

Supporting information

Metal-free synthesis of new azocines via the addition reaction of enaminones with acenaphthoquinone followed by oxidative-cleavage of the corresponding vicinal diols

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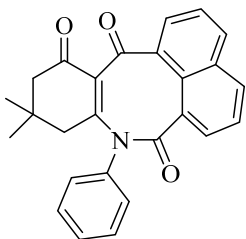
General information: The chemicals used in this work were purchased from Merck and Sigma-Aldrich chemical companies and were used without purification. The progress of the reaction and the purity of compounds were monitored by TLC analytical silica gel plates (Merck 60 F₂₅₀). Melting points were determined on an Electro thermal 9100 apparatus. IR spectra were recorded using a Shimadzu IR-470 spectrometer with KBr plates. ¹H-NMR and ¹³C-NMR spectra were recorded on a Bruker DRX-400 AVANCE spectrometer in DMSO-d₆ as solvent.

General procedure for the preparation of enaminones 1a-t

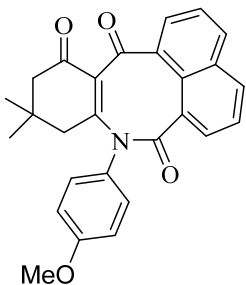
1,3-Diketone (5 mmol), amine (5 mmol), I₂ (0.1 mmol), and CH₃CN (5 mL) were added to a reaction tube. The tube was then sealed and stirred at room temperature for 1 h. In the most cases, enaminones **1** were precipitate from the reaction mixture as white crystals, which were collected on filter paper and further purified by washing with cool acetonitrile (2×2 mL). When the products were soluble in acetonitrile, the solvent was removed under reduced pressure, then water (10 mL) added and products extracted by ethyl acetate (3×3 mL). The organic layers were collected, washed with aqueous Na₂S₂O₃ solution and dried over anhydrous Na₂SO₄. After partial vaporization of solvent, the products were precipitated and the mixture was filtered to give the pure enaminones **1** as white solids.

General procedure for the synthesis of azocine derivatives 3a-t:

A mixture of enaminone **3** (1 mmol), Et₃N (1 mmol), and acenaphthoquinone **2** (1 mmol) in EtOH (4 mL) was placed in the flask and the mixture stirred 12 hours at reflux condition. The progress of the reaction was monitored by thin layer chromatography using EtOAc/*n*-hexane as eluent. After completion of the reaction, the reaction mixture was cooled to room temperature and H₅IO₆ (1 mmol) was added to the flask and the mixture stirred for additional 1 hour. The reaction mixture was filtered and the crude product was recrystallized from ethanol to afford the pure product **3**.

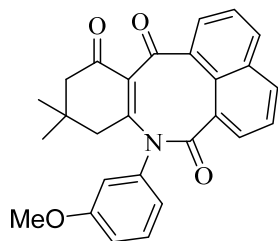


10,10-Dimethyl-8-phenyl-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine 7,12,13(8H,9H)-trione (**3a**): isolated yield = 95%; white powder; mp = 224 °C; IR cm^{-1} : 1306, 1358, 1666, 1681, 1692, 1712, 2957, 3304; ^1H NMR (400 MHz, DMSO-*d*₆) δ 0.60 (s, 3H, CH₃), 0.84 (s, 3H, CH₃), 2.03 (d, *J* = 16.5 Hz, 1H, H-CH), 2.10-2.25 (m, 2H, CH₂), 2.33 (d, *J* = 17.7 Hz, 1H, H-CH), 7.40–7.55 (m, 3H, arom), 7.60 (t, *J* = 7.8 Hz, 2H, arom), 7.66–7.82 (m, 3H, arom), 7.94 (d, *J* = 7.1 Hz, 1H, arom), 8.19 (t, *J* = 8.1 Hz, 2H, arom); ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 27.1, 27.4, 33.3, 50.3, 126.7, 127.2, 127.3, 127.6, 127.7, 128.7, 129.1, 130.1, 130.6, 132.4, 132.8, 134.7, 136.5, 137.0, 137.8, 151.6, 168.0, 196.1, 198.2. HRMS (ESI) calcd for C₂₆H₂₁NO₃ [M+H]⁺: 396.1600; Found: 396.1595.

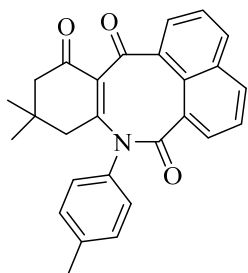


8-(4-Methoxyphenyl)-10,10-dimethyl-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8H,9H)-trione (**3b**): isolated yield = 90%; white powder; mp >230 °C; IR cm^{-1} : 767, 810, 1356, 1639, 1671, 1693, 2923; ^1H NMR (400 MHz, DMSO-*d*₆) δ 0.62 (s, 3H, CH₃), 0.84 (s, 3H, CH₃), 2.02 (d, *J* = 16.5 Hz, 1H, H-CH), 2.12-2.24 (m, 2H, CH₂), 2.33 (d, *J* = 17.6 Hz, 1H, H-CH), 3.83 (s, 3H, OCH₃), 7.14 (d, *J* = 8.5 Hz, 2H, arom), 7.41 (d, *J* = 8.5 Hz, 2H, arom), 7.57–7.80 (m, 3H, arom), 7.91 (d, *J* = 6.9 Hz, 1H, arom), 8.18 (t, *J* = 8.6 Hz, 2H, arom); ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 27.0, 27.4, 33.3, 50.3, 55.9, 115.3, 126.6, 127.2, 127.3, 127.6, 128.7, 128.9, 130.2, 130.6, 132.4, 132.8, 134.8,

136.0, 137.0, 151.8, 159.6, 168.1, 196.1, 198.4. HRMS (ESI) calcd for C₂₇H₂₃NO₄ [M+H]⁺: 426.1705; found: 426.1700.

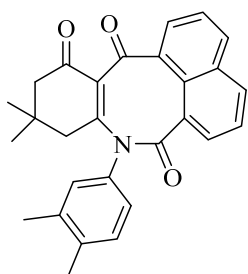


8-(3-Methoxyphenyl)-10,10-dimethyl-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3c**): isolated yield = 90%; white powder; mp >230 °C; IR cm⁻¹: 766, 826, 1638, 1654, 1685, 2967, 3296; ¹H NMR (400 MHz, DMSO-*d*₆) δ 0.61 (s, 3H, CH₃), 0.85 (s, 3H, CH₃), 2.03 (d, *J* = 16.5 Hz, 1H, H-CH), 2.15-2.26 (m, 2H, CH₂), 2.35 (d, *J* = 17.6 Hz, 1H, CH), 3.84 (s, 3H, OCH₃), 7.00-7.14 (m, 3H, arom), 7.50 (t, *J* = 7.9 Hz, 1H, arom), 7.67-7.80 (m, 3H, arom), 7.96 (d, *J* = 6.9 Hz, 1 H, arom), 8.18 (t, *J* = 8.4 Hz, 2H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 26.9, 27.5, 33.3, 50.3, 55.9, 113.7, 114.5, 119.7, 126.6, 127.2, 127.3, 127.7, 128.7, 130.6, 130.9, 132.4, 132.8, 134.7, 136.4, 137.0, 138.8, 151.6, 160.5, 167.9, 196.1, 198.2. HRMS (ESI) calcd for C₂₇H₂₃NO₄ [M+H]⁺: 426.1705; found: 426.1699.

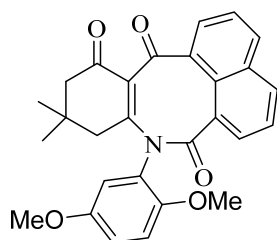


10,10-Dimethyl-8-(*p*-tolyl)-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3d**): isolated yield = 95%; white powder; mp = 225-227 °C; IR cm⁻¹: 765, 808, 1302, 1357, 1653, 2341,

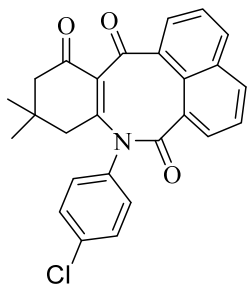
2360, 3030; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 0.61 (s, 3H, CH_3), 0.84 (s, 3H, CH_3), 2.02 (d, $J = 16.5$ Hz, 1H, H-CH), 2.11-2.24 (m, 2H, CH_2), 2.32 (d, $J = 17.7$ Hz, 1H, H-CH), 2.38 (s, 3H, CH_3), 7.33-7.42 (m, 4H, arom), 7.66-7.77 (m, 3H, arom), 7.92 (d, $J = 7.0$ Hz, 1H, arom), 8.18 (t, $J = 8.4$ Hz, 2H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 21.2, 27.0, 27.4, 33.3, 50.3, 126.6, 127.2, 127.3, 127.4, 127.7, 128.6, 130.5, 130.6, 132.4, 132.8, 134.8, 135.2, 136.2, 137.0, 138.7, 151.7, 168.0, 196.0, 198.3. HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{23}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 410.1756; found: 410.1751.



8-(3,4-Dimethylphenyl)-10,10-dimethyl-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3e**): isolated yield = 95%; white powder; mp = 227-229 °C; IR cm^{-1} : 773, 833, 1302, 1356, 1627, 1660, 1677, 2955; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 0.60 (s, 3H, CH_3), 0.84 (s, 3H, CH_3), 2.02 (d, $J = 16.5$ Hz, 1H, H-CH), 2.12-2.24 (m, 2H, CH_2), 2.29 (s, 3H, CH_3), 2.31 (s, 3H, CH_3), 2-27-2.32 (d overlapped with CH_3 singnals, 1H, H-CH), 7.18 (dd, $J_1 = 7.9$ Hz, $J_2 = 1.6$ Hz, 1H, Aarom), 7.28 (s, 1H, arom), 7.34 (d, $J = 8.0$ Hz, 1H, arom) 7.66-7.76 (m, 3H, arom), 7.92 (d, $J = 6.8$ Hz, 1H, arom), 8.18 (t, $J = 8.5$ Hz, 2H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 19.6, 19.9, 27.0, 27.4, 33.3, 50.3, 124.7, 126.6, 127.2, 127.4, 127.6, 128.4, 128.5, 130.5, 130.9, 132.4, 132.8, 134.9, 135.4, 136.1, 137.1, 137.4, 138.2, 151.7, 168.0, 196.1, 198.3. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{25}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 424.1913; found: 424.1904.

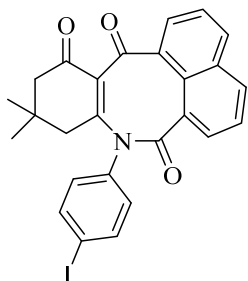


8-(2,5-Dimethoxyphenyl)-10,10-dimethyl-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3f**): isolated yield = 87%; white powder; mp >230 °C; IR cm^{-1} : 777, 1042, 1277, 1306, 1359, 1508, 1637, 1665, 1689, 2957; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 0.64 (s, 3H, CH_3), 0.85 (s, 3H, CH_3), 2.04 (d, $J = 16.5$ Hz, 1H, H-CH), 2.11-2.19 (m, 2H, CH_2), 2.23 (d, $J = 17.6$ Hz, 1H, H-CH), 3.78 (s, 3H, OCH_3), 3.92 (s, 3H, OCH_3), 6.8 (broad peak, 1H, arom), 7.08 (dd, $J_1 = 9.0$ Hz, $J_2 = 3.1$ Hz, 1H, arom), 7.22 (d, $J = 9.0$ Hz, 1H, arom) 7.68-7.79 (m, 4H, arom), 8.11-8.25 (m, 2H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 26.8, 27.6, 33.2, 50.3, 56.2, 57.0, 114.3, 115.2, 119.6, 126.7, 126.9, 127.3, 127.4, 127.5, 128.0, 130.4, 132.3, 132.5, 132.9, 135.0, 135.9, 137.3, 149.7, 151.8, 153.8, 167.5, 195.9. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{25}\text{NO}_5$ $[\text{M}+\text{H}]^+$: 456.1811; found: 456.1801.



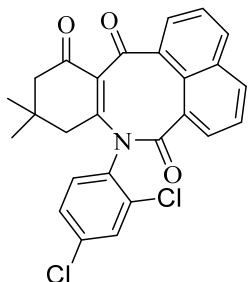
8-(4-Chlorophenyl)-10,10-dimethyl-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3g**): isolated yield = 92%; white powder; mp = 225-227 °C; IR cm^{-1} : 808, 1303, 1670, 2342, 2360, 2954; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 0.60 (s, 3H, CH_3), 0.85 (s, 3H, CH_3), 2.03 (d, $J = 16.4$ Hz, 1H, H-CH), 2.17-2.25 (m, 2H, CH_2), 2.34 (d, $J = 17.7$ Hz, 1H, H-CH), 7.53 (d, $J = 8.5$ Hz, 2H, arom), 7.67-7.73 (m, 3H, arom), 7.76 (d, $J = 7.3$ Hz, 2H, arom), 7.96 (d, $J = 7.3$ Hz, 1H, arom), 8.19 (t, $J = 7.7$ Hz, 2H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 27.1, 27.3, 33.3, 50.2, 126.7, 127.2,

127.3, 127.8, 128.8, 129.4, 130.2, 130.8, 132.4, 132.9, 134.4, 136.6, 136.7, 136.8, 151.2, 168.0, 196.1, 198.1. HRMS (ESI) calcd for C₂₆H₂₀ClNO₃ [M+H]⁺: 430.1210; found: 430.1202.



8-(4-Iodophenyl)-10,10-dimethyl-10,11-dihydro-7H-benzo[b]naphtho[1,8-ef]azocine-

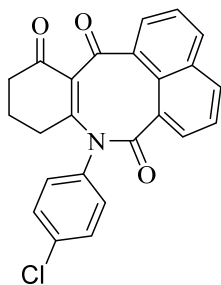
7,12,13(8H,9H)-trione (**3h**): isolated yield = 94%; white powder; mp >230 °C; IR cm⁻¹: 764, 1646, 2341, 2359, 2955; ¹H NMR (400 MHz, DMSO-*d*₆) δ 0.60 (s, 3H, CH₃), 0.86 (s, 3H, CH₃), 2.03 (d, *J* = 16.3 Hz, 1H, H-CH), 2.16-2.26 (m, 2H, CH₂), 2.32 (d, *J* = 17.5 Hz, 1H, H-CH), 7.31 (d, *J* = 8.2 Hz, 2H, arom), 7.68-7.73 (m, 1H, arom), 7.76 (d, *J* = 7.2 Hz, 2H, arom), 7.94-7.98 (m, 3H, arom), 8.19 (t, *J* = 7.9 Hz, 2H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 27.1, 27.3, 33.3, 50.3, 95.1, 126.7, 127.2, 127.3, 127.8, 128.8, 129.7, 130.7, 132.4, 132.9, 134.5, 136.8, 136.9, 137.5, 138.9, 151.1, 167.9, 196.0, 198.1. HRMS (ESI) calcd for C₂₆H₂₀NO₃I [M+H]⁺: 522.0566; found: 522.0554.



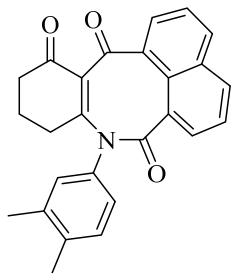
8-(2,4-Dichlorophenyl)-10,10-dimethyl-10,11-dihydro-7H-benzo[b]naphtho[1,8-ef]azocine-

7,12,13(8H,9H)-trione (**3i**): isolated yield = 92%; white powder; mp >230 °C; IR cm⁻¹: 769, 817, 1306, 1360, 1634, 1671, 2360, 2951; ¹H NMR (400 MHz, DMSO-*d*₆) δ 0.69 (s, 3H, CH₃), 0.82 (s, 3H, CH₃), 2.08-2.15 (m, 3H, CH₂+H-CH), 2.36 (d, *J* = 17.7 Hz, 1H, H-CH), 7.39 (d, *J* = 8.4 Hz, 1H, arom), 7.70-

7.78 (m, 4H, arom) 7.85 (d, $J = 6.7$ Hz, 1H, arom), 8.00 (s, 1H, arom), 8.21 (d, $J = 7.8$ Hz, 2H, arom); ^{13}C NMR (100 MHz, DMSO- d_6) δ 26.3, 27.8, 30.1, 50.2, 126.8, 127.3, 127.4, 127.6, 128.4, 129.7, 130.2, 130.8, 131.0, 132.4, 133.9, 134.1, 134.2, 135.1, 136.1, 136.8, 150.7, 167.1, 196.0, 198.6. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{19}\text{NO}_3\text{Cl}_2$ $[\text{M}+\text{H}]^+$: 464.0820; found: 464.0808.

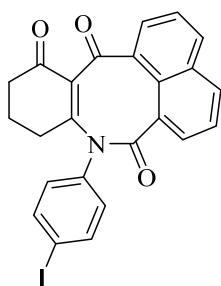


8-(4-Chlorophenyl)-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3j**): isolated yield = 92%; white powder; mp = 229 °C; IR cm^{-1} : 777, 1291, 1361, 1637, 1647, 1685, 1707, 1718, 2345, 2917; ^1H NMR (400 MHz, DMSO- d_6) δ 1.68-1.82 (m, 2H, CH_2), 1.96-2.19 (m, 2H, CH_2), 2.20-2.32 (m, 2H, CH_2), 7.51-7.59 (m, 2H, arom), 7.64-7.80 (m, 5H, arom), 7.98 (dd, $J_1 = 7.8$ Hz, $J_2 = 0.8$ Hz, 1H, arom), 8.14-8.24 (m, 2H, arom); ^{13}C NMR (100 MHz, DMSO- d_6) δ 20.9, 27.0, 36.9, 126.6, 127.2, 127.3, 127.8, 128.8, 129.3, 130.2, 130.7, 132.4, 132.8, 133.4, 134.3, 136.9, 137.0, 137.7, 153.3, 168.0, 196.0, 198.0. HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{18}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 402.0897; found: 402.0891.

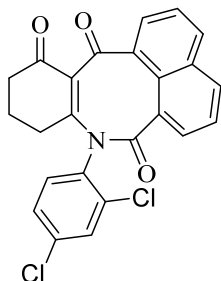


8-(3,4-Dimethylphenyl)-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3k**): isolated yield = 95%; white powder; mp = 225-227 °C; IR cm^{-1} : 779, 1295, 1361, 1637, 1653, 1693, 2341, 2360, 2962; ^1H NMR (400 MHz, DMSO- d_6) δ 1.64-1.81 (m, 2H, CH_2), 2.03-2.15 (m, 1H,

CH), 2.15-2.28 (m, 2H, CH₂), 2.29 (s, 3H, CH₃), 2.31 (s, 3H, CH₃), 2.38-2.45 (m, 1H, CH), 7.20 (dd, $J_1 = 7.5$ Hz, $J_2 = 2.2$ Hz, 1H, arom), 7.29 (s, 1H, arom), 7.3 (d, $J = 7.8$ Hz, 1H, arom), 7.64-7.80 (m, 3H, arom), 7.92 (d, $J = 6.7$ Hz, 1H, arom), 8.18 (t, $J = 7.6$ Hz, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 19.6, 19.9, 20.9, 27.1, 37.0, 124.6, 126.6, 127.2, 127.3, 127.7, 128.2, 128.5, 130.5, 131.0, 132.4, 132.7, 134.8, 135.8, 137.0, 137.1, 137.4, 138.3, 153.9, 168.1, 196.0, 198.5. HRMS (ESI) calcd for C₂₆H₂₁NO₃ [M+H]⁺: 396.1600; found: 396.1592.

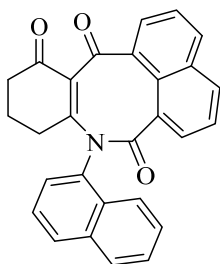


8-(4-Iodophenyl)-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (31): isolated yield = 90%; white powder; mp >230 °C; IR cm⁻¹: 797, 1004, 1651, 2341, 2359, 2871, 3052; ¹H NMR (400 MHz, DMSO-*d*₆) δ 1.66-1.85 (m, 2H, CH₂), 2.04-2.16 (m, 1H, H-CH), 2.18-2.33 (m, 2H, CH₂), 2.39-2.47 (m, 1H, H-CH), 7.33 (d, $J = 8.2$ Hz, 2H, arom), 7.60-7.80 (m, 3H, arom), 7.90-7.80 (m, 3H, arom), 7.86-8.01 (m, 3H, arom), 8.19 (t, $J = 6.8$ Hz, 2H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 20.8, 27.0, 36.9, 95.0, 126.6, 127.2, 127.3, 127.8, 128.7, 129.6, 130.7, 132.4, 132.8, 134.3, 136.9, 137.7, 137.9, 139.0, 153.2, 167.9, 196.0, 198.3. HRMS (ESI) calcd for C₂₄H₁₆NO₃I [M+H]⁺: 494.253; found: 494.0242.



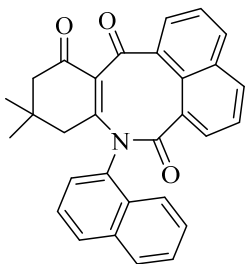
8-(2,4-Dichlorophenyl)-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione

(**3m**): isolated yield = 90%; white powder; mp >230 °C; IR cm⁻¹: 769, 811, 1127, 1644, 2349, 2359, 2889, 3089; ¹H NMR (400 MHz, DMSO-*d*₆) δ 1.60-1.86 (m, 2H, CH₂), 2.06-2.29 (m, 3H, CH₂), 2.41-2.49 (m, 1H, H-CH), 7.42 (d, *J* = 8.3 Hz, 1H, arom), 7.65-7.80 (m, 3H, arom), 7.78 (d, *J* = 7.2 Hz, 2H, arom), 7.85 (d, *J* = 6.8 Hz, 1H, arom), 8.00 (brs, 1H, arom), 8.21 (d, *J* = 8.0 Hz, 2H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 20.6, 26.3, 36.9, 126.7, 127.3, 127.4, 127.7, 128.5, 129.8, 130.3, 130.7, 131.0, 132.4, 132.8, 133.8, 134.1, 134.7, 135.1, 136.8, 136.9, 152.9, 167.2, 196.02, 198.8. HRMS (ESI) calcd for C₂₄H₁₅NO₃Cl₂ [M+H]⁺: 436.0507; found: 436.0497.

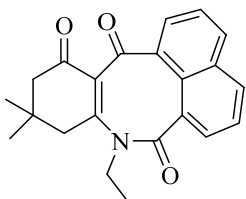


8-(Naphthalen-1-yl)-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3n**):

isolated yield = 90%; white powder; mp >230 °C; IR cm⁻¹: 793, 1015, 1191, 1295, 1363, 1610, 1634, 1661, 1690, 2358, 2873, 3050; The ¹H NMR (400 MHz, DMSO-*d*₆) spectra of this compound shows the existence of two of diastereomers (conformers) which their ratio was calculated from the integrals of the relative peaks as 1:0.21; δ 1.45-1.82 (m, 2.4H, CH₂ of major and minor conformers), 1.89-2.08 (m, 1.2H, H-CH of both conformers), 2.10-2.31 (m, 2.42H, CH₂ of both conformers), 2.54-2.64 (m, 1.21H, H-CH of both conformers), 7.58 (d, *J* = 6.9 Hz, 1H, arom), 7.63-7.95 (m, 7.26H, arom), 7.97-8.33 (m, 7.47H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 20.5, 20.8, 26.7, 27.0, 36.9, 122.4, 122.7, 125.5, 126.6, 126.6, 126.7, 126.8, 127.2, 127.3, 127.4, 127.5, 127.8, 127.9, 128.1, 128.4, 128.7, 128.9, 129.1, 129.2, 130.1, 130.2, 130.3, 130.6, 130.9, 131.7, 132.3, 132.5, 132.7, 132.8, 134.4, 134.5, 134.7, 134.9, 135.1, 135.2, 136.5, 137.0, 137.4, 154.2, 154.3, 168.1, 170.9, 196.2, 199.0. HRMS (ESI) calcd for C₂₈H₁₉NO₃ [M+H]⁺: 418.1443; found: 418.1439.

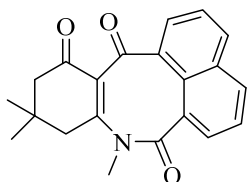


10,10-Dimethyl-8-(naphthalen-1-yl)-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3o**): isolated yield = 88%; white powder; mp >230 °C; IR cm^{-1} : 770, 805, 1313, 1357, 1680, 1633, 1666, 1680, 2360, 2952; The ^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectra of this compound shows the existence of two of diastereomers (conformers) which their ratio was calculated from the integrals of the relative peaks as 1:0.18; δ 0.60 (s, 0.57H, CH_3 of minor conformer), 0.64 (s, 0.57H, CH_3 of minor conformer), 0.67 (s, 3H, CH_3 of major conformer), 0.74 (s, 3H, CH_3 of major conformer), 1.80-2.03 (m, 1.5H, H-CH of minor and major conformers), 2.03-2.20 (m, 2.38H, H- CH_2 of minor and major conformers), 3.31-2.49 (m, 1H, H-CH of major conformer), 7.57 (d, $J = 7.2$ Hz, 1H, arom), 7.65-7.77 (m, 3.55H, arom), 7.78-7.90 (m, 3.73H, arom), 8.06 ($J = 6.9$ Hz, 1H, arom) 8.13 (d, $J = 9.3$ Hz, 2H, aqrom), 8.02-8.16 (m, 0.54H, arom), 8.18 (d, $J = 8.5$ Hz, 1H, arom), 8.23 (d, $J = 8.1$ Hz, 2H, arom), 8.17-8.26 (m, 0.55H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 26.0, 27.0, 27.6, 28.0, 32.8, 32.9, 49.9, 50.3, 56.5, 122.7, 125.4, 125.5, 126.4, 126.8, 127.2, 127.4, 127.5, 127.7, 127.8, 128.5, 128.6, 128.8, 129.1, 129.3, 130.1, 130.3, 130.9, 132.5, 132.7, 132.8, 133.0, 134.4, 134.6, 134.7, 135.9, 137.0 151.9, 168.0, 196.1, 198.8. HRMS (ESI) calculated for $\text{C}_{30}\text{H}_{23}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 446.1756; found: 446.1748.

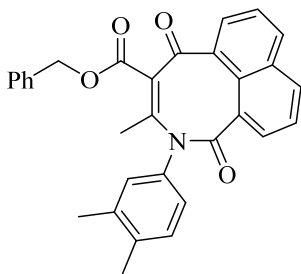


8-Ethyl-10,10-dimethyl-10,11-dihydro-7*H*-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8*H*,9*H*)-trione (**3p**): isolated yield = 85%; white powder; mp = 226-228 °C; IR cm^{-1} : 669, 1284, 1634, 1689, 2341,

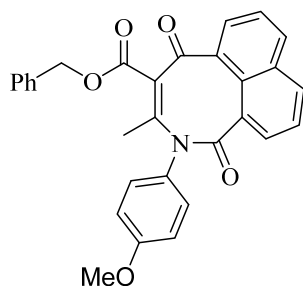
2359, 2959; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 0.57 (s, 3H, CH_3), 1.00 (s, 3H, CH_3), 1.18 (t, $J = 6.9$ Hz, 3H, CH_3), 1.96 (d, $J = 16.5$ Hz, 1H, H-CH), 2.21-2.32 (m, 2H, CH_2), 2.86 (d, $J = 17.5$ Hz, 1H, H-CH), 3.54 (m, 1H, H-CH_{Et}), 4.06 (dq, $J_1 = 14.1$ Hz, $J_2 = 7.1$ Hz, 1H, H-CH_{Et}), 7.59 (d, $J = 6.9$ Hz, 1H, arom), 7.63-7.70 (m, 3H, arom), 8.05-8.22 (m, 2H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 12.9, 26.9, 28.0, 33.6, 50.3, 126.5, 127.2, 127.3, 127.8, 128.6, 130.4, 132.3, 132.8, 135.1, 136.2, 137.0, 152.2, 167.1, 196.1, 197.7, 136.1, 136.8, 150.7, 167.1, 196.0, 198.6. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{21}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 348.1600; found: 348.1593.



8,10,10-Trimethyl-10,11-dihydro-7H-benzo[*b*]naphtho[1,8-*ef*]azocine-7,12,13(8H,9H)-trione (**3q**): isolated yield = 85%; white powder; mp = 222-224 °C; IR cm^{-1} : 777, 1362, 1636, 1684, 2342, 2360, 2957; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 0.57 (s, 3H, CH_3), 1.00 (s, 3H, CH_3), 1.97 (d, $J = 16.4$ Hz, 1H, H-CH), 2.17-2.36 (m, 2H, CH_2), 2.88 (d, $J = 17.5$ Hz, 1H, H-CH), 3.35 (s, 3H, CH_3), 7.59-7.73 (m, 5H, arom), 8.13 (dd, $J_1 = 8.1$ Hz, $J_2 = 1.2$ Hz, 1H, arom), 8.16 (dd, $J_1 = 7.8$ Hz, $J_2 = 1.8$ Hz, 1H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 26.8, 28.0, 33.2, 33.6, 50.3, 126.4, 127.1, 127.3, 127.8, 129.1, 130.6, 132.4, 132.9, 134.8, 135.3, 137.1, 153.5, 167.6, 196.1, 197.6. HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{19}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 334.1443; found: 334.1439.

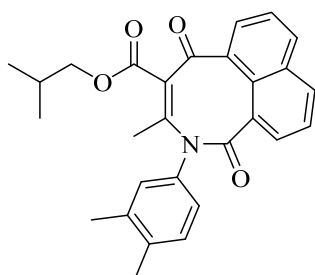


Benzyl (E)-2-(3,4-dimethylphenyl)-3-methyl-1,5-dioxo-2,5-dihydro-1H-naphtho[1,8-cd]azocine-4-carboxylate (**3r**): isolated yield = 94%; white powder; mp >230 °C; IR cm^{-1} : 778, 818, 1224, 1251, 1509, 1644, 1658, 1686, 1719, 2341, 2360, 2956, 3006; The ^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectra of this compound shows the existence of two of diastereomers (conformers) which their ratio was calculated from the integrals of the relative peaks as 1:0.2; δ 1.91 (s, 0.59H, CH_3 of minor conformer), 2.12 (s, 3H, CH_3 of major conformer), 2.14 (s, 1.2H, 2^*CH_3 of minor conformer), 2.28 (s, 3H, CH_3 of major conformer), 2.30 (s, 3H, CH_3 of major conformer), 4.92 (d, $J = 12.4$ Hz, 1H, H-CH of major conformer), 4.99 (d, $J = 12.4$ Hz, 1H, CH_2 of major conformer), 5.19 (s, 0.42H, CH_2 of minor conformer), 6.19 (dd, $J_1 = 8.0$ Hz, $J_2 = 2.0$ Hz, 0.21H, arom), 6.28 (d, $J = 2.0$ Hz, 0.21H, arom), 6.93-7.08 (m, 2.12H, arom), 7.09-7.17 (m, 0.49H, arom), 7.18-7.45 (m, 6.65H, arom), 7.53-7.64 (m, 2H, arom), 7.74 (t, $J = 8.0$ Hz, 1H, arom), 7.84-8.02 (m, 1.59H, arom), 8.14-8.27 (m, 2H, arom), 8.37 (dd, $J_1 = 7.2$ Hz, $J_2 = 1.2$ Hz, 0.53H, arom), 8.52 (dd, $J_1 = 8.4$ Hz, $J_2 = 1.2$ Hz, 0.53H, arom); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 18.2, 18.7, 19.2, 19.6, 19.8, 19.9, 66.8, 67.7, 85.3, 116.4, 120.3, 124.8, 126.4, 127.3, 127.4, 127.5, 127.8, 127.9, 128.3, 128.4, 128.5, 128.5, 128.6, 128.6, 128.7, 128.8, 129.0, 130.4, 130.5, 130.7, 130.8, 131.1, 132.2, 132.4, 132.6, 133.0, 134.8, 135.4, 135.6, 135.7, 135.8, 136.2, 137.3, 137.4, 138.0, 147.3, 149.5, 161.9, 165.5, 166.2, 167.1, 189.8, 196.7. HRMS (ESI) calcd for $\text{C}_{31}\text{H}_{25}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 476.1784; found: (No expected molecular ion was observed).



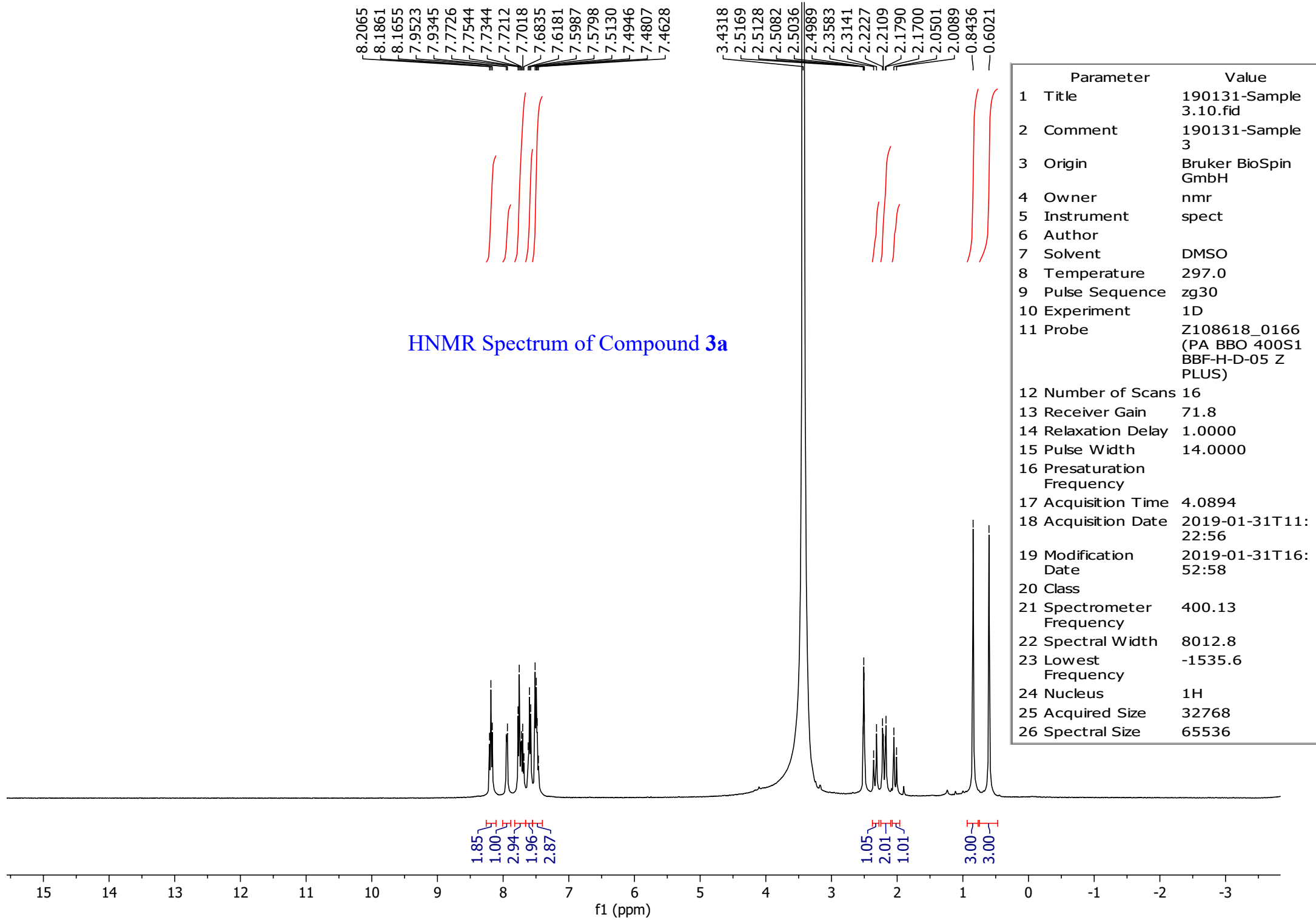
Benzyl(E)-2-(4-methoxyphenyl)-3-methyl-1,5-dioxo-2,5-dihydro-1H-naphtho[1,8-cd]azocine-4-carboxylate (**3s**): isolated yield = 92%; white powder; mp >230 °C; IR cm^{-1} : 778, 818, 1061, 1224, 1251, 1509, 1644, 1648, 1686, 1719, 2360, 2956, 3033; The ^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectra of this compound shows the existence of two of diastereomers (conformers) which their ratio was

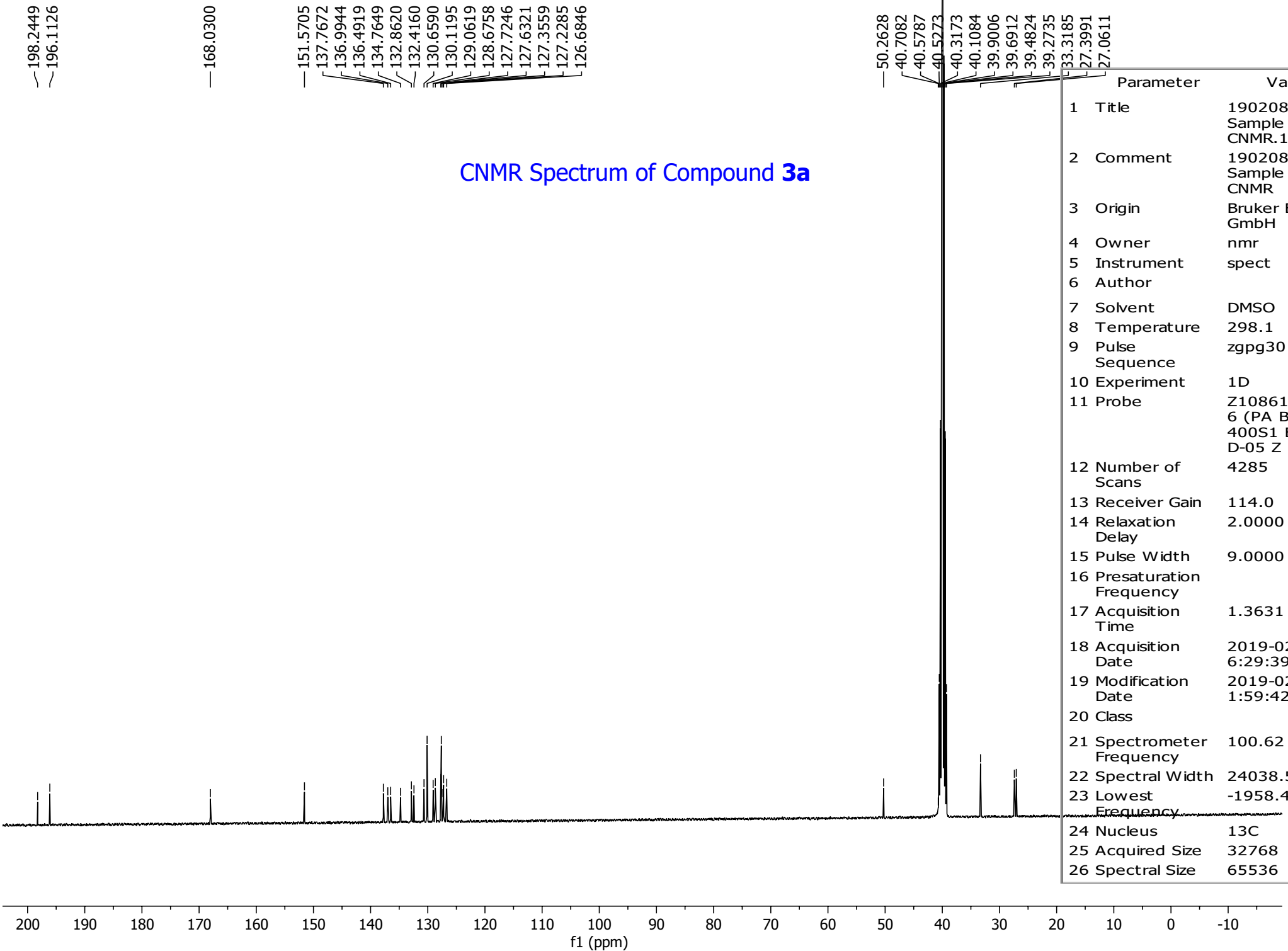
calculated from the integrals of the relative peaks as 1:0.2; δ 1.94 (s, 0.6H, CH₃ of minor conformer), 2.12 (s, 3H, CH₃ of major conformer), 3.70 (s, 0.6H, OCH₃ of minor conformer), 3.82 (s, 3H, CH₃, OCH₃ of major conformer), 4.93 (d, J = 12.4 Hz, 1H, CH₂ of major conformer), 5.00 (d, J = 12.4 Hz, 1H, CH₂ of major conformer), 5.19 (s, 0.4 H, CH₂ of minor conformer), 6.45 (dd, J_1 = 6.6 Hz, J_2 =2.0 Hz, 0.4H, arom), 6.86 (dd, J_1 = 6.8 Hz, J_2 =2.0 Hz, 0.4H, arom), 6.93-7.00 (m, 2H, arom), 7.08-7.18 (m, 2.42H, arom), 7.23-7.27 (m, 0.6H, arom), 7.28-7.36 (m, 3H, arom), 7.41-7.48 (m, 2.H, arom), 7.56 (d, J =7.6 Hz, 1H, arom), 7.59 (d, J = 7.2 Hz, 1H, arom), 7.4 (t, J = 7.2 Hz, 1H, arom), 7.86-7.92 (m+dd, J_1 = 7.2 Hz, J_2 =0.8 Hz, 1.4H, arom), 8.14-8.24 (m, 2H, arom), 8.37 (dd, J_1 = 7.2 Hz, J_2 = 1.2 Hz, 0.4H, arom), 8.52 (dd, J_1 = 8.0 Hz, J_2 = 1.2 Hz, 0.4H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 18.1, 18.7, 55.6, 55.9, 66.8, 67.6, 114.7, 115.2, 120.9, 126.4, 127.3, 127.8, 127.4, 127.5, 127.8, 128.3, 128.4, 128.5, 128.6, 128.7, 128.8, 128.8, 128.9, 129.0, 130.5, 130.6, 131.0, 132.4, 132.6, 133.0, 134.7, 135.4, 135.6, 135.7, 142.3, 149.5, 156.5, 159.4, 161.9, 165.6, 166.4, 167.2, 189.8, 196.8. HRMS (ESI) calcd for C₃₀H₂₃NO₅ [M+H]⁺: 478.1674; found: 478.1574.



Isobutyl (E)-2-(3,4-dimethylphenyl)-3-methyl-1,5-dioxo-2,5-dihydro-1H-naphtho[1,8-cd]azocine-4-carboxylate (**3t**): isolated yield = 90%; white powder; mp >230 °C; IR cm⁻¹: 780, 1064, 1233, 1324, 1501, 1644, 1661, 1686, 1717, 2360, 2890, 2962; The ¹H NMR (400 MHz, DMSO-*d*₆) spectra of this compound shows the existence of two of diastereomers (conformers) which their ratio was calculated from the integrals of the relative peaks as 1:0.2; δ 0.60 (d, J = 6.8 Hz, 3H, CH₃ of major conformer), 0.66-0.80 (m+d, J = 6.8 Hz, 3.5H, CH₃ of major and minor conformers), 1.65 (two overlapped septets, J = 6.8 Hz, 1.11H, CH of major and minor conformers), 1.90 (s, 0.33H, CH₃ of minor conformer), 2.12 (s, 3H, CH₃ of major conformer), 2.14 (s, 0.6H, CH₃ of minor conformer), 2.28 (s, 3H, CH₃ of major

conformer), 2.30 (s, 3H, CH₃ of major conformer), 3.60-3.74 (m, 2H, CH₂ of major conformer), 3.88 (d, J= 6.0 Hz, CH₂ of minor conformer), 6.20 (d, J= 7.6 Hz, 0.11H, arom), 6.3 (s, 0.11H, arom), 7.03 (s, 0.11H, arom), 7.11-7.45 (m, 3H, arom), 7.68-7.93 (m, 4H, arom), 8.18 (d, J= 8.4 Hz, 1H, arom), 8.21 (d, J= 7.6 Hz, 1H, arom), 8.38 (d, J= 7.6 Hz, 0.33H, arom), 8.52 (d, J= 8.4 Hz, 0.33H, arom); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 19.1, 19.2, 19.4, 20.1, 24.4, 27.8, 28.0, 30.7, 71.6, 107.3, 117.0, 120.7, 126.8, 127.5, 129.6, 129.9, 131.1, 131.9, 135.1, 136.6, 137.5, 168.4, 170.9. HRMS (ESI) calcd for C₂₈H₂₇NO₄ [M+H]⁺: 442.1940; found: (No expected molecular ion was observed).





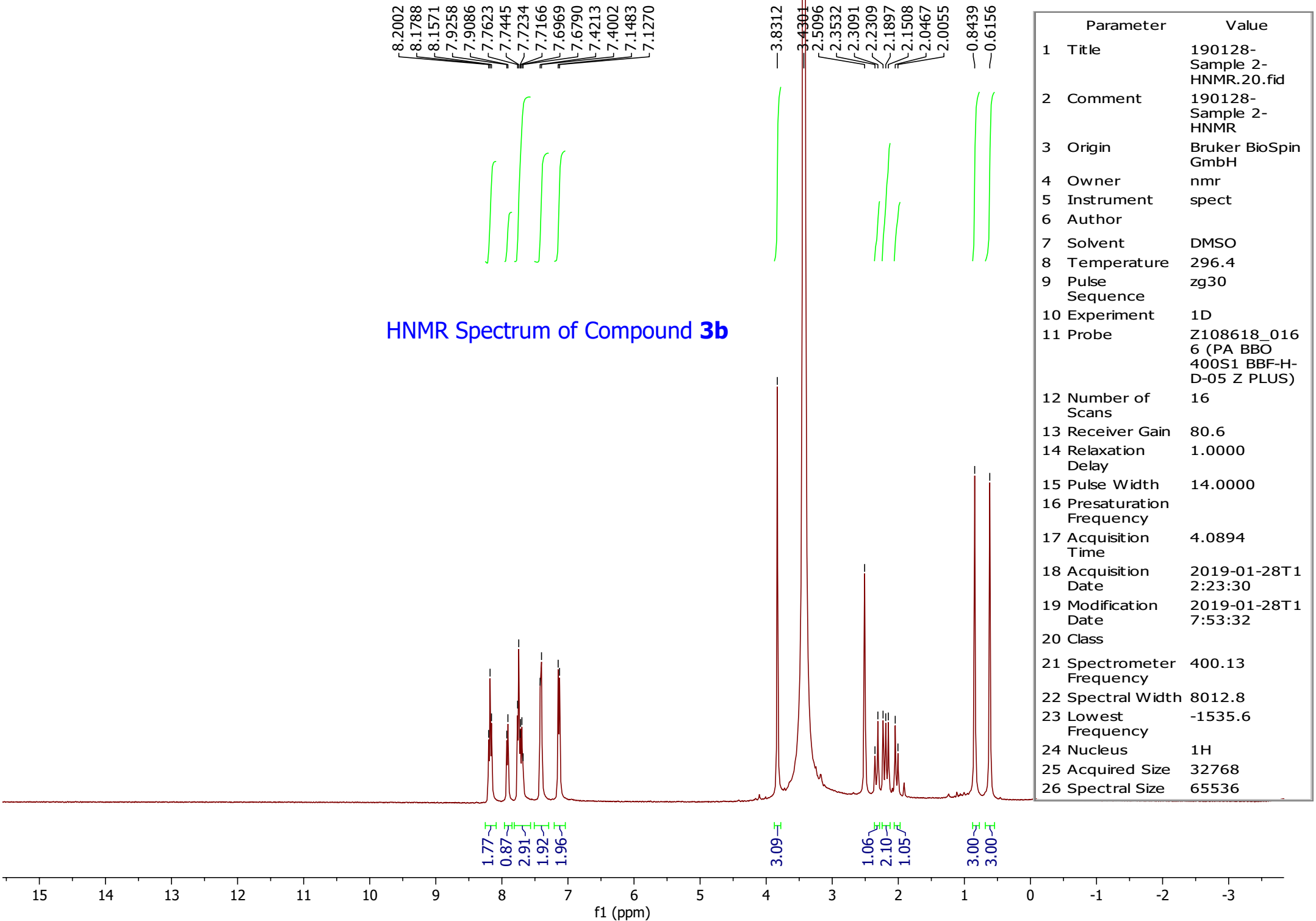
CNMR Spectrum of Compound 3a

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2 Comment	190208-Sample 3-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.1
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4285
13 Receiver Gain	114.0
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15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-08T16:29:39
19 Modification Date	2019-02-08T21:59:42
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

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HNMR Spectrum of Compound **3b**



Parameter	Value
1 Title	190128-Sample 2-HNMR.20.fid
2 Comment	190128-Sample 2-HNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.4
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
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14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-01-28T12:23:30
19 Modification Date	2019-01-28T17:53:32
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536

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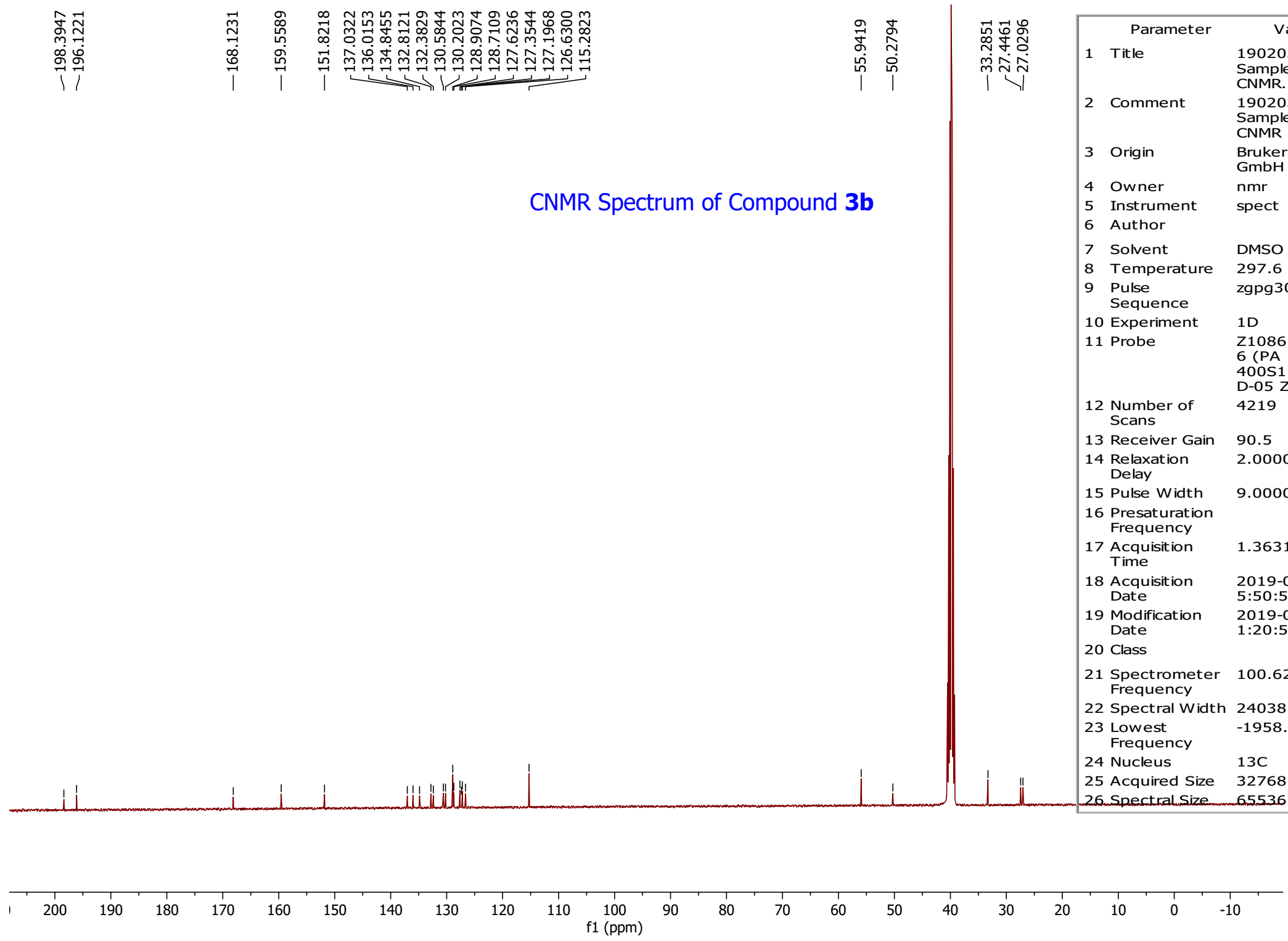
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CNMR Spectrum of Compound **3b**

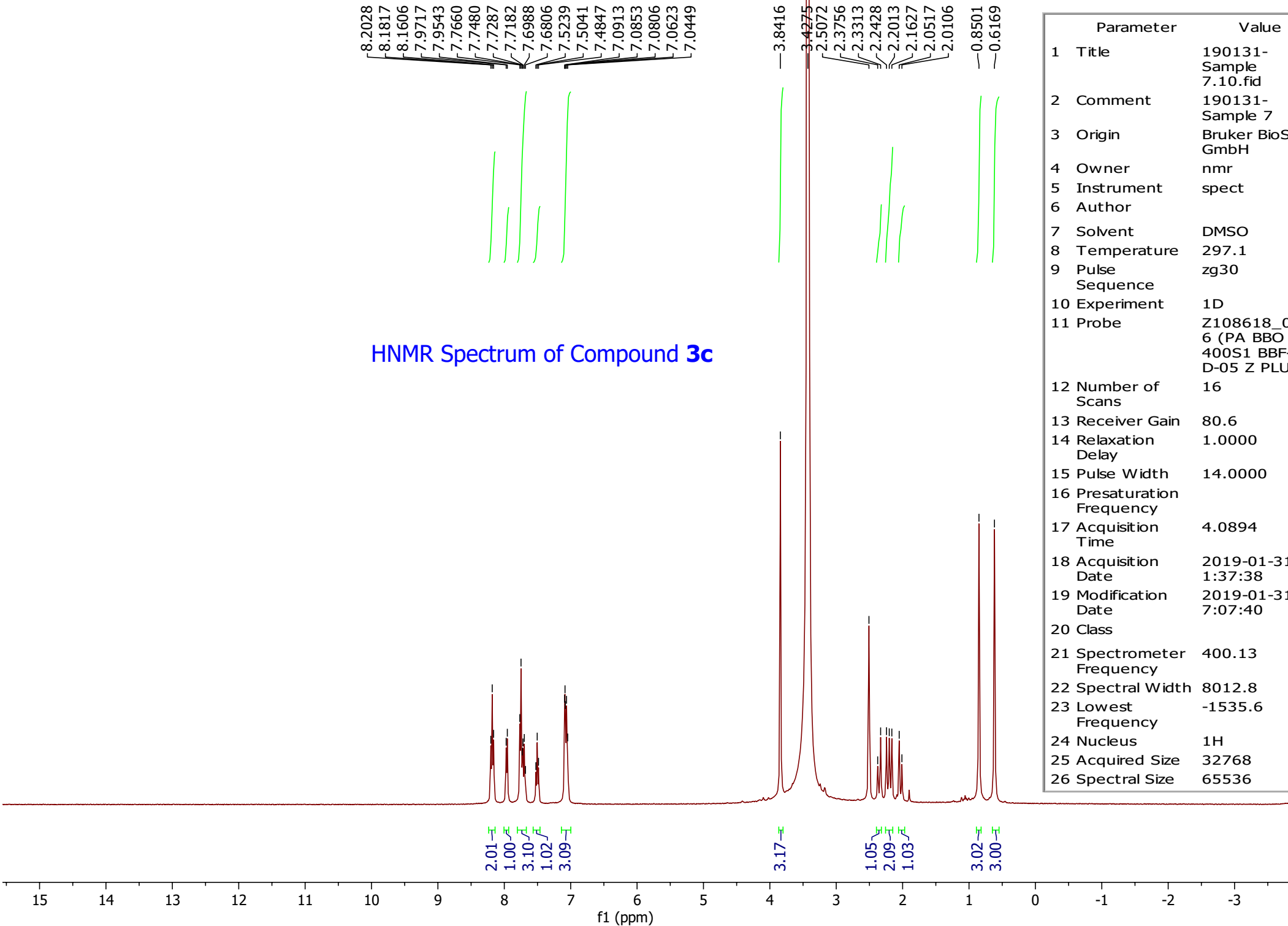


Parameter	Value
1 Title	190203-Sample 2-CNMR.10.fid
2 Comment	190203-Sample 2-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	297.6
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4219
13 Receiver Gain	90.5
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-03T15:50:55
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20 Class	
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22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

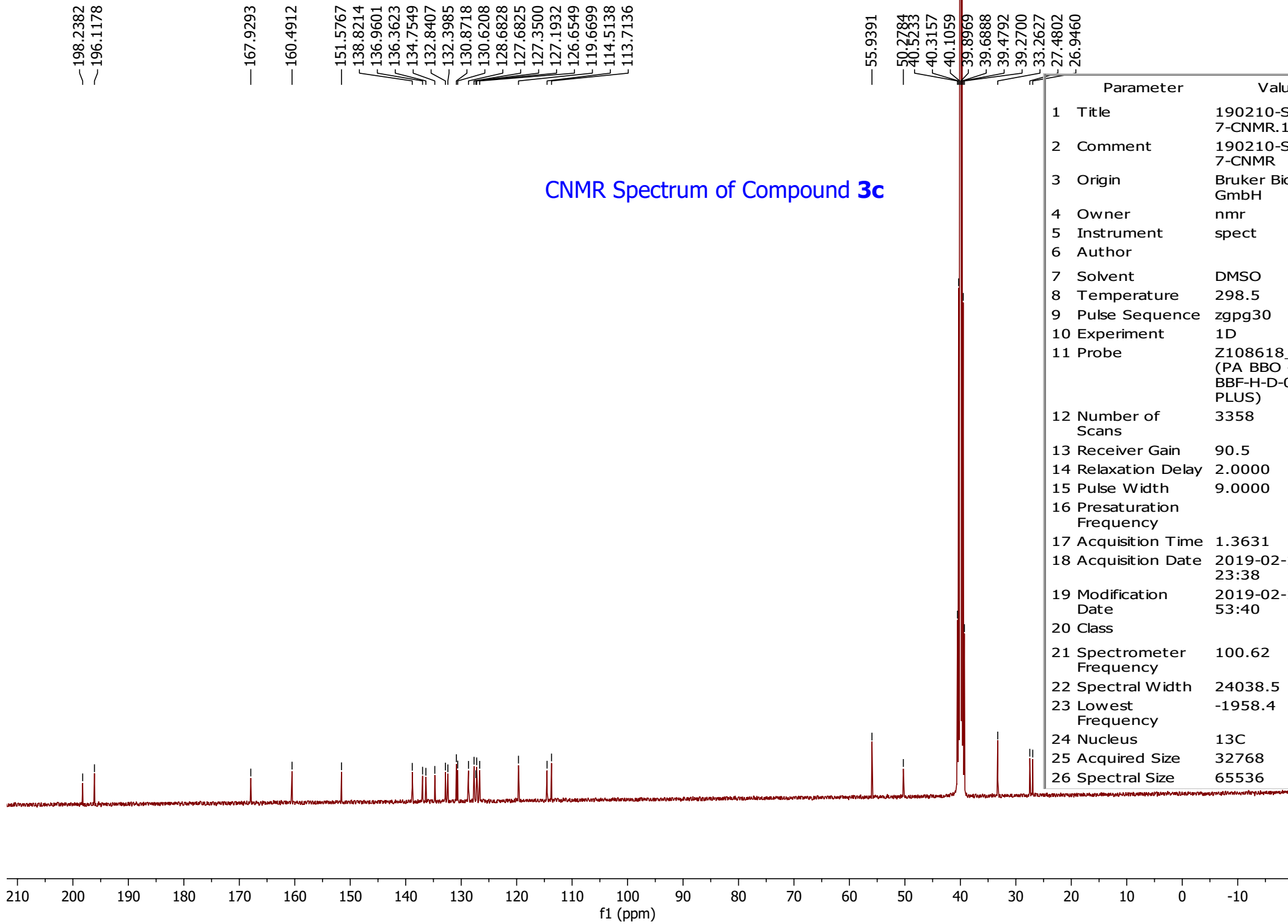
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HNMR Spectrum of Compound **3c**



Parameter	Value
1 Title	190131-Sample 7.10.fid
2 Comment	190131-Sample 7
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	297.1
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	80.6
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-01-31T1 1:37:38
19 Modification Date	2019-01-31T1 7:07:40
20 Class	
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22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536

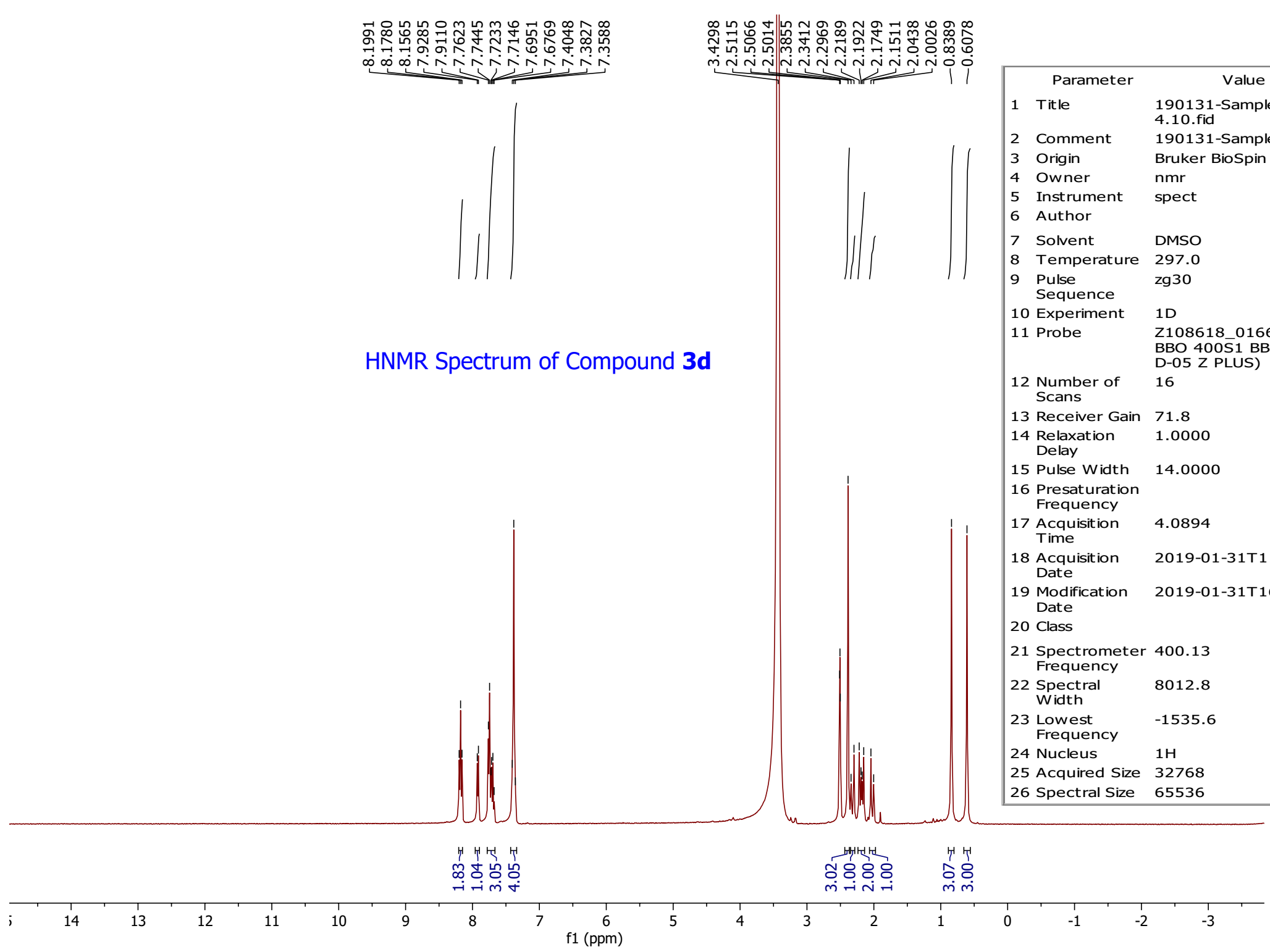


Parameter	Value
1 Title	190210-Sample 7-CNMR.10.fid
2 Comment	190210-Sample 7-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.5
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	3358
13 Receiver Gain	90.5
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-10T15:23:38
19 Modification Date	2019-02-10T20:53:40
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

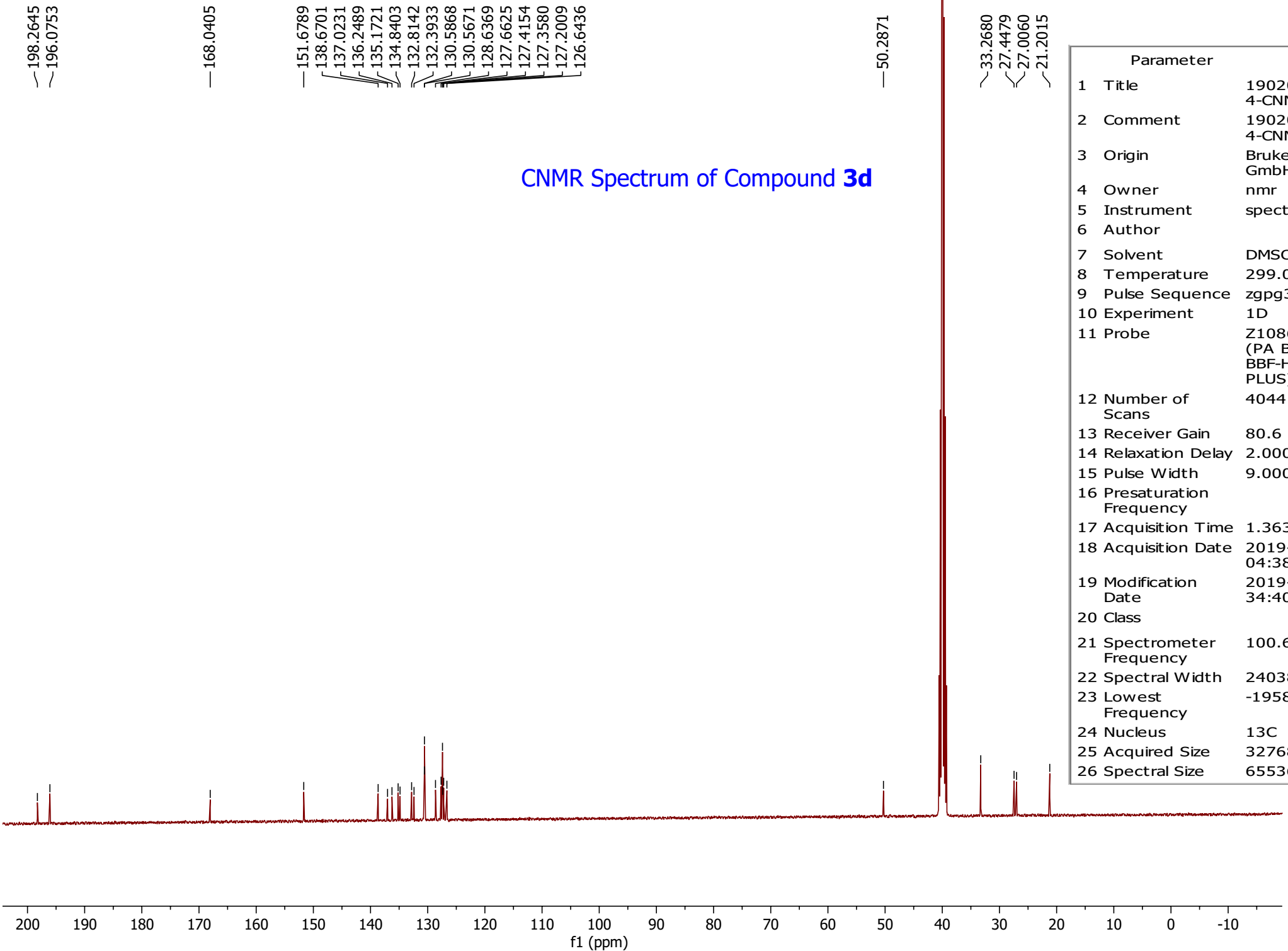
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8.1565
7.9285
7.9110
7.7623
7.7445
7.7233
7.7146
7.6951
7.6769
7.4048
7.3827
7.3588

3.4298
2.5115
2.5066
2.5014
2.3855
2.3412
2.2969
2.2189
2.1922
2.1749
2.1511
2.0438
2.0026
-0.8389
-0.6078

HNMR Spectrum of Compound **3d**



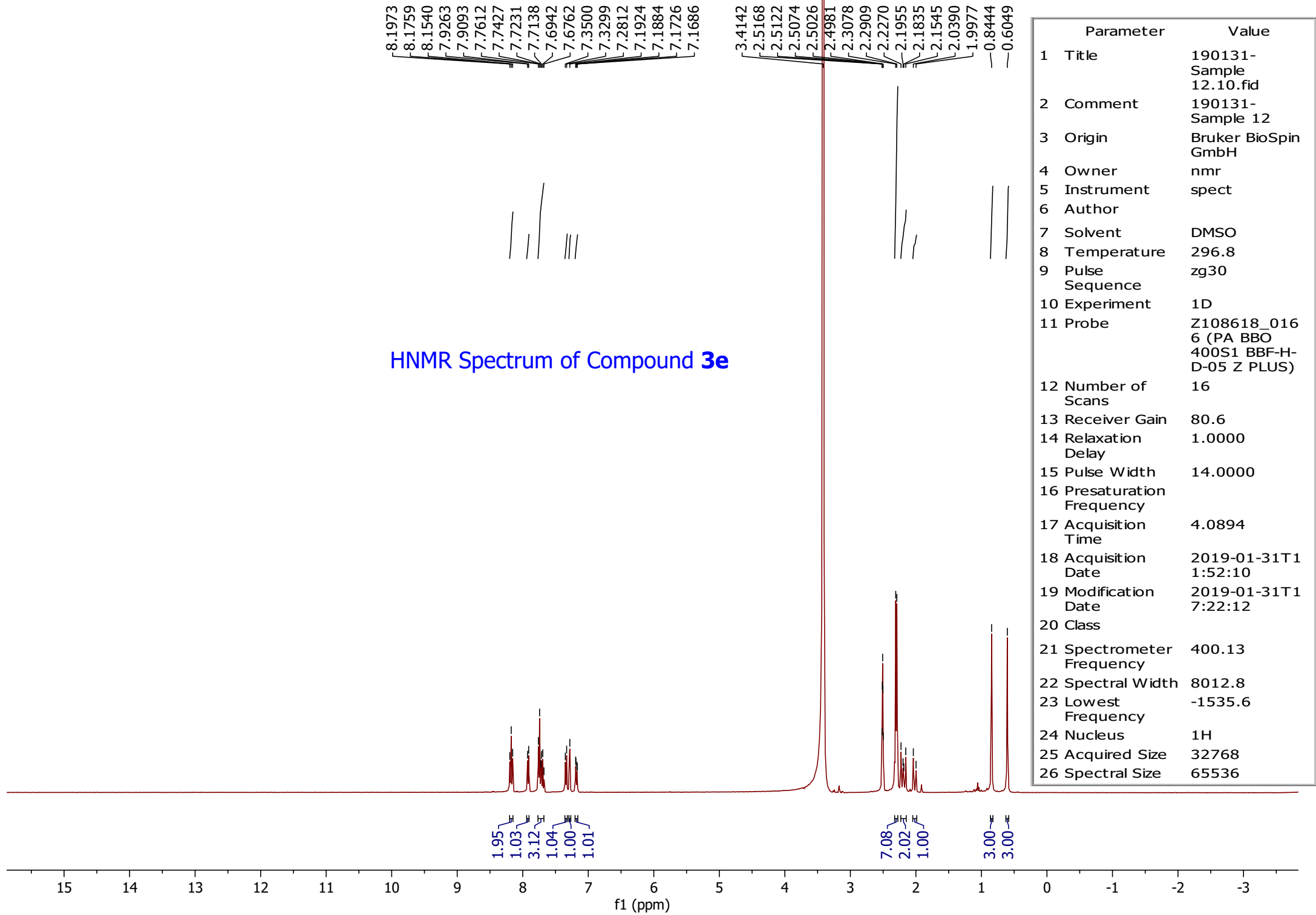
Parameter	Value
1 Title	190131-Sample 4.10.fid
2 Comment	190131-Sample 4
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	297.0
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	71.8
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-01-31T11:28:16
19 Modification Date	2019-01-31T16:58:18
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536



CNMR Spectrum of Compound 3d

Parameter	Value
1 Title	190209-Sample 4-CNMR.10.fid
2 Comment	190209-Sample 4-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	299.0
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4044
13 Receiver Gain	80.6
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-09T13:04:38
19 Modification Date	2019-02-09T18:34:40
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

HNMR Spectrum of Compound **3e**



~198.2931
~196.0780

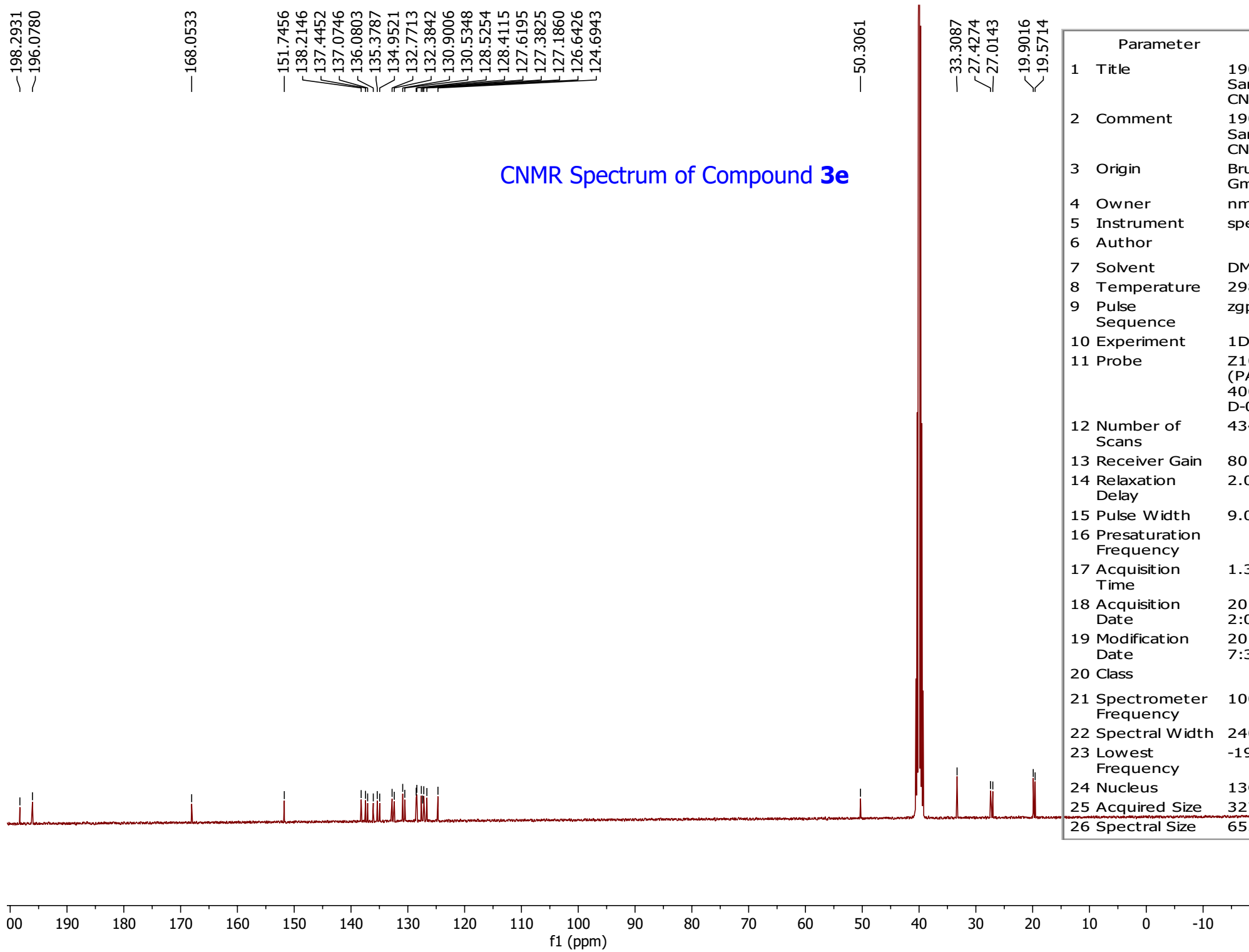
—168.0533

—151.7456
138.2146
137.4452
137.0746
136.0803
135.3787
134.9521
132.7713
132.3842
130.9006
130.5348
128.5254
128.4115
127.6195
127.3825
127.1860
126.6426
124.6943

—50.3061

—33.3087
27.4274
27.0143
19.9016
19.5714

CNMR Spectrum of Compound 3e

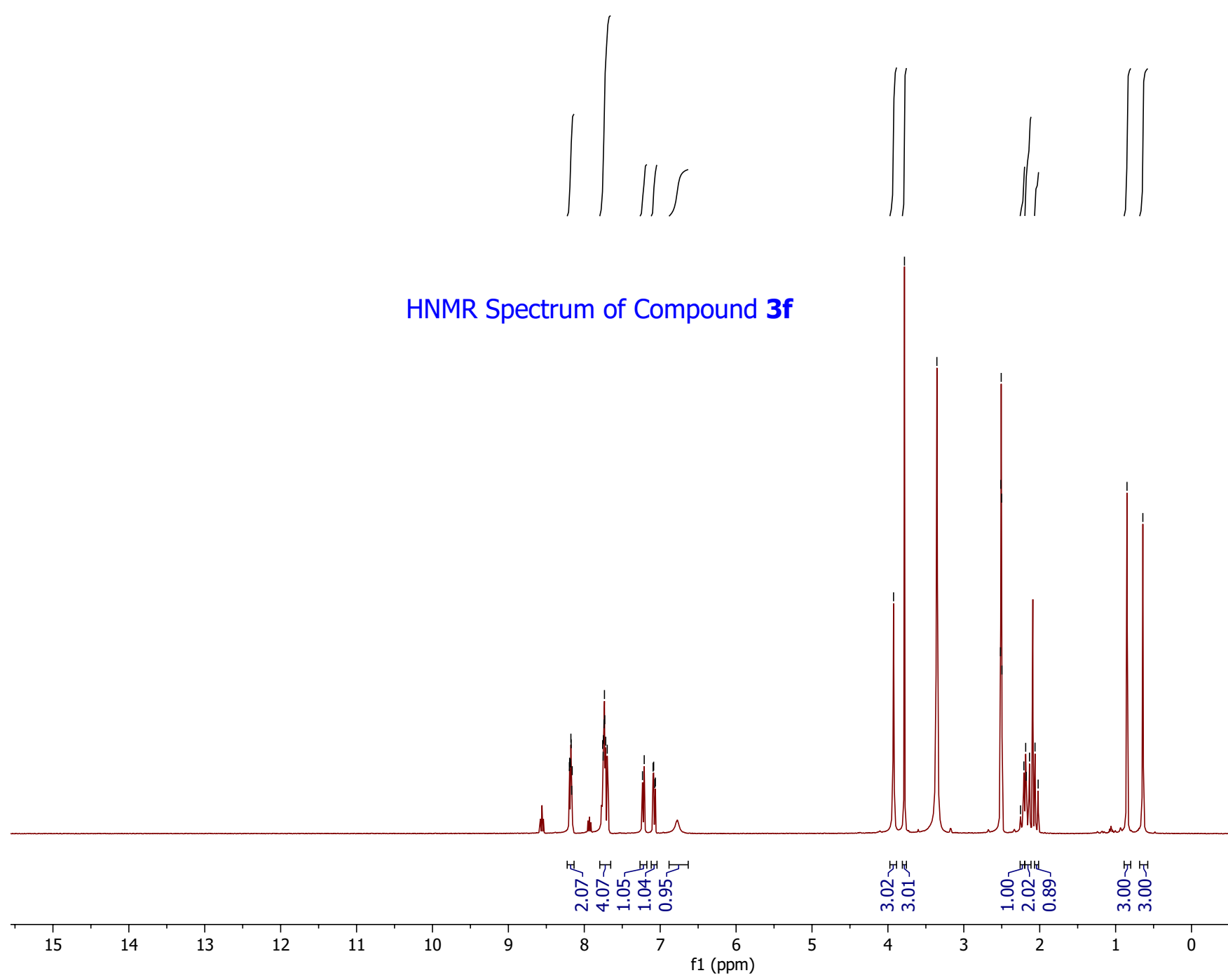


Parameter	Value
1 Title	190216-Sample 12-CNMR.10.fid
2 Comment	190216-Sample 12-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.7
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4347
13 Receiver Gain	80.6
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-16T12:06:52
19 Modification Date	2019-02-16T17:36:54
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

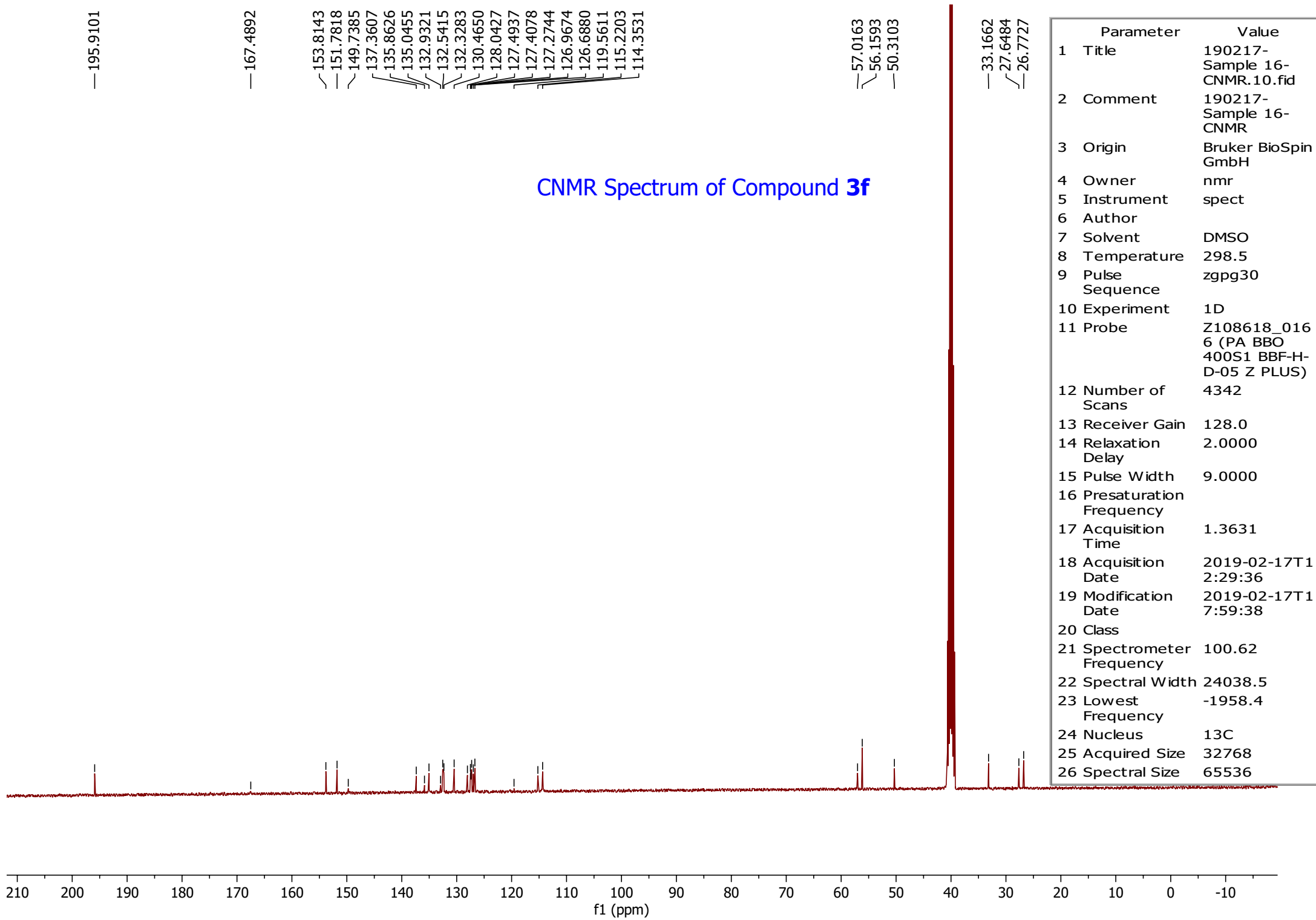
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8.1928
8.1824
8.1763
8.1731
8.1659
8.1592
7.7575
7.7512
7.7476
7.7394
7.7342
7.7308
7.7173
7.6977
7.2329
7.2103
7.0934
7.0857
7.0708
7.0632

3.9255
3.7819
3.3547
2.5165
2.5119
2.5072
2.5026
2.4980
2.2528
2.2088
2.1849
2.1765
2.1347
2.0618
2.0206
0.8500
0.6412

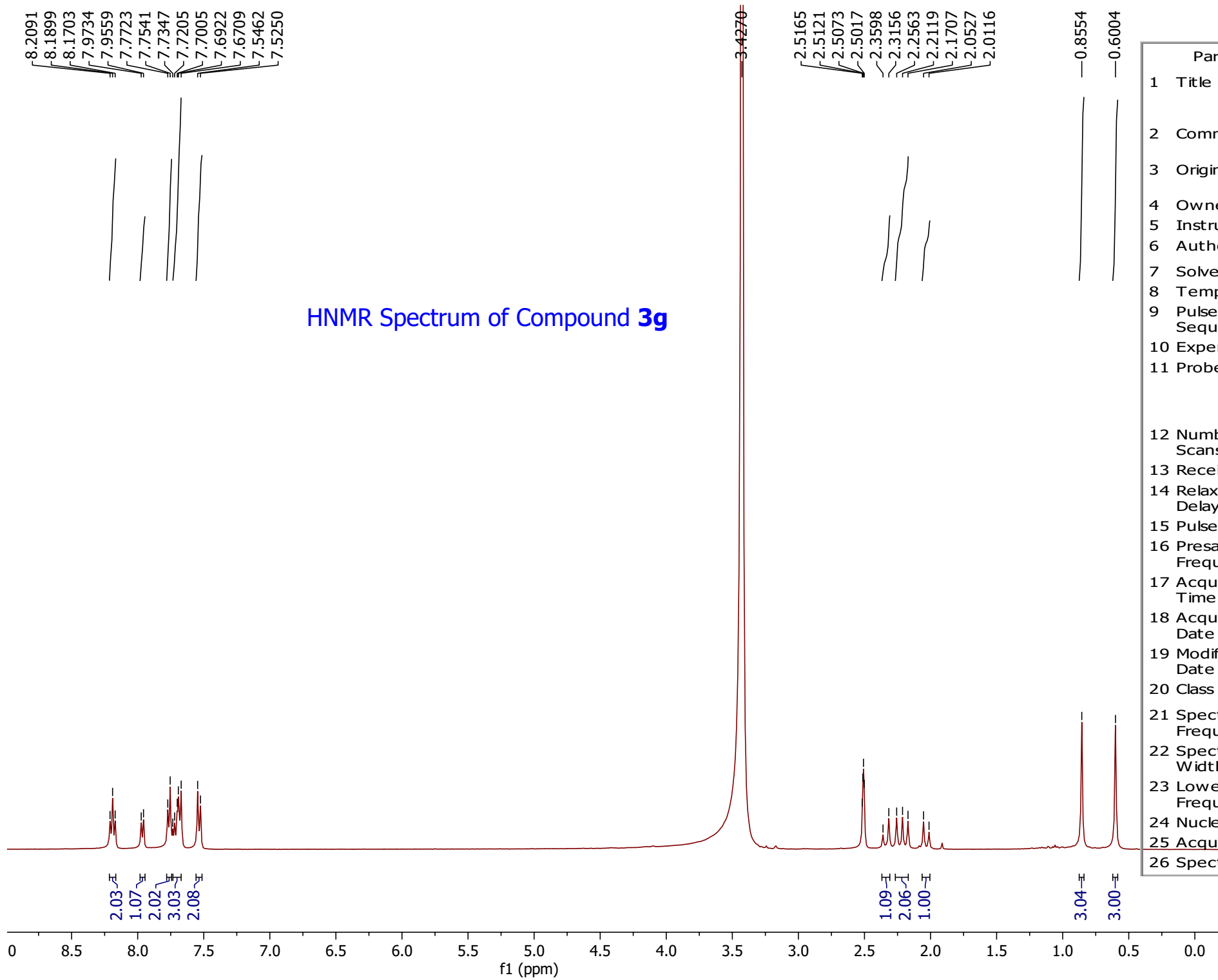
HNMR Spectrum of Compound **3f**



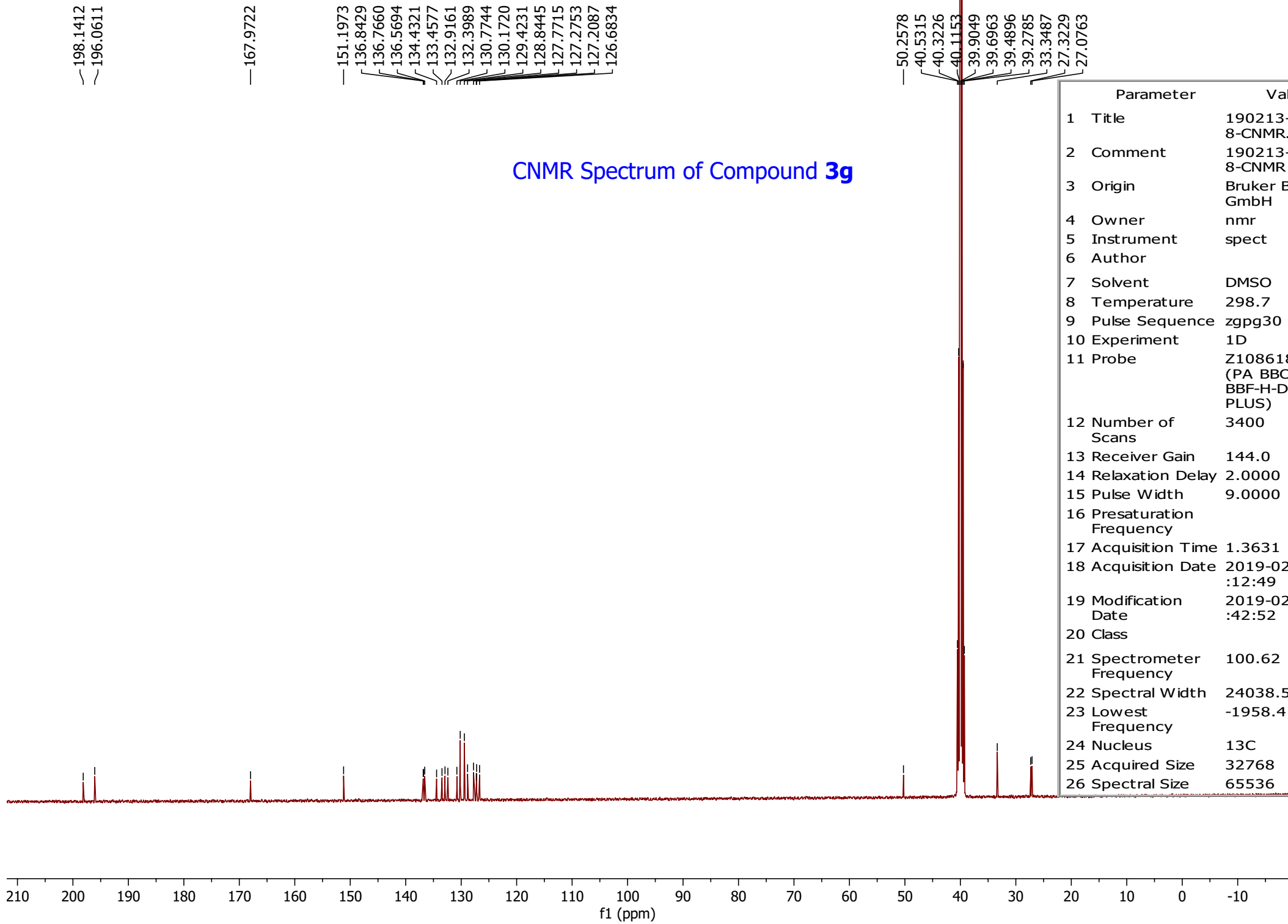
Parameter	Value
1 Title	190201-Sample 16-HNMR.10.fid
2 Comment	190201-Sample 16-HNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.6
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_01 66 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	161.0
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-02-01T 08:55:45
19 Modification Date	2019-02-01T 14:25:46
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	¹ H
25 Acquired Size	32768
26 Spectral Size	65536



Parameter	Value
1 Title	190217-Sample 16-CNMR.10.fid
2 Comment	190217-Sample 16-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.5
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4342
13 Receiver Gain	128.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-17T12:29:36
19 Modification Date	2019-02-17T17:59:38
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

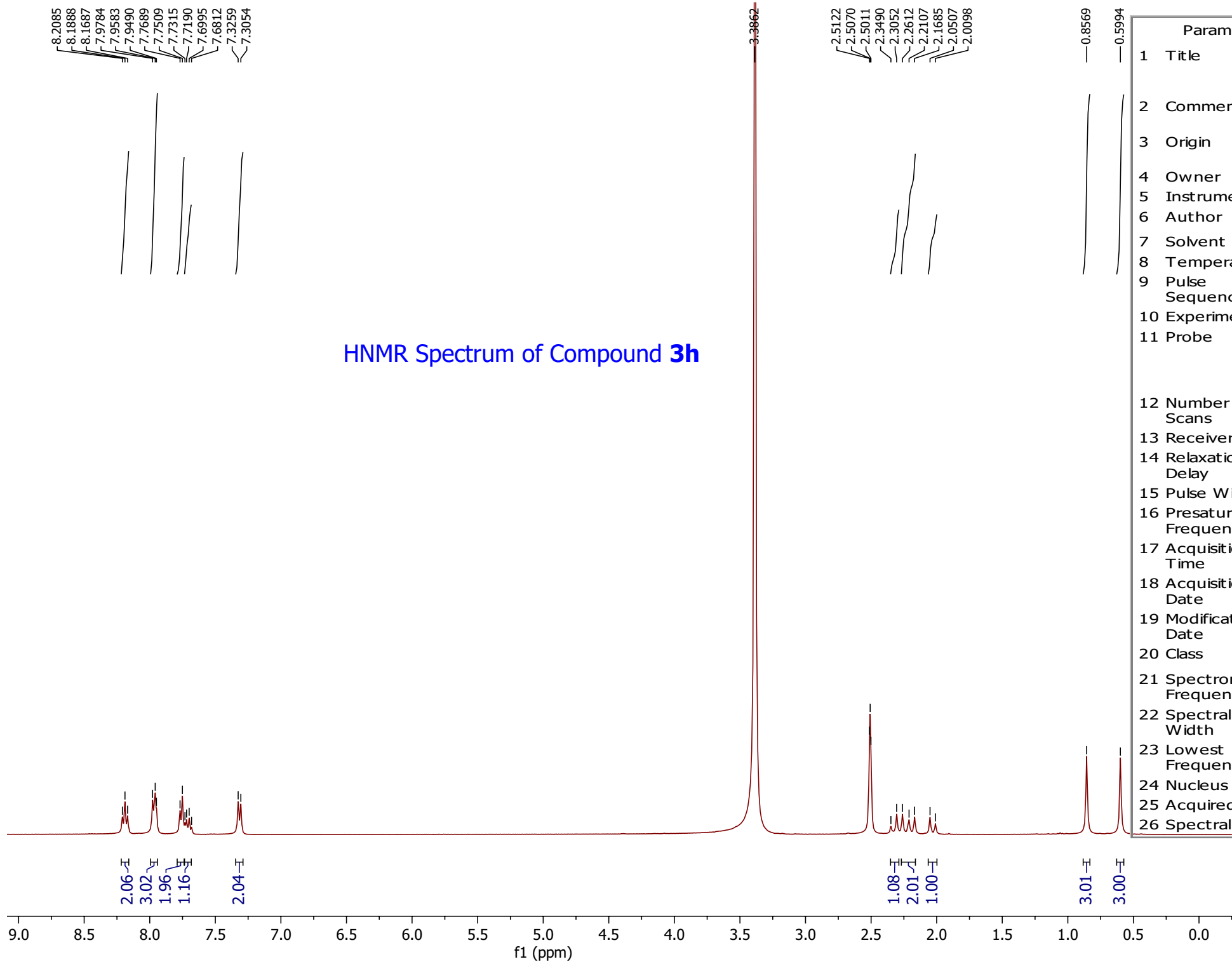


Parameter	Value
1 Title	190131-Sample 8.10.fid
2 Comment	190131-Sample 8
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	297.1
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_01 66 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	80.6
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-01-31T 11:42:41
19 Modification Date	2019-01-31T 17:12:44
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536



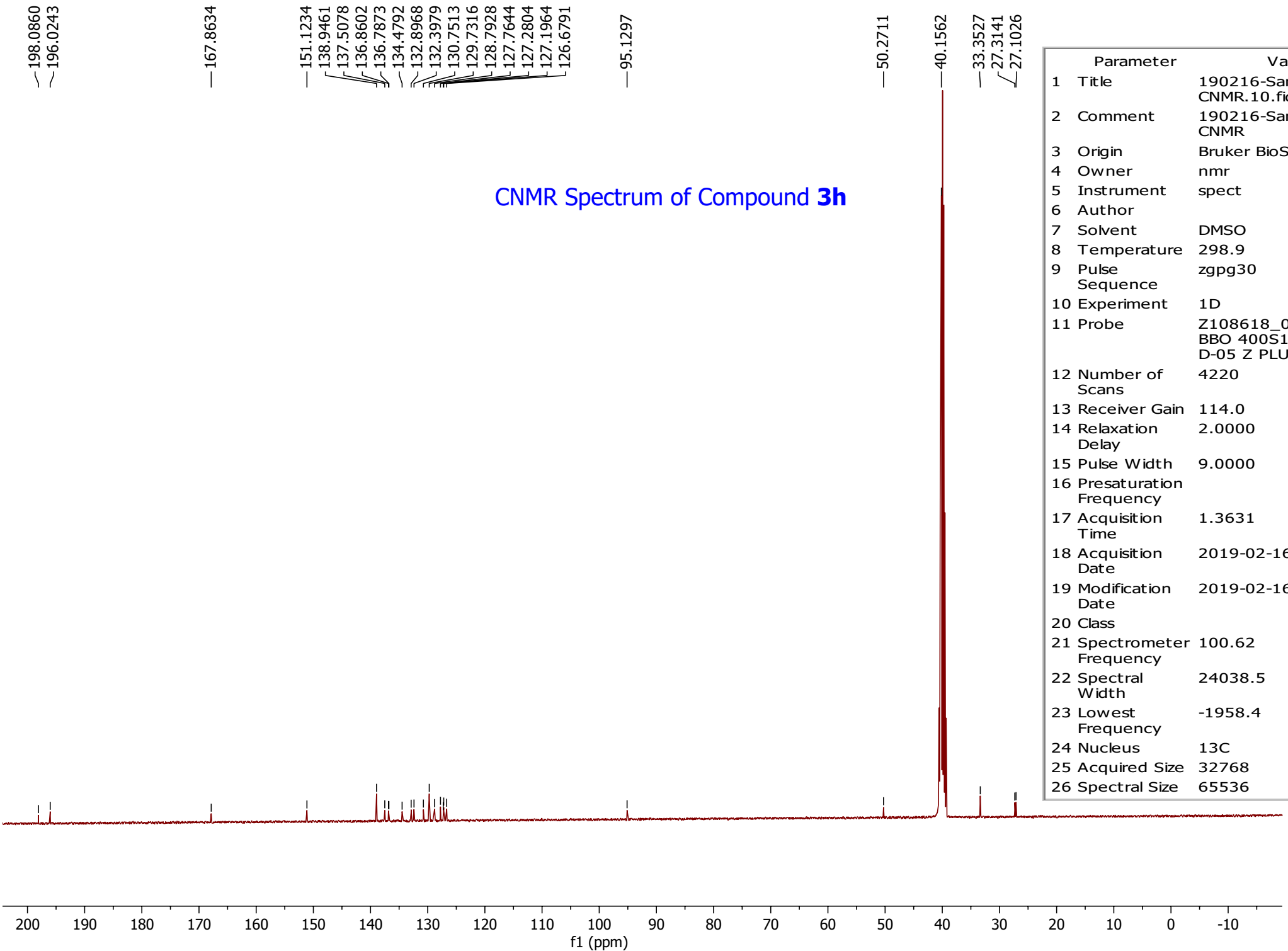
CNMR Spectrum of Compound **3g**

Parameter	Value
1 Title	190213-Sample 8-CNMR.10.fid
2 Comment	190213-Sample 8-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.7
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	3400
13 Receiver Gain	144.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-13T15:12:49
19 Modification Date	2019-02-13T20:42:52
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536

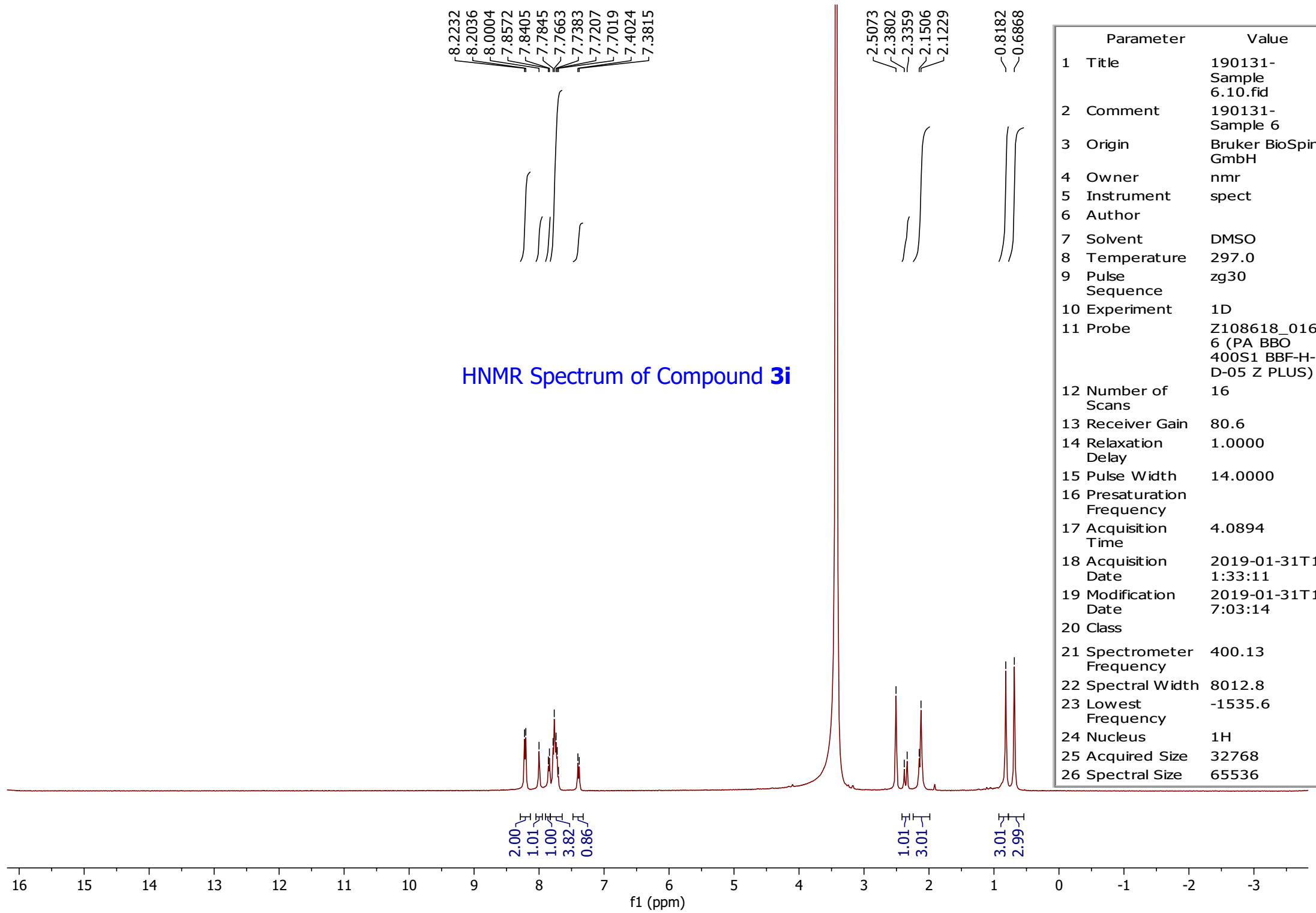


HNMR Spectrum of Compound **3h**

Parameter	Value
1 Title	190131-Sample 13.10.fid
2 Comment	190131-Sample 13
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.7
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	114.0
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-01-31T 11:57:13
19 Modification Date	2019-01-31T 17:27:14
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536



Parameter	Value
1 Title	190216-Sample 13-CNMR.10.fid
2 Comment	190216-Sample 13-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.9
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4220
13 Receiver Gain	114.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-16T16:16:10
19 Modification Date	2019-02-16T21:46:12
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536



8.2232
8.2036
8.0004
7.8572
7.8405
7.7845
7.7663
7.7383
7.7207
7.7019
7.4024
7.3815

2.5073
2.3802
2.3359
2.1506
2.1229

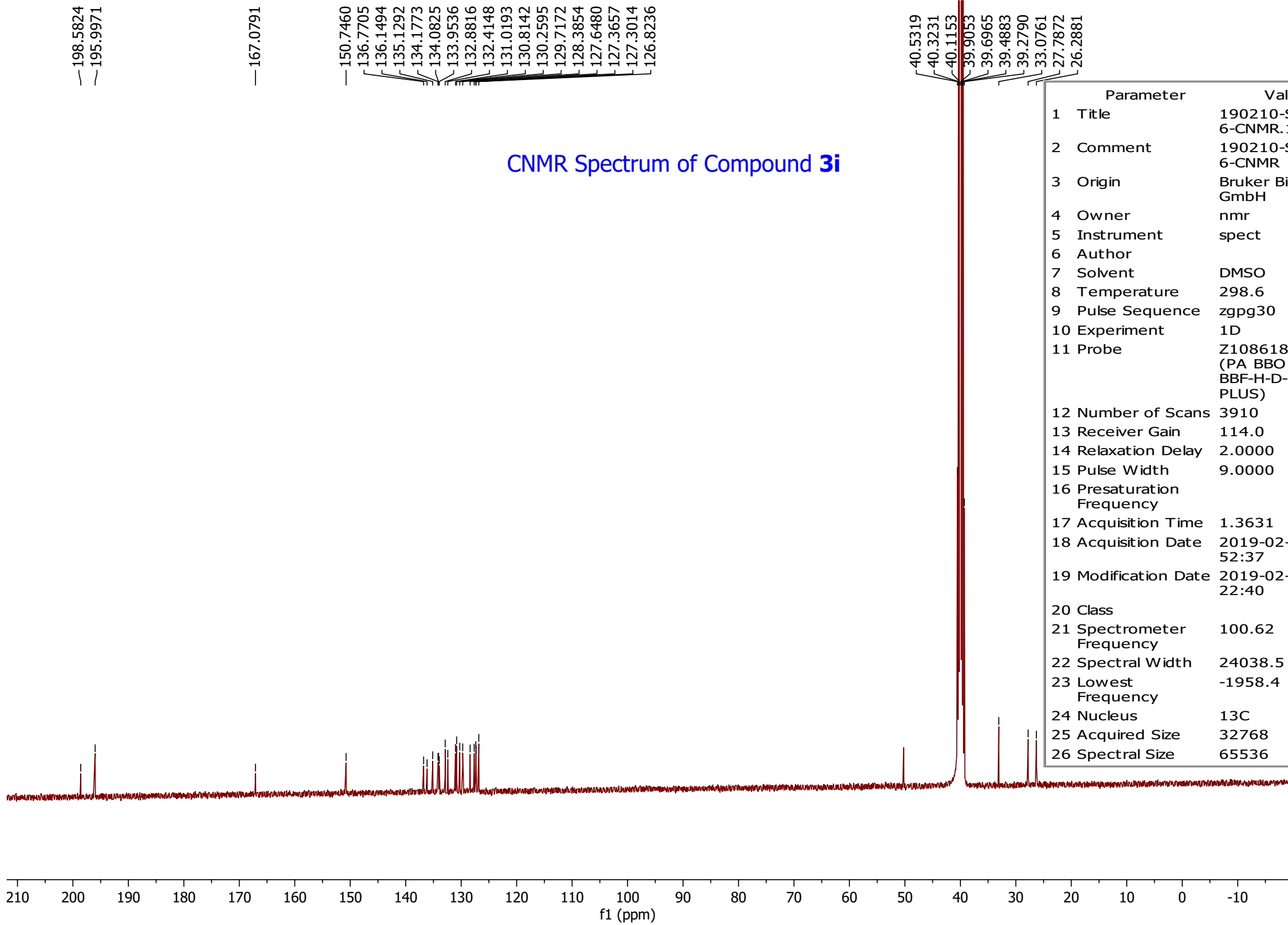
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0.6868

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1.01
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3.82
0.86

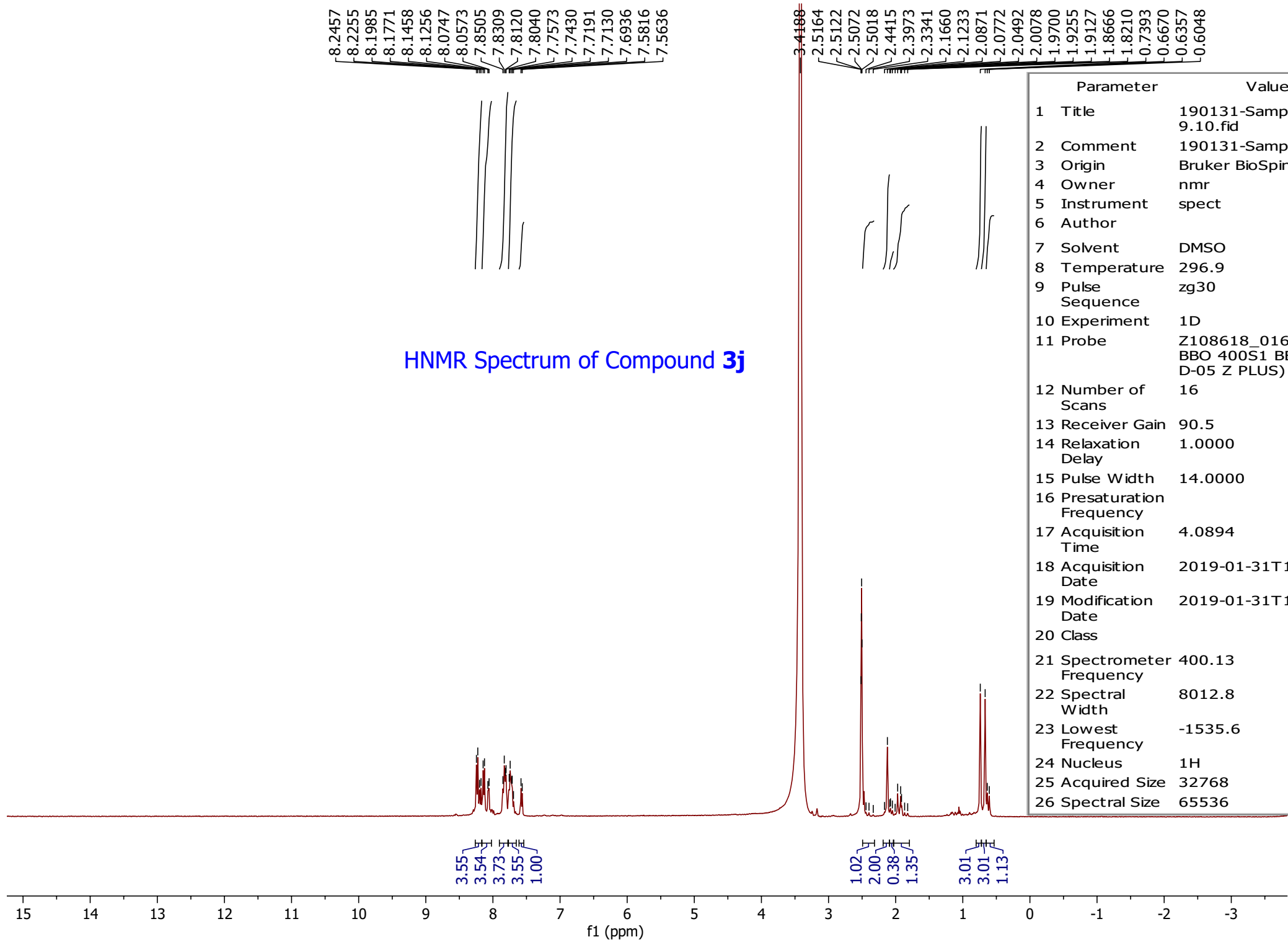
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3.01

3.01
2.99

f1 (ppm)



Parameter	Value
1 Title	190210-Sample 6-CNMR.10.fid
2 Comment	190210-Sample 6-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.6
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	3910
13 Receiver Gain	114.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-10T10:52:37
19 Modification Date	2019-02-10T16:22:40
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536



8.2457
8.2255
8.1985
8.1771
8.1458
8.1256
8.0747
8.0573
7.8505
7.8309
7.8120
7.8040
7.7573
7.7430
7.7191
7.7130
7.6936
7.5816
7.5636

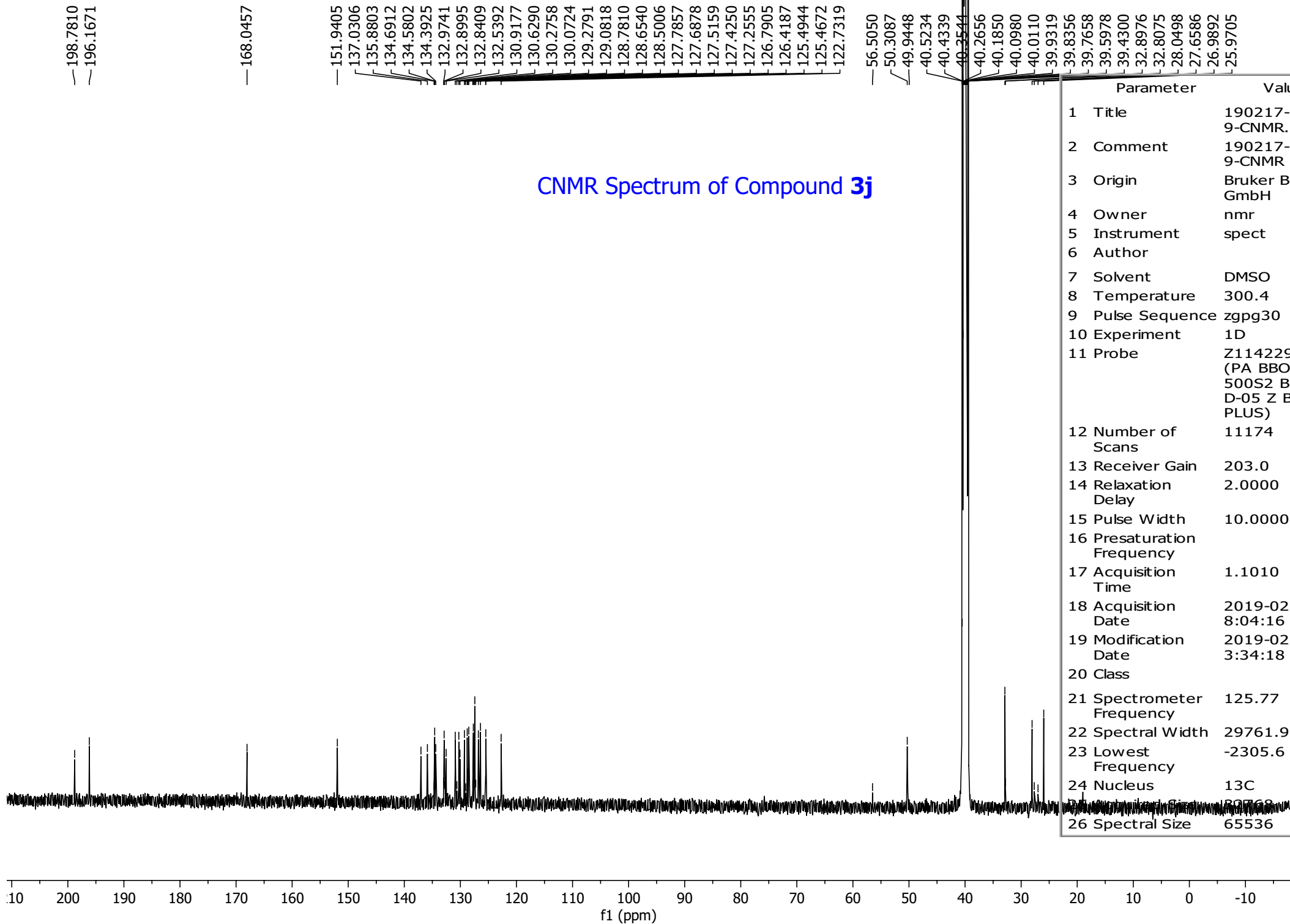
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2.5122
2.5072
2.5018
2.4415
2.3973
2.3341
2.1660
2.1233
2.0871
2.0772
2.0492
2.0078
1.9700
1.9255
1.9127
1.8666
1.8210
0.7393
0.6670
0.6357
0.6048

3.55
3.54
3.73
3.55
1.00

1.02
2.00
0.38
1.35

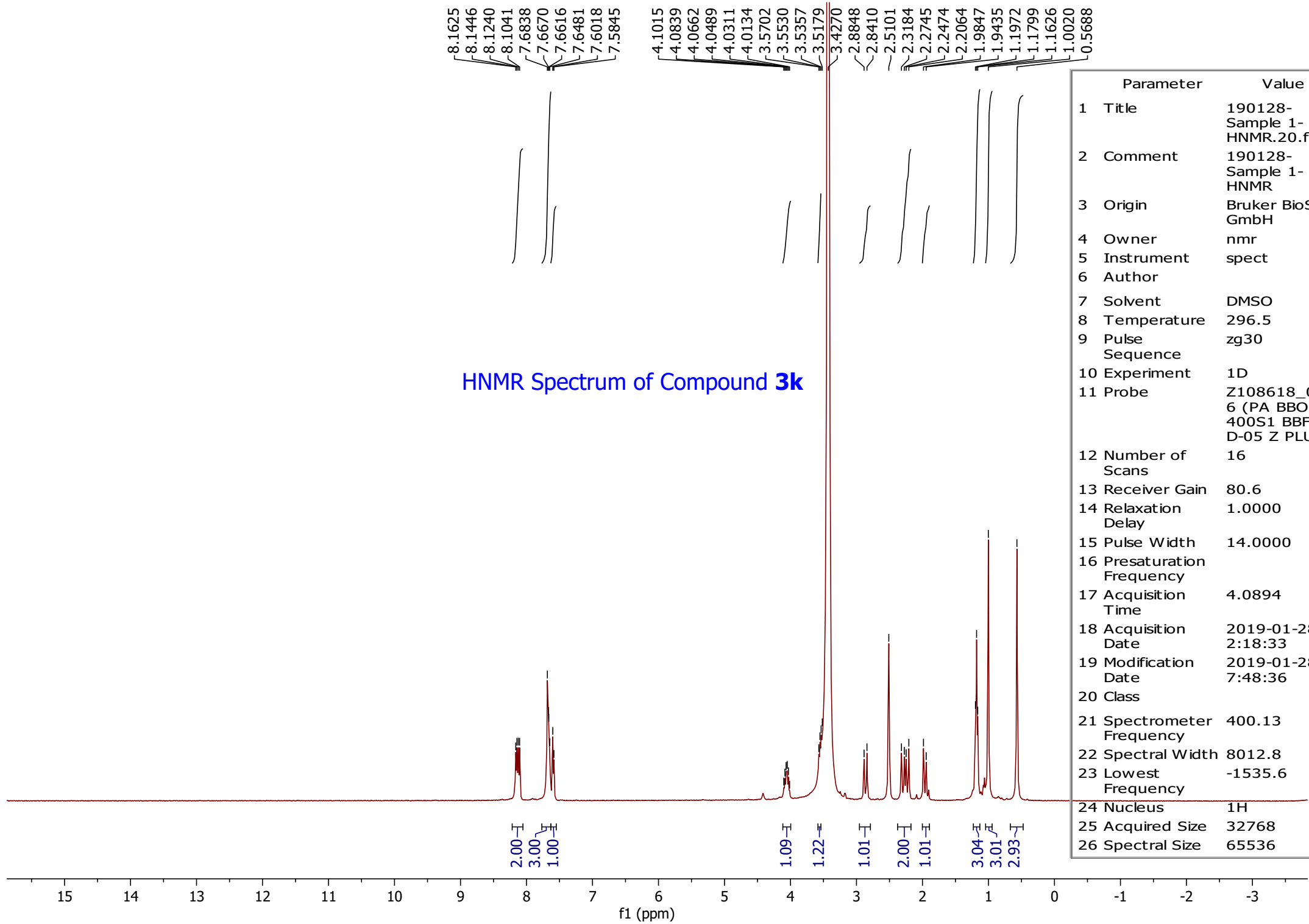
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3.01
1.13

f1 (ppm)



- 198.7810
- 196.1671
- 168.0457
- 151.9405
- 137.0306
- 135.8803
- 134.6912
- 134.5802
- 134.3925
- 132.9741
- 132.8995
- 132.8409
- 132.5392
- 130.9177
- 130.6290
- 130.2758
- 130.0724
- 129.2791
- 129.0818
- 128.7810
- 128.6540
- 128.5006
- 127.7857
- 127.6878
- 127.5159
- 127.4250
- 127.2555
- 126.7905
- 126.4187
- 125.4944
- 125.4672
- 122.7319
- 56.5050
- 50.3087
- 49.9448
- 40.5234
- 40.4339
- 40.3544
- 40.2656
- 40.1850
- 40.0980
- 40.0110
- 39.9319
- 39.8356
- 39.7658
- 39.5978
- 39.4300
- 32.8976
- 32.8075
- 28.0498
- 27.6586
- 26.9892
- 25.9705

Parameter	Value
1 Title	190217-Sample 9-CNMR.10.fid
2 Comment	190217-Sample 9-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	300.4
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z114229_0007 (PA BBO 500S2 BBF-H-D-05 Z BTO PLUS)
12 Number of Scans	11174
13 Receiver Gain	203.0
14 Relaxation Delay	2.0000
15 Pulse Width	10.0000
16 Presaturation Frequency	
17 Acquisition Time	1.1010
18 Acquisition Date	2019-02-17T18:04:16
19 Modification Date	2019-02-17T23:34:18
20 Class	
21 Spectrometer Frequency	125.77
22 Spectral Width	29761.9
23 Lowest Frequency	-2305.6
24 Nucleus	13C
25 File Size	80768
26 Spectral Size	65536



8.1625
8.1446
8.1240
8.1041
7.6838
7.6670
7.6616
7.6481
7.6018
7.5845

4.1015
4.0839
4.0662
4.0489
4.0311
4.0134
3.5702
3.5530
3.5357
3.5179
3.4270
2.8848
2.8410
2.5101
2.3184
2.2745
2.2474
2.2064
1.9847
1.9435
1.1972
1.1799
1.1626
1.0020
0.5688

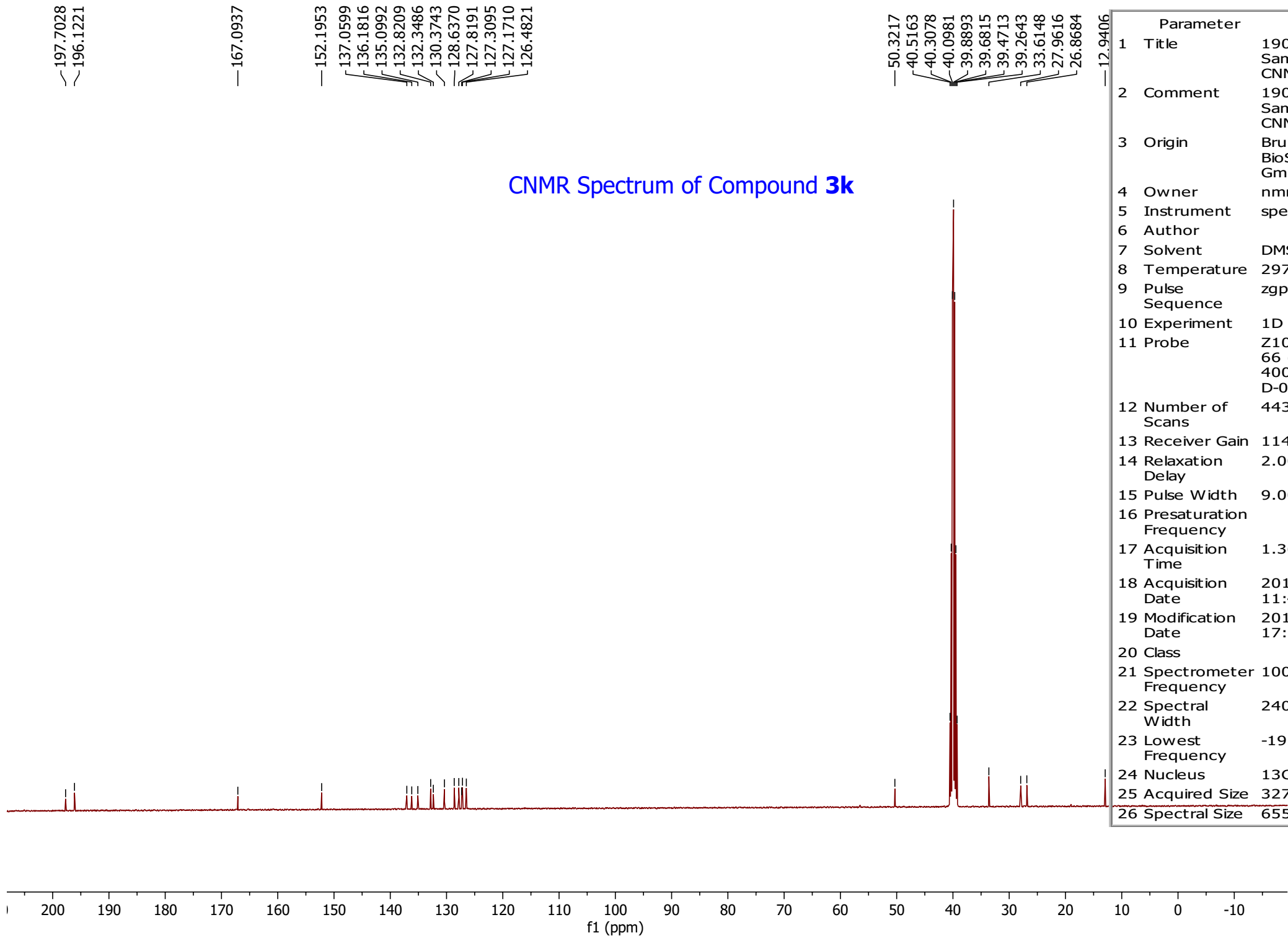
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3.00
1.00

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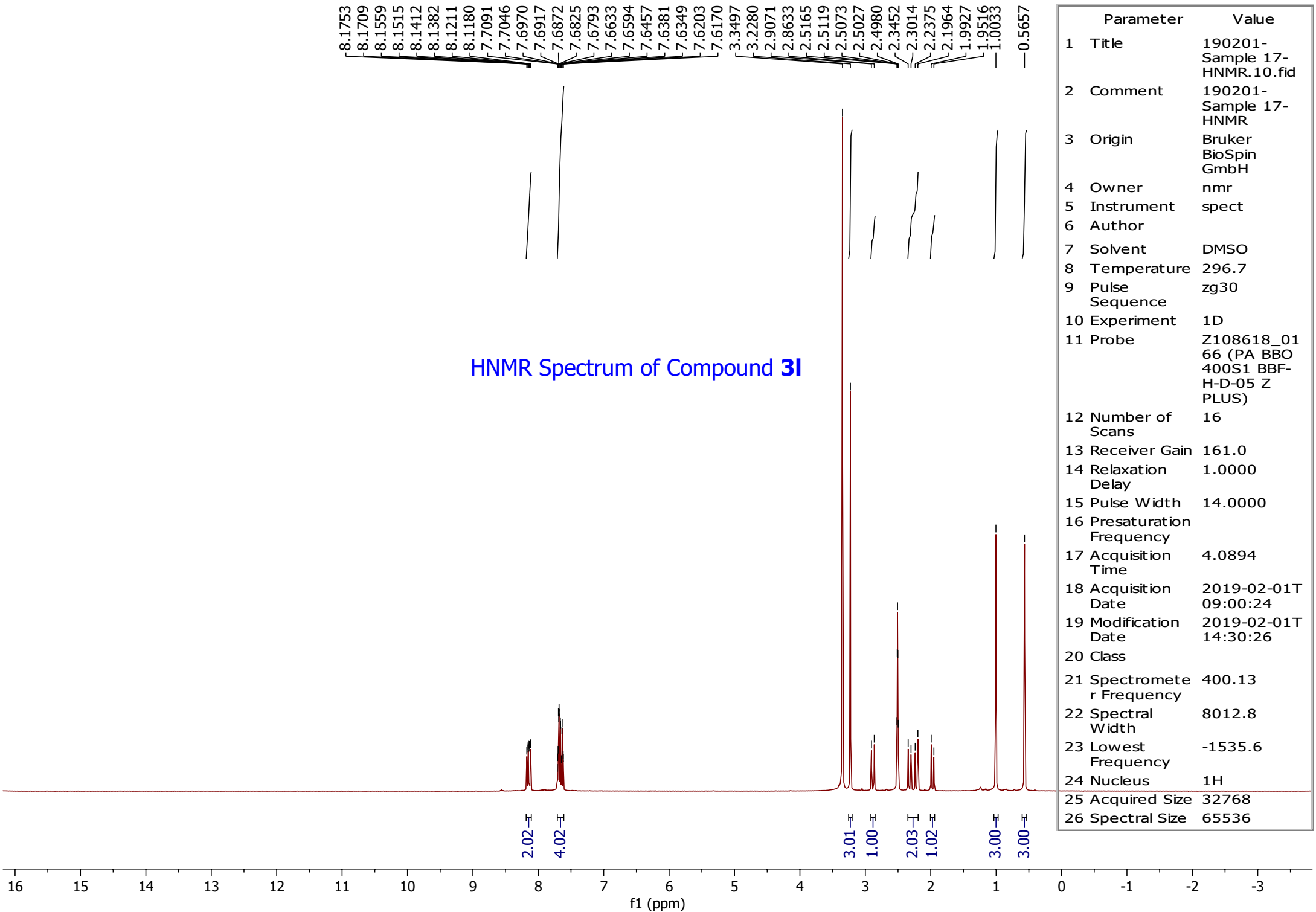
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1.01

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2.93

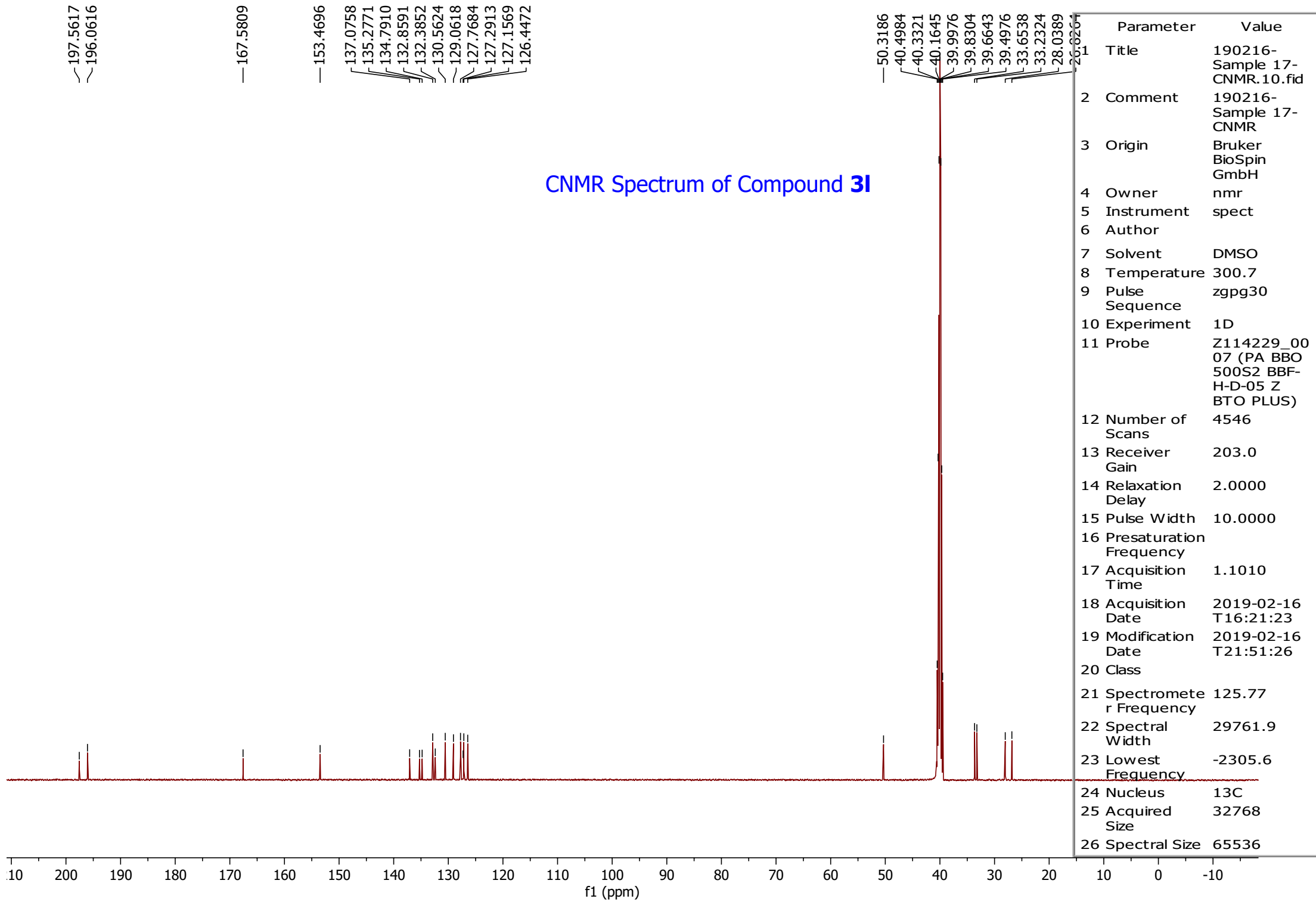
f1 (ppm)



Parameter	Value
1 Title	190203-Sample 1-CNMR.10.fid
2 Comment	190203-Sample 1-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	297.8
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4431
13 Receiver Gain	114.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-03T 11:43:44
19 Modification Date	2019-02-03T 17:13:46
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536



Parameter	Value
1 Title	190201-Sample 17-HNMR.10.fid
2 Comment	190201-Sample 17-HNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.7
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	161.0
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-02-01T 09:00:24
19 Modification Date	2019-02-01T 14:30:26
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536



197.5617
196.0616

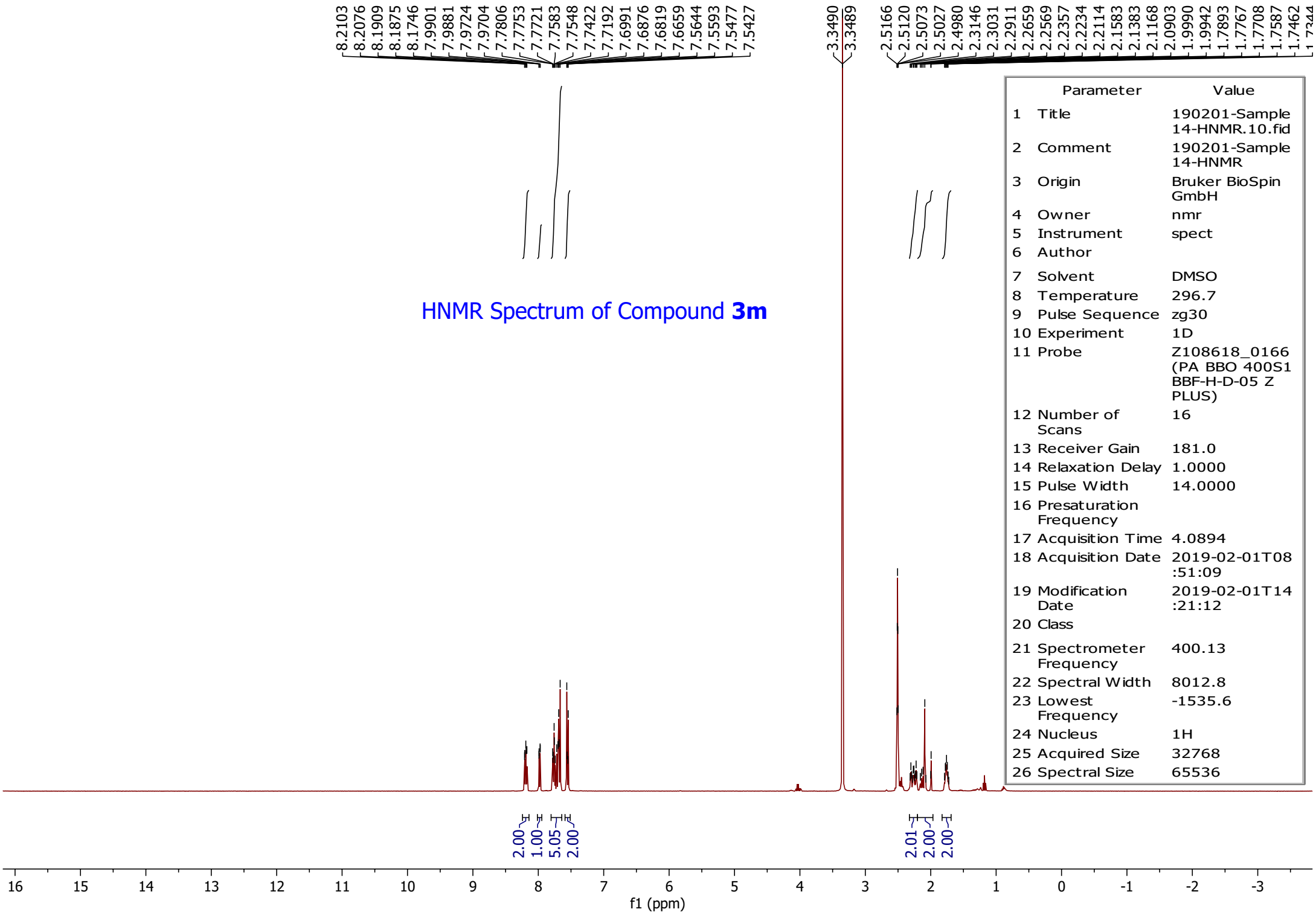
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153.4696

137.0758
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134.7910
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127.2913
127.1569
126.4472

50.3186
40.4984
40.3321
40.1645
39.9976
39.8304
39.6643
39.4976
33.6538
33.2324
28.0389
26.6204

f1 (ppm)



HNMR Spectrum of Compound 3m

Parameter	Value
1 Title	190201-Sample 14-HNMR.10.fid
2 Comment	190201-Sample 14-HNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.7
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	181.0
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-02-01T08:51:09
19 Modification Date	2019-02-01T14:21:12
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536

8.2103
8.2076
8.1909
8.1875
8.1746
7.9901
7.9881
7.9724
7.9704
7.7806
7.7753
7.7721
7.7583
7.7548
7.7422
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7.6876
7.6819
7.6659
7.5644
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7.5427

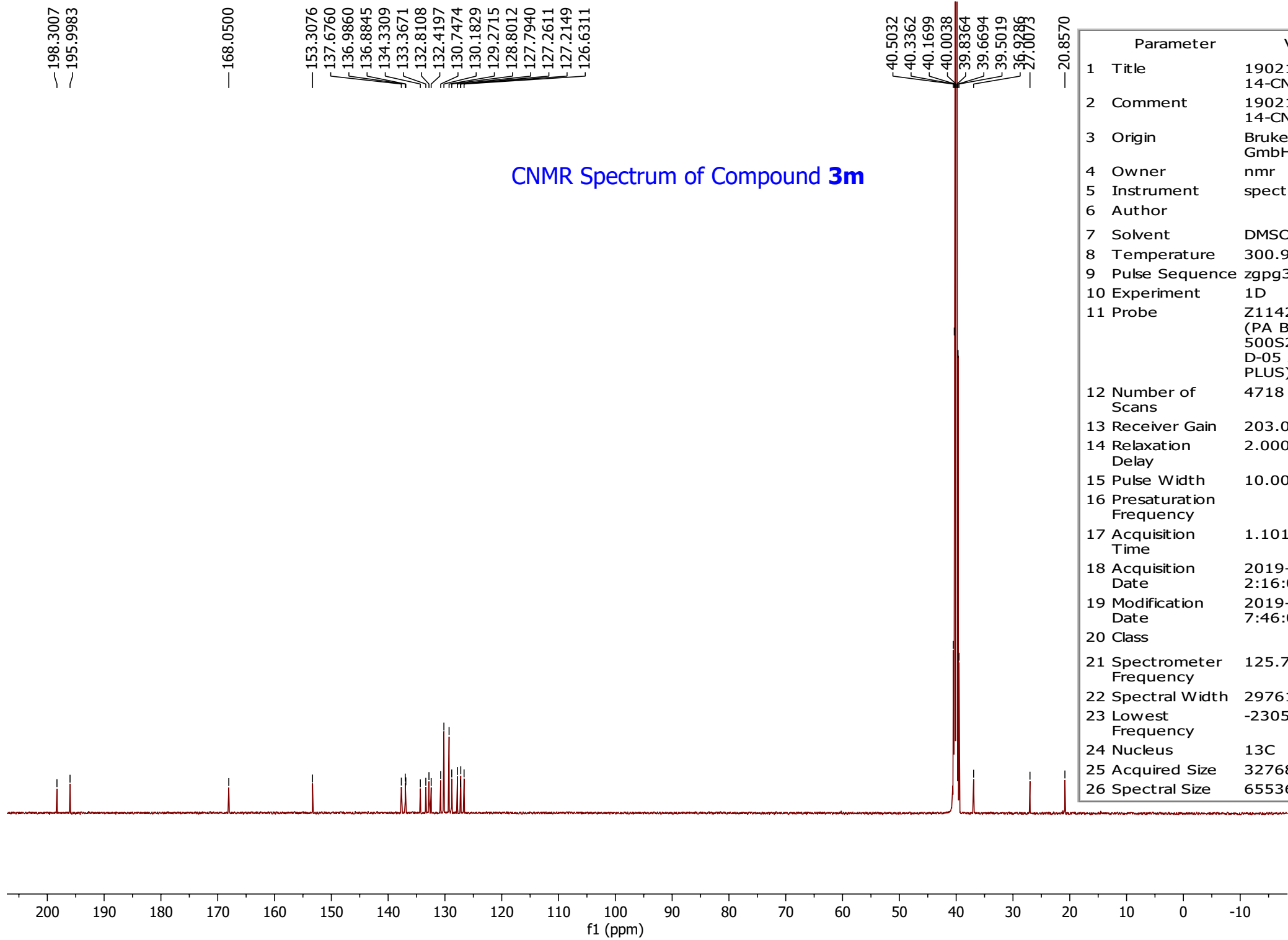
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2.3031
2.2911
2.2659
2.2569
2.2357
2.2234
2.2114
2.1583
2.1383
2.1168
2.0903
1.9990
1.9942
1.7893
1.7767
1.7708
1.7587
1.7462
1.7344

2.00
1.00
5.05
2.00

2.01
2.00
2.00

16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

f1 (ppm)



198.3007
195.9983

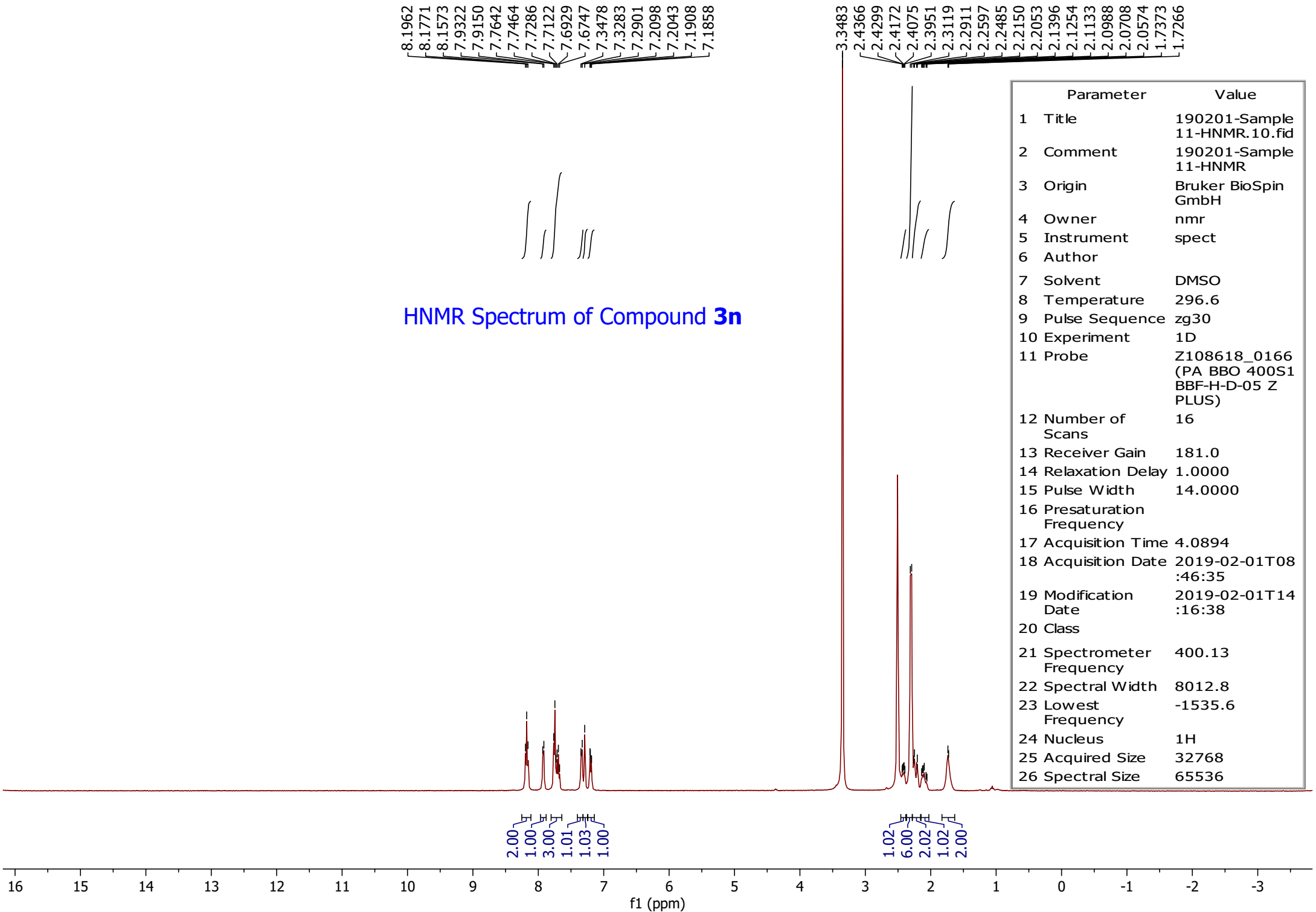
168.0500

153.3076
137.6760
136.9860
136.8845
134.3309
133.3671
132.8108
132.4197
130.7474
130.1829
129.2715
128.8012
127.7940
127.2611
127.2149
126.6311

40.5032
40.3362
40.1699
40.0038
39.8364
39.6694
39.5019
36.9286
27.0073

20.8570

f1 (ppm)



8.1962
8.1771
8.1573
7.9322
7.9150
7.7642
7.7464
7.7286
7.7122
7.6929
7.6747
7.3478
7.3283
7.2901
7.2098
7.2043
7.1908
7.1858

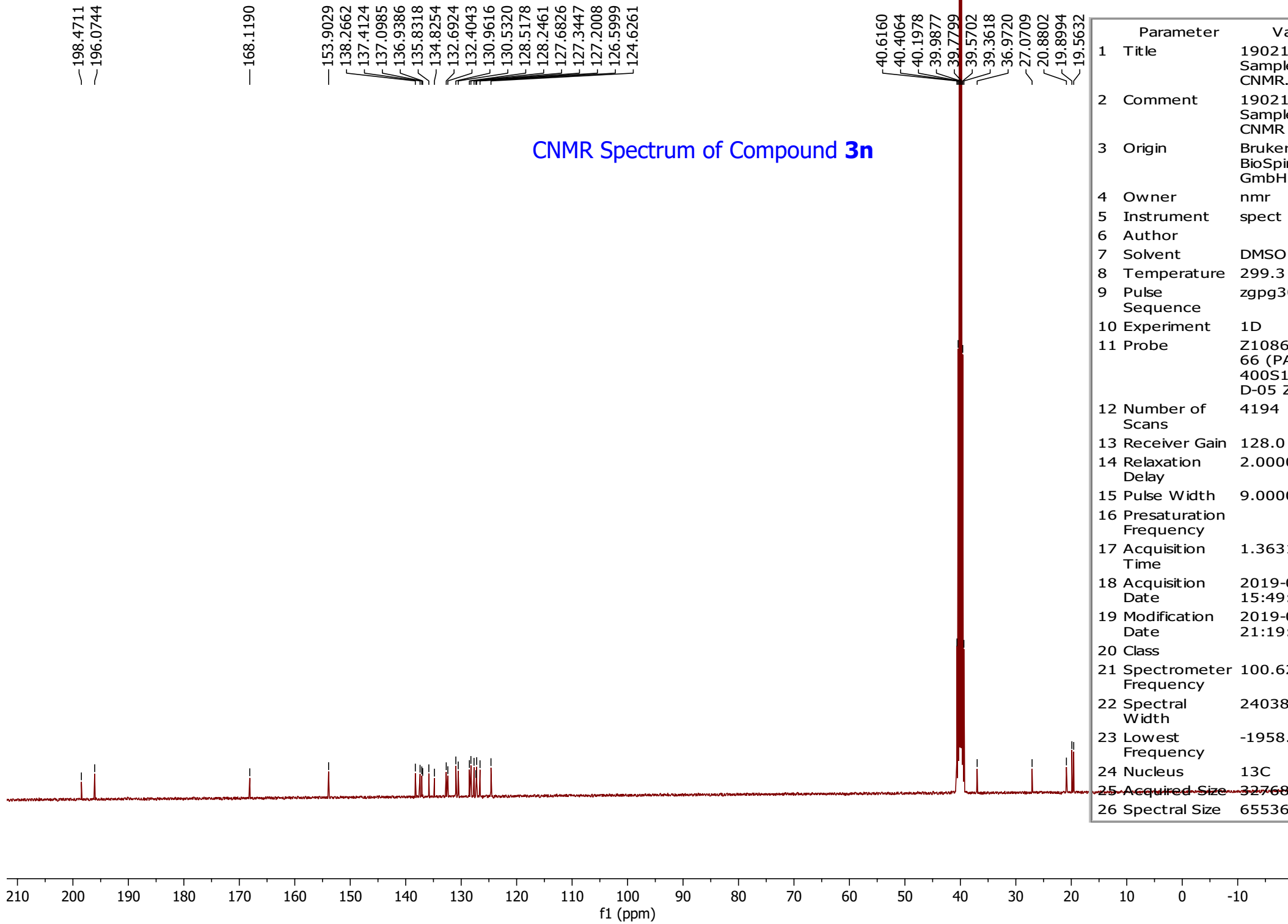
3.3483
2.4366
2.4299
2.4172
2.4075
2.3951
2.3119
2.2911
2.2597
2.2485
2.2150
2.2053
2.1396
2.1254
2.1133
2.0988
2.0708
2.0574
1.7373
1.7266

2.00
1.00
3.00
1.01
1.03
1.00

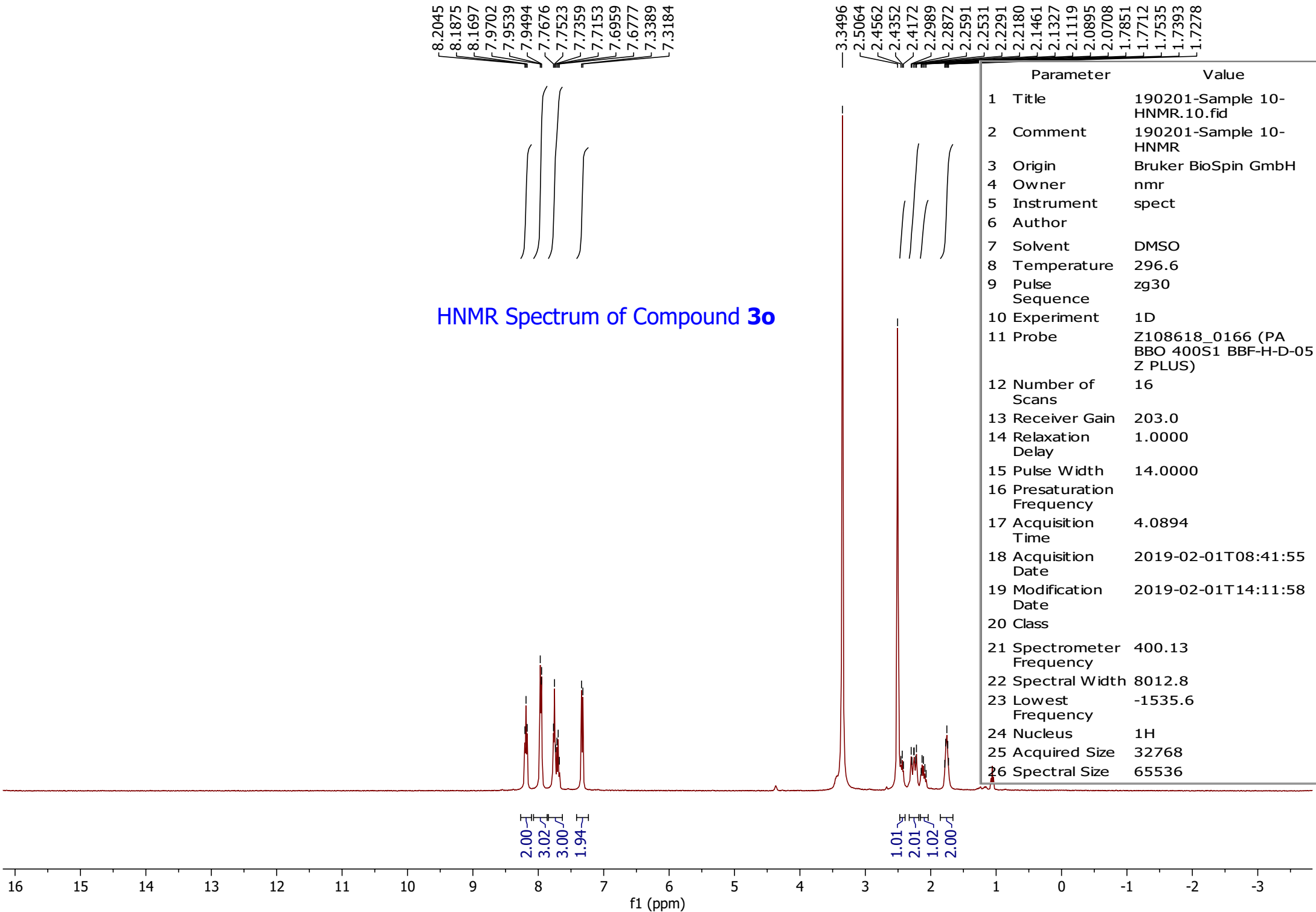
1.02
6.00
2.02
1.02
2.00

16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

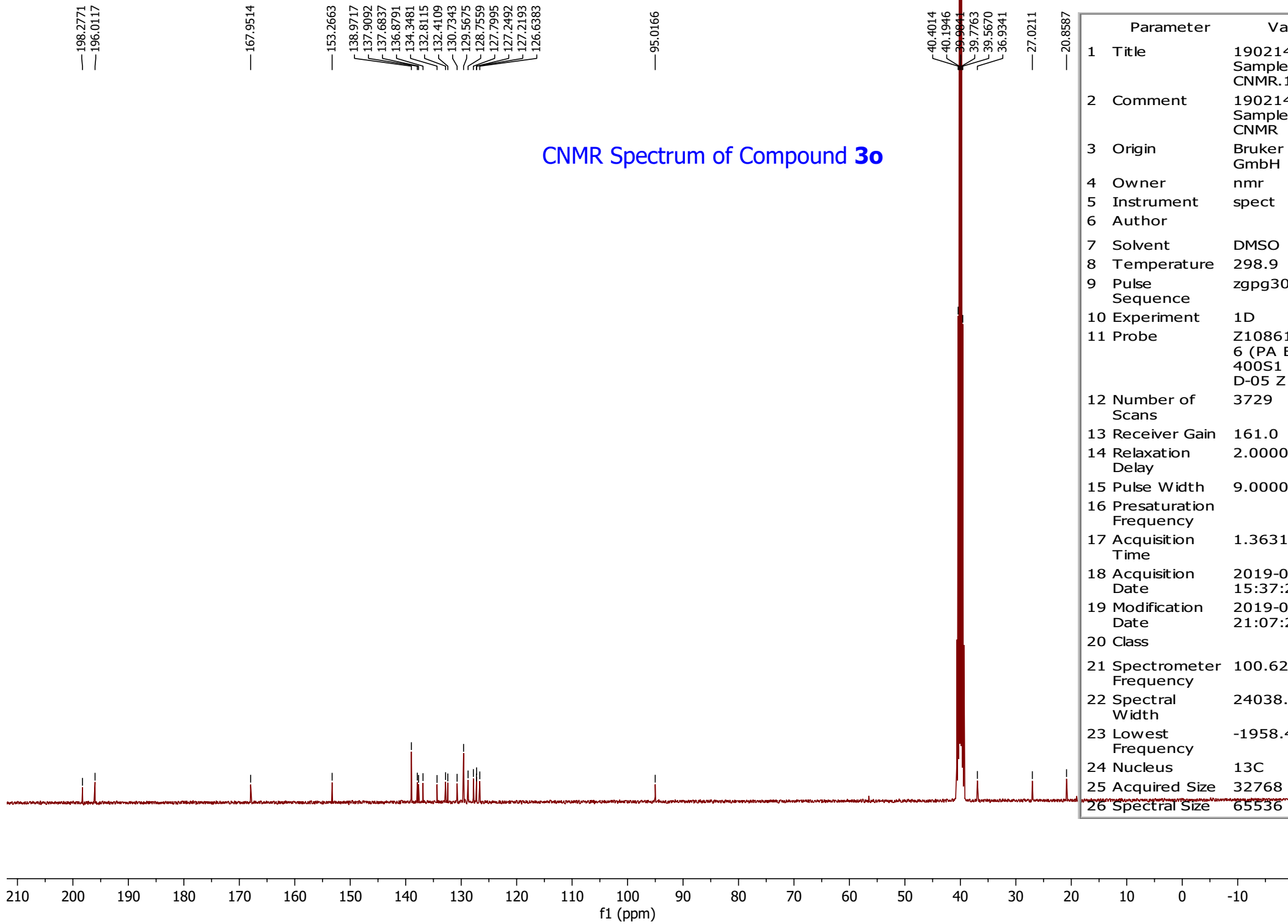
f1 (ppm)



Parameter	Value
1 Title	190215-Sample 11-CNMR.10.fid
2 Comment	190215-Sample 11-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	299.3
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	4194
13 Receiver Gain	128.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-15T15:49:17
19 Modification Date	2019-02-15T21:19:20
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536



Parameter	Value
1 Title	190201-Sample 10-HNMR.10.fid
2 Comment	190201-Sample 10-HNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.6
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	203.0
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-02-01T08:41:55
19 Modification Date	2019-02-01T14:11:58
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536



— 198.2771
— 196.0117

— 167.9514

— 153.2663

138.9717
137.9092
137.6837
136.8791
134.3481
132.8115
132.4109
130.7343
129.5675
128.7559
127.7995
127.2492
127.2193
126.6383

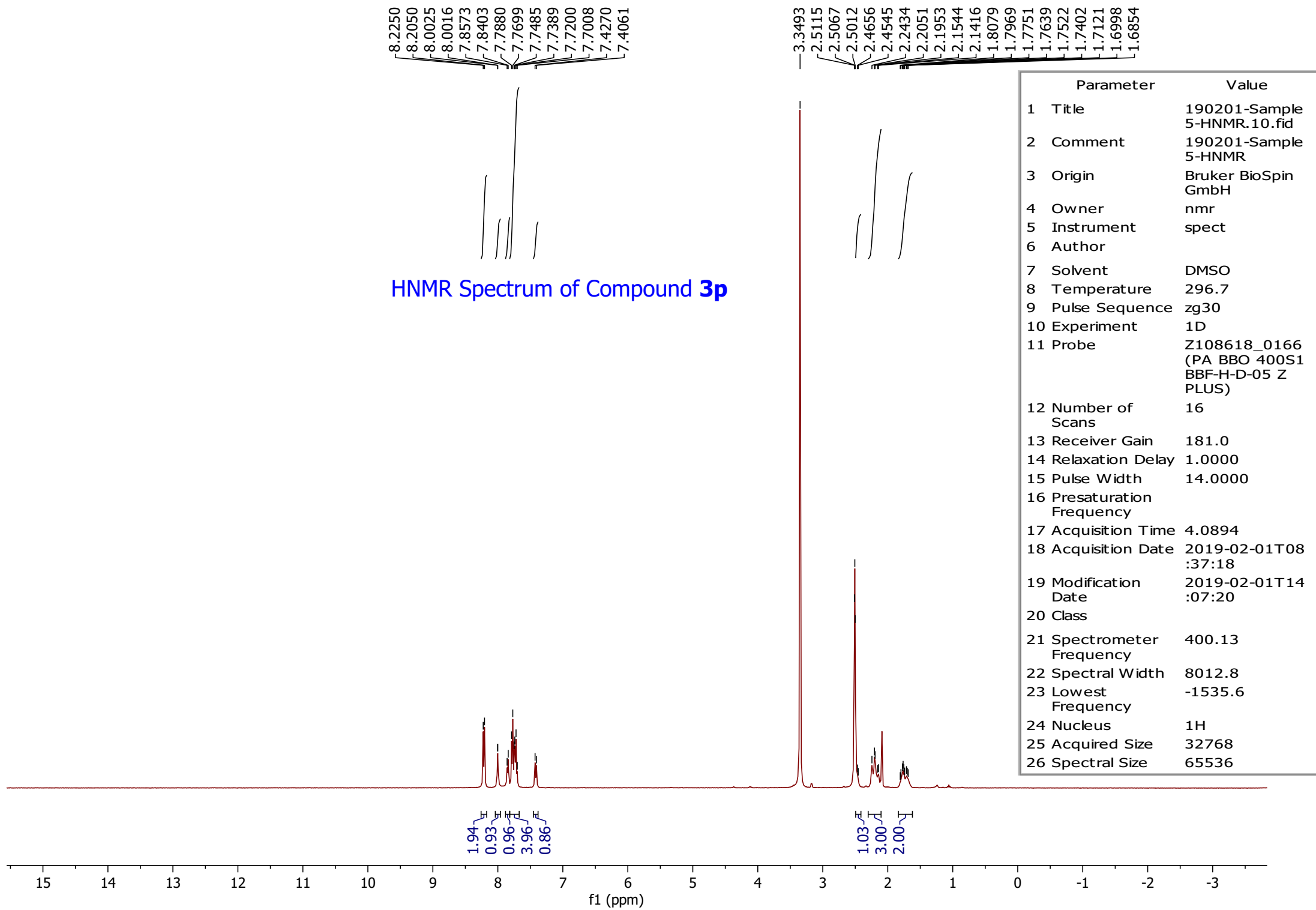
— 95.0166

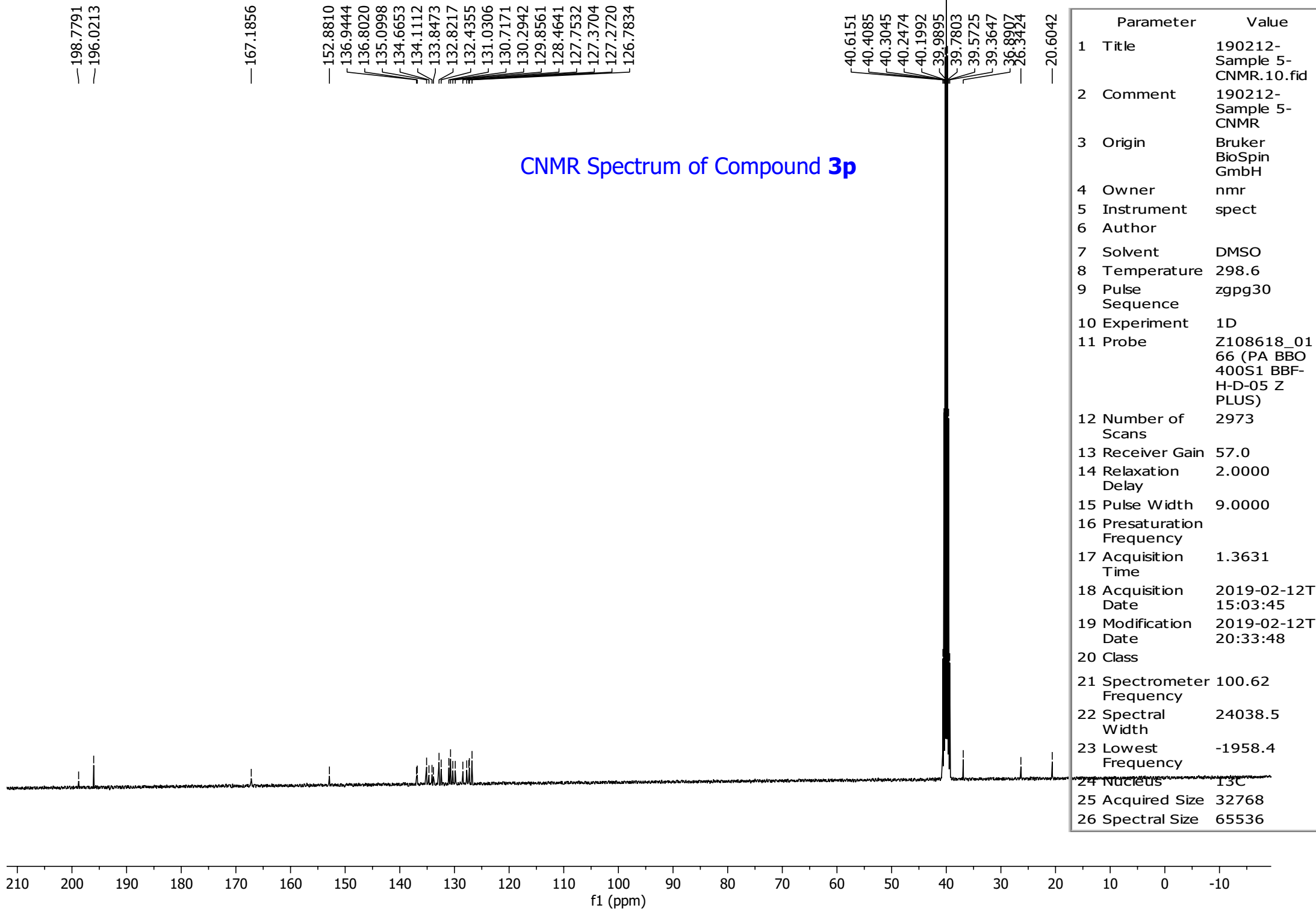
40.4014
40.1946
39.9641
39.7763
39.5670
36.9341

— 27.0211

— 20.8587

Parameter	Value
1 Title	190214-Sample 10-CNMR.10.fid
2 Comment	190214-Sample 10-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.9
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	3729
13 Receiver Gain	161.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-14T 15:37:26
19 Modification Date	2019-02-14T 21:07:28
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	¹³ C
25 Acquired Size	32768
26 Spectral Size	65536





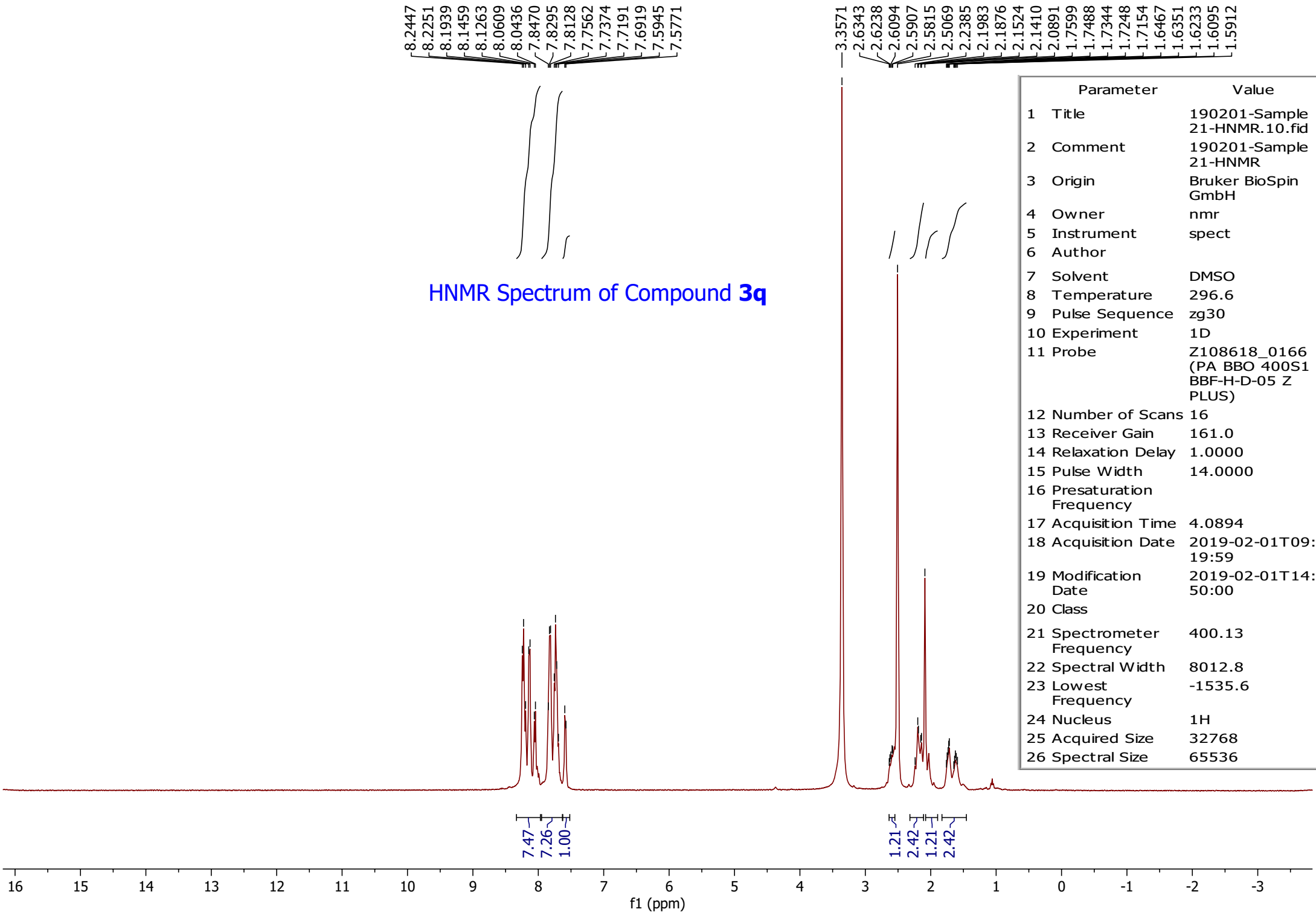
—198.7791
—196.0213

—167.1856

—152.8810
—136.9444
—136.8020
—135.0998
—134.6653
—134.1112
—133.8473
—132.8217
—132.4355
—131.0306
—130.7171
—130.2942
—129.8561
—128.4641
—127.7532
—127.3704
—127.2720
—126.7834

—40.6151
—40.4085
—40.3045
—40.2474
—40.1992
—39.9895
—39.7803
—39.5725
—39.3647
—36.8907
—26.3424
—20.6042

f1 (ppm)



HNMR Spectrum of Compound **3q**

Parameter	Value
1 Title	190201-Sample 21-HNMR.10.fid
2 Comment	190201-Sample 21-HNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	296.6
9 Pulse Sequence	zg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	16
13 Receiver Gain	161.0
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Presaturation Frequency	
17 Acquisition Time	4.0894
18 Acquisition Date	2019-02-01T09:19:59
19 Modification Date	2019-02-01T14:50:00
20 Class	
21 Spectrometer Frequency	400.13
22 Spectral Width	8012.8
23 Lowest Frequency	-1535.6
24 Nucleus	1H
25 Acquired Size	32768
26 Spectral Size	65536

8.2447
8.2251
8.1939
8.1459
8.1263
8.0609
8.0436
7.8470
7.8295
7.8128
7.7562
7.7374
7.7191
7.6919
7.5945
7.5771

3.3571
2.6343
2.6238
2.6094
2.5907
2.5815
2.5069
2.2385
2.1983
2.1876
2.1524
2.1410
2.0891
1.7599
1.7488
1.7344
1.7248
1.7154
1.6467
1.6351
1.6233
1.6095
1.5912

7.47
7.26
1.00

1.21
2.42
1.21
2.42

16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

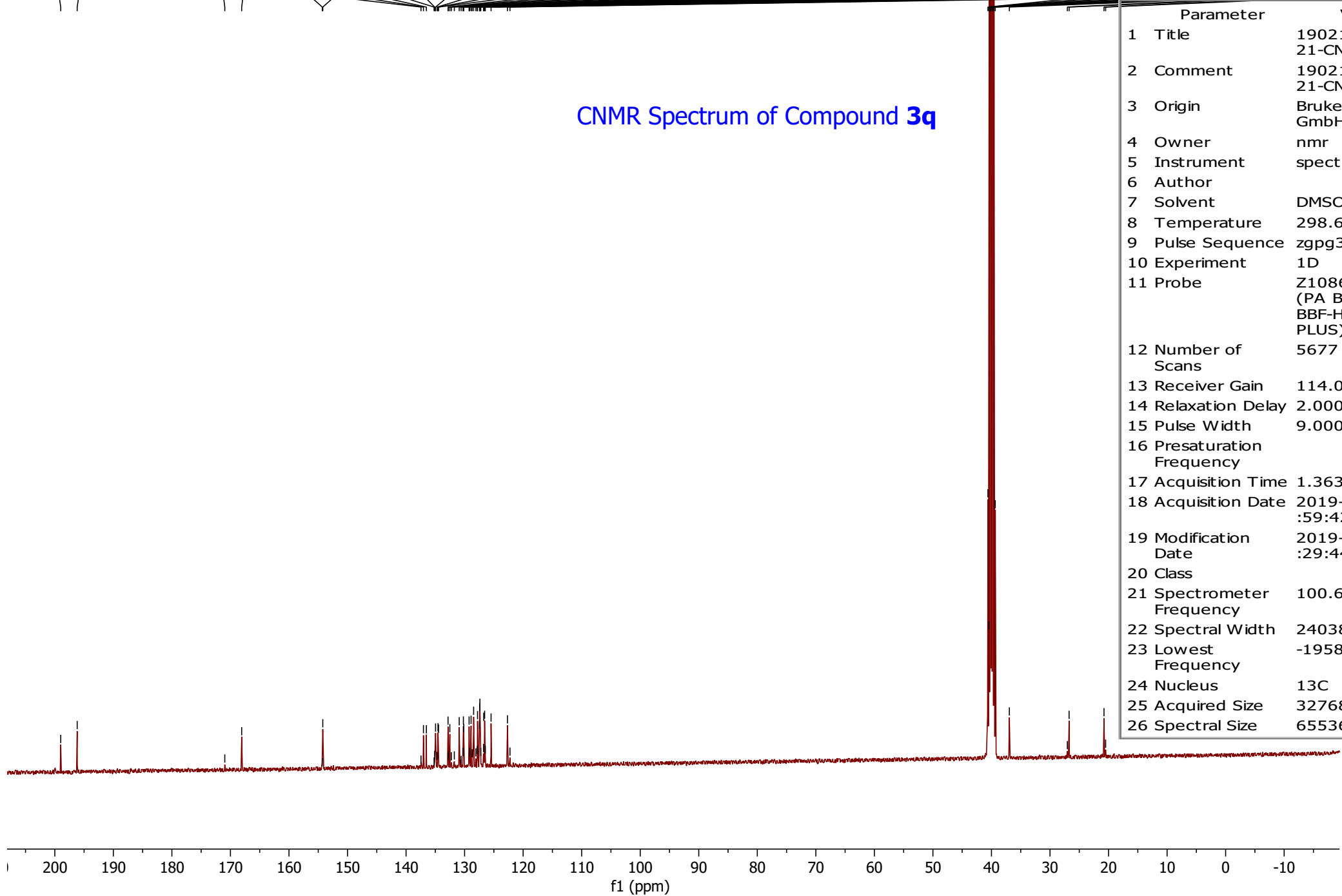
f1 (ppm)

— 198.986
— 196.176

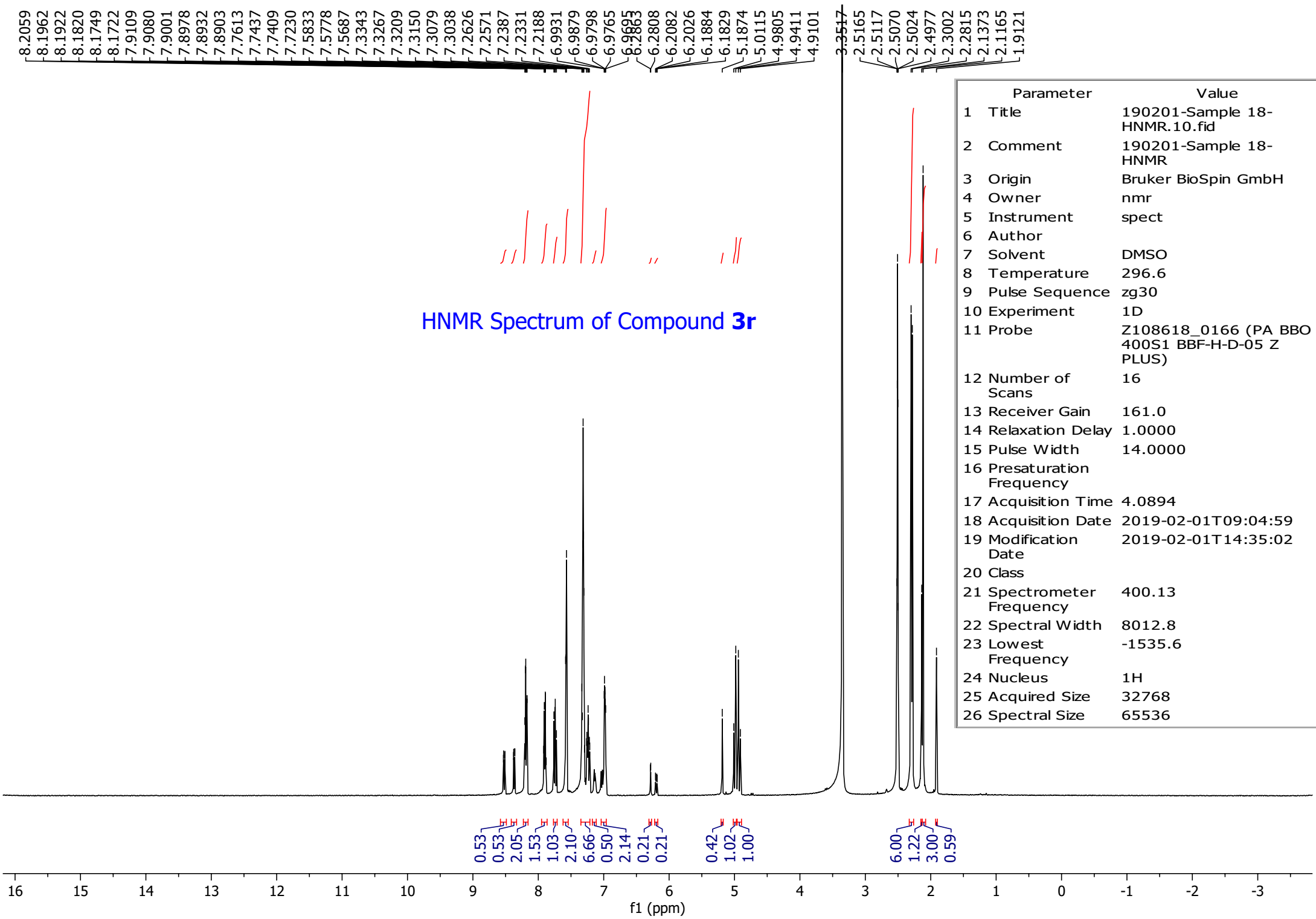
— 170.962
— 168.076

154.307
154.223
137.445
137.028
136.528
135.211
135.103
134.959
134.691
134.543
134.450
132.829
132.768
132.520
132.261
131.754
130.918
130.593
130.266
130.203
130.137
129.231
129.134
128.890
128.702
128.454
128.072
127.831
127.786
127.444
127.408
127.386
127.237
126.770
126.731
126.577
126.461
125.478
122.676
122.248
40.6038
40.4451
40.3955
40.1876
-39.9772
-39.7690
-39.5612
-39.3513
-36.9436
-27.0365
-26.7383
-20.7751
-20.4950

CNMR Spectrum of Compound 3q



Parameter	Value
1 Title	190217-Sample 21-CNMR. 10.fid
2 Comment	190217-Sample 21-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.6
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	5677
13 Receiver Gain	114.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-17T17:59:42
19 Modification Date	2019-02-17T23:29:44
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536



8.2059
8.1962
8.1922
8.1820
8.1749
8.1722
7.9109
7.9080
7.9001
7.8978
7.8932
7.8903
7.7613
7.7437
7.7409
7.7230
7.5833
7.5778
7.5687
7.3343
7.3267
7.3209
7.3150
7.3079
7.3038
7.2626
7.2571
7.2387
7.2331
7.2188
6.9931
6.9879
6.9798
6.9765
6.9695
6.9663
6.2808
6.2082
6.2026
6.1884
6.1829
5.1874
5.0115
4.9805
4.9411
4.9101

3.5517
2.5165
2.5117
2.5070
2.5024
2.4977
2.3002
2.2815
2.1373
2.1165
1.9121

Red integration lines and values for the aromatic region peaks.

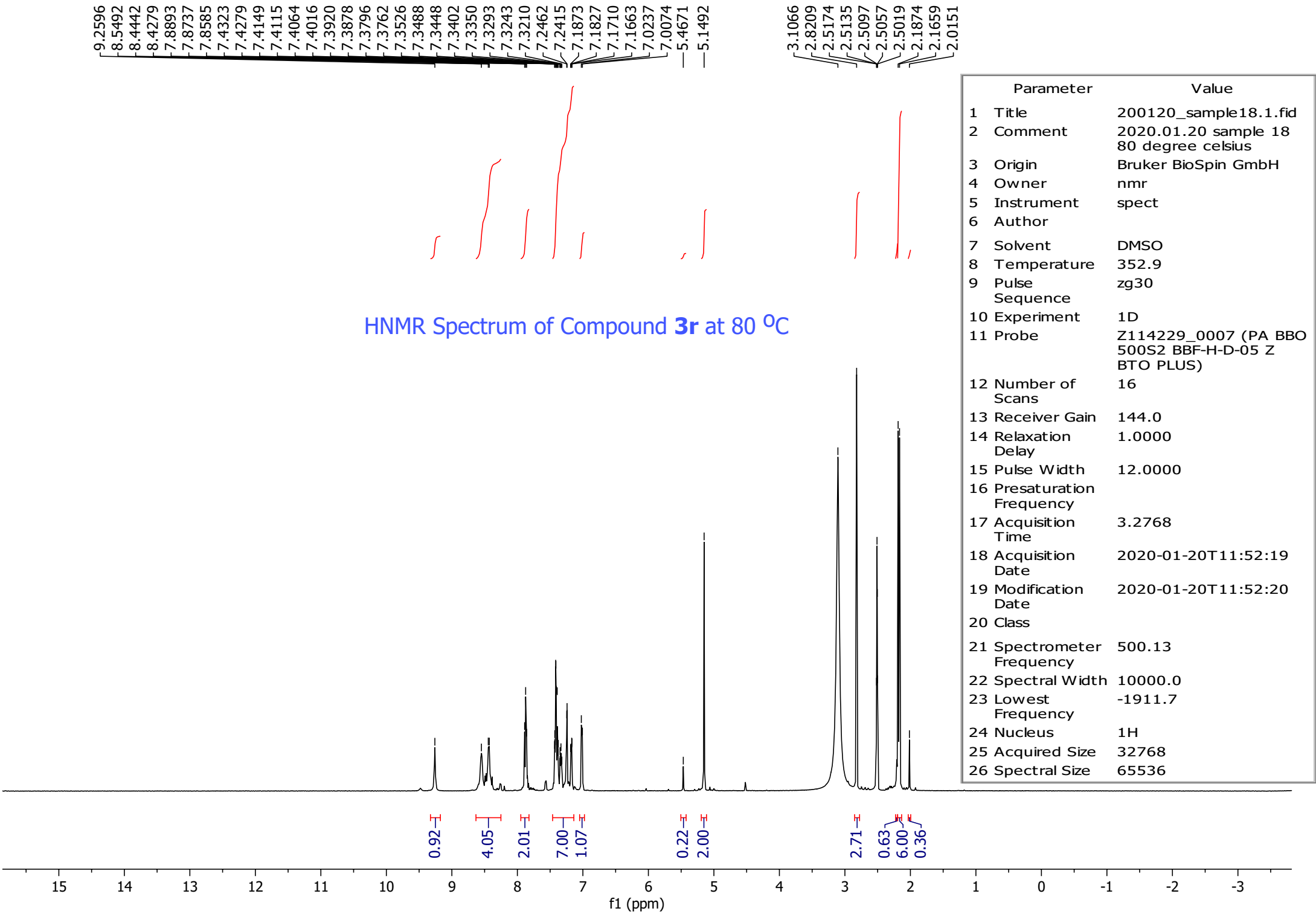
HNMR Spectrum of Compound 3r

Integration values for the aromatic region peaks: 0.53, 0.53, 2.05, 1.53, 1.03, 2.10, 6.66, 0.50, 2.14, 0.21, 0.21, 0.42, 1.02, 1.00

Integration values for the aliphatic region peaks: 6.00, 1.22, 3.00, 0.59

16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

f1 (ppm)



9.2596
8.5492
8.4442
8.4279
7.8893
7.8737
7.8585
7.4323
7.4279
7.4149
7.4115
7.4064
7.4016
7.3920
7.3878
7.3796
7.3762
7.3526
7.3488
7.3448
7.3402
7.3350
7.3293
7.3243
7.3210
7.2462
7.2415
7.1873
7.1827
7.1710
7.1663
7.0237
7.0074
—5.4671
—5.1492

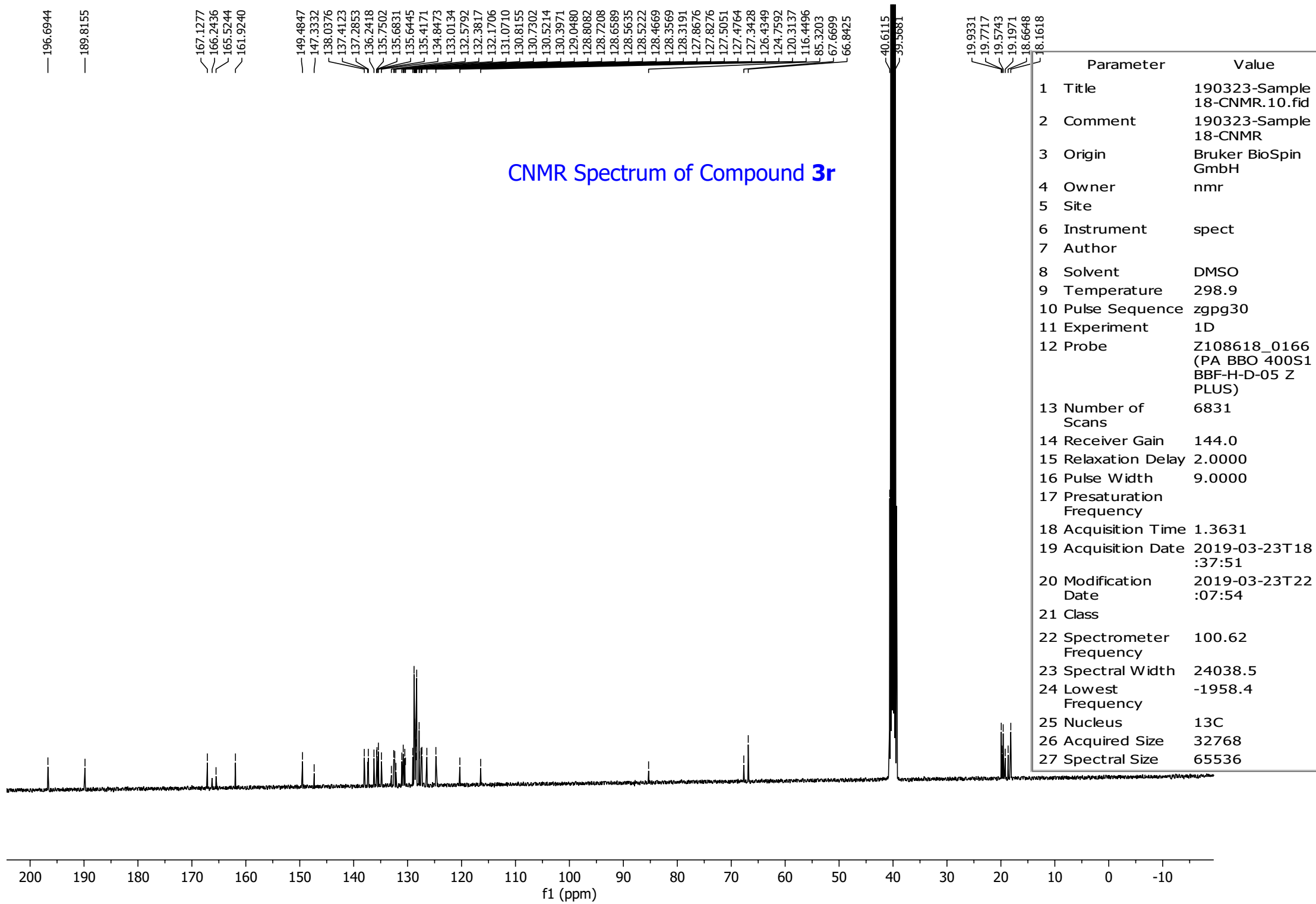
3.1066
2.8209
2.5174
2.5135
2.5097
2.5057
2.5019
2.1874
2.1659
2.0151



0.92
4.05
2.01
7.00
1.07
0.22
2.00
2.71
0.63
6.00
0.36

15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

f1 (ppm)



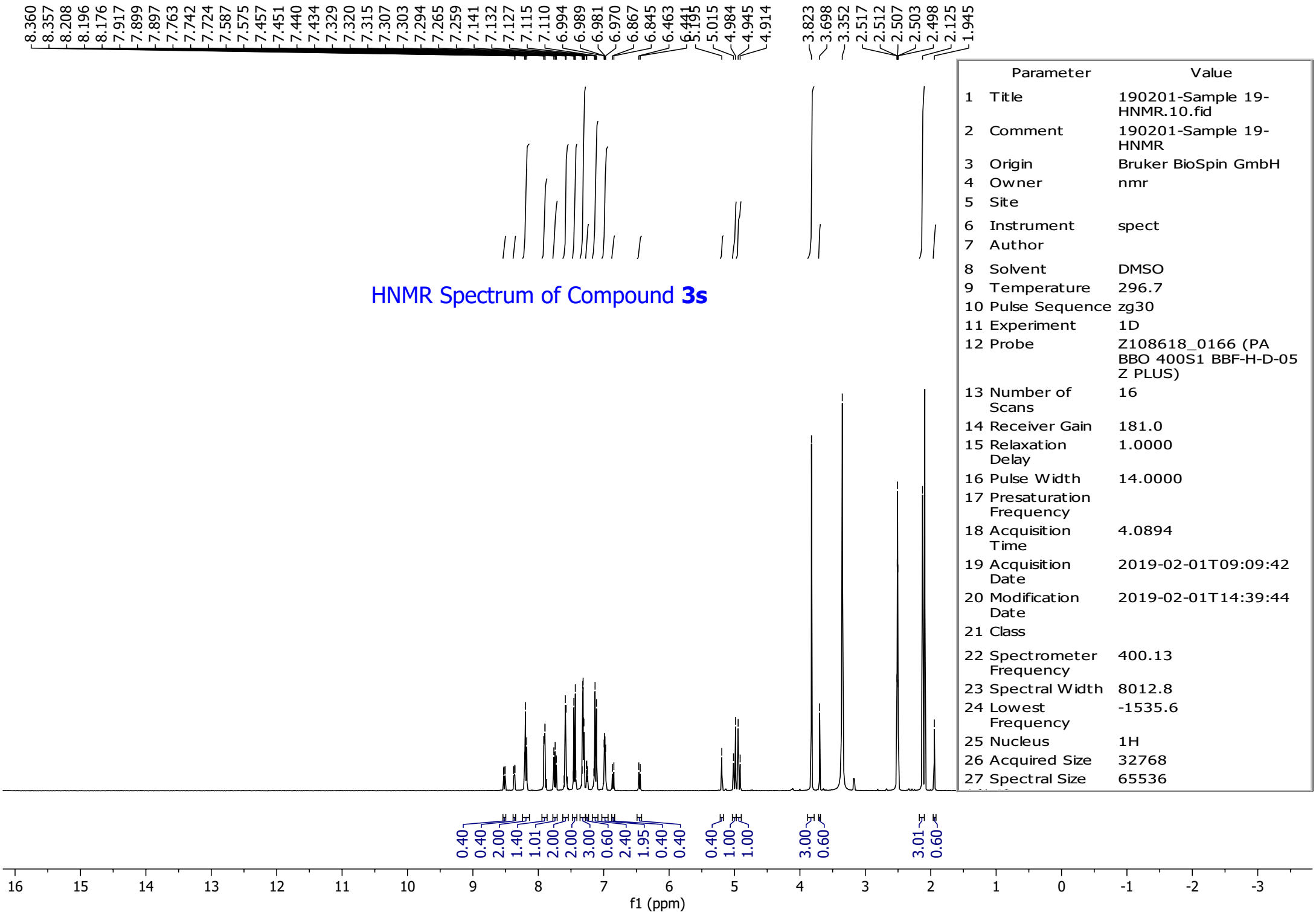
196.6944
189.8155
167.1277
166.2436
165.5244
161.9240
149.4847
147.3332
138.0376
137.4123
137.2853
136.2418
135.7502
135.6831
135.6445
135.4171
134.8473
133.0134
132.5792
132.3817
132.1706
131.0710
130.8155
130.7302
130.5214
130.3971
129.0480
128.8082
128.7208
128.6589
128.5635
128.5222
128.4669
128.3569
128.3191
127.8676
127.8276
127.5051
127.4764
127.3428
126.4349
124.7592
120.3137
116.4496
85.3203
67.6699
66.8425

40.6115
39.5681

19.9331
19.7717
19.5743
19.1971
18.6648
18.1618

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)



8.360
8.357
8.208
8.196
8.176
7.917
7.899
7.897
7.763
7.742
7.724
7.587
7.575
7.457
7.451
7.440
7.434
7.329
7.320
7.315
7.307
7.303
7.294
7.265
7.259
7.141
7.132
7.127
7.115
7.110
6.994
6.989
6.981
6.970
6.867
6.845
6.463
6.441
5.195
5.015
4.984
4.945
4.914

3.823
3.698
3.352
2.517
2.512
2.507
2.503
2.498
2.125
1.945

0.40
0.40
2.00
1.40
1.01
2.00
2.00
3.00
0.60
2.40
1.95
0.40
0.40

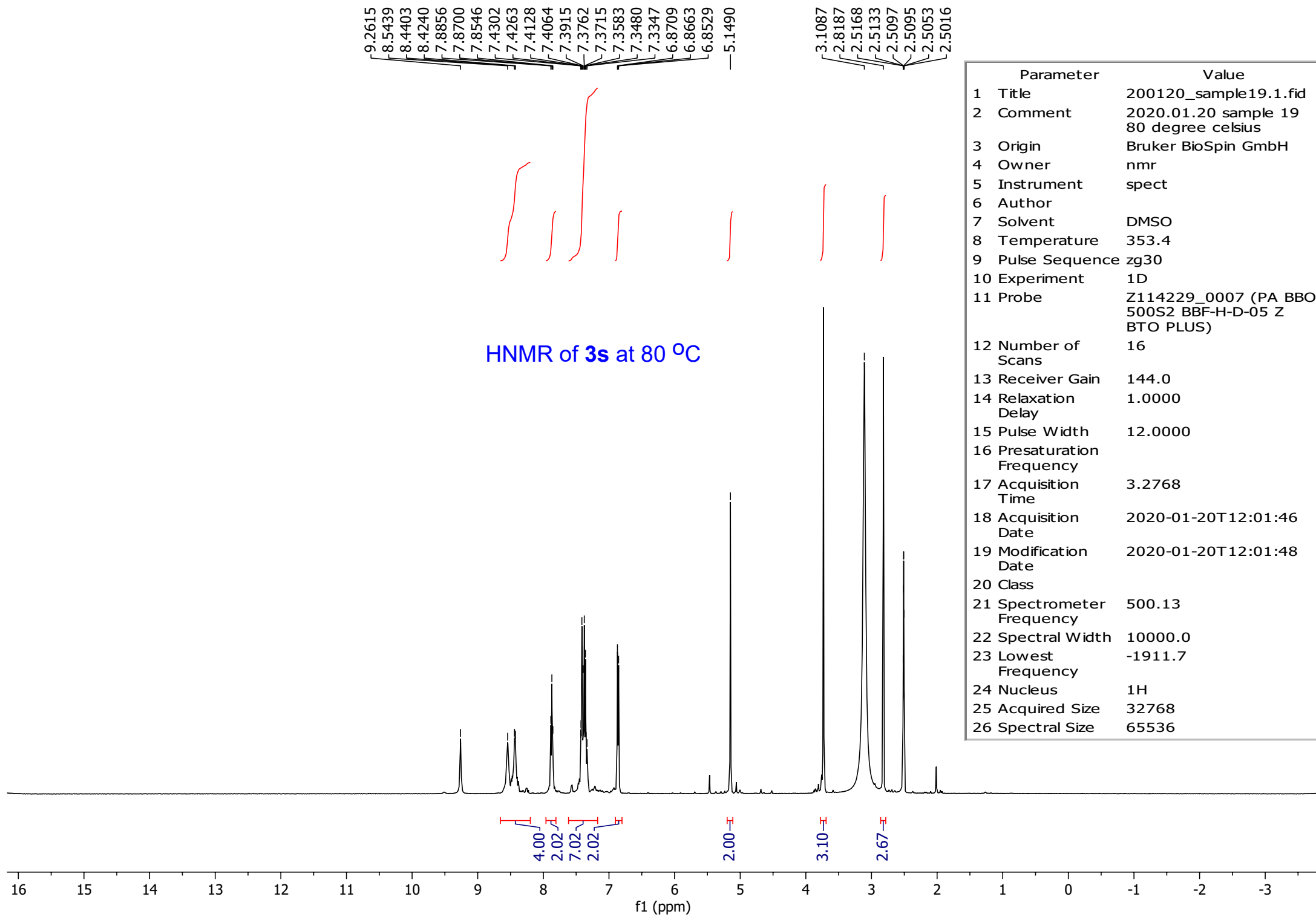
0.40
1.00
1.00

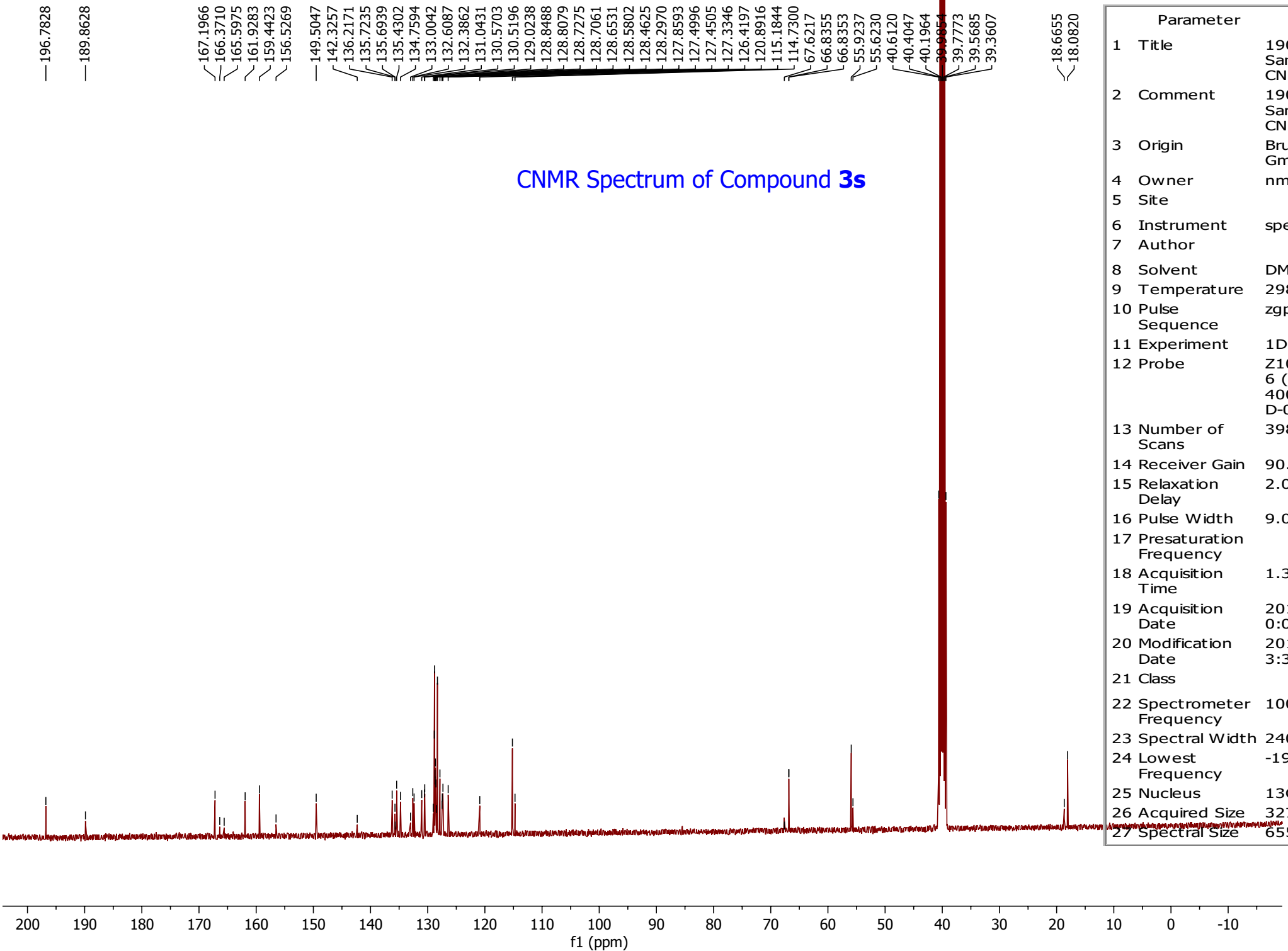
3.00
0.60

3.01
0.60

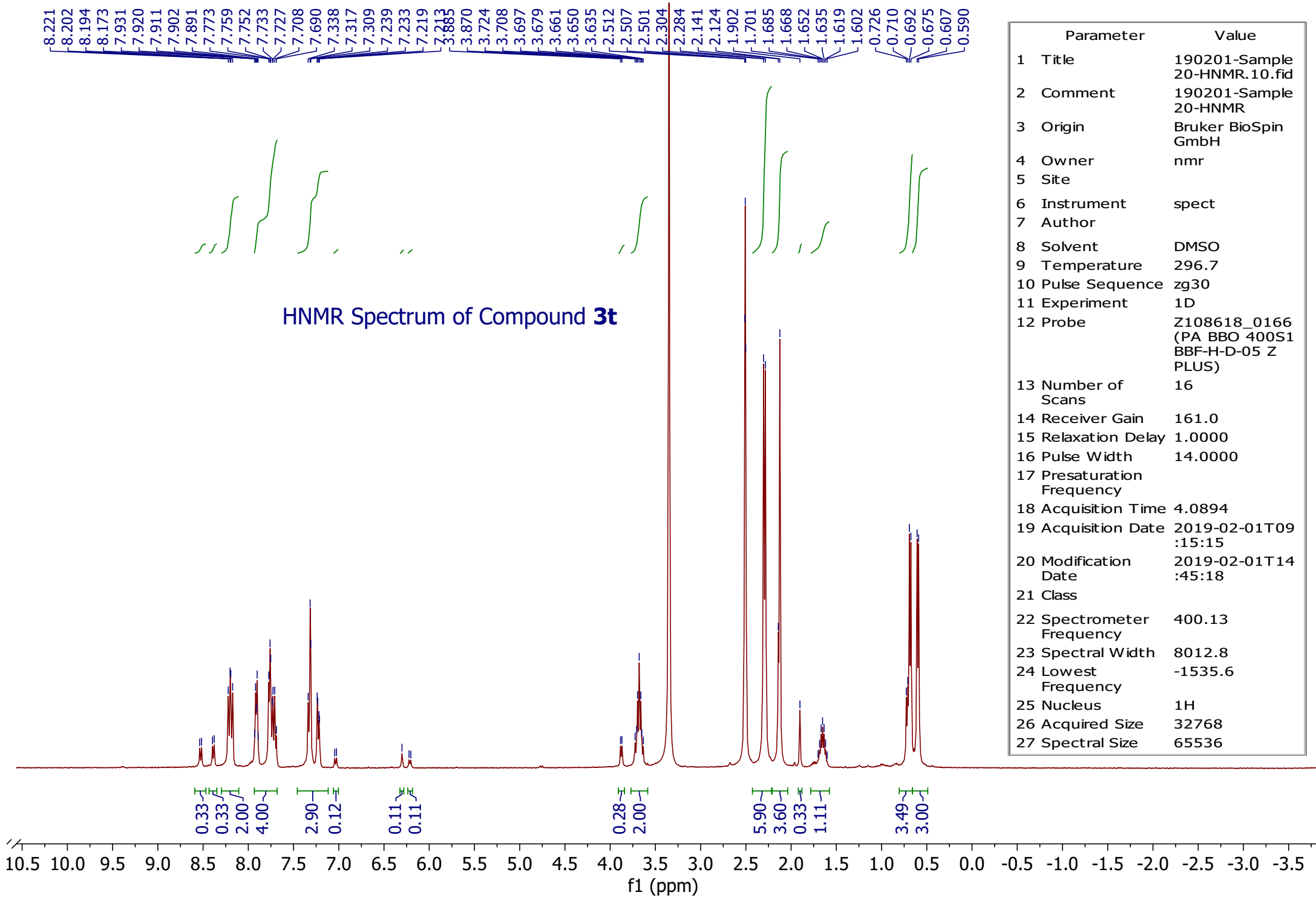
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

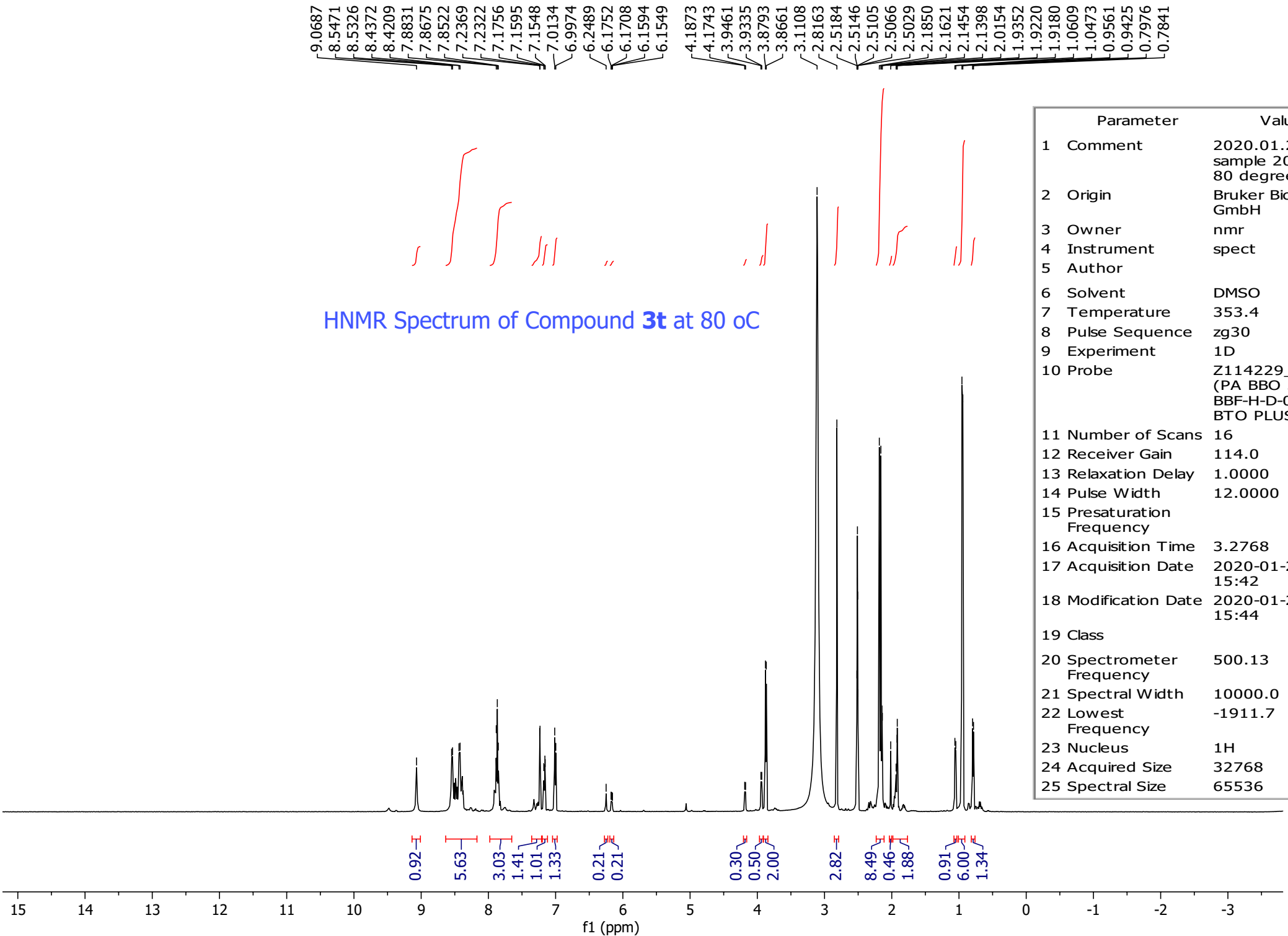
f1 (ppm)



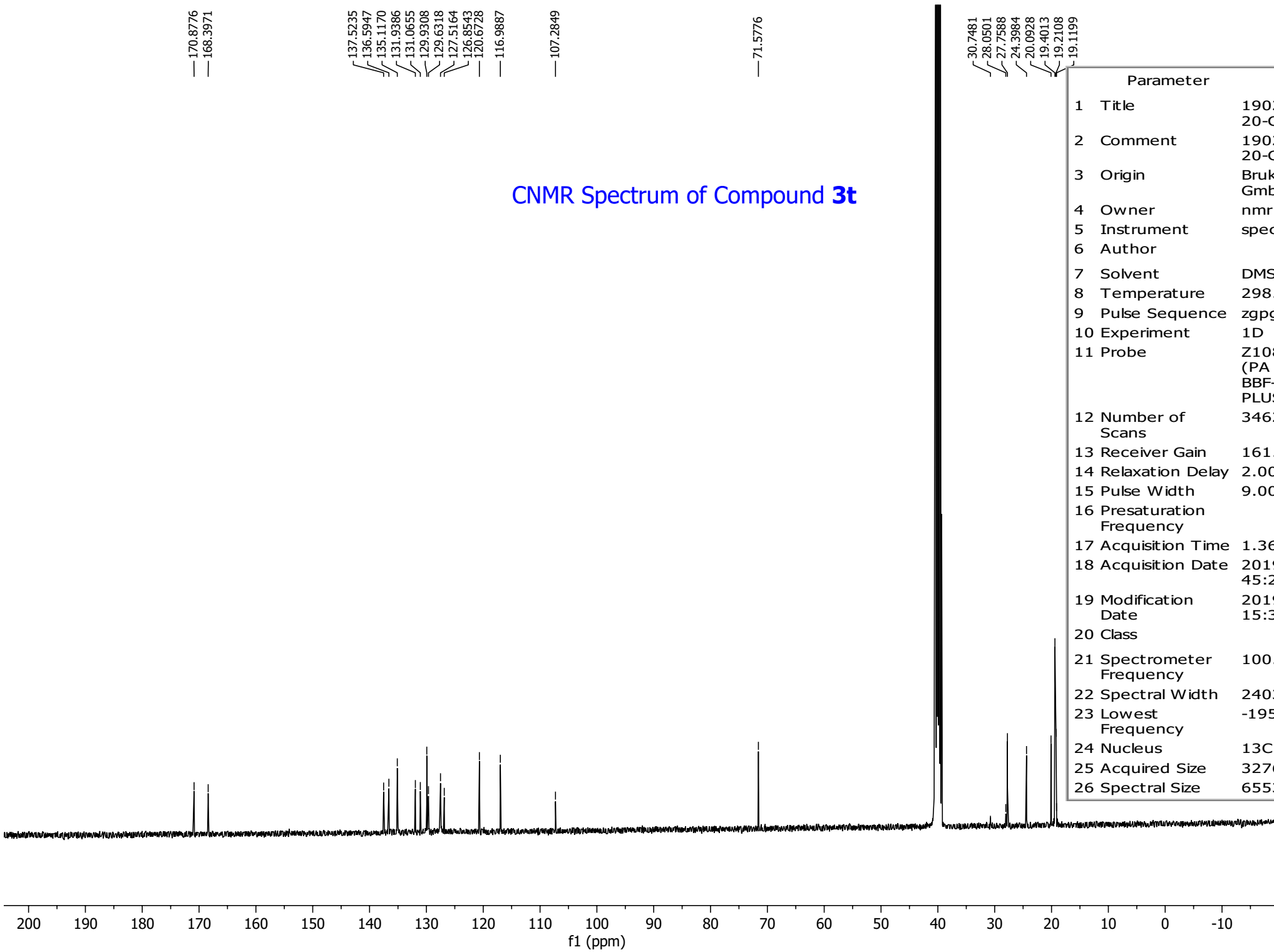


Parameter	Value
1 Title	190322-Sample 19-CNMR.10.fid
2 Comment	190322-Sample 19-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	DMSO
9 Temperature	298.9
10 Pulse Sequence	zgpg30
11 Experiment	1D
12 Probe	Z108618_016 6 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
13 Number of Scans	3988
14 Receiver Gain	90.5
15 Relaxation Delay	2.0000
16 Pulse Width	9.0000
17 Presaturation Frequency	
18 Acquisition Time	1.3631
19 Acquisition Date	2019-03-22T10:07:18
20 Modification Date	2019-03-22T13:37:20
21 Class	
22 Spectrometer Frequency	100.62
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	¹³ C
26 Acquired Size	32768
27 Spectral Size	65536





Parameter	Value
1 Comment	2020.01.20 sample 20 80 degree celsius
2 Origin	Bruker BioSpin GmbH
3 Owner	nmr
4 Instrument	spect
5 Author	
6 Solvent	DMSO
7 Temperature	353.4
8 Pulse Sequence	zg30
9 Experiment	1D
10 Probe	Z114229_0007 (PA BBO 500S2 BBF-H-D-05 Z BTO PLUS)
11 Number of Scans	16
12 Receiver Gain	114.0
13 Relaxation Delay	1.0000
14 Pulse Width	12.0000
15 Presaturation Frequency	
16 Acquisition Time	3.2768
17 Acquisition Date	2020-01-20T12:15:42
18 Modification Date	2020-01-20T12:15:44
19 Class	
20 Spectrometer Frequency	500.13
21 Spectral Width	10000.0
22 Lowest Frequency	-1911.7
23 Nucleus	1H
24 Acquired Size	32768
25 Spectral Size	65536



Parameter	Value
1 Title	190225-Sample 20-CNMR.10.fid
2 Comment	190225-Sample 20-CNMR
3 Origin	Bruker BioSpin GmbH
4 Owner	nmr
5 Instrument	spect
6 Author	
7 Solvent	DMSO
8 Temperature	298.9
9 Pulse Sequence	zgpg30
10 Experiment	1D
11 Probe	Z108618_0166 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
12 Number of Scans	3462
13 Receiver Gain	161.0
14 Relaxation Delay	2.0000
15 Pulse Width	9.0000
16 Presaturation Frequency	
17 Acquisition Time	1.3631
18 Acquisition Date	2019-02-25T14:45:28
19 Modification Date	2019-02-25T20:15:30
20 Class	
21 Spectrometer Frequency	100.62
22 Spectral Width	24038.5
23 Lowest Frequency	-1958.4
24 Nucleus	13C
25 Acquired Size	32768
26 Spectral Size	65536