Electronic Supplementary Information

Pd-catalyzed regioselective C-H halogenation of quinazolinones and benzoxazinones

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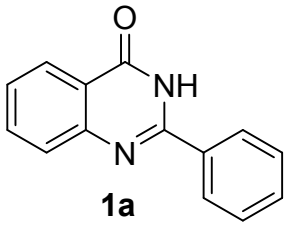
Experimental section

General

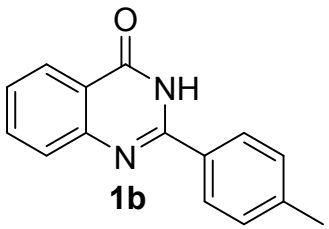
All starting materials were obtained from Merck Millipore or Sigma-Aldrich, and were used without further purification. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Mass spectra were recorded on an Agilent Technology (HP) 5973 Network Mass Selective Detector operating at an ionization potential of 70 eV. IR spectra were recorded on a Bomem MB-Series FT-IR spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKERDRX-300AVANCEspectrometer at 300 and 75 MHz and 500AVANCEspectrometer at 500 and 126 MHz, respectively. ^{1}H and ^{13}CNMR spectra were obtained in DMSO-d_6 using TMS as internal standard. Elemental analyses were performed using a Heraeus CHN-O Rapid analyzer.

Procedure for the Synthesis of quinazolin-4(3*H*)-one derivatives

Anthranilamide (15 mmol) and an aldehyde (18 mmol) were dissolved in DMSO (40 mL). Then, the reaction mixture was stirred at 100 ^{o}C in an open flask and monitored by TLC. After complete consumption of the starting materials, the reaction mixture was cooled to room temperature. When water (100 mL) was added to the reaction mixture, the precipitate was formed and collected by filtration. Recrystallization in ethanol afforded quinazolinone.[S1]

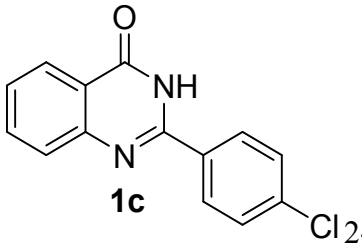


1a 2-Phenylquinazolin-4(3*H*)-one (1a) White solid, Yield: 84%, mp: 240-242 °C (lit.[S2] mp: 235-236 °C); ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.56 (br s, 1H), 8.20 - 8.15 (m, 3H), 7.91 – 7.80 (m, 1H), 7.75 (d, J = 8.2 Hz, 1H), 7.60 - 7.50 (m, 4H).

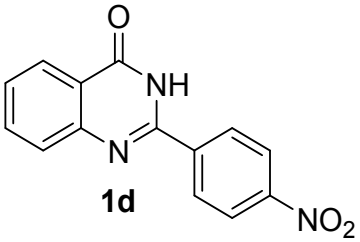


1b 2-(*p*-Tolyl)quinazolin-4(3*H*)-one (1b) White solid, Yield: 78%, mp: 235-237 °C (lit.[S2] mp: 237-238 °C); ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.47 (br s, 1H), 8.09-8.16

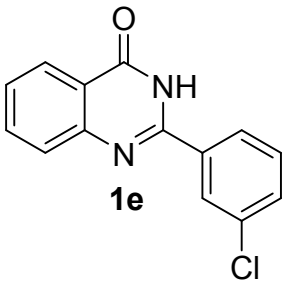
(m, 3H), 7.86 – 7.79 (m, 1H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 2H), 2.39 (s, 3H).



2-(4-Chlorophenyl)quinazolin-4(3*H*)-one (1c) White solid, Yield: 65%, mp: 305-307 °C (lit.[S3] mp: 306-308 °C); ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.61 (br s, 1H), 8.26 – 8.11 (m, 3H), 7.83 (d, *J* = 7.5 Hz, 1H), 7.74 (d, *J* = 7.4 Hz, 1H), 7.67 – 7.58 (m, 2H), 7.52 (d, *J* = 7.3 Hz, 1H).



2-(4-Nitrophenyl)quinazolin-4(3*H*)-one (1d) White solid, Yield: 52%, mp: 361-363 °C (lit.[S3] mp: 362-364 °C); ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.78 (br s, 1H), 8.44 – 8.35 (m, 4H), 8.26 -8.17 (m, 1H), 7.90 – 7.73 (m, 2H), 7.58 (t, *J* = 7.7 Hz, 1H).

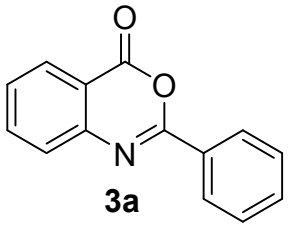


2-(3-Chlorophenyl)quinazolin-4(3*H*)-one (1e) White solid, Yield: 56%, mp: 292-295 °C (lit.[S4] mp: 292-294 °C); ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.53 (s, 1H), 8.15 – 8.21 (m, 3H), 7.91 – 7.71 (m, 2H), 7.50-7.64 (m, 3H).

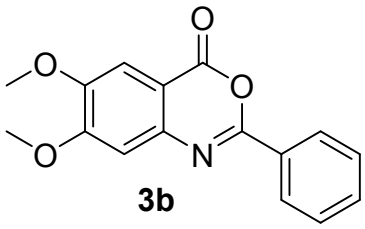
Procedure for the Synthesis of benzo[*d*][1,3]oxazin-4-one derivatives

To a stirred solution of anthranilic acid derivative (15 mmol) in pyridine (50 mL), arenoyl chloride (15 mmol) was added drop wise, maintaining the temperature near 0-6 °C for 1 h. The reaction

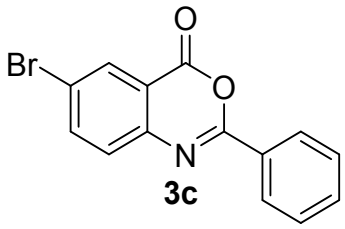
mixture was stirred for another 3 h at room temperature until a solid product was separated. The reaction mixture was neutralized with saturated sodium bicarbonate solution and the pale yellow solid which separated was filtered, washed with water and recrystallised from ethanol.[S5]



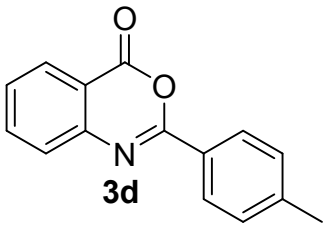
3a 2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (3a) White solid, Yield: 89%, mp: 121-122 °C (lit.[S5a] mp: 123-125 °C). ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.28 – 8.08 (m, 3H), 7.99 – 7.89 (m, 1H), 7.77 – 7.52 (m, 5H).



3b 6,7-dimethoxy-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (3b) White solid, Yield: 80%, mp: 198-201 °C (lit.[S6] mp: 197-198 °C); ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.18 – 8.08 (m, 2H), 7.66 – 7.54 (m, 2H), 7.46 – 7.38 (m, 2H), 7.17 – 7.16 (m, 1H), 3.94 (s, 3H), 3.89 (s, 3H)

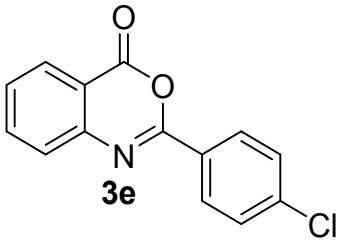


3c 6-bromo-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (3c) White solid, Yield: 72%, mp: 196-197 °C (lit.[S7] mp: 200-201 °C). ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.28 – 8.16 (m, 3H), 8.12 – 8.08 (m, 1H), 7.75 – 7.55 (m, 4H).



3d 2-(p-tolyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (3d) White solid, Yield: 69%, mp: 152-154 °C (lit.[S7] mp: 155-156 °C). ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.14 – 8.06 (m, 3H),

7.94 (t, *J* = 7.7, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 2.40 (s, 3H).



2-(4-chlorophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (3e)

White solid, Yield: 81%, mp: 191-193 °C (lit.[S7] mp: 192-193 °C).^{1}H NMR (300 MHz,) δ 8.25 – 8.11 (m, 3H), 7.99 – 7.93 (m, 1H), 7.77 – 7.58 (m, 4H).

Procedure for the Preparation of *N*-Iodosuccinimide (NIS).

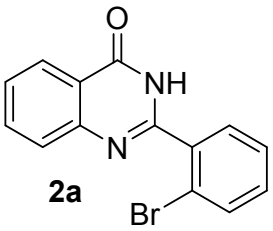
NaI (10 mmol) and NCS (10 mmol) were individually dissolved in acetone (25 mL). The two solutions were mixed in a 100 mL round-bottomed flask equipped with a magnetic stirring bar. After it stirred for 15 min, the NaCl formed during the course of reaction was filtered; the filtrate was concentrated under reduced pressure. To remove iodine from the crude product, the solid was washed several times with 15 mL portions of diethyl ether until a bright yellow-colored powder was obtained. The NIS produced using this procedure was used without further purification.[S8] White solid, Yield: 89%; mp. = 197−200 °C [lit.[S8] mp: 196-198 °C].

General procedure for the halogenation of quinazolinones and benzoxazinones

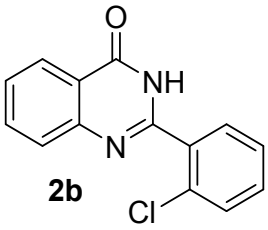
To a 10 mL single-neck round-bottom flask equipped with a magnetic stir bar were added the quinazolinones or benzoxazinones (1 mmol), NXS (1.2 mmol), *p*-TsOH.H_2O (0.5 mmol) and $Pd(OAc)_2$ (0.1 mmol,) in turn. Subsequently, the solvent (DCE, 3 mL) was added. The reaction mixture was stirred at 100 °C, and the completion of the reaction was monitored using TLC. After the reaction had been completed, the solvent was evaporated under vacuum. The residue diluted with ethyl acetate (2 × 10 mL) and the organic layer was further washed with brine solution. The organic layers were dried over Na_2SO_4, filtered, and concentrated. Finally, the halogenated product was purified by thin layer chromatography to afford the desired pure coupling product.

Procedure for Gram-Scale Reaction.

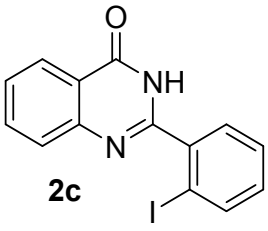
2-phenylquinazolin-4(3*H*)-one (7 mmol, 1.54 g), NBS (8.4 mmol, 1.49 g), *p*-TsOH.H_2O (3.5 mmol, 0.67 g), and $Pd(OAc)_2$ (0.7 mmol, 0.16 g) were added to a balloon equipped with a magnetic stirring bar followed by the addition of DCE (15 mL). The reaction mixture was stirred at 100 °C and the completion of the reaction was monitored using TLC (*n*-hexane). After the reaction was completed, the solvent was evaporated by vacuum. The residue diluted with ethyl acetate (2 × 20 mL) and the organic layer was further washed with brine solution. Afterward, the organic layer was separated and dried over anhydrous Na_2SO_4 and concentrated under reduced pressure using a rotary evaporator. Purification was accomplished using column chromatography using silica gel as the stationary phase (*n*-hexane).



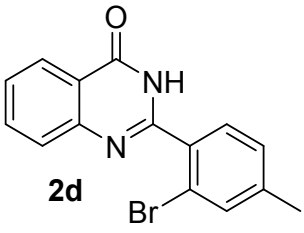
2a 2-(2-Bromophenyl)quinazolin-4(3*H*)-one (2a) White solid, Yield: 76%, mp: 171-173 °C (lit.[S9] mp: 175-179 °C); IR (KBr) (ν_{max} /cm^{-1}): 1672, 1602, 1469, 1296; ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.63 (br s, 1H), 8.19 (d, J = 8.0 Hz, 1H), 7.87 (t, J = 7.8 Hz, 1H), 7.78 (d, J = 7.9 Hz, 1H), 7.72 (d, J = 8.2 Hz, 1H), 7.66 – 7.64 (m, 1H), 7.62 – 7.46 (m, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 161.90, 153.81, 149.04, 136.34, 135.05, 133.11, 132.13, 131.25, 128.14, 127.95, 127.51, 126.31, 121.75, 121.45; MS (m/z): 300 (M^+); Anal. Calcd for $C_{14}H_9BrN_2O$: C, 55.84; H, 3.01; N, 9.30. Found: C, 56.03; H, 3.09; N, 9.21.



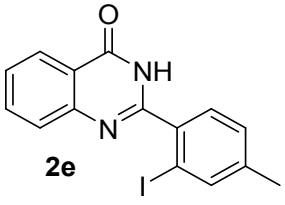
2b 2-(2-Chlorophenyl)quinazolin-4(3*H*)-one (2b) White solid, Yield: 63%, mp: 167-169 °C (lit.[s9] mp: 172-174 °C); IR (KBr) (ν_{max} /cm^{-1}): 1673, 1605, 1434, 1295; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.67 (br s, 1H), 8.18 (d, J = 7.8 Hz, 1H), 7.87 (d, J = 7.4 Hz, 1H), 7.73 – 7.46 (m, 5H); ^{13}C NMR (126 MHz, , DMSO-d_6) δ 162.21, 153.33, 148.80, 135.84, 134.22, 132.86, 132.35, 130.52, 129.57, 129.06, 128.17, 126.81, 122.03, 121.59; MS (m/z): 255 (M^+-1); Anal. Calcd for $C_{14}H_9ClN_2O$: C, 65.51; H, 3.53; N, 10.91. Found: C, 65.68; H, 3.59; N, 11.02.



2c 2-(2-Iodophenyl)quinazolin-4(3*H*)-one (2c) White solid, Yield: 79%, mp: 213-215 °C (lit.[S10] mp: 217-219 °C); IR (KBr) (ν_{max} /cm^{-1}): 1660, 1606, 1465, 1415; ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.69 (br s, 1H), 8.21 (d, J = 7.8 Hz, 1H), 8.01 – 7.99 (m, 2H), 7.92 – 7.54 (m, 4H), 7.00 (t, J = 7.9 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 162.09, 157.10, 149.01, 138.77, 135.14, 133.27, 133.19, 132.63, 128.11, 127.77, 126.32, 124.66, 121.91, 97.21; MS (m/z): 347 (M$^+$-1); Anal. Calcd for $C_{14}H_9IN_2O$: C, 48.30; H, 2.61; N, 8.05. Found: C, 48.45; H, 2.71; N, 8.00.

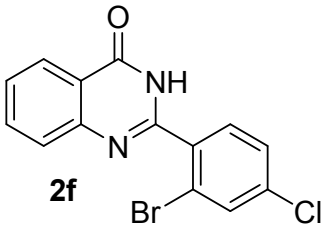


2d 2-(2-Bromo-4-methylphenyl)quinazolin-4(3*H*)-one (2d) White solid, Yield: 83%, mp: 175-177 °C IR (KBr) (ν_{max} /cm^{-1}): 1656, 1599, 1461, 1295; ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.56 (br s, 1H), 8.18 (dd, J = 7.9, 1.9 Hz, 1H), 7.89 – 7.82 (m, 1H), 7.74 – 7.65 (m, 1H), 7.64 – 7.49 (m, 3H), 7.34 (dd, J = 7.9, 1.6 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 162.26, 154.24, 149.50, 142.73, 135.45, 133.77, 132.95, 131.43, 129.13, 128.35, 127.86, 126.71, 122.10, 121.63, 21.36; MS (m/z): 314 (M$^+$); Anal. Calcd for $C_{15}H_{11}BrN_2O$: C, 57.16; H, 3.52; N, 8.89. Found: C, 57.34; H, 3.57; N, 8.83.

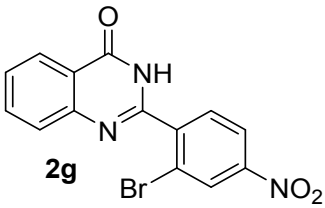


2e 2-(2-iodo-4-methylphenyl)quinazolin-4(3*H*)-one (2e) White solide, Yield: 76%, mp: 224-226 °C; IR (KBr) (ν_{max} /cm^{-1}): 1674, 1602, 1550, 1466, 1289; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.43 (br s, 1H), 8.15 – 8.08 (m, 2H), 7.82 (t, J = 7.6 Hz, 1H), 7.72 (d, J = 8.1 Hz, 1H), 7.50 (t, J = 7.6 Hz, 1H), 7.35 (d, J = 7.8 Hz, 2H), 2.39 (s, 3H).^{13}C NMR (75 MHz, DMSO-d_6) δ 161.89, 152.25, 147.36, 140.03, 135.18, 132.56, 132.16, 132.00, 130.15, 128.93, 127.59,

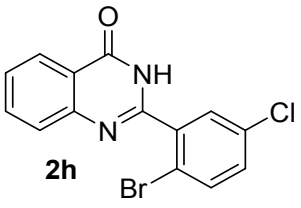
126.30, 125.07, 121.51, 20.17. MS (m/z): 362 (M^+); Anal. Calcd for $C_{15}H_{11}IN_2O$: C, 49.75; H, 3.06; N, 7.74. Found: C, 49.98; H, 3.10; N, 7.71.



2f 2-(2-Bromo-4-chlorophenyl)quinazolin-4(3*H*)-one (2f) White solid, Yield: 75%, mp: 264-266 °C; IR (KBr) (ν_{max} /cm^{-1}): 1676, 1607, 1469, 1296; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.62 (br s, 1H), 8.26 - 8.15 (m, 1H), 7.98 – 7.79 (m, 2H), 7.79 – 7.48 (m, 4H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 161.86, 152.96, 148.94, 135.82, 135.26, 135.11, 132.53, 132.44, 128.33, 127.97, 127.67, 126.32, 122.42, 121.77; MS (m/z): 336 (M^+); Anal. Calcd for $C_{14}H_8BrClN_2O$: C, 50.11; H, 2.40; N, 8.35. Found: C, 50.36; H, 2.49; N, 8.26.

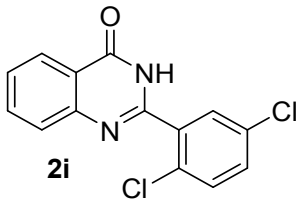


2g 2-(2-Bromo-4-nitrophenyl)quinazolin-4(3*H*)-one (2g) White solid, Yield: 51%, mp: 255-266 °C; IR (KBr) (ν_{max} /cm^{-1}): 1677, 1608, 1525, 1469, 1350; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.81 (br s, 1H), 8.65 - 8.59 (m, 1H), 8.38 (dd, J = 8.4, 2.3 Hz, 1H), 8.24 – 8.18 (m, 1H), 7.97 (d, J = 8.4 Hz, 1H), 7.93 – 7.85 (m, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.62 (t, J = 7.4 Hz, 1H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 166.12, 160.97, 149.54, 149.21, 135.66, 132.92, 128.47, 128.26, 127.51, 126.81, 124.13, 123.63, 122.78, 122.35; MS (m/z): 345 (M^+); Anal. Calcd for $C_{14}H_8BrN_3O_3$: C, 48.58; H, 2.33; N, 12.14. Found: C, 48.73; H, 2.38; N, 12.04.



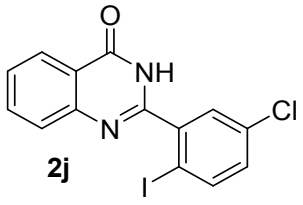
2h 2-(2-Bromo-5-chlorophenyl)quinazolin-4(3*H*)-one (2h) White solid, Yield: 64%, mp: 227-229 °C IR (KBr) (ν_{max} /cm^{-1}): 1674, 1606, 1468, 1295; ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.65 (br s, 1H), 8.22 – 8.16 (m, 1H), 7.91 – 7.77 (m, 3H), 7.72 (dt, J = 7.9, 3.8 Hz, 1H), 7.62 – 7.54 (m, 2H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 162.09, 157.02, 149.30, 138.26, 135.55, 135.22, 132.47, 132.35, 131.42, 130.02, 128.41, 126.74, 122.28, 119.18; MS (m/z): 336

(M^+); Anal. Calcd for $C_{14}H_8BrClN_2O$: C, 50.11; H, 2.40; N, 8.35. Found: C, 50.32; H, 2.46; N, 8.26.



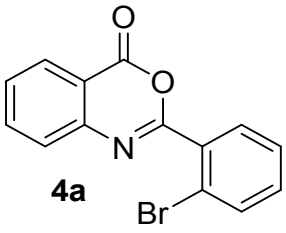
2i

2-(2,5-Dichlorophenyl)quinazolin-4(3*H*)-one (2i) White solid, Yield: 50%, mp: 220-225 °C; IR (KBr) (ν_{max} /cm^{-1}): 1695, 1606, 1554, 1468, 1295; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.71 (br s, 1H), 8.19 (d, J = 8.0 Hz, 1H), 7.94 – 7.50 (m, 6H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 162.25, 151.91, 149.10, 136.01, 135.58, 132.68, 132.31, 132.21, 131.44, 131.27, 128.50, 128.21, 126.77, 122.18; MS (m/z): 290 (M^+); Anal. Calcd for $C_{14}H_8Cl_2N_2O$: C, 57.76; H, 2.77; N, 9.62. Found: C, 57.89; H, 2.81; N, 9.59.



2j

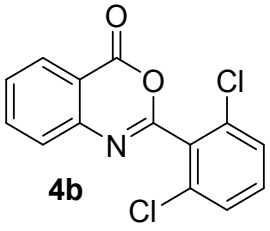
2-(5-chloro-2-iodophenyl)quinazolin-4(3*H*)-one (2j) White solide, Yield: 55%, mp: 218-220 °C; IR (KBr) (ν_{max} /cm^{-1}): 1670, 1606, 1464, 1247; ^{1}H NMR (300 MHz,) δ 12.92 (br s, 1H), 8.09 (d, J = 7.9 Hz, 1H), 7.88 (s, 1H), 7.77 (t, J = 7.6 Hz, 1H), 7.64 – 7.45 (m, 3H), 7.21 (d, J = 8.3 Hz, 1H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 160.42, 152.09, 147.11, 136.08, 133.97, 133.88, 132.16, 130.98, 129.47, 128.72, 126.36, 126.03, 125.08, 120.10. MS (m/z): 381 (M^+-1); Anal. Calcd for $C_{14}H_8ClIN_2O$: C, 43.95; H, 2.11; N, 7.32. Found: C, 44.12; H, 2.13; N, 7.30.



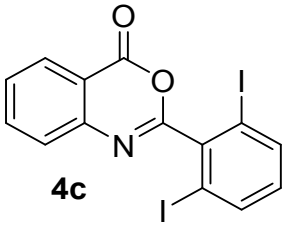
4a

2-(2-Bromophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (4a) White solid, Yield: 61%, mp: 127-130 °C (lit.[S11] mp: 118 °C); IR (KBr) (ν_{max} /cm^{-1}): 1759, 1617, 1593, 1435; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.26 – 8.23 (m, 3H), 8.18 – 8.13 (m, 1H), 7.71 (dd, J = 8.5, 6.0 Hz, 1H), 7.68 – 7.58 (m, 2H), 7.56 – 7.49 (m, 1H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 159.31, 157.94, 145.02, 140.79, 134.04, 130.80, 130.26, 130.02, 128.94, 128.89, 128.58, 122.09, 119.89, 114.14;

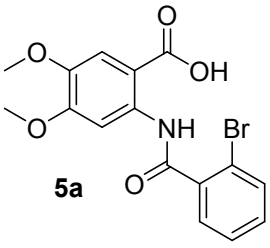
MS (m/z): 301 (M^+); Anal. Calcd for $C_{14}H_8BrNO_2$: C, 55.66; H, 2.67; N, 4.64. Found: C, 55.72; H, 2.76; N, 4.58.



4b 2-(2,6-Dichlorophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (4b) White solid, Yield: 45%, mp: 161-162 °C (lit.[S12] mp: 167-169 °C); IR (KBr) (ν_{max} /cm^{-1}): 1779, 1680, 1600, 1435; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.67 (d, J = 8.9 Hz, 1H), 8.02 (d, J = 6.9 Hz, 2H), 7.64 – 7.50 (m, 3H), 7.12 (d, J = 8.0 Hz, 1H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 160.21, 157.19, 147.78, 137.61, 131.70, 130.52, 130.44, 129.19, 127.92, 122.77, 119.13; MS (m/z): 291 (M^+). Anal. Calcd for $C_{14}H_7Cl_2NO_2$: C, 57.56; H, 2.42; N, 4.80. Found: C, 57.64; H, 2.44; N, 4.71.

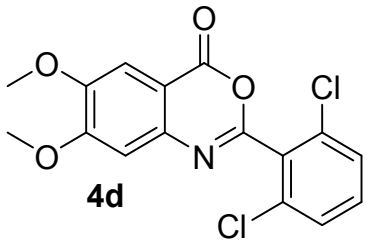


4c 2-(2-iodophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (4c) White solid, Yield: 49%, mp: 175-180 °C; IR (KBr) (ν_{max} /cm^{-1}): 1753, 1643, 1600, 1099; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.33 – 8.26 (m, 1H), 8.13 – 8.07 (m, 1H), 8.04 (d, J = 8.1 Hz, 2H), 7.88 – 7.77 (m, 2H), 7.08 (t, J = 7.9 Hz, 1H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 159.84, 159.62, 146.19, 142.06, 139.29, 138.86, 134.49, 131.26, 129.42, 128.40, 117.06, 97.24. MS (m/z): 475 (M^+). Anal. Calcd for $C_{14}H_7I_2NO_2$: C, 35.40; H, 1.49; N, 2.95. Found: C, 35.49; H, 1.51; N, 2.94.

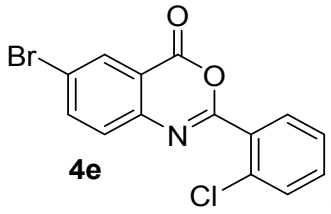


5a 2-(2-Bromobenzamido)-4,5-dimethoxybenzoic acid (5a) White solid, Yield: 41%, mp: 275-280 °C decomp. IR (KBr) (ν_{max} /cm^{-1}): 1693, 1674, 1657, 1607, 1525, 1481; ^{1}H NMR (500 MHz, DMSO-d_6) δ 11.69 (br s, 1H), 8.41 (s, 1H), 7.76 (d, J = 7.9 Hz, 1H), 7.69 – 7.63 (m, 1H), 7.58 – 7.43 (m, 3H), 3.88 (s, 3H), 3.79 (s, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 170.22, 166.20, 154.14, 144.93, 139.25, 137.16, 134.26, 132.86, 129.80, 129.04, 119.59, 113.79, 108.90,

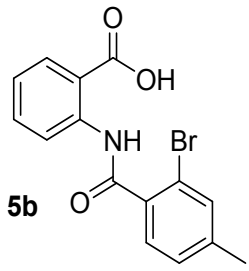
104.02, 56.57, 56.54; MS (m/z): 379 (M^+); Anal. Calcd for $C_{16}H_{14}BrNO_5$: C, 50.55; H, 3.71; N, 3.68. Found: C, 55.78; H, 3.79; N, 3.62.



2-(2,6-Dichlorophenyl)-6,7-dimethoxy-4*H*-benzo[*d*][1,3]oxazin-4-one (4d) White solid, Yield: 43%, mp: 206-210 °C; IR (KBr) (ν_{max} /cm^{-1}): 1743, 1609, 1509, 1289, ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.65-7.73 (m, 3H), 7.55 (s, 1H), 7.35 (s, 1H), 3.97 (s, 3H), 3.95 (s, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 159.03, 157.59, 153.44, 153.39, 151.33, 142.28, 134.08, 131.39, 129.50, 109.70, 109.57, 108.35, 57.42, 57.09; MS (m/z): 351 (M^+); Anal. Calcd for $C_{16}H_{11}Cl_2NO_4$: C, 54.57; H, 3.15; N, 3.98. Found: C, 54.71; H, 3.21; N, 3.93.

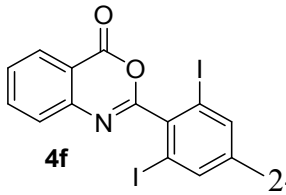


6-Bromo-2-(2-chlorophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (4e) White solid, Yield: 54%, mp: 188-191 °C; IR (KBr) (ν_{max} /cm^{-1}): 1767, 1608, 1573, 1451; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.38 (d, J = 2.3 Hz, 1H), 8.28 – 8.17 (m, 3H), 7.75 – 7.58 (m, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 160.00, 158.18, 143.26, 139.48, 134.26, 132.69, 130.53, 130.08, 129.96, 129.00, 125.42, 121.22, 120.91; MS (m/z): 335 (M^+); Anal. Calcd for $C_{14}H_7BrClNO_2$: C, 49.96; H, 2.10; N, 4.16. Found: C, 50.19; H, 2.17; N, 4.23.



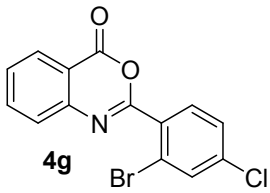
2-(2-bromo-4-methylbenzamido)benzoic acid (5b) White solid, Yield: 46%, mp: 168-170 °C; IR (KBr) (ν_{max} /cm^{-1}): 1742, 1641, 1506, 1380; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.17 – 8.02 (m, 2H), 7.99 – 7.87 (m, 1H), 7.77 – 7.52 (m, 2H), 7.44 – 7.37 (m, 2H), 2.41 (s, 3H);^{13}C NMR (126 MHz, DMSO-d_6) δ 171.47, 166.25, 142.78, 136.89, 134.39, 133.13, 132.20,

130.24, 129.47, 128.03, 123.02, 120.15, 119.95, 119.66, 21.89; MS (m/z): 334 (M^+); Anal. Calcd for $C_{15}H_{12}BrNO_3$: C, 53.91; H, 3.62; N, 4.19. Found: C, 54.16; H, 3.67; N, 4.15.



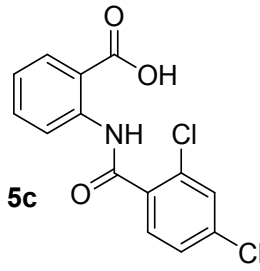
4f 2-(2-iodo-4-methylphenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (4f)

White solid, Yield: 51%, mp: 168-170 °C; IR (KBr) (ν_{max} /cm^{-1}): 1761, 1643, 1593, 1518, 1468; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.29 (dd, J = 7.9, 1.5 Hz, 1H), 8.12 – 8.04 (m, 1H), 7.89 (s, 2H), 7.79 (t, J = 7.6 Hz, 1H), 7.46 (d, J = 8.2 Hz, 1H), 2.32 (s, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 159.91, 159.72, 146.24, 144.98, 139.42, 138.77, 131.17, 129.64, 129.37, 128.37, 117.04, 96.78, 20.59; MS (m/z): 489 (M^+); Anal. Calcd for $C_{15}H_9I_2NO_2$: C, 36.84; H, 1.86; N, 2.86. Found: C, 36.95; H, 1.90; N, 2.82.



4g 2-(2-bromo-4-chlorophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (4g)

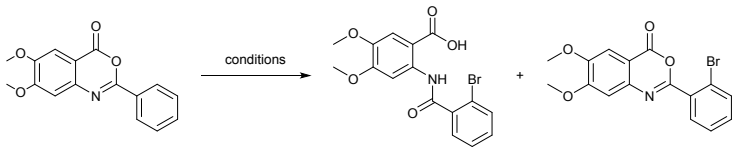
White solid, Yield: 72%, mp: 147-150 °C; IR (KBr) (ν_{max} /cm^{-1}): 1703, 1581, 1506, 1384; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.58 (d, J = 8.2 Hz, 1H), 8.27 – 8.17 (m, 1H), 7.88 (d, J = 1.8 Hz, 1H), 7.68 – 7.56 (m, 2H), 7.46 – 7.39 (m, 1H), 7.08 (t, J = 7.4 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 159.18, 156.95, 144.26, 140.42, 138.52, 135.45, 132.94, 131.95, 131.69, 130.40, 128.58, 123.61, 120.33, 119.18, MS (m/z): 335 (M^+); Anal. Calcd for $C_{14}H_7BrClNO_2$: C, 49.96; H, 2.10; N, 4.16. Found: C, 50.14; H, 2.15; N, 4.19.



5c 2-(2,4-dichlorobenzamido)benzoic acid (5c)

White solid, Yield: 45%, mp: 117-119 °C; IR (KBr) (ν_{max} /cm^{-1}): 1703, 1581, 1506, 1384; ^{1}H NMR (300 MHz, DMSO-d_6) δ 13.96 (br s, 1H), 11.11 (br s, 1H), 8.70 – 8.66 (m, 1H), 8.03 – 7.99 (m, 2H), 7.65 – 7.51 (m, 2H), 7.50 – 7.38 (m, 1H), 7.12 – 7.05 (m, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 171.80, 163.62, 141.25, 140.72, 136.96, 134.41, 131.83, 130.63, 130.11, 129.48, 129.26, 122.58, 119.17; MS (m/z): 310 (M^+); Anal. Calcd for $C_{14}H_9Cl_2NO_3$: C, 54.22; H, 2.93; N, 4.52. Found: C, 54.38; H, 2.98; N, 4.49.

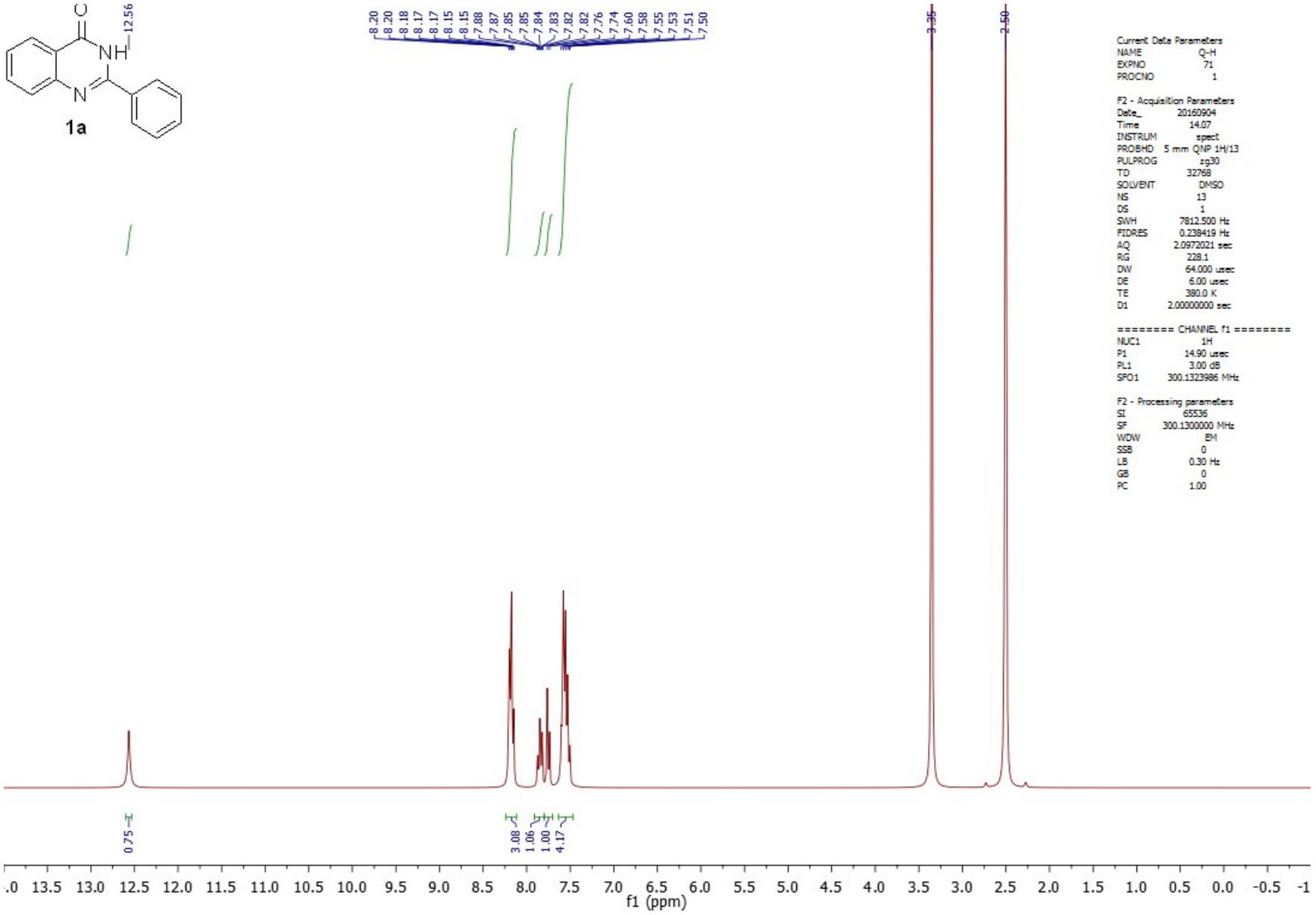
Table S1. Optimization of the reaction conditions[a]



3b 5a 4i

Entry	Solvent	Temp.	Time	Yield[b] 5a	Yield[b] 4i
1	DCE	100	4	41%	Trace
2	CH_2Cl_2	100	4	Trace	Trace
3	CH_3CN	100	4	10%	Trace
4	$PhCH_3$	100	4	Trace	Trace
5	DCE	80	4	26%	Trace
6	DCE	60	4	Trace	Trace
7	DCE	100	2	25%	Trace

[a] 6,7-dimethoxy-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (1 mmol), NBS (1.2 mol), $Pd(OAc)_2$ (10 mol %), *p*-TsOH (0.5 mmol). and solvent (3 mL); [b] Isolated yield.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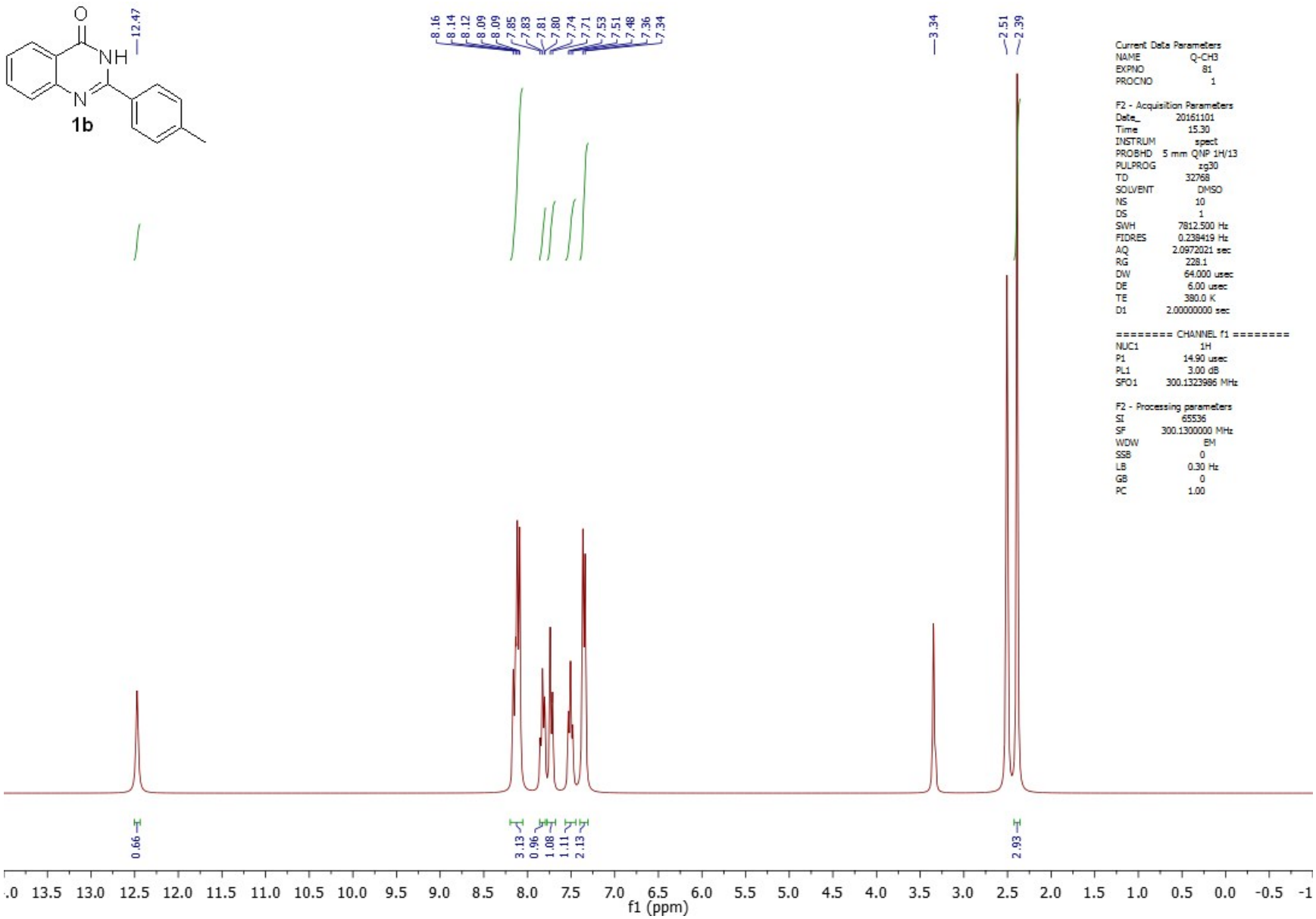
12.56
8.20
8.20
8.18
8.17
8.17
8.15
8.15
7.88
7.87
7.85
7.85
7.84
7.83
7.82
7.82
7.76
7.74
7.60
7.58
7.55
7.53
7.51
7.50
3.35
2.50
Current Data Parameters
NAME Q-H
EXPNO 71
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160904
Time 14.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 13
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.75
3.08
1.06
1.00
4.17
f1 (ppm)


1b
12.47
8.16
8.14
8.12
8.09
8.09
7.85
7.83
7.81
7.80
7.74
7.71
7.53
7.51
7.48
7.36
7.34
3.34
2.51
2.39
Current Data Parameters
NAME Q-CH3
EXPNO 81
PROCNO 1
F2 - Acquisition Parameters
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Time 15.30
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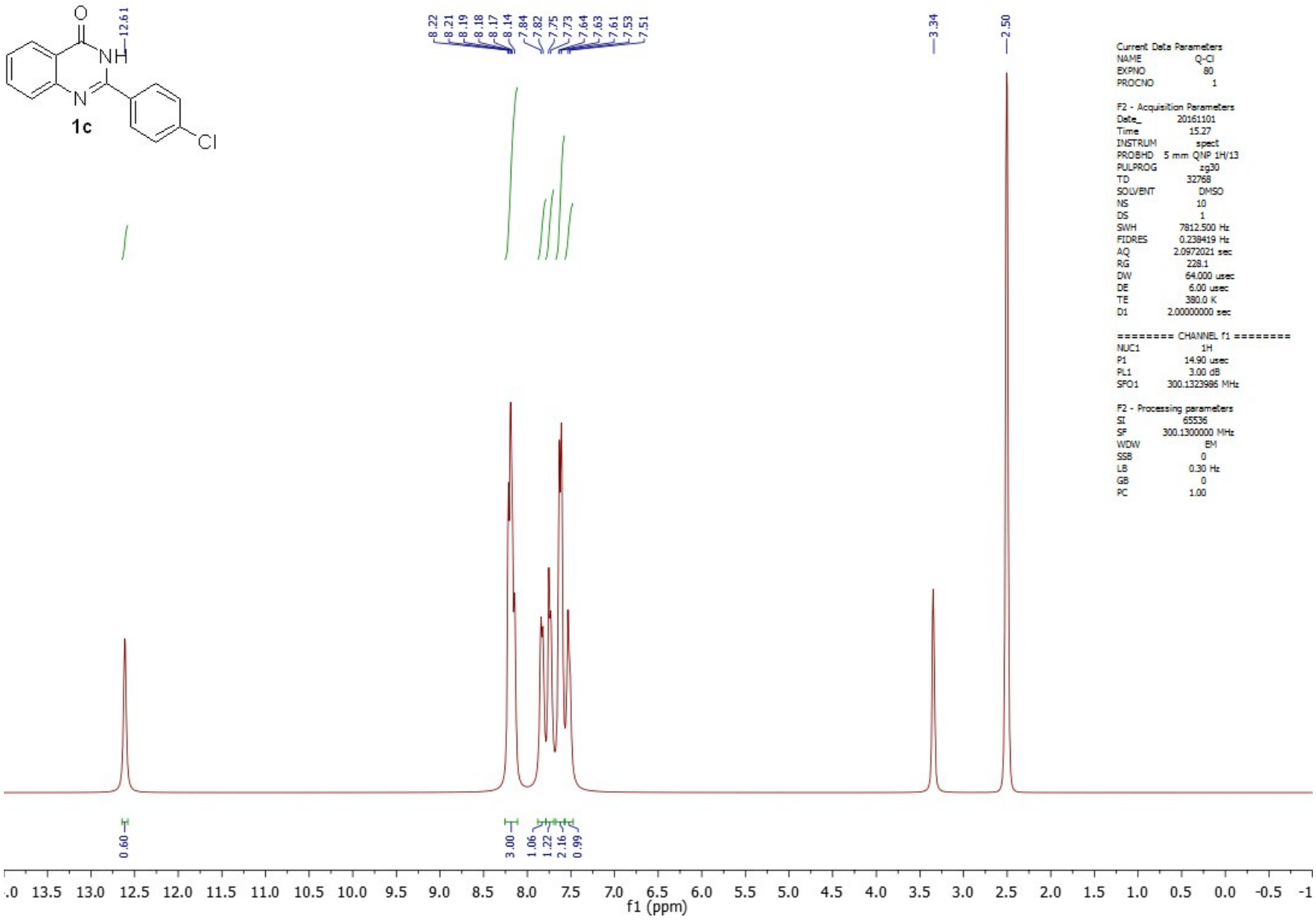
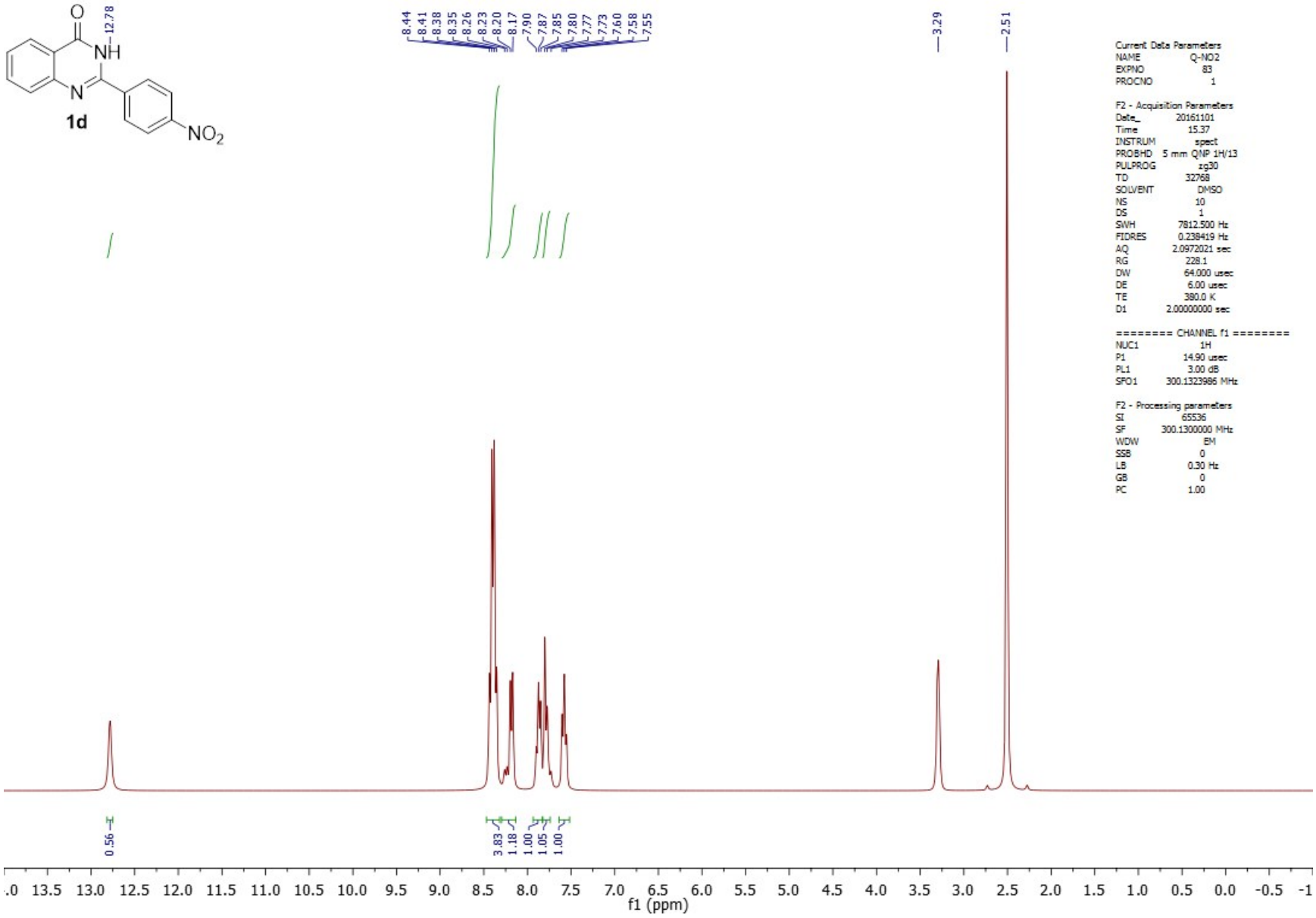
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.66
3.13
0.96
1.08
1.11
2.13
2.93
f1 (ppm)


1c
12.61
8.22
8.21
8.19
8.18
8.17
8.14
7.84
7.82
7.75
7.73
7.64
7.63
7.61
7.53
7.51
3.34
2.50
Current Data Parameters
NAME Q-Cl
EXPNO 80
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161101
Time 15.27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.60
3.00
1.06
1.22
2.16
0.99
f1 (ppm)


1d
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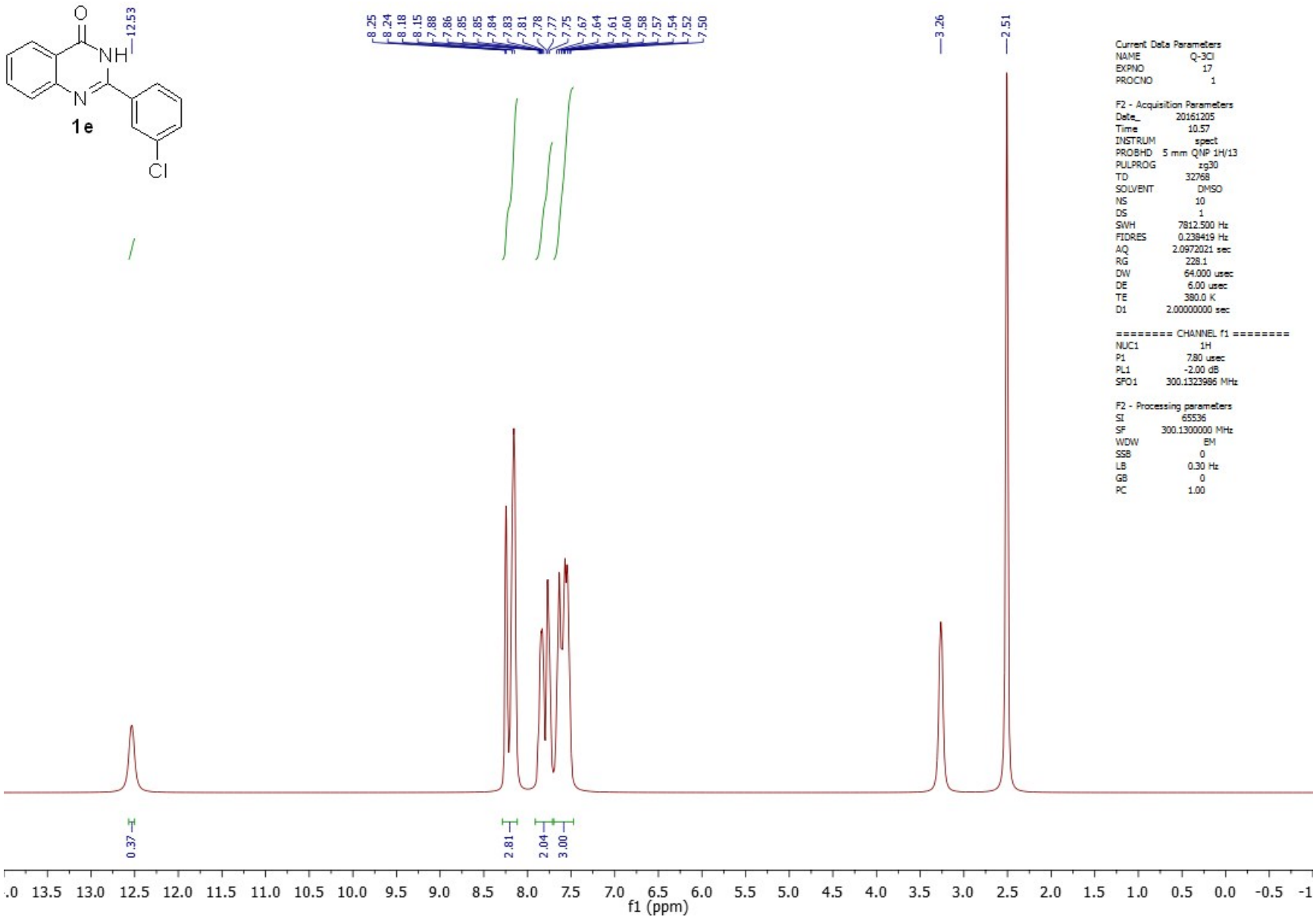
8.38
8.35
8.26
8.23
8.20
8.17
7.90
7.87
7.85
7.80
7.77
7.73
7.60
7.58
7.55
3.29
2.51
Current Data Parameters
NAME Q-NO2
EXPNO 83
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161101
Time 15.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.56
3.83
1.18
1.00
1.05
1.00
f1 (ppm)


1e
12.53
8.25
8.24
8.18
8.15
7.88
7.86
7.85
7.85
7.84
7.83
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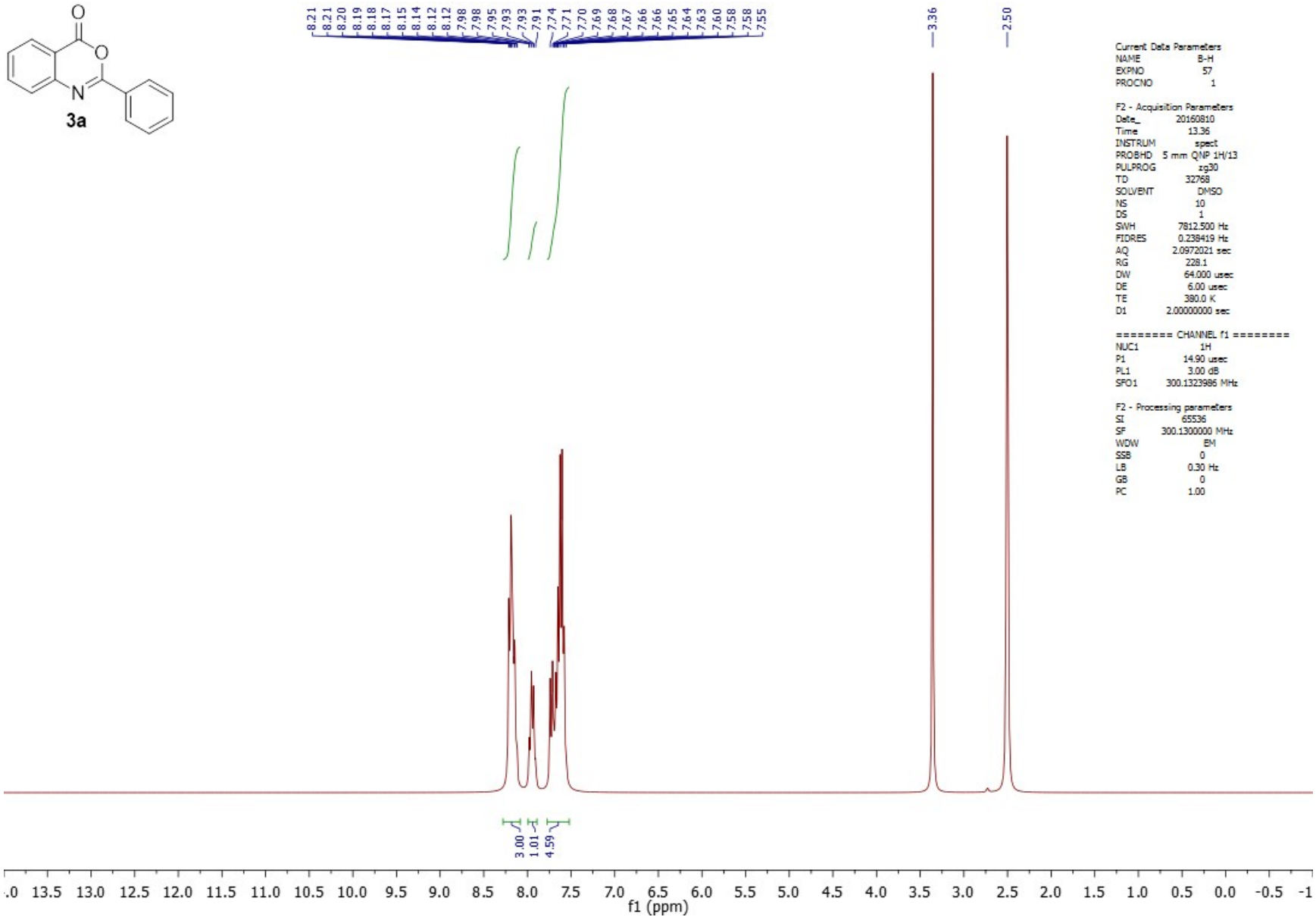
7.78
7.77
7.75
7.67
7.64
7.61
7.60
7.58
7.57
7.54
7.52
7.50
3.26
2.51
Current Data Parameters
NAME Q-3Cl
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161205
Time 10.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.37
2.81
2.04
3.00
f1 (ppm)


3a
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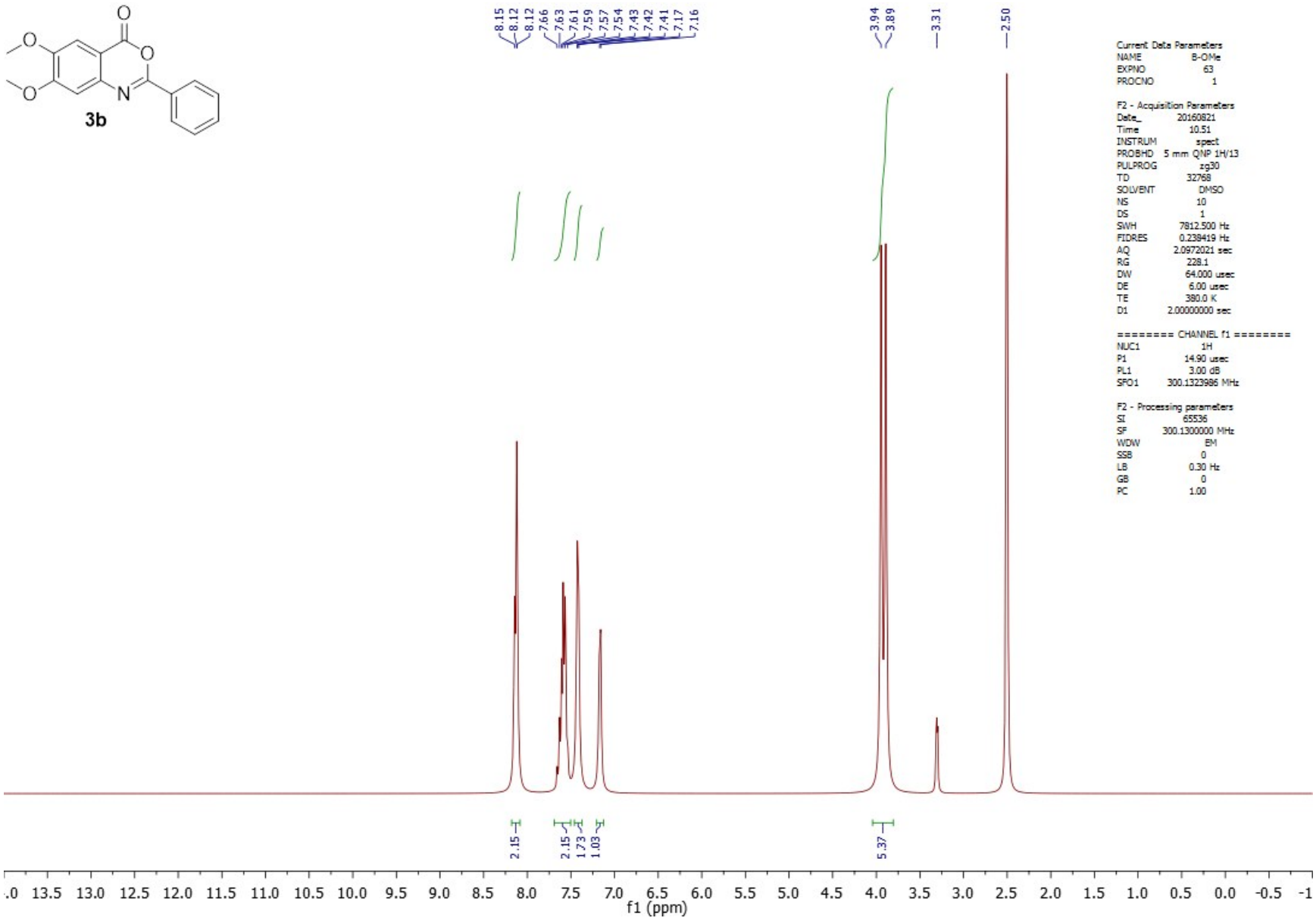
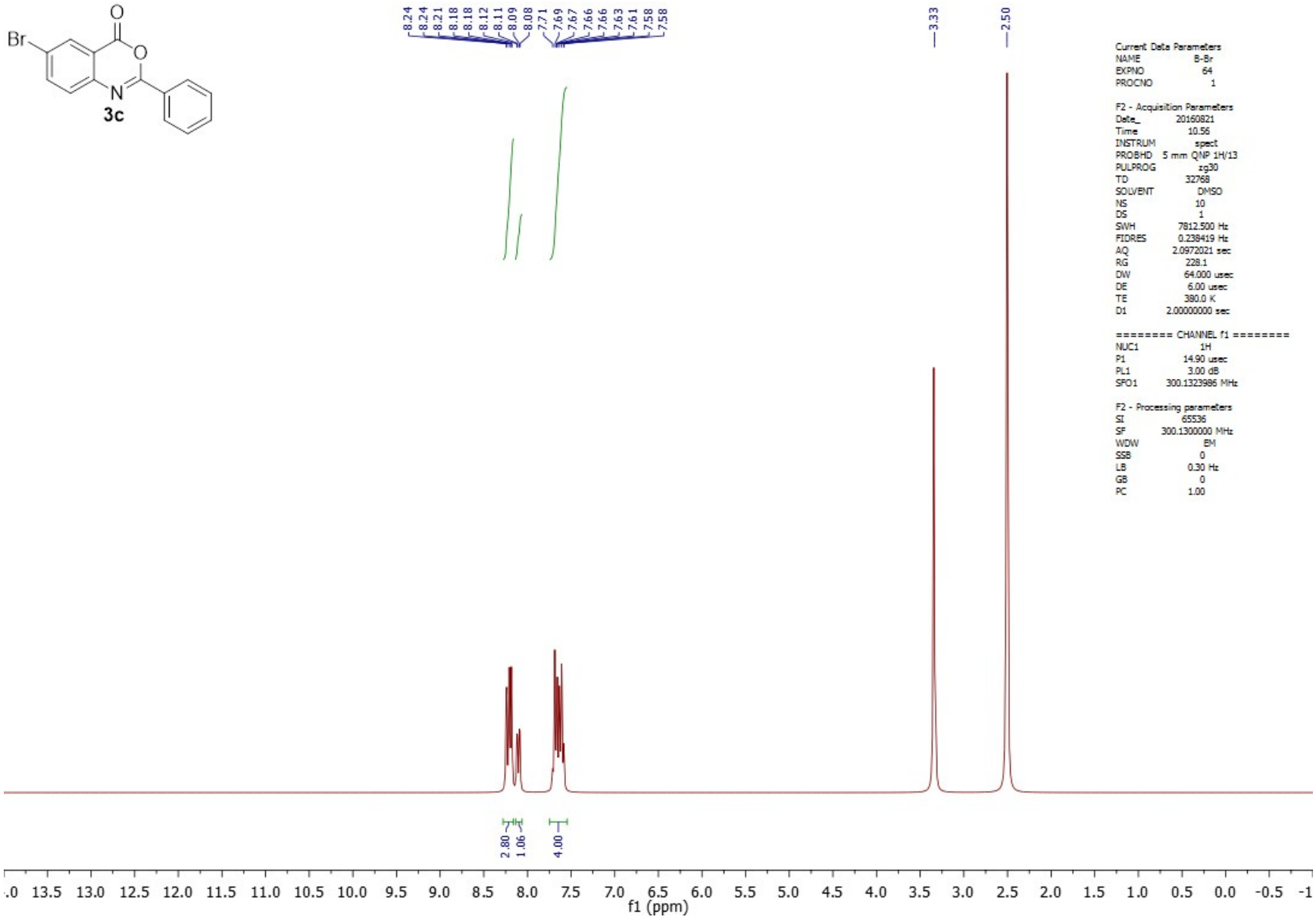
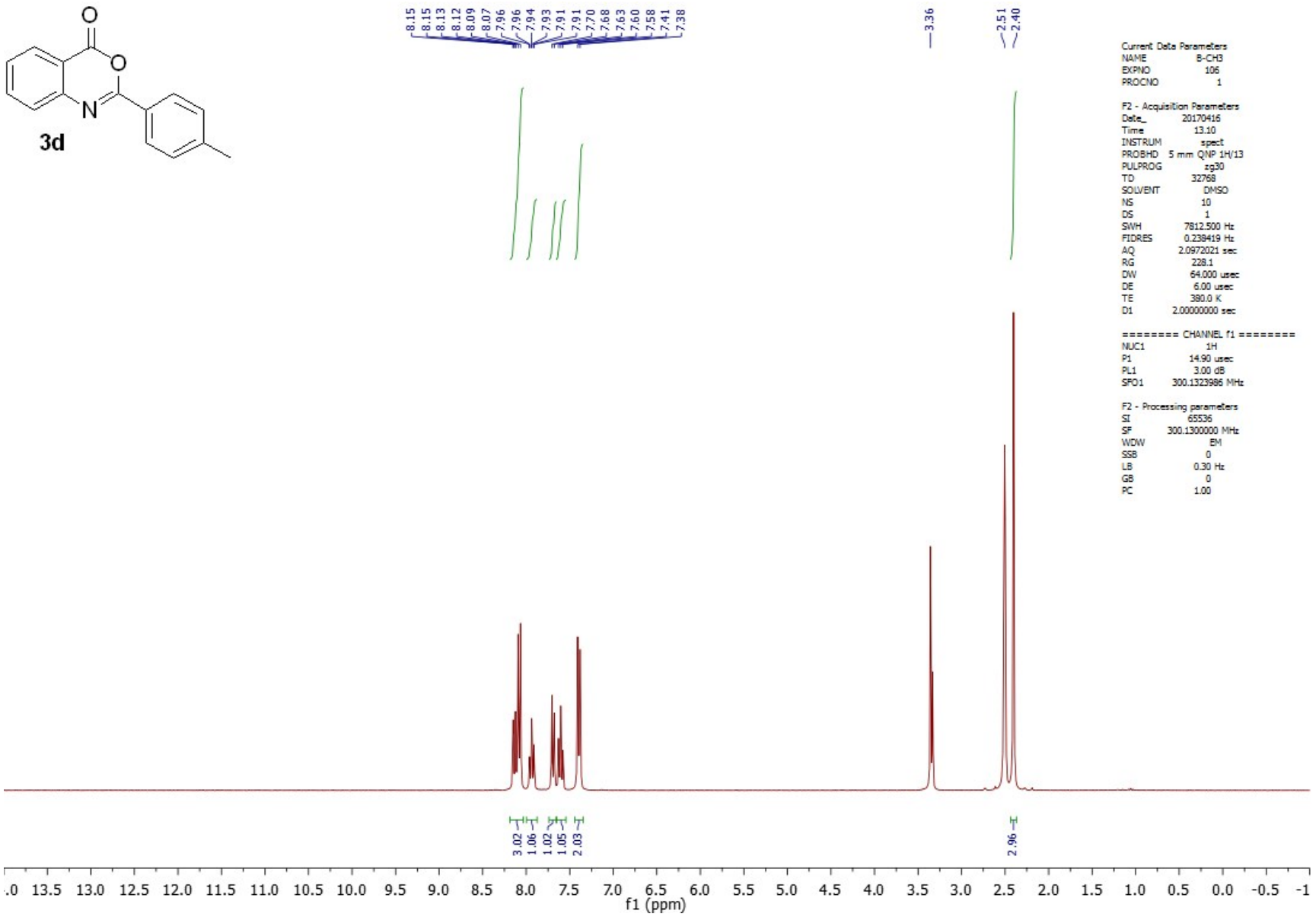
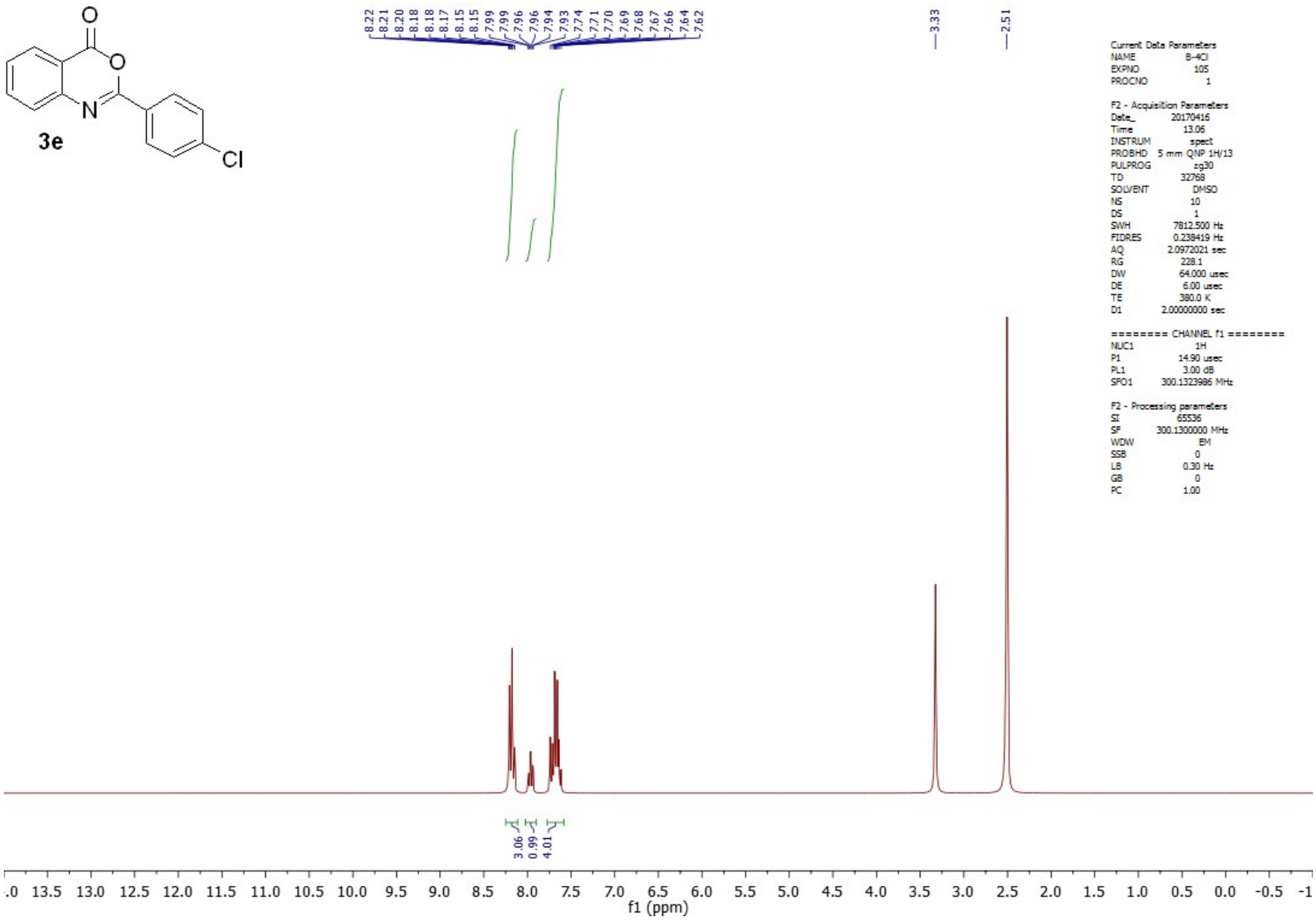
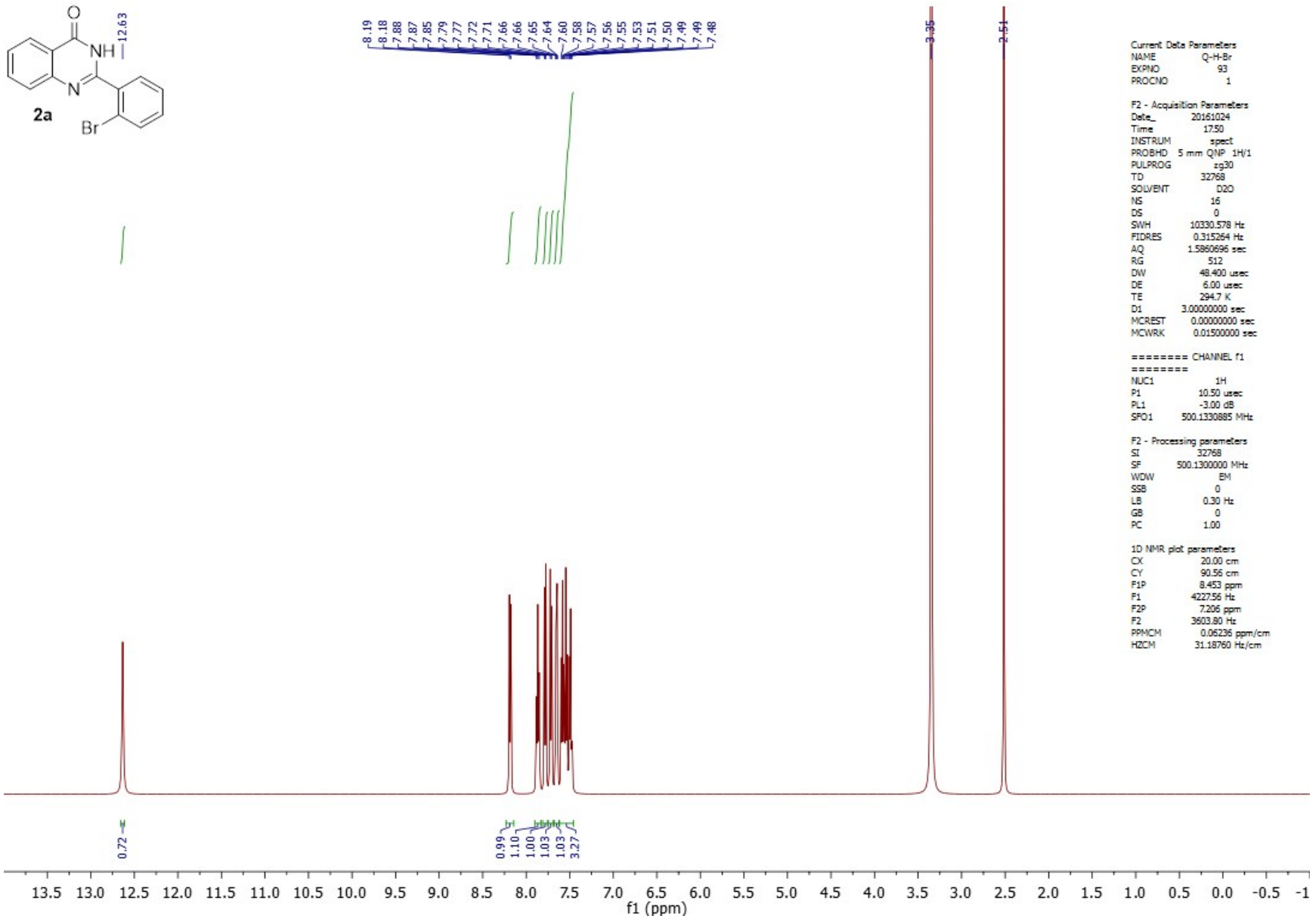
8.21
8.20
8.19
8.18
8.17
8.15
8.14
8.12
8.12
7.98
7.98
7.95
7.93
7.93
7.91
7.74
7.71
7.70
7.69
7.68
7.67
7.66
7.66
7.65
7.64
7.63
7.60
7.58
7.55
3.36
2.50
Current Data Parameters
NAME B-H
EXPNO 57
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160810
Time 13.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.00
1.01
4.59
f1 (ppm)

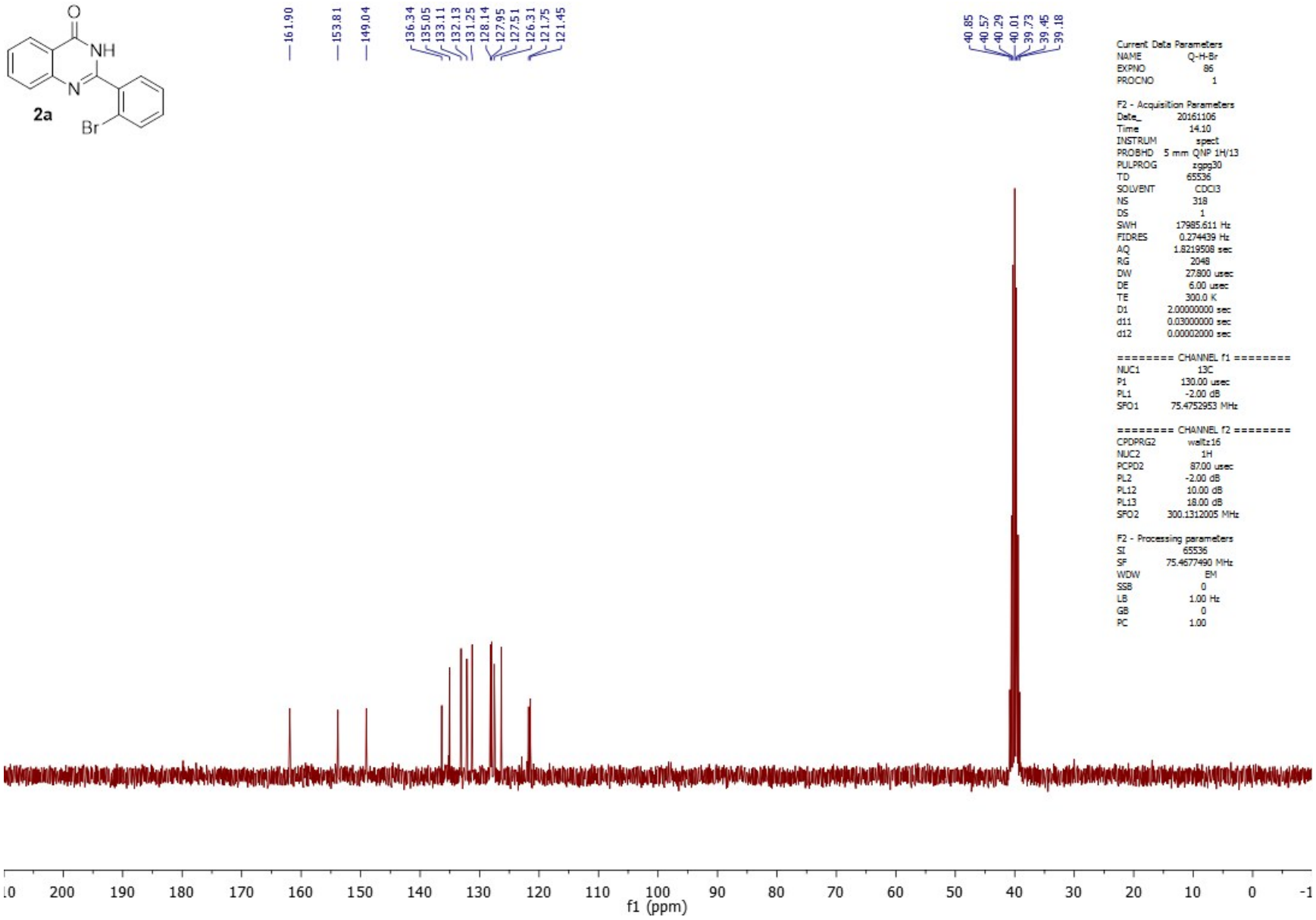

3b
8.15
8.12
8.12
7.66
7.63
7.61
7.59
7.57
7.54
7.43
7.42
7.41
7.17
7.16
3.94
3.89
3.31
2.50
2.15
2.15
1.73
1.03
5.37
f1 (ppm)
Current Data Parameters
NAME B-OMe
EXPNO 63
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160821
Time 10.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

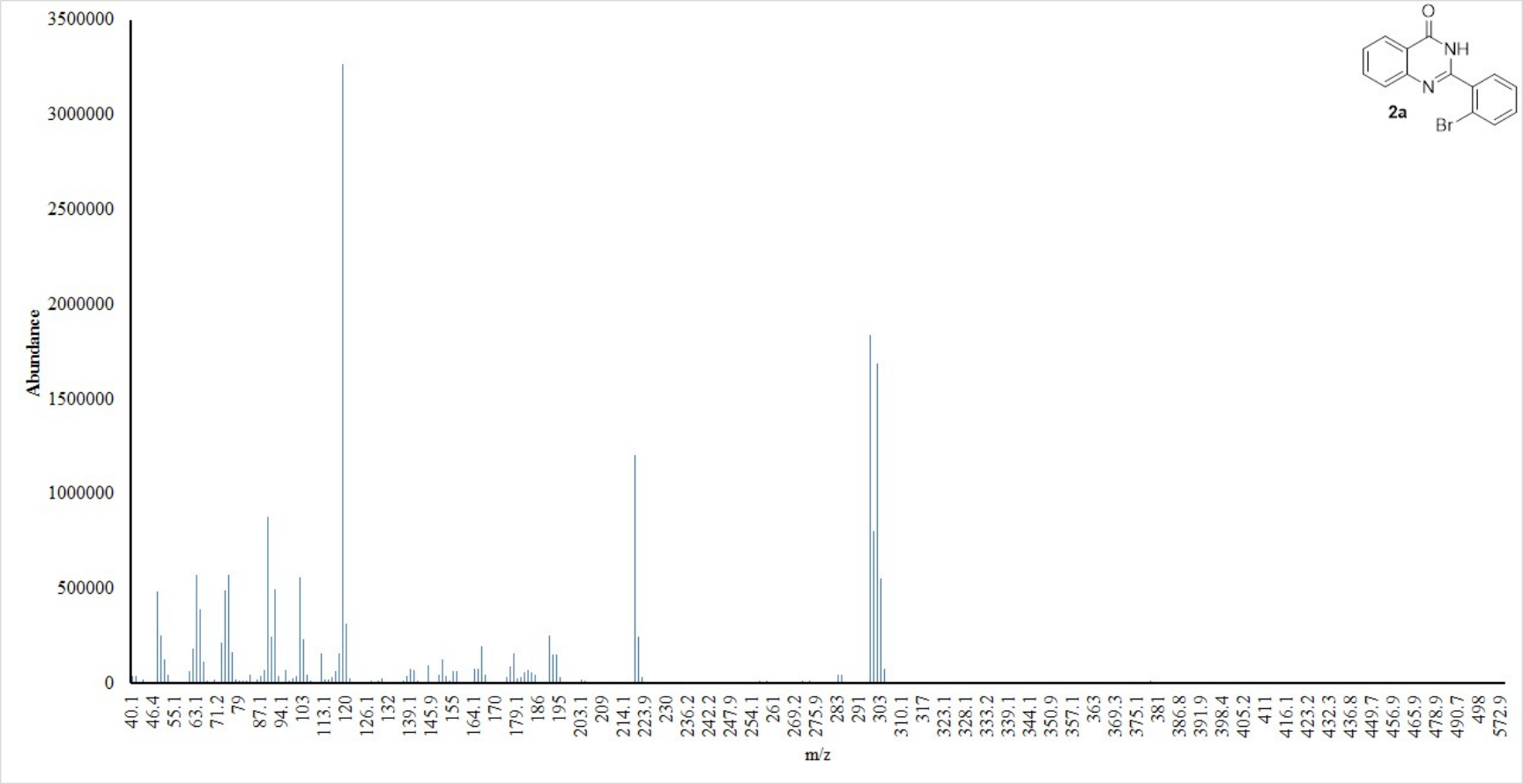

Br
O
O
N
3c
8.24
8.24
8.21
8.18
8.18
8.12
8.11
8.09
8.08
7.71
7.69
7.67
7.66
7.66
7.63
7.61
7.58
7.58
3.33
2.50
Current Data Parameters
NAME B-Br
EXPNO 64
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160821
Time 10.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.80
1.06
4.00
.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

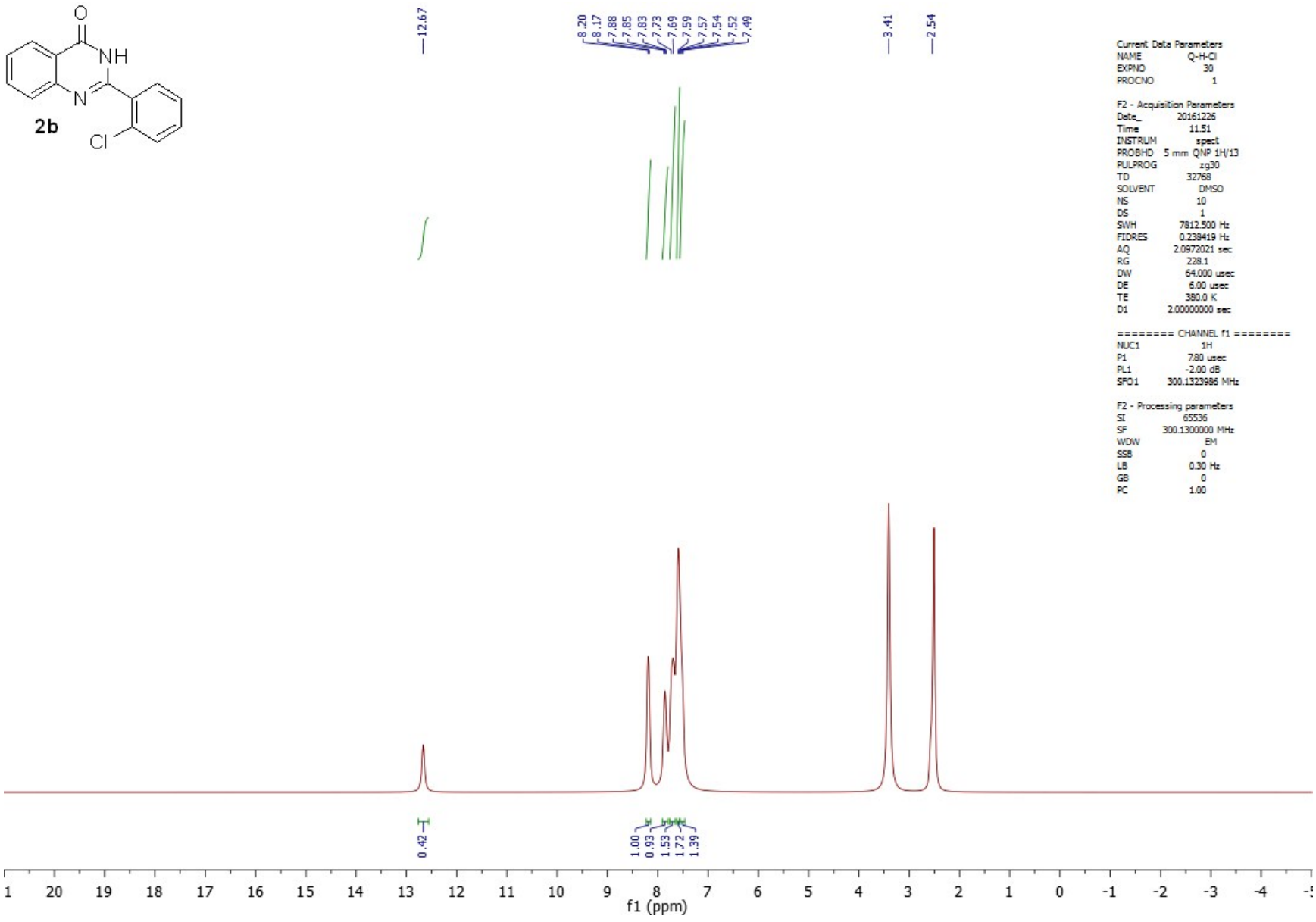

3d
8.15
8.15
8.13
8.12
8.09
8.07
7.96
7.96
7.94
7.93
7.91
7.91
7.70
7.68
7.63
7.60
7.58
7.41
7.38
3.36
2.51
2.40
Current Data Parameters
NAME B-CH3
EXPNO 106
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170416
Time 13.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.02
1.06
1.02
1.05
2.03
2.96
f1 (ppm)

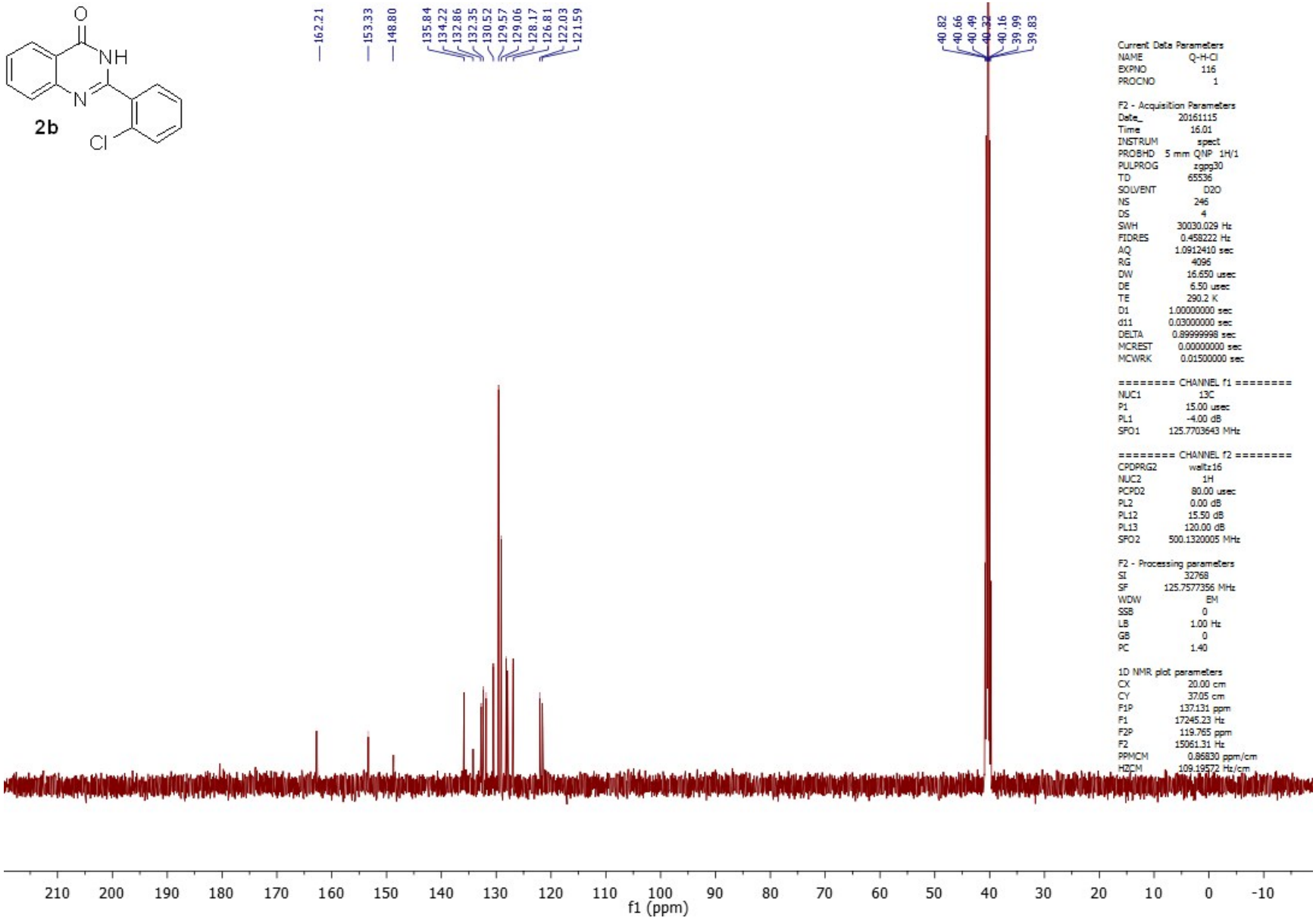

3e
Cl
8.22
8.21
8.20
8.18
8.17
8.15
8.15
7.99
7.99
7.96
7.96
7.94
7.93
7.74
7.71
7.70
7.69
7.68
7.67
7.66
7.64
7.62
3.33
2.51
Current Data Parameters
NAME 8-4Cl
EXPNO 105
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170416
Time 13.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.06
0.99
4.01
f1 (ppm)

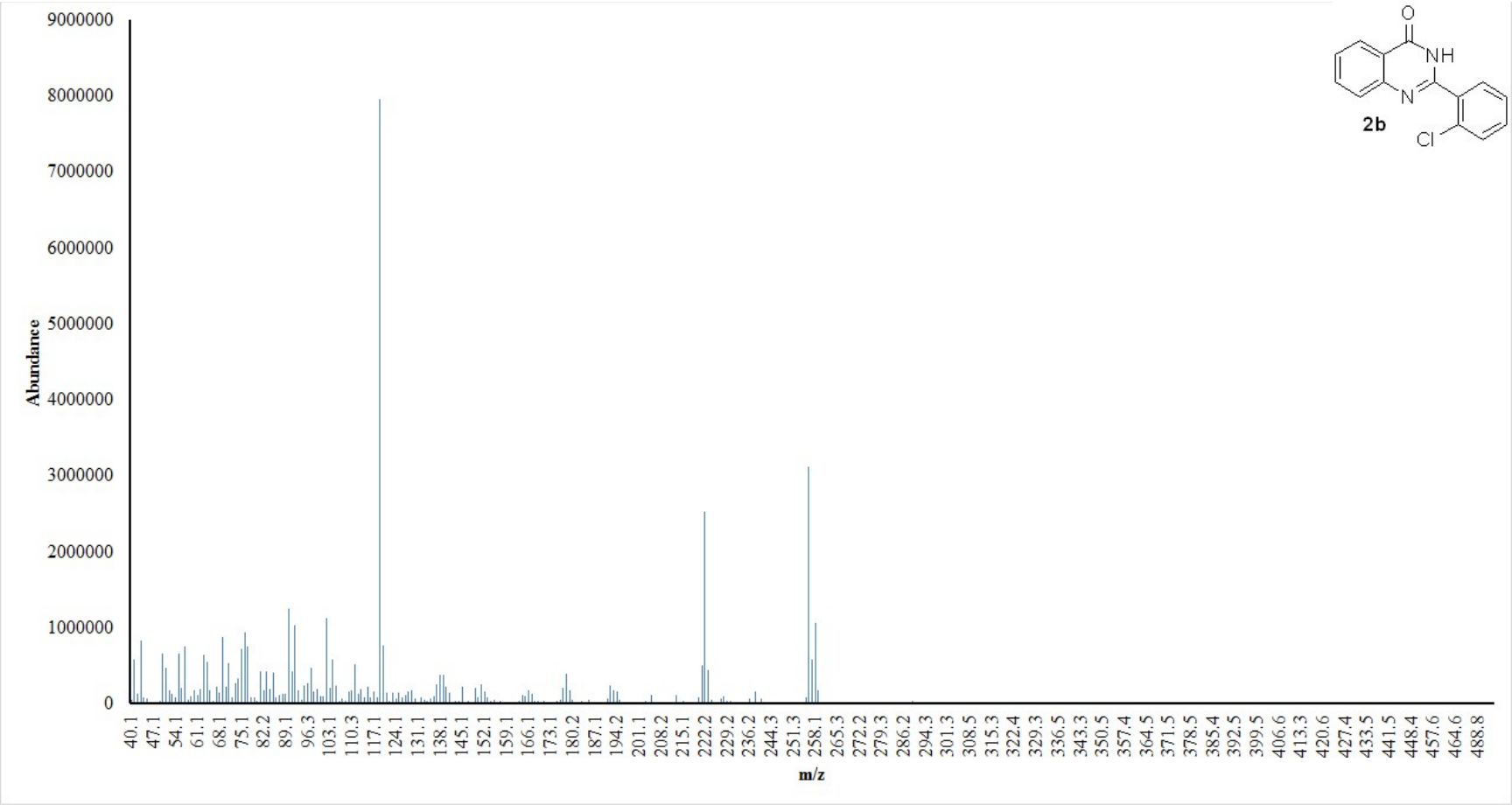

2a
12.63
8.19
8.18
7.88
7.87
7.85
7.79
7.77
7.72
7.71
7.66
7.66
7.65
7.64
7.60
7.58
7.57
7.56
7.55
7.53
7.51
7.50
7.49
7.49
7.48
3.35
2.51
Current Data Parameters
NAME Q-H-Br
EXPNO 93
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161024
Time 17.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT D2O
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 512
DW 48.400 usec
DE 6.00 usec
TE 294.7 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 90.56 cm
F1P 8.453 ppm
F1 4227.56 Hz
F2P 7.206 ppm
F2 3603.80 Hz
PPMCM 0.06236 ppm/cm
HZCM 31.18760 Hz/cm
0.72
0.99
1.10
1.00
1.03
1.03
3.27
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

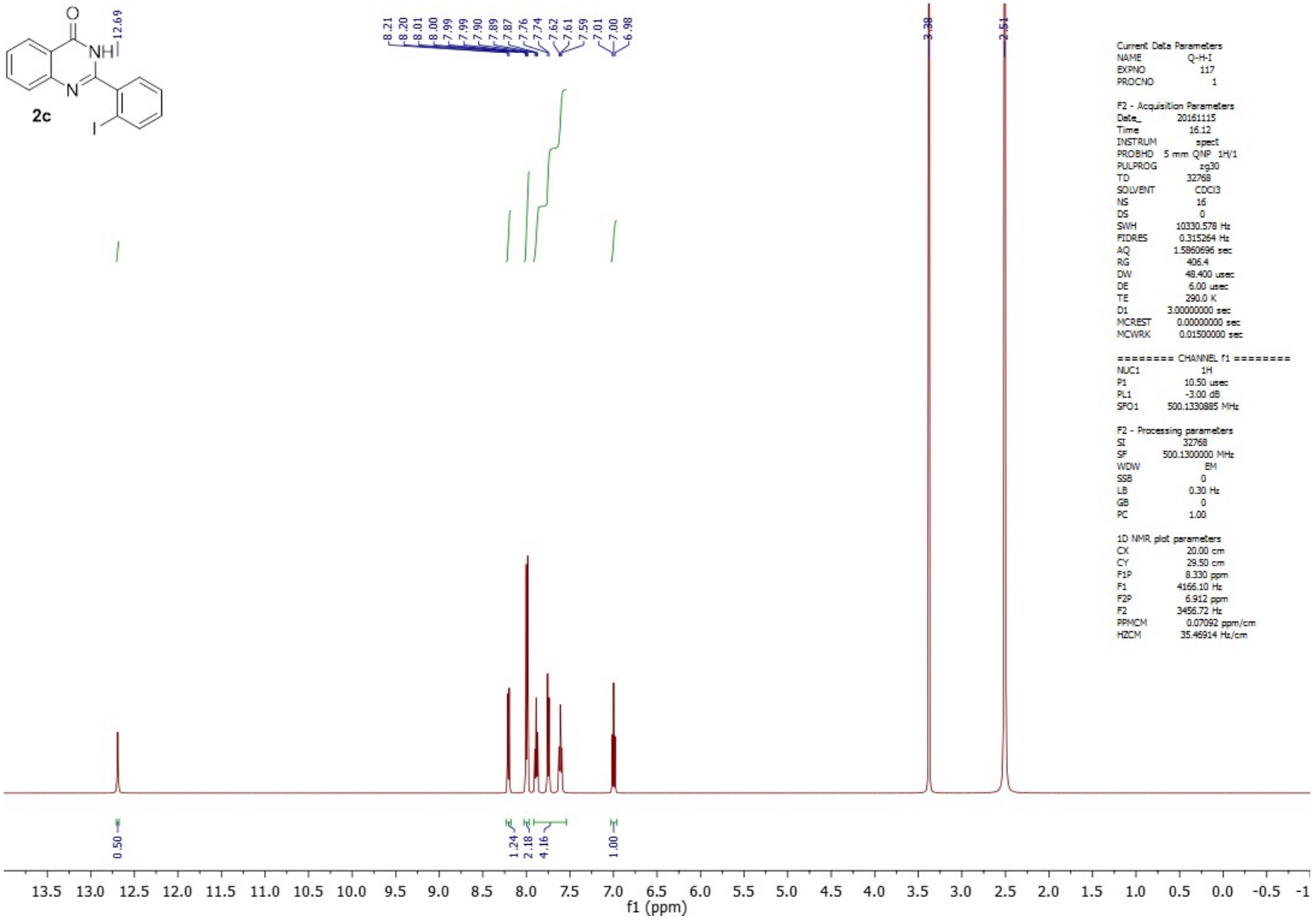

2a
161.90
153.81
149.04
136.34
135.05
133.11
132.13
131.25
128.14
127.95
127.51
126.31
121.75
121.45
40.85
40.57
40.29
40.01
39.73
39.45
39.18
Current Data Parameters
NAME Q-H-Br
EXPNO 86
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161106
Time 14.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 318
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

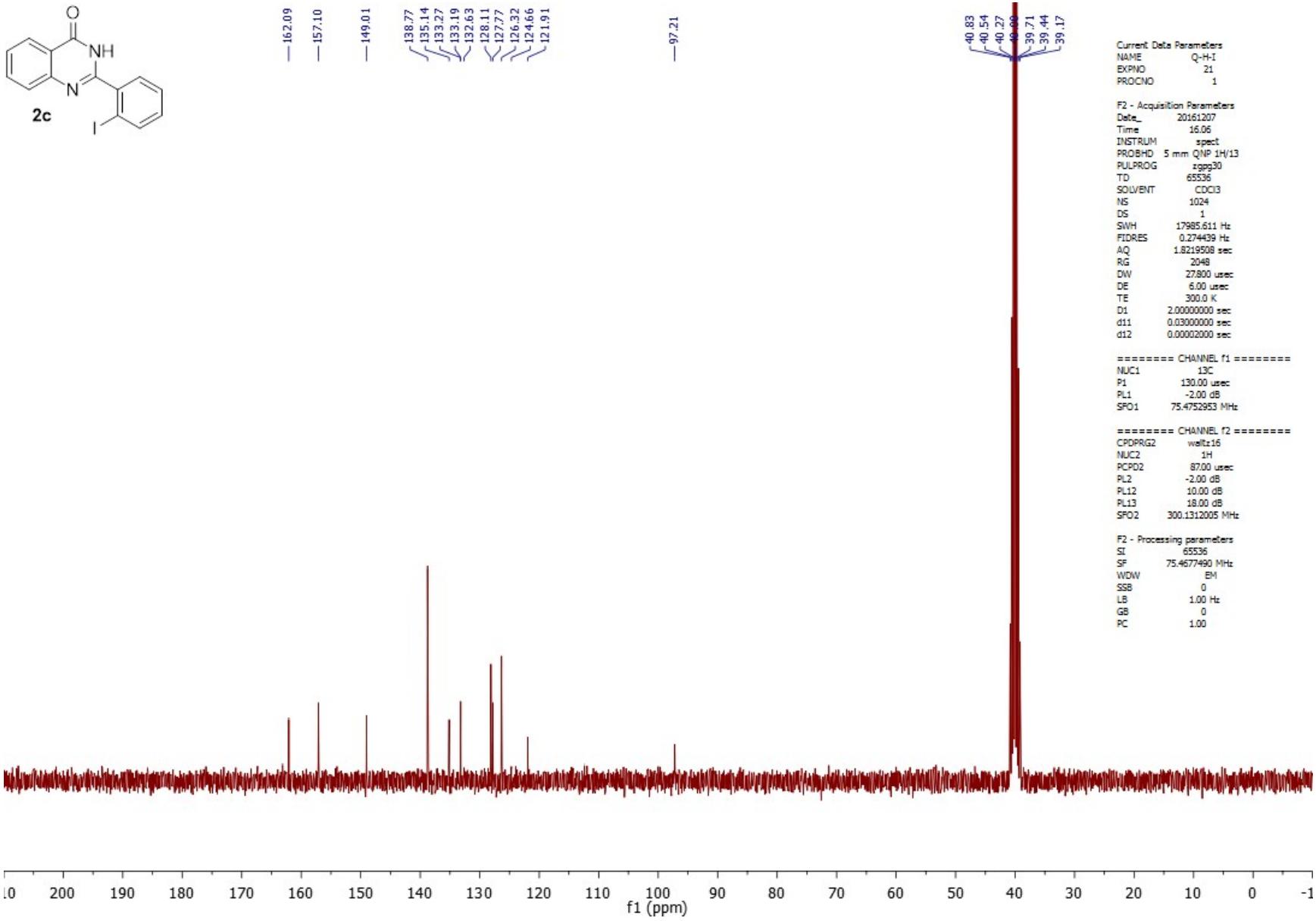

Abundance
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
m/z
40.1 46.4 55.1 63.1 71.2 79 87.1 94.1 103 113.1 120 126.1 132 139.1 145.9 155 164.1 170 179.1 186 195 203.1 209 214.1 223.9 230 236.2 242.2 247.9 254.1 261 269.2 275.9 283 291 303 310.1 317 323.1 328.1 333.2 339.1 344.1 350.9 357.1 363 369.3 375.1 381 386.8 391.9 398.4 405.2 411 416.1 423.2 432.3 436.8 449.7 456.9 465.9 478.9 490.7 498 572.9
O
NH
N
Br
2a

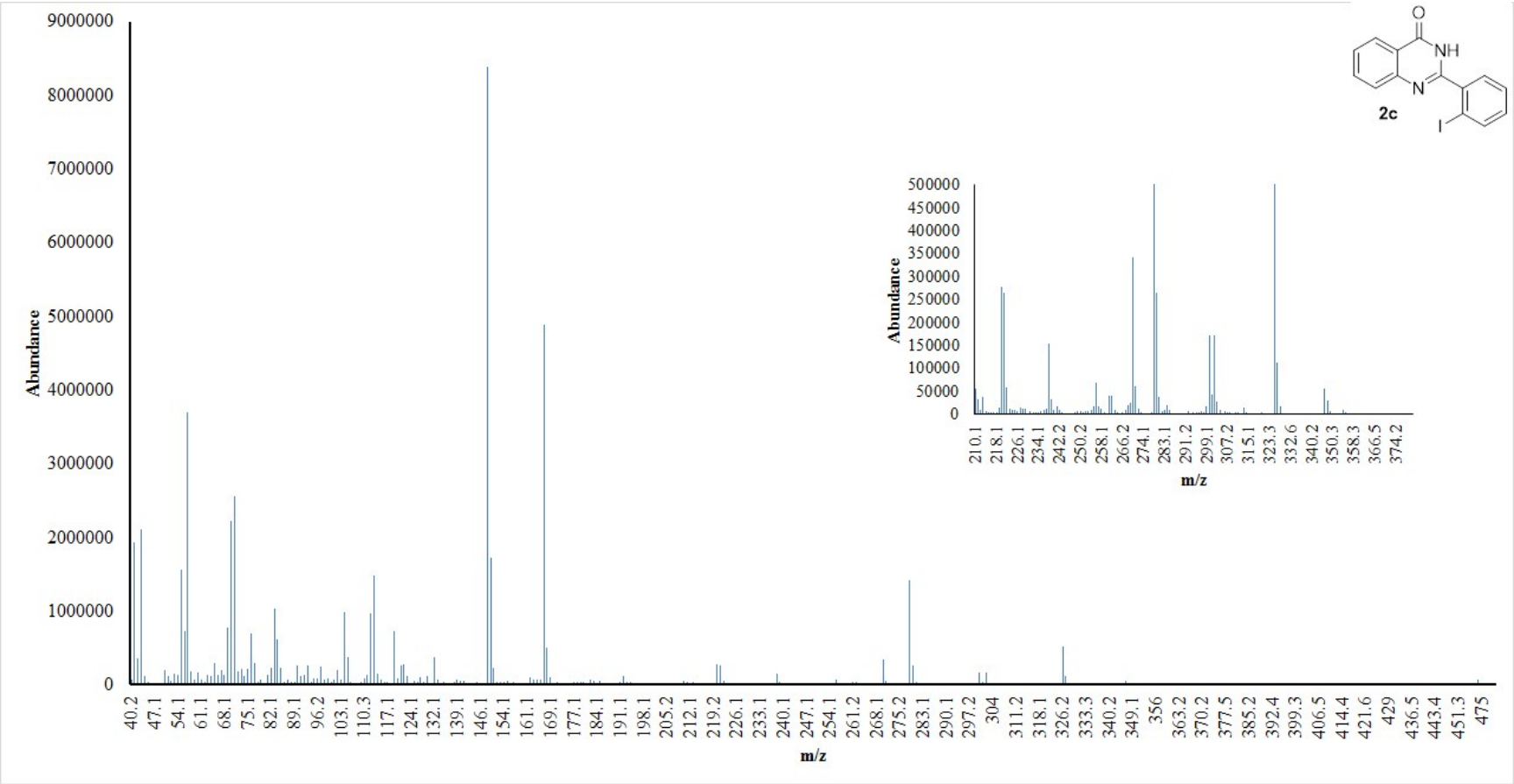

2b
12.67
8.20
8.17
7.88
7.85
7.83
7.73
7.69
7.59
7.57
7.54
7.52
7.49
3.41
2.54
Current Data Parameters
NAME Q-H-Cl
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161226
Time 11.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.42
1.00
0.93
1.53
1.72
1.39
f1 (ppm)

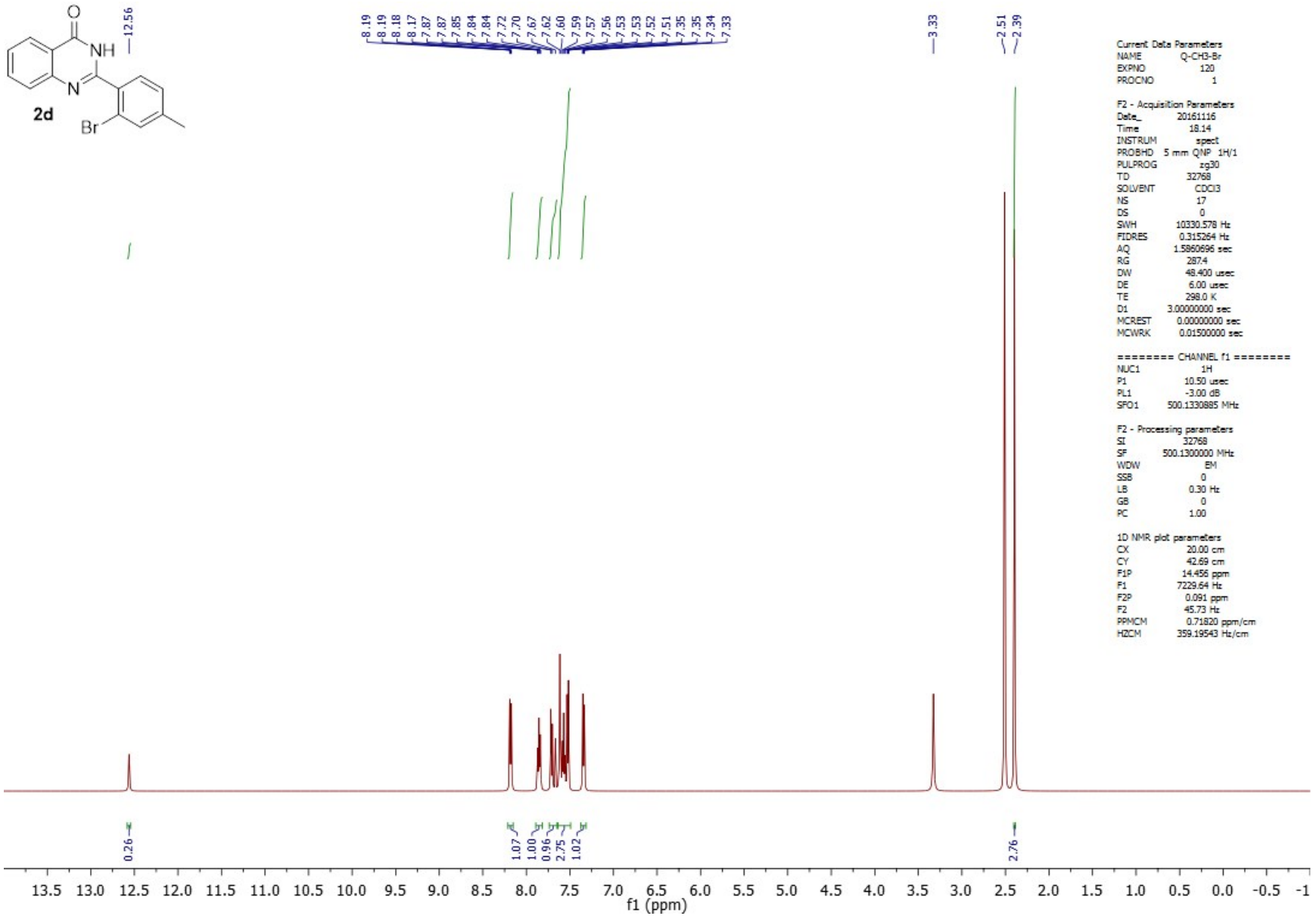

O
NH
N
Cl
2b
162.21
153.33
148.80
135.84
134.22
132.86
132.35
130.52
129.57
129.06
128.17
126.81
122.03
121.59
40.82
40.66
40.49
40.32
40.16
39.99
39.83
Current Data Parameters
NAME Q-H-Cl
EXPNO 116
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161115
Time 16.01
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 246
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 290.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 37.05 cm
F1P 137.131 ppm
F1 17245.23 Hz
F2P 119.765 ppm
F2 15061.31 Hz
PPMCM 0.86830 ppm/cm
HZCM 109.19572 Hz/cm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

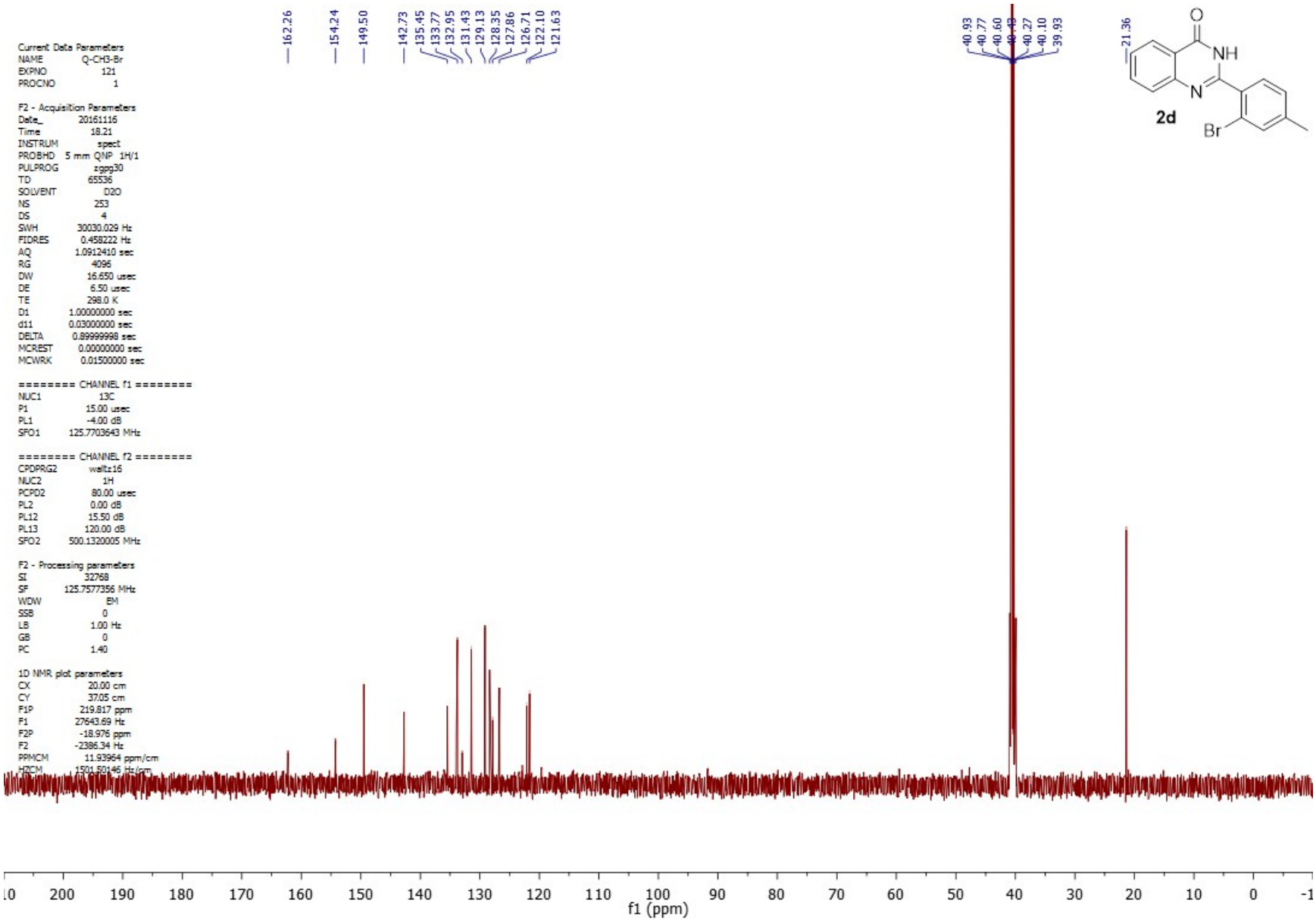

9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Abundance
40.1
47.1
54.1
61.1
68.1
75.1
82.2
89.1
96.3
103.1
110.3
117.1
124.1
131.1
138.1
145.1
152.1
159.1
166.1
173.1
180.2
187.1
194.2
201.1
208.2
215.1
222.2
229.2
236.2
244.3
251.3
258.1
265.3
272.2
279.3
286.2
294.3
301.3
308.5
315.3
322.4
329.3
336.5
343.3
350.5
357.4
364.5
371.5
378.5
385.4
392.5
399.5
406.6
413.3
420.6
427.4
433.5
441.5
448.4
457.6
464.6
488.8
m/z
O
NH
N
Cl
2b

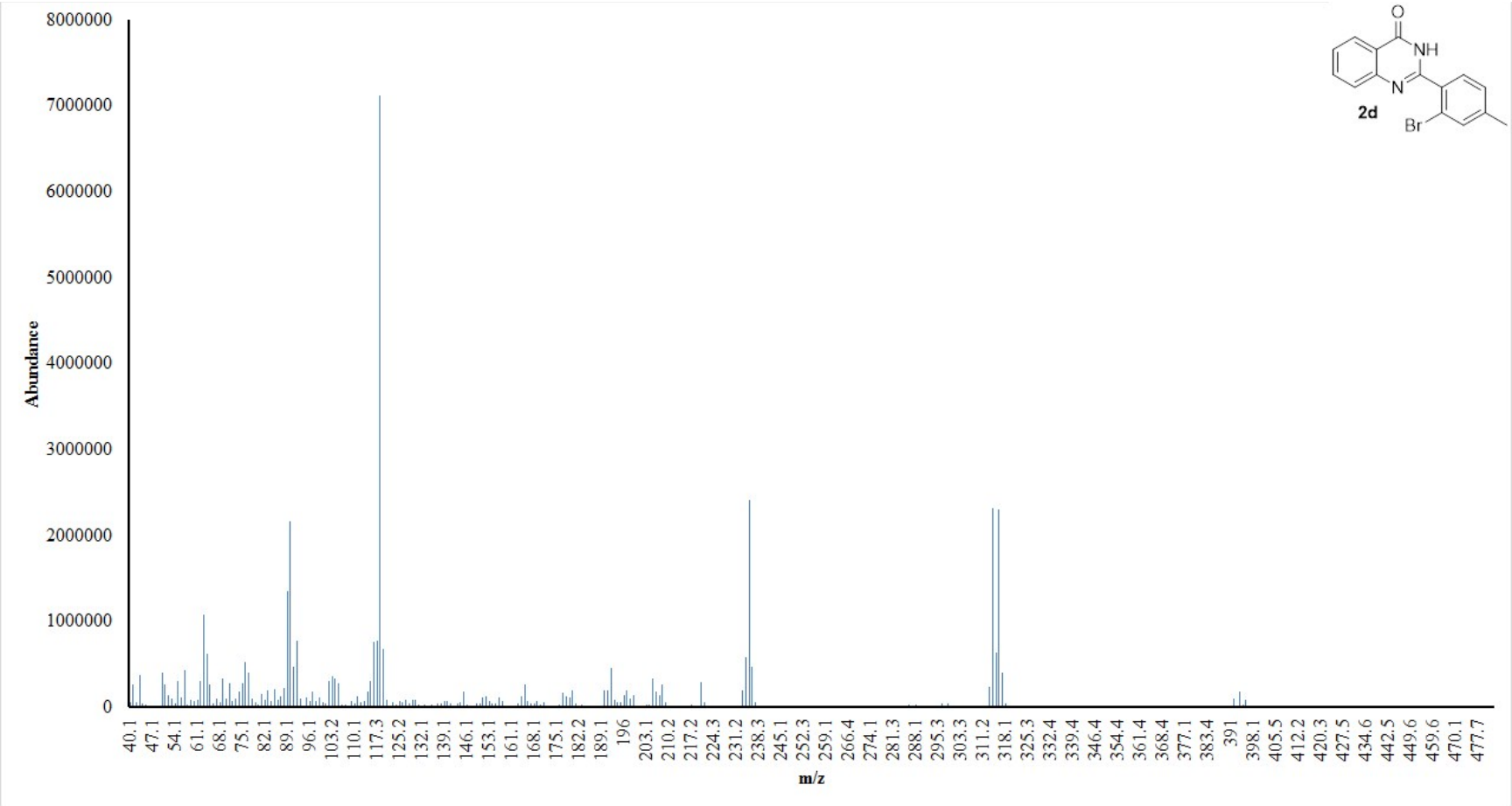

2c
12.69
8.21
8.20
8.01
8.00
7.99
7.99
7.90
7.89
7.87
7.76
7.74
7.62
7.61
7.59
7.01
7.00
6.98
3.38
2.51
0.50
1.24
2.18
4.16
1.00
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)
Current Data Parameters
NAME Q-H-I
EXPNO 117
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161115
Time 16.12
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 406.4
DW 48.400 usec
DE 6.00 usec
TE 290.0 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 29.50 cm
F1P 8.330 ppm
F1 4166.10 Hz
F2P 6.912 ppm
F2 3456.72 Hz
PPMCM 0.07092 ppm/cm
HZCM 35.46914 Hz/cm

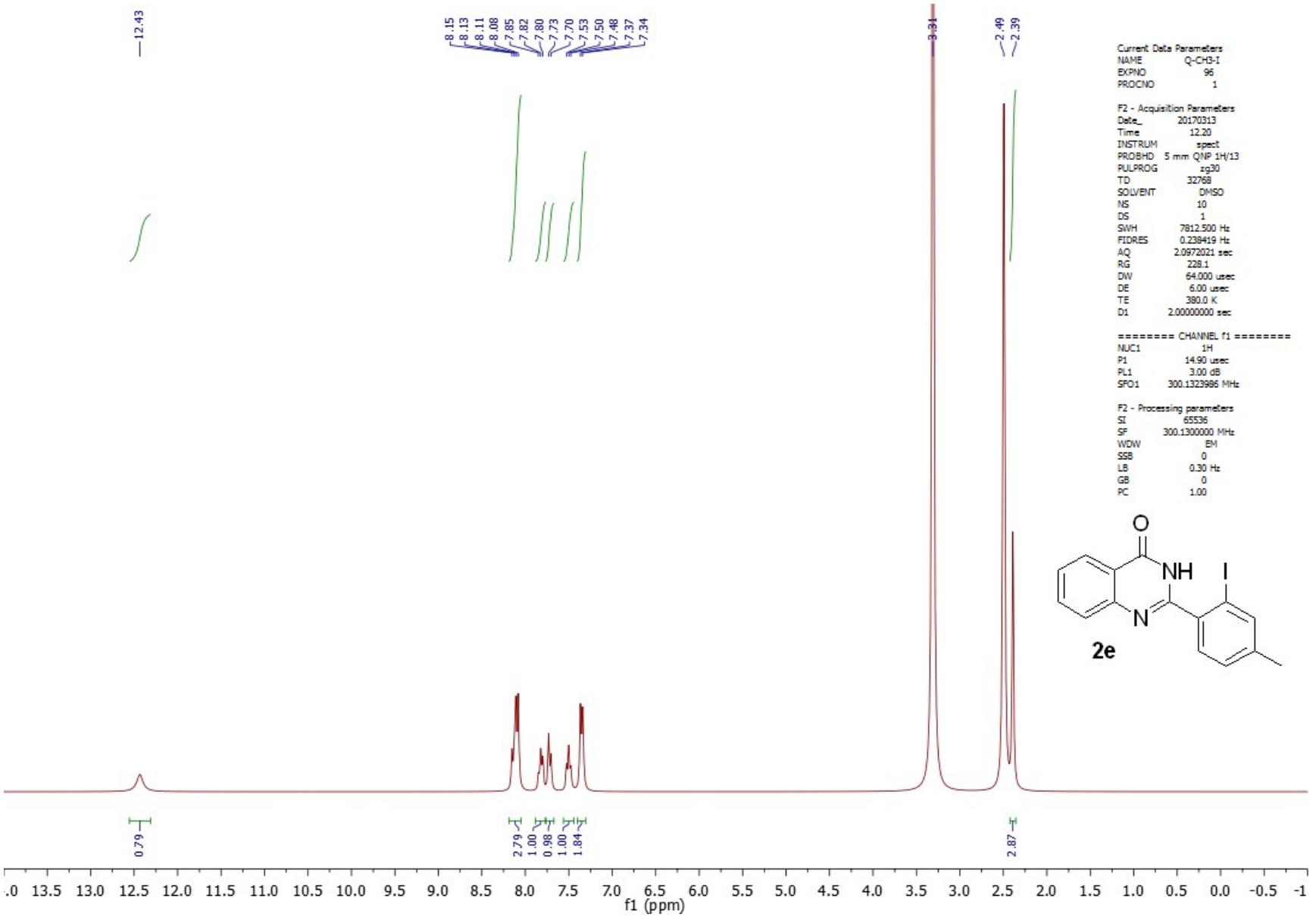

2c
162.09
157.10
149.01
138.77
135.14
133.27
133.19
132.63
128.11
127.77
126.32
124.66
121.91
97.21
40.83
40.54
40.27
40.00
39.71
39.44
39.17
Current Data Parameters
NAME Q-H-I
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161207
Time 16.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

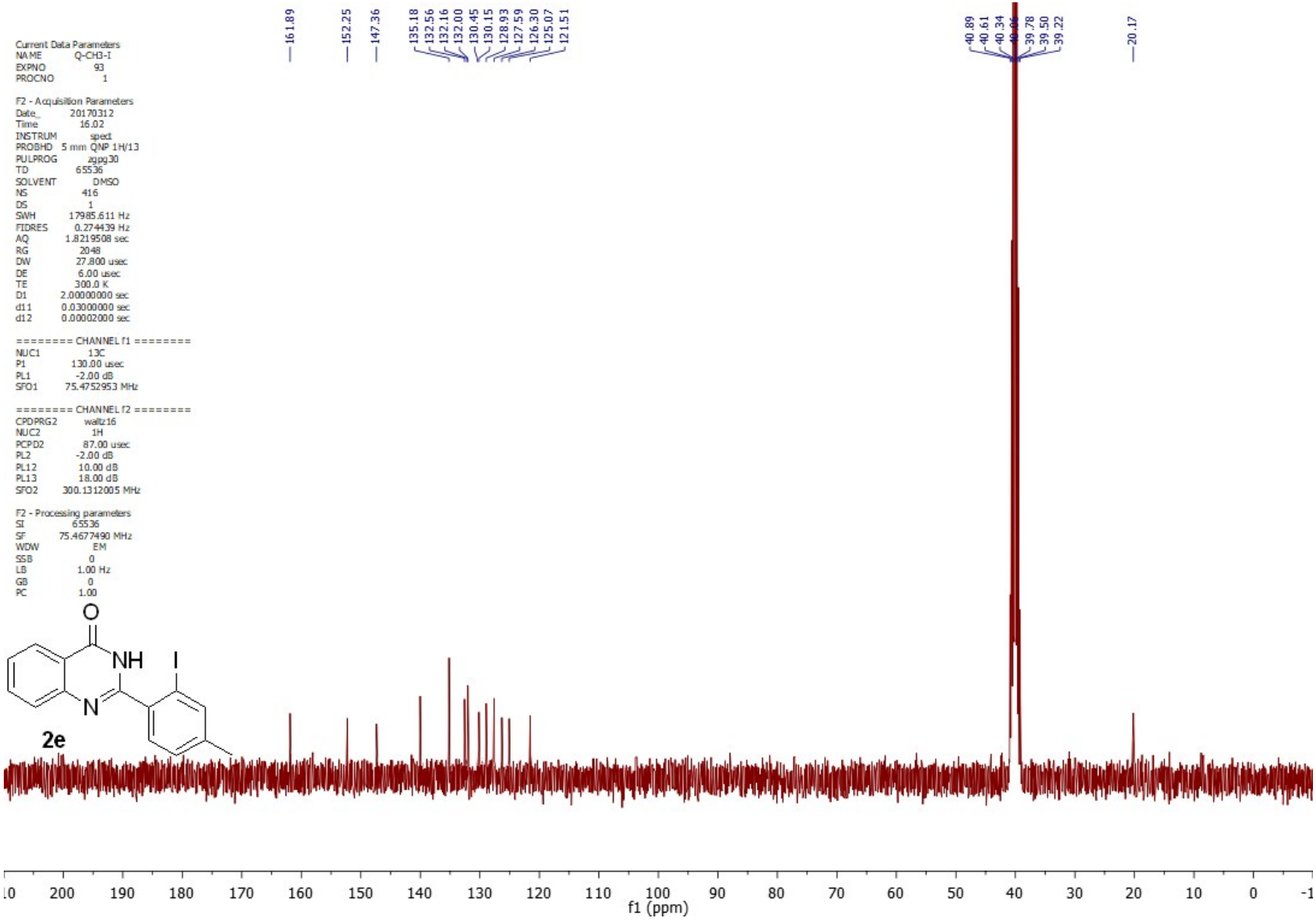

Abundance
m/z
2c
O
NH
N
I

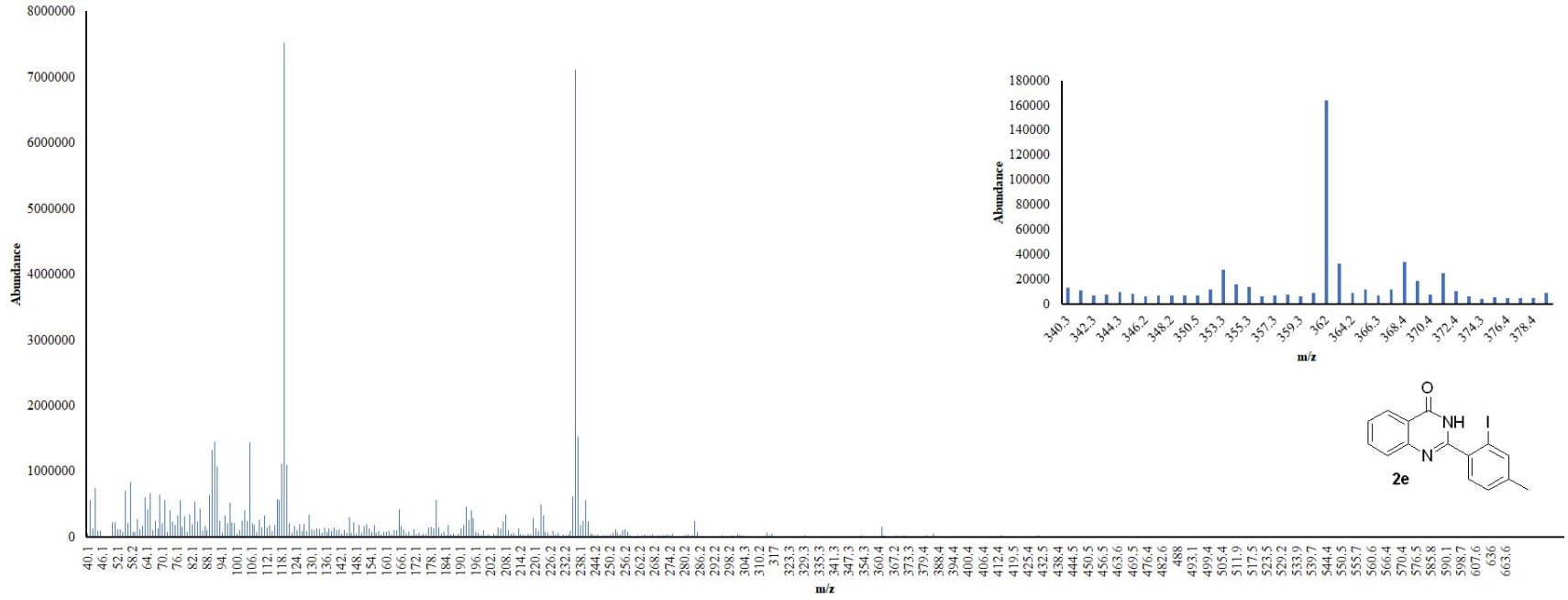

2d
12.56
8.19
8.19
8.18
8.17
7.87
7.87
7.85
7.84
7.84
7.72
7.70
7.67
7.62
7.60
7.59
7.57
7.56
7.53
7.53
7.52
7.51
7.35
7.35
7.34
7.33
3.33
2.51
2.39
Current Data Parameters
NAME Q-CH3-Br
EXPNO 120
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161116
Time 18.14
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 17
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 287.4
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 42.69 cm
F1P 14.456 ppm
F1 7229.64 Hz
F2P 0.091 ppm
F2 45.73 Hz
PPMCM 0.71820 ppm/cm
HZCM 359.19543 Hz/cm
0.26
1.07
1.00
0.96
2.75
1.02
2.76
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

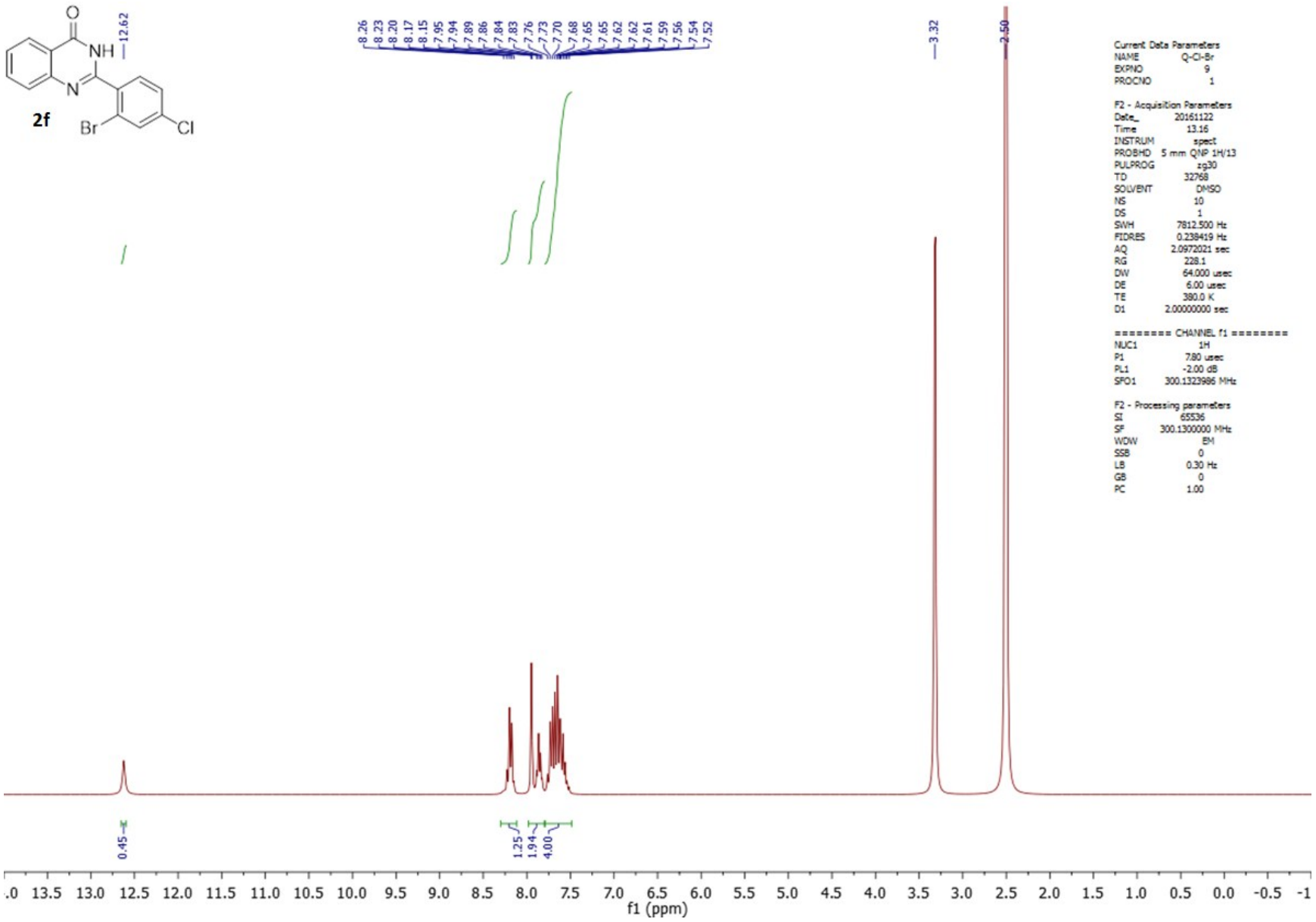

Current Data Parameters
NAME Q-CH3-Br
EXPNO 121
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161116
Time 18.21
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 253
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 37.05 cm
F1P 219.817 ppm
F1 27643.69 Hz
F2P -18.976 ppm
F2 -2386.34 Hz
PPMCM 11.93964 ppm/cm
HZCM 1501.50146 Hz/cm
162.26
154.24
149.50
142.73
135.45
133.77
132.95
131.43
129.13
128.35
127.86
126.71
122.10
121.63
40.93
40.77
40.60
40.43
40.27
40.10
39.93
21.36
O
NH
N
Br
2d
f1 (ppm)

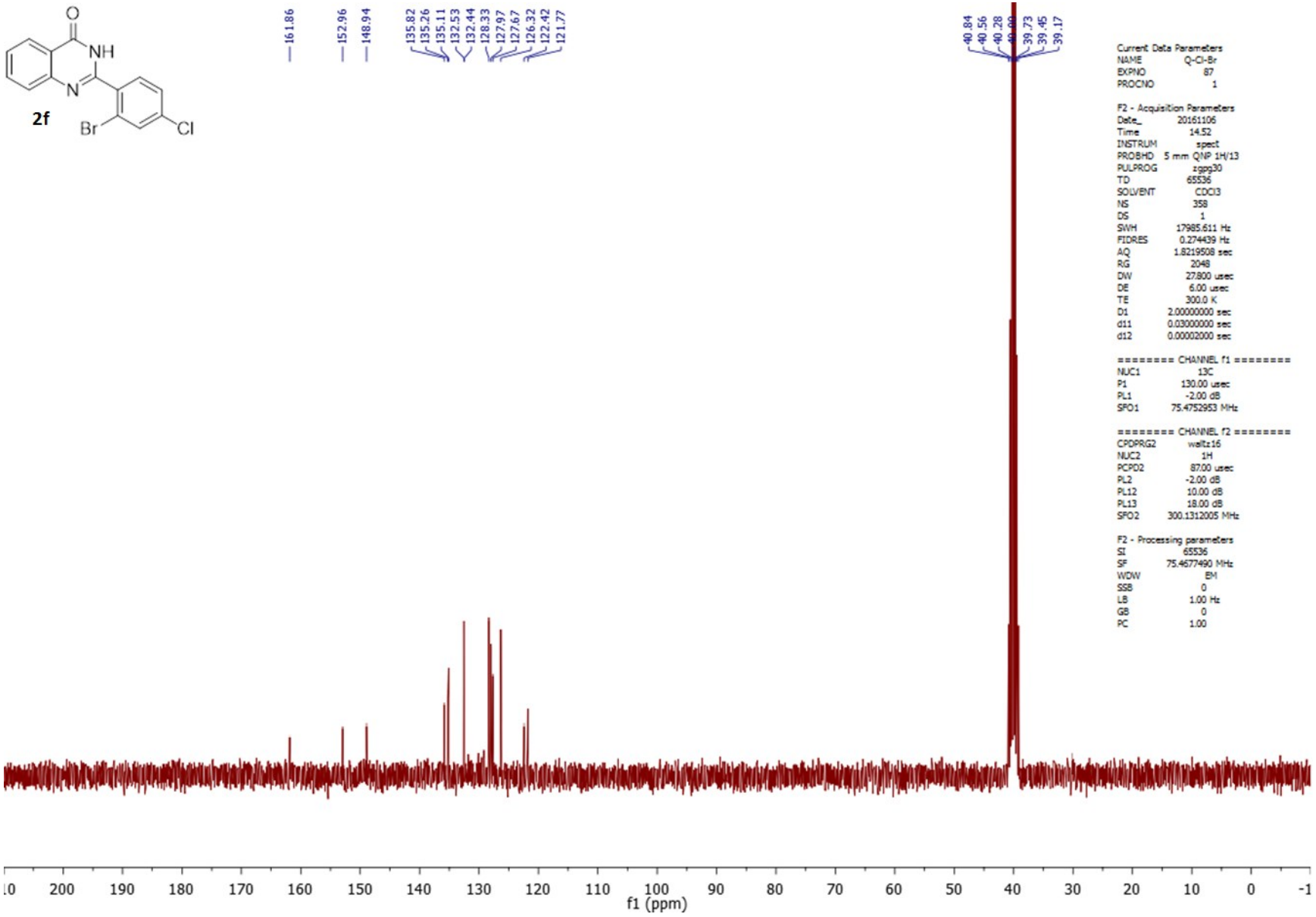

8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Abundance
40.1 47.1 54.1 61.1 68.1 75.1 82.1 89.1 96.1 103.2 110.1 117.3 125.2 132.1 139.1 146.1 153.1 161.1 168.1 175.1 182.2 189.1 196 203.1 210.2 217.2 224.3 231.2 238.3 245.1 252.3 259.1 266.4 274.1 281.3 288.1 295.3 303.3 311.2 318.1 325.3 332.4 339.4 346.4 354.4 361.4 368.4 377.1 383.4 391 398.1 405.5 412.2 420.3 427.5 434.6 442.5 449.6 459.6 470.1 477.7
m/z
O
NH
N
Br
2d

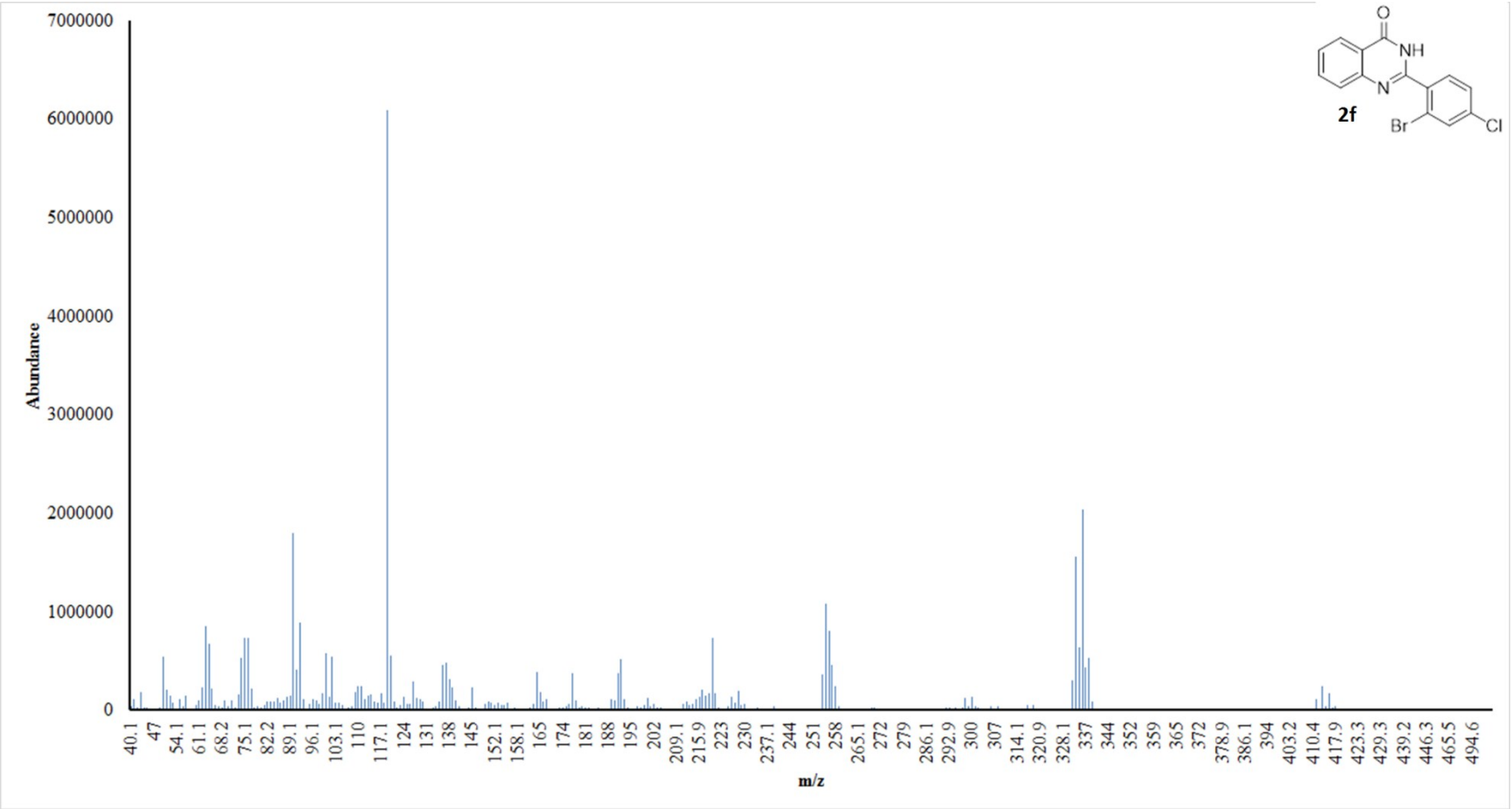

12.43
8.15
8.13
8.11
8.08
7.85
7.82
7.80
7.73
7.70
7.53
7.50
7.48
7.37
7.34
3.34
2.49
2.39
Current Data Parameters
NAME Q-CH3-I
EXPNO 96
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 12.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
NH
I
N
2e
0.79
2.79
1.00
0.98
1.00
1.84
2.87
f1 (ppm)

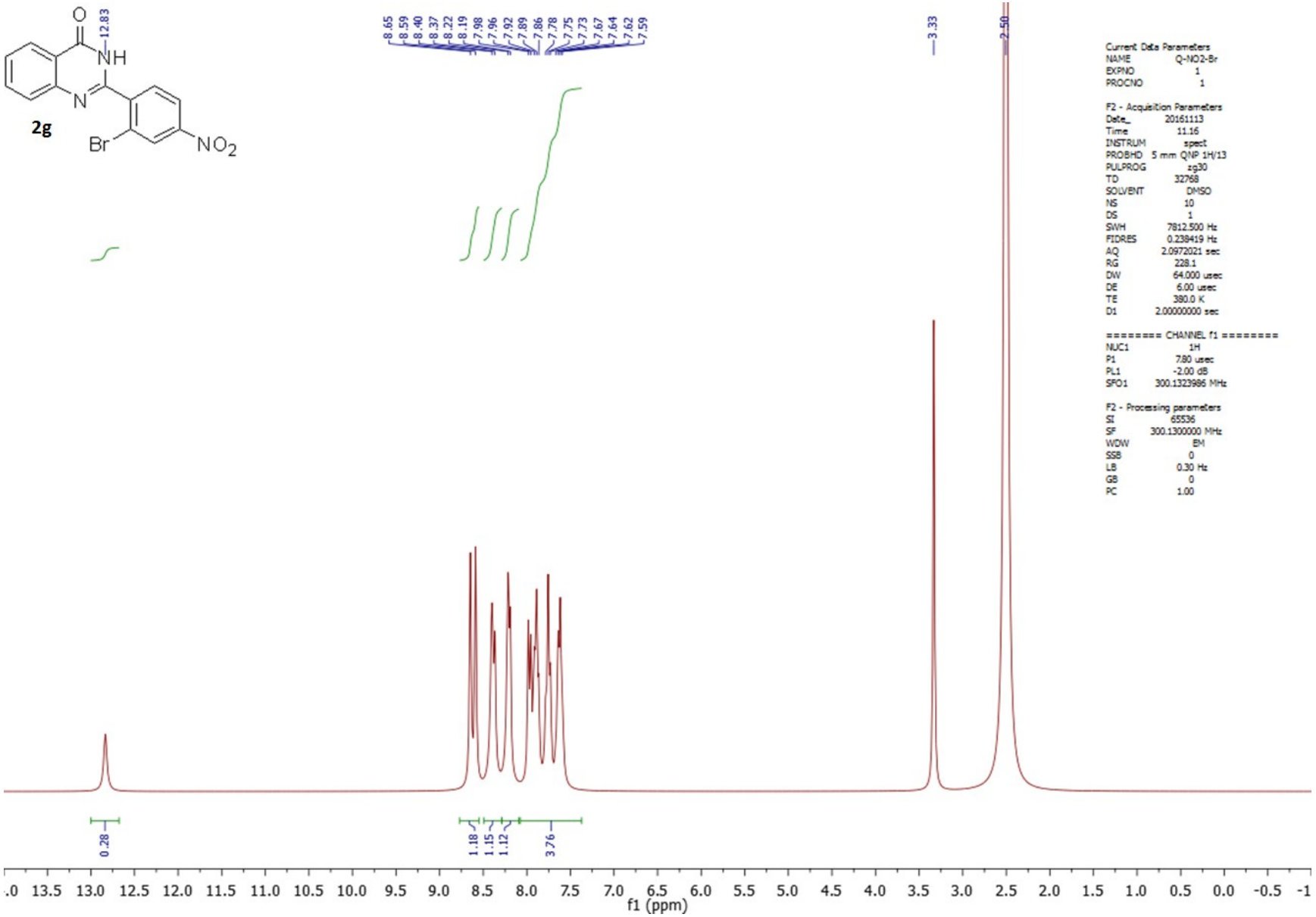

Current Data Parameters
NAME Q-CH3-I
EXPNO 93
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170312
Time 16.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 416
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
161.89
152.25
147.36
135.18
132.56
132.16
132.00
130.45
130.15
128.93
127.59
126.30
125.07
121.51
40.89
40.61
40.34
39.78
39.50
39.22
20.17
O
NH
I
N
2e
f1 (ppm)

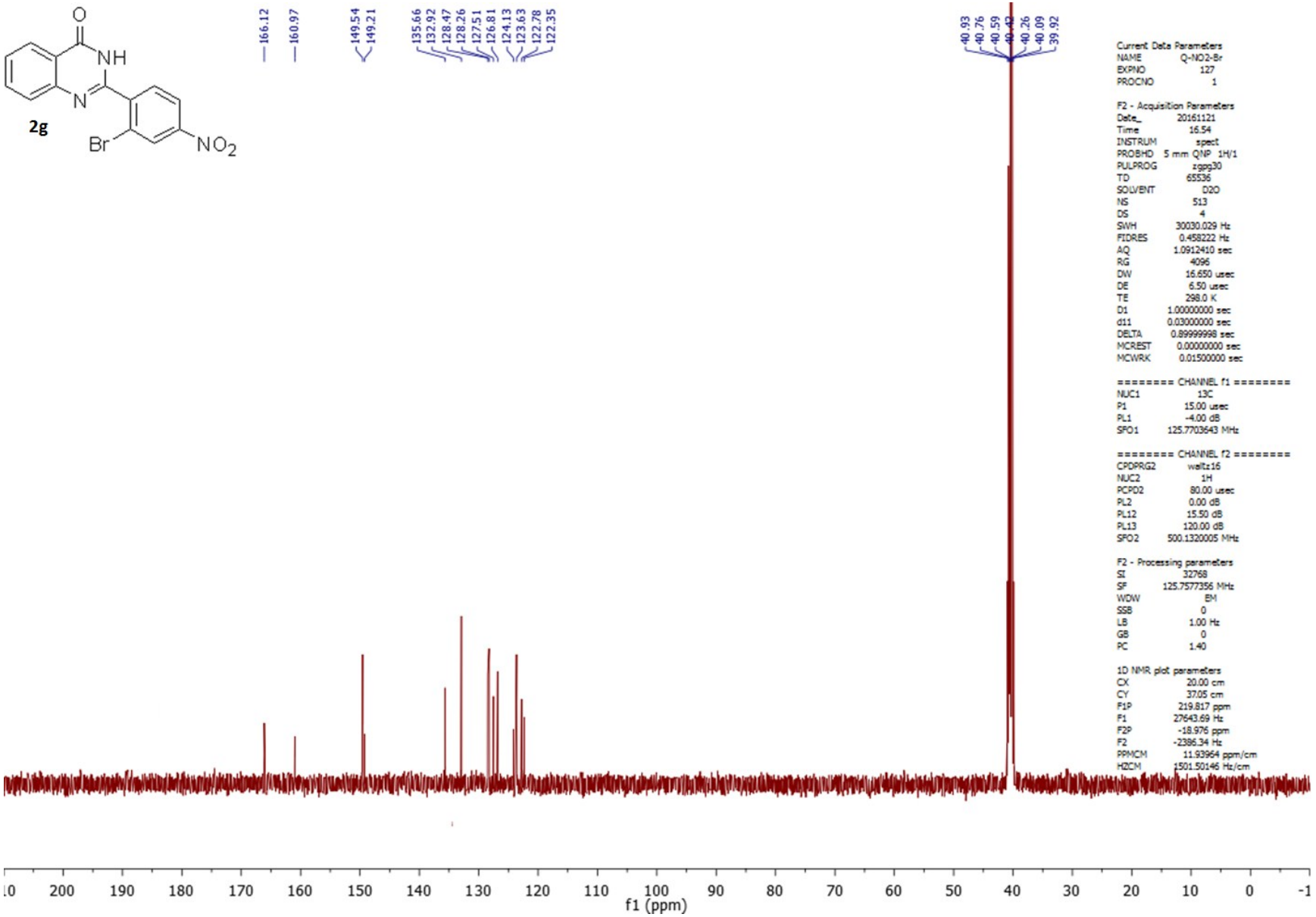

Abundance
m/z
O
NH
I
N
2e

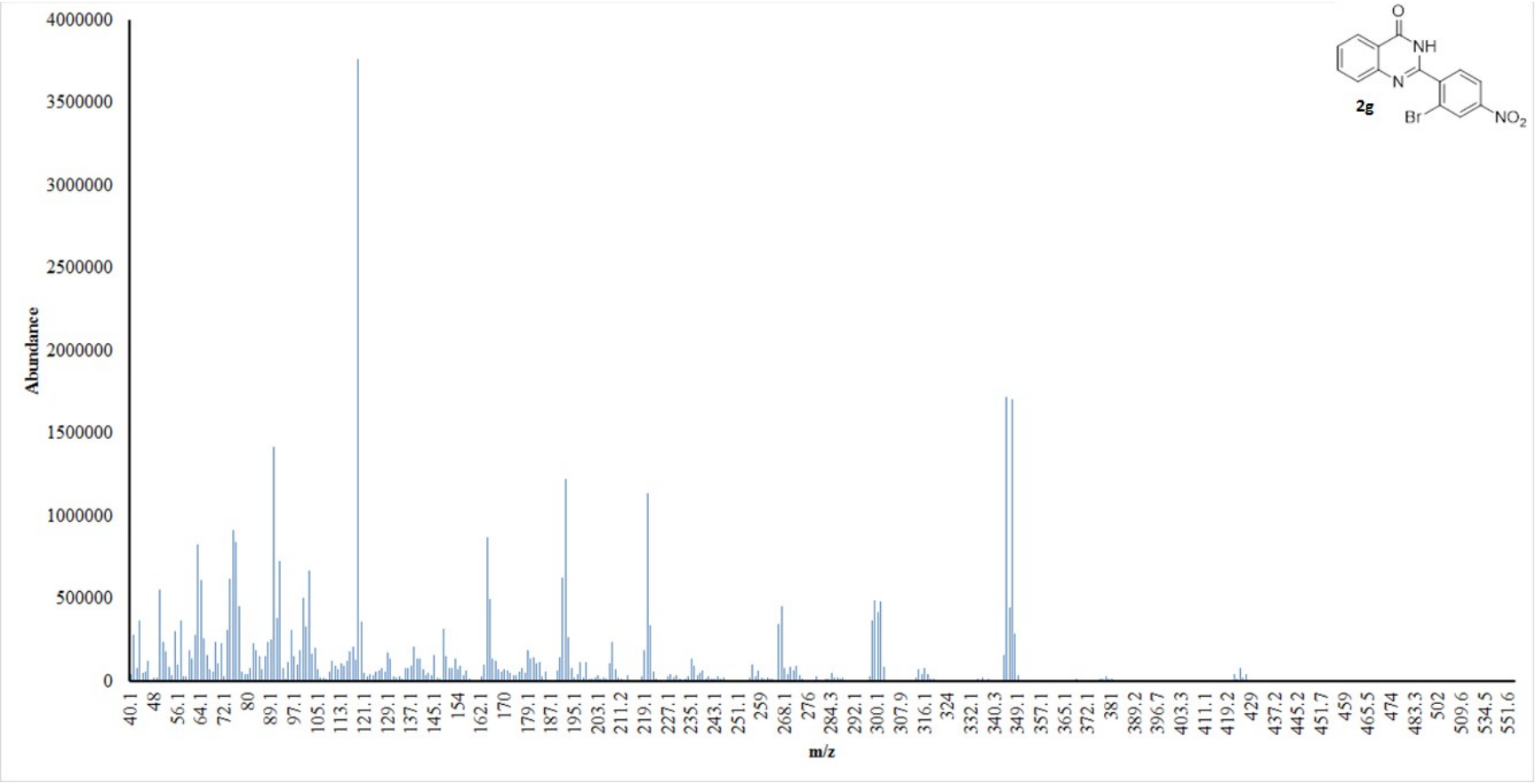

2f
12.62
8.26
8.23
8.20
8.17
8.15
7.95
7.94
7.89
7.86
7.84
7.83
7.76
7.73
7.70
7.68
7.65
7.65
7.62
7.62
7.61
7.59
7.56
7.54
7.52
3.32
2.50
0.45
1.25
1.94
4.00
f1 (ppm)
Current Data Parameters
NAME Q-Cl-Br
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161122
Time 13.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

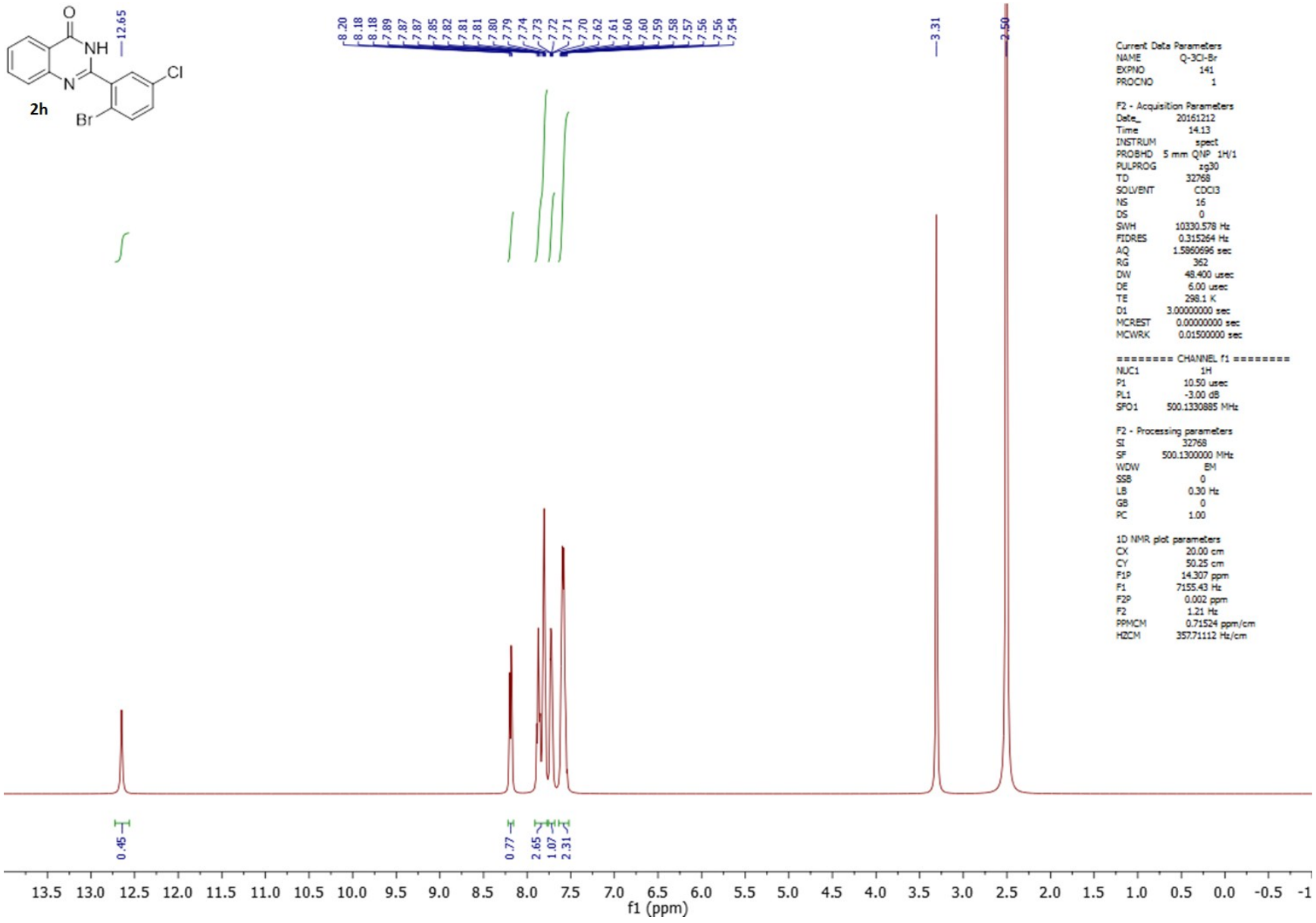

2f
O
NH
N
Br
Cl
161.86
152.96
148.94
135.82
135.26
135.11
132.53
132.44
128.33
127.97
127.67
126.32
122.42
121.77
40.84
40.56
40.28
40.00
39.73
39.45
39.17
Current Data Parameters
NAME Q-Cl-Br
EXPNO 87
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161106
Time 14.52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 358
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

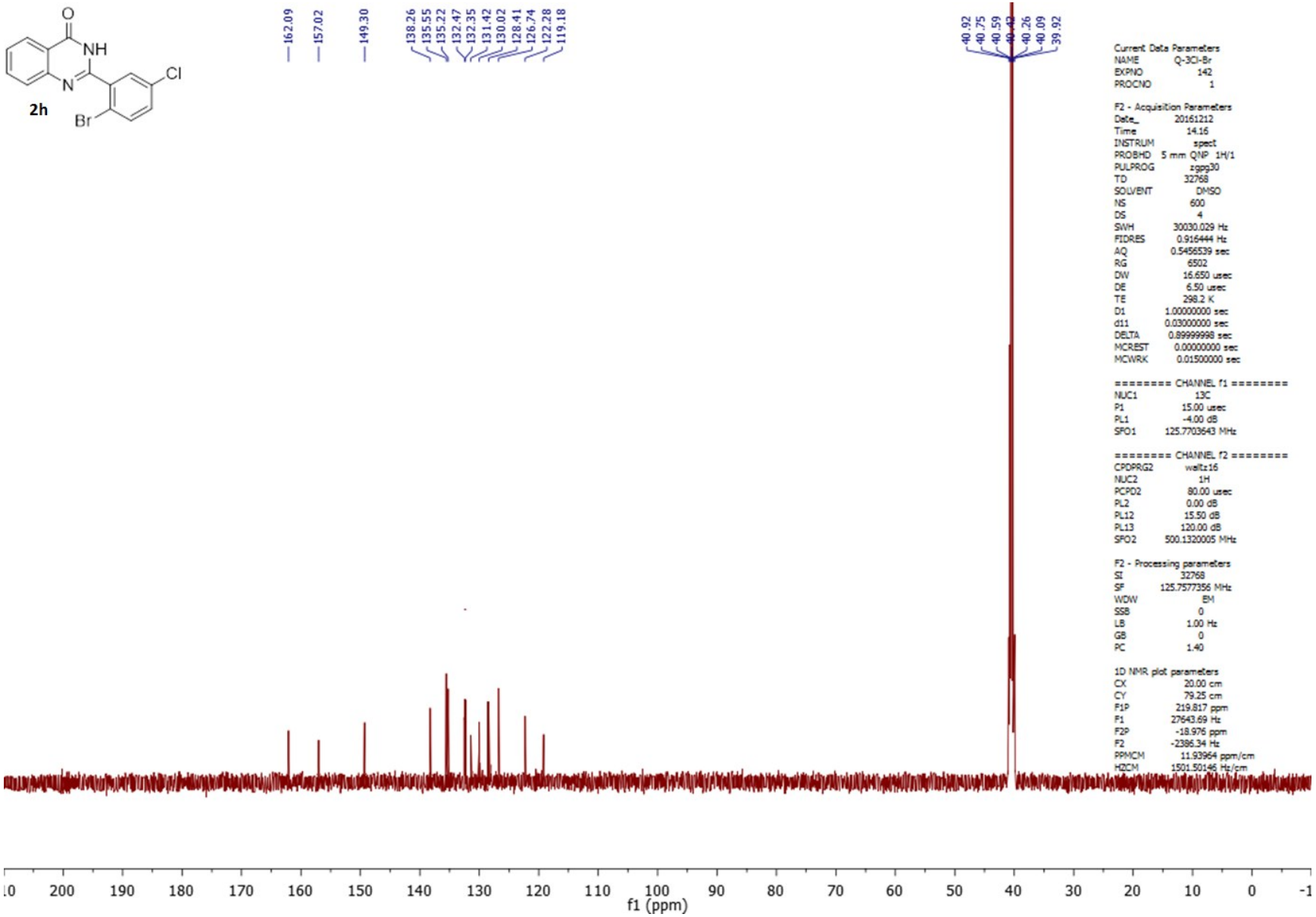

2f
Abundance
m/z
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
40.1
47
54.1
61.1
68.2
75.1
82.2
89.1
96.1
103.1
110
117.1
124
131
138
145
152.1
158.1
165
174
181
188
195
202
209.1
215.9
223
230
237.1
244
251
258
265.1
272
279
286.1
292.9
300
307
314.1
320.9
328.1
337
344
352
359
365
372
378.9
386.1
394
403.2
410.4
417.9
423.3
429.3
439.2
446.3
465.5
494.6

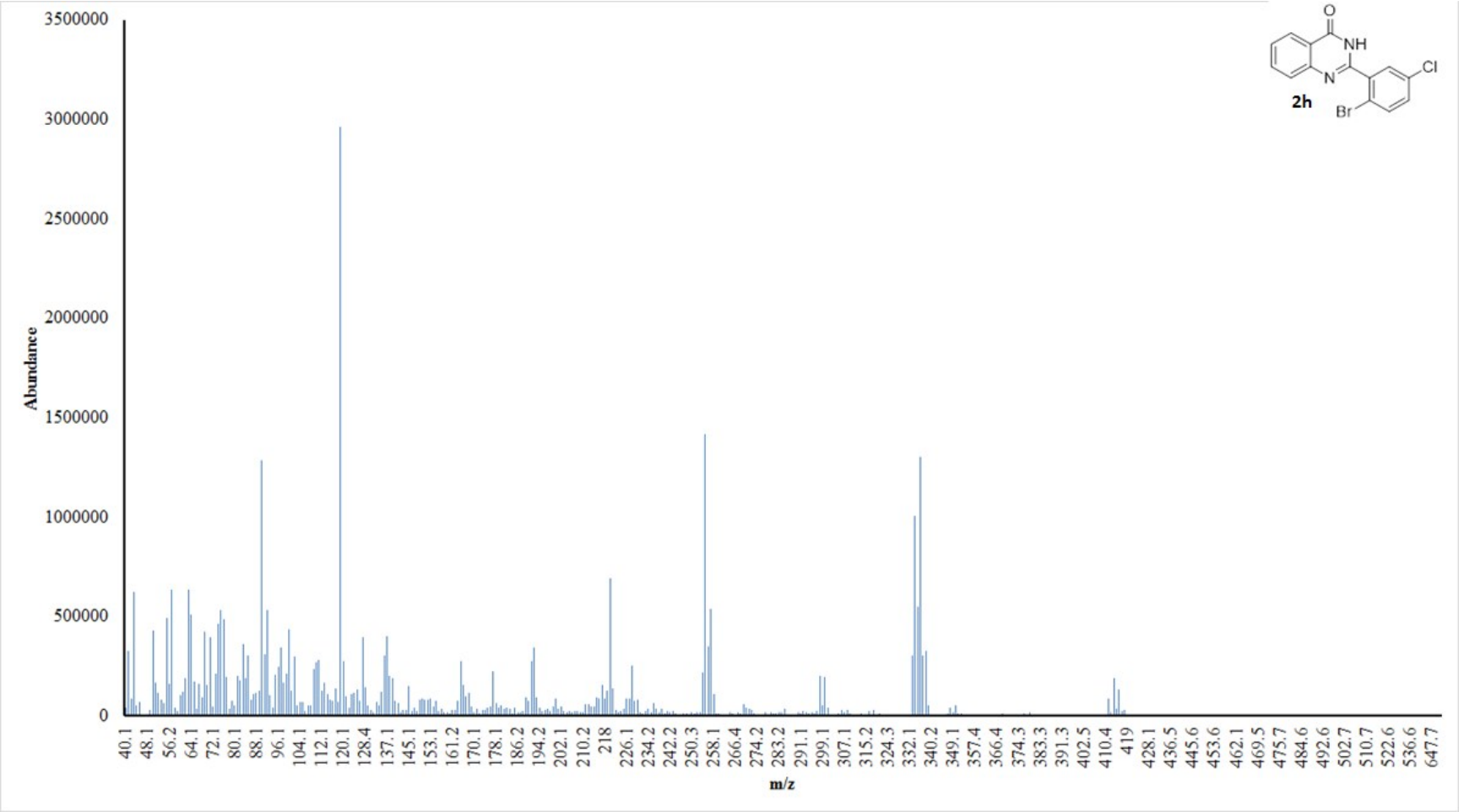

2g
12.83
8.65
8.59
8.40
8.37
8.22
8.19
7.98
7.96
7.92
7.89
7.86
7.78
7.75
7.73
7.67
7.64
7.62
7.59
3.33
2.50
Current Data Parameters
NAME Q-NO2-Br
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161113
Time 11.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.28
1.18
1.15
1.12
3.76
f1 (ppm)

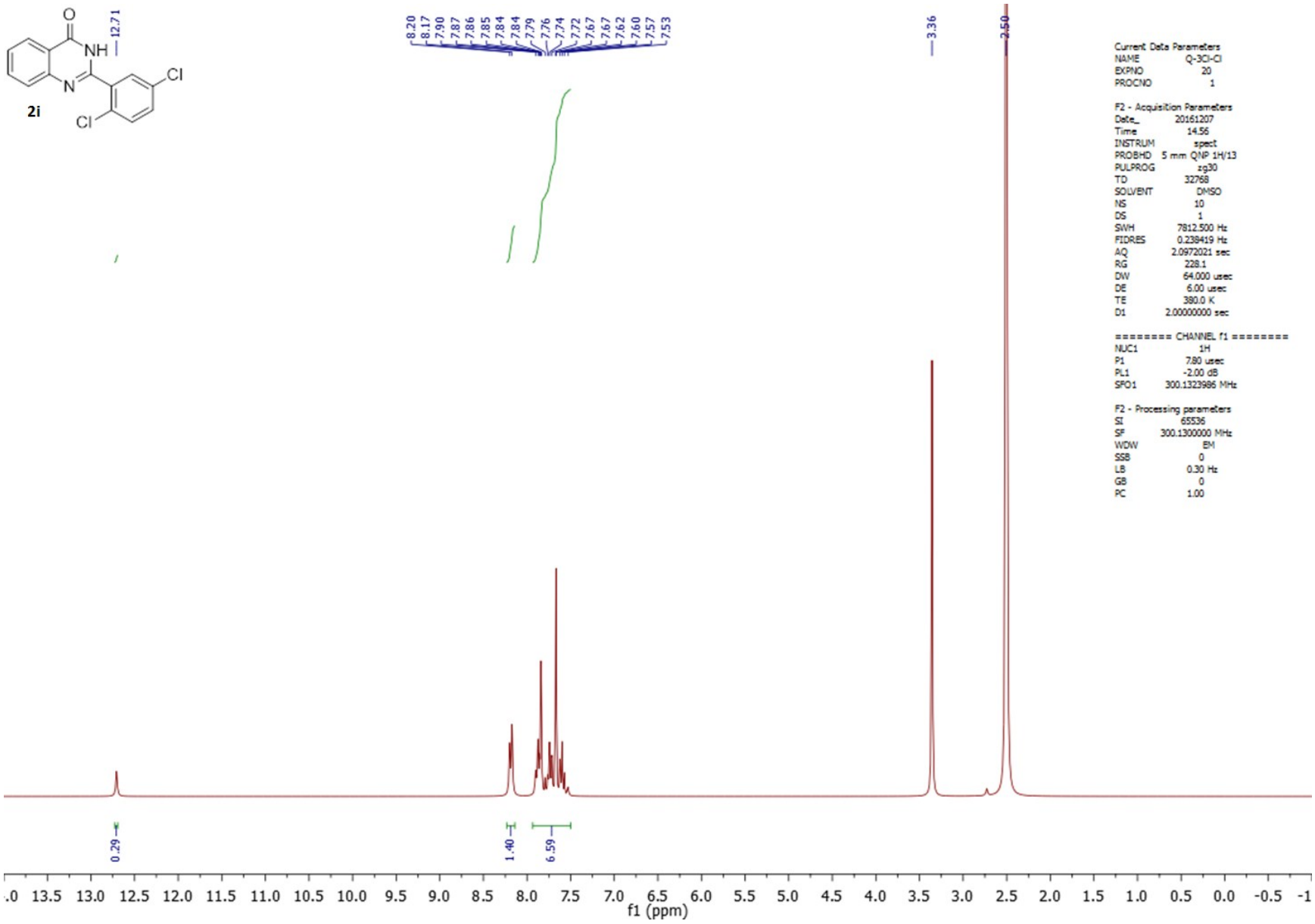

O
NH
N
Br
NO2
2g
166.12
160.97
149.54
149.21
135.66
132.92
128.47
128.26
127.51
126.81
124.13
123.63
122.78
122.35
40.93
40.76
40.59
40.43
40.26
40.09
39.92
Current Data Parameters
NAME Q-NO2-Br
EXPNO 127
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161121
Time 16.54
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 513
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 37.05 cm
F1P 219.817 ppm
F1 27643.69 Hz
F2P -18.976 ppm
F2 -2386.34 Hz
PPMCM 11.93964 ppm/cm
HZCM 1501.50146 Hz/cm
f1 (ppm)

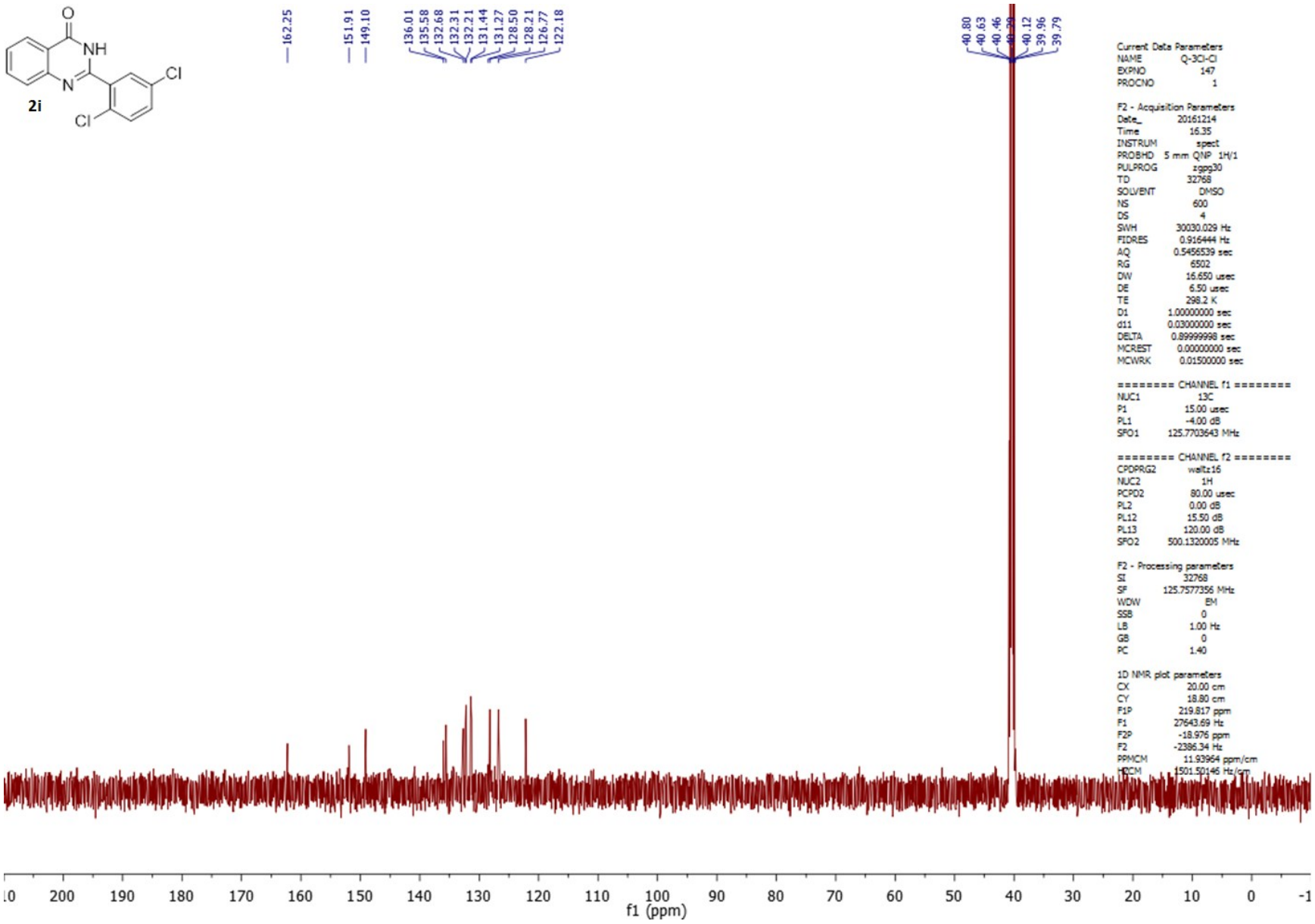

Abundance
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
40.1
48
56.1
64.1
72.1
80
89.1
97.1
105.1
113.1
121.1
129.1
137.1
145.1
154
162.1
170
179.1
187.1
195.1
203.1
211.2
219.1
227.1
235.1
243.1
251.1
259
268.1
276
284.3
292.1
300.1
307.9
316.1
324
332.1
340.3
349.1
357.1
365.1
372.1
381
389.2
396.7
403.3
411.1
419.2
429
437.2
445.2
451.7
459
465.5
474
483.3
502
509.6
534.5
551.6
m/z
O
NH
N
Br
NO2
2g

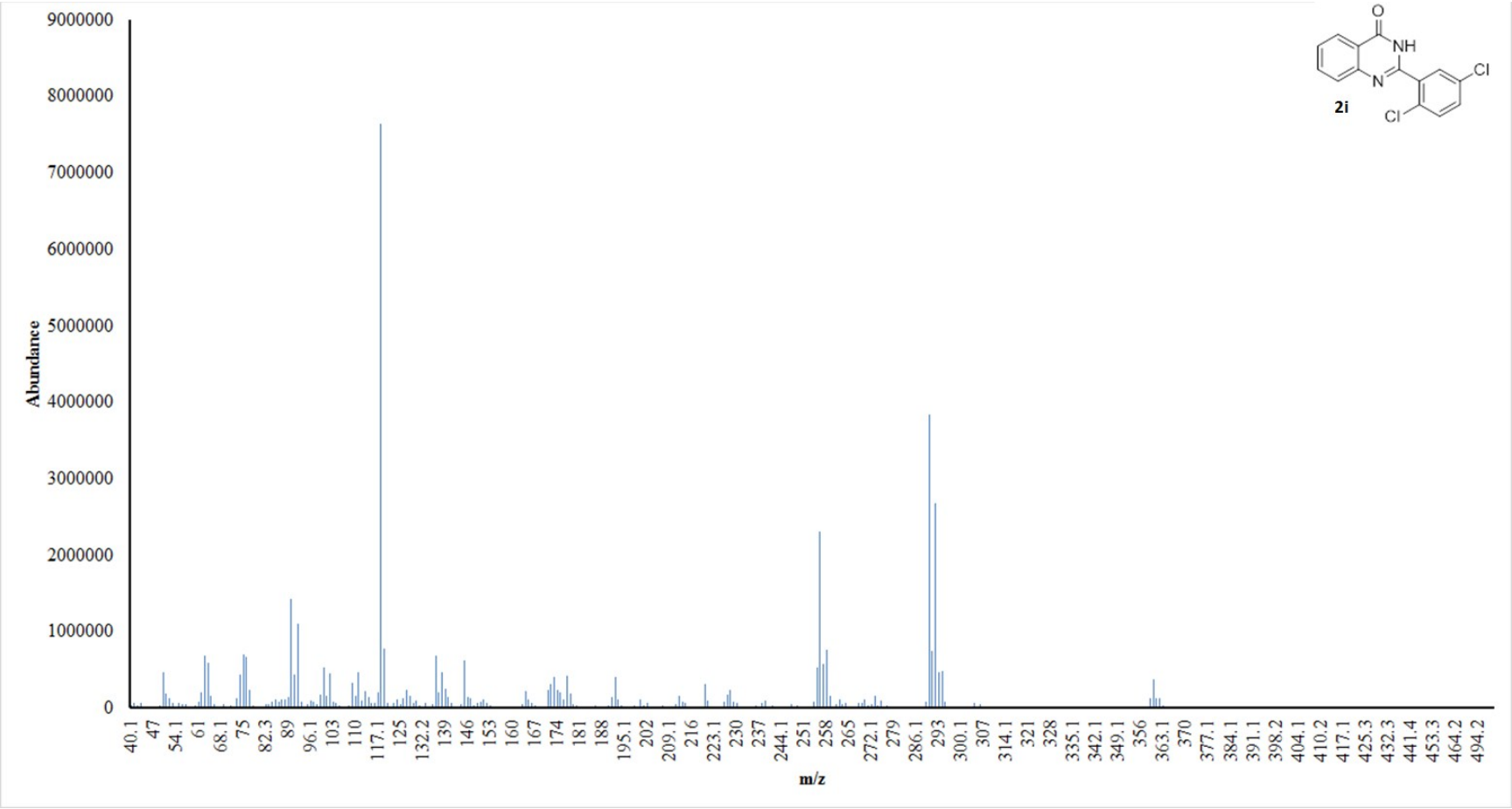

O
NH
N
Cl
Br
2h
12.65
8.20
8.18
8.18
7.89
7.87
7.87
7.85
7.82
7.81
7.81
7.80
7.79
7.74
7.73
7.72
7.71
7.70
7.62
7.61
7.60
7.60
7.59
7.58
7.57
7.56
7.56
7.54
3.31
2.50
Current Data Parameters
NAME Q-3Cl-Br
EXPNO 141
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161212
Time 14.13
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 362
DW 48.400 usec
DE 6.00 usec
TE 298.1 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 50.25 cm
F1P 14.307 ppm
F1 7155.43 Hz
F2P 0.002 ppm
F2 1.21 Hz
PPMCM 0.71524 ppm/cm
HZCM 357.71112 Hz/cm
0.45
0.77
2.65
1.07
2.31
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

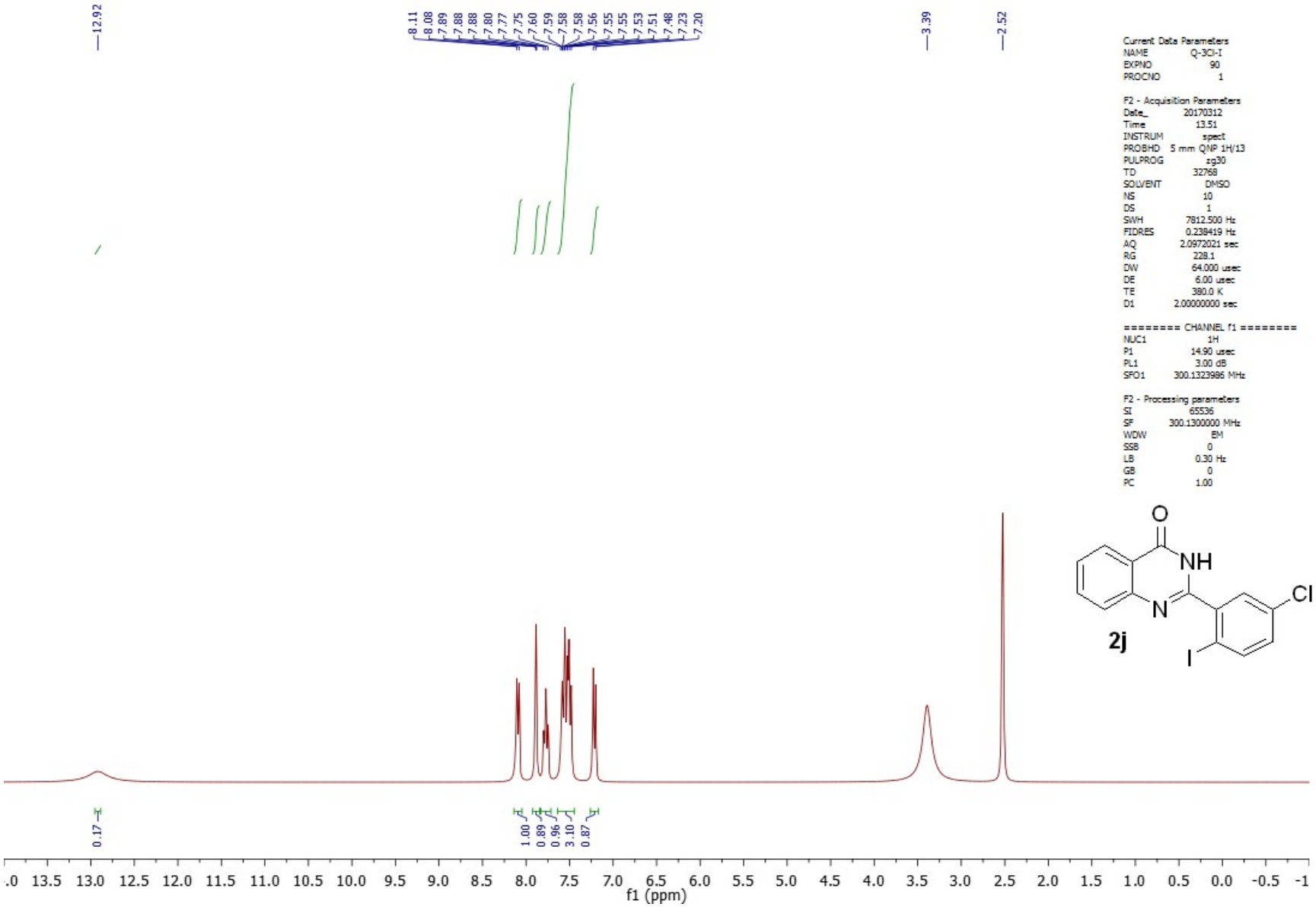

O
NH
N
Cl
Br
2h
162.09
157.02
149.30
138.26
135.55
135.22
132.47
132.35
131.42
130.02
128.41
126.74
122.28
119.18
40.92
40.75
40.59
40.43
40.26
40.09
39.92
Current Data Parameters
NAME Q-3Cl-Br
EXPNO 142
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161212
Time 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 600
DS 4
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 6502
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 79.25 cm
F1P 219.817 ppm
F1 27643.69 Hz
F2P -18.976 ppm
F2 -2386.34 Hz
PPMCM 11.93964 ppm/cm
HZCM 1501.50146 Hz/cm
f1 (ppm)

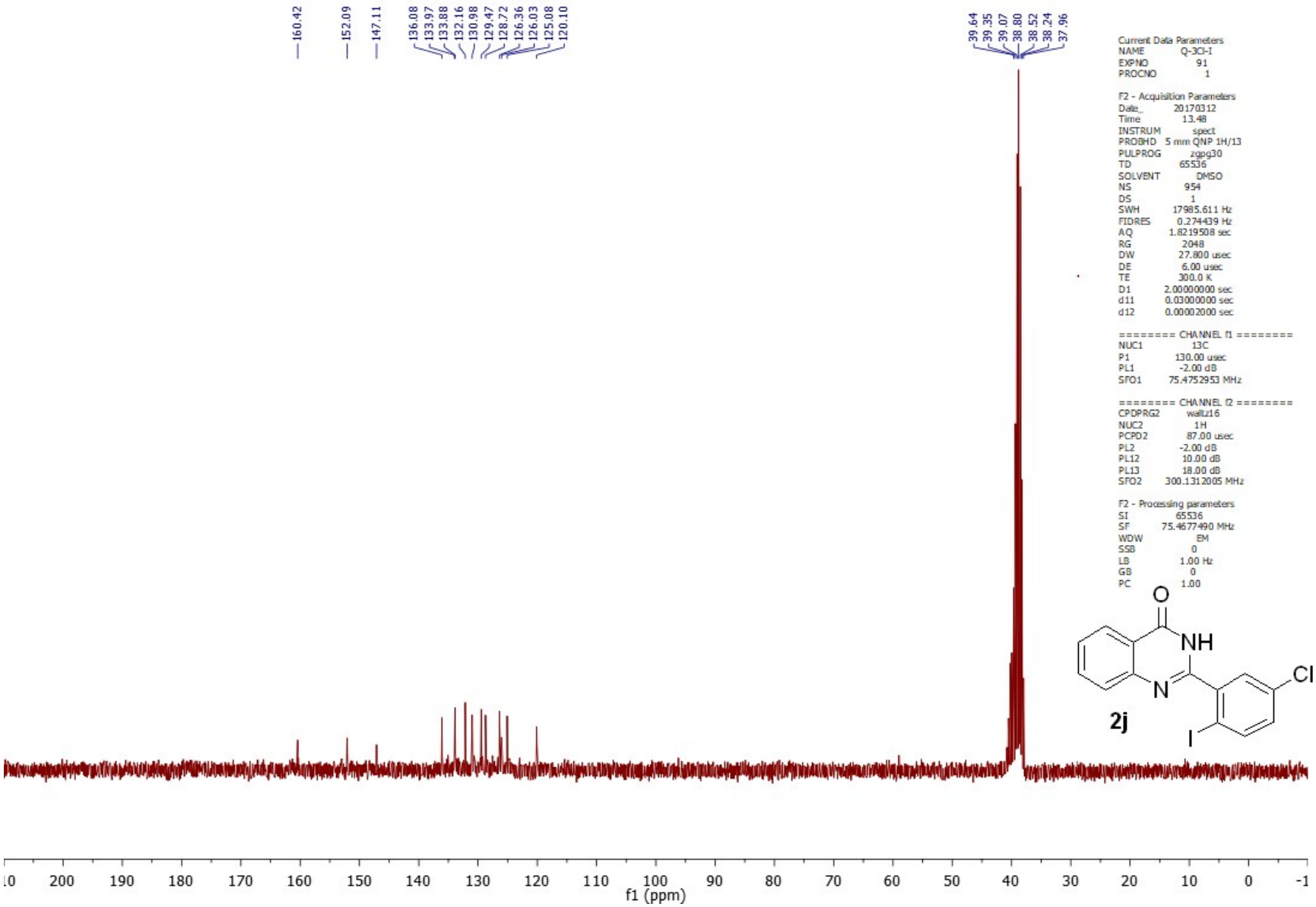

Abundance
m/z
2h
O
NH
N
Cl
Br

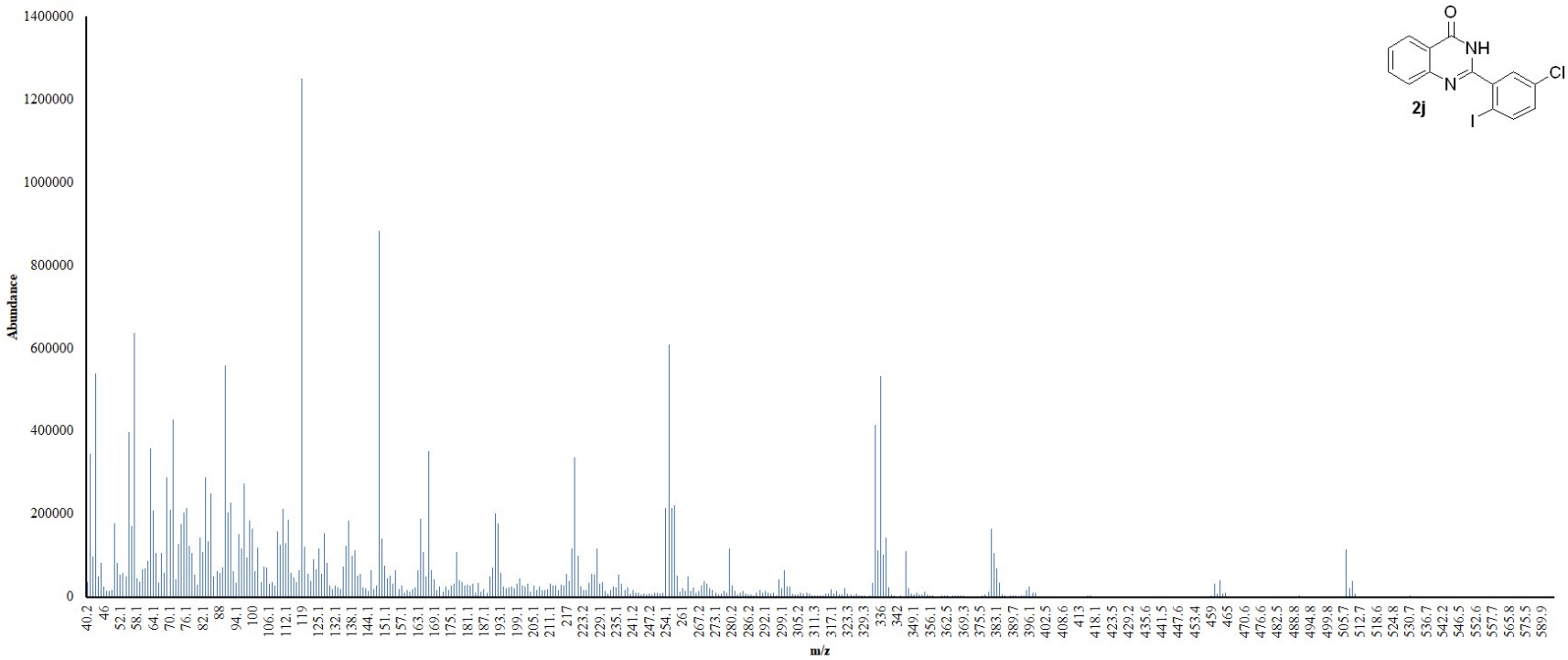

2i
12.71
8.20
8.17
7.90
7.87
7.86
7.85
7.84
7.84
7.84
7.79
7.76
7.74
7.72
7.67
7.67
7.62
7.60
7.57
7.53
3.36
2.50
Current Data Parameters
NAME Q-3Cl-Cl
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161207
Time 14.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.29
1.40
6.59
f1 (ppm)

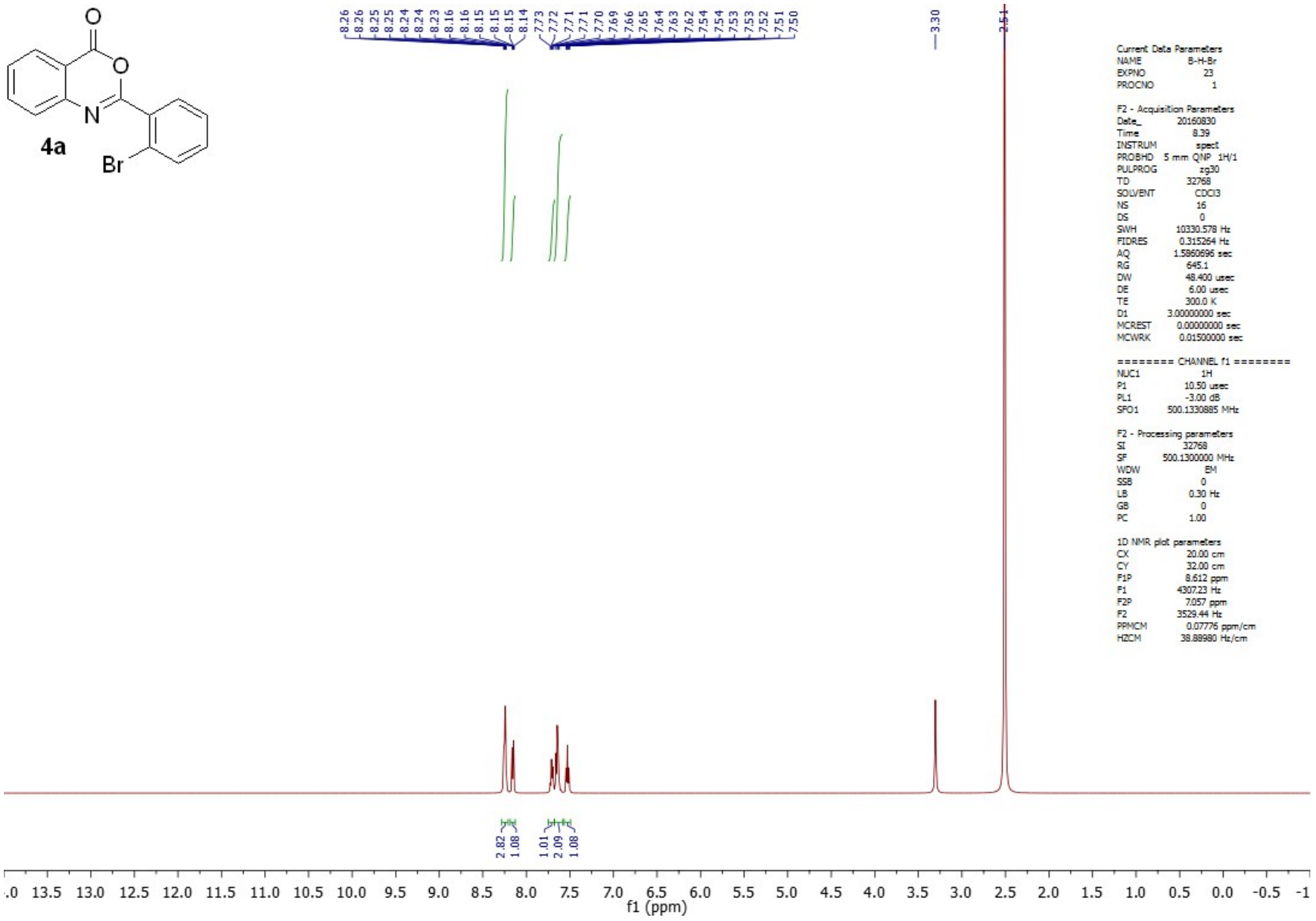

2i
162.25
151.91
149.10
136.01
135.58
132.68
132.31
132.21
131.44
131.27
128.50
128.21
126.77
122.18
40.80
40.63
40.46
40.29
40.12
39.96
39.79
Current Data Parameters
NAME Q-3Cl-Cl
EXPNO 147
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161214
Time 16.35
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 600
DS 4
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 6502
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 18.80 cm
F1P 219.817 ppm
F1 27643.69 Hz
F2P -18.976 ppm
F2 -2386.34 Hz
PPMCM 11.93964 ppm/cm
HZCM 1501.50146 Hz/cm
f1 (ppm)

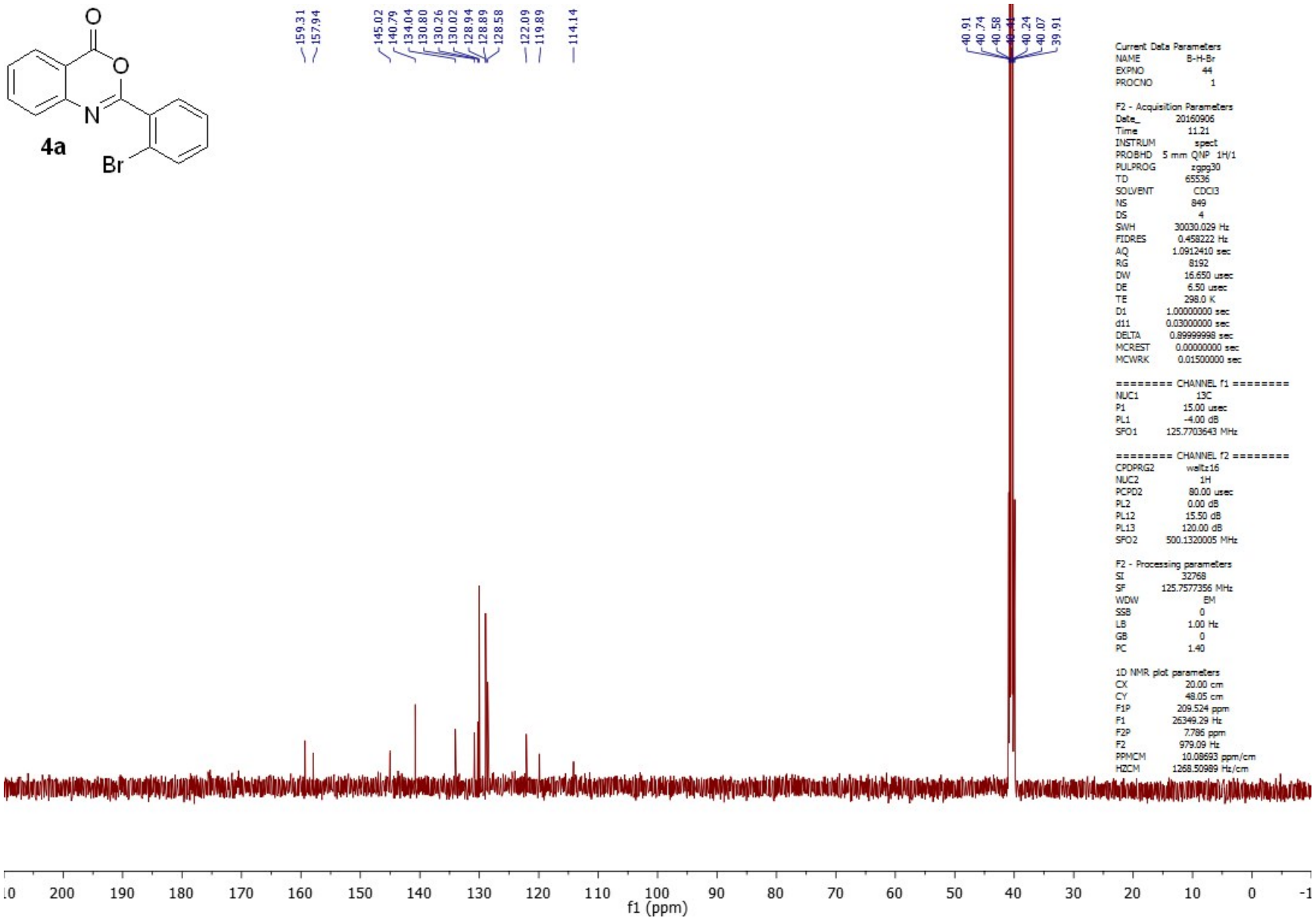

O
NH
N
Cl
Cl
2i
Abundance
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
40.1 47 54.1 61 68.1 75 82.3 89 96.1 103 110 117.1 125 132.2 139 146 153 160 167 174 181 188 195.1 202 209.1 216 223.1 230 237 244.1 251 258 265 272.1 279 286.1 293 300.1 307 314.1 321 328 335.1 342.1 349.1 356 363.1 370 377.1 384.1 391.1 398.2 404.1 410.2 417.1 425.3 432.3 441.4 453.3 464.2 494.2
m/z

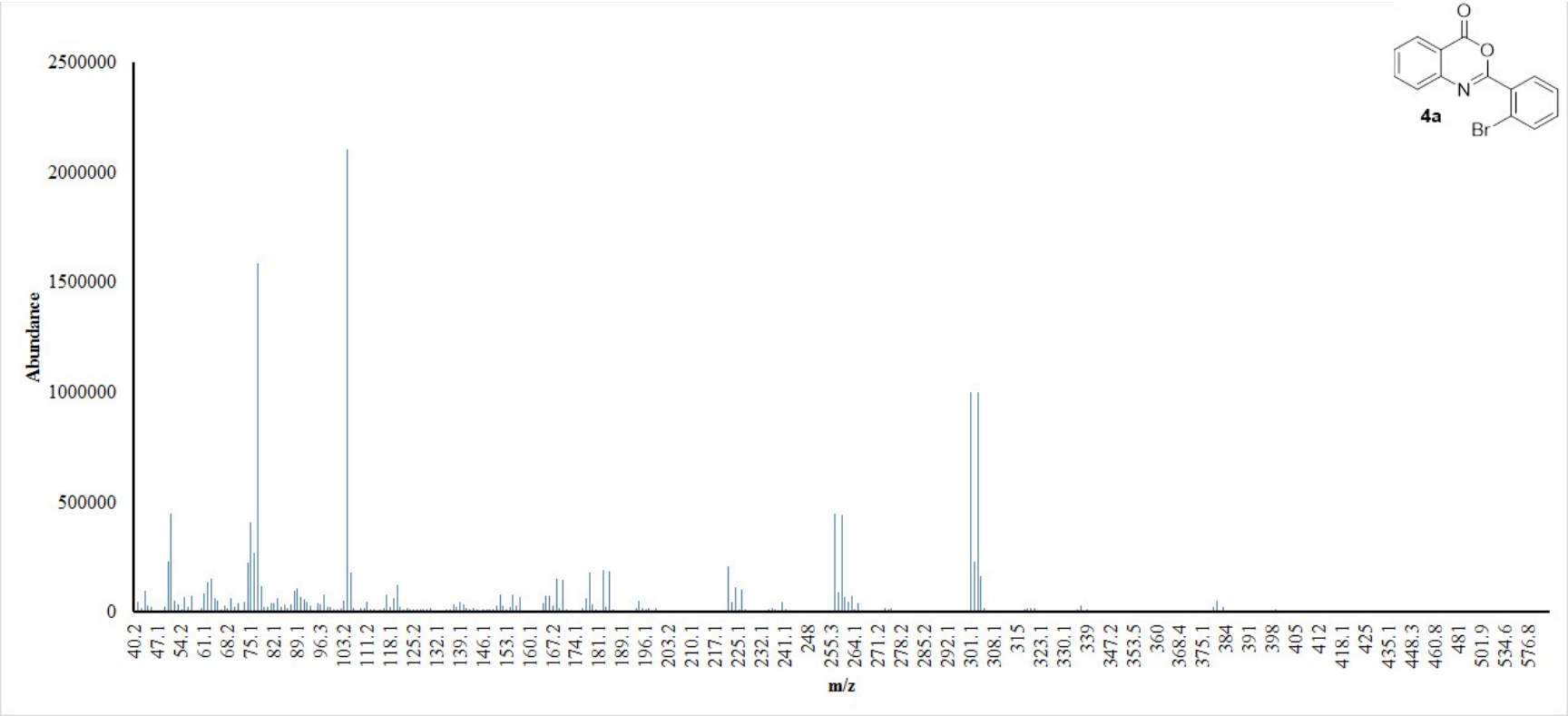

12.92
8.11
8.08
7.89
7.88
7.88
7.80
7.77
7.75
7.60
7.59
7.58
7.58
7.56
7.55
7.55
7.53
7.51
7.48
7.23
7.20
3.39
2.52
Current Data Parameters
NAME Q-3Cl-I
EXPNO 90
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170312
Time 13.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
NH
N
Cl
I
2j
0.17
1.00
0.89
0.96
3.10
0.87
f1 (ppm)

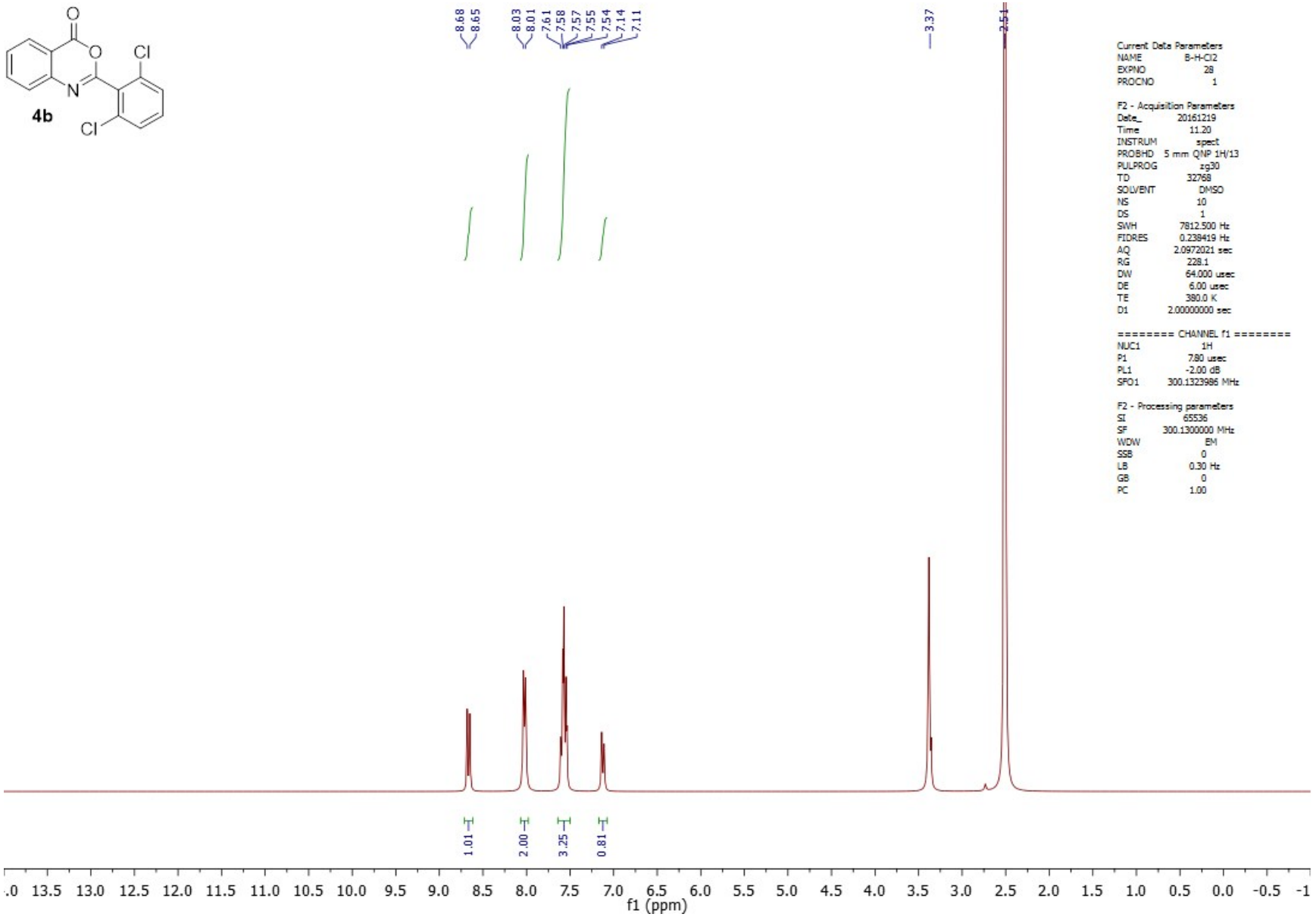

160.42
152.09
147.11
136.08
133.97
133.88
132.16
130.98
129.47
128.72
126.36
126.03
125.08
120.10
39.64
39.35
39.07
38.80
38.52
38.24
37.96
Current Data Parameters
NAME Q-3Cl-I
EXPNO 91
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170312
Time 13.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 954
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
NH
N
Cl
I
2j
f1 (ppm)

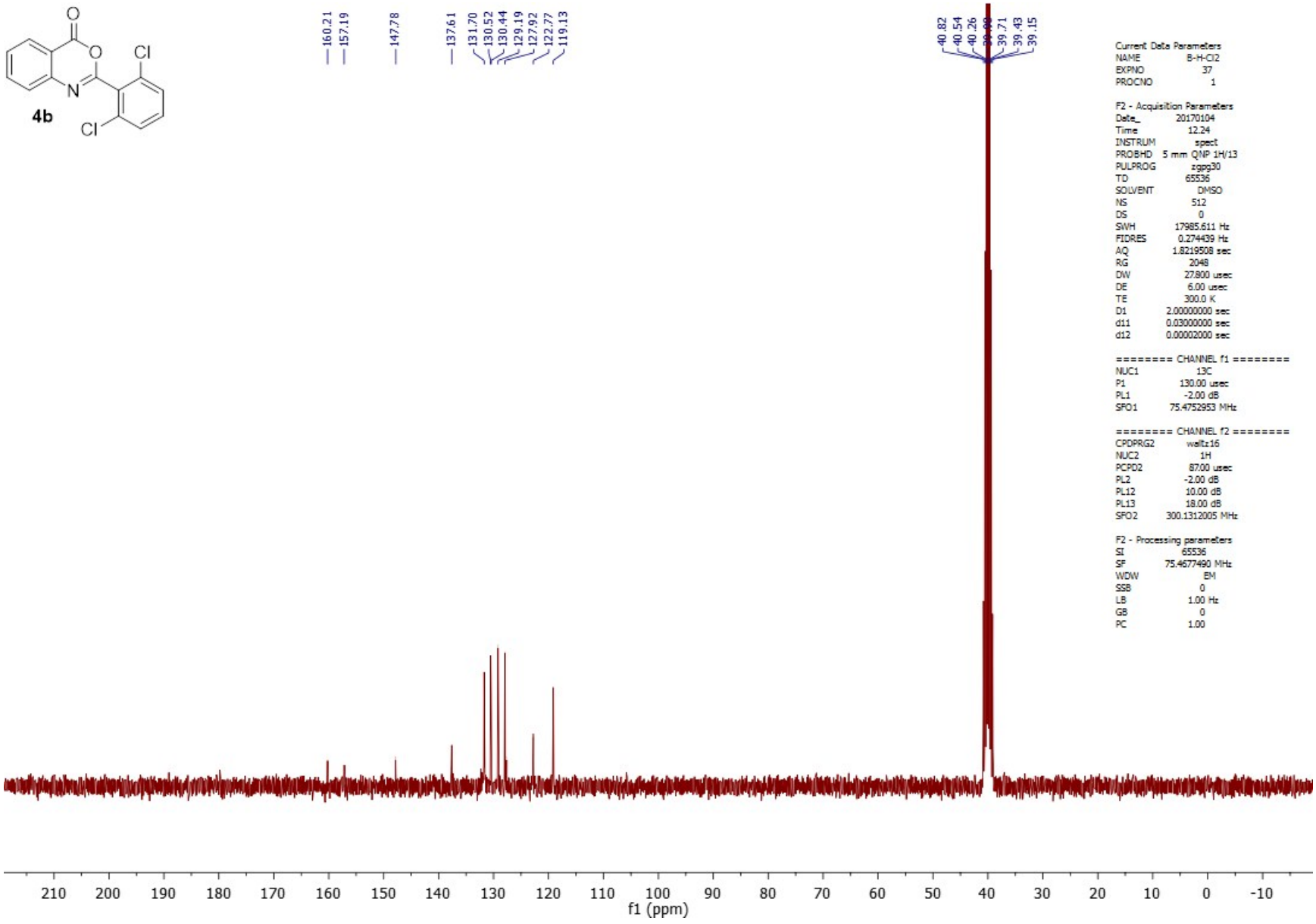

Abundance
m/z
2j

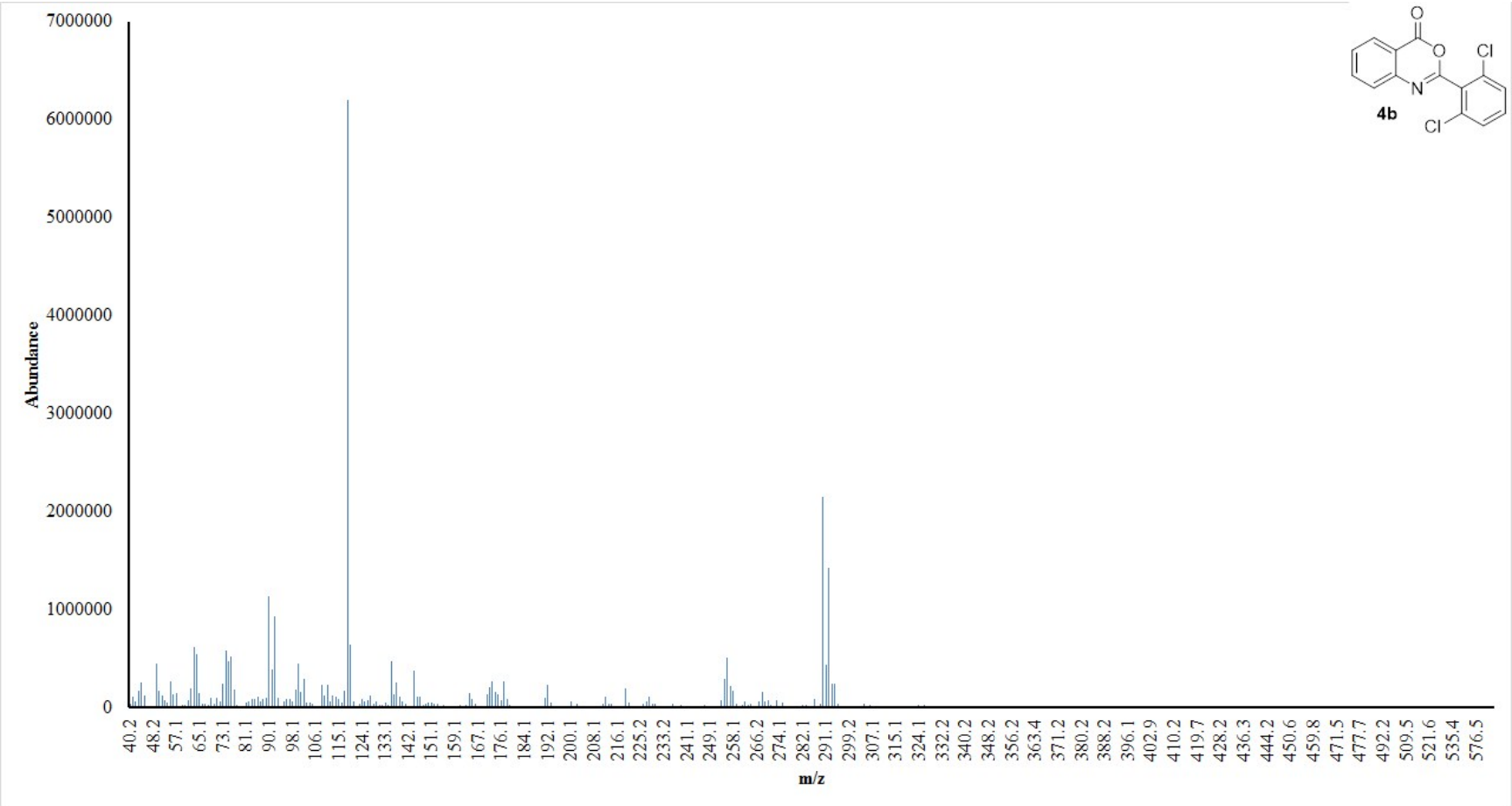

4a
Br
8.26 8.26 8.25 8.25 8.24 8.24 8.23 8.16 8.16 8.15 8.15 8.15 8.14 7.73 7.72 7.71 7.71 7.70 7.69 7.66 7.65 7.64 7.63 7.62 7.54 7.54 7.53 7.53 7.52 7.51 7.50
3.30
2.51
2.82 1.08 1.01 2.09 1.08
f1 (ppm)
Current Data Parameters
NAME B-H-Br
EXPNO 23
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160830
Time 8.39
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 645.1
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 32.00 cm
F1P 8.612 ppm
F1 4307.23 Hz
F2P 7.057 ppm
F2 3529.44 Hz
PPMCM 0.07776 ppm/cm
HZCM 38.88980 Hz/cm

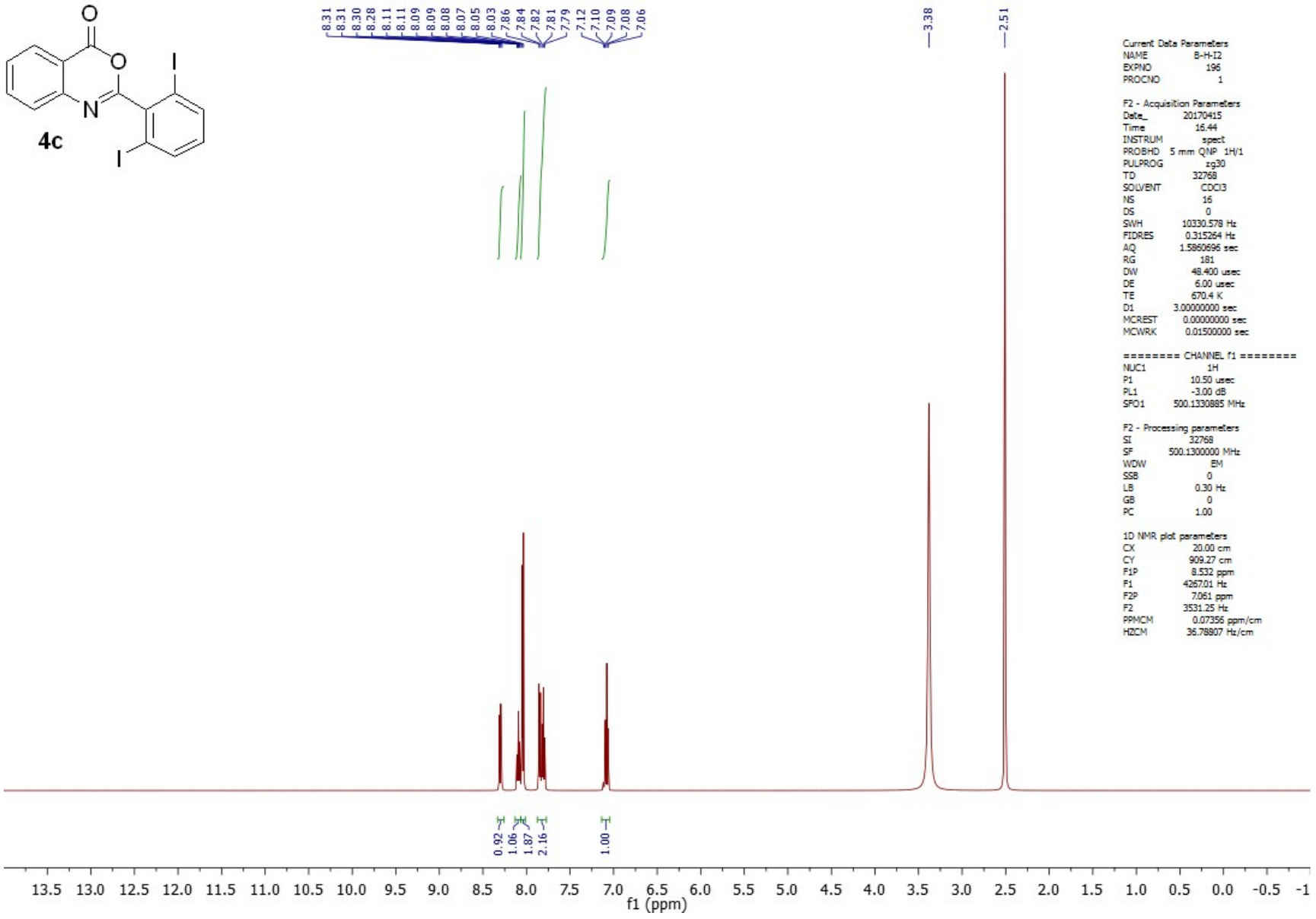

4a
Br
159.31
157.94
145.02
140.79
134.04
130.80
130.26
130.02
128.94
128.89
128.58
122.09
119.89
114.14
40.91
40.74
40.58
40.24
40.07
39.91
Current Data Parameters
NAME B-H-Br
EXPNO 44
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160906
Time 11.21
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 849
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 48.05 cm
F1P 209.524 ppm
F1 26349.29 Hz
F2P 7.786 ppm
F2 979.09 Hz
PPMCM 10.08693 ppm/cm
HZCM 1268.50989 Hz/cm
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

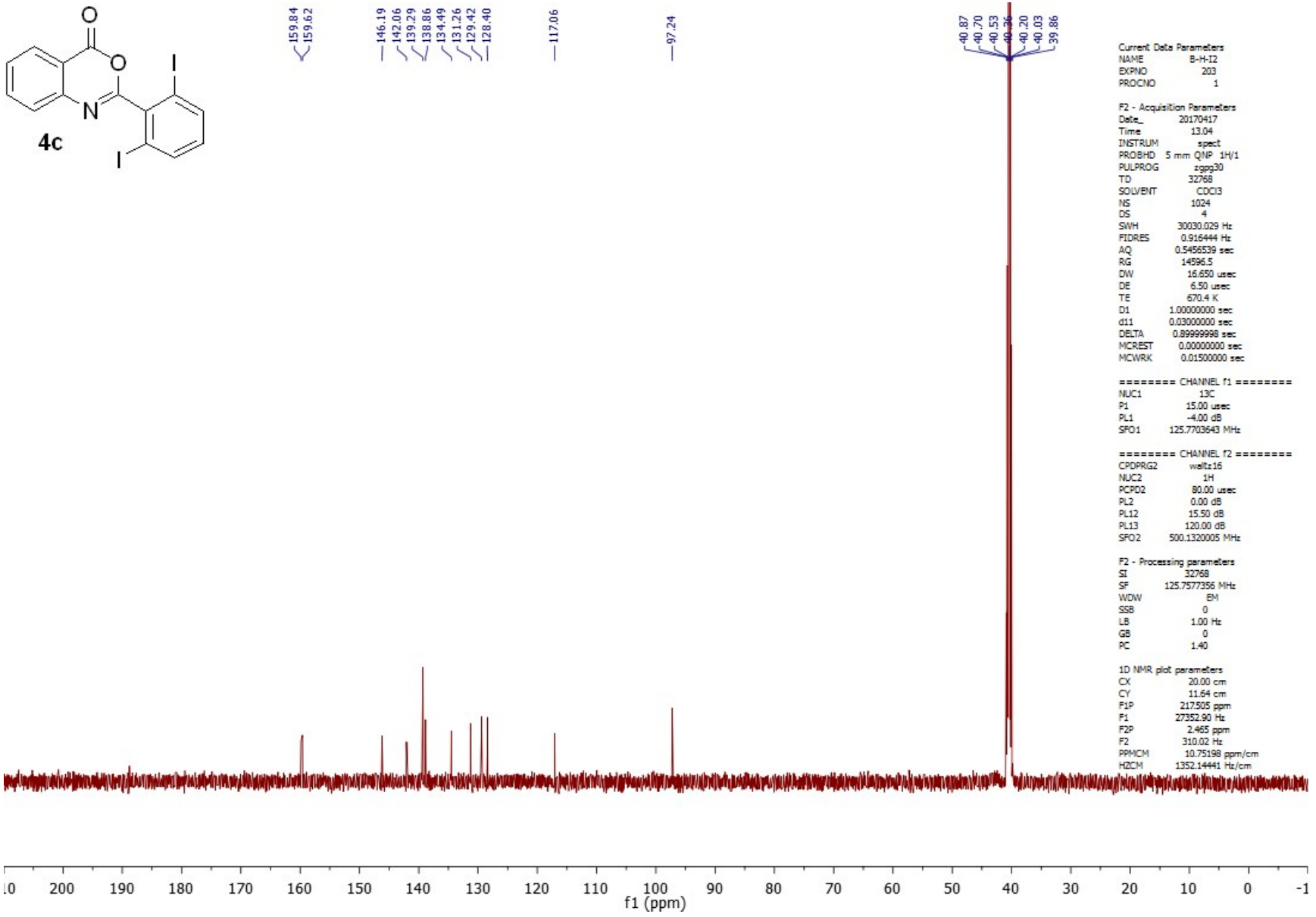

4a
Abundance
2500000
2000000
1500000
1000000
500000
0
m/z
40.2 47.1 54.2 61.1 68.2 75.1 82.1 89.1 96.3 103.2 111.2 118.1 125.2 132.1 139.1 146.1 153.1 160.1 167.2 174.1 181.1 189.1 196.1 203.2 210.1 217.1 225.1 232.1 241.1 248 255.3 264.1 271.2 278.2 285.2 292.1 301.1 308.1 315 323.1 330.1 339 347.2 353.5 360 368.4 375.1 384 391 398 405 412 418.1 425 435.1 448.3 460.8 481 501.9 534.6 576.8

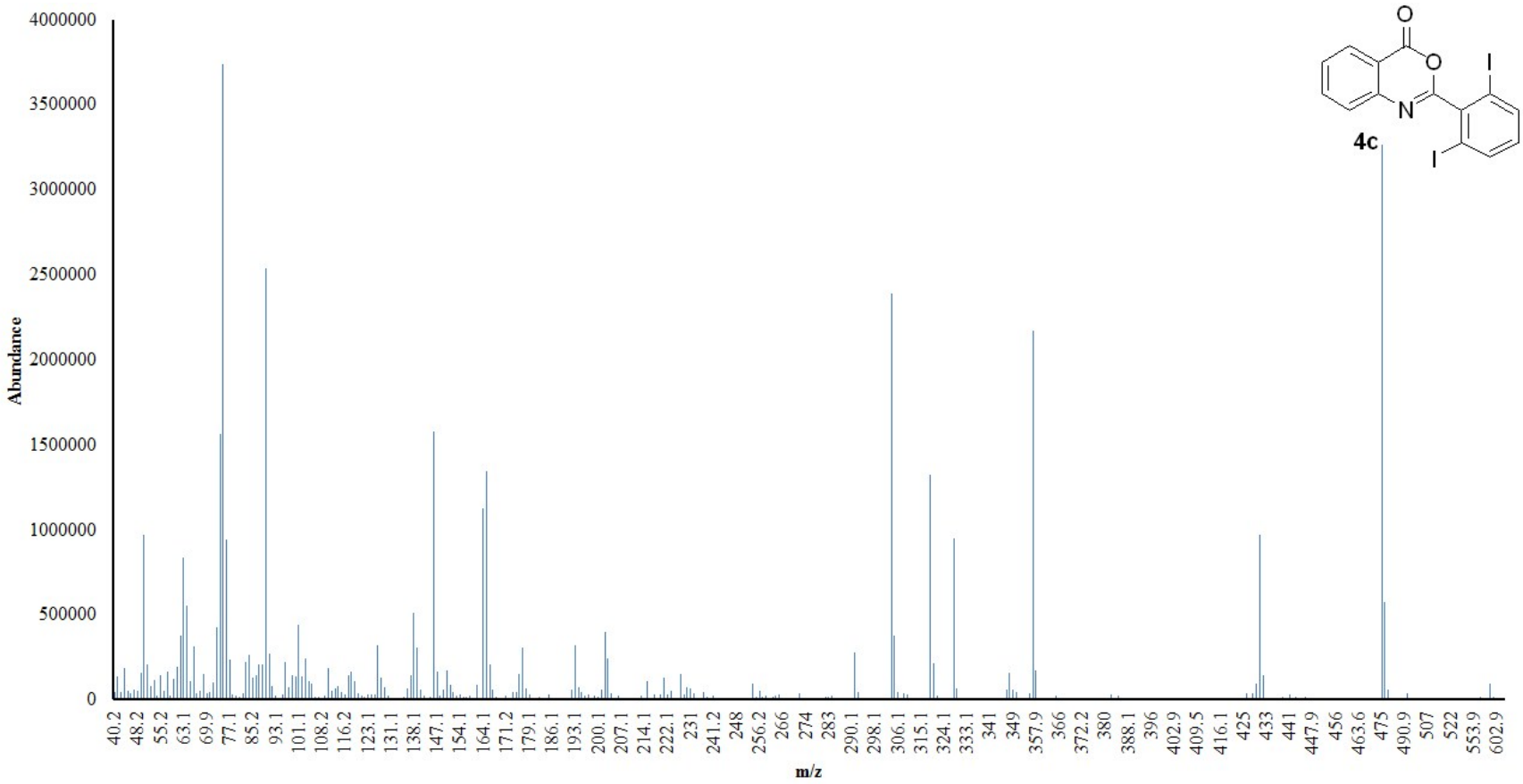

4b
8.68
8.65
8.03
8.01
7.61
7.58
7.57
7.55
7.54
7.14
7.11
3.37
2.51
Current Data Parameters
NAME B-H-Cl2
EXPNO 28
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161219
Time 11.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.01
2.00
3.25
0.81
f1 (ppm)

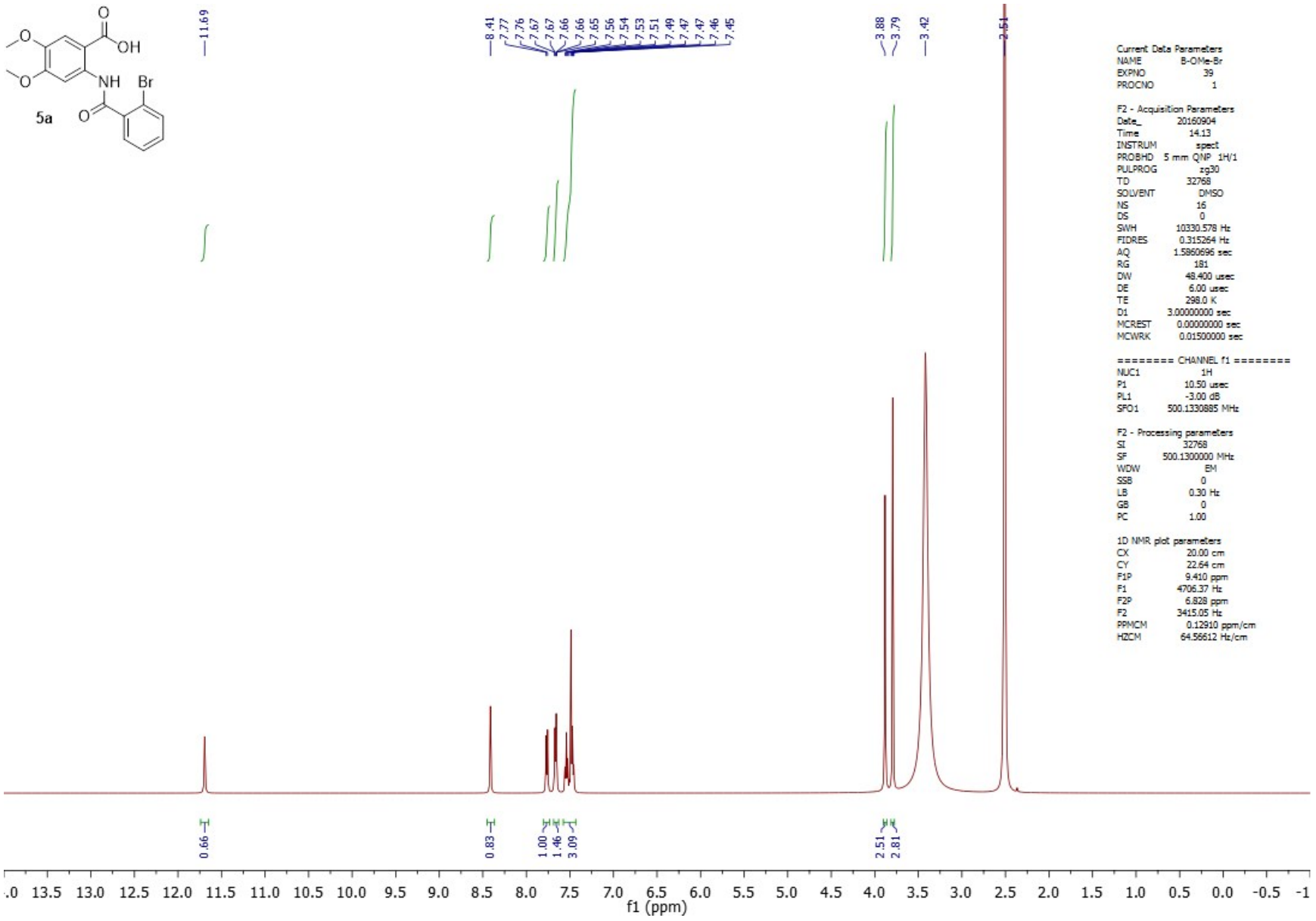

4b
160.21
157.19
147.78
137.61
131.70
130.52
130.44
129.19
127.92
122.77
119.13
40.82
40.54
40.26
39.98
39.71
39.43
39.15
Current Data Parameters
NAME B-H-Cl2
EXPNO 37
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170104
Time 12.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 0
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

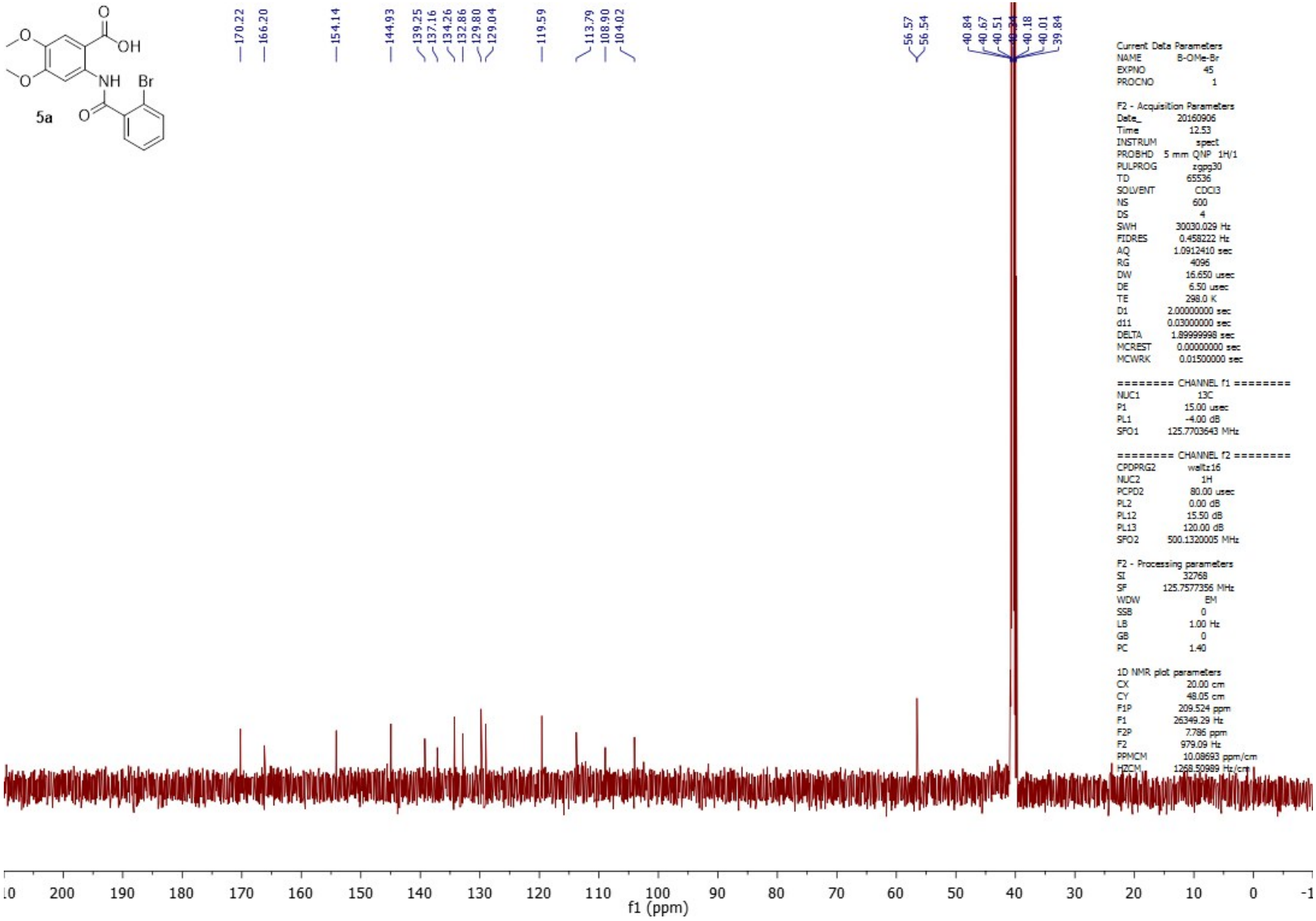

Abundance
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
40.2
48.2
57.1
65.1
73.1
81.1
90.1
98.1
106.1
115.1
124.1
133.1
142.1
151.1
159.1
167.1
176.1
184.1
192.1
200.1
208.1
216.1
225.2
233.2
241.1
249.1
258.1
266.2
274.1
282.1
291.1
299.2
307.1
315.1
324.1
332.2
340.2
348.2
356.2
363.4
371.2
380.2
388.2
396.1
402.9
410.2
419.7
428.2
436.3
444.2
450.6
459.8
471.5
477.7
492.2
509.5
521.6
535.4
576.5
m/z
O
O
N
Cl
Cl
4b

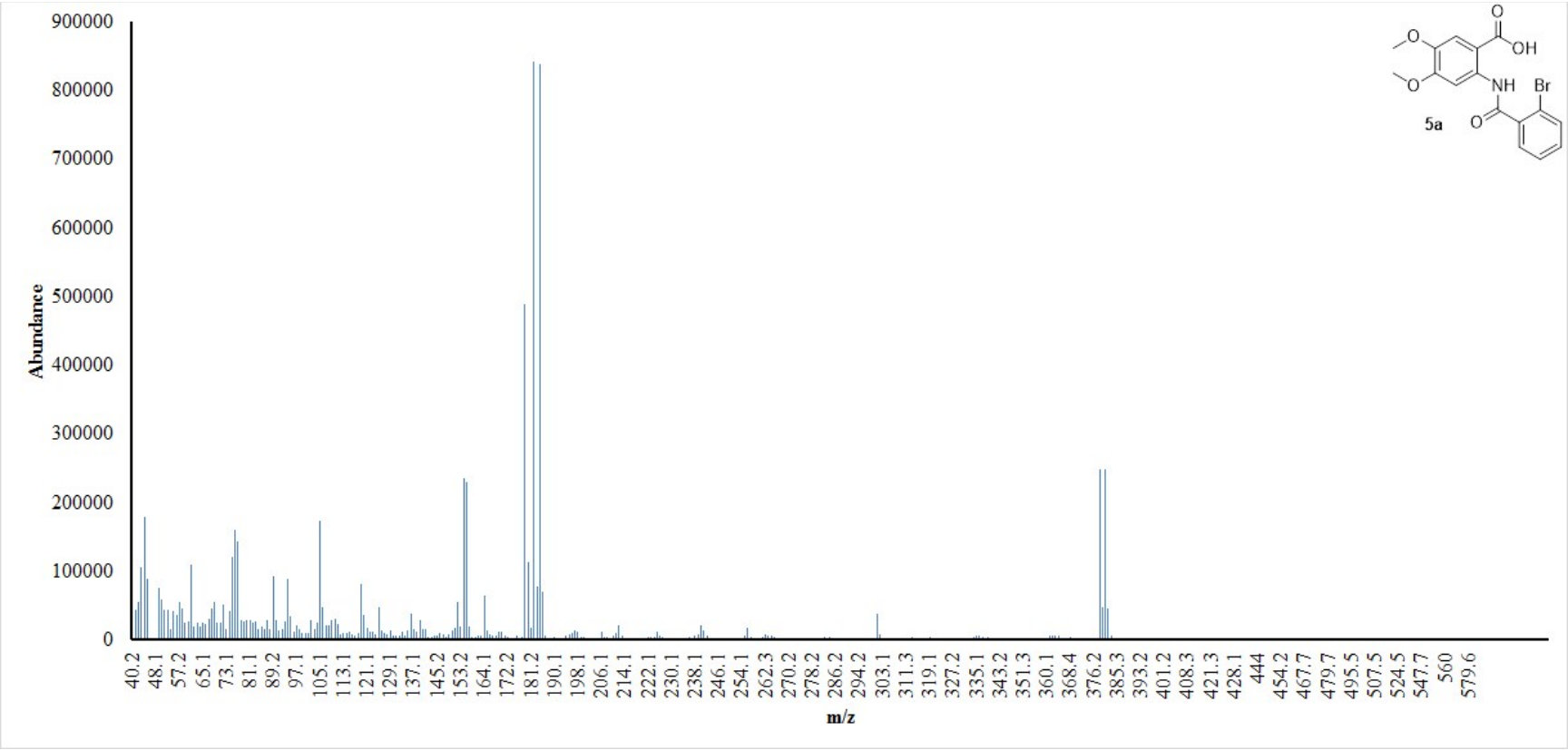

4c
8.31
8.31
8.30
8.28
8.11
8.11
8.09
8.09
8.08
8.07
8.05
8.03
7.86
7.84
7.82
7.81
7.79
7.12
7.10
7.09
7.08
7.06
3.38
2.51
Current Data Parameters
NAME B-H-I2
EXPNO 196
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170415
Time 16.44
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 670.4 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 909.27 cm
F1P 8.532 ppm
F1 4267.01 Hz
F2P 7.061 ppm
F2 3531.25 Hz
PPMCM 0.07356 ppm/cm
HZCM 36.78807 Hz/cm
0.92
1.06
1.87
2.16
1.00
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

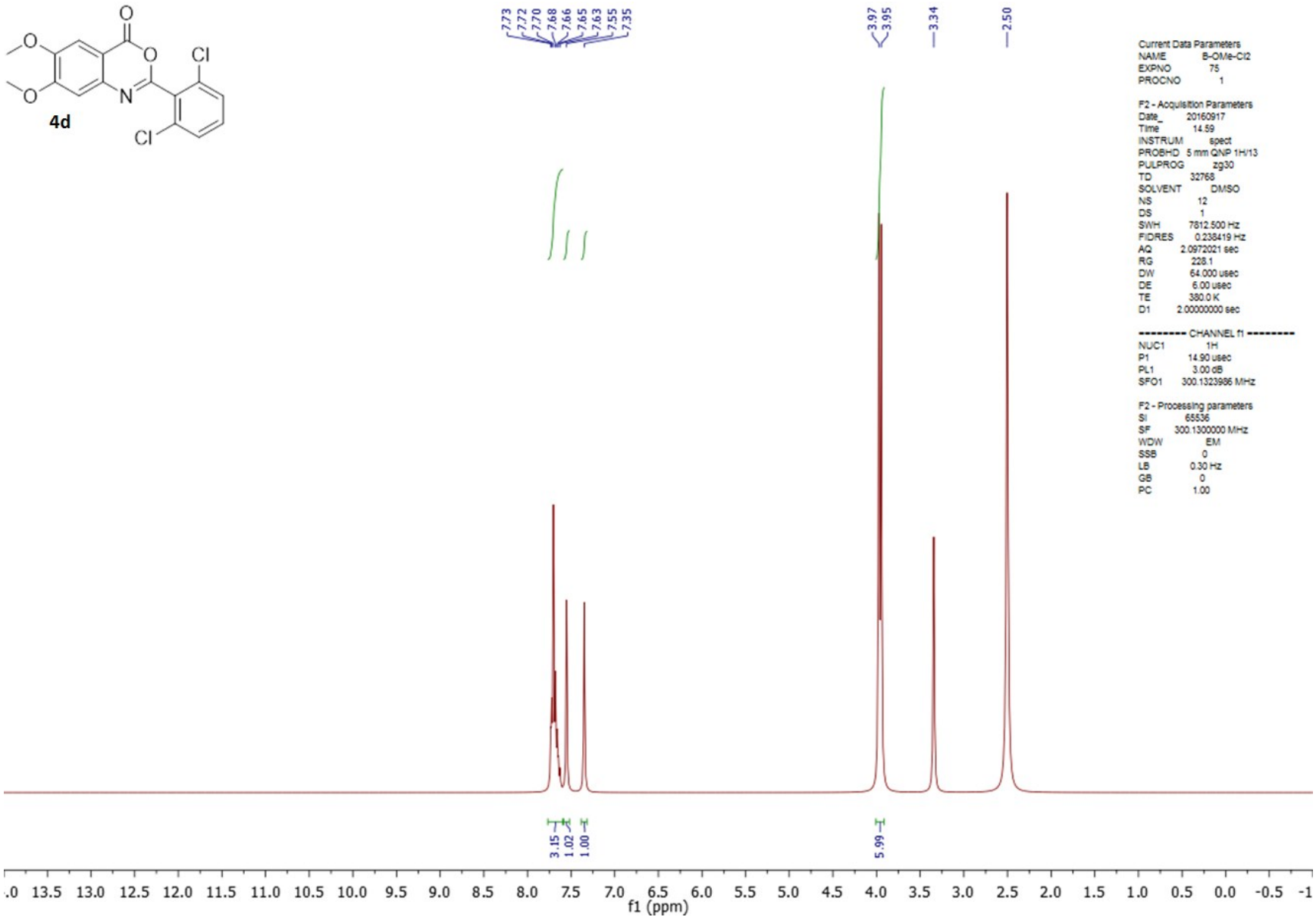

4c
159.84
159.62
146.19
142.06
139.29
138.86
134.49
131.26
129.42
128.40
117.06
97.24
40.87
40.70
40.53
40.36
40.20
40.03
39.86
Current Data Parameters
NAME B-H-I2
EXPNO 203
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170417
Time 13.04
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 14596.5
DW 16.650 usec
DE 6.50 usec
TE 670.4 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 11.64 cm
F1P 217.505 ppm
F1 27352.90 Hz
F2P 2.465 ppm
F2 310.02 Hz
PPMCM 10.75198 ppm/cm
HZCM 1352.14441 Hz/cm
f1 (ppm)

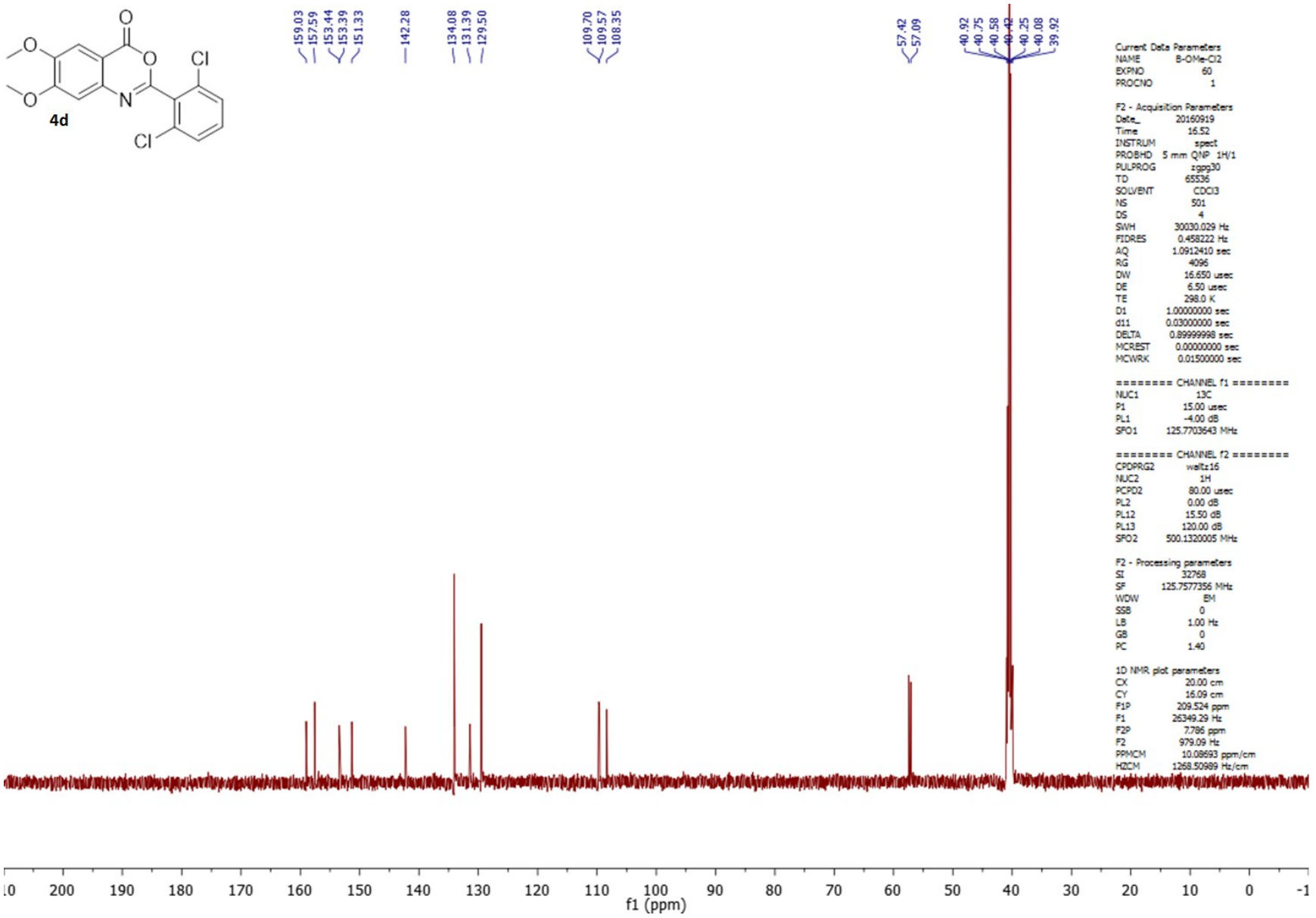

4c
Abundance
m/z
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
40.2
48.2
55.2
63.1
69.9
77.1
85.2
93.1
101.1
108.2
116.2
123.1
131.1
138.1
147.1
154.1
164.1
171.2
179.1
186.1
193.1
200.1
207.1
214.1
222.1
231
241.2
248
256.2
266
274
283
290.1
298.1
306.1
315.1
324.1
333.1
341
349
357.9
366
372.2
380
388.1
396
402.9
409.5
416.1
425
433
441
447.9
456
463.6
475
490.9
507
522
553.9
602.9

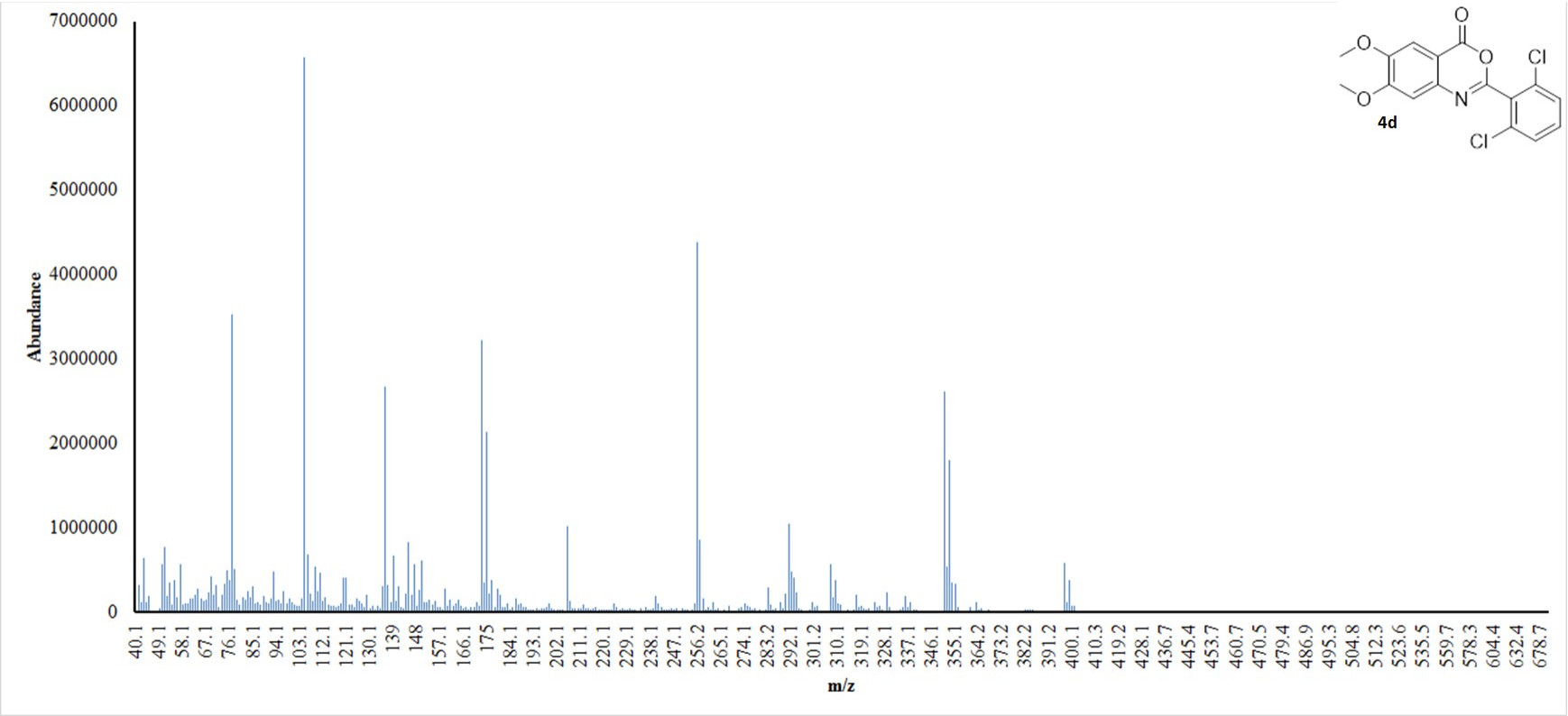

5a
11.69
8.41
7.77
7.76
7.67
7.67
7.66
7.66
7.65
7.56
7.54
7.53
7.51
7.49
7.47
7.47
7.46
7.45
3.88
3.79
3.42
2.51
0.66
0.83
1.00
1.46
3.09
2.51
2.81
f1 (ppm)
Current Data Parameters
NAME B-OMe-Br
EXPNO 39
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160904
Time 14.13
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 22.64 cm
F1P 9.410 ppm
F1 4706.37 Hz
F2P 6.828 ppm
F2 3415.05 Hz
PPMCM 0.12910 ppm/cm
HZCM 64.56612 Hz/cm

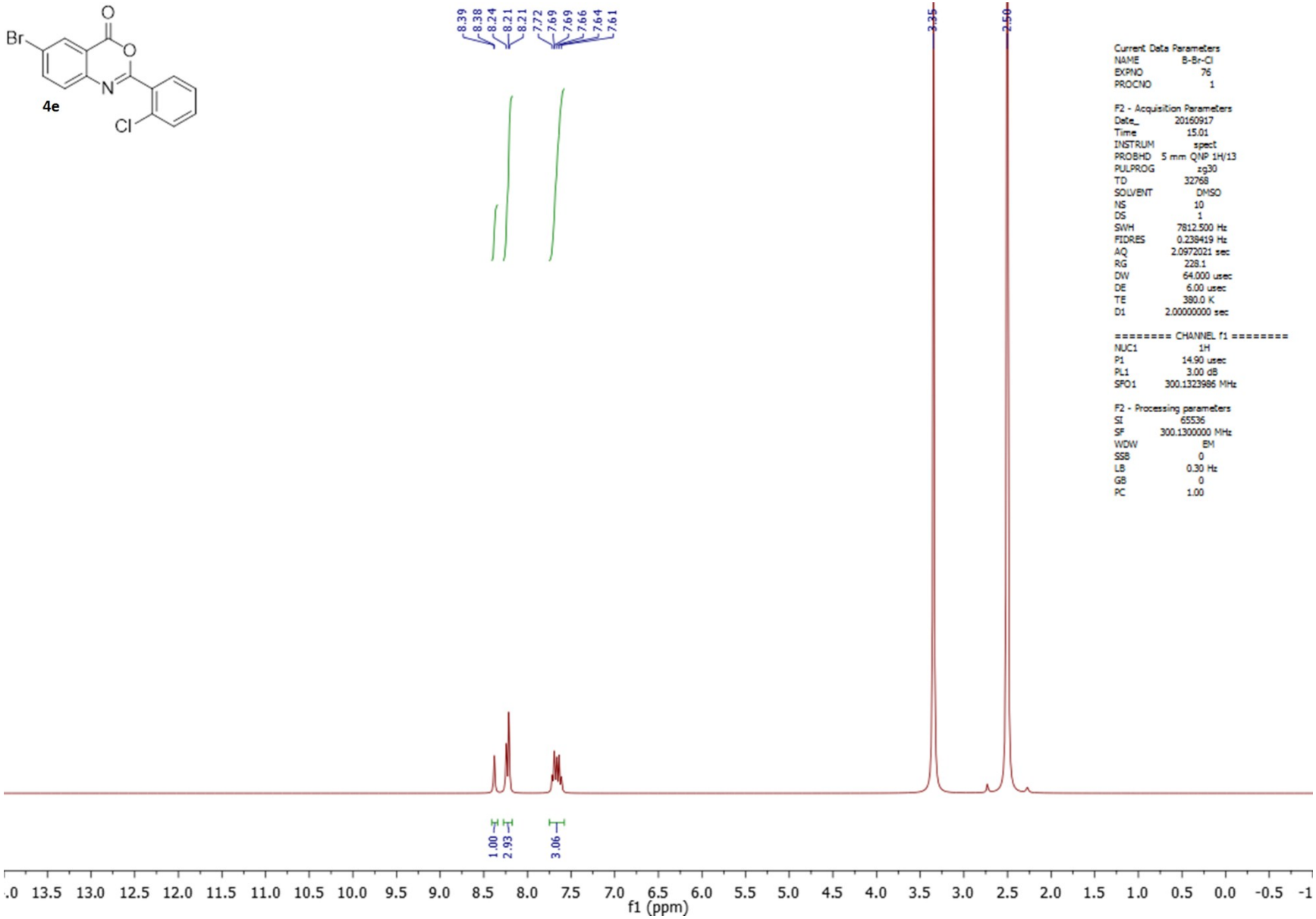

5a
170.22
166.20
154.14
144.93
139.25
137.16
134.26
132.86
129.80
129.04
119.59
113.79
108.90
104.02
56.57
56.54
40.84
40.67
40.51
40.34
40.18
40.01
39.84
Current Data Parameters
NAME B-OMe-Br
EXPNO 45
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160906
Time 12.53
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 48.05 cm
F1P 209.524 ppm
F1 26349.29 Hz
F2P 7.786 ppm
F2 979.09 Hz
PPMCM 10.08693 ppm/cm
HZCM 1268.50989 Hz/cm
f1 (ppm)

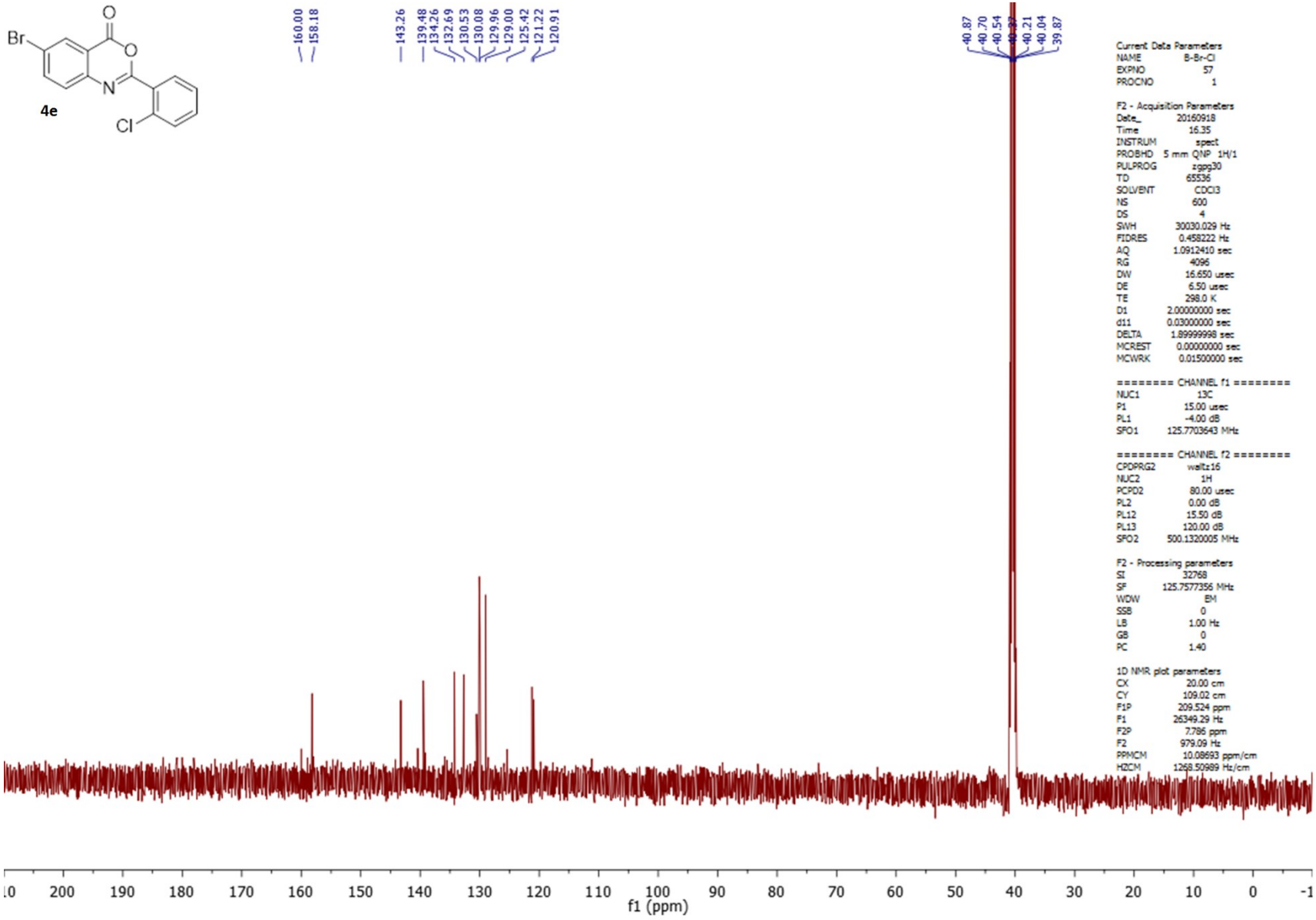

Abundance
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
40.2
48.1
57.2
65.1
73.1
81.1
89.2
97.1
105.1
113.1
121.1
129.1
137.1
145.2
153.2
164.1
172.2
181.2
190.1
198.1
206.1
214.1
222.1
230.1
238.1
246.1
254.1
262.3
270.2
278.2
286.2
294.2
303.1
311.3
319.1
327.2
335.1
343.2
351.3
360.1
368.4
376.2
385.3
393.2
401.2
408.3
421.3
428.1
444
454.2
467.7
479.7
495.5
507.5
524.5
547.7
560
579.6
m/z
O
OH
O
O
NH
Br
O
5a

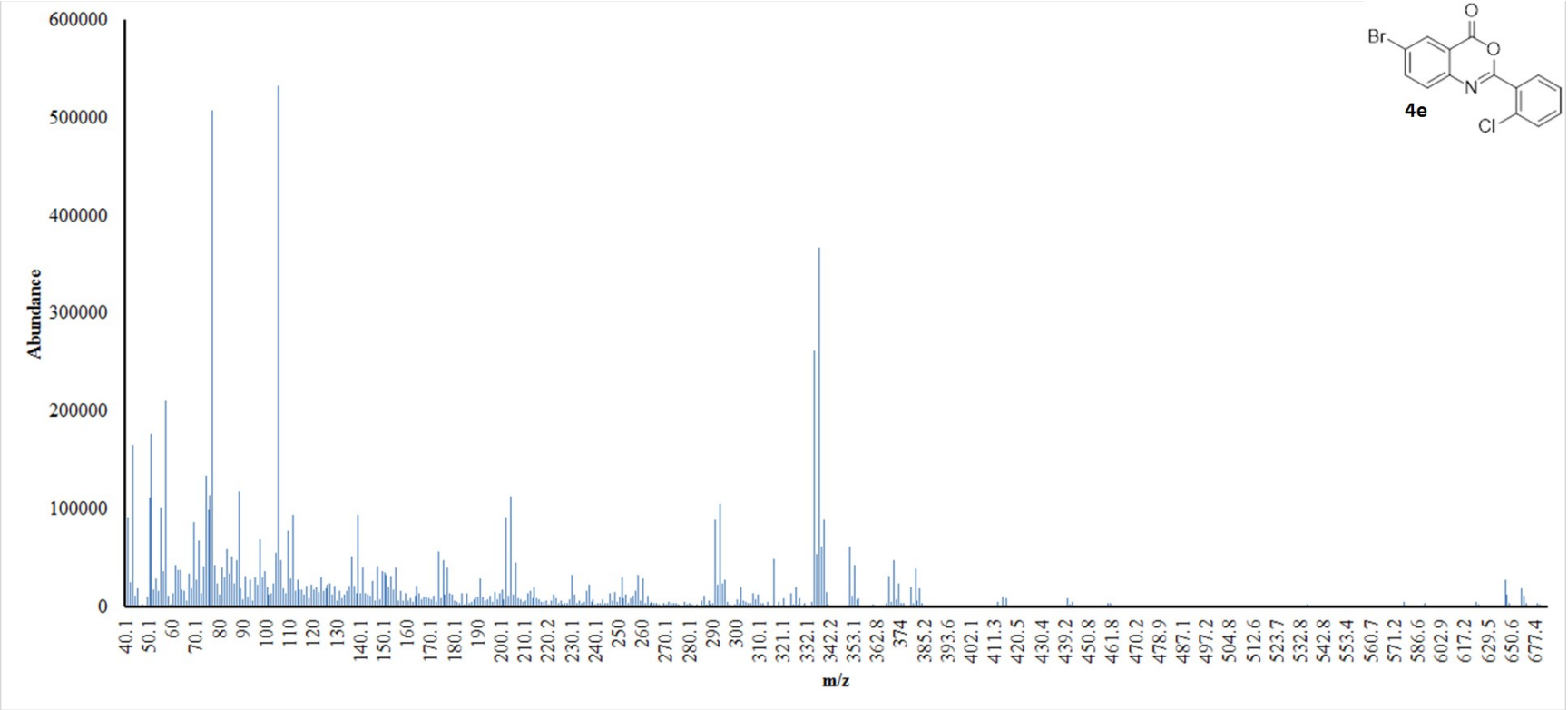

4d
7.73
7.72
7.70
7.68
7.66
7.65
7.63
7.55
7.35
3.97
3.95
3.34
2.50
Current Data Parameters
NAME B-OMe-Cl2
EXPNO 75
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160917
Time 14.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 12
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
--------- CHANNEL f1 ---------
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.15
1.02
1.00
5.99
f1 (ppm)

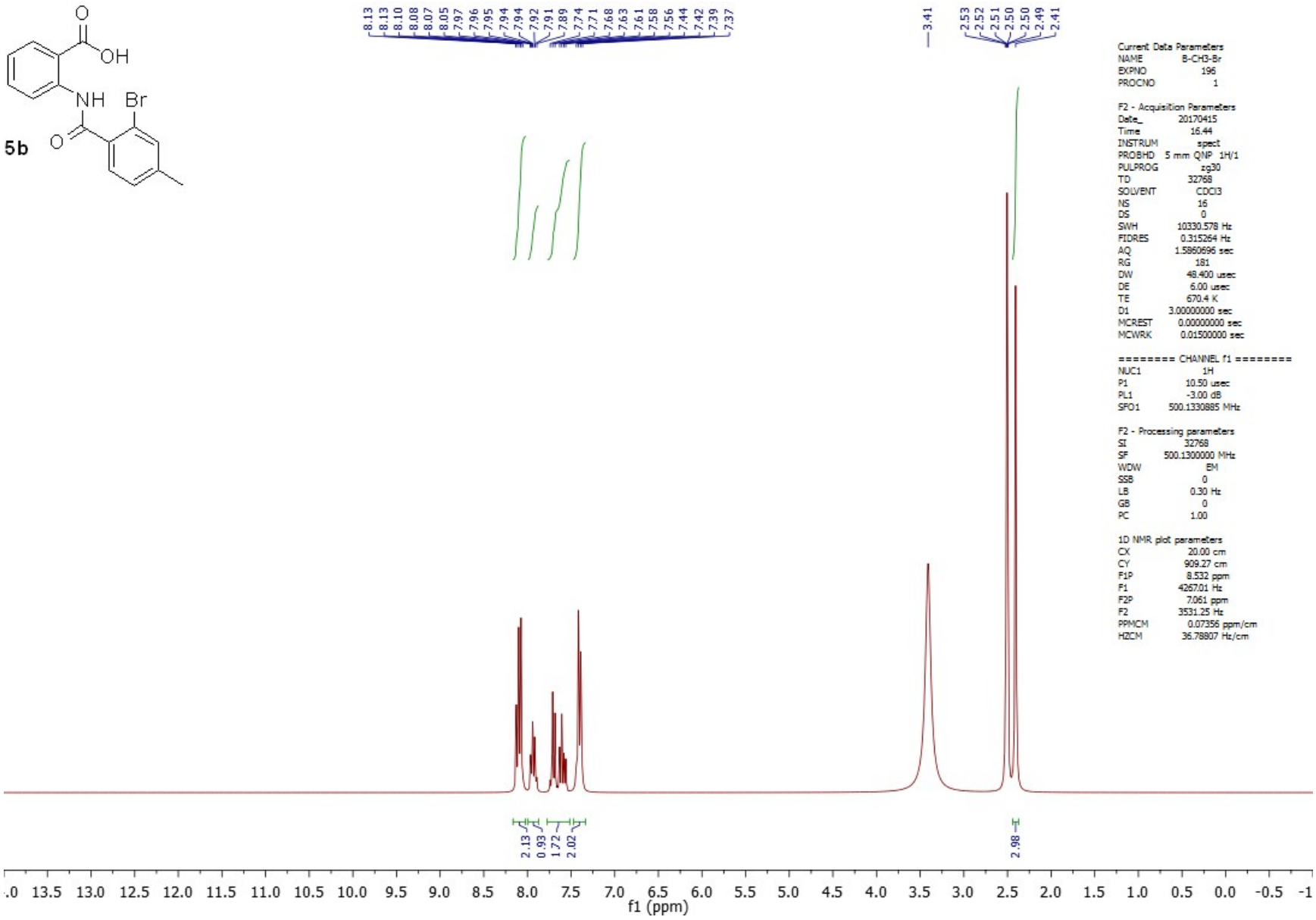

4d
159.03
157.59
153.44
153.39
151.33
142.28
134.08
131.39
129.50
109.70
109.57
108.35
57.42
57.09
40.92
40.75
40.58
40.42
40.25
40.08
39.92
Current Data Parameters
NAME B-OMe-Cl2
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160919
Time 16.52
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 501
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 16.09 cm
F1P 209.524 ppm
F1 26349.29 Hz
F2P 7.786 ppm
F2 979.09 Hz
PPMCM 10.08693 ppm/cm
HZCM 1268.50989 Hz/cm
f1 (ppm)

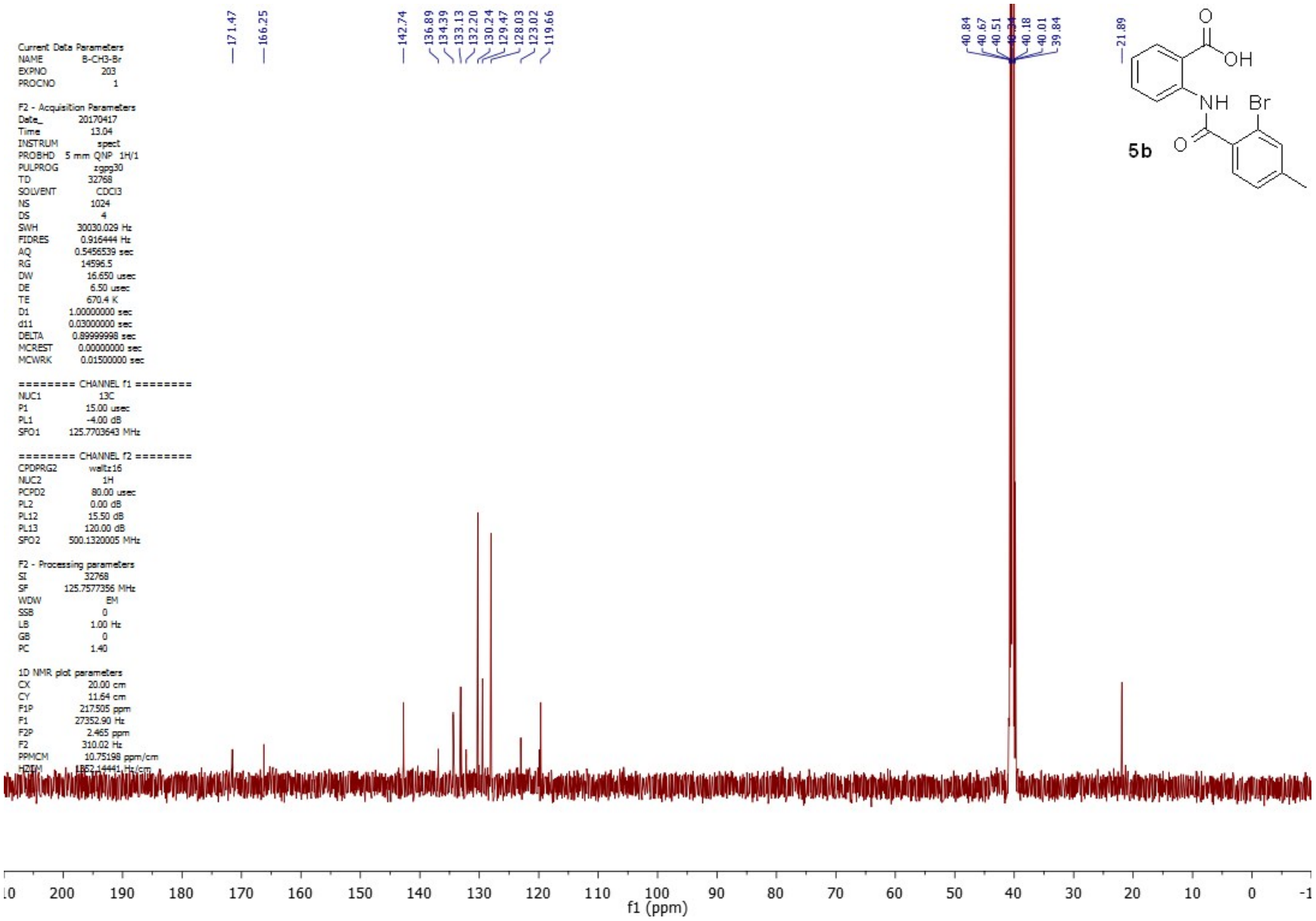

Abundance
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
40.1 49.1 58.1 67.1 76.1 85.1 94.1 103.1 112.1 121.1 130.1 139 148 157.1 166.1 175 184.1 193.1 202.1 211.1 220.1 229.1 238.1 247.1 256.2 265.1 274.1 283.2 292.1 301.2 310.1 319.1 328.1 337.1 346.1 355.1 364.2 373.2 382.2 391.2 400.1 410.3 419.2 428.1 436.7 445.4 453.7 460.7 470.5 479.4 486.9 495.3 504.8 512.3 523.6 535.5 559.7 578.3 604.4 632.4 678.7
m/z
O
O
O
O
N
Cl
Cl
4d

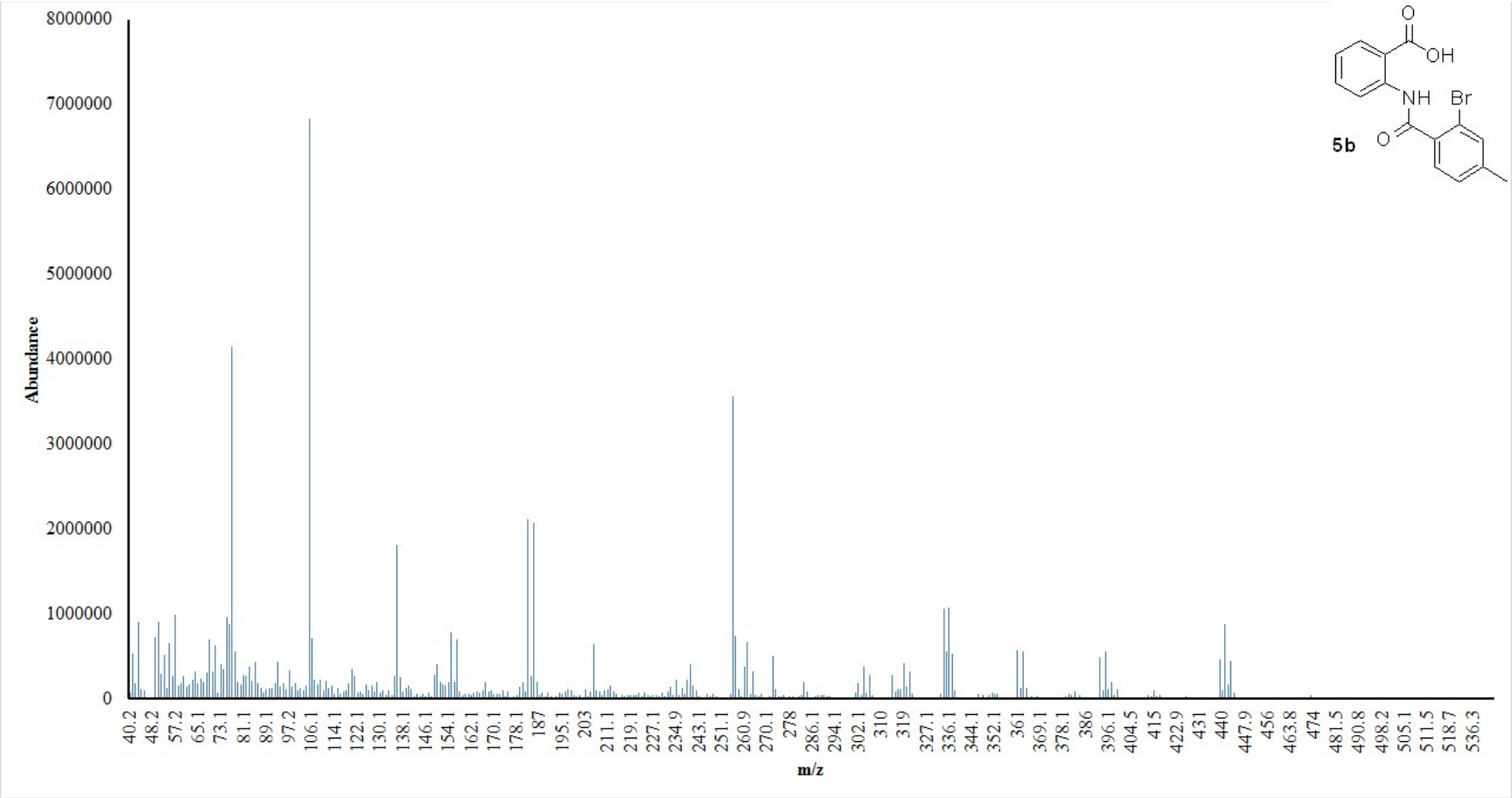

Br
O
O
N
Cl
4e
8.39
8.38
8.24
8.21
8.21
7.72
7.69
7.69
7.66
7.64
7.61
3.35
2.50
1.00
2.93
3.06
f1 (ppm)
Current Data Parameters
NAME B-Br-Cl
EXPNO 76
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160917
Time 15.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

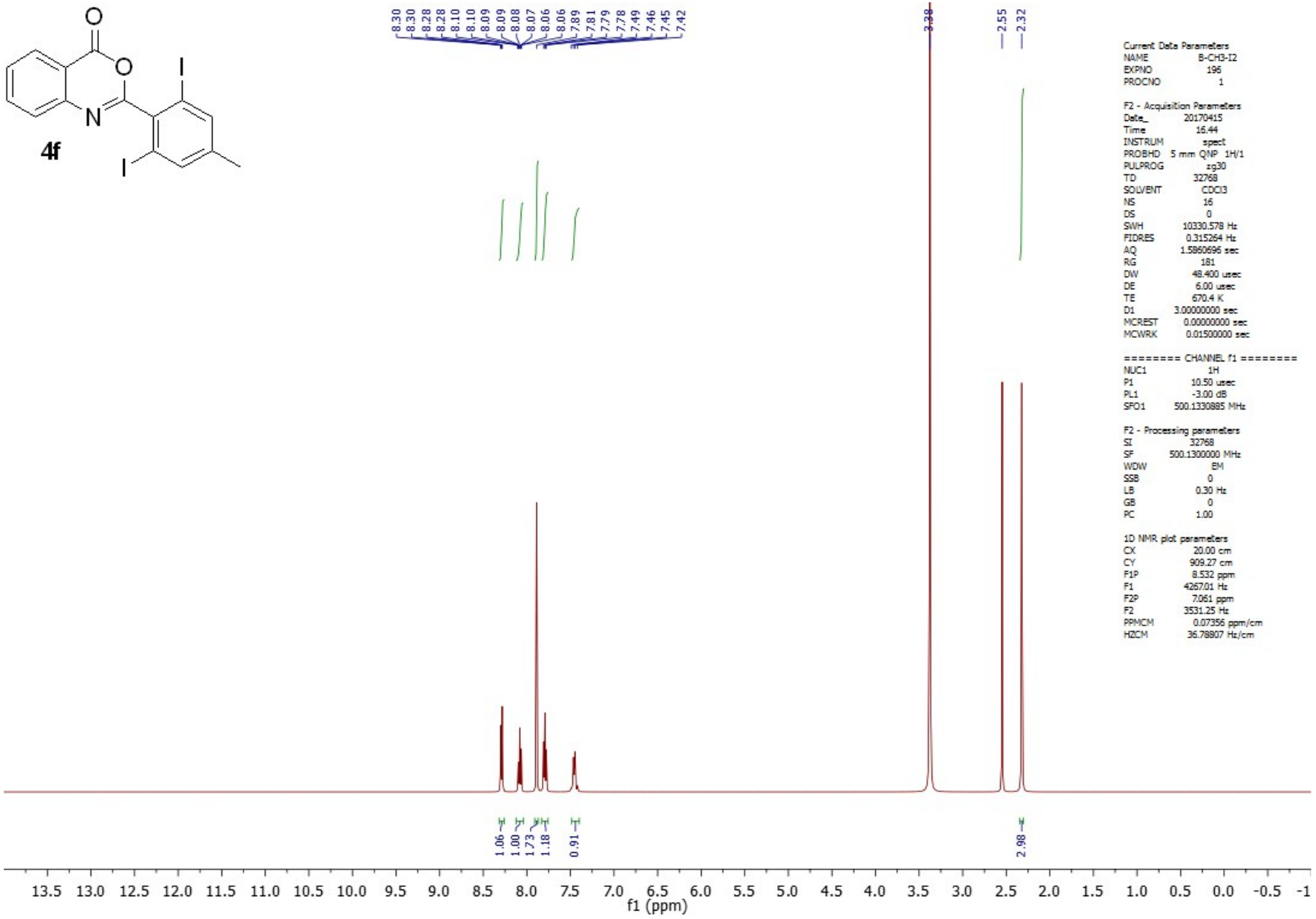

Br
O
O
N
Cl
4e
160.00
158.18
143.26
139.48
134.26
132.69
130.53
130.08
129.96
129.00
125.42
121.22
120.91
40.87
40.70
40.54
40.38
40.21
40.04
39.87
Current Data Parameters
NAME B-Br-Cl
EXPNO 57
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160918
Time 16.35
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 109.02 cm
F1P 209.524 ppm
F1 26349.29 Hz
F2P 7.786 ppm
F2 979.09 Hz
PPMCM 10.08693 ppm/cm
HZCM 1268.50989 Hz/cm
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

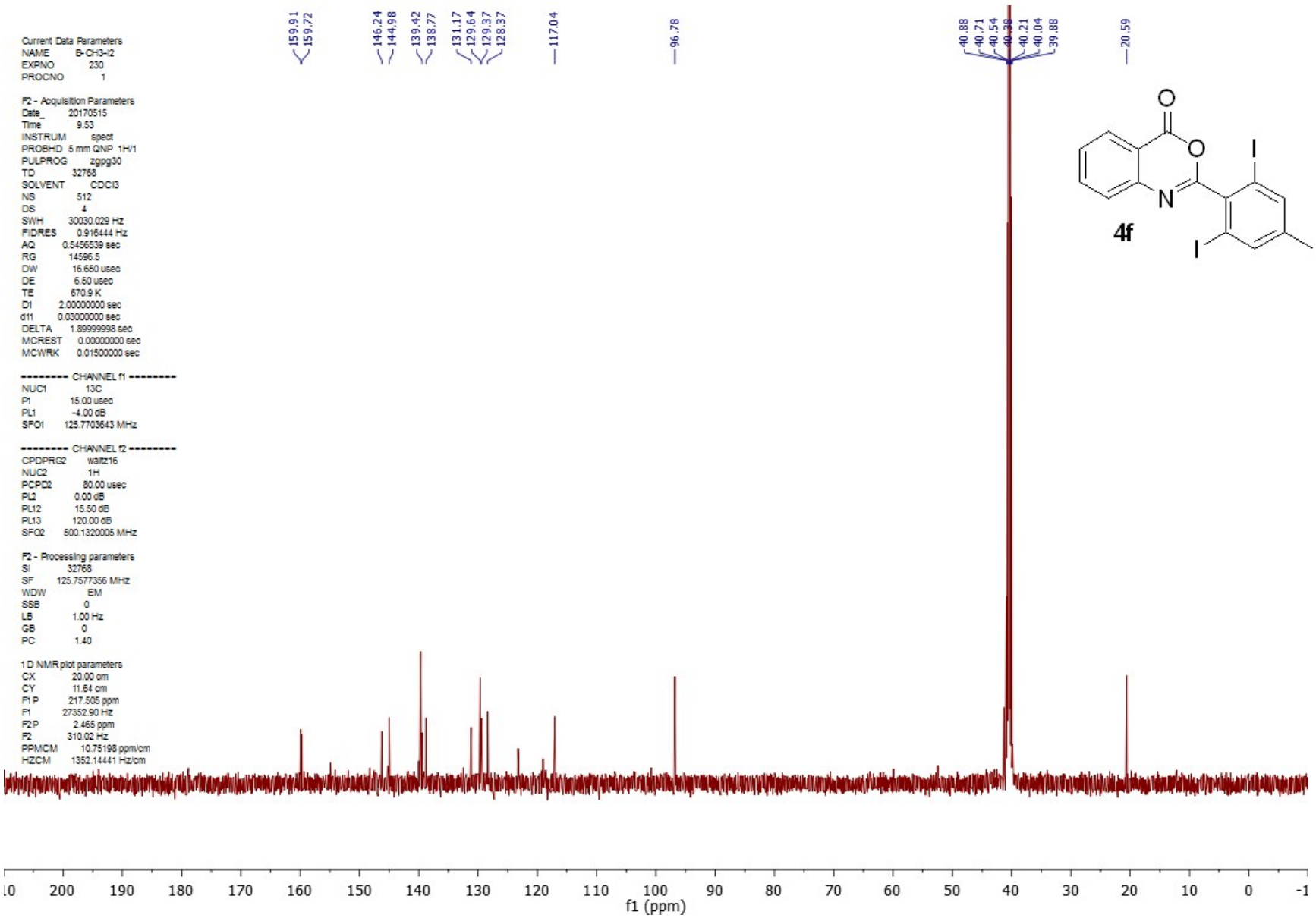

Br
O
O
N
Cl
4e
Abundance
600000
500000
400000
300000
200000
100000
0
m/z
40.1 50.1 60 70.1 80 90 100 110 120 130 140.1 150.1 160 170.1 180.1 190 200.1 210.1 220.2 230.1 240.1 250 260 270.1 280.1 290 300 310.1 321.1 332.1 342.2 353.1 362.8 374 385.2 393.6 402.1 411.3 420.5 430.4 439.2 450.8 461.8 470.2 478.9 487.1 497.2 504.8 512.6 523.7 532.8 542.8 553.4 560.7 571.2 586.6 602.9 617.2 629.5 650.6 677.4

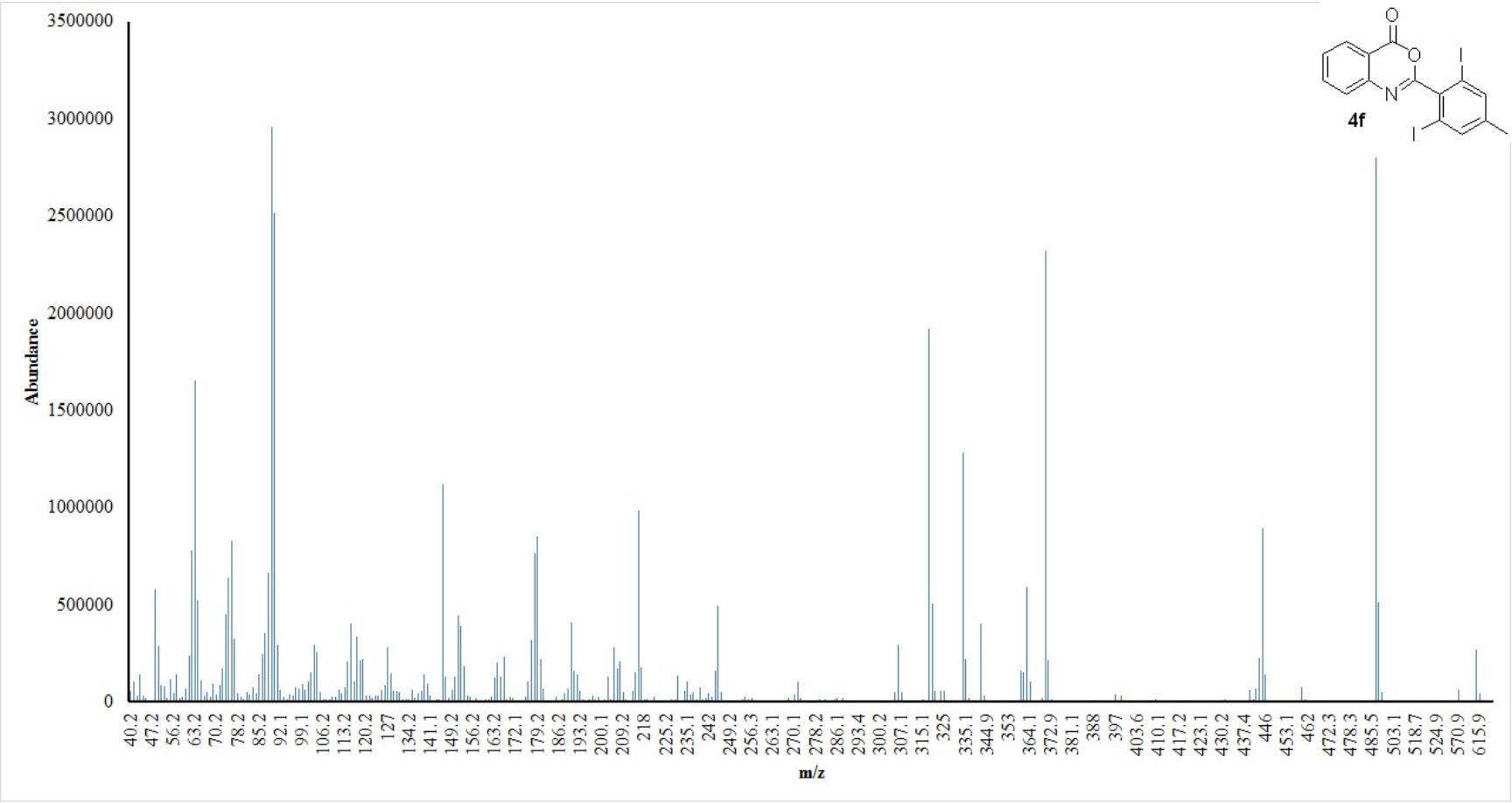

5b
O
OH
NH
Br
O
8.13
8.13
8.10
8.08
8.07
8.05
7.97
7.96
7.95
7.94
7.94
7.92
7.91
7.89
7.74
7.71
7.68
7.63
7.61
7.58
7.56
7.44
7.42
7.39
7.37
3.41
2.53
2.52
2.51
2.50
2.50
2.49
2.41
Current Data Parameters
NAME B-CH3-Br
EXPNO 196
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170415
Time 16.44
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 670.4 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 909.27 cm
F1P 8.532 ppm
F1 4267.01 Hz
F2P 7.061 ppm
F2 3531.25 Hz
PPMCM 0.07356 ppm/cm
HZCM 36.78807 Hz/cm
2.13
0.93
1.72
2.02
2.98
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

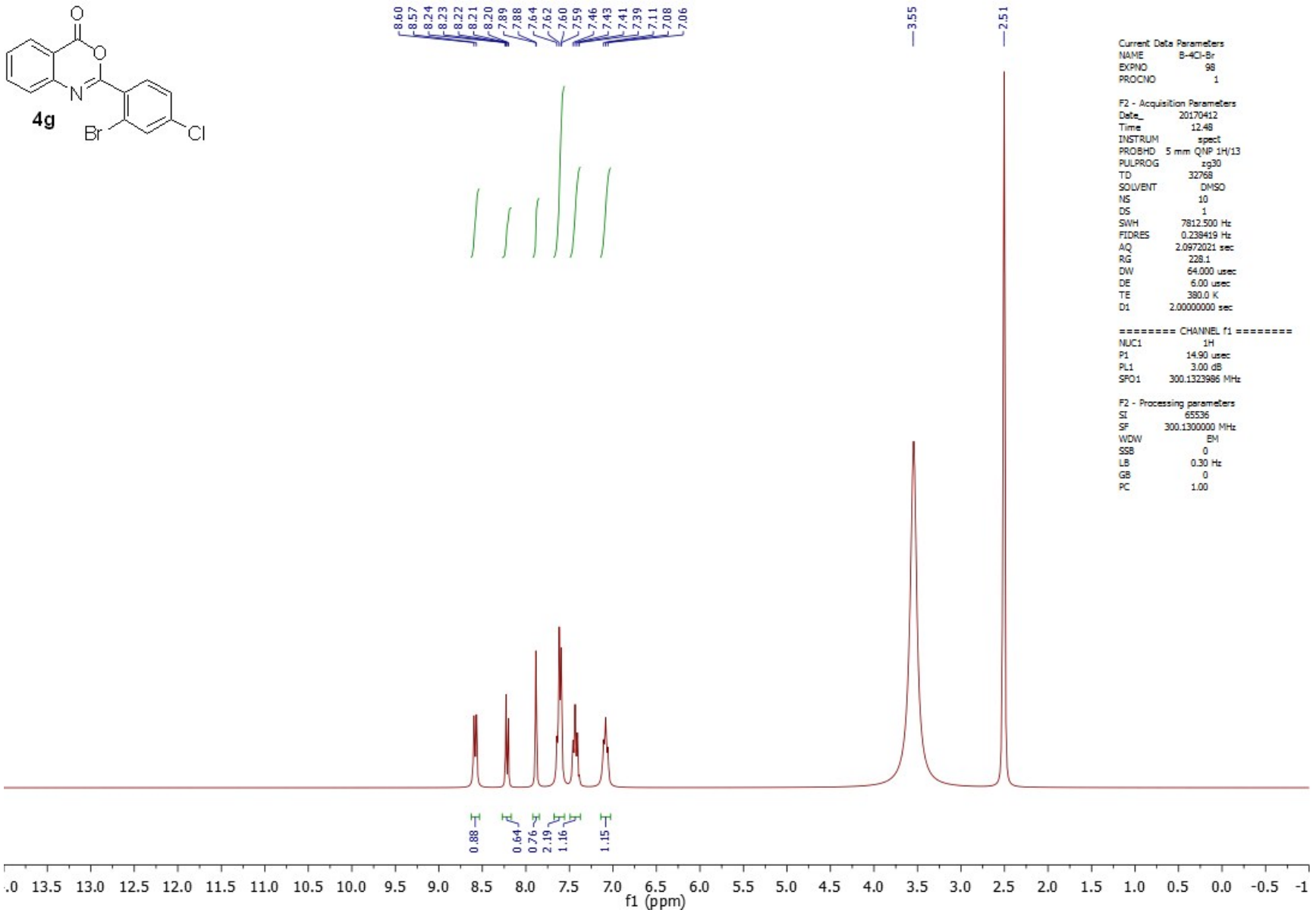

171.47
166.25
142.74
136.89
134.39
133.13
132.20
130.24
129.47
128.03
123.02
119.66
40.84
40.67
40.51
40.34
40.18
40.01
39.84
21.89
Current Data Parameters
NAME B-CH3-Br
EXPNO 203
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170417
Time 13.04
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 14596.5
DW 16.650 usec
DE 6.50 usec
TE 670.4 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 11.64 cm
F1P 217.505 ppm
F1 27352.90 Hz
F2P 2.465 ppm
F2 310.02 Hz
PPMCM 10.75198 ppm/cm
HZCM 1352.14441 Hz/cm
O
OH
NH
Br
O
5b
f1 (ppm)

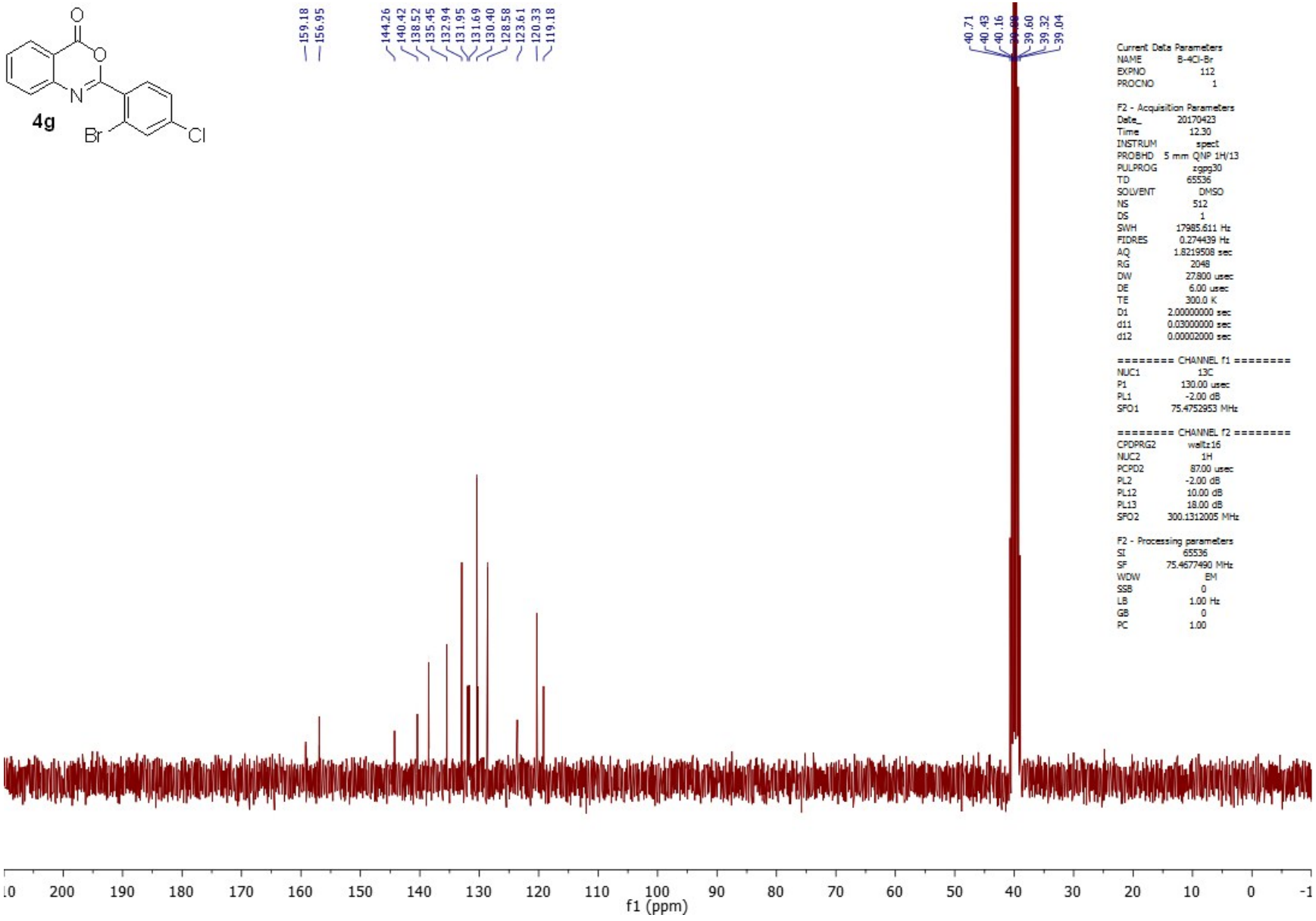

8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Abundance
40.2
48.2
57.2
65.1
73.1
81.1
89.1
97.2
106.1
114.1
122.1
130.1
138.1
146.1
154.1
162.1
170.1
178.1
187
195.1
203
211.1
219.1
227.1
234.9
243.1
251.1
260.9
270.1
278
286.1
294.1
302.1
310
319
327.1
336.1
344.1
352.1
361
369.1
378.1
386
396.1
404.5
415
422.9
431
440
447.9
456
463.8
474
481.5
490.8
498.2
505.1
511.5
518.7
536.3
m/z
O
OH
NH
Br
O
5b

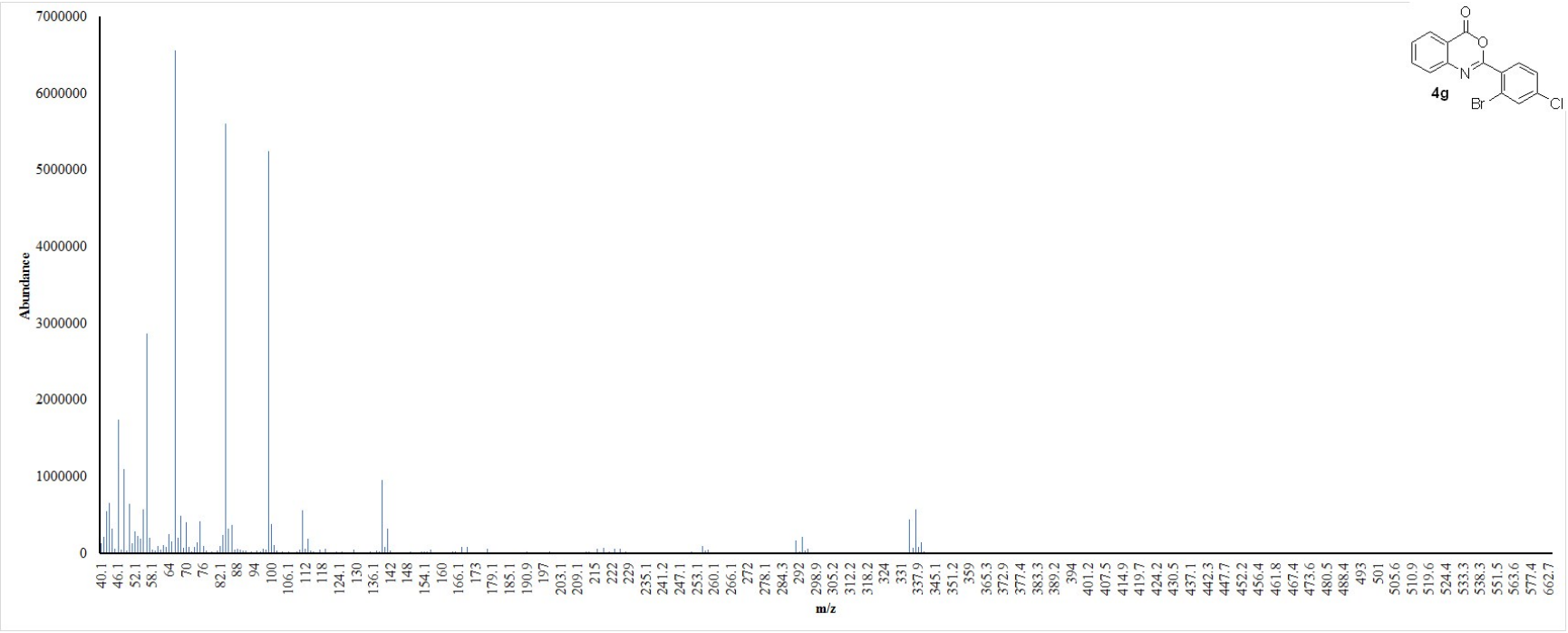

4f
8.30
8.30
8.28
8.28
8.10
8.10
8.09
8.09
8.08
8.07
8.06
8.06
7.89
7.81
7.79
7.78
7.49
7.46
7.45
7.42
3.38
2.55
2.32
Current Data Parameters
NAME B-CH3-I2
EXPNO 196
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170415
Time 16.44
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 670.4 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 909.27 cm
F1P 8.532 ppm
F1 4267.01 Hz
F2P 7.061 ppm
F2 3531.25 Hz
PPMCM 0.07356 ppm/cm
HZCM 36.78807 Hz/cm
1.06
1.00
1.73
1.18
0.91
2.98
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.5 -1
f1 (ppm)

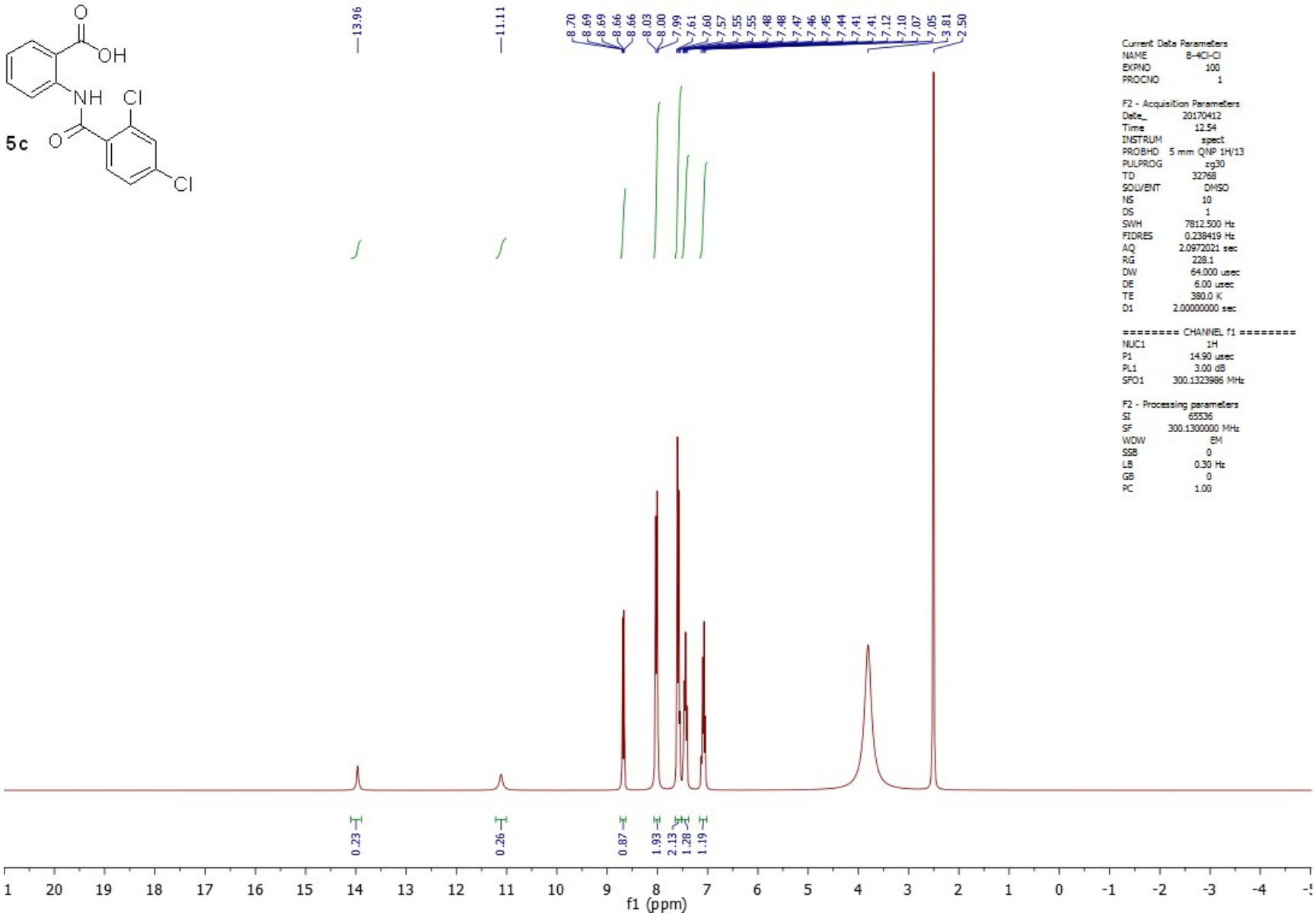

159.91
159.72
146.24
144.98
139.42
138.77
131.17
129.64
129.37
128.37
117.04
96.78
40.88
40.71
40.54
40.38
40.21
40.04
39.88
20.59
Current Data Parameters
NAME B-CH3-I2
EXPNO 230
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170515
Time 9.53
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 14596.5
DW 16.650 usec
DE 6.50 usec
TE 670.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
--------- CHANNEL f1 ---------
NUC1 13C
P1 15.00 usec
PL1 -4.00 dB
SFO1 125.7703643 MHz
--------- CHANNEL f2 ---------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 11.64 cm
F1P 217.505 ppm
F1 27352.90 Hz
F2P 2.465 ppm
F2 310.02 Hz
PPMCM 10.75198 ppm/cm
HZCM 1352.14441 Hz/cm
O
O
N
I
I
4f
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

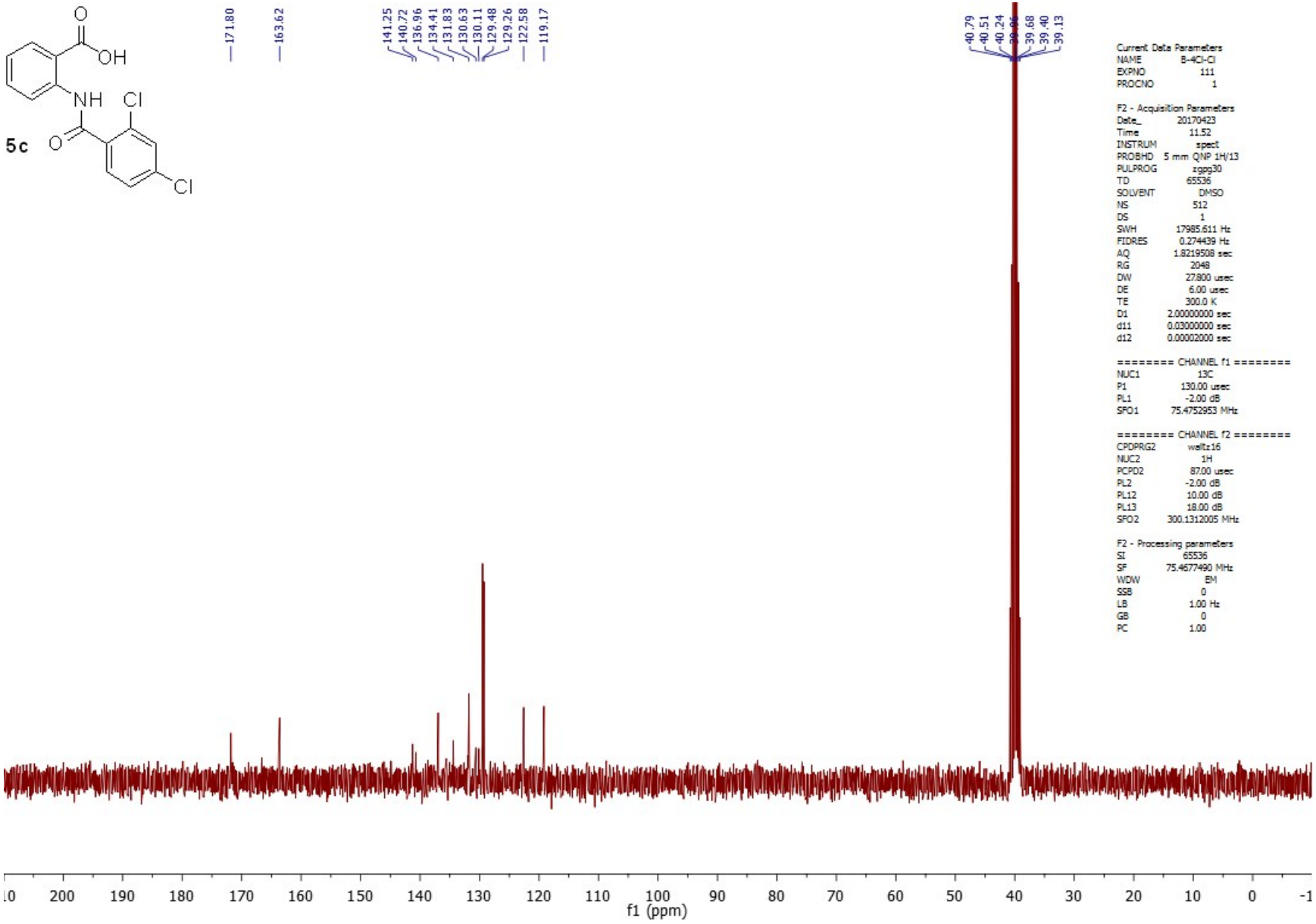

Abundance
m/z
4f

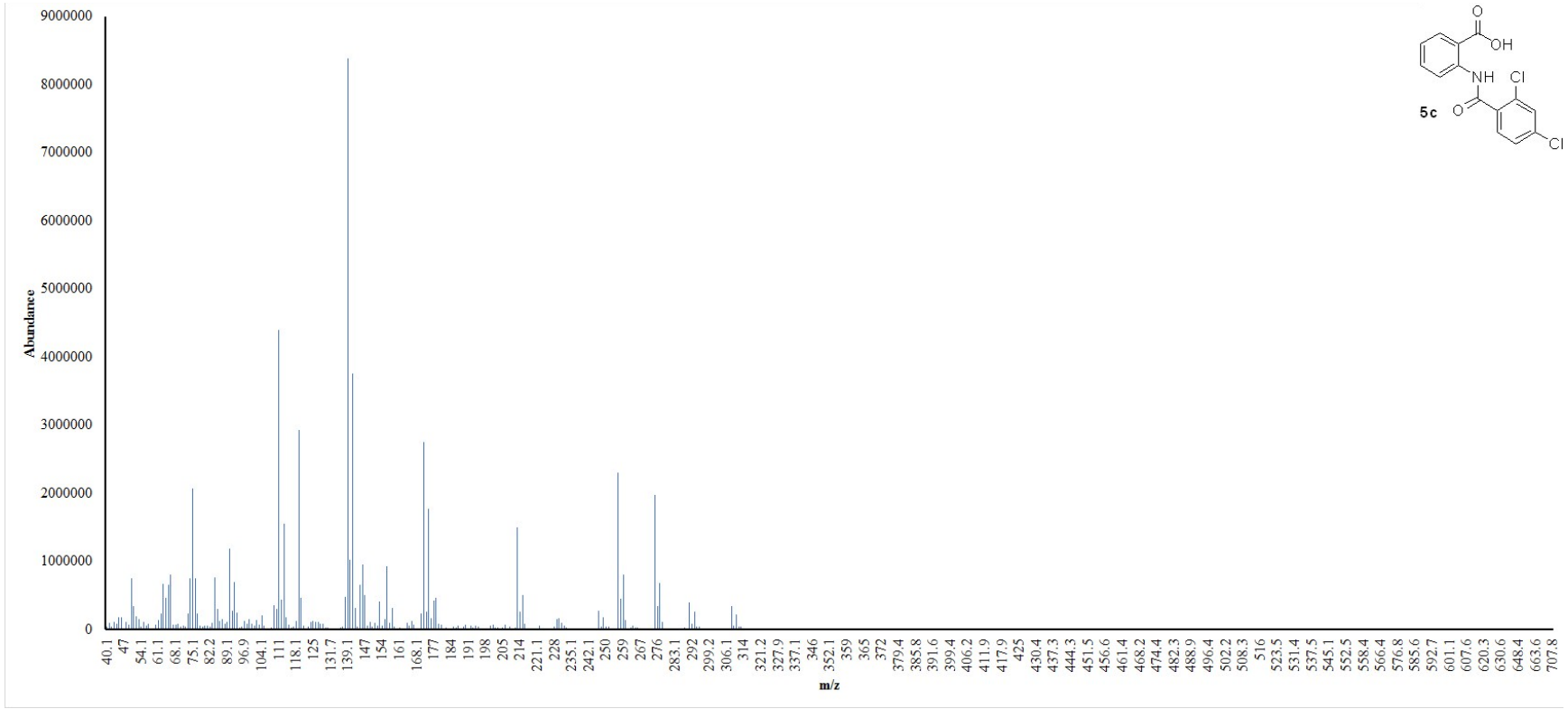

4g
Br
Cl
8.60
8.57
8.24
8.23
8.22
8.21
8.20
7.89
7.88
7.64
7.62
7.60
7.59
7.46
7.43
7.41
7.39
7.11
7.08
7.06
3.55
2.51
Current Data Parameters
NAME B-4Cl-Br
EXPNO 98
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170412
Time 12.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.88
0.64
0.76
2.19
1.16
1.15
f1 (ppm)


4g
Br
Cl
159.18
156.95
144.26
140.42
138.52
135.45
132.94
131.95
131.69
130.40
128.58
123.61
120.33
119.18
40.71
40.43
40.16
39.60
39.32
39.04
Current Data Parameters
NAME B-4Cl-Br
EXPNO 112
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170423
Time 12.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


Abundance
m/z
4g
Br
Cl


5c
O
OH
NH
Cl
O
Cl
13.96
11.11
8.70
8.69
8.69
8.66
8.66
8.03
8.00
7.99
7.61
7.60
7.57
7.55
7.55
7.48
7.48
7.47
7.46
7.45
7.44
7.41
7.41
7.12
7.10
7.07
7.05
3.81
2.50
Current Data Parameters
NAME B-4Cl-Cl
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170412
Time 12.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 3.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.23
0.26
0.87
1.93
2.13
1.28
1.19
1 20 19 18 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


5c
171.80
163.62
141.25
140.72
136.96
134.41
131.83
130.63
130.11
129.48
129.26
122.58
119.17
40.79
40.51
40.24
39.96
39.68
39.40
39.13
Current Data Parameters
NAME B-4Cl-Cl
EXPNO 111
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170423
Time 11.52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 1
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 130.00 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 10.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
f1 (ppm)


Abundance
m/z
5c

X-ray crystal structure analysis of 2d

Crystal	2d
Empirical formula	C15 H11 Br1 N2 O1
Color, habit	Colorless , Needle
Crystal system	Triclinic
Symmetry space group	*P-1*
Z	2
a (Å)	8.3615(14)
b(Å)	8.6790(16)
c(Å)	9.7121(16)
α (°)	87.261(14)
β (°)	86.850(13)
γ (°)	74.088(13)
Temperature (K)	298(2)
Volume ($Å^3$)	676.4(2)
D_{calcd} ($Mg\ m^{-3}$)	1.547
Radiation (Å)	Mo Kα 0.71073
Absorption coefficient (μ) (mm^{-1})	3.031
Absorption correction min	0.4521
Absorption correction max	0.8754
F (000)	316
θ range for data collection (°)	2.44-27.00
index ranges (h;k;l)	-10,10,-9,11,-12,12
R factor all	0.0851
R factor gt	0.0776
wR factor ref	0.1702
wR factor gt	0.1464
goodness-of-fit	0.978

X-ray crystal structure analysis of 4d

Crystal	4d
Empirical formula	C16 H11 Cl2 N1 O4
Color, habit	Colorless , Needle
Crystal system	Triclinic
Symmetry space group	*P-1*
Z	2
a (Å)	8.079(2)
b(Å)	8.088(3)
c(Å)	12.700(4)
α (°)	87.80(2)
β (°)	83.42(2)
γ (°)	71.65(2)
Temperature (K)	298(2)
Volume ($Å^3$)	782.5(4)
D_{calcd} (Mg m^{-3})	1.547
Radiation (Å)	Mo Kα 0.71073
Absorption coefficient (μ) (mm^{-1})	0.434
Absorption correction min	0.5215
Absorption correction max	0.8456
F (000)	360
θ range for data collection (°)	2.67-27.00
index ranges (h;k;l)	-10,10,-10,10,-16,16
R factor all	0.0895
R factor gt	0.0521
wR factor ref	0.1751
wR factor gt	0.1512
goodness-of-fit	1.100

Crystallographic data (excluding structure factors) for the structures reported in this work have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC: 1529594 (**2d**) and 1529593 (**4d**). Copy of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge DB2 1EZ,UK (fax:+ 44 (1223) 336033; e-mail: deposit@ccdc.cam.ac.uk).

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