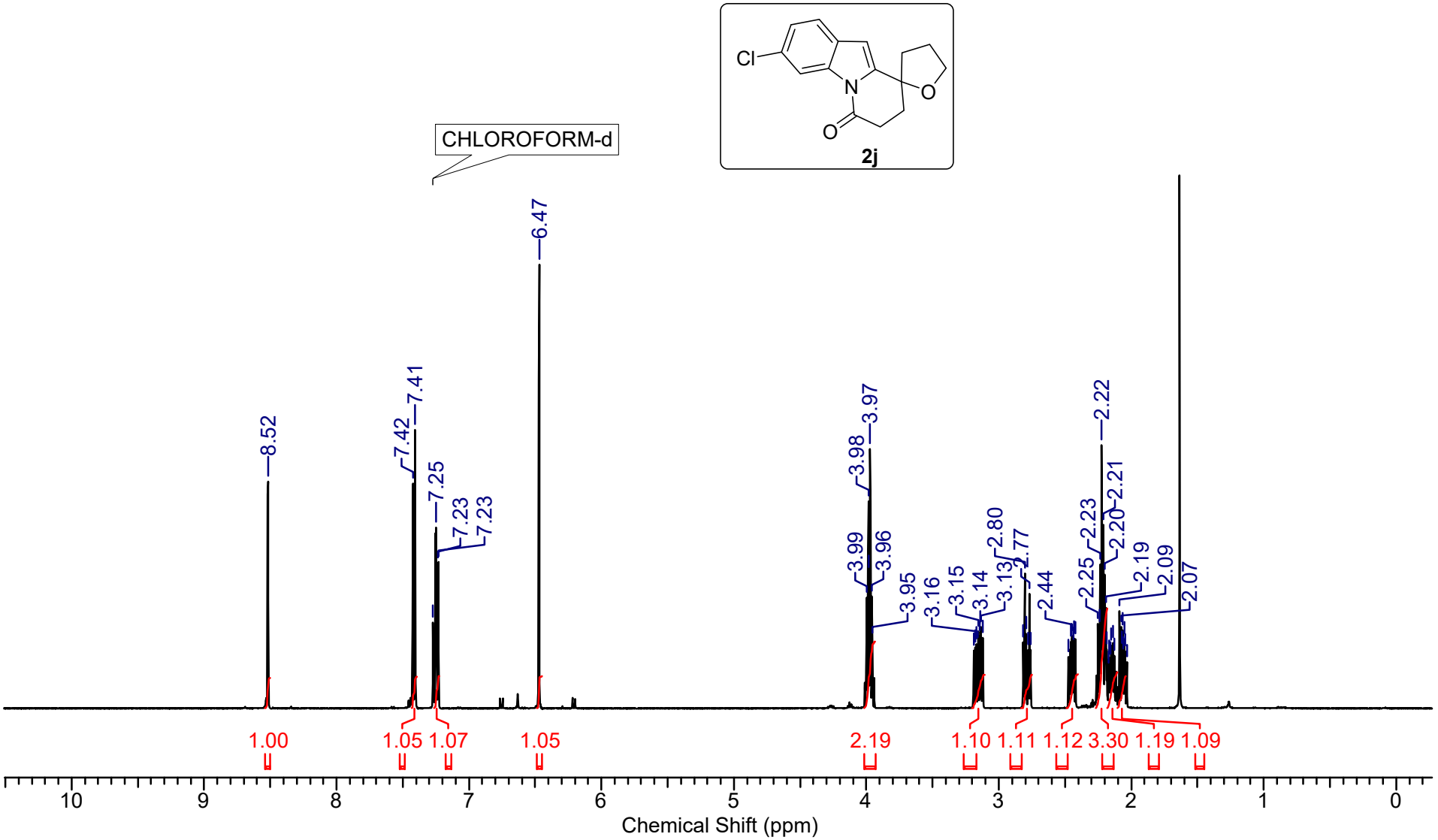

CHLOROFORM-d
77.32
77.00
76.69
77.47
104.32
104.09
103.81
121.03
120.93
112.23
111.99
125.23
135.31
135.18
169.14
162.32
159.93
141.81
67.38
35.98
33.71
31.22
25.88
F
N
O
O
2i
Chemical Shift (ppm)
Chemical Shift (ppm)

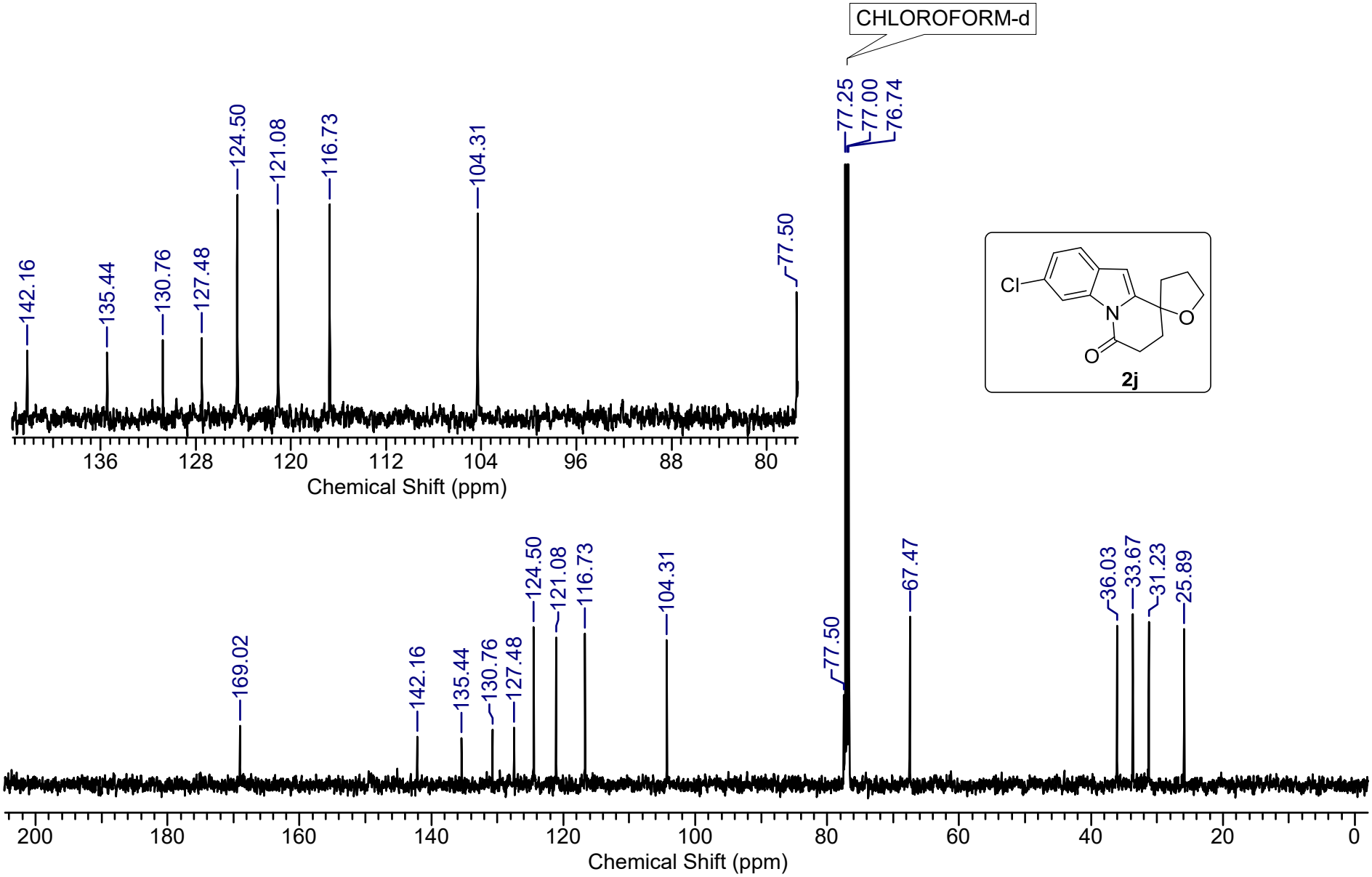

F
N
O
O
2i
120.92
112.22
111.98
104.32
103.80
67.37
35.97
33.71
31.21
25.88
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

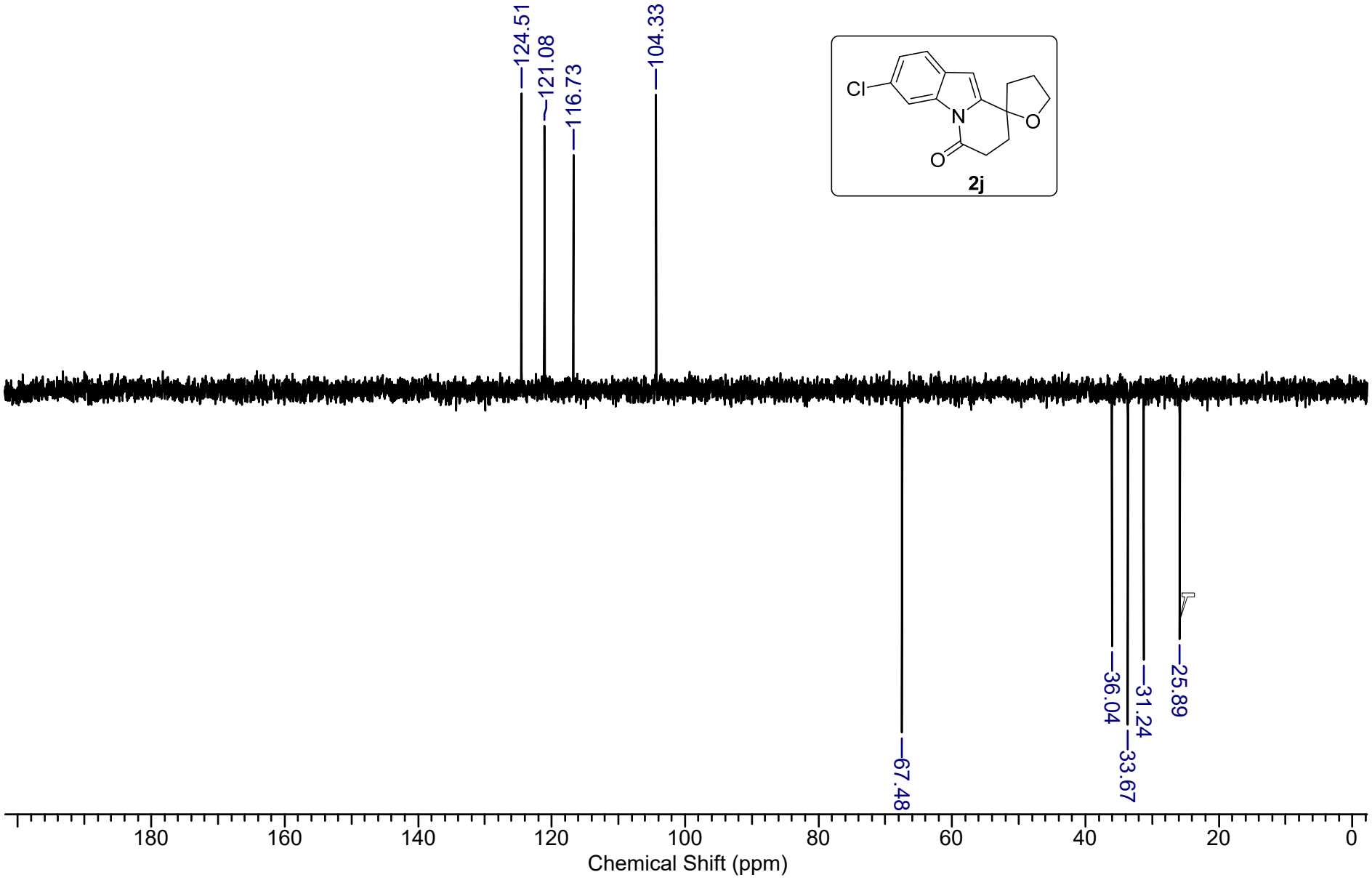

-116.47
F
N
O
O
2i
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
Chemical Shift (ppm)

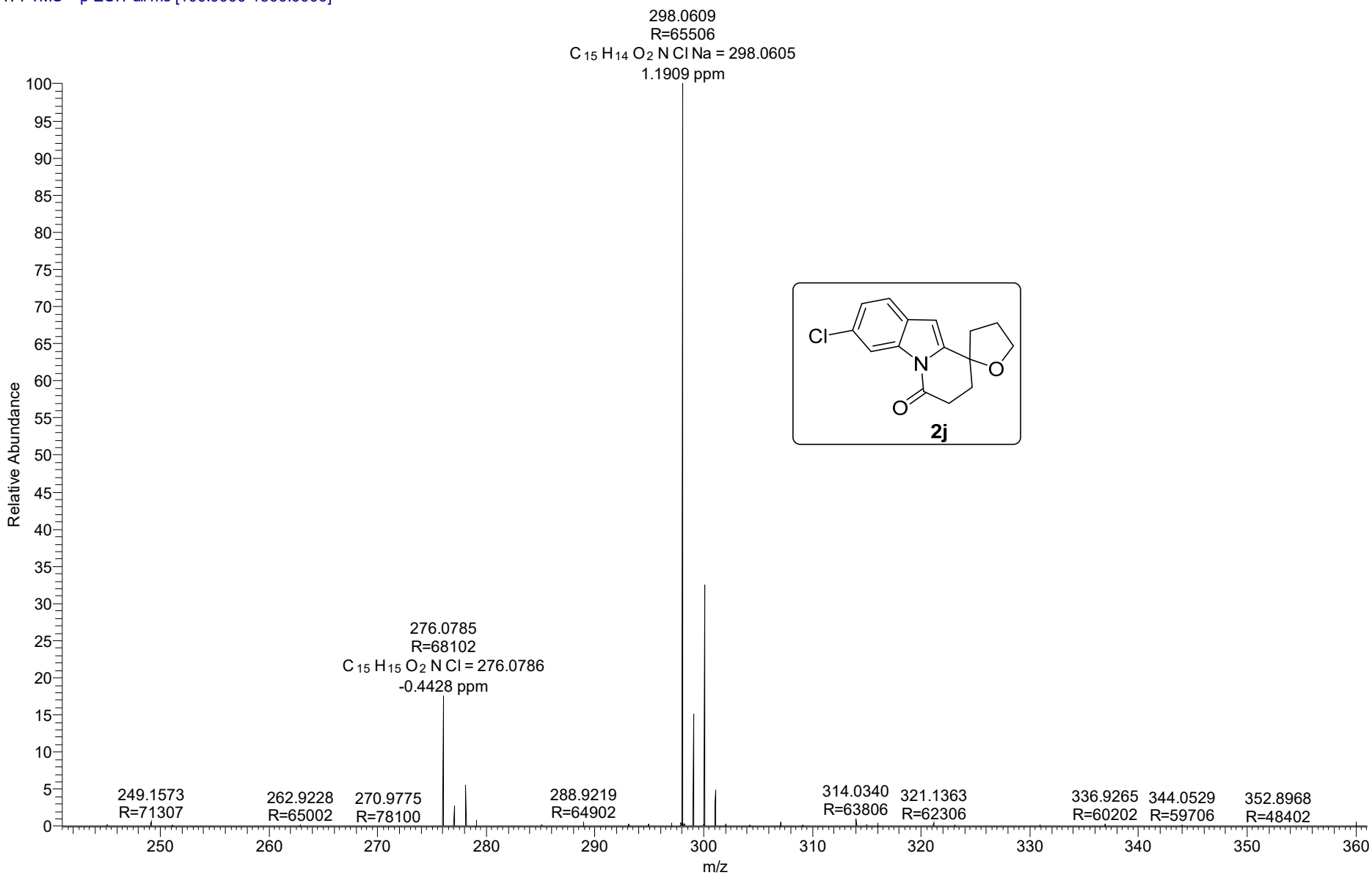
MSH-43 #275 RT: 1.50 AV: 1 NL: 1.11E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]

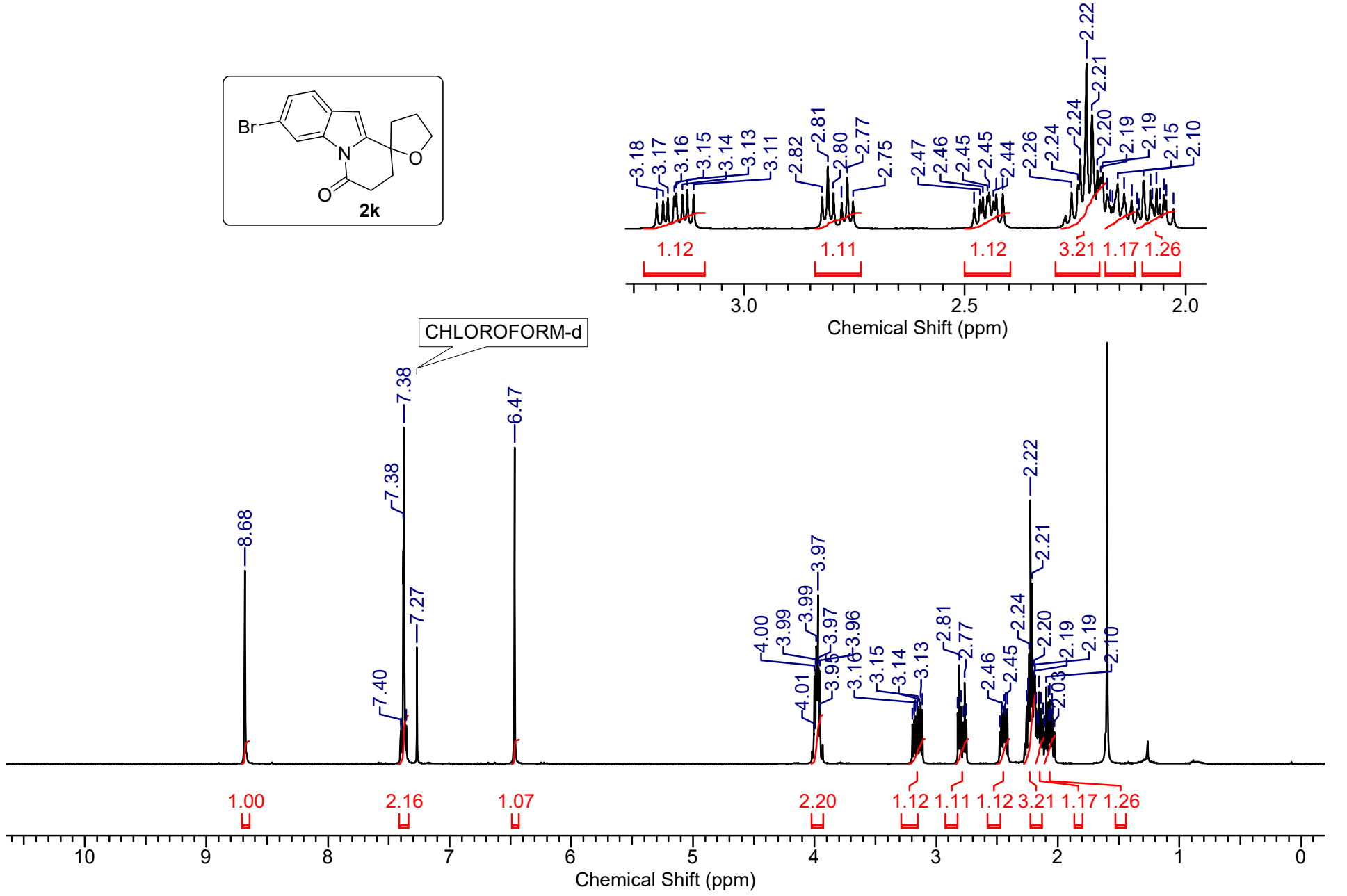


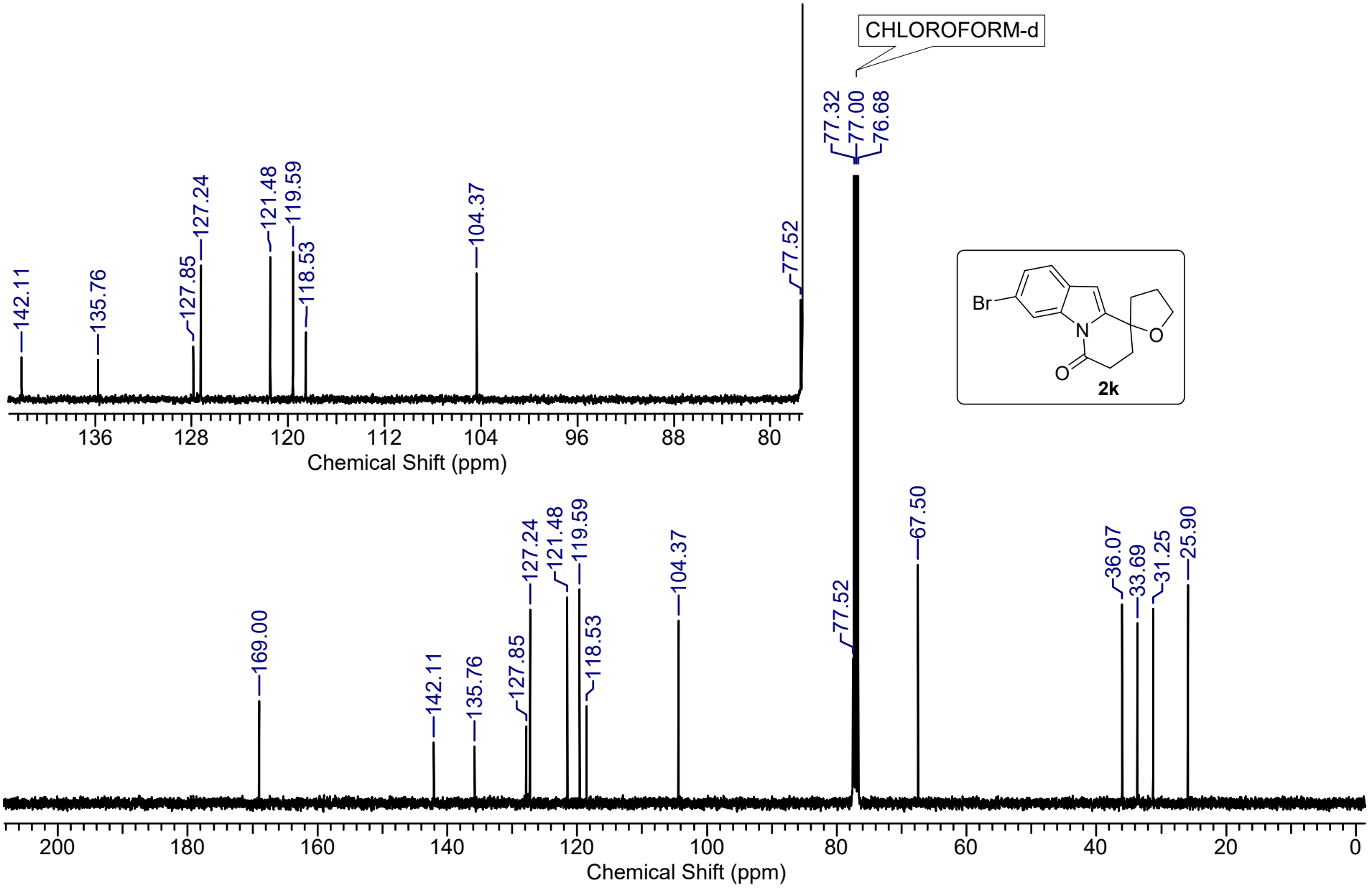

Cl
N
O
O
2j
CHLOROFORM-d
8.52
7.42
7.41
7.25
7.23
7.23
6.47
3.98
3.97
3.99
3.96
3.95
3.16
3.15
3.14
3.13
2.80
2.77
2.44
2.25
2.23
2.22
2.20
2.21
2.19
2.09
2.07
1.00
1.05
1.07
1.05
2.19
1.10
1.11
1.12
3.30
1.19
1.09
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

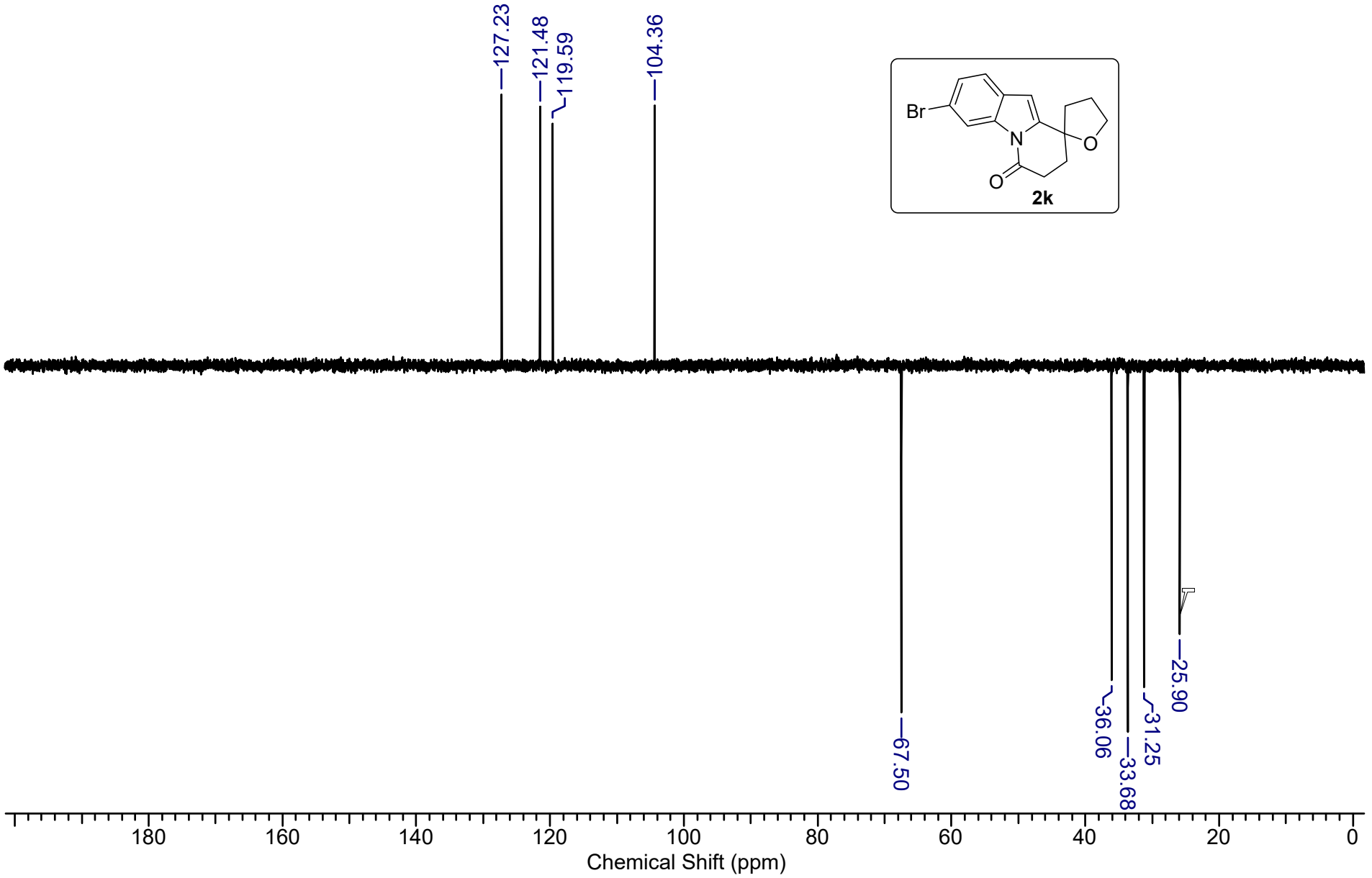

CHLOROFORM-d
77.25
77.00
76.74
142.16
135.44
130.76
127.48
124.50
121.08
116.73
104.31
77.50
Cl
N
O
O
2j
136
128
120
112
104
96
88
80
Chemical Shift (ppm)
169.02
142.16
135.44
130.76
127.48
124.50
121.08
116.73
104.31
77.50
67.47
36.03
33.67
31.23
25.89
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

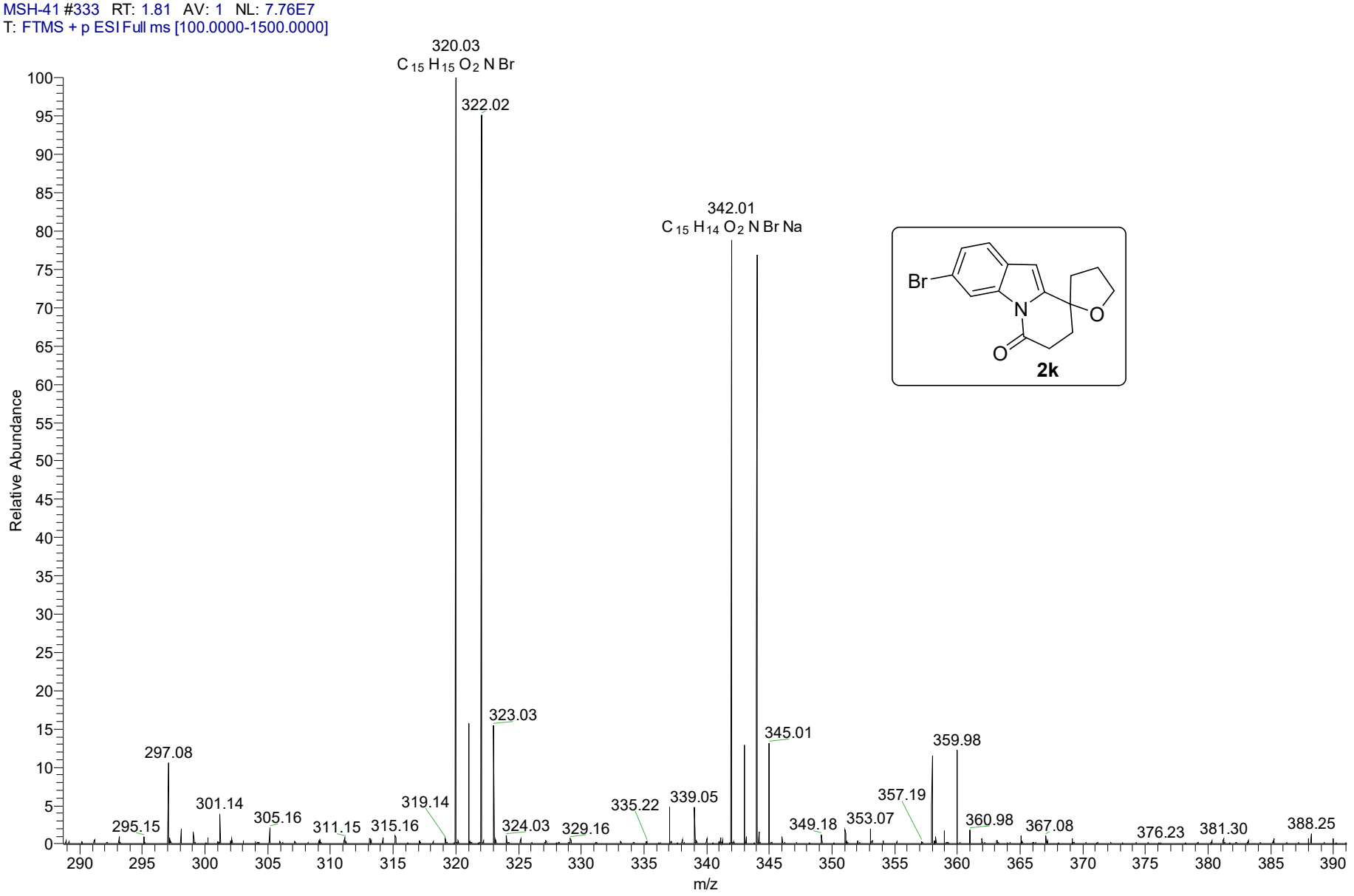

124.51
121.08
116.73
104.33
Cl
N
O
O
2j
67.48
36.04
33.67
31.24
25.89
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

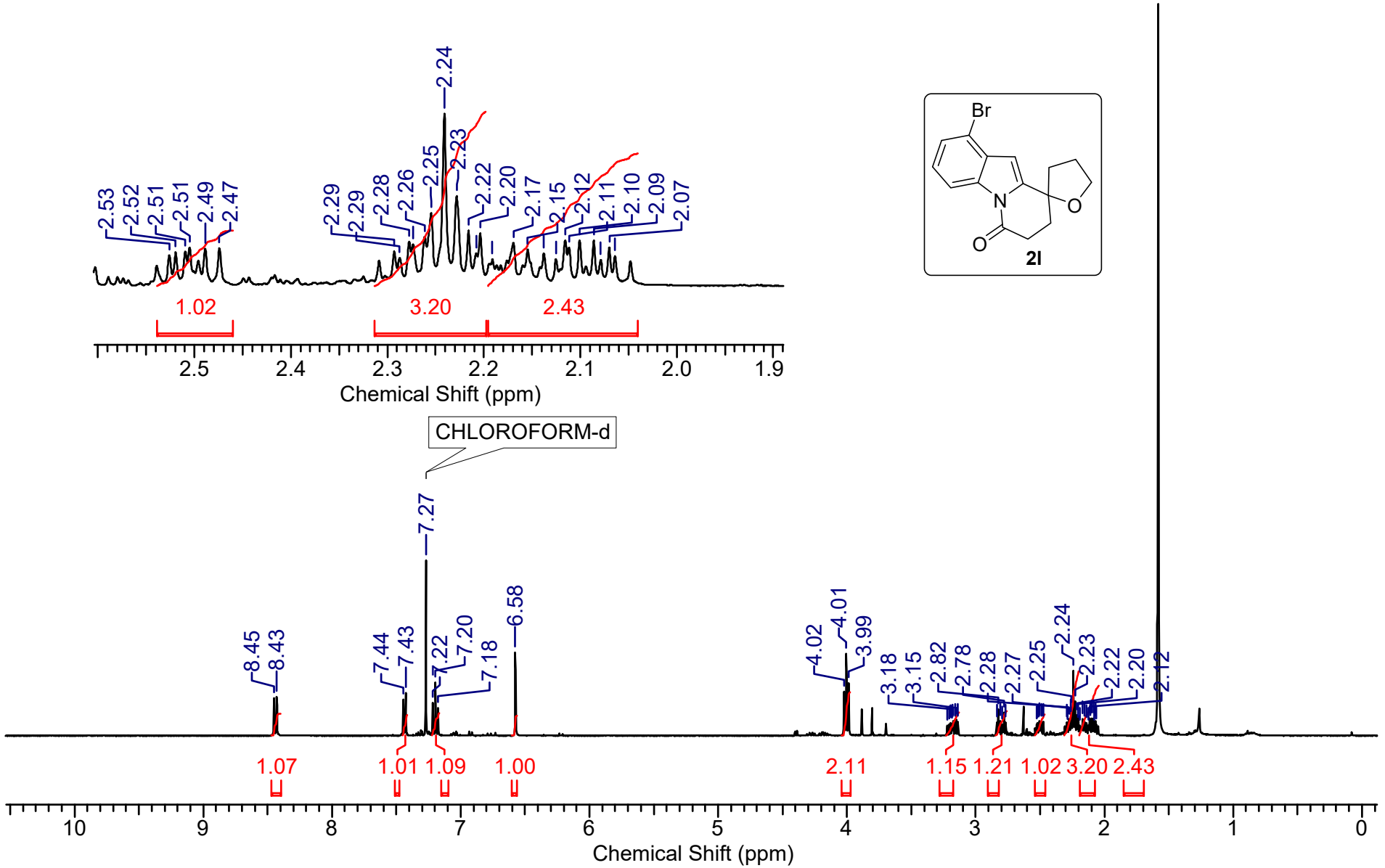

MSH-53 #308 RT: 1.67 AV: 1 NL: 5.54E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
298.0609
R=65506
C 15 H 14 O 2 N Cl Na = 298.0605
1.1909 ppm
276.0785
R=68102
C 15 H 15 O 2 N Cl = 276.0786
-0.4428 ppm
249.1573
R=71307
262.9228
R=65002
270.9775
R=78100
288.9219
R=64902
314.0340
R=63806
321.1363
R=62306
336.9265
R=60202
344.0529
R=59706
352.8968
R=48402
Relative Abundance
m/z
Cl
N
O
O
2j

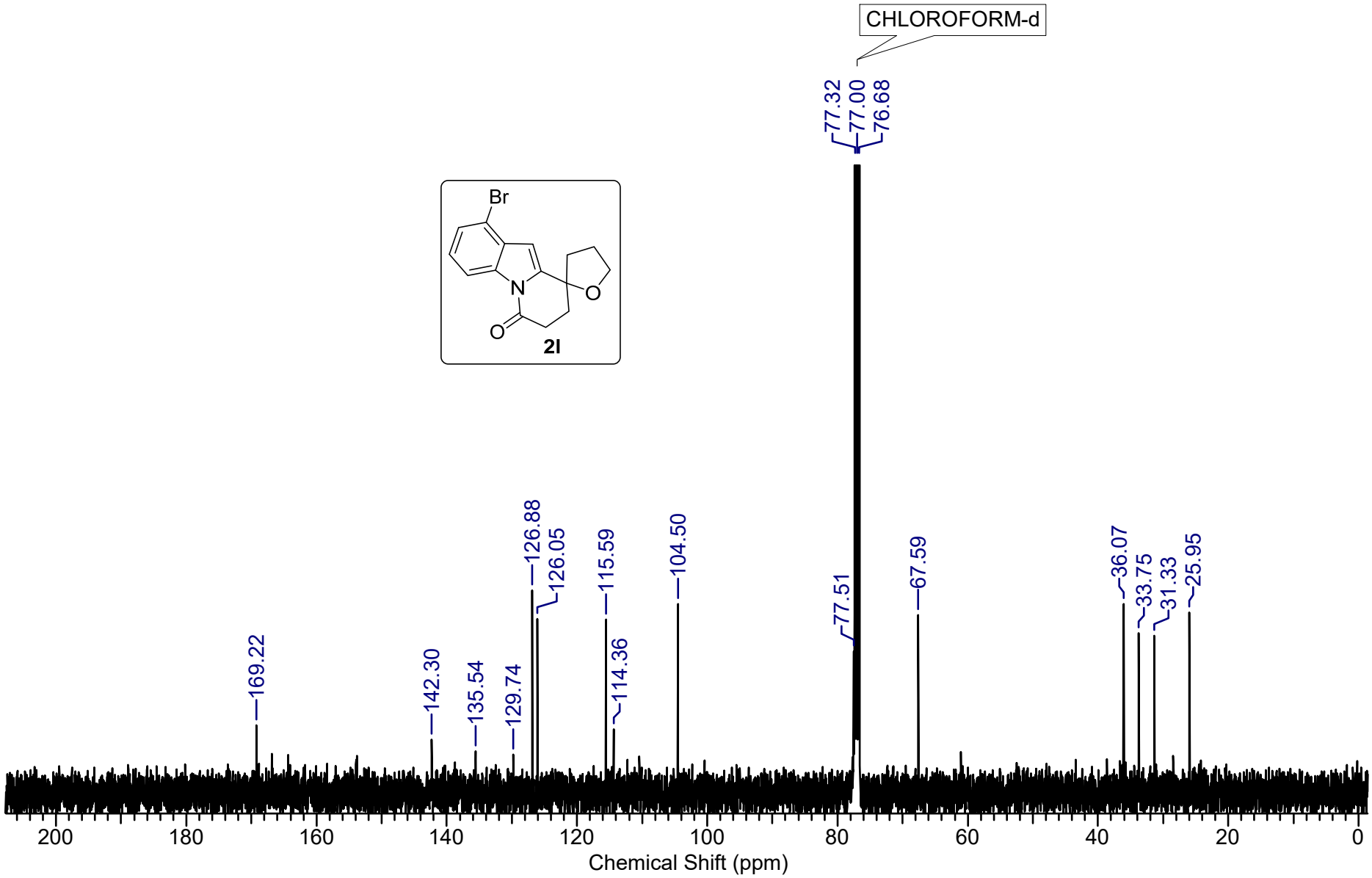

Br
N
O
O
2k
3.18
3.17
3.16
3.15
3.14
3.13
3.11
2.82
2.81
2.80
2.77
2.75
2.47
2.46
2.45
2.45
2.44
2.26
2.24
2.22
2.21
2.20
2.19
2.19
2.15
2.10
1.12
1.11
1.12
3.21
1.17
1.26
3.0
2.5
2.0
Chemical Shift (ppm)
CHLOROFORM-d
8.68
7.40
7.38
7.38
7.27
6.47
4.01
4.00
3.99
3.99
3.97
3.97
3.96
3.95
3.16
3.15
3.14
3.13
2.81
2.77
2.46
2.45
2.24
2.22
2.21
2.20
2.19
2.19
2.10
2.03
1.00
2.16
1.07
2.20
1.12
1.11
1.12
3.21
1.17
1.26
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

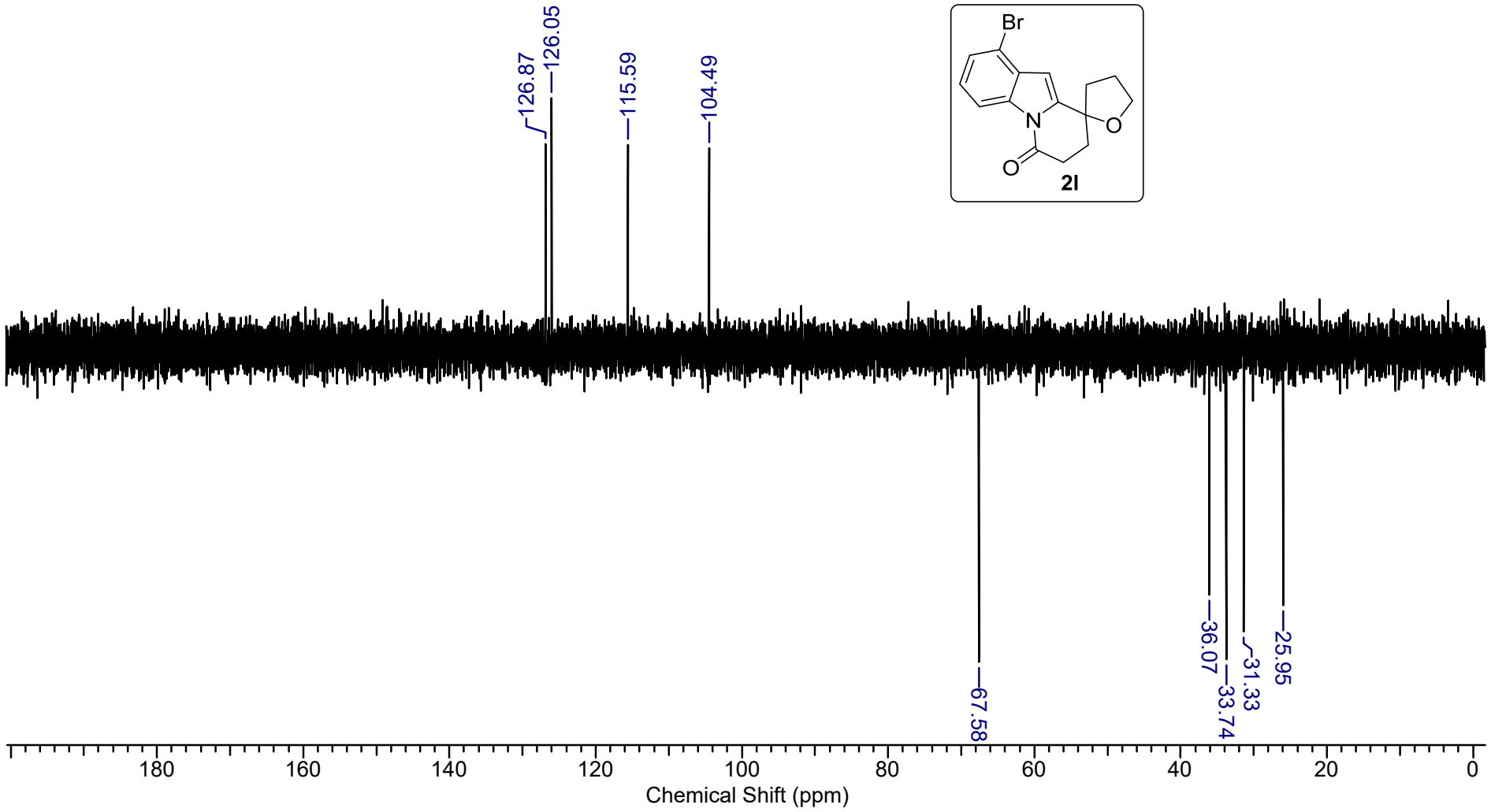

CHLOROFORM-d
77.32
77.00
76.68
142.11
135.76
127.85
127.24
121.48
119.59
118.53
104.37
77.52
Br
N
O
O
2k
136
128
120
112
104
96
88
80
Chemical Shift (ppm)
169.00
142.11
135.76
127.85
127.24
121.48
119.59
118.53
104.37
77.52
67.50
36.07
33.69
31.25
25.90
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


127.23
121.48
119.59
104.36
Br
N
O
O
2k
67.50
36.06
33.68
31.25
25.90
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

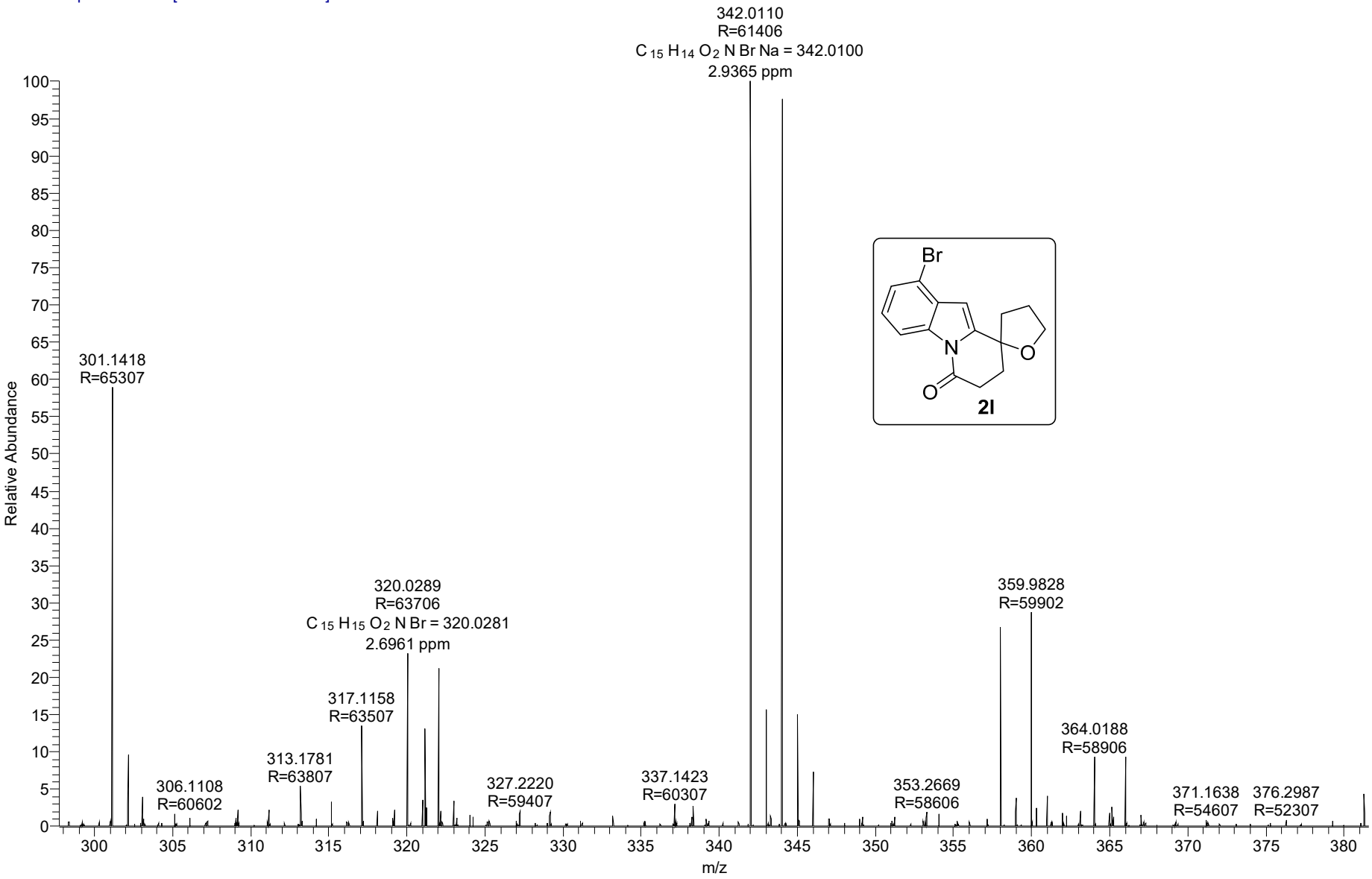

MSH-41 #333 RT: 1.81 AV: 1 NL: 7.76E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
320.03
C 15 H 15 O 2 N Br
322.02
342.01
C 15 H 14 O 2 N Br Na
323.03
345.01
297.08
359.98
301.14
319.14
339.05
357.19
305.16
335.22
295.15
311.15
315.16
324.03
329.16
349.18
353.07
360.98
367.08
376.23
381.30
388.25
Relative Abundance
m/z
Br
N
O
O
2k

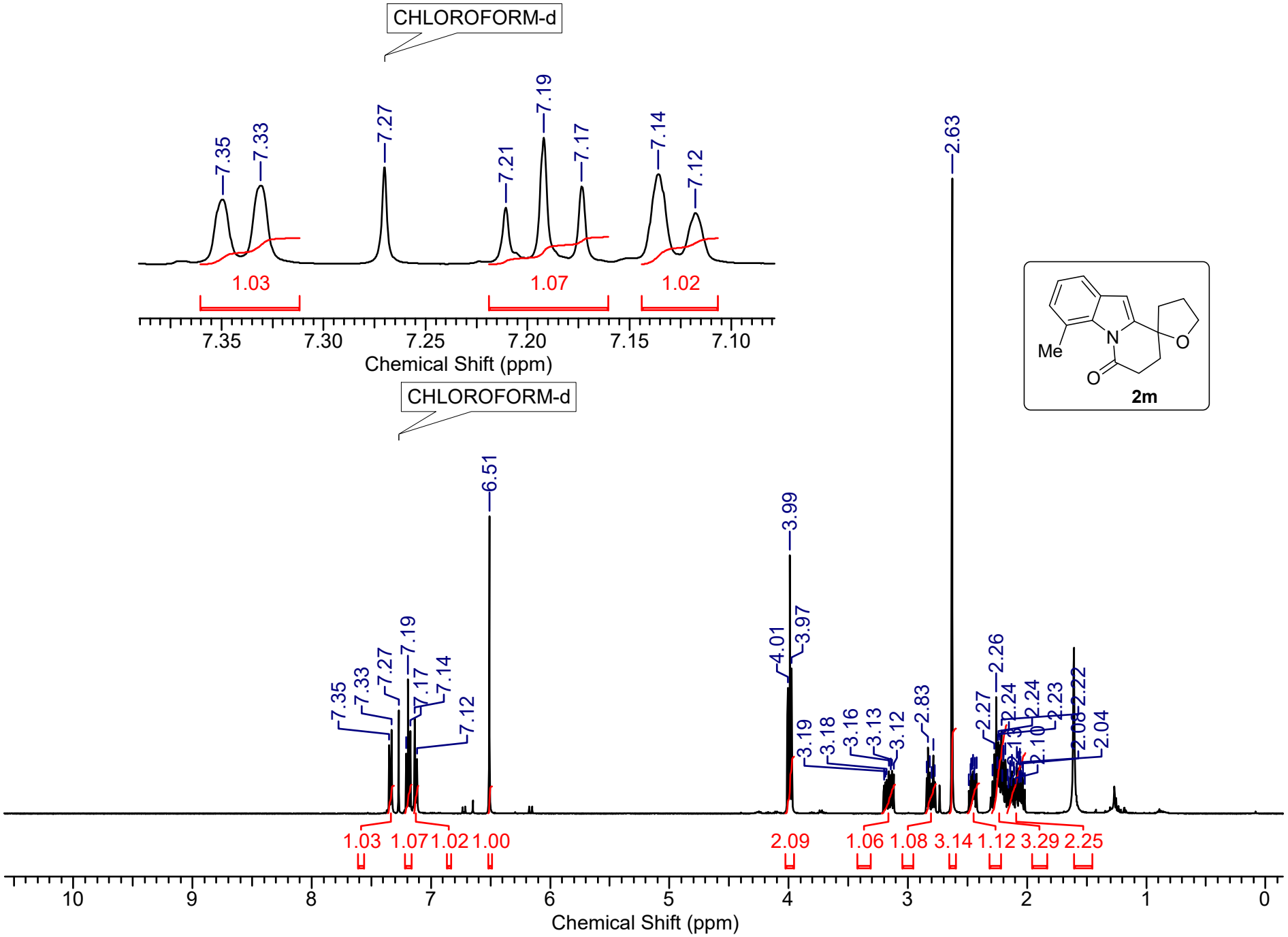

Br
N
O
O
2l
CHLOROFORM-d
Chemical Shift (ppm)
Chemical Shift (ppm)

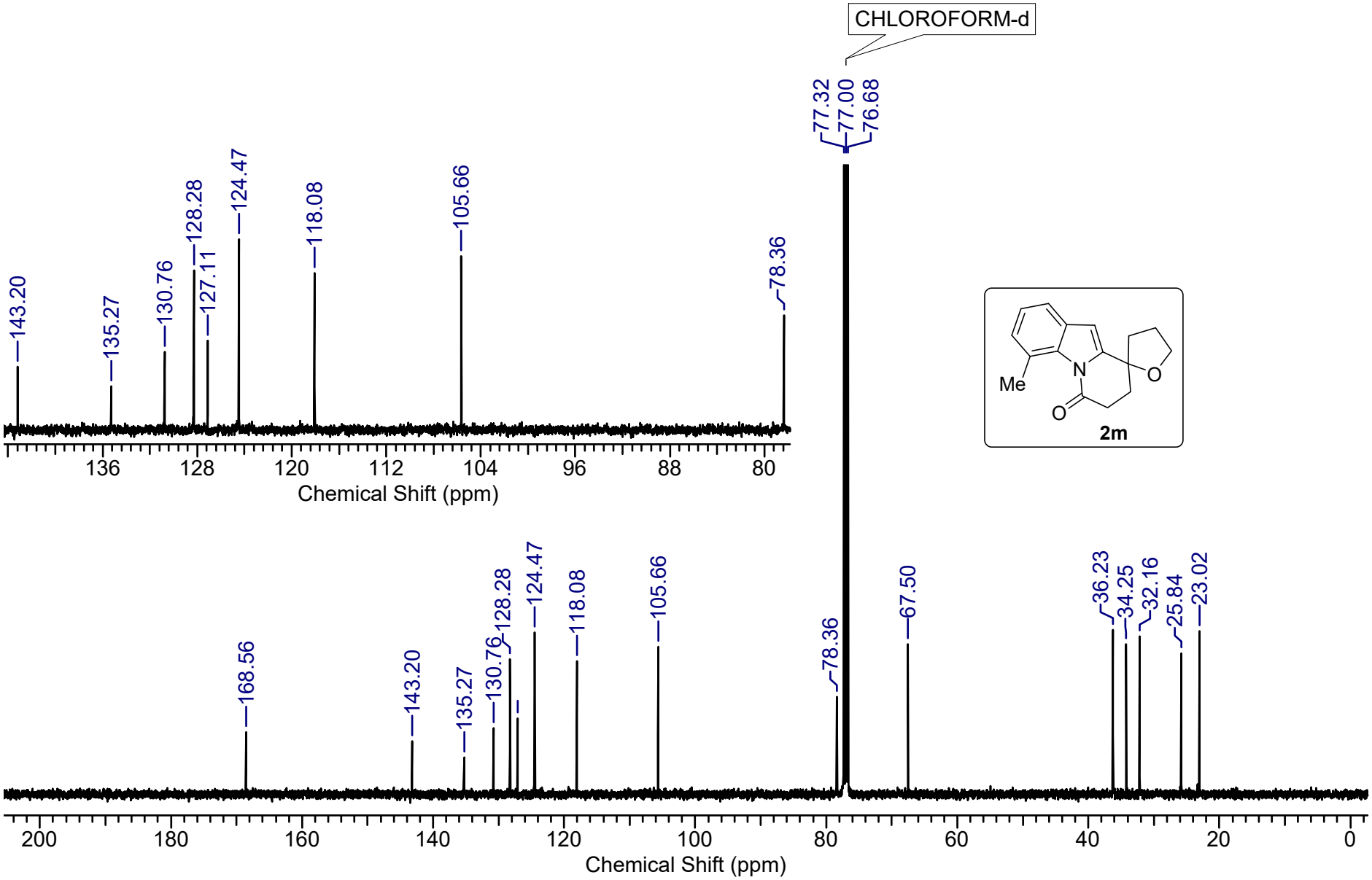

CHLOROFORM-d
77.32
77.00
76.68
Br
N
O
O
2l
169.22
142.30
135.54
129.74
126.88
126.05
115.59
114.36
104.50
77.51
67.59
36.07
33.75
31.33
25.95
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

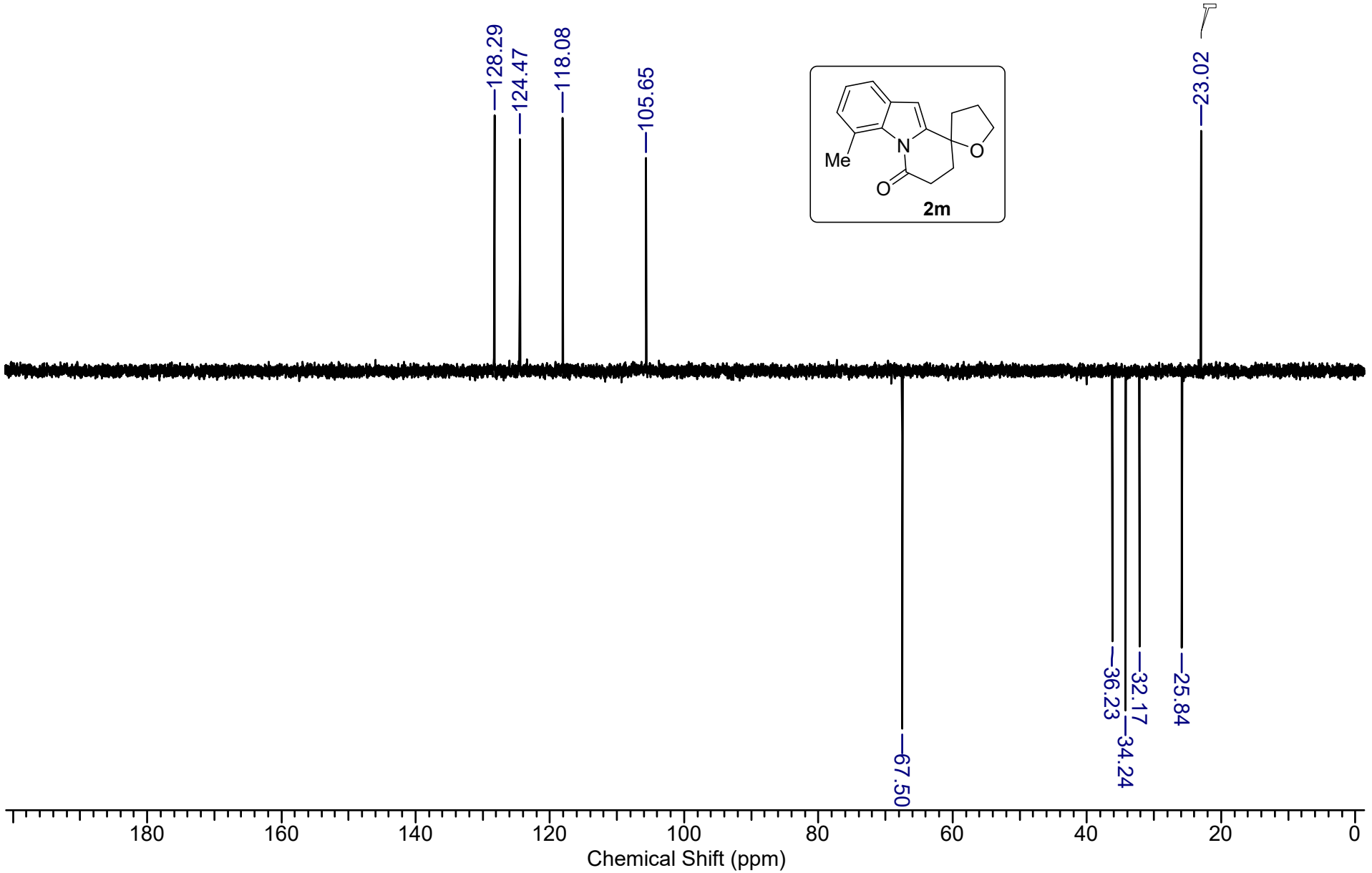

126.87
126.05
115.59
104.49
67.58
36.07
33.74
31.33
25.95
Br
N
O
O
2l
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

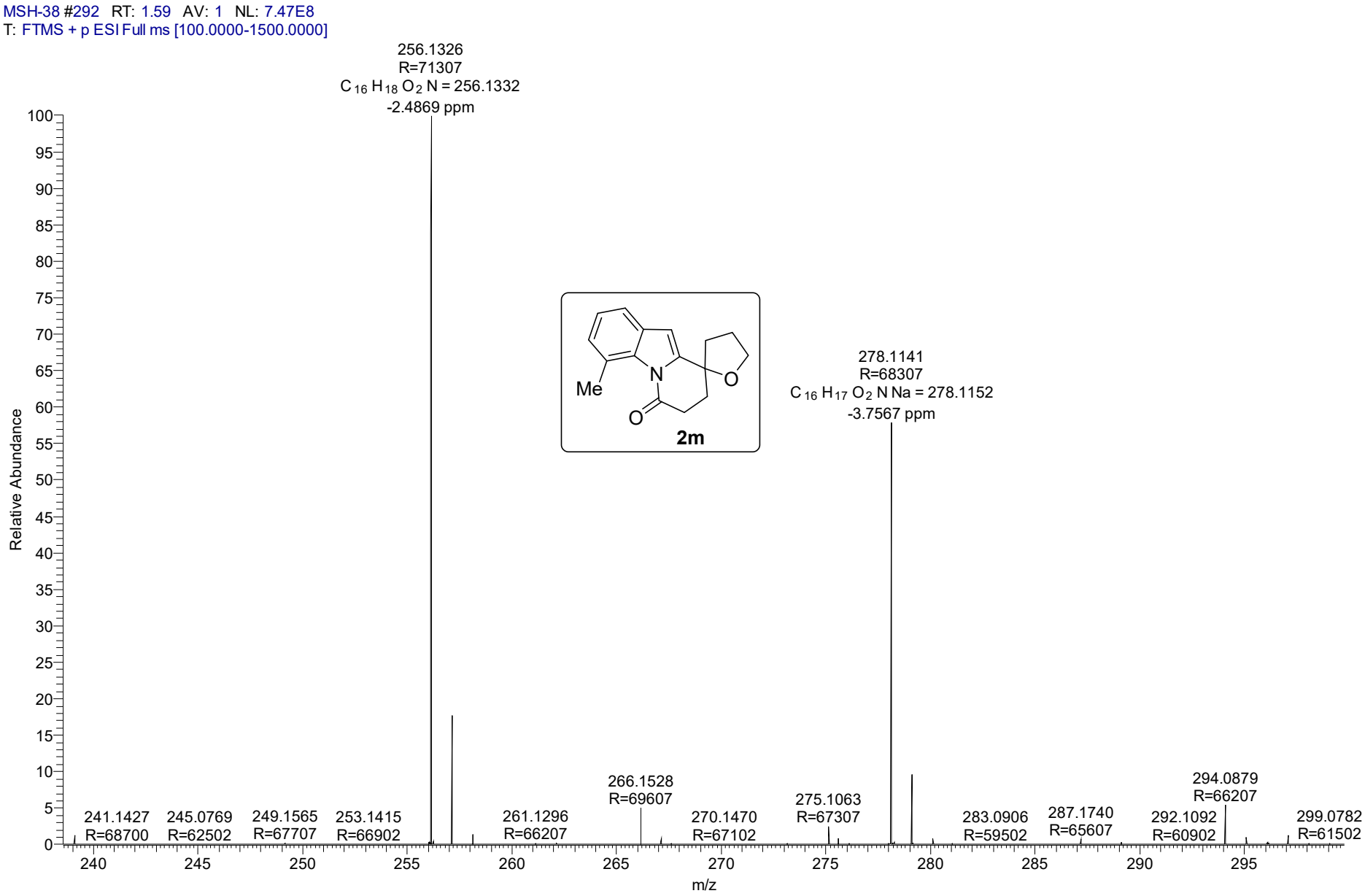
MS-11a #313 RT: 1.68 AV: 1 NL: 2.30E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

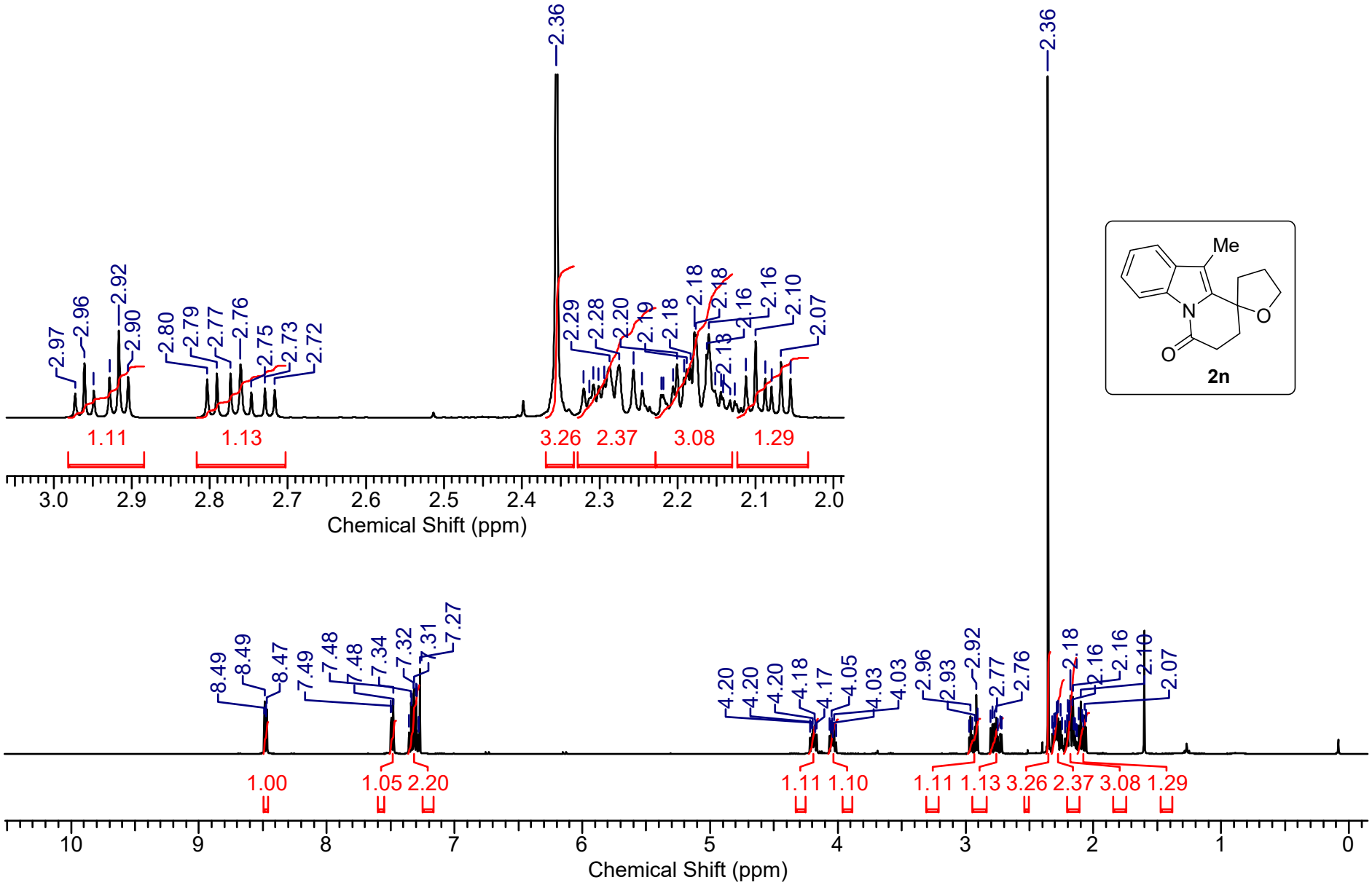


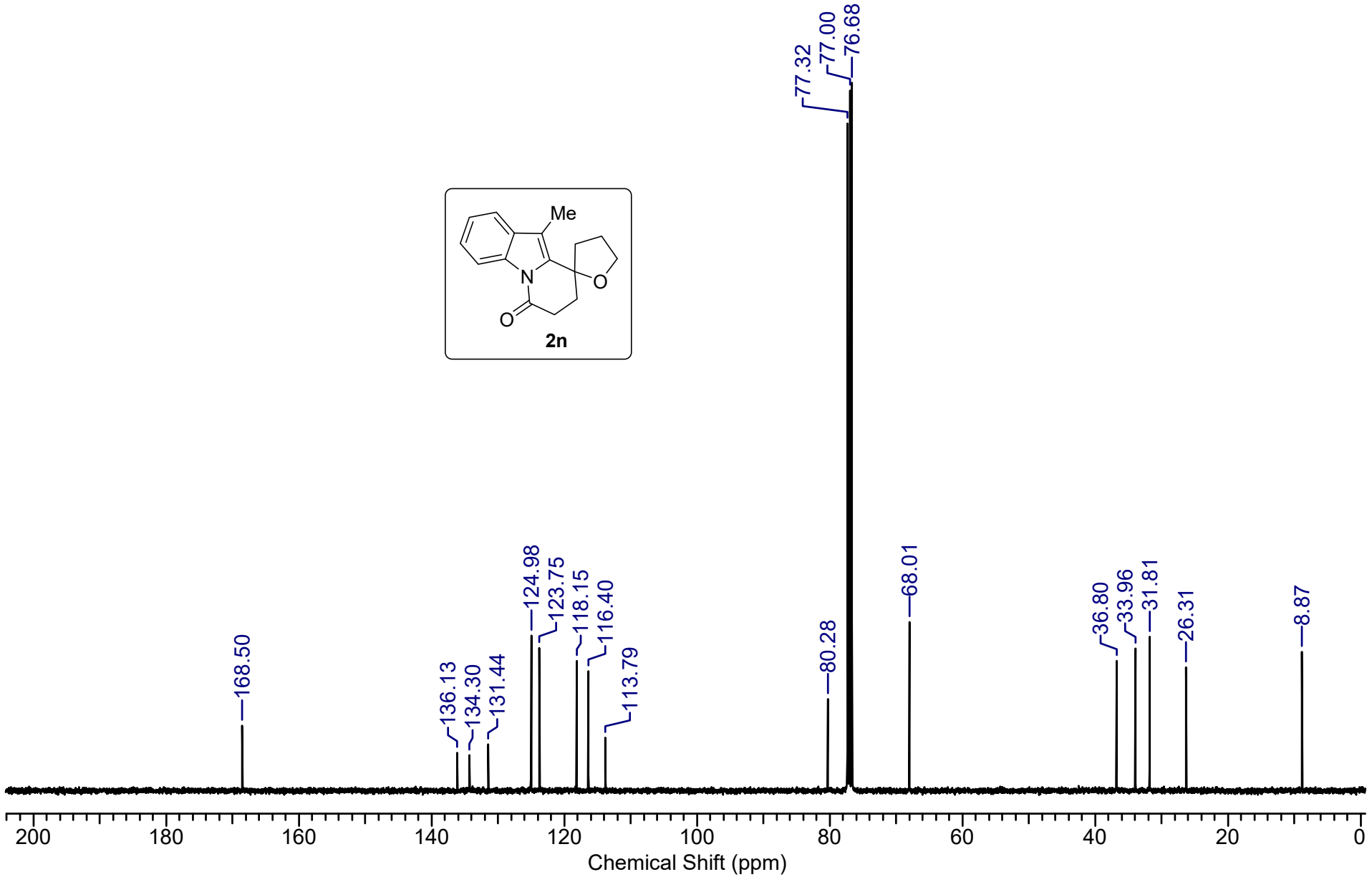

CHLOROFORM-d
7.35
7.33
7.27
7.21
7.19
7.17
7.14
7.12
1.03
1.07
1.02
7.35 7.30 7.25 7.20 7.15 7.10
Chemical Shift (ppm)
CHLOROFORM-d
6.51
3.99
4.01
3.97
3.19
3.18
3.16
3.13
3.12
2.83
2.63
2.27
2.26
2.24
2.24
2.23
2.22
2.10
2.08
2.04
1.03 1.07 1.02 1.00
2.09 1.06 1.08 3.14 1.12 3.29 2.25
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)
Me
N
O
O
2m

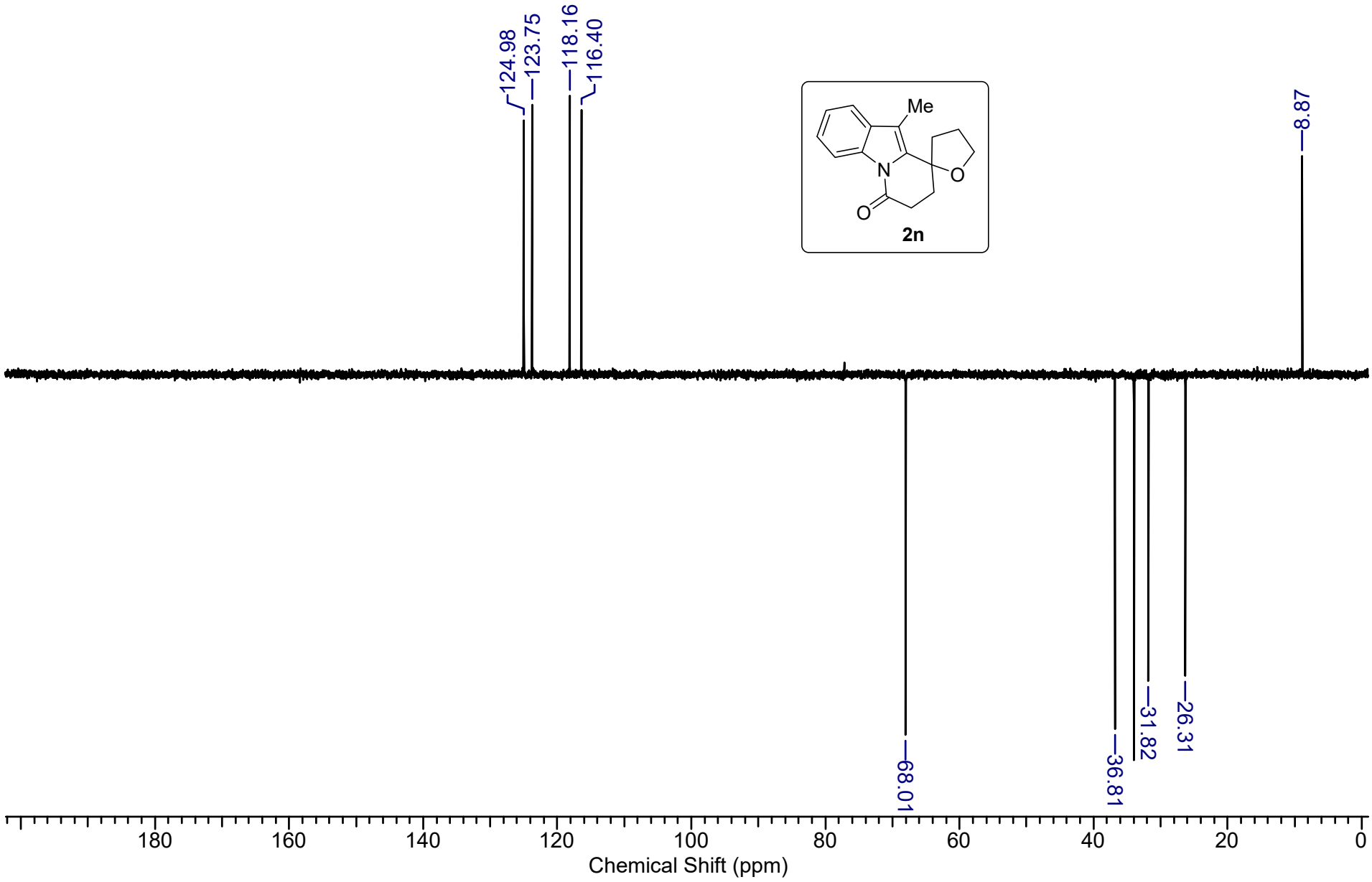

CHLOROFORM-d
77.32
77.00
76.68
143.20
135.27
130.76
128.28
127.11
124.47
118.08
105.66
78.36
Me
N
O
O
2m
136
128
120
112
104
96
88
80
Chemical Shift (ppm)
168.56
143.20
135.27
130.76
128.28
124.47
118.08
105.66
78.36
67.50
36.23
34.25
32.16
25.84
23.02
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


128.29
124.47
118.08
105.65
23.02
67.50
36.23
34.24
32.17
25.84
Me
N
O
O
2m
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

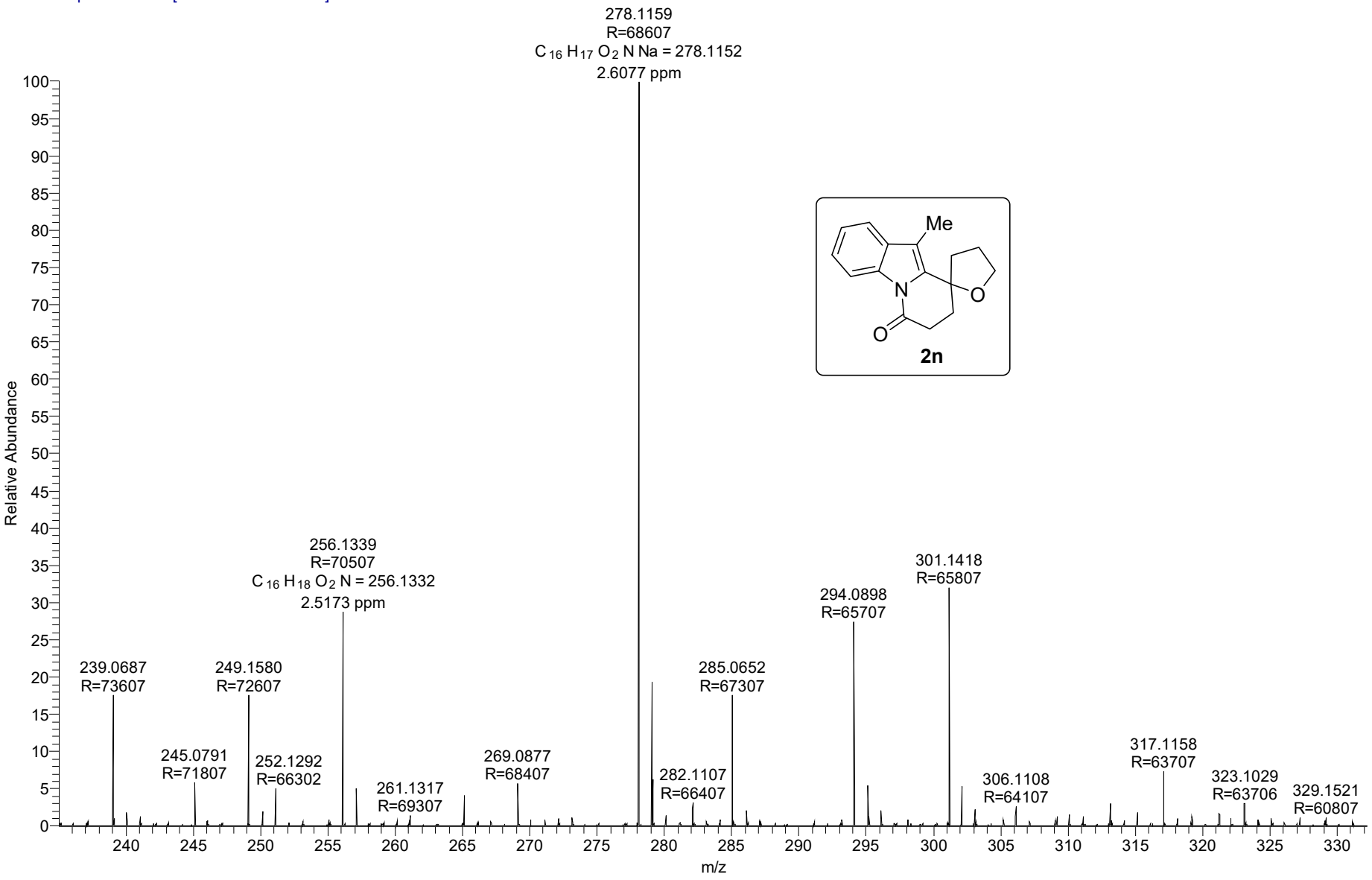

MSH-38 #292 RT: 1.59 AV: 1 NL: 7.47E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
256.1326
R=71307
C 16 H 18 O 2 N = 256.1332
-2.4869 ppm
278.1141
R=68307
C 16 H 17 O 2 N Na = 278.1152
-3.7567 ppm
Me
N
O
O
2m
Relative Abundance
m/z
241.1427 R=68700
245.0769 R=62502
249.1565 R=67707
253.1415 R=66902
261.1296 R=66207
266.1528 R=69607
270.1470 R=67102
275.1063 R=67307
283.0906 R=59502
287.1740 R=65607
292.1092 R=60902
294.0879 R=66207
299.0782 R=61502

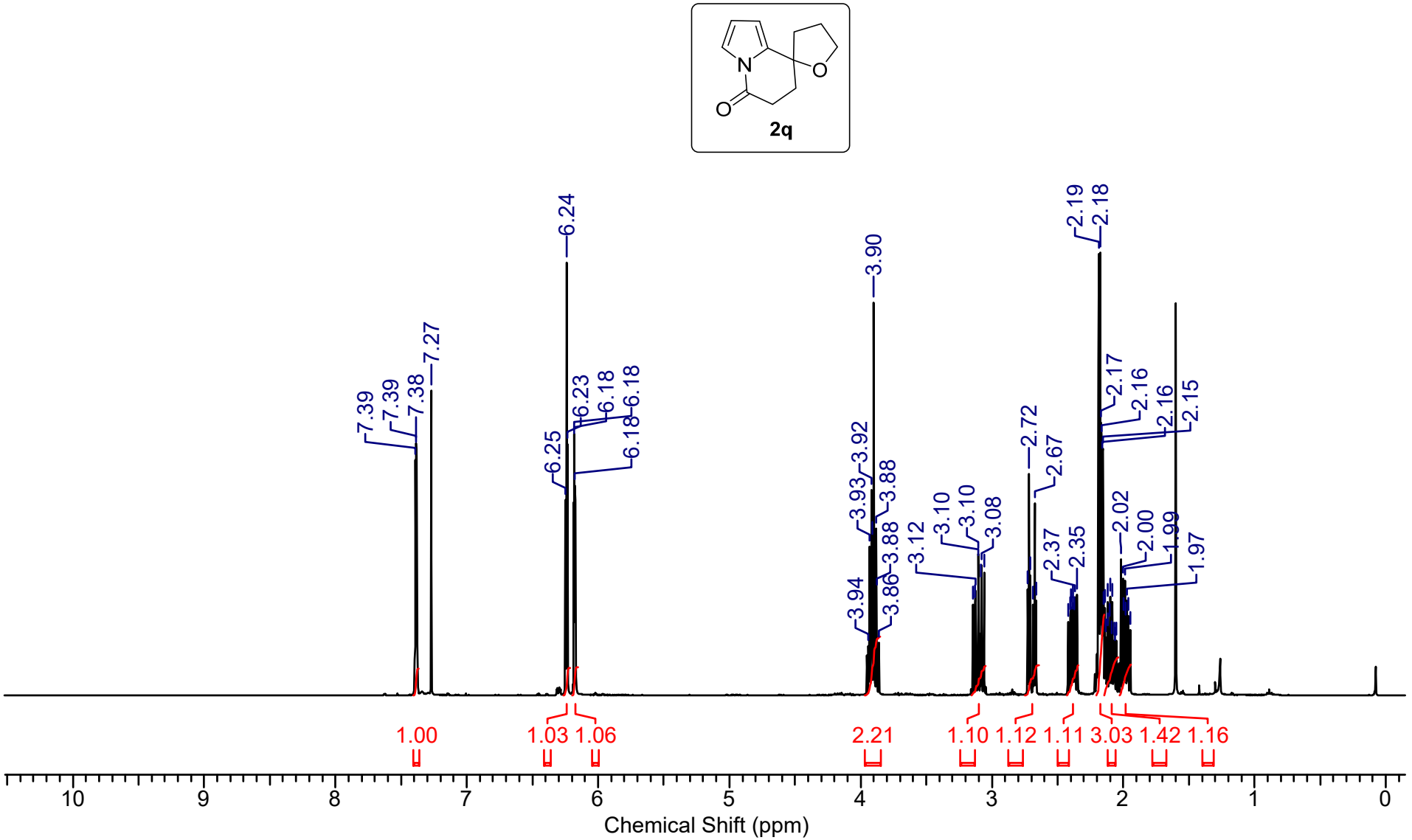

2.36
2.97
2.96
2.92
2.90
2.80
2.79
2.77
2.76
2.75
2.73
2.72
2.29
2.28
2.20
2.19
2.18
2.18
2.18
2.16
2.16
2.10
2.07
1.11
1.13
3.26
2.37
3.08
1.29
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
Chemical Shift (ppm)
Me
N
O
O
2n
8.49
8.49
8.47
7.49
7.48
7.48
7.34
7.32
7.31
7.27
4.20
4.20
4.20
4.18
4.17
4.05
4.03
4.03
2.96
2.93
2.92
2.77
2.76
1.00
1.05
2.20
1.11
1.10
1.11
1.13
3.26
2.37
3.08
1.29
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

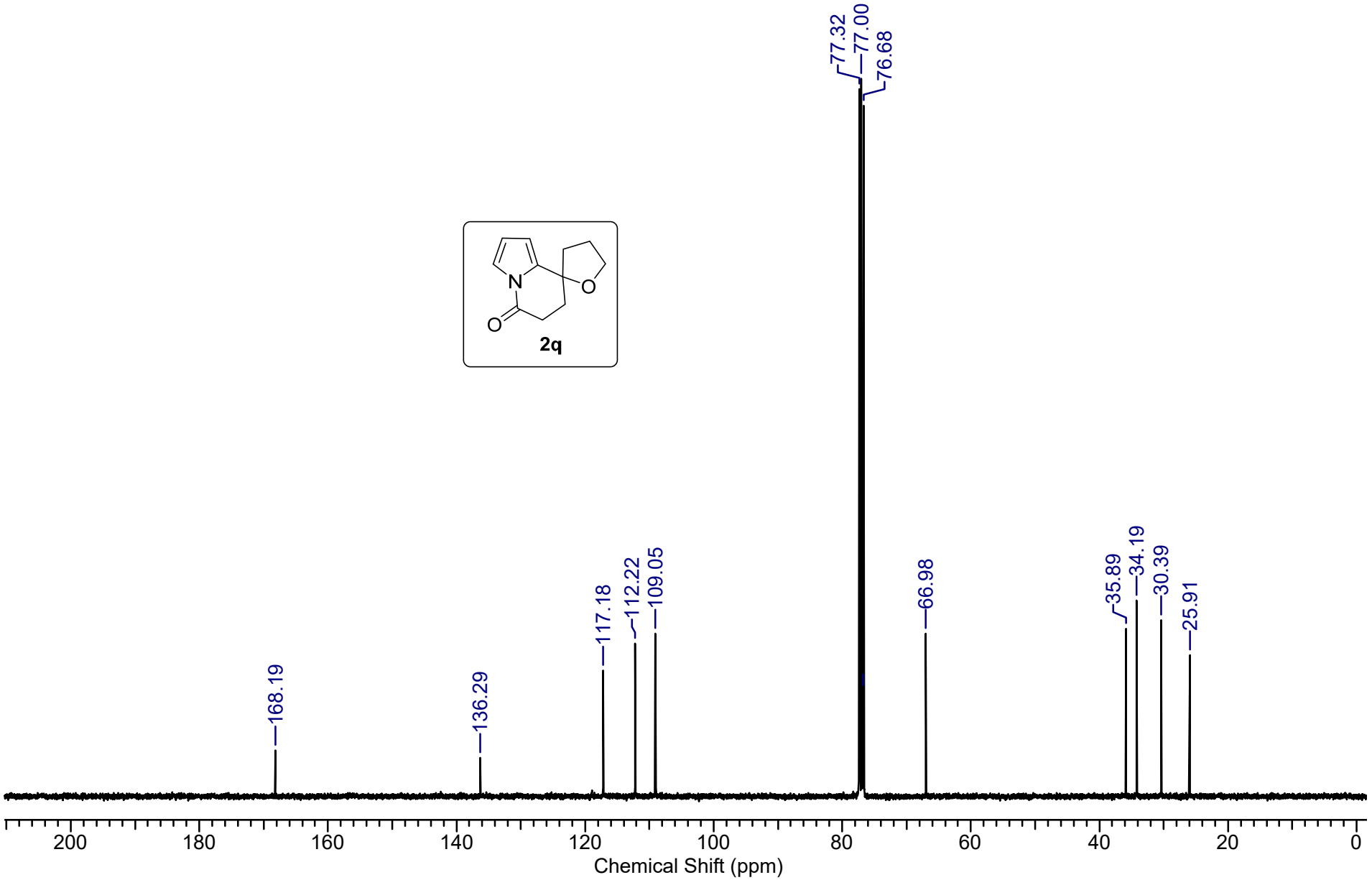

Me
N
O
O
2n
77.32
77.00
76.68
168.50
136.13
134.30
131.44
124.98
123.75
118.15
116.40
113.79
80.28
68.01
36.80
33.96
31.81
26.31
8.87
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

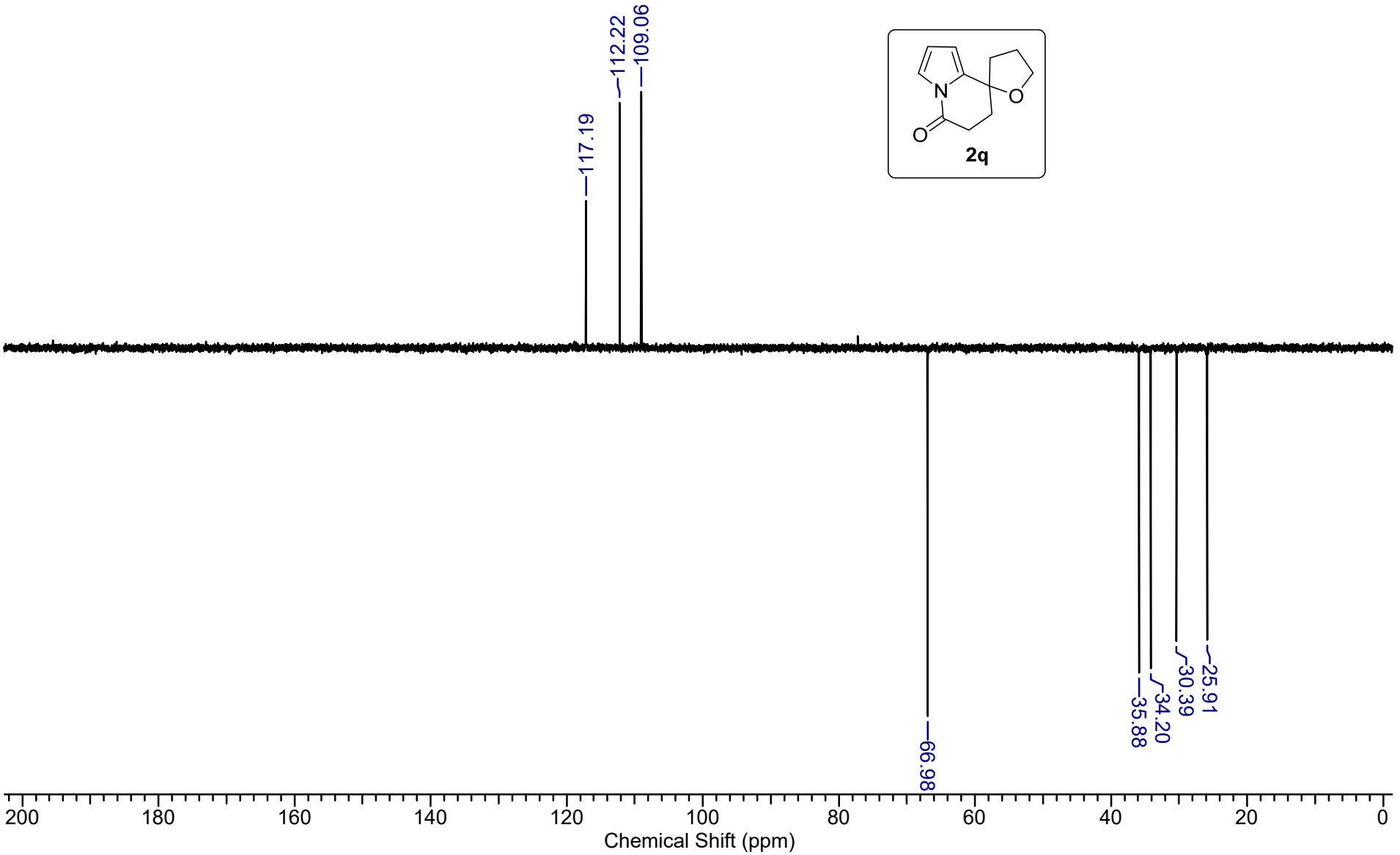

124.98
123.75
118.16
116.40
8.87
68.01
36.81
31.82
26.31
Me
N
O
O
2n
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-10a #312 RT: 1.67 AV: 1 NL: 4.32E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

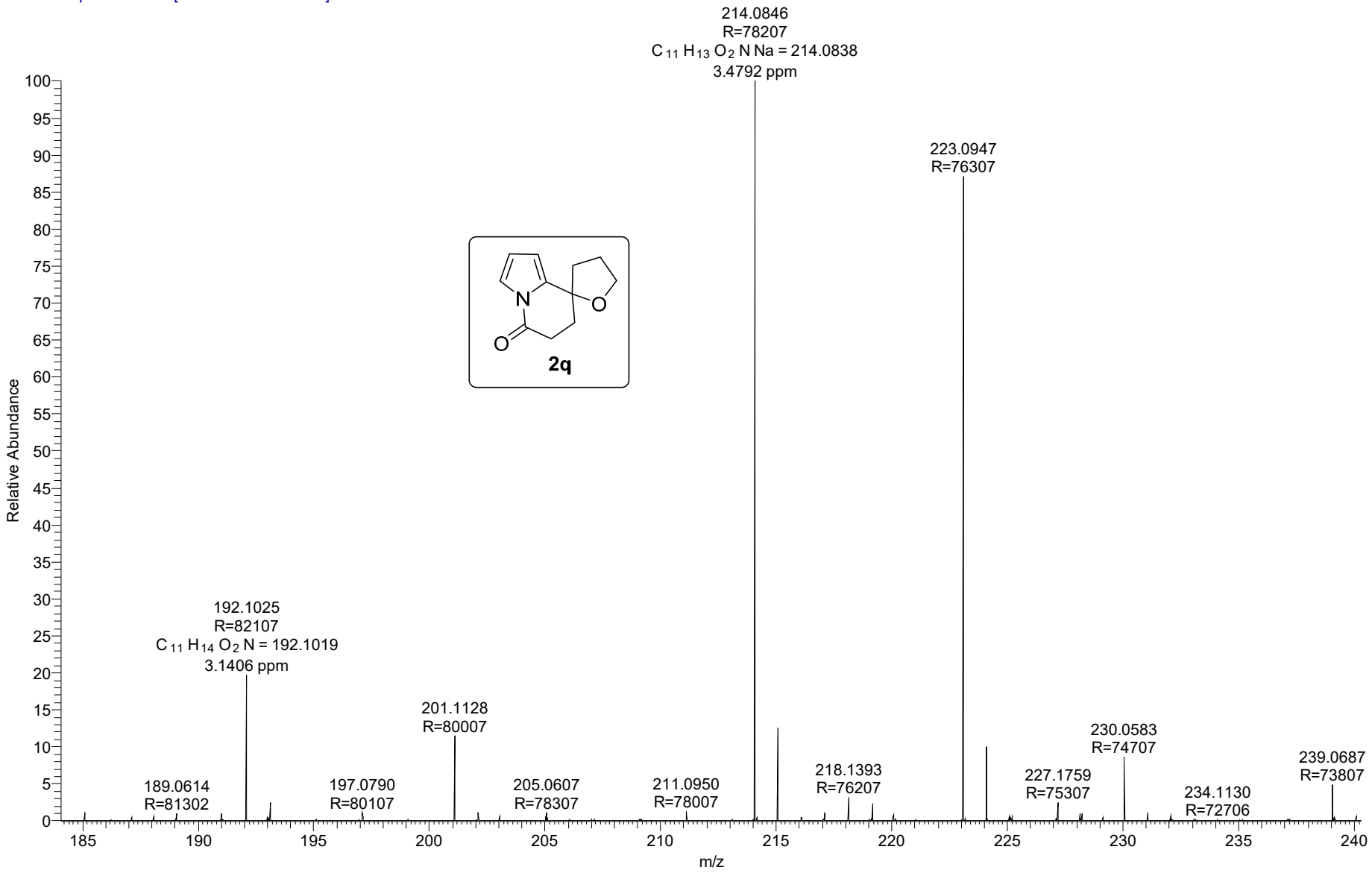


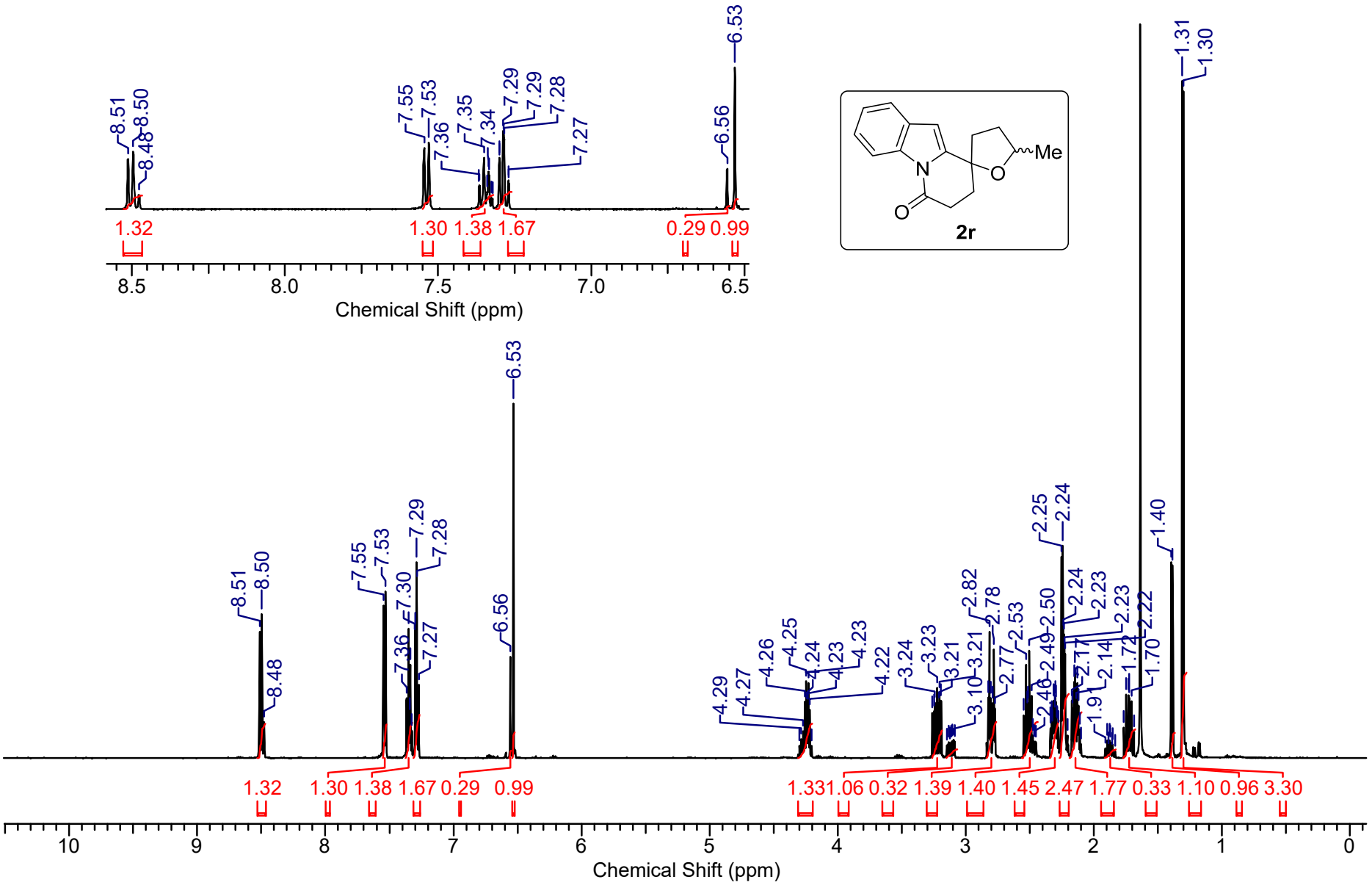

N
O
O
2q
7.39
7.39
7.38
7.27
6.25
6.24
6.23
6.18
6.18
6.18
3.94
3.93
3.92
3.90
3.88
3.88
3.86
3.12
3.10
3.10
3.08
2.72
2.67
2.37
2.35
2.19
2.18
2.17
2.16
2.16
2.15
2.02
2.00
1.99
1.97
1.00
1.03
1.06
2.21
1.10
1.12
1.11
3.03
1.42
1.16
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

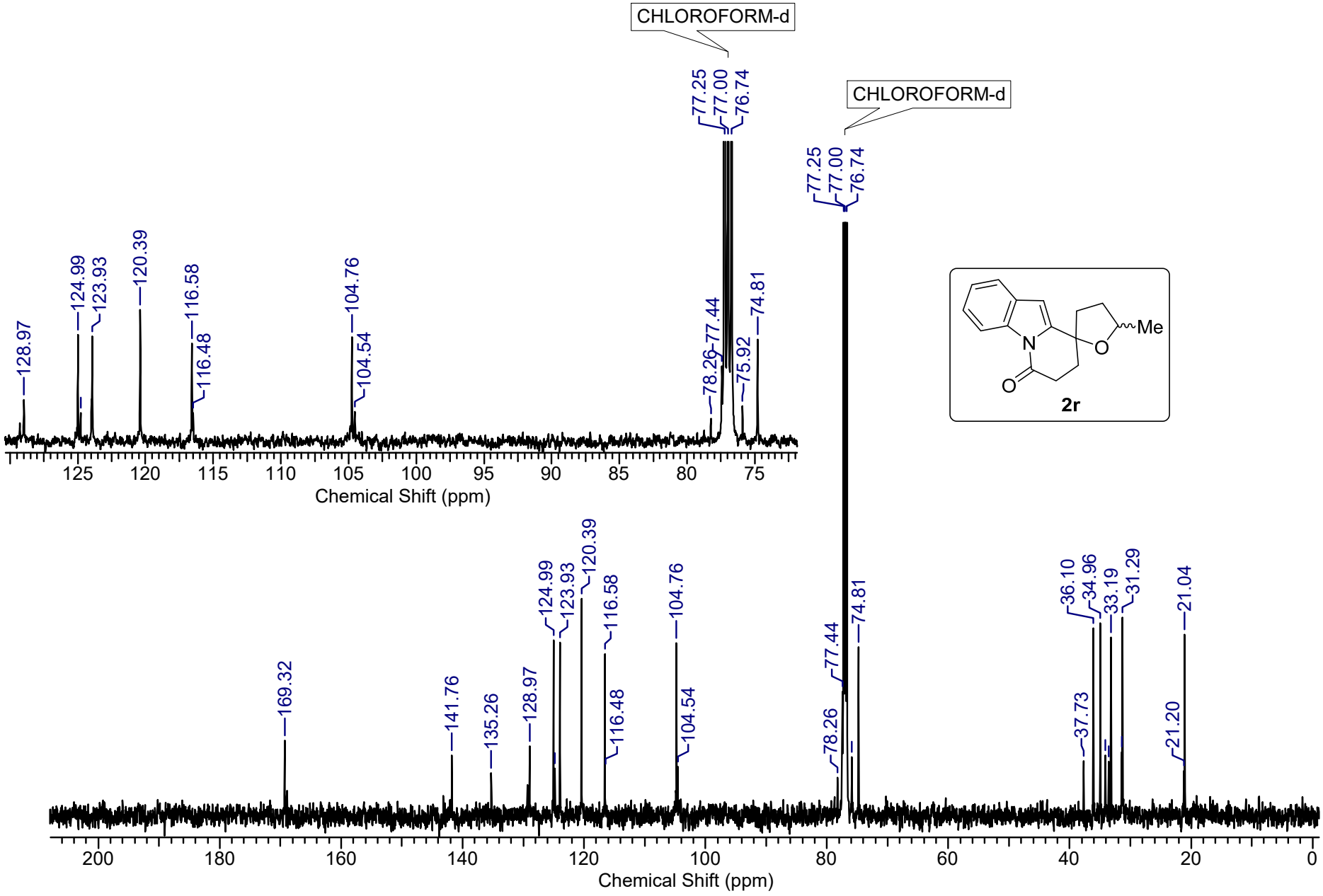

77.32
77.00
76.68
168.19
136.29
117.18
112.22
109.05
66.98
35.89
34.19
30.39
25.91
N
O
O
2q
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

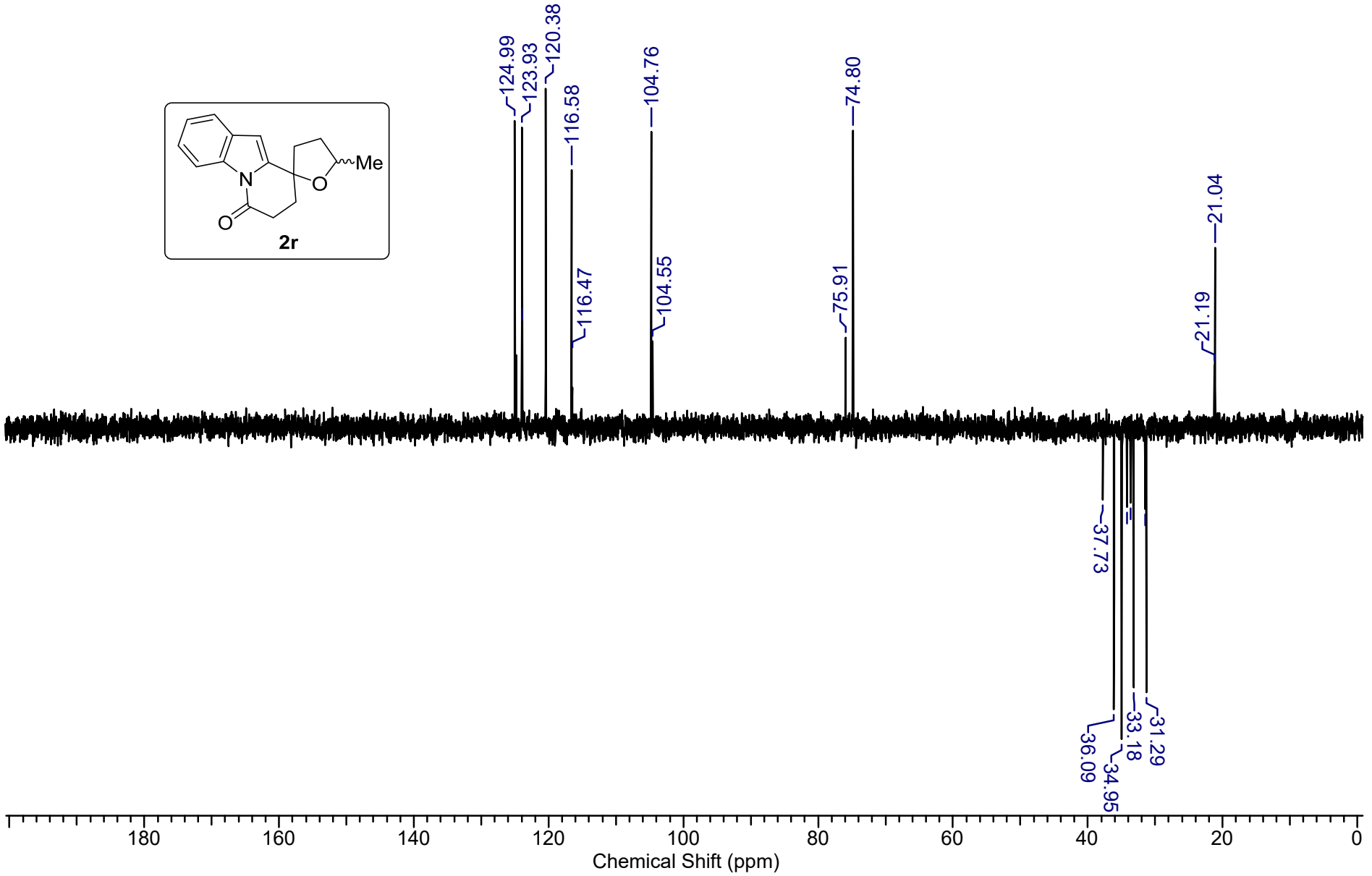

117.19
112.22
109.06
N
O
O
2q
66.98
35.88
34.20
30.39
25.91
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

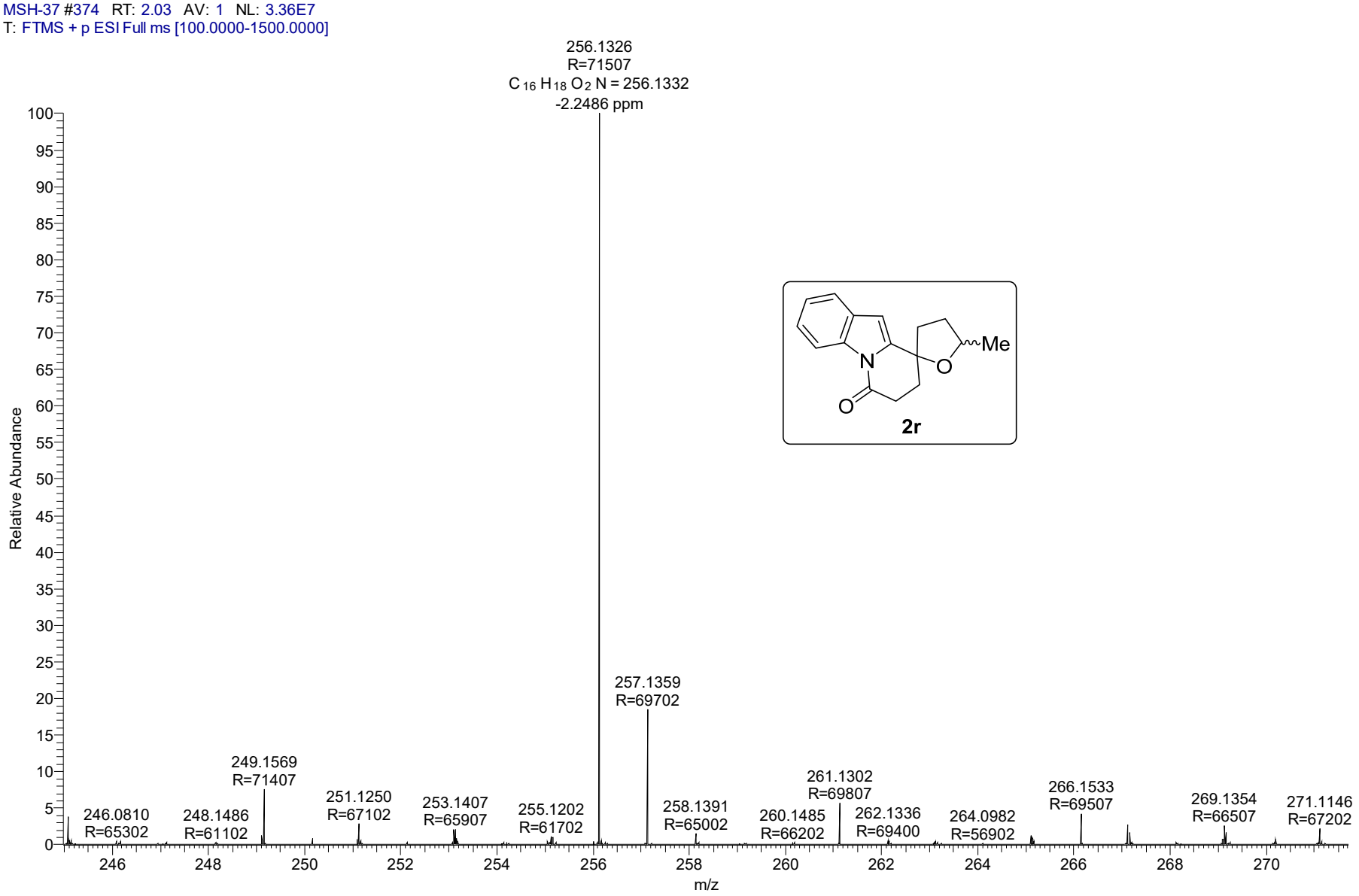
MS-14a #276 RT: 1.48 AV: 1 NL: 1.26E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

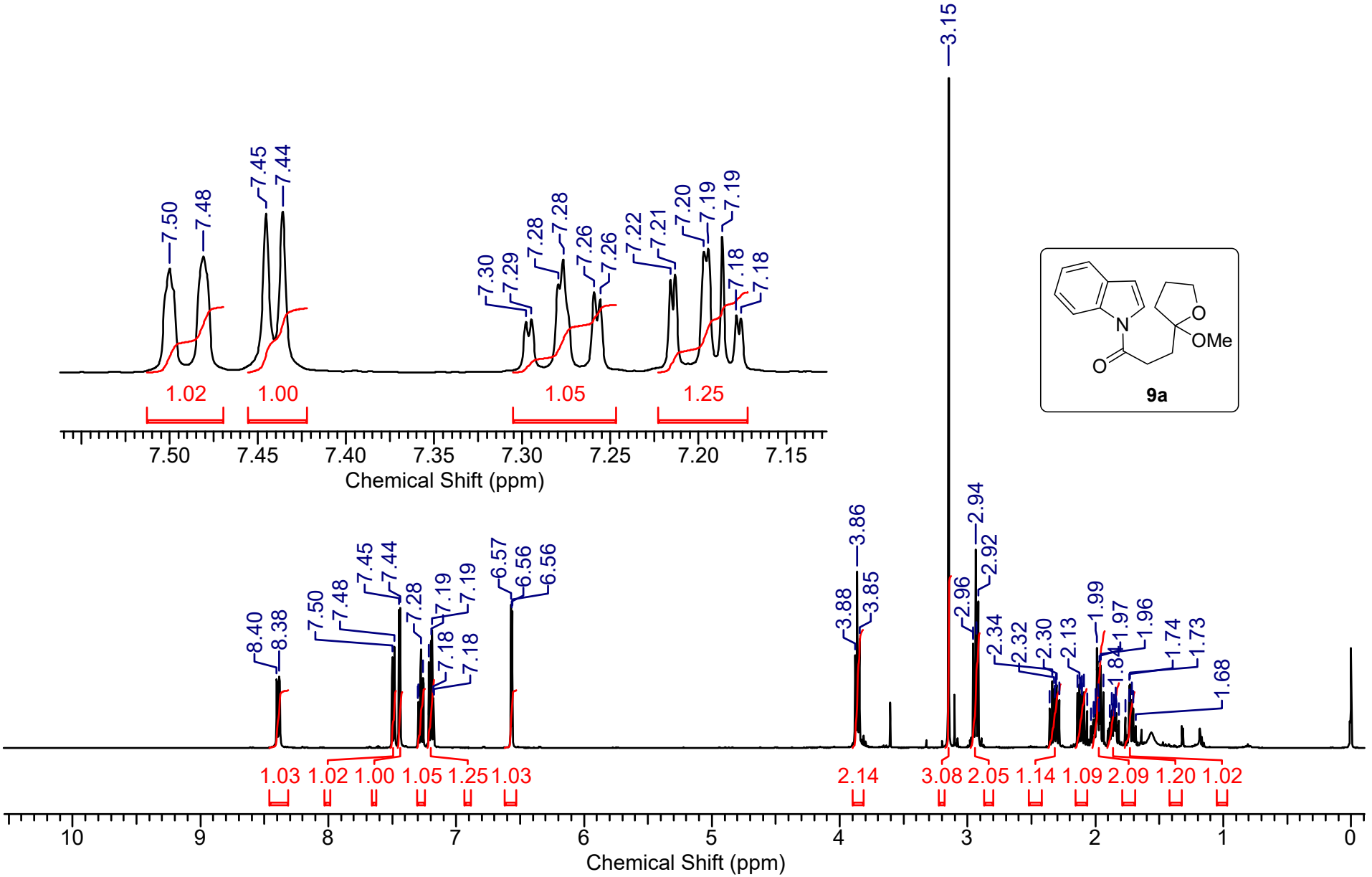


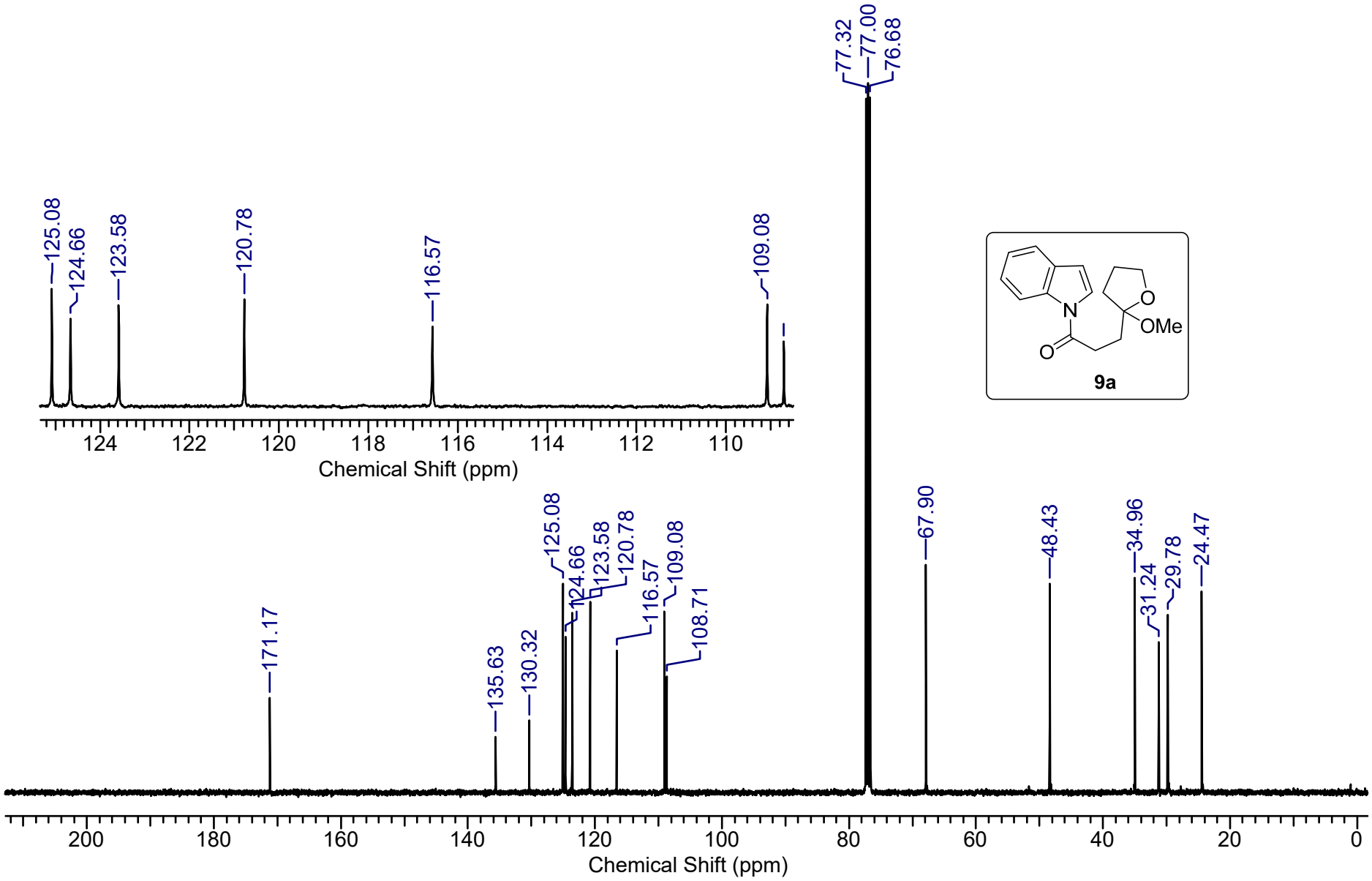

8.51
8.50
8.48
7.55
7.53
7.36
7.35
7.34
7.29
7.29
7.28
7.27
6.56
6.53
1.32
1.30
1.38
1.67
0.29
0.99
8.5
8.0
7.5
7.0
6.5
Chemical Shift (ppm)
Me
N
O
O
2r
1.31
1.30
1.40
2.25
2.24
2.24
2.23
2.23
2.22
2.50
2.49
2.82
2.78
2.77
2.53
2.71
2.14
1.91
1.74
1.72
1.70
4.29
4.27
4.26
4.25
4.24
4.23
4.23
4.22
3.24
3.23
3.21
3.21
3.10
2.48
1.32
1.30
1.38
1.67
0.29
0.99
1.33
1.06
0.32
1.39
1.40
1.45
2.47
1.77
0.33
1.10
0.96
3.30
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

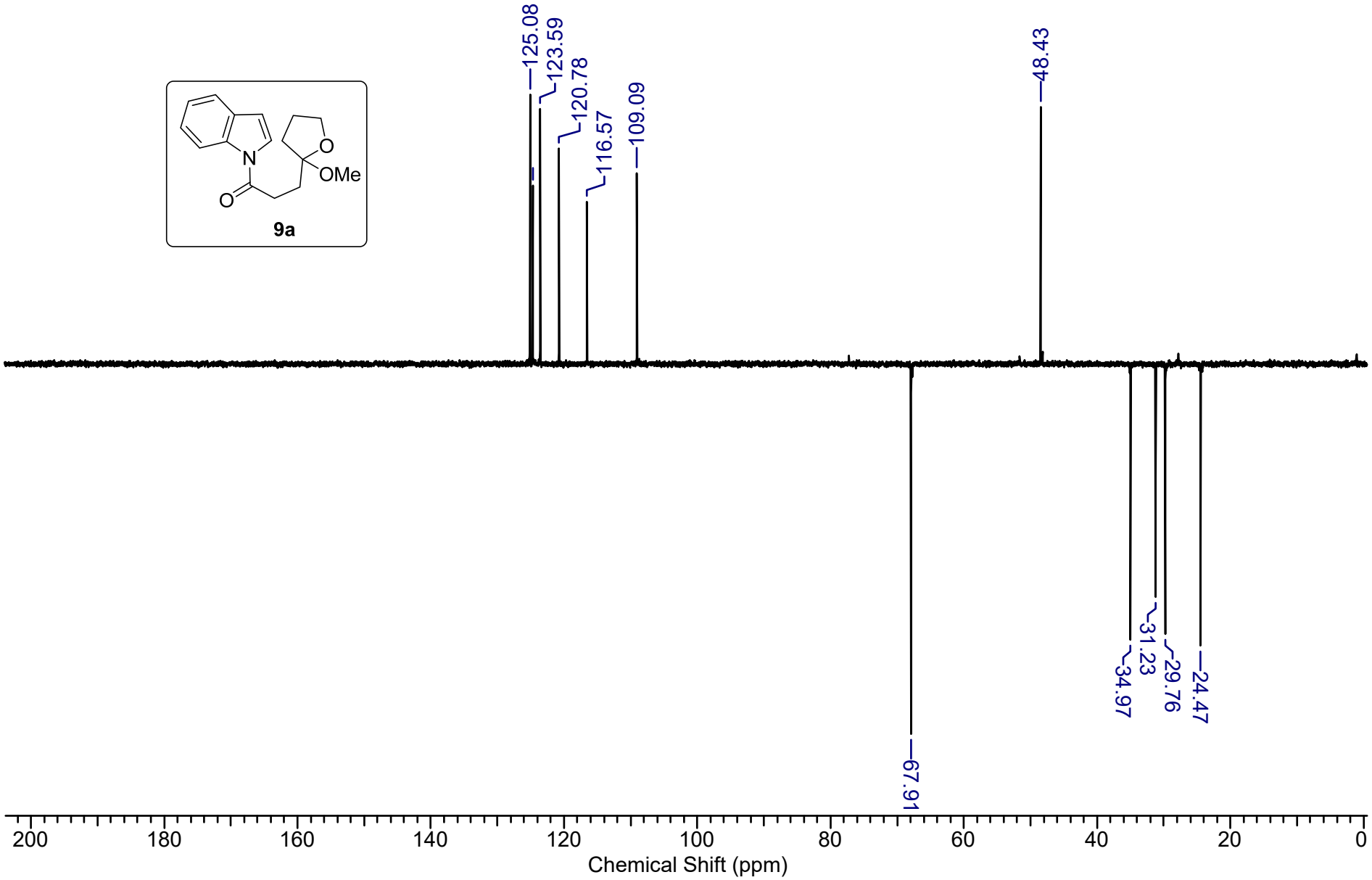

CHLOROFORM-d
77.25
77.00
76.74
CHLOROFORM-d
77.25
77.00
76.74
128.97
124.99
123.93
120.39
116.58
116.48
104.76
104.54
78.26
77.44
74.81
75.92
Me
N
O
O
2r
Chemical Shift (ppm)
169.32
141.76
135.26
128.97
124.99
123.93
120.39
116.58
116.48
104.76
104.54
78.26
77.44
74.81
36.10
34.96
33.19
31.29
37.73
21.04
21.20
Chemical Shift (ppm)

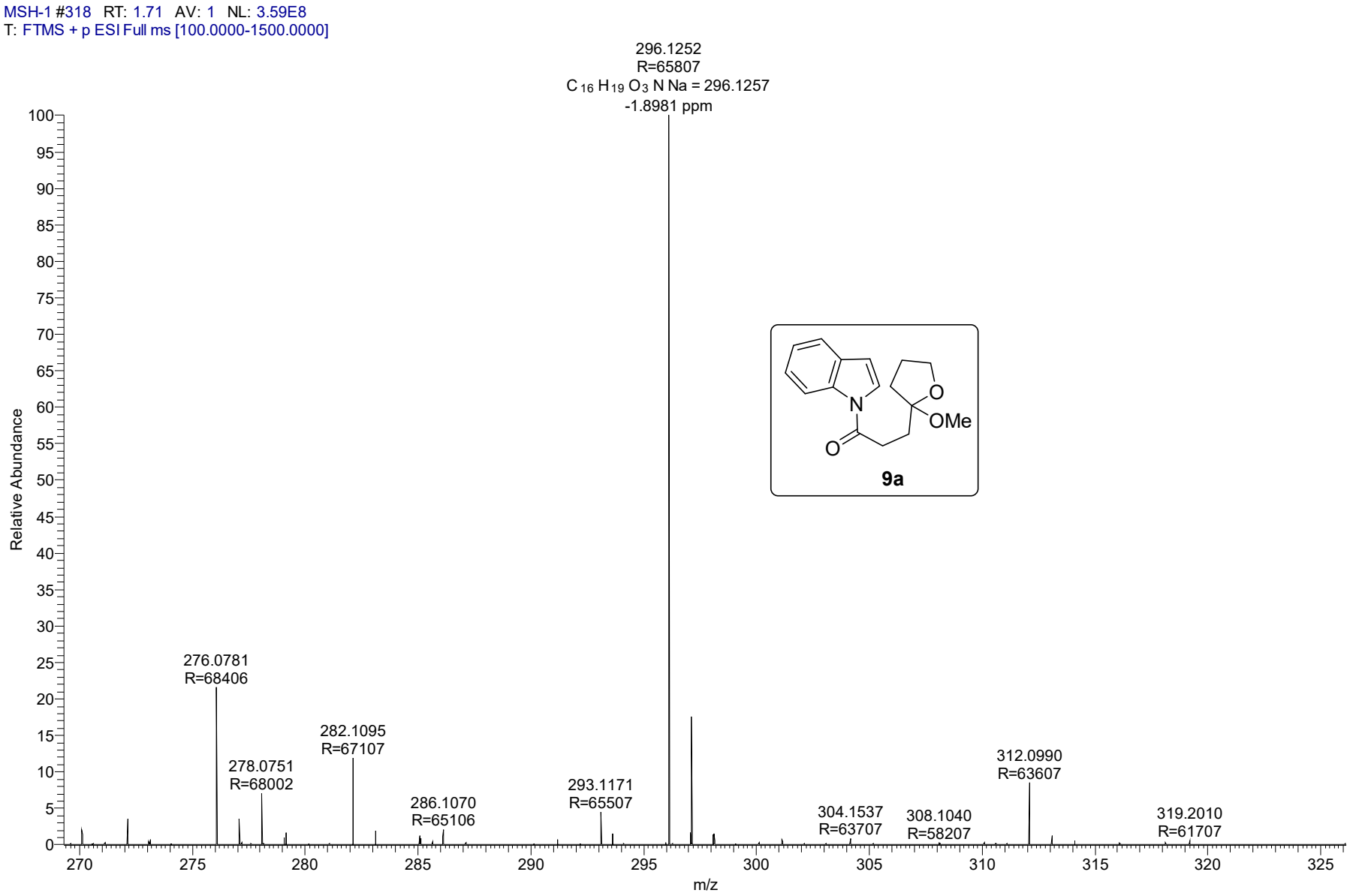

124.99
123.93
120.38
116.58
116.47
104.76
104.55
75.91
74.80
21.19
21.04
37.73
36.09
34.95
33.18
31.29
Me
N
O
O
2r
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

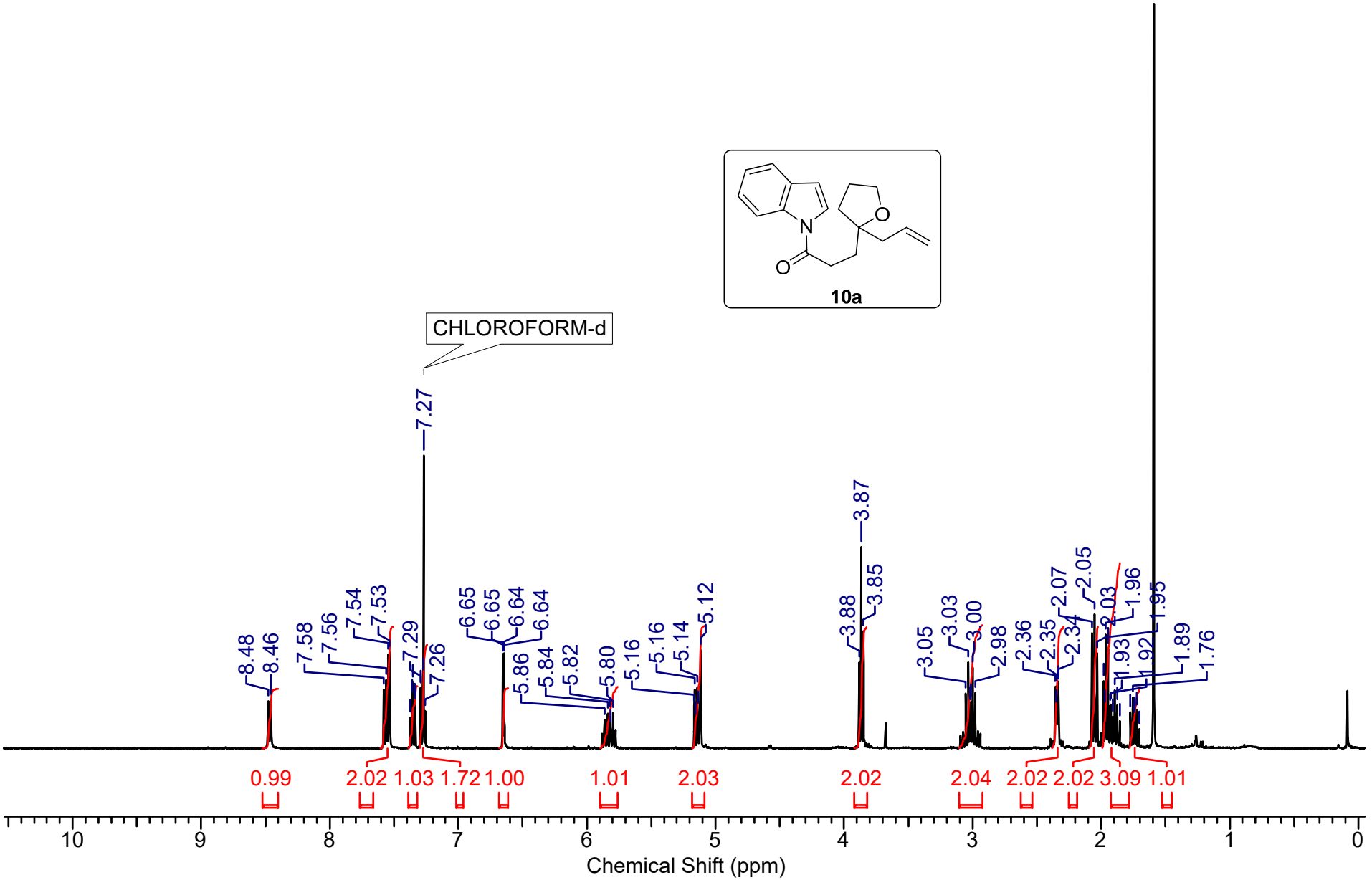

MSH-37 #374 RT: 2.03 AV: 1 NL: 3.36E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]
256.1326
R=71507
C 16 H 18 O 2 N = 256.1332
-2.2486 ppm
Relative Abundance
m/z
246.0810
R=65302
248.1486
R=61102
249.1569
R=71407
251.1250
R=67102
253.1407
R=65907
255.1202
R=61702
257.1359
R=69702
258.1391
R=65002
260.1485
R=66202
261.1302
R=69807
262.1336
R=69400
264.0982
R=56902
266.1533
R=69507
269.1354
R=66507
271.1146
R=67202
N
O
O
Me
2r

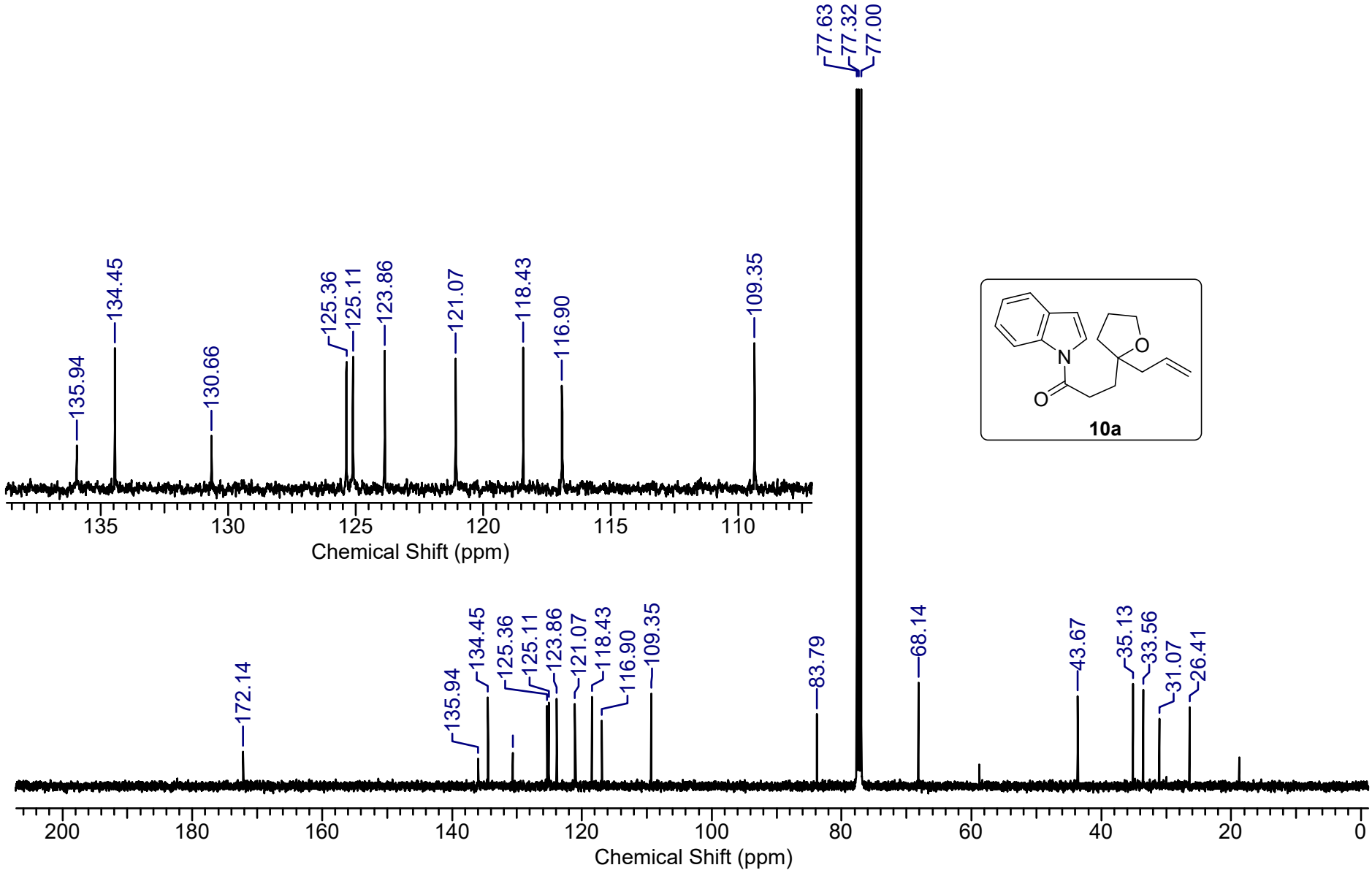

7.50
7.48
7.45
7.44
7.30
7.29
7.28
7.28
7.26
7.26
7.22
7.21
7.20
7.19
7.19
7.18
7.18
1.02
1.00
1.05
1.25
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.15
Chemical Shift (ppm)
3.15
3.86
2.94
3.88
3.85
2.92
2.96
2.34
2.32
2.30
2.13
1.99
1.97
1.96
1.84
1.74
1.73
1.68
8.40
8.38
7.50
7.48
7.45
7.44
7.28
7.19
7.19
7.18
7.18
6.57
6.56
6.56
1.03
1.02
1.00
1.05
1.25
1.03
2.14
3.08
2.05
1.14
1.09
2.09
1.20
1.02
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
N
O
O
OMe
9a

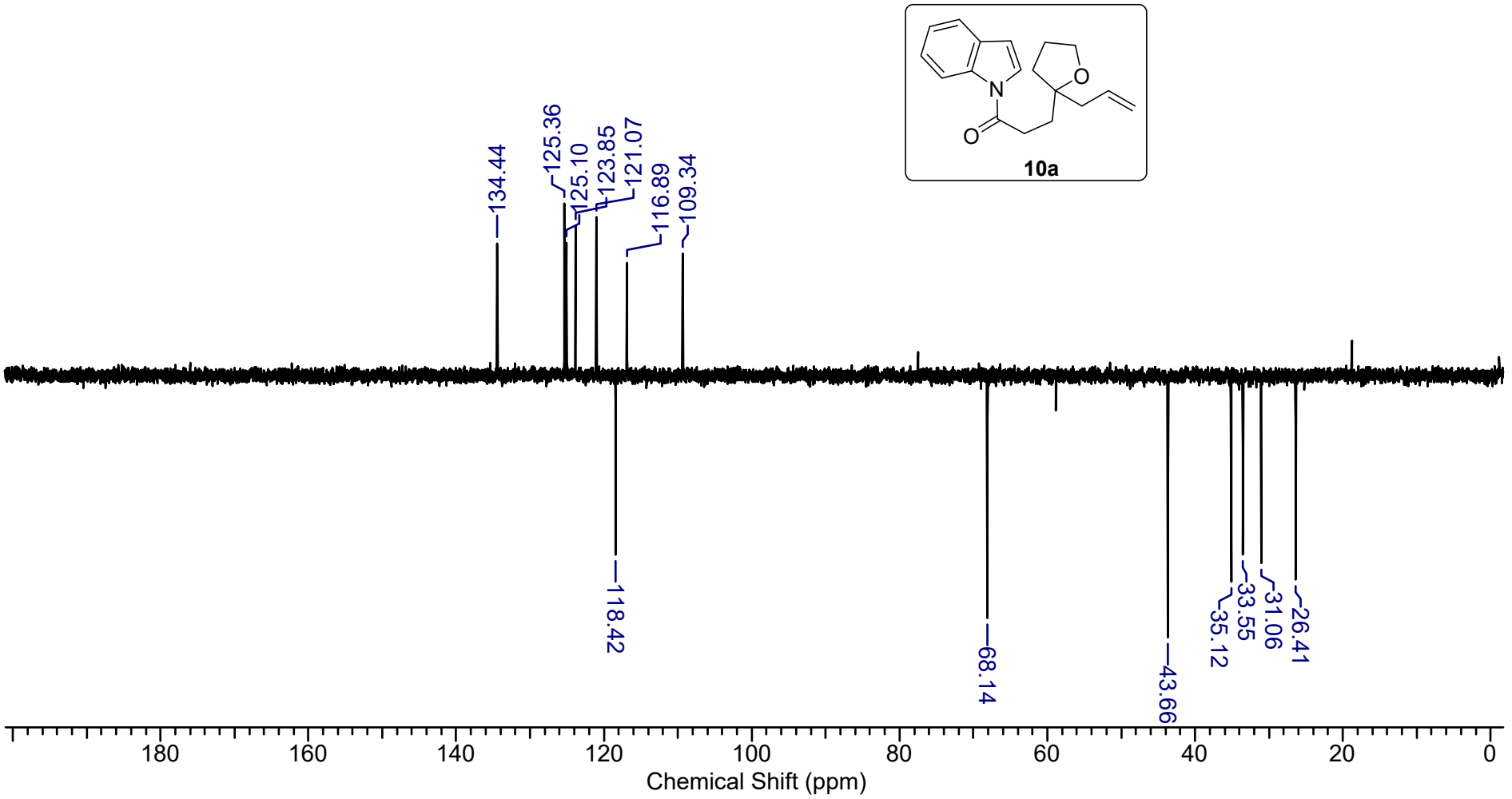

77.32
77.00
76.68
125.08
124.66
123.58
120.78
116.57
109.08
N
O
O
OMe
9a
124
122
120
118
116
114
112
110
Chemical Shift (ppm)
171.17
135.63
130.32
125.08
124.66
123.58
120.78
116.57
109.08
108.71
67.90
48.43
34.96
31.24
29.78
24.47
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


125.08
123.59
120.78
116.57
109.09
48.43
67.91
34.97
31.23
29.76
24.47
N
O
O
OMe
9a
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

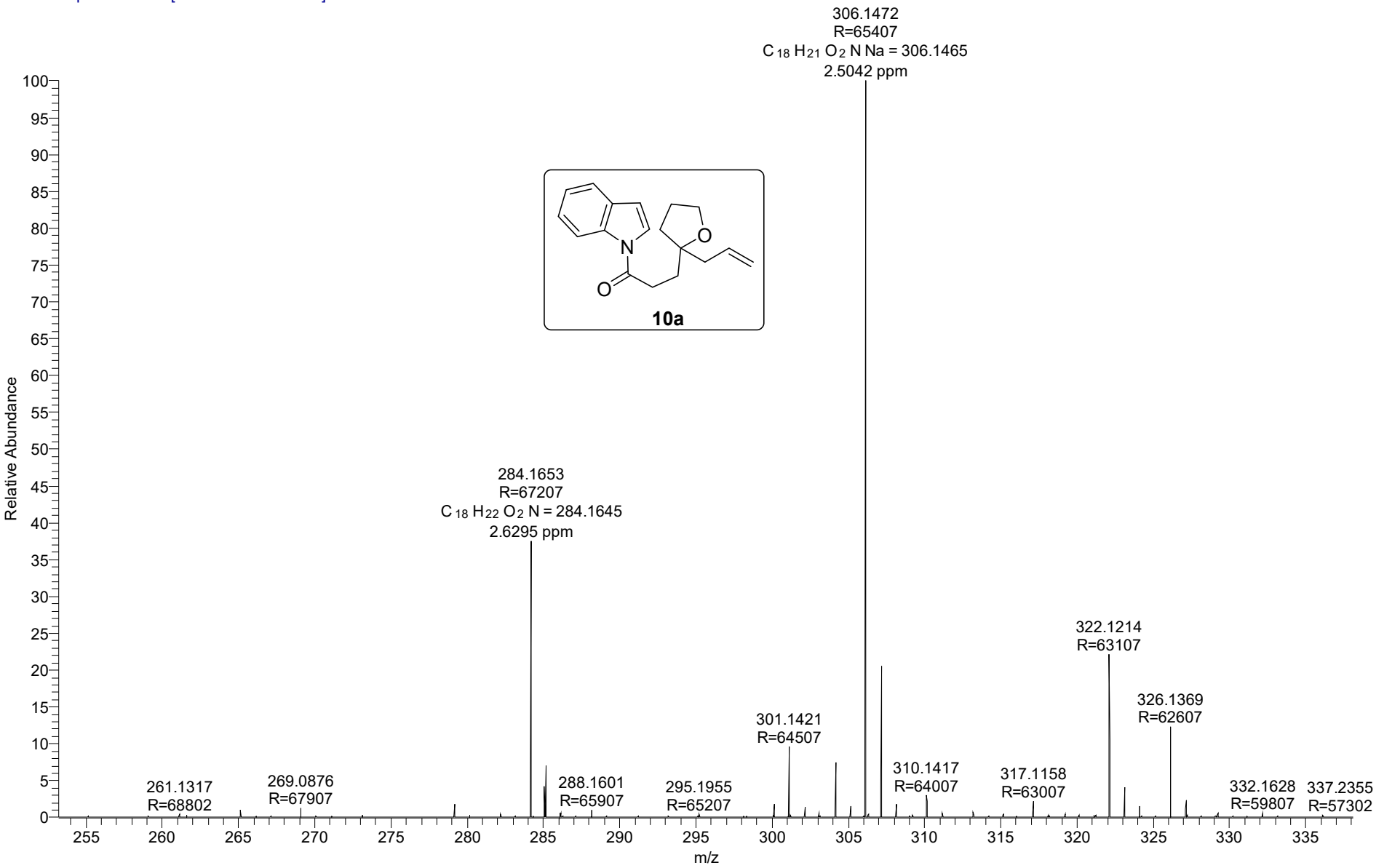

MSH-1 #318 RT: 1.71 AV: 1 NL: 3.59E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
296.1252
R=65807
C 16 H 19 O 3 N Na = 296.1257
-1.8981 ppm
276.0781
R=68406
278.0751
R=68002
282.1095
R=67107
286.1070
R=65106
293.1171
R=65507
304.1537
R=63707
308.1040
R=58207
312.0990
R=63607
319.2010
R=61707
Relative Abundance
m/z
N
O
O
OMe
9a

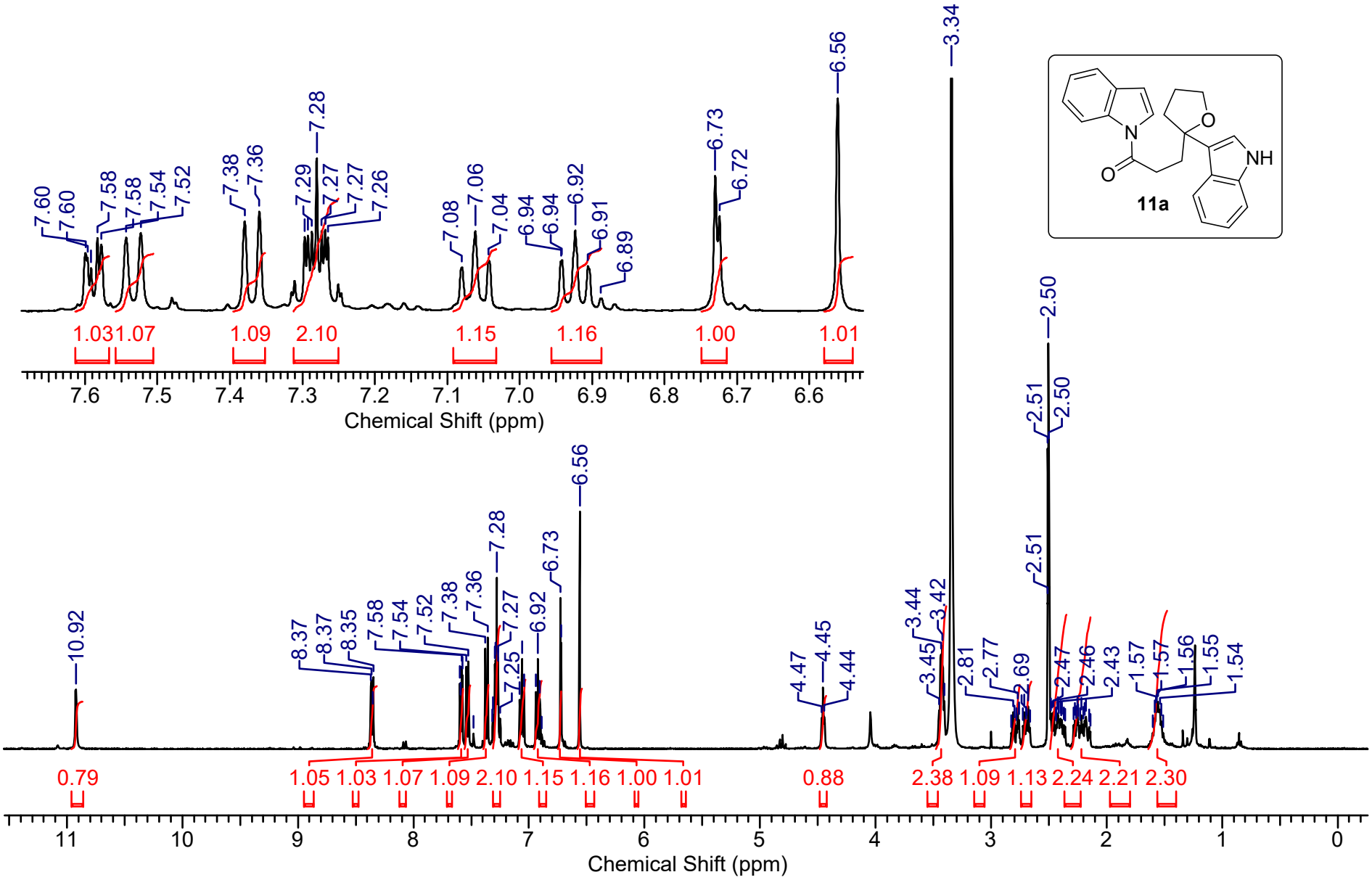

10a
CHLOROFORM-d
8.48
8.46
7.58
7.56
7.54
7.53
7.29
7.27
7.26
6.65
6.65
6.64
6.64
5.86
5.84
5.82
5.80
5.16
5.16
5.14
5.12
3.87
3.88
3.85
3.05
3.03
3.00
2.98
2.36
2.35
2.34
2.07
2.05
2.03
1.96
1.95
1.93
1.92
1.89
1.76
0.99
2.02
1.03
1.72
1.00
1.01
2.03
2.02
2.04
2.02
2.02
3.09
1.01
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

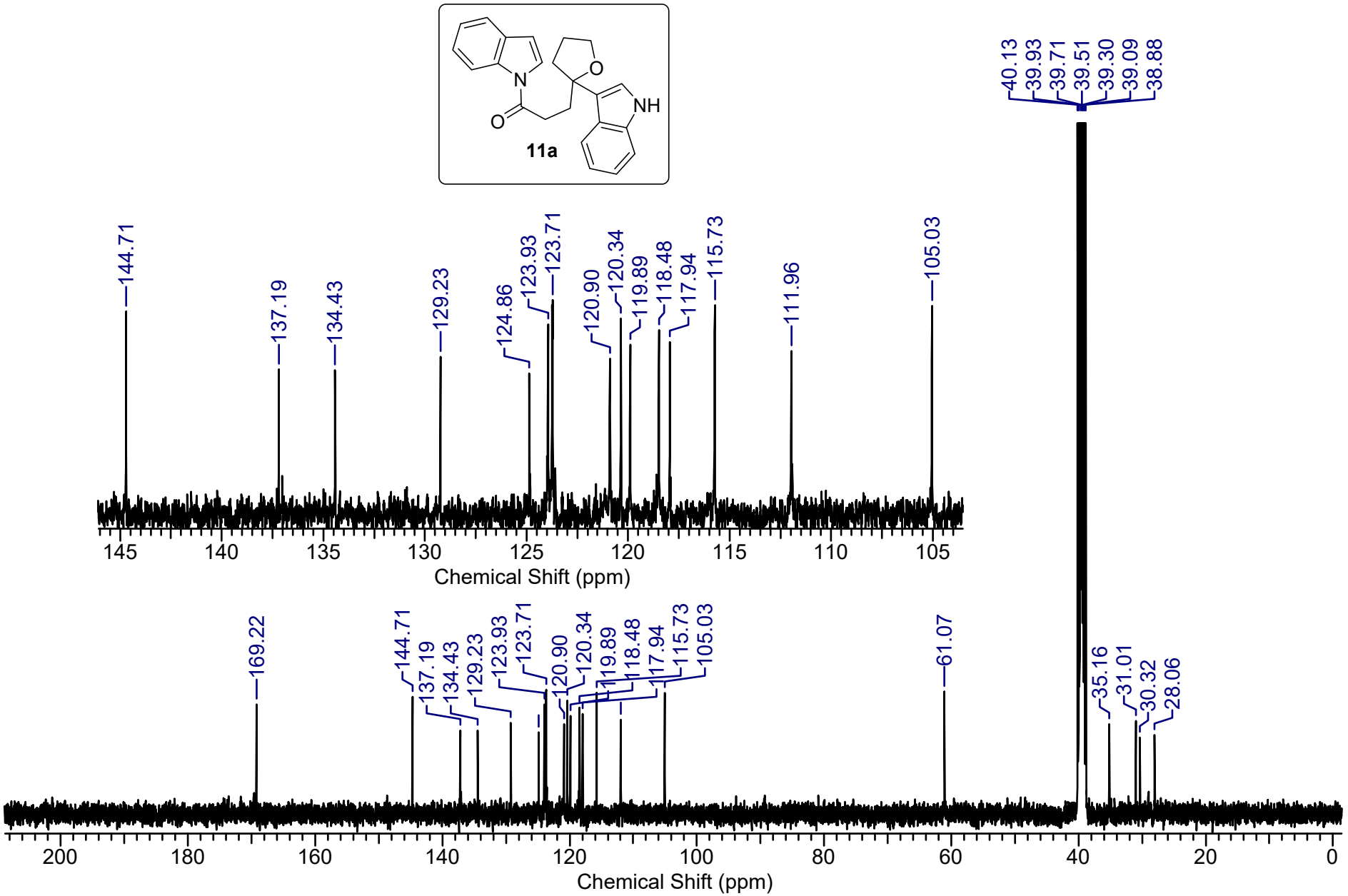

77.63
77.32
77.00
135.94
134.45
130.66
125.36
125.11
123.86
121.07
118.43
116.90
109.35
135
130
125
120
115
110
Chemical Shift (ppm)
N
O
O
10a
172.14
135.94
134.45
125.36
125.11
123.86
121.07
118.43
116.90
109.35
83.79
68.14
43.67
35.13
33.56
31.07
26.41
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

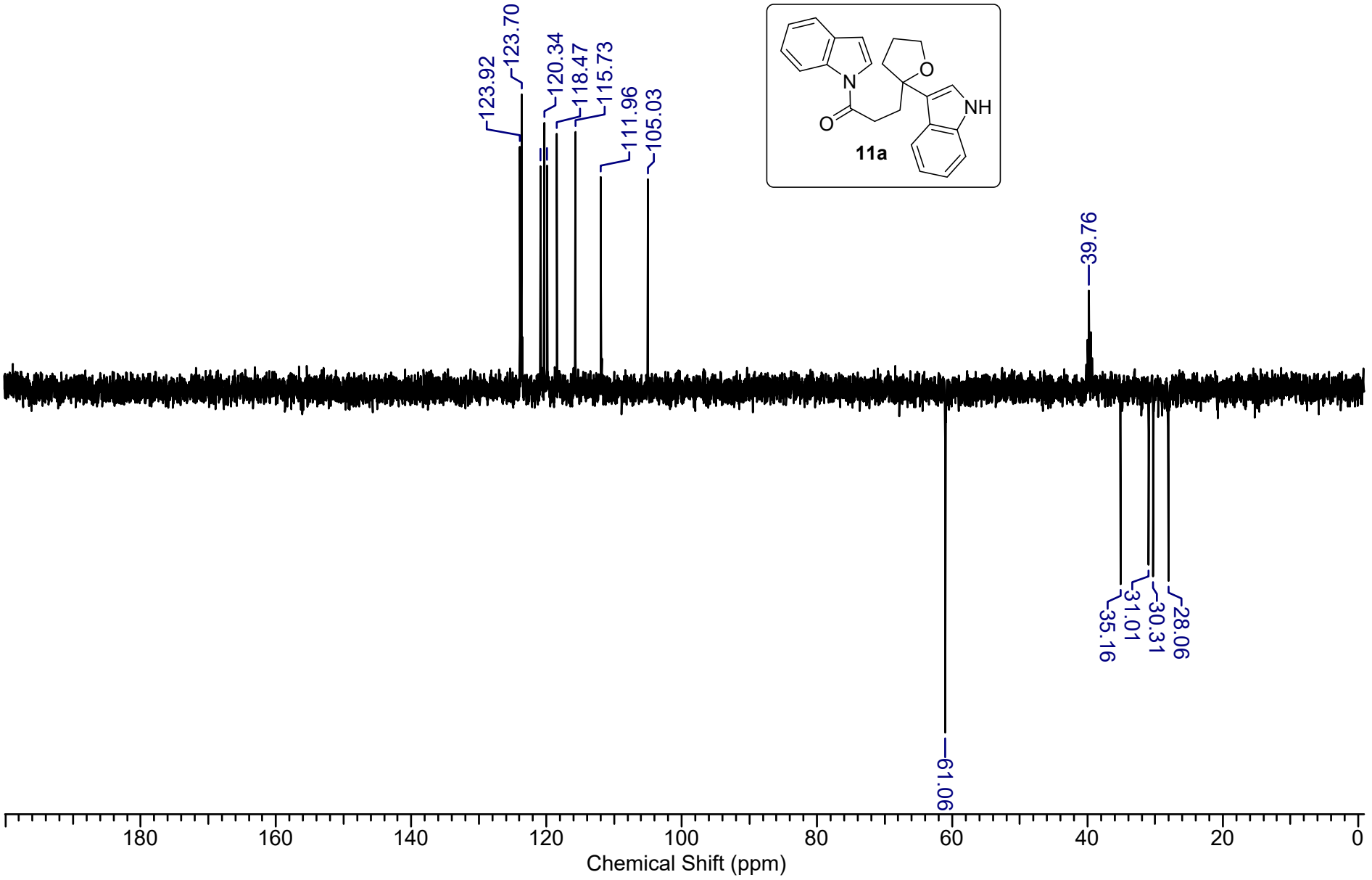

134.44
125.36
125.10
123.85
121.07
116.89
109.34
118.42
68.14
43.66
35.12
33.55
31.06
26.41
N
O
O
10a
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

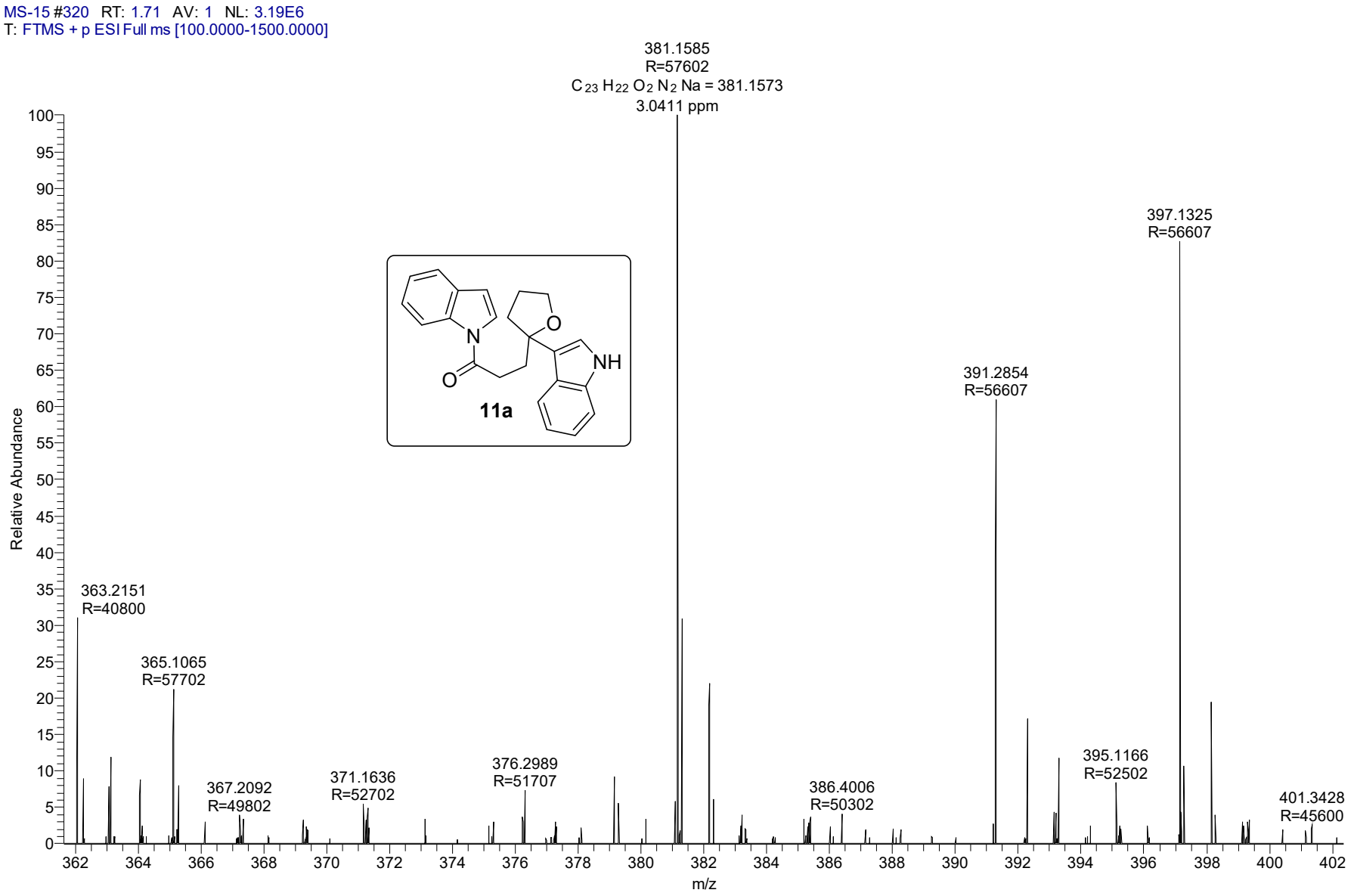
MS-16 #316 RT: 1.69 AV: 1 NL: 1.45E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

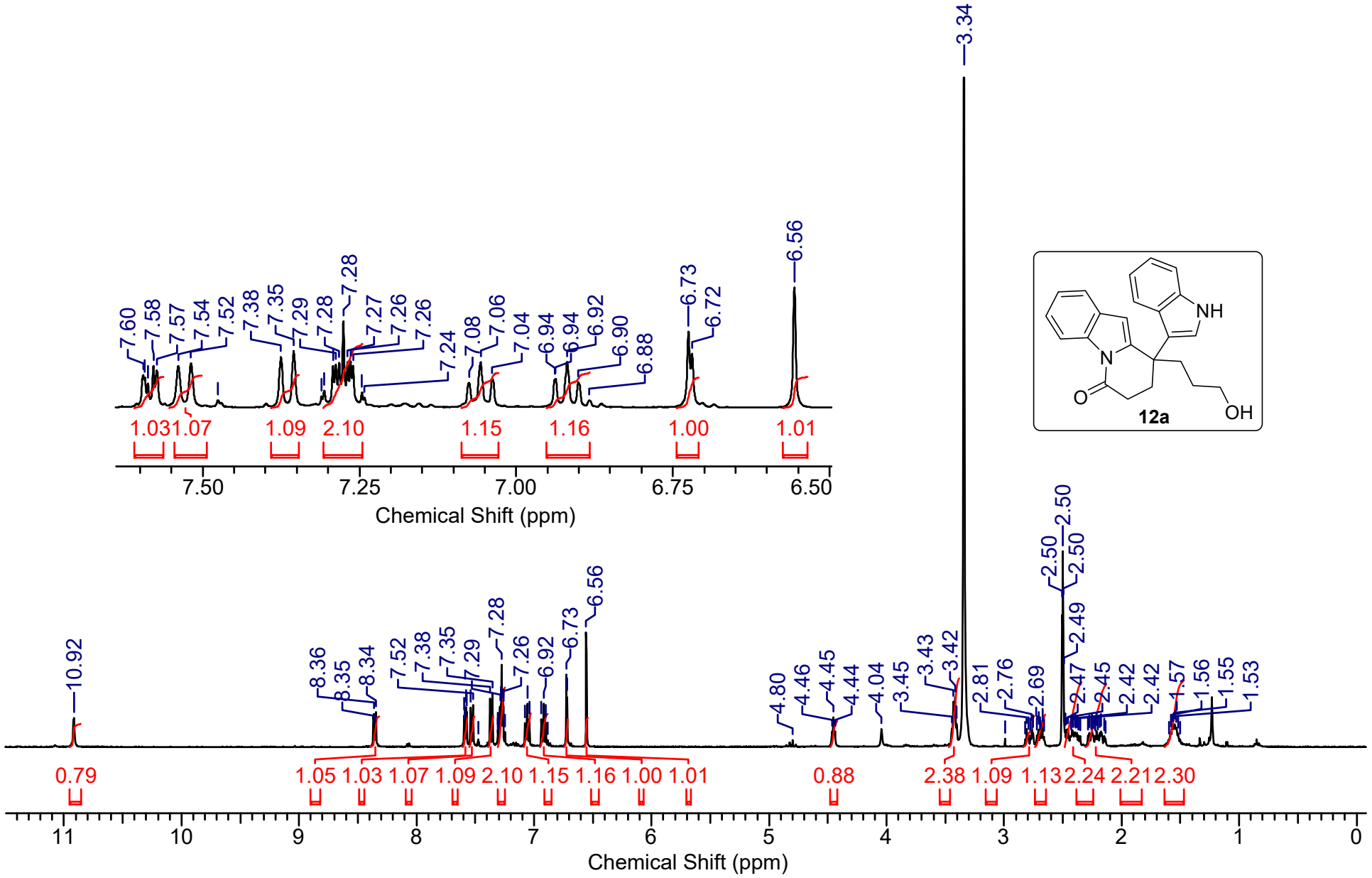


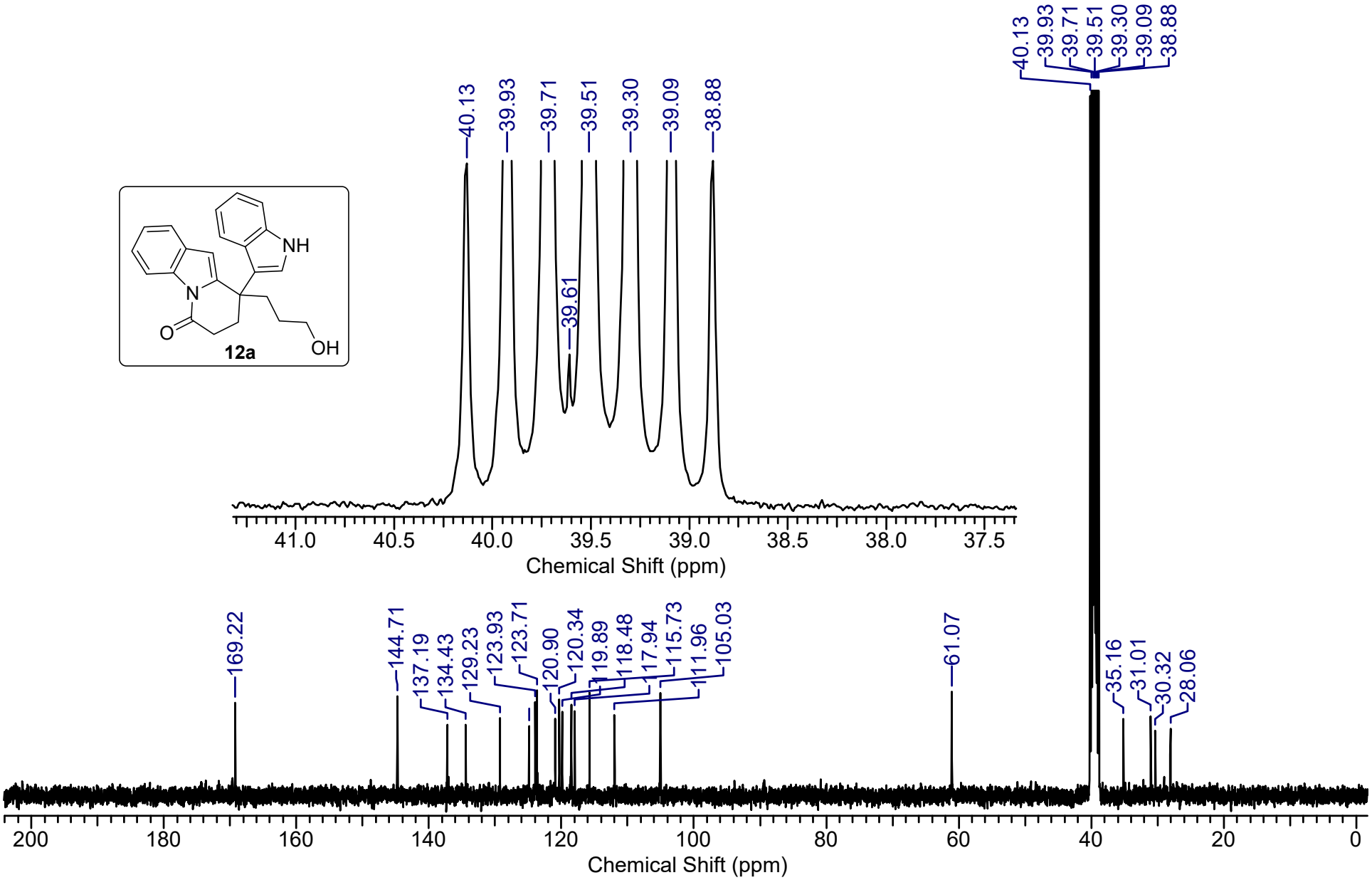

11a
Chemical Shift (ppm)
Chemical Shift (ppm)

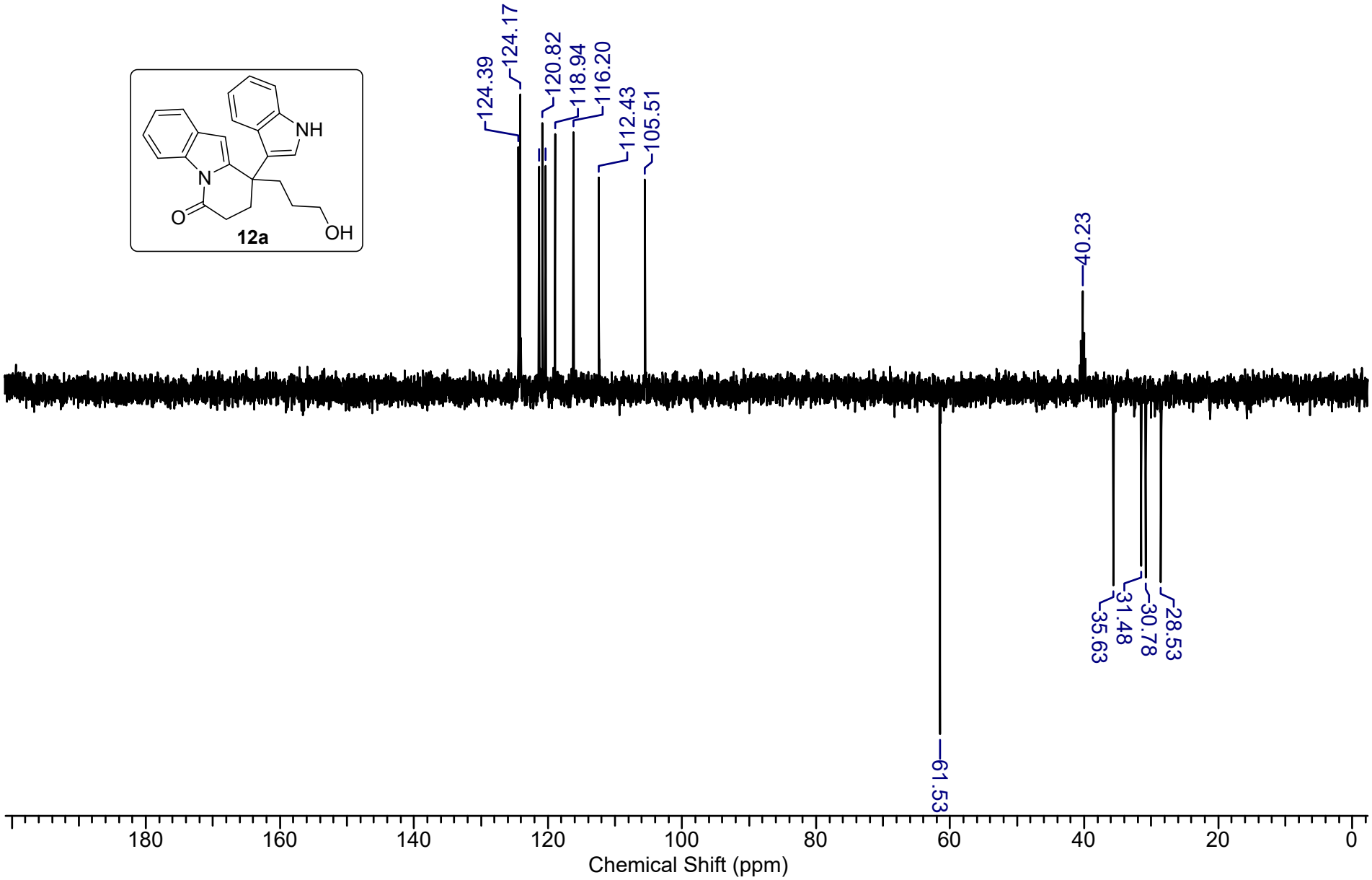

11a
144.71
137.19
134.43
129.23
124.86
123.93
123.71
120.90
120.34
119.89
118.48
117.94
115.73
111.96
105.03
40.13
39.93
39.71
39.51
39.30
39.09
38.88
Chemical Shift (ppm)
169.22
144.71
137.19
134.43
129.23
123.93
123.71
120.90
120.34
119.89
118.48
117.94
115.73
105.03
61.07
35.16
31.01
30.32
28.06
Chemical Shift (ppm)


123.92
123.70
120.34
118.47
115.73
111.96
105.03
39.76
35.16
31.01
30.31
28.06
61.06
11a
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

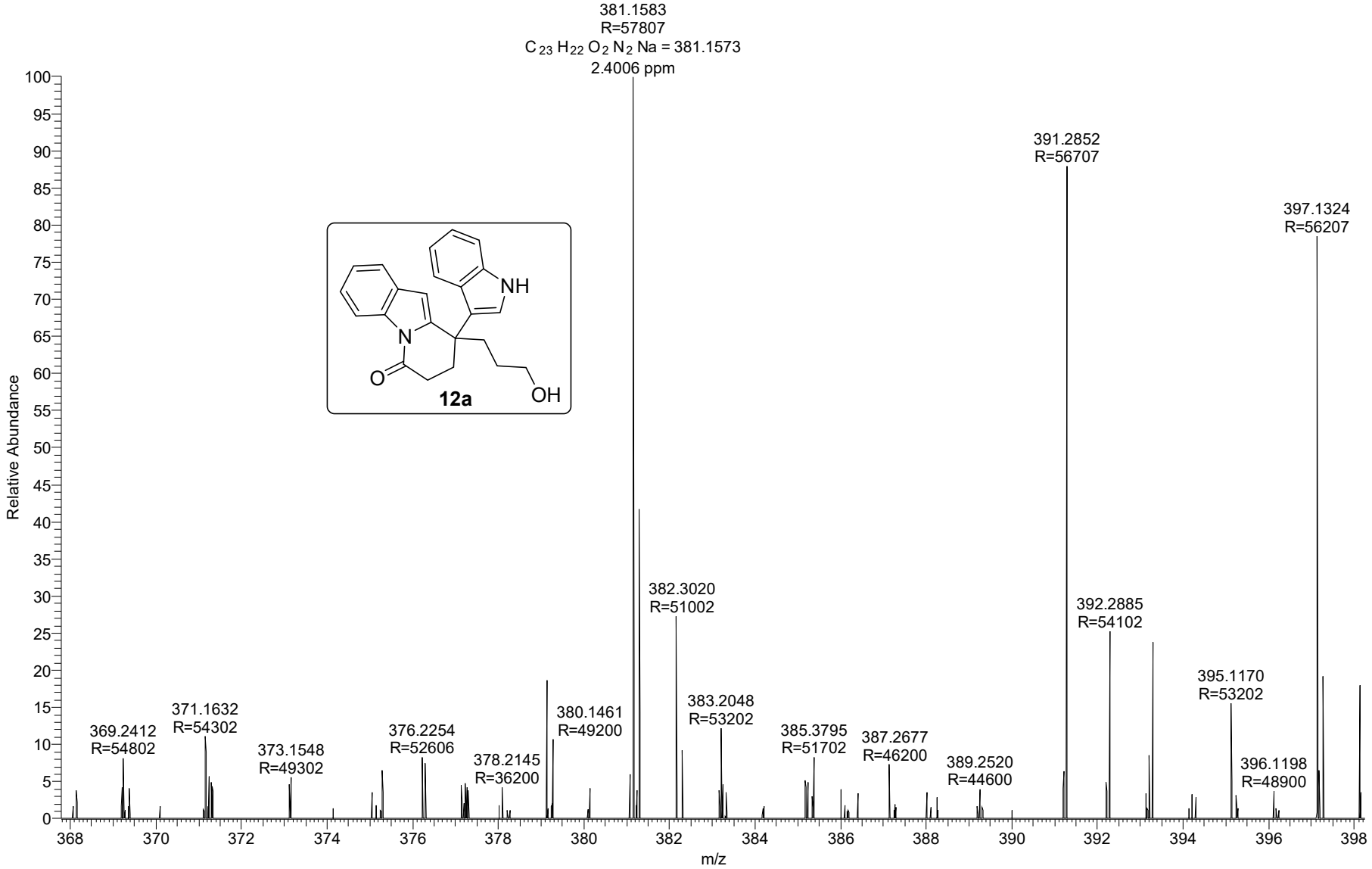

MS-15 #320 RT: 1.71 AV: 1 NL: 3.19E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]
381.1585
R=57602
C 23 H 22 O 2 N 2 Na = 381.1573
3.0411 ppm
397.1325
R=56607
391.2854
R=56607
363.2151
R=40800
365.1065
R=57702
367.2092
R=49802
371.1636
R=52702
376.2989
R=51707
386.4006
R=50302
395.1166
R=52502
401.3428
R=45600
11a
Relative Abundance
m/z

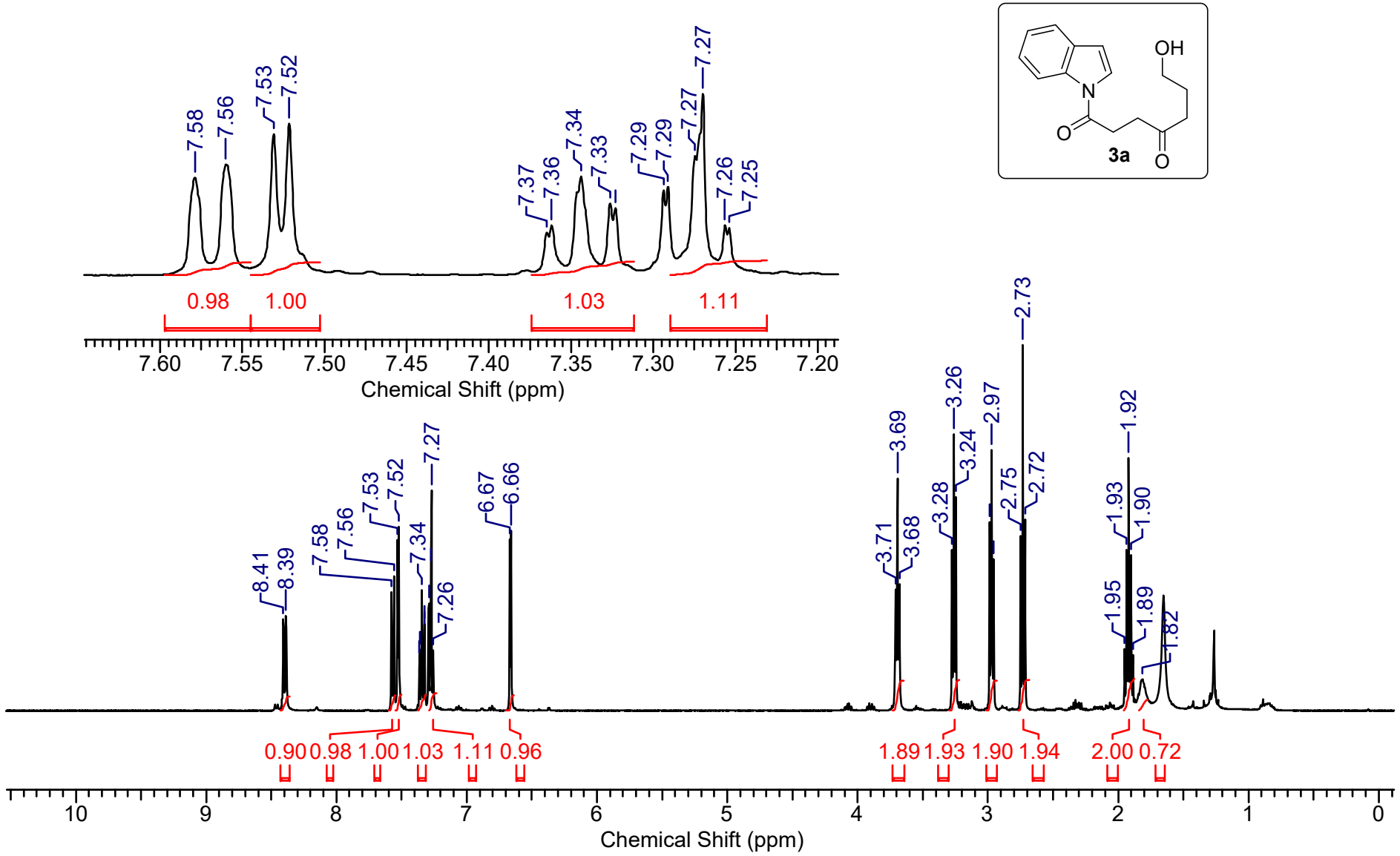

12a
NH
N
O
OH
Chemical Shift (ppm)
Chemical Shift (ppm)

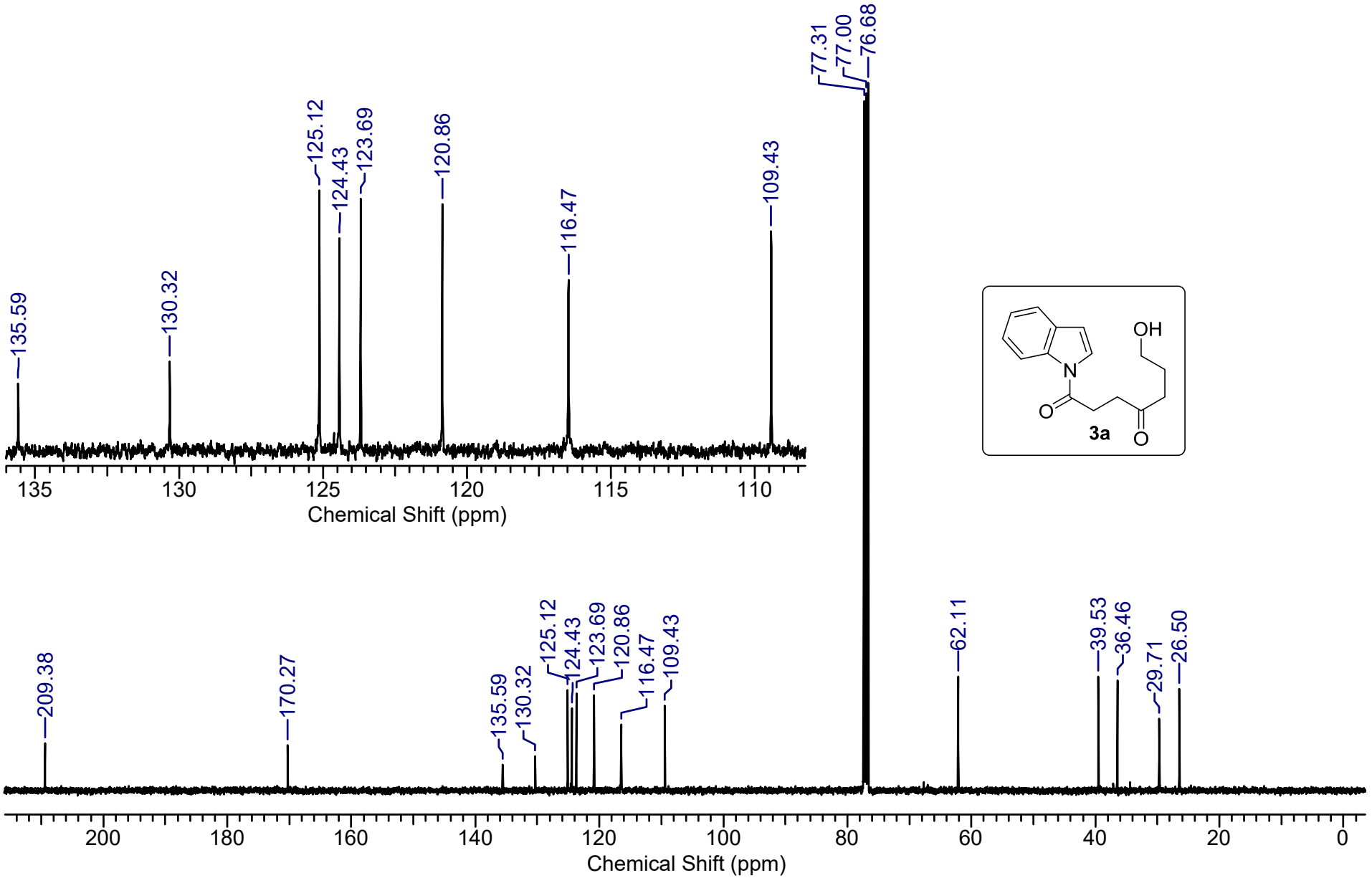

12a
NH
N
O
OH
40.13
39.93
39.71
39.61
39.51
39.30
39.09
38.88
41.0
40.5
40.0
39.5
39.0
38.5
38.0
37.5
Chemical Shift (ppm)
169.22
144.71
137.19
134.43
129.23
123.93
123.71
120.90
120.34
119.89
118.48
117.94
115.73
111.96
105.03
61.07
35.16
31.01
30.32
28.06
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

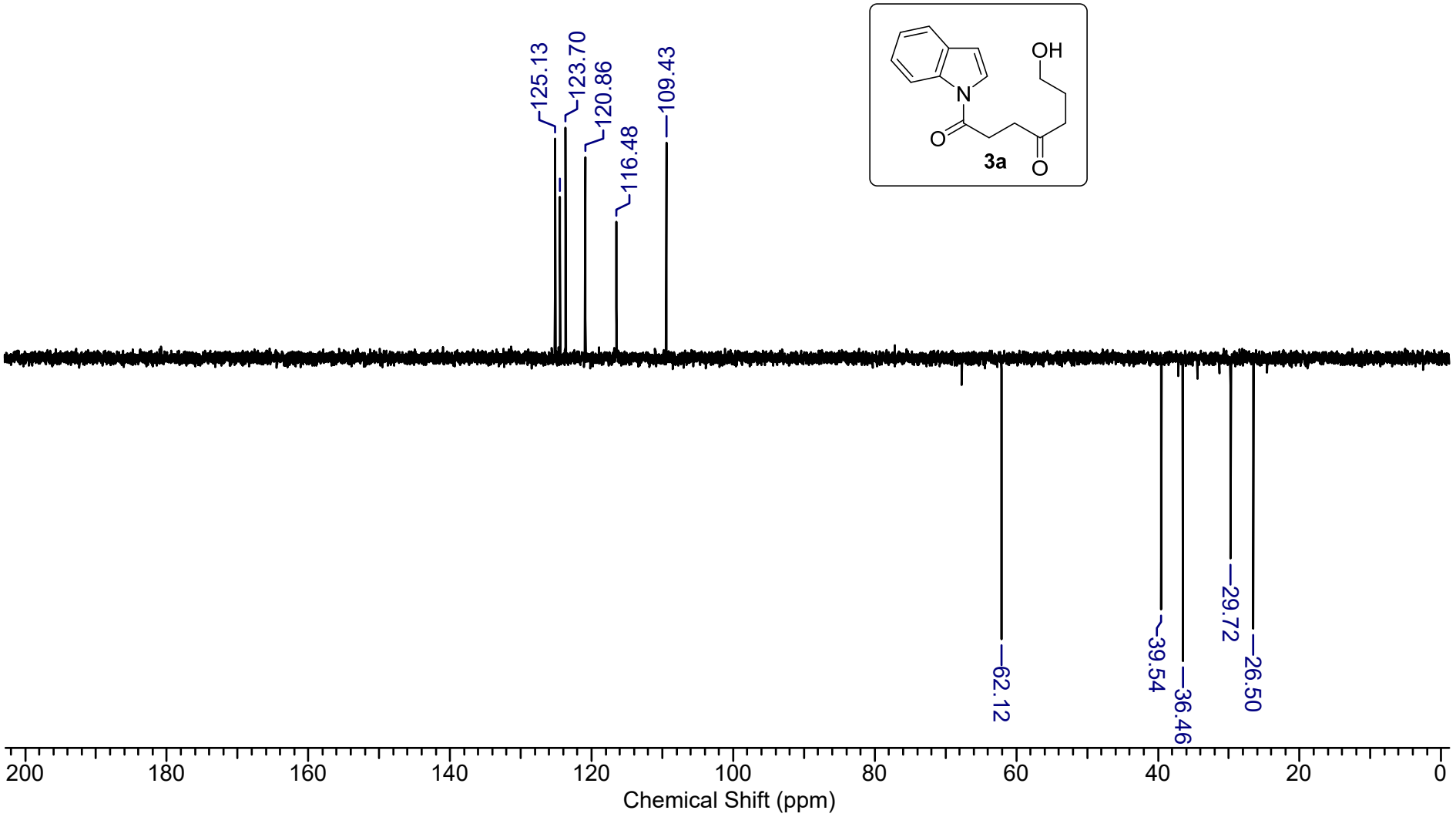

124.39
124.17
120.82
118.94
116.20
112.43
105.51
40.23
35.63
31.48
30.78
28.53
61.53
NH
N
O
OH
12a
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-15a #310 RT: 1.66 AV: 1 NL: 2.58E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]

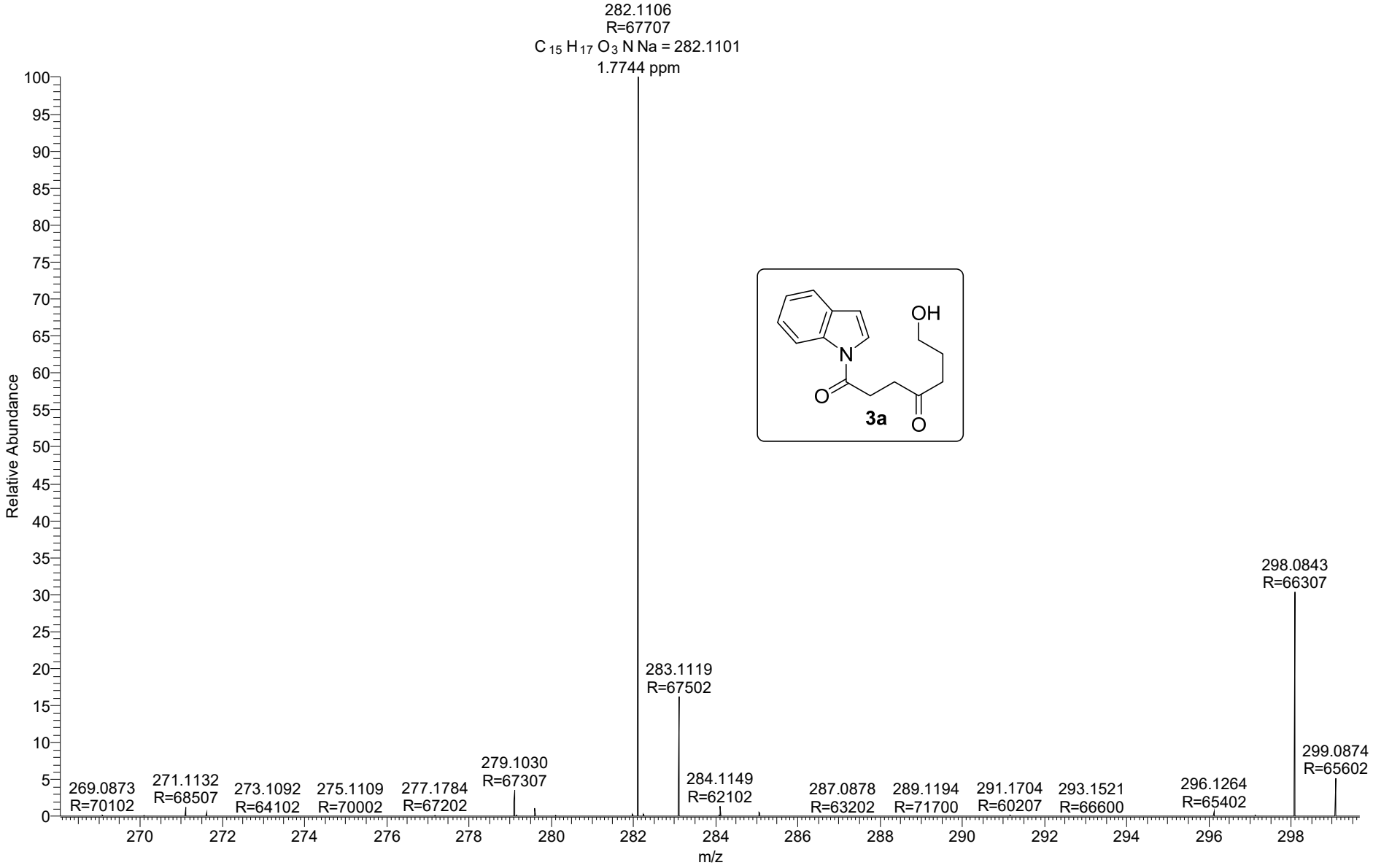


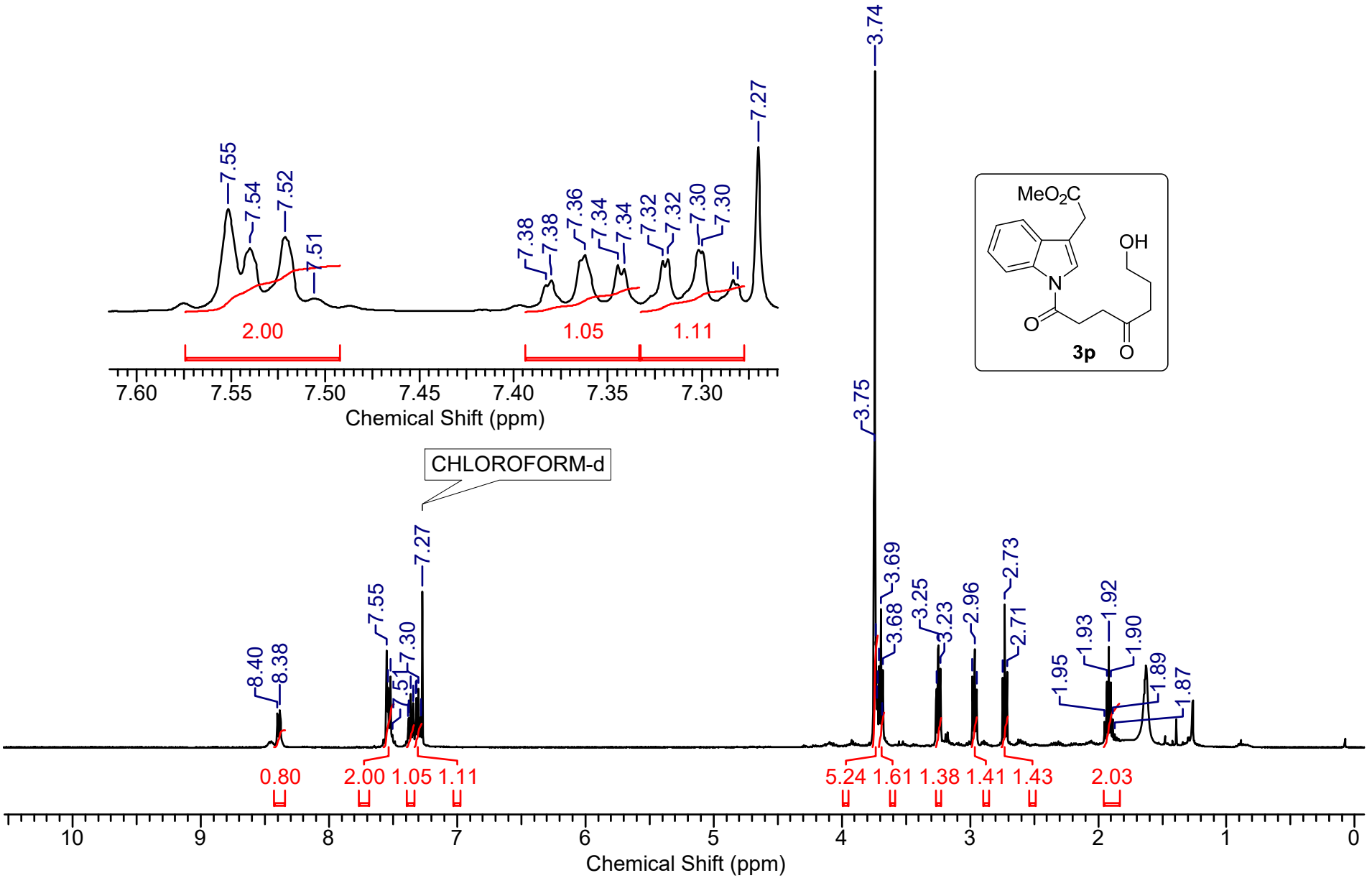

OH
N
O
O
3a
7.58
7.56
7.53
7.52
7.37
7.36
7.34
7.33
7.29
7.29
7.27
7.27
7.26
7.25
0.98
1.00
1.03
1.11
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
Chemical Shift (ppm)
8.41
8.39
7.58
7.56
7.53
7.52
7.34
7.27
7.26
6.67
6.66
3.69
3.71
3.68
3.26
3.28
3.24
2.97
2.75
2.73
2.72
1.92
1.93
1.90
1.95
1.89
1.82
0.90
0.98
1.00
1.03
1.11
0.96
1.89
1.93
1.90
1.94
2.00
0.72
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

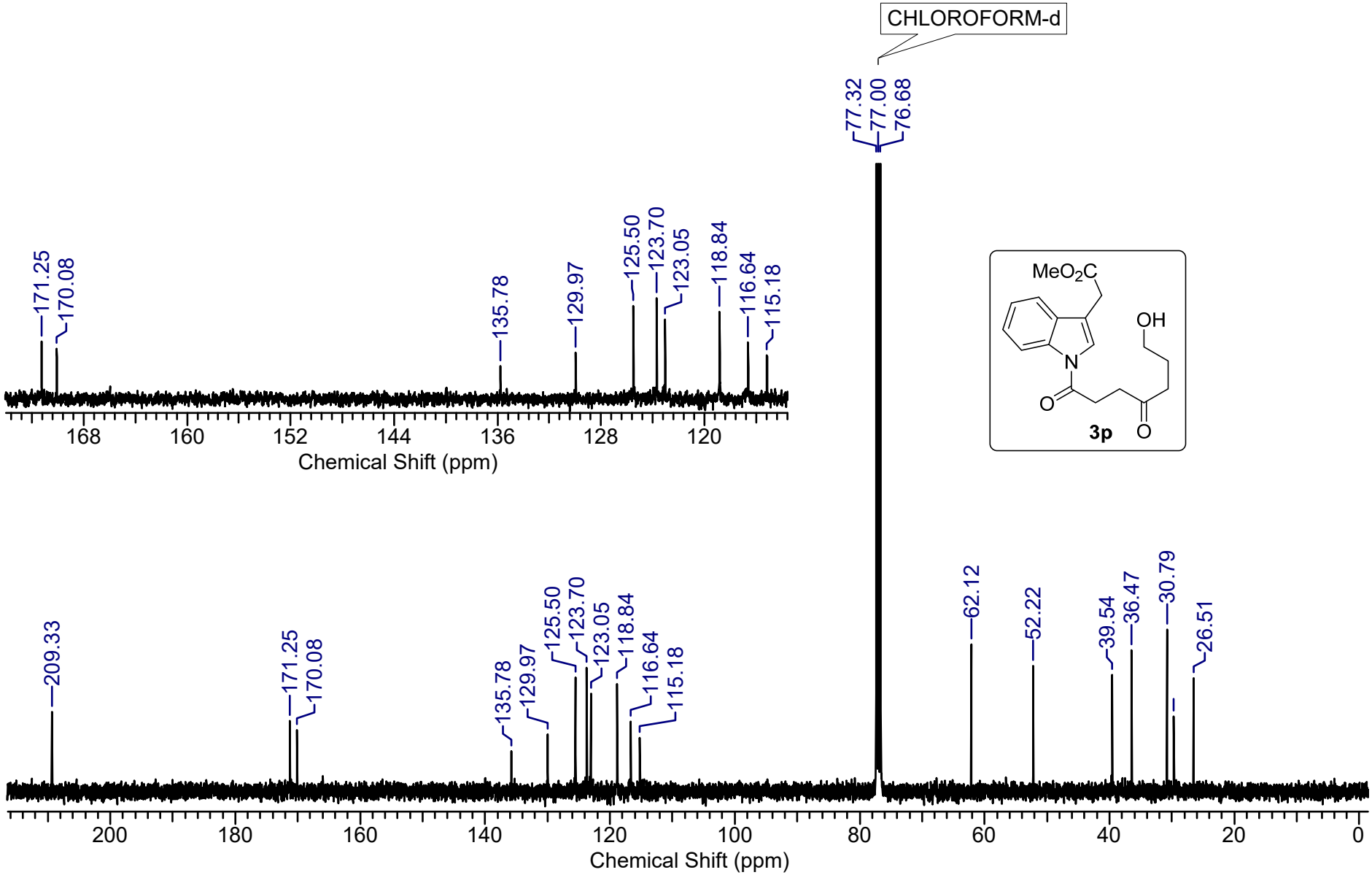

135.59
130.32
125.12
124.43
123.69
120.86
116.47
109.43
77.31
77.00
76.68
135
130
125
120
115
110
Chemical Shift (ppm)
OH
N
O
O
3a
209.38
170.27
135.59
130.32
125.12
124.43
123.69
120.86
116.47
109.43
62.11
39.53
36.46
29.71
26.50
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

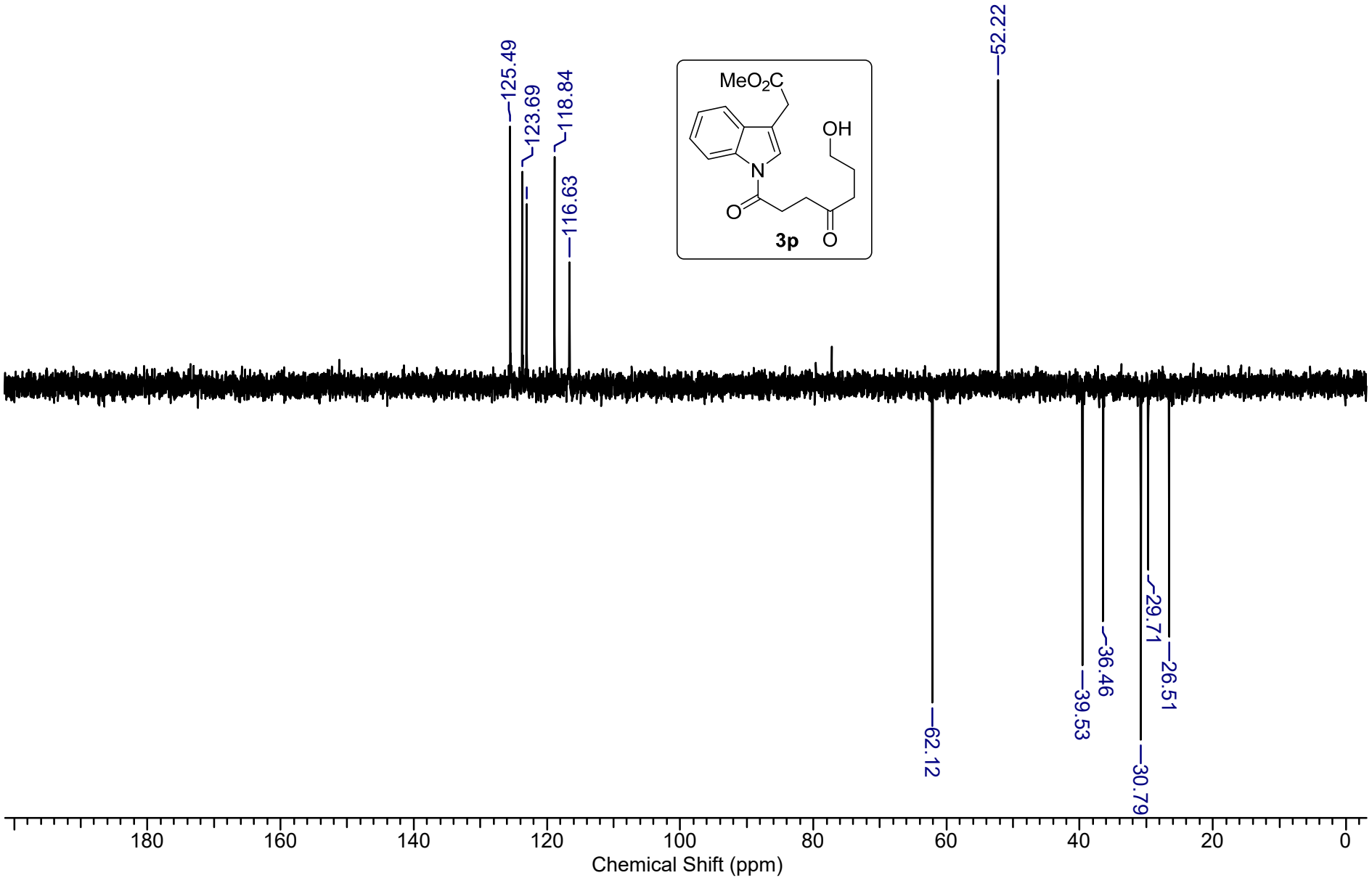

125.13
123.70
120.86
116.48
109.43
OH
N
O
O
3a
62.12
39.54
36.46
29.72
26.50
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-17 #267 RT: 1.43 AV: 1 NL: 7.22E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

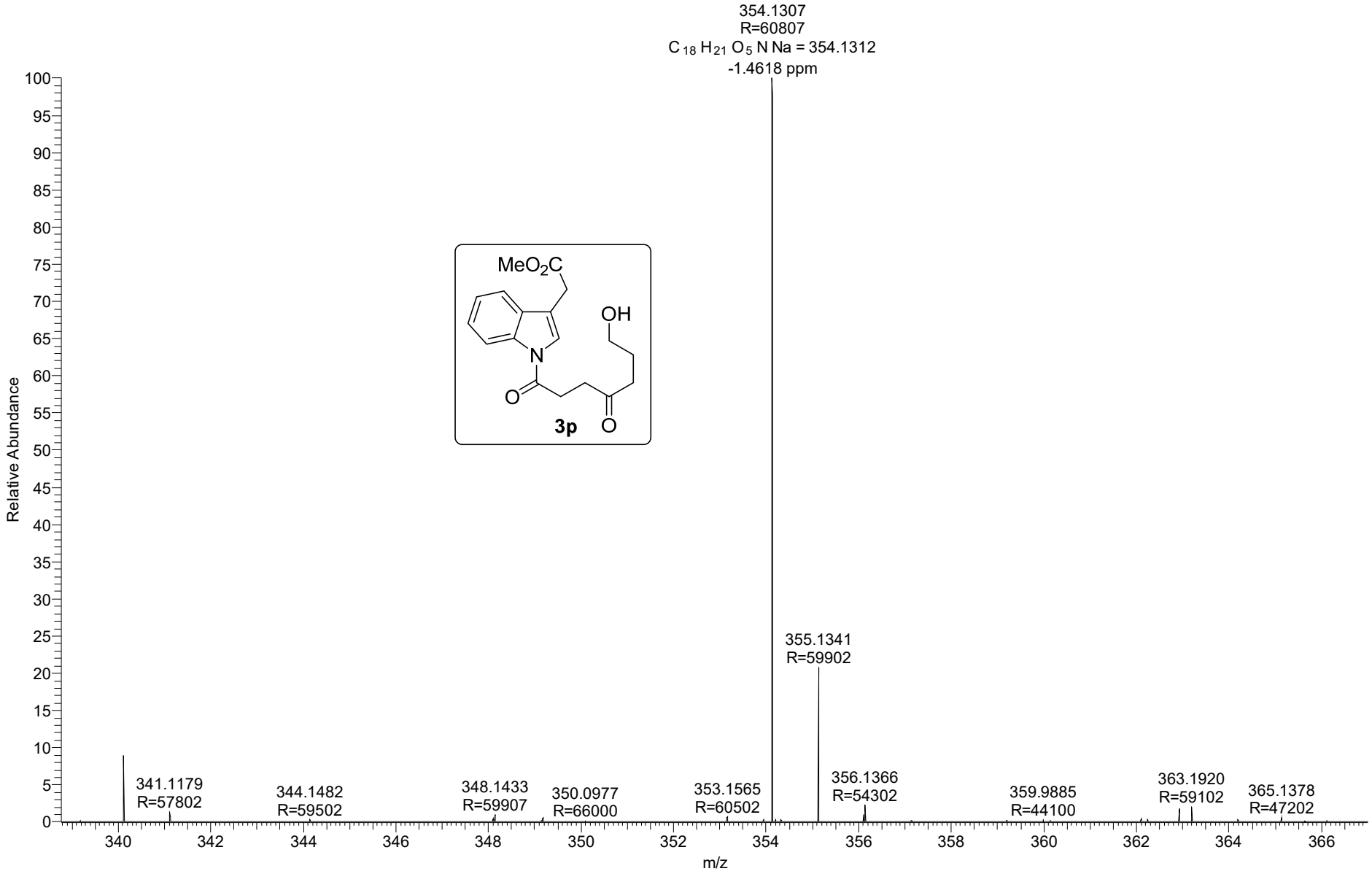


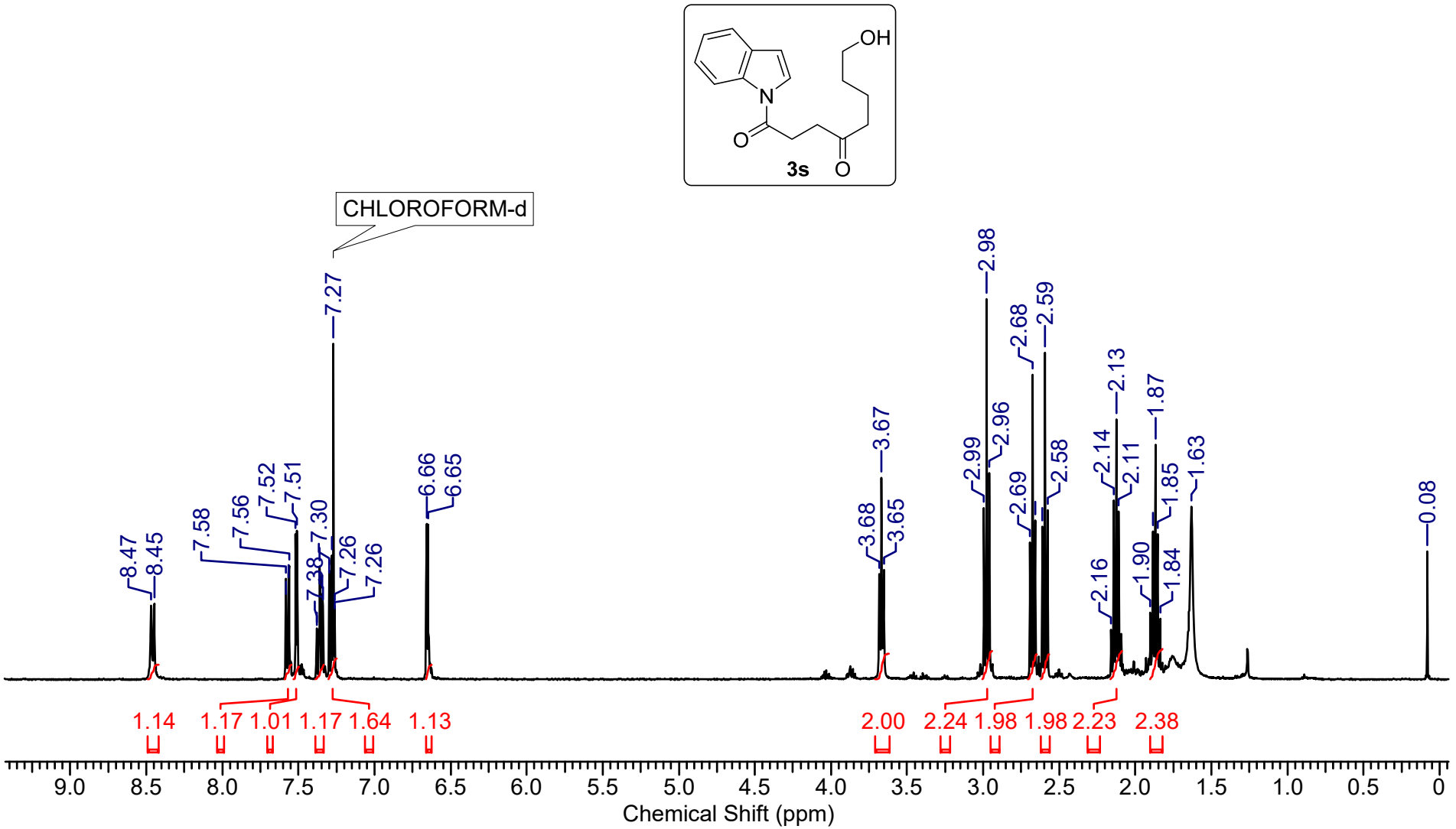

7.55
7.54
7.52
7.51
7.38
7.38
7.36
7.34
7.34
7.32
7.32
7.30
7.30
7.27
2.00
1.05
1.11
7.60
7.55
7.50
7.45
7.40
7.35
7.30
Chemical Shift (ppm)
MeO2C
OH
N
O
O
3p
3.74
3.75
CHLOROFORM-d
8.40
8.38
7.55
7.51
7.30
7.27
3.69
3.68
3.25
3.23
2.96
2.73
2.71
1.95
1.93
1.92
1.90
1.89
1.87
0.80
2.00
1.05
1.11
5.24
1.61
1.38
1.41
1.43
2.03
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

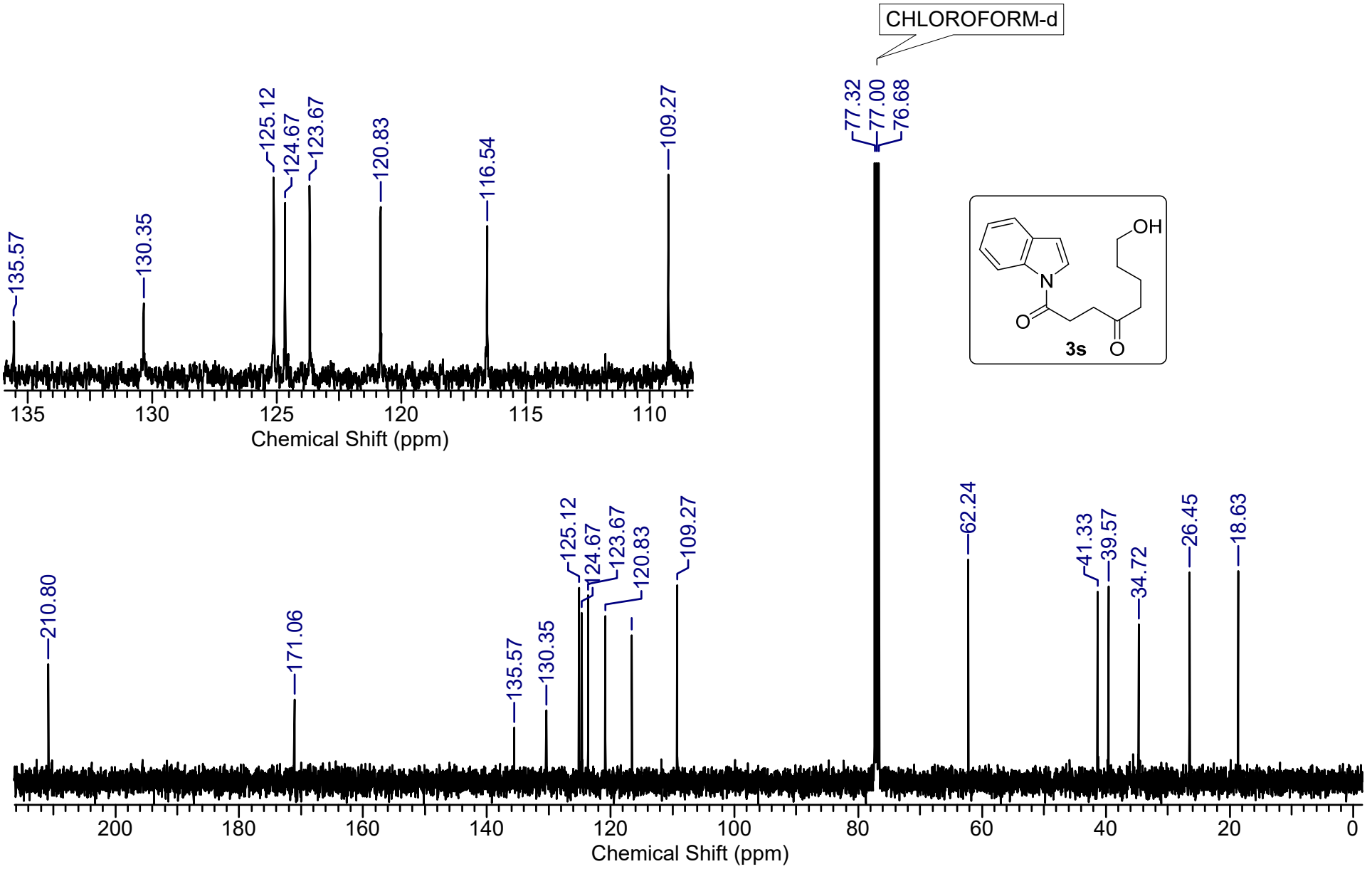

CHLOROFORM-d
77.32
77.00
76.68
MeO2C
OH
N
O
O
3p
209.33
171.25
170.08
135.78
129.97
125.50
123.70
123.05
118.84
116.64
115.18
62.12
52.22
39.54
36.47
30.79
26.51
Chemical Shift (ppm)
Chemical Shift (ppm)

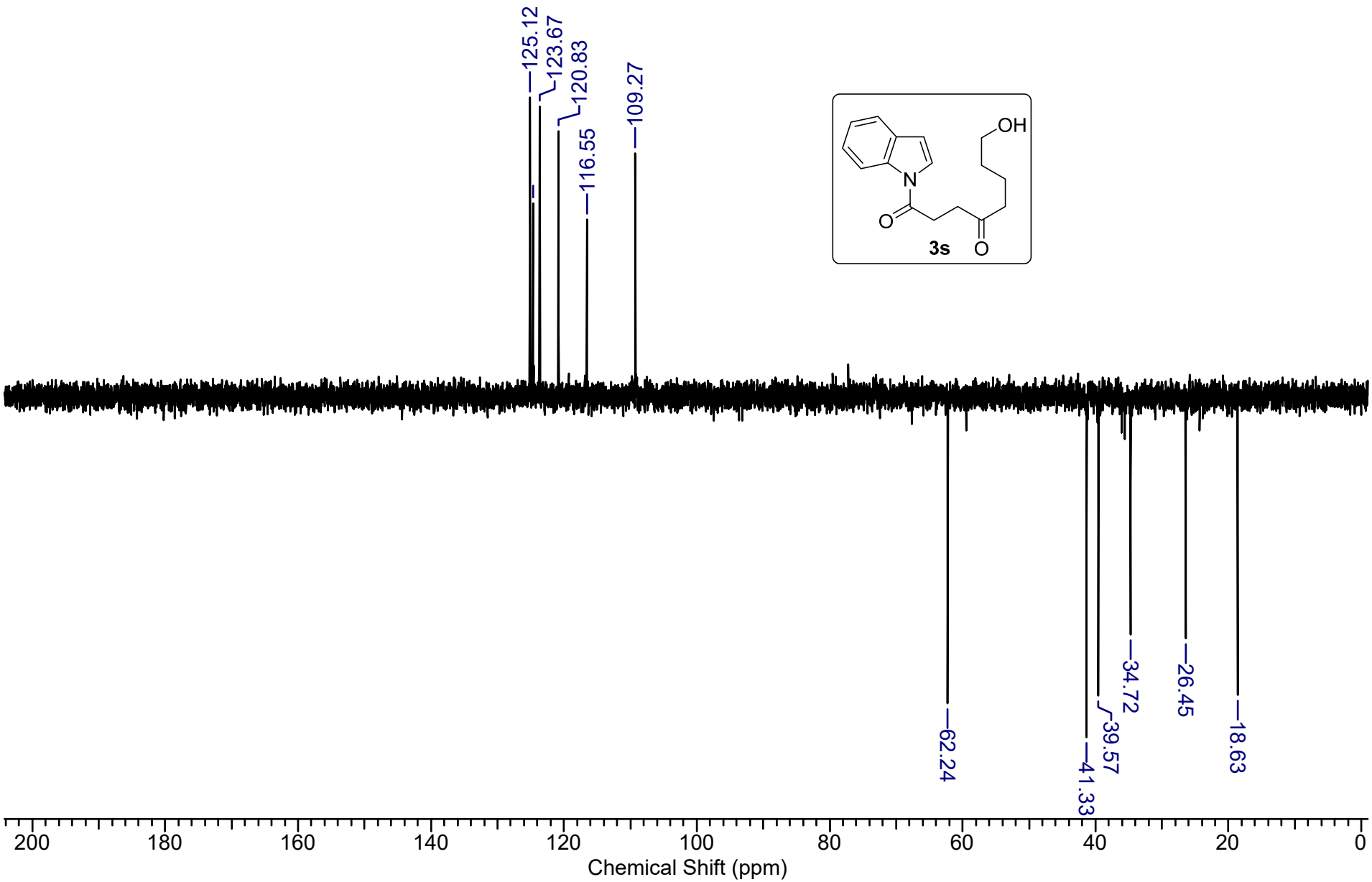

125.49
123.69
118.84
116.63
52.22
62.12
39.53
36.46
30.79
29.71
26.51
MeO2C
OH
N
O
O
3p
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MSH-102 #334 RT: 1.81 AV: 1 NL: 1.12E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

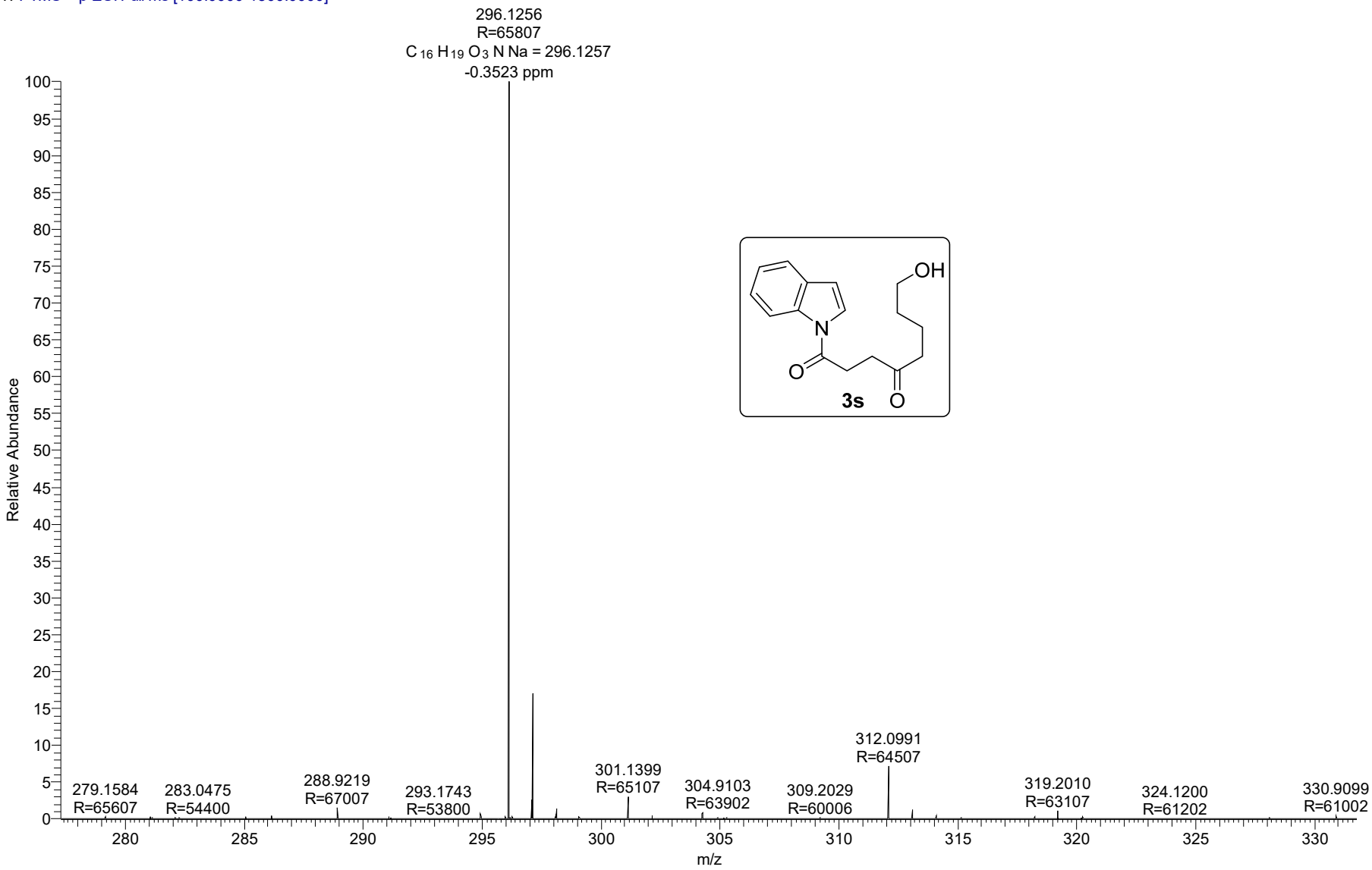


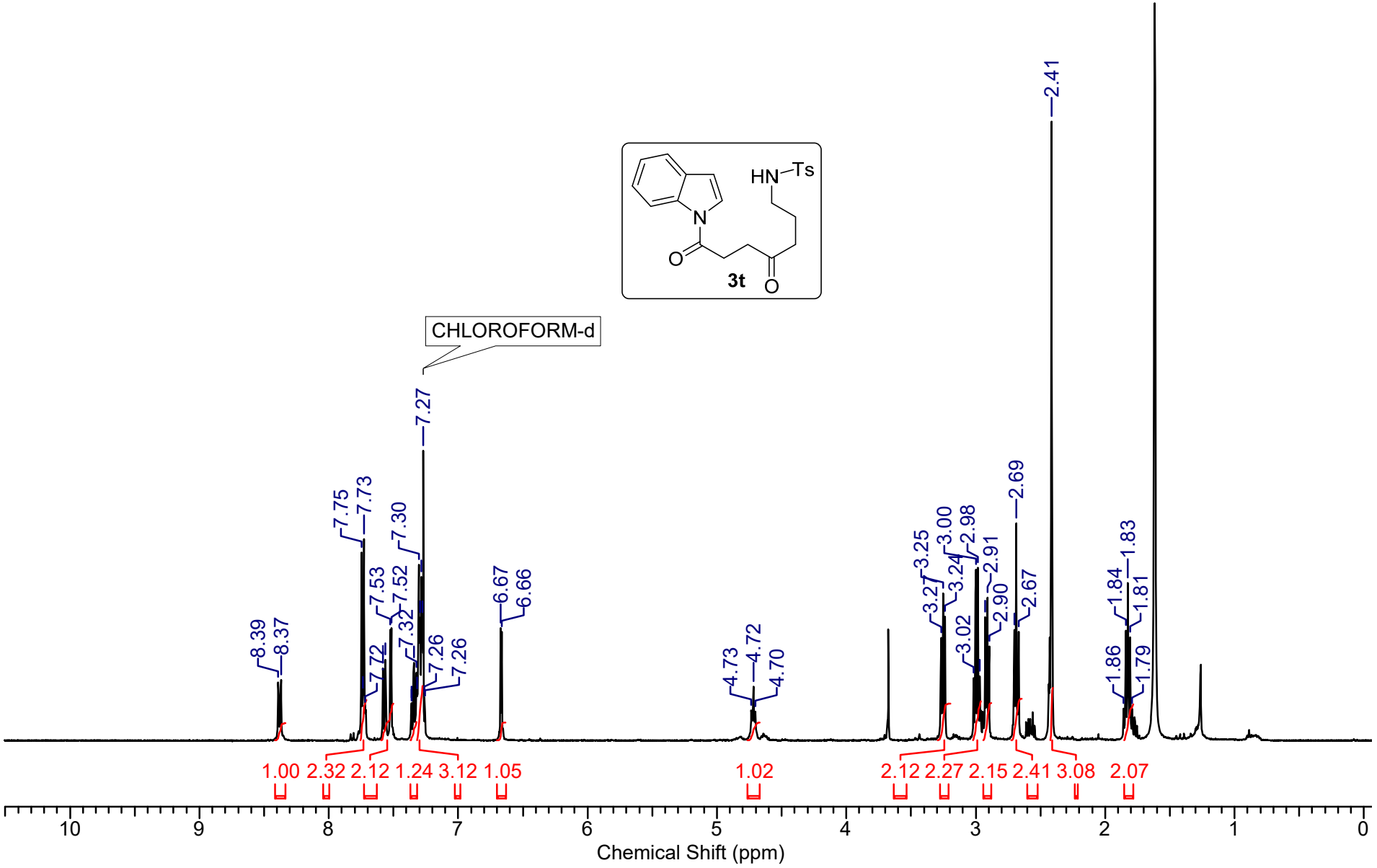

OH
N
O
O
3s
CHLOROFORM-d
8.47
8.45
7.58
7.56
7.52
7.51
7.30
7.38
7.27
7.26
7.26
6.66
6.65
3.68
3.67
3.65
2.99
2.98
2.96
2.69
2.68
2.59
2.58
2.16
2.14
2.13
2.11
1.90
1.87
1.85
1.84
1.63
0.08
1.14
1.17
1.01
1.17
1.64
1.13
2.00
2.24
1.98
1.98
2.23
2.38
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

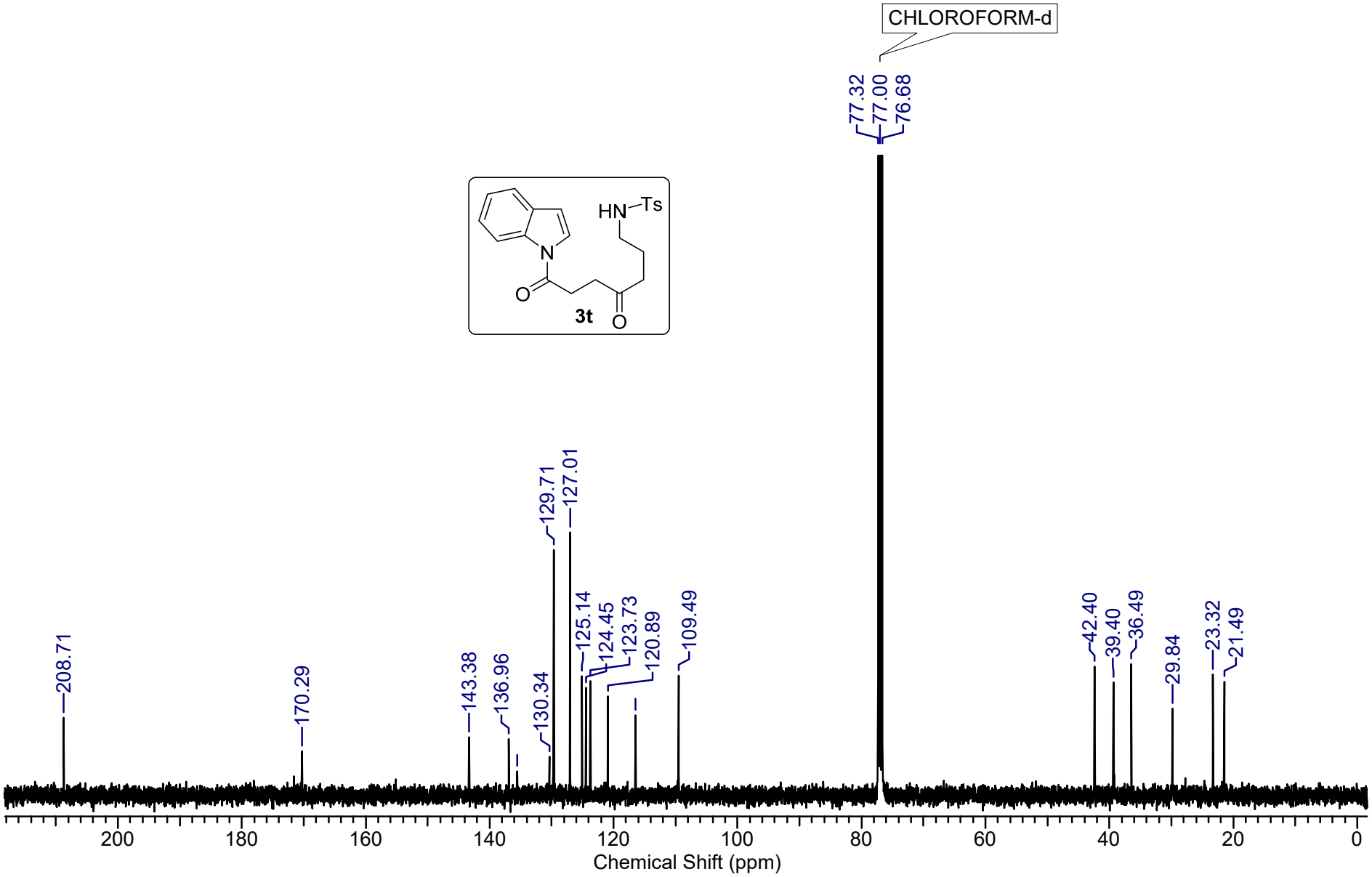

CHLOROFORM-d
77.32
77.00
76.68
135.57
130.35
125.12
124.67
123.67
120.83
116.54
109.27
OH
N
O
O
3s
Chemical Shift (ppm)
210.80
171.06
135.57
130.35
125.12
124.67
123.67
120.83
109.27
62.24
41.33
39.57
34.72
26.45
18.63
Chemical Shift (ppm)

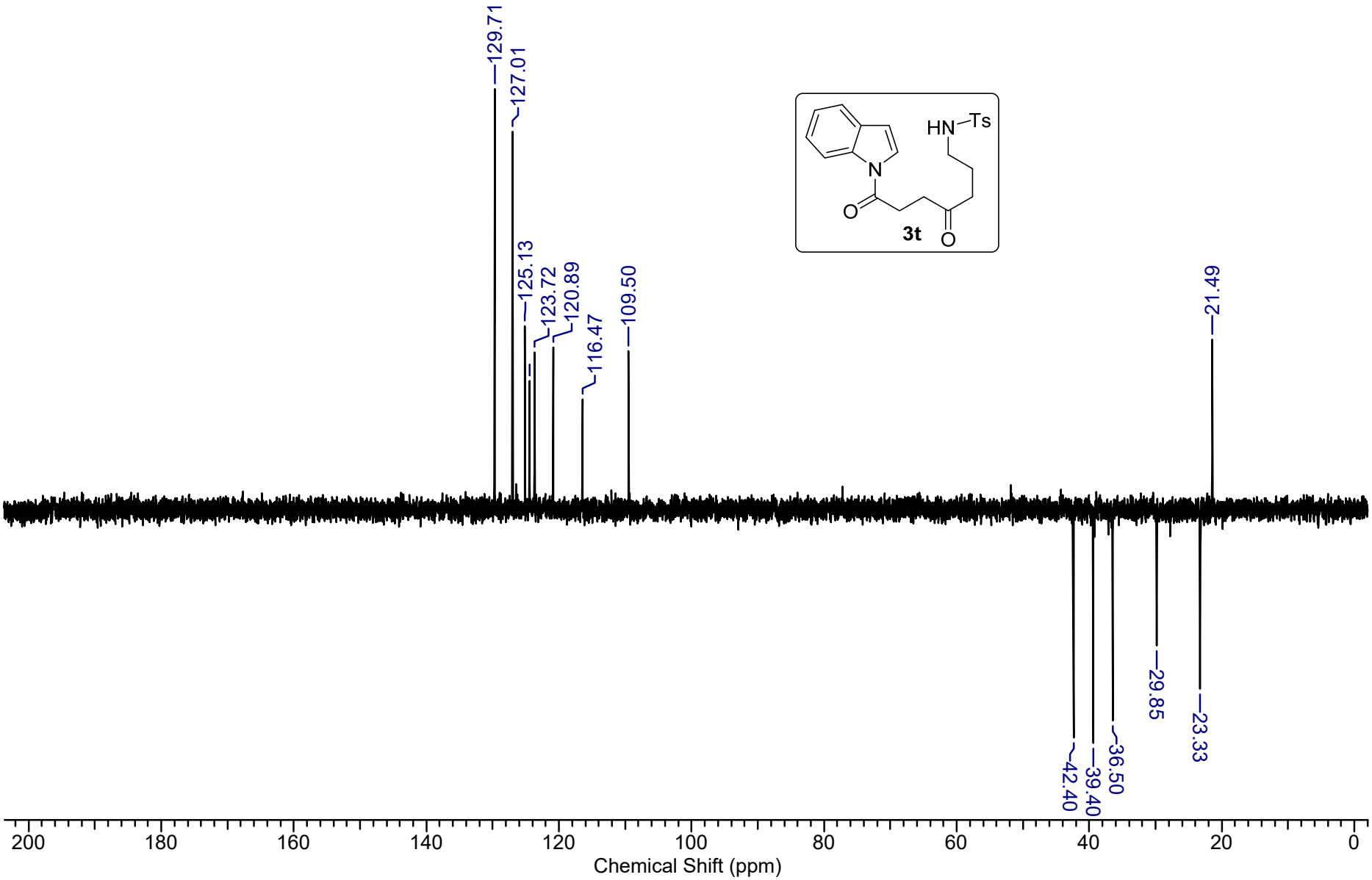

125.12
123.67
120.83
116.55
109.27
OH
N
O
O
3s
62.24
41.33
39.57
34.72
26.45
18.63
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MSH-59 #322 RT: 1.75 AV: 1 NL: 1.86E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]




HN
Ts
N
O
O
3t
CHLOROFORM-d
8.39
8.37
7.75
7.73
7.72
7.53
7.52
7.32
7.30
7.27
7.26
7.26
6.67
6.66
4.73
4.72
4.70
3.25
3.27
3.24
3.00
2.98
3.02
2.91
2.90
2.69
2.67
2.41
1.86
1.84
1.83
1.81
1.79
1.00 2.32 2.12 1.24 3.12 1.05
1.02
2.12 2.27 2.15 2.41 3.08 2.07
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


CHLOROFORM-d
77.32
77.00
76.68
HN
Ts
N
O
O
O
3t
208.71
170.29
143.38
136.96
130.34
129.71
127.01
125.14
124.45
123.73
120.89
109.49
42.40
39.40
36.49
29.84
23.32
21.49
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


129.71
127.01
125.13
123.72
120.89
116.47
109.50
21.49
42.40
39.40
36.50
29.85
23.33
HN
Ts
N
O
O
O
3t
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

MS-18 #253 RT: 1.35 AV: 1 NL: 6.78E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]