Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Solid-phase synthesis of coralmycin A/epi-coralmycin A and desmethoxycoralmycin A

Paige M. E. Hawkins,[a,b] Dennis Y. Liu,[c] Roger G. Linington,[c] and Richard J. Payne*[a,b]

[a] School of Chemistry, The University of Sydney, Sydney, NSW, 2006, Australia.
[b] Australian Research Council Centre of Excellence for Innovations in Peptide and Protein Science, The University of Sydney, NSW 2006, Australia
[c] Department of Chemistry, Simon Fraser University, Burnaby, BC V5A 1S6, Canada.

Table of Contents

General Methods and Materials

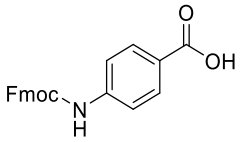
All reactions were carried out under an argon atmosphere and at room temperature (22 °C) unless the reaction was performed under aqueous conditions or unless otherwise specified. The reactions carried out at 0 °C employed a bath of water and ice and reactions carried at -40 °C employed a bath of dry ice and MeCN. Anhydrous THF, CH_2Cl_2, MeOH, MeCN, DMF, and toluene were obtained using a PureSolv® solvent purification system. The reactions were monitored by thin layer chromatography (TLC) on aluminium backed silica plates (Merck Silica Gel 60 F254). Visualisation of TLC plates was undertaken with an ultraviolet (UV) light at λ = 254 nm and staining with solutions of vanillin, ninhydrin, and potassium permanganate followed by exposure of the stained plates to heat. Silica flash column chromatography (Merck Silica Gel 60 40 – 63 µm) was undertaken to purify crude reaction mixtures using solvents as specified. Fractions were collected manually or with a Biotage Isolera Spektra automated flash purification system.

All commercially available reagents were used as obtained from Sigma-Aldrich, Merck, Acros Organics or AK Scientific. Amino acids, coupling reagents and Trityl-OH ChemMatrix® resins were obtained from NovaBiochem, PCAS, GL Biochem, or Mimotopes and peptide synthesis grade DMF was obtained from Merck or Labscan. All non-commercially available reagents were synthesized according to literature procedures as referenced.

^{1}H NMR spectra were obtained using a Bruker DRX 300, DRX 400 or DRX 500 at frequencies of 300 MHz, 400 MHz or 500 MHz respectively in $CDCl_3$, MeOD, acetone-d^6, or DMSO-d^6 Chemical shifts are reported in parts per million (ppm) and coupling constants in Hertz (Hz). ^{1}H NMR data is reported as follows: chemical shift values (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant(s) and relative integral. ^{13}C NMR spectra were obtained using a Bruker DRX 400 or DRX 500 at 100 MHz or 125 MHz in $CDCl_3$, MeOD, CD_3CN, DMSO-d^6, and acetone-d^6 unless otherwise specified. ^{13}C NMR data is reported as chemical shift values (ppm). Any rotamers were confirmed *via* variable temperature NMR experiments or saturation transfer NMR experiments. Low resolution mass spectra for novel compounds were recorded on a Bruker amaZon SL mass spectrometer (ESI) operating in positive/negative mode or on a Shimadzu 2020 (ESI) mass spectrometer operating in positive/negative mode. High resolution mass spectra were recorded on a Bruker-Daltronics Apex Ultra 7.0T Fourier transform (FTICR) mass spectrometer.

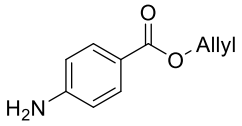
LC-MS was performed on a Shimadzu UPLC-MS instrument with an LC-M20A pump, SPD-M30A diode array detector and a Shimadzu 2020 (ESI) mass spectrometer operating in positive mode. Separations on the UPLC system were performed on a Waters Acquity 1.7 µm, 2.1 x 50 mm (C18) column. These separations were performed using a mobile phase of 0.1 vol % trifluoroacetic acid in water (Solvent A) and 0.1 vol % trifluoroacetic acid in MeCN (Solvent B) using linear gradients. Preparative reverse-phase HPLC was performed using a Waters 500 pump with a 2996 photodiode array detector and a Waters 600 Multisolvent Delivery System.

Synthesis of Fmoc-PABA-OH (7)



4-Aminobenzoic acid (1.00 g, 7.29 mmol), Fmoc-OSu (2.58 g, 7.66 mmol) and $NaHCO_3$ (1.67 g) were dissolved in 1,4-dioxane/water (1:1, v/v, 40 mL) and the reaction solution was stirred at room temperature for 16 h. The resulting reaction mixture was acidified to pH 1 *via* addition of 2 M aqueous HCl and the resulting precipitate was collected and dried *in vacuo* to afford the title compound (2.14 g, 5.94 mmol, 81%) as a white solid. **^{1}H NMR** (DMSO-d^6, 400 MHz) δ (ppm); 12.63 (br s, 1H), 10.08 (s, 1H), 7.91 (m, 2H), 7.85 (m, 2H), 7.76 (m, 2H), 7.56 (m, 2H), 7.43 (m, 2H), 7.35 (m, 2H), 4.53 (d, *J* = 6.4 Hz, 2H), 4,33 (t, *J* = 6.4 Hz, 1H); **^{13}C NMR** (DMSO-d^6, 125 MHz) δ (ppm); 166.9, 153.3, 143.7, 143.2, 140.8, 130.4, 127.7, 127.1, 125.1, 124.5, 120.2, 117.4, 65.8, 46.6; **LR-MS**: (-ESI) *m/z* 358 $[M-H]^-$; **HR-MS**: (+ESI) Calc. for $C_{22}H_{18}NO_4$: 382.1050 $[M+Na]^+$, Found: 382.1048 $[M+Na]^+$; **IR (ATR):** ν_{max} = 3347, 2971, 2941, 1709, 1676, 1610, 1592, 1526, 1511, 1450, 1412, 1311, 1282, 1223 cm^{-1}; **mp:** 240 – 241 °C. This data is in agreement with that previously reported by Van der Plas *et al*.[1]

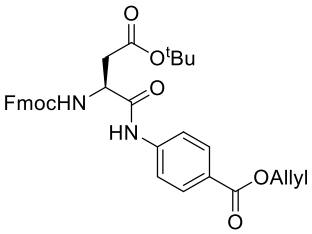
Synthesis of PABA-OAll (8)



4-Aminobenzoic acid (3.98 g, 29.0 mmol) and K_2CO_3 (4.00 g, 29.0 mmol) were dissolved in DMF (116 mL) and to this solution was added allyl bromide (3.80 mL, 43.6 mmol). The resulting reaction mixture was stirred at room temperature for 16 h. The reaction mixture was diluted with water (300 mL) and extracted with ethyl acetate (2 x 150 mL). The combined organic extracts were washed with saturated aqueous $NaHCO_3$ solution (100 mL), water (100 mL) and brine (100 mL), dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The crude residue was purified by flash chromatography (30 – 40% v/v EtOAc in *n*-hexanes) to afford allyl 4-aminobenzoate (3.43 g, 19.3 mmol, 67%) as a yellow solid. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ (ppm); 7.87 (m, 2H), 6.63 (m, 2H), 6.02 (ddt, *J* = 17.5, 10.4, 5.6, 1H), 5.38 (m, 1H), 5.25 (m, 1H), 4.77 (dt, *J* = 5.6, 1.4), 4.10 (br s, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ (ppm); 166.4, 151.1, 132.8, 131.8, 119.7, 117.8, 113.9, 65.1; **LR-MS**: (+ESI) *m/z* 178 $[M+H]^+$; **HR-MS**: (+ESI) Calc. for $C_{10}H_{11}NO_2$: 178.0863 $[M+H]^+$, Found: 178.0859 $[M+H]^+$; **IR (ATR):** ν_{max} = 3418, 3330, 3214, 1685, 1636, 1596, 1574, 1513, 1459, 1443, 1359, 1309, 1276, 1237 cm^{-1}; **mp:** 53 – 54 °C. This data is in agreement with that previously reported by Garrido-Hernandez *et al*.[2]

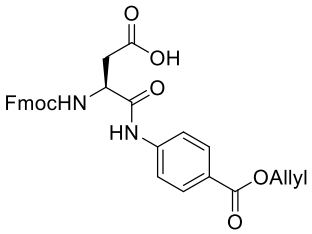
Synthesis of Fmoc-L-Asp-PABA-OAll (6)

*Allyl (S)-4-(2-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-4-(tert-butoxy)-4-oxobutanamido)benzoate (**S1**)*



Fmoc-L-aspartic acid 4-*tert*-butyl ester (1.23 g, 2.99 mmol), HATU (1.14 g, 2.99 mmol) and HOAt (813 mg, 5.97 mmol) were dissolved in DMF (4 mL) and to this solution was added *i*Pr_2NEt (1.04 mL, 5.97 mmol). The resulting reaction mixture was stirred at room temperature for 10 minutes at which point PABA-OAll (**8**) (353 mg, 1.99 mmol) was added. The reaction mixture was stirred at room temperature for 16 h. The reaction solution was washed with saturated aqueous $NaHCO_3$ (50 mL) and extracted with EtOAc (2 x 25 mL). The combined organic extracts were washed with brine (50 mL), dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The crude residue was purified by flash chromatography (20 – 30% v/v EtOAc in *n*-hexane) to afford the title compound (998 mg, 1.75 mmol, 88%) as a pale brown solid. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm); 8.82 (br s, 1H), 8.02 (m, 2H), 7.76 (m, 2H), 7.56-7.59 (m, 4H), 7.39 (m, 2H), 7.28 (m, 2H), 6.11 (br s, 1H), 6.04 (ddt, *J* = 17.2, 10.5, 5.6 Hz, 1H), 5.40 (m, 1H), 5.29 (m, 1H), 4.81 (dt, *J* = 5.6, 1.5 Hz, 2H), 4.67 (br s, 1H), 4.48 (d, *J* = 6.7 Hz, 2H), 4.22 (t, *J* = 6.7 Hz, 1H), 2.95 (dd, *J* = 16.6, 3.0 Hz, 1H), 2.70 (dd, *J* = 16.6, 6.4 Hz, 1H), 1.46 (s, 9H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm); 171.4, 169.0, 165.8, 143.7, 143.6, 141.8, 141.5, 132.4, 131.0, 128.0, 127.2, 126.2, 125.0, 120.2, 119.2, 118.3, 82.5, 67.5, 65.6, 52.0, 47.3, 37.4, 28.1; **LR-MS**: (+ESI) *m/z* 593 $[M+Na]^+$; **HR-MS**: (+ESI) Calc. for $C_{33}H_{34}N_2O_7$: 593.2258 $[M+Na]^+$, Found: 593.2256 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -18.0° (c = 0.63 in $CHCl_3$); **IR (ATR):** ν_{max} = 3324, 3306, 3066, 2977, 2935, 1716, 1694, 1601, 1537 cm^{-1}; **mp:** 143 – 144 °C.

*Fmoc-L-Asp-PABA-OAll (**6**)*

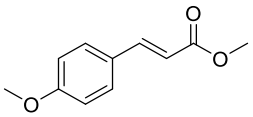


Allyl (*S*)-4-(2-((((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)-4-(*tert*-butoxy)-4-oxobutanamido)benzoate ester (**S1**) (2.62 g, 4.60 mmol) was dissolved in CH_2Cl_2/TFA (1:1, v/v, 8 mL) and stirred at room

temperature for 1 h. The reaction mixture was concentrated *in vacuo*, azeotroping with toluene to afford the title compound (2.37 g, 4.60 mmol, quantitative) as a brown solid. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm); 9.70 (br s, 1H), 7.80 (m, 2H), 7.82 (m, 2H), 7.68 (m, 2H), 7.37 (m, 2H), 7.28 (m, 2H), 7.14 (br d, *J* = 8.2 Hz, 1H), 6.07 (ddt, *J* = 17.0, 10.7, 5.5 Hz, 1H), 5.41 (m, 1H), 5.25 (m, 1H), 4.78-4.81 (m, 3H), 4.33-4.43 (m, 2H), 4.22 (t, *J* = 6.9 Hz, 1H), 3.05 (dd, *J* = 16.8, 6.3 Hz, 1H), 2.90 (dd, *J* = 16.8, 6.9 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm); 172.4, 170.6, 165.9, 157.2, 144.8, 144.0, 142.0, 133.7, 131.2, 128.5, 127.9, 126.1, 126.0, 120.7, 119.8, 118.0, 67.5, 65.7, 53.4, 47.9, 36.5; **LR-MS**: (+ESI) *m/z* 515 $[M+H]^+$; **HR-MS**: (+ESI) Calc. for $C_{29}H_{26}N_2O_7$: 537.1632 $[M+Na]^+$, Found: 537.1632 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -18.3° (c = 1.0 in DMSO); **IR (ATR):** ν_{max} = 3326, 3068, 3047, 1703, 1599, 1540, 1512 cm^{-1}; **mp:** 182 – 183 °C.

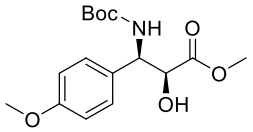
Synthesis of a suitably protected *erythro*-β-methoxy-L-Asp-PABA-OAll compound (25)

*Methyl (E)-3-(4-methoxyphenyl)acrylate (**S2**)*



Thionyl chloride (4.06 mL) was added dropwise to anhydrous MeOH at 0 °C followed by *E*-methoxy cinnamic acid (4.99 g, 28.0 mmol). The resulting reaction mixture was stirred at reflux for 1.5 h. The reaction mixture was concentrated *in vacuo* redissolved in EtOAc (75 mL) and washed with water (50 mL) and saturated aqueous $NaHCO_3$ solution (50 mL). The organic layer was then dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo* to afford the title compound as a white flaky solid which was sufficiently pure to be used in the next step without purification (4.89 g, 25.5 mmol, 91%). **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm); 7.65 (d, *J* = 16.1 Hz, 1H), 7.47 (m, 2H) 6.90 (m, 2H), 6.31 (d, *J* = 16.1 Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H); **LR-MS**: (+ESI) *m/z* 193 $[M+H]^+$; **mp:** 91 – 92 °C. This data is in agreement with that previously reported by Zhang *et al*.[3]

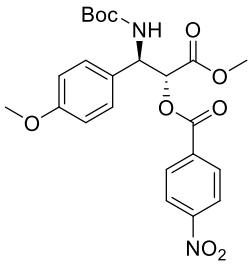
*Methyl (2S,3R)-3-((tert-butoxycarbonyl)amino)-2-hydroxy-3-(4-methoxyphenyl)propanoate (**S3**)*



tert-Butyl carbamate (1.34 g, 11.4 mmol) was dissolved in *n*-PrOH (19 mL) and a freshly prepared aqueous NaOH solution (0.31 M, 36 mL) was added. To this mixture was added *tert*-butyl hypochlorite (7.4 M, 1.51 mL) dropwise followed by $(DHQD)_2PHAL$ in *n*-PrOH (17 mL). The resulting reaction mixture was stirred at 25 °C for 10 min after which methyl (*E*)-3-(4-methoxyphenyl)acrylate (**S2**) (916 mg, 4.77

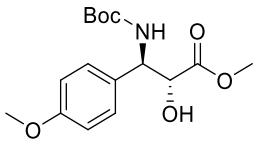
mmol) was added and mixture was sonicated until all the acrylate was dissolved. Upon dissolution of the acrylate, potassium osmate (VI) dihydrate was added in one portion causing the solution to turn black. The resulting reaction mixture was stirred at room temperature for 16 h providing a golden-brown solution. The reaction mixture was extracted with EtOAc (3 x 50 mL) and washed with water (75 mL) and brine (75 mL), dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The crude residue was purified by flash chromatography (20 – 30% v/v EtOAc in *n*-hexanes) to afford the title compound as a white flaky solid (1.17 g, 3.60 mmol, 75%). **^{1}H NMR** ($CDCl_3$, 500 MHz) δ (ppm); 7.29 (m, 2H), 6.88 (m, 2H), 5.32 (d, *J* = 9.1 Hz, 1H), 5.15 (d, *J* = 9.1 Hz, 1H), 4.43 (s, 1H), 3.83 (s, 3H), 3.79 (s, 3H), 3.13 (br s, 1H), 1.41 (s, 9H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ (ppm); 173.6, 159.3, 155.3, 128.1, 127.4, 114.2, 80.0, 73.8, 55.4, 53.2, 28.4, 28.4; **LR-MS**: (+ESI) *m/z* 348 $[M+Na]^+$; **HR-MS**: (+ESI) Calc. for $C_{16}H_{23}NO_6$: 348.1418 $[M+Na]^+$, Found: 348.1417 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -4.7° (c = 0.96 in MeOH); **IR (ATR):** ν_{max} = 3373, 2977, 2935, 1694, 1612, 1512, 1392, 1366, 1246 cm^{-1}; **mp:** 81 – 83 °C. This data is in agreement with that previously reported by Mishra *et al*.[4]

*(1R,2R)-1-((tert-Butoxycarbonyl)amino)-3-methoxy-1-(4-methoxyphenyl)-3-oxopropan-2-yl 4-nitrobenzoate (**S4**)*



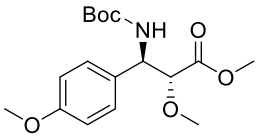
Methyl (2*S*,3*R*)-3-((*tert*-butoxycarbonyl)amino)-2-hydroxy-3-(4-methoxyphenyl)propanoate (**S3**) (1.33 g, 4.10 mmol), triphenylphosphine (2.37 g, 9.02 mmol) and *p*-nitrobenzoic acid (1.51 g, 9.02 mmol) was dissolved in THF (55 mL) and to this solution was added DIAD (1.78 mL, 9.02 mmol) at 0 °C. The resulting reaction mixture was protected from light, warmed to room temperature and stirred for 16 h. The reaction mixture was concentrated *in vacuo*. The crude material was purified by repeated flash chromatography (50% v/v CH_2Cl_2 in *n*-hexanes to 2% v/v MeOH in CH_2Cl_2) to afford the title compound as a white crystalline solid (1.42 g, 2.99 mmol, 73%). **^{1}H NMR** ($CDCl_3$, 500 MHz) δ (ppm); 8.28 (m, 2H), 8.18 (m, 2H), 7.28 (m, 2H), 6.90 (m, 2H), 5.65 (d, *J* = 4.8 Hz, 1H), 5.38 (br s, 1H), 5.32 (br d, *J* = 7.9 Hz, 1H), 3.80 (s, 3H), 3.68 (s, 3H), 1.42 (s, 9H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ (ppm); 167.9, 164.0, 159.7, 155.0, 151.0, 134.6, 131.2, 128.7, 128.4, 123.8, 114.3, 80.5, 75.5, 55.4, 54.6, 52.7, 28.4; **LR-MS**: (+ESI) *m/z* 497 $[M+Na]^+$; **HR-MS**: (+ESI) Calc. for $C_{23}H_{26}N_2O_9$: 497.1531 $[M+Na]^+$, Found: 497.1530 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -24.9° (c = 0.52 in $CHCl_3$); **IR (ATR):** ν_{max} = 3323, 3062, 2976, 1698, 1601, 1536 cm^{-1}; **mp:** 142 – 144 °C. This data is in agreement with that previously reported by Mishra *et al*.[4]

*Methyl (2R,3R)-3-((tert-butoxycarbonyl)amino)-2-hydroxy-3-(4-methoxyphenyl)propanoate (**S5**)*



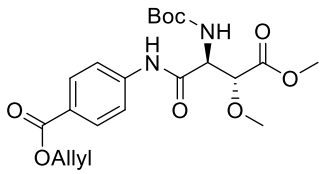
(1*R*,2*R*)-1-((*tert*-Butoxycarbonyl)amino)-3-methoxy-1-(4-methoxyphenyl)-3-oxopropan-2-yl 4-nitrobenzoate (**S4**) (1.51 g, 3.17 mmol) was dissolved in anhydrous MeOH (45 mL) and to this solution was added sodium azide (1.03 g, 15.9 mmol). The resulting reaction mixture was stirred at 45 °C for 48 h. The reaction mixture was concentrated *in vacuo*. The crude material was purified by flash chromatography (30% v/v EtOAc in *n*-hexanes) to afford the title compound as a white solid (956 mg, 2.94 mmol, 93%). **^{1}H NMR** ($CDCl_3$, 500 MHz) δ (ppm); 7.16 (m, 2H), 6.82 (m, 2H), 5.54 (br d, *J* = 7.6 Hz, 1H), 5.04 (br d, *J* = 7.6 Hz, 1H), 4.57 (br s, 1H), 3.77 (s, 3H), 3.70 (s, 3H), 2.90 (br s, 1H), 1.42 (s, 9H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ (ppm); 172.6, 159.5, 155.1, 129.1, 128.6, 114.0, 80.0, 73.5, 56.3, 55.3, 52.7, 28.5; **LR-MS**: (+ESI) *m/z* 348 $[M+Na]^+$; **HR-MS**: (+ESI) Calc. for $C_{16}H_{23}NO_6$: 348.1418 $[M+Na]^+$, Found: 348.1422 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -47.0° (c = 2.0 in MeOH); **IR (ATR):** ν_{max} = 3394, 2976, 2937, 2843, 1740, 1712, 1613, 1512, 1367, 1295, 1248 cm^{-1}; **mp:** 151 – 152 °C. This data is in agreement with that previously reported by Mishra *et al*.[4]

*Methyl (2R,3R)-3-((tert-butoxycarbonyl)amino)-2-methoxy-3-(4-methoxyphenyl)propanoate (**S6**)*



Methyl (2*R*,3*R*)-3-((*tert*-butoxycarbonyl)amino)-2-hydroxy-3-(4-methoxyphenyl)propanoate (**S5**) (227 mg, 0.70 mmol) was dissolved in anhydrous MeCN (6 mL) and to this solution was added silver (I) oxide (827 mg, 3.57 mmol) and iodomethane (443 µL, 7.14 mmol). The resulting reaction mixture was protected from the light and stirred at room temperature for 16 h after which additional silver (I) oxide (827 mg, 3.57 mmol) was added. The reaction mixture was stirred for a further 16 h then filtered through Celite® eluting with EtOAc (100 mL) and concentrated in vacuo. The crude material was purified by flash chromatography (20% v/v EtOAc in n-hexanes) to afford the title compound as a white solid (230 mg, 0.68 mmol, 97%). **^{1}H NMR** ($CDCl_3$, 500 MHz) δ (ppm); 7.20 (m, 2H), 6.81 (m, 2H), 5.45 (br d, J = 6.9 Hz, 1H), 5.06 (br s, 1H), 4.41 (br s, 1H), 3.77 (s, 3H), 3.60 (s, 3H), 3.46 (s, 3H), 1.42 (s, 9H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ (ppm); 170.4, 159.3, 155.1, 130.0, 128.8, 113.9, 83.0, 79.9, 59.4, 55.4, 55.3, 52.0, 28.5; **LR-MS**: (+ESI) m/z 362 $[M+Na]^+$; **HR-MS**: (+ESI) Calc. for $C_{17}H_{25}NO_6$: 362.1574 $[M+Na]^+$, Found: 362.1573 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -5.1° (c = 1.46, $CHCl_3$); **IR (ATR):** ν_{max} = 3370, 2975, 2956, 2934, 2837, 1753, 1709, 1612, 1511 cm^{-1}; **mp:** 90 – 91 °C.

*Allyl 4-((2S,3R)-2-((tert-butoxycarbonyl)amino)-3,4-dimethoxy-4-oxobutanamido)benzoate (**S7**)*

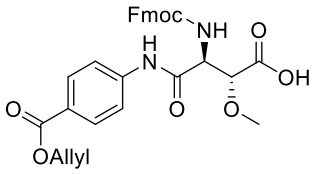


Oxidation:

Methyl (2*R*,3*R*)-3-((*tert*-butoxycarbonyl)amino)-2-methoxy-3-(4-methoxyphenyl)propanoate (**S6**) (504 mg, 1.48 mmol) was dissolved in EtOAc/MeCN (1:1, v/v, 34 mL) and to this solution was added sodium periodate (5.75 g, 26.9 mmol) in water (135 mL). The resulting reaction mixture was stirred mechanically at room temperature for 30 min after which ruthenium (III) trichloride trihydrate (77 mg, 0.30 mmol) was added followed by $NaHCO_3$ (485 mg, 5.80 mmol). The resulting reaction mixture was stirred mechanically at room temperature for 16 h. The reaction mixture was poured onto saturated aqueous $NaHCO_3$ solution (600 mL) and washed with CH_2Cl_2 (2 x 200 mL). The organic layer was further extracted with saturated aqueous $NaHCO_3$ solution (600 mL). Aqueous extracts were combined, acidified to pH 1 via addition of 2 M aqueous HCl, extracted with EtOAc (6 x 300 mL), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo) to afford (2*S*,3*R*)-2-((*tert*-butoxycarbonyl)amino)-3,4-dimethoxy-4-oxobutanoic acid as a brown foam which was sufficiently pure to be used in the next step without purification.

Amidation: The crude residue, HATU (562 mg, 1.48 mmol), HOAt (402 mg, 2.96 mmol) and allyl 4-aminobenzoate (**8**) (1.05 g, 5.92 mmol) were dissolved in DMF (15 mL) and to this solution was added *i*Pr_2NEt (516 µL, 2.96 mmol) at 0 °C. The reaction mixture was warmed to room temperature and stirred for 16 h. The reaction mixture was poured onto water (100 mL) and extracted with EtOAc (2 x 50 mL). Organic extracts were combined and washed with 2 M HCl (100 mL), saturated aqueous $NaHCO_3$ solution (100 mL), water (100 mL), brine (100 mL), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The crude material was purified by flash chromatography (10 – 30% v/v EtOAc in n-hexanes) to afford the title compound (419 mg, 0.96 mmol, 65% over 2 steps) as a pale brown solid. ^{1}H NMR spectroscopic analysis indicated a 5:1 mixture of rotamers through integration of the singlets at δ 3.81 and 3.77 ppm. **^{1}H NMR** ($CDCl_3$, 400 MHz, rotamers) δ (ppm); 8.51 (br s, 1H), 8.04 (m, 0.3H), 8.01 (m, 1.7H), 7.62 (m, 0.3H), 7.59 (m, 1.7H), 6.03 (ddt, J = 17.2, 10.5, 5.6 Hz, 1H), 5.61 (br d, 0.8H), 5.51 (br d, 0.2H), 5.40 (m, 1H), 5.28 (m, 1H), 4.80 (dt, J = 5.7, 1.4 Hz, 2H), 4.78 (m, 1H), 4.15 (d, J = 5.0 Hz, 1H), 3.81 (s, 2.5H), 3.77 (s, 0.5H), 3.52 (s, 2.5H), 3.49 (s, 0.5H), 1.48 (s, 7H), 1.47 (s, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz, rotamers) δ (ppm); 170.6, 167.3, 165.8, 141.6, 132.4, 131.0, 126.2, 119.3, 119.2, 118.3, 80.1, 65.6, 59.7, 59.5, 52.7, 28.4, 28.3, 1.2; **HR-MS**: (+ESI) Calc. for $C_{21}H_{28}N_2O_8$: 459.1738 $[M+Na]^+$, Found: 459.1739 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -3.0° (c = 1.15, $CHCl_3$); **IR (ATR):** ν_{max} = 3331, 2977, 2962, 2905, 2836, 1714, 1689, 1600, 1530, 1514 cm^{-1}; **mp:** 143 – 144 °C.

*(2R,3S)-3-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-4-((4-((allyloxy)carbonyl)phenyl)amino)-2-methoxy-4-oxobutanoic acid (**25**)*



Methyl ester deprotection: Allyl 4-((2*S*,3*R*)-2-((*tert*-butoxycarbonyl)amino)-3,4-dimethoxy-4-oxobutanamido)benzoate (**S7**) (392 mg, 0.90 mmol) was dissolved in THF (15 mL) and to this solution was added a 0.6 M aqueous LiOH solution (1.8 mL) at 0 °C. The resulting reaction mixture was stirred at 0 °C for 40 min, poured onto 2 M HCl (25 mL) and extracted with EtOAc (2 x 25 mL). The organic layer dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo.

Boc-deprotection: The crude residue was dissolved in trifluoroacetic acid:CH_2Cl_2 (1:1, v/v, 6 mL) and stirred at room temperature for 1 h. The reaction mixture was concentrated in vacuo.

Fmoc-protection: The crude residue was suspended in THF/saturated aqueous $NaHCO_3$ solution (2:1 v/v, 6 mL) and to this suspension was added Fmoc-OSu (353 mg, 0.95 mmol). The resulting reaction mixture was stirred at room temperature for 16 h. The reaction mixture was then acidified to pH 1 via addition of 2 M aqueous HCl and extracted with ethyl acetate (3 x 25 mL). The combined organic extracts were washed with brine (50 mL), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The crude residue was purified by flash chromatography (0 – 10% v/v MeOH in CH_2Cl_2) to afford the title compound (272 mg, 0.51 mmol, 57% over 3 steps) as a yellow solid. ^{1}H NMR spectroscopic analysis indicated a 1:3 mixture of rotamers through integration of the singlets at δ 3.43 and 3.41 ppm. **^{1}H NMR** (MeOD, 400 MHz, rotamers) δ (ppm); 8.00-7.93 (m, 2H), 7.78-7.64 (m, 6H), 7.38-7.27 (m, 4H), 6.05 (ddt, J = 17.3, 10.5, 5.4 Hz, 1H), 5.39 (br dd, J = 1.5, 17.3 Hz, 1H), 5.27 (br dd, J = 1.4, 10.5 Hz, 1H), 4.79 (br dt, J = 1.3, 5.5 Hz, 2H), 4.71 (br d, J = 5.8 Hz, 1H), 4.44-4.33 (m, 2H), 4.22 (br t, J = 6.8 Hz, 1H), 4.02 (br d, J = 5.4 Hz, 1H), 3.43 (s, 0.8H), 3.41 (s, 2.2H); **^{13}C NMR** (MeOD, 100 MHz) δ (ppm); 167.2, 145.2, 145.1, 144.0, 142.6, 133.8, 131.5, 131.5, 128.8, 128.2, 126.7, 126.3, 126.2, 120.9, 120.7, 120.6, 118.4, 68.4, 68.3, 66.5, 59.4, 59.2, 58.7; **LR-MS**: (+ESI) m/z 545 $[M+H]^+$; **HR-MS**: (+ESI) Calc. for $C_{30}H_{28}N_2O_8$: 567.1738 $[M+Na]^+$, Found: 567.1737 $[M+Na]^+$; **$[\alpha]_D^{20}$** = -13.0° (c = 0.6 in MeOH); **IR (ATR):** ν_{max} = 3308, 2938, 1715, 1695, 1600, 1529, 1450, 1410, 1271, 1251 cm^{-1}; **mp:** 126 – 129 °C.

NMR Data for Coralmycin A (1)

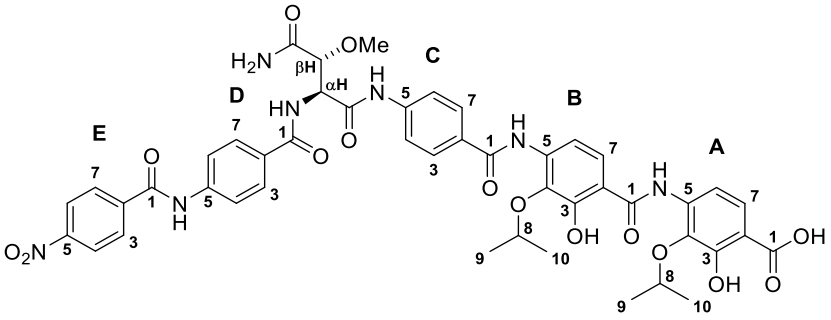


Table S1 ^{1}H-NMR (500 MHz) and ^{13}C-NMR (125 MHz) of synthetic coralmycin A (**1**) in DMSO-d^{6} referenced at 2.50 and 39.52 ppm, respectively. ^{13}C signals were extracted from the HSQC and HMBC spectra. All assignments were made based on COSY, HSQC and HMBC data, in comparison the isolated natural product.[5] Only the correct diastereomer was assigned. *Assignment difficult due to signal overlap. n.d. = not determined (due to overlapping signals, preventing assignment). ^value reported by Kim *et al*. does not match their included spectra.[5]

Residue	Position	Natural δ ^{1}H /ppm (mult, *J* Hz)	Synthetic δ ^{1}H /ppm (mult, *J* Hz)	Δ/ppm	Natural δ ^{13}C /ppm	Synthetic δ ^{13}C /ppm	Δ/ppm
A	1				n.d.	n.d.	
	2				n.d.	n.d.	
	3				n.d.	n.d.	
	4				n.d.	n.d.	
	5				n.d.	n.d.	
	6	n.d.	n.d.		n.d.	n.d.	
	7	n.d.	n.d.		n.d.	n.d.	
	8	4.72 (m)	4.69 (m)	0.03	72.3	72.7*	0.4
	9,10	1.23 (d, 6.0)	1.23 (d, 6.1)	0	21.9	22*	0.1
	NH	n.d.	n.d.				
B	1				n.d.	n.d.	
	2				n.d.	n.d.	
	3				n.d.	n.d.	
	4				n.d.	n.d.	
	5				n.d.	n.d.	
	6	n.d.	n.d.		n.d.	n.d.	
	7	n.d.	n.d.		n.d.	n.d.	
	8	4.38 (m)^	4.65 (m)		75.2	72.7*	0.5
	9.10	1.26 (d, 6.5)	1.25 (d, 6.0)	0.01	22	22*	0
	NH	9.53 (br s)	n.d.				
C	1				n.d.	n.d.	

	2				128.6	n.d.	
	3,7	7.96 (d, 8.5)	n.d.		128.4	n.d.	
	4,6	7.83 (d, 8.5)	7.82 (m)	0.01	118.8	119.0	0.2
	5				142	n.d.	
	NH	10.56 (s)	n.d.				
L-Asn	α C=O				n.d.	n.d.	
	α CH	4.92 (dd, 8.0, 8.5)	4.92 (m)	0	55.7	55.7	0
	β CH	4.10 (d, 8.5)	4.14 (d, 7.9)	0.04	79.9	80.0	0.1
	β OMe	3.31 (s)	3.30 (s)	0.01	57.9	57.8	0.1
	β C=O				n.d.	n.d.	
	α NH	8.48 (d, 8.0)	n.d.				
	γ NH_2	7.48 (br s)	n.d.				
		7.56 (br s)	n.d.				
D	1				n.d.	n.d.	
	2				128.5	n.d.	
	3,7	7.91 (m)	n.d.		128.2	n.d.	
	4,6	7.91 (m)	7.92 (m)	0.01	119.4	119.5	0.1
	5				141.8	n.d.	
	NH	10.8 (s)	n.d.				
E	1				164.2	n.d.	
	2				140.2	140.1	0.1
	3,7	8.21 (d, 8.5)	8.22 (m)	0.01	129.3	129.1	0.2
	4,6	8.39 (d, 8.5)	8.36 (d, 7.5)	0.03	123.4	123.4	0
	5				149.2	149.1	0.1

Analytical HPLC Trace of Coralmycin A (1/*epi*-1)

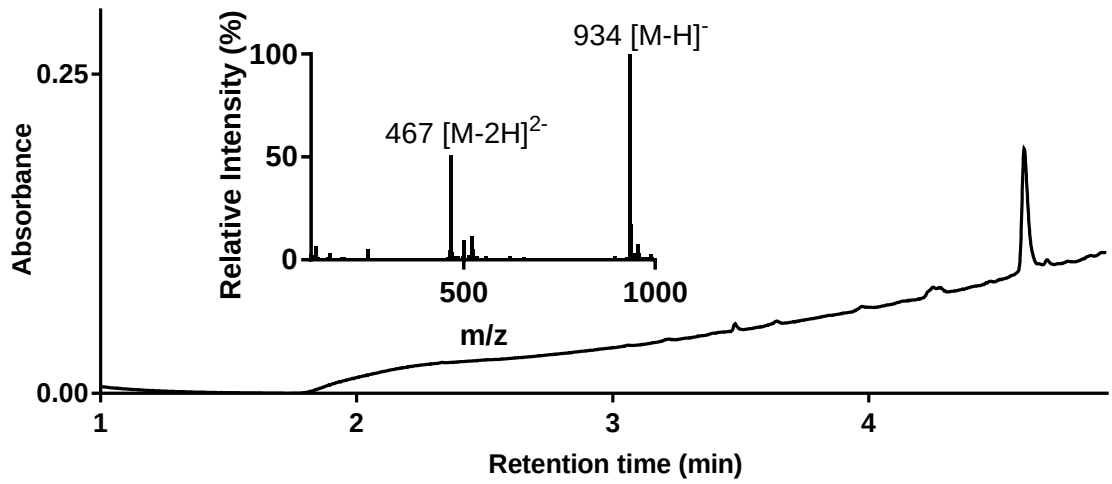


Figure S1 RP-HPLC trace of coralmycin A (**1**/***epi*-1**) with mass spectrum (+ESI) insert. Gradient: 0 – 100 vol % MeCN:water (0.1% TFA) linear gradient over 5 min. λ = 220 nm.

^{1}H NMR Spectrum of Coralmycin A (1/*epi*-1) showing diastereomeric ratio

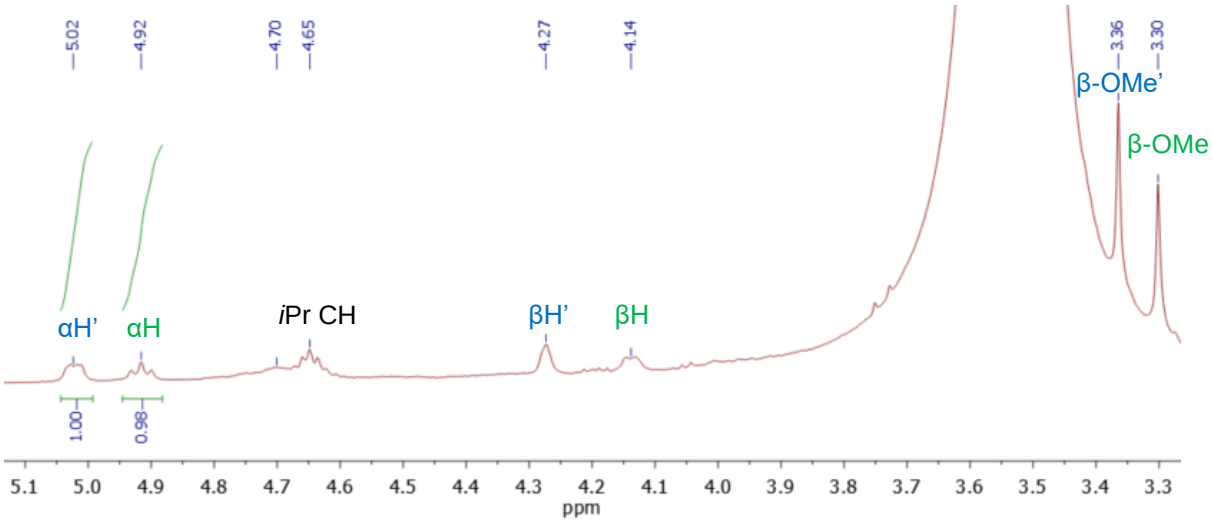


Figure S2 ^{1}H spectrum of synthetic coralmycin A (**1**/***epi*-1**) showing expected signals (green) and additional signals observed (blue).

NMR Data for Desmethoxycoralmycin (5)

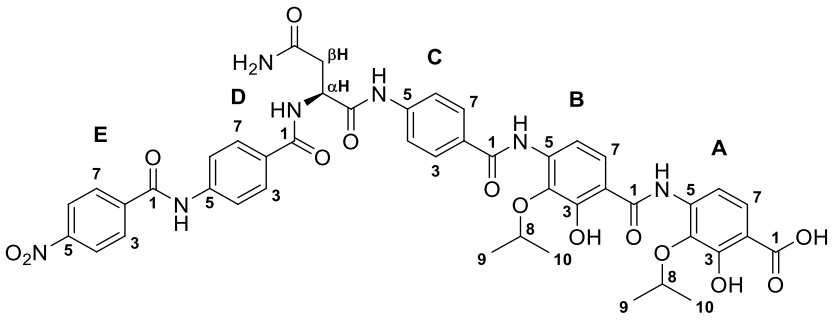


Table S2 ^{1}H-NMR (500 MHz) and ^{13}C-NMR (125 MHz) of **5** in DMSO-d^{6} referenced at 2.50 and 39.52 ppm, respectively. ^{13}C signals were extracted from the HSQC and HMBC spectra. All assignments were made based on COSY, HSQC and HMBC data. *Assignment difficult due to signal overlap.

Residue	Position	δ ^{1}H /ppm (mult, *J* Hz)	δ ^{13}C /ppm
A	1		163.5
	2		111.1
	3		151.7
	4		135.2
	5		137.3
	6	7.99 (m)*	117.3
	7	7.42 (m)*	123.7
	8	4.32 (m)	71.5*
	9,10	1.25 (m)*	21.9
	NH	11.28 (s)	
B	1		163.6
	2		116.3
	3		150.4
	4		138.2
	5		136.2
	6	7.51 (d, 8.7)	115.0
	7	7.79 (m)*	124.8
	8	4.70 (m)	71.4*
	9,10	1.25 (m)*	21.9
	NH	9.6 (m)	
C	1		164.3
	2		128.4
	3,7	7.82 (m)*	128.4
	4,6	7.98 (m)*	118.8
	5		142.3
	NH	7.79 (m)*	
L-Asn	α C=O		170.6

	α CH	4.92 (dd, 7.1, 7.1)	51.7
	β CH_2	2.70 (d, 6.9)	36.8
	β C=O		171.3
	α NH	8.69 (d, 7.3)	
	γ NH_2	7.42 (s)	
		6.98 (s)	
D	1		165.7
	2		129.2
	3,7	7.96 (m)*	128.3
	4,6	7.92 (m)*	119.6
	5		141.5
	NH	10.81 (s)	
E	1		164.2
	2		140.3
	3,7	8.22 (d, 8.4)	129.3
	4,6	8.38 (d, 8.4)	123.5
	5		149.3

Analytical HPLC Trace of Desmethoxycoralmycin A (5)

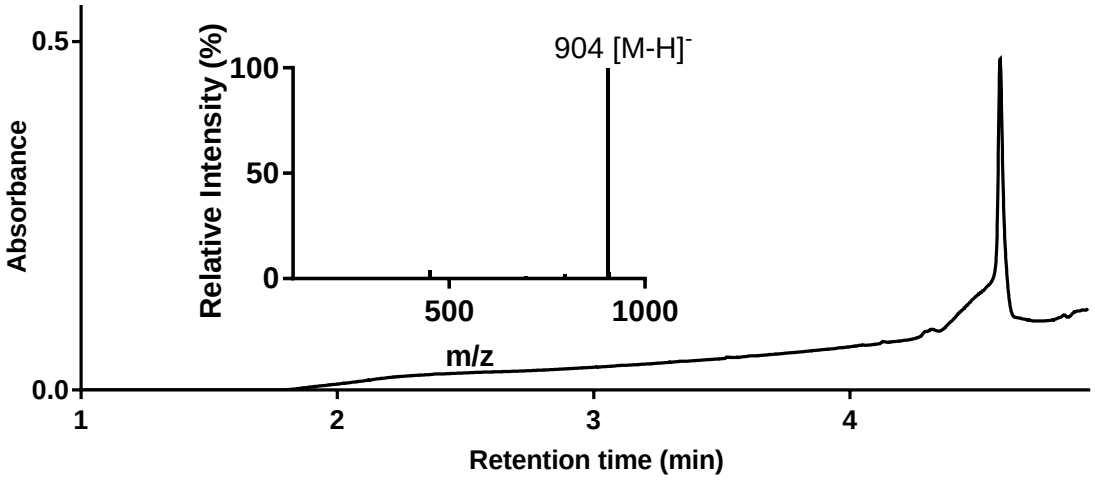


Figure S3 RP-HPLC trace of desmethoxycoralmycin A (**5**) with mass spectrum (+ESI) insert. Gradient: 0 – 100 vol % MeCN:water (0.1% TFA) linear gradient over 5 min. λ = 220 nm.

Analytical chiral LC Trace of Desmethoxycoralmycin A (5)

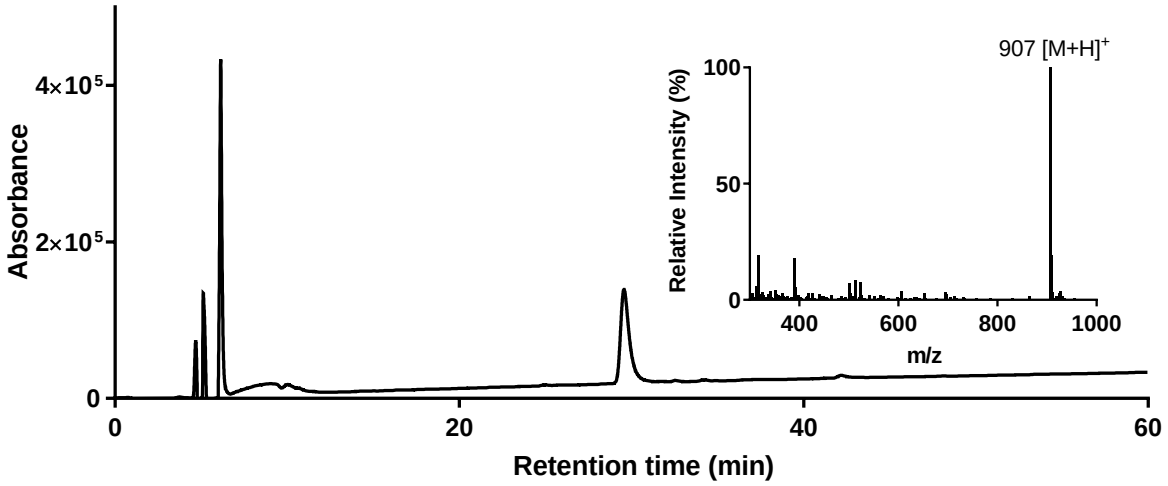
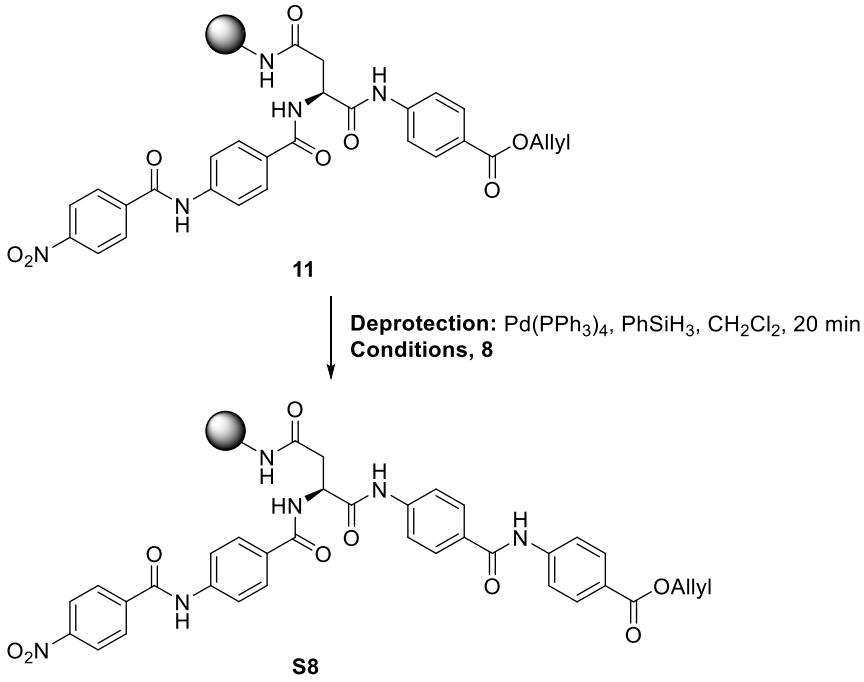


Figure S4 Liquid chromatography-MS chiral trace of desmethoxycoralmycin A using a Daicel Chiral CHIRALCEL® OJ-RH analytical column (pore size 5 µm, 4.6 x 150 mm) with mass spectrum (+ESI) insert. Gradient: 30 – 70 vol% MeCN:H_2O (0.1% formic acid) linear gradient over 60 min (0.2 mL/min). λ = 210 nm.

Conditions for the attempted amidation of resin-bound 11 with PABA-OAll (8)



Scheme S1 Attempted synthesis of resin-bound **S8**.

Table S3 Conditions trialled for the amidation of **11**. Results based on integration of peaks *via* UPLC-MS analysis. SM = starting material; HATU = 1-[*bis*(dimethylamino)methylene]-1*H*-1,2,3-triazolo[4,5b]pyridinium 3-oxid hexafluorophosphate; HOAt = 1-hydroxy-7-azabenzotriazole; DMTMM.BF_4 = 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium tetrafluoroborate; TFFH = tetramethylfluoroformamidinium hexafluorophosphate.

Entry	Conditions	Result
1	HATU (4 eq.), HOAt (8 eq.), *i*Pr_2NEt (8 eq.), in DMF, rt, 16 h	SM:HOAt active ester (7:3)
2	HATU (4 eq,), HOAt (8 eq.), *i*Pr_2NEt (8 eq.), cat. DMAP in DMF, rt, 16 h x 2	Product:HOAt active ester:dimethylamide product^ (6:14:4)
3	HATU (8 eq.), HOAt (16 eq.), *i*Pr_2NEt (16 eq.), cat. DMAP in DMF, µW, 50 °C, 1 h	HOAt active ester:dimethylamide product^ (7:3)
4	*N*-ethoxycarbonyl-2-ethoxy-1,2-dihydroquinoline (4 eq.), DMF, rt, 16 h	SM
5*	DMTMM.BF_4 (4 eq.), *i*Pr_2NEt (5 eq.) in DMF, µW, 50 °C, 2 h	SM:HOAt active ester (8:2)
6*	TFFH (4 eq,), *i*Pr_2NEt (4 eq.) in DMF, rt, 16 h	SM:acid fluoride (17:3)

*Reaction trialled on desnitro **11**

^Product arising from the amidation of **11** with dimethylamine impurities (present in low quantities in DMF)

Antimicrobial data

Table S4 Target Panel Bacterial Strains and Growth Conditions

Strain Name	Strain Designation	BSL	Growth Medium	Growth Condition
Gram-Positive				
Bacillus subtilis	ATCC 6051	1	NB	37°C
Enterococcus faecalis	ATCC 29212	2	BHI	37°C
Enterococcus faecium	ATCC 6569	2	BHI	37°C
Staphylococcus aureus (Methicillin-Resistant/Multidrug-Resistant)	BAA-44	2	TSB	37°C
Staphylococcus aureus (Methicillin-Sensitive)	ATCC 29213	2	TSB	37°C
Staphylococcus epidermidis	ATCC 14990	1	TSB	37°C
Streptococcus pneumoniae	ATCC 49619	2	BHI	37°C; 5% CO_2
Gram-Negative				
Acinetobacter baumanii	ATCC 19606	2	TSB	37°C
Escherichia coli	K-12 MG1655	1	NB	37°C
Klebsiella aerogenes	ATCC 35029	1	NB	37°C
Klebsiella pneumoniae	ATCC 700603	2	NB	37°C
Providencia alcalifaciens	ATCC 9886	1	TSB	37°C
Pseudomonas aeruginosa	ATCC 27853	2	TSB	37°C
Salmonella enterica	ATCC 13311	2	NB	37°C
Shigella sonnei	ATCC 25931	2	NB	37°C
Vibrio cholerae	A1552 El Tor	2	TSB	37°C
Yersinia pseudotuberculosis	ATCC 6904	2	BHI	37°C

Table S5 Biological activity of coralmycin A (**1**/***epi*-1**), desmethoxycoralmycin A (**5**), and various controls against a range of Gram-negative and Gram-positive bacteria.

	MIC (μM)								
Gram-negative Bacteria	Coralmycin A (**1**/***epi*-1**)	Desmethoxy coralmycin A (**5**)	Ampicillin	Gentamicin		Ciprofloxacin	Levofloxacin	Sparfloxacin	Norfloxacin
E. coli	2	0.83	0.09	0.25		0.17	0.10	0.13	0.25
K. aerogenes	21	1.7	>128	0.5		2.8	0.33	0.25	2
K. pneumoniae	128	21	>128	1		2	4	8	5.8
P. alcalifaciens	19	1	1.8	9.3		0.13	0.42	1.7	0.25
S. enterica	4	1	0.02	2		0.05	0.06	0.06	0.17
S. sonnei	1.5	0.15	0.03	0.25		0.08	0.13	0.06	0.25
Y. pseudotuberculosis	32	5.3	0.02	1.3		0.14	0.13	0.06	0.5
A. baumanii	8	1	>128	1.7		2	1	0.08	32
P. aeruginosa	128	21	>128	1.7		0.5	3.3	4	5.3
V. cholerae	1.3	0.46	0.75	0.83		0.02	0.02	0.004	0.15
Gram-positive Bacteria			Penicillin G	Vancomycin	Azithromycin				
B. subtilis	2.2	0.5	2.13	0.19	12	0.38	0.19	0.19	128
MSSA	2.8	0.38	14	1.5	1.3	1.2	1	0.25	8

MRSA	3.3	0.8	>128	2	>128	64	32	128	>128
S. epidermidis	2.7	0.5	>128	4	1	1.3	1.7	1	8
E. faecium	0.33	0.06	8	1	10.7	0.67	1	0.5	2
E. faecalis	2	0.33	8	26.7	4	1	2.7	1.3	8
S. pneumoniae	0.13	0.05	0.33	32	0.1	>128	0.83	1	2

References

[1] S. E. Van der Plas, A. Gea, S. Figaroli, P. J. De Clercq, A. Madder, *Eur. J. Org. Chem.* **2008**, *2008*, 1582.

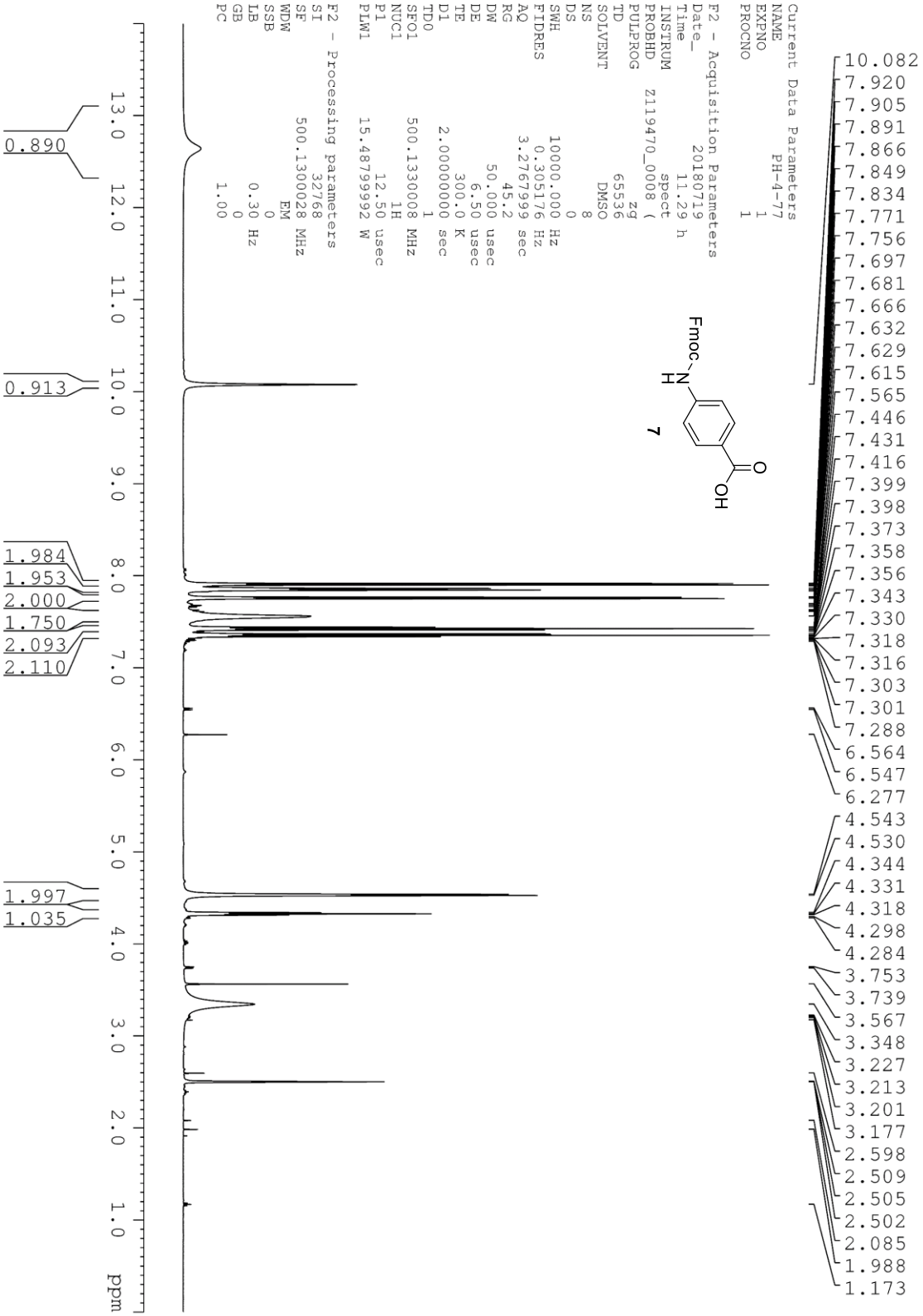
[2] H. Garrido-Hernandez, K. D. Moon, R. L. Geahlen, R. F. Borch, *J. Med. Chem.* **2006**, *49*, 3368.

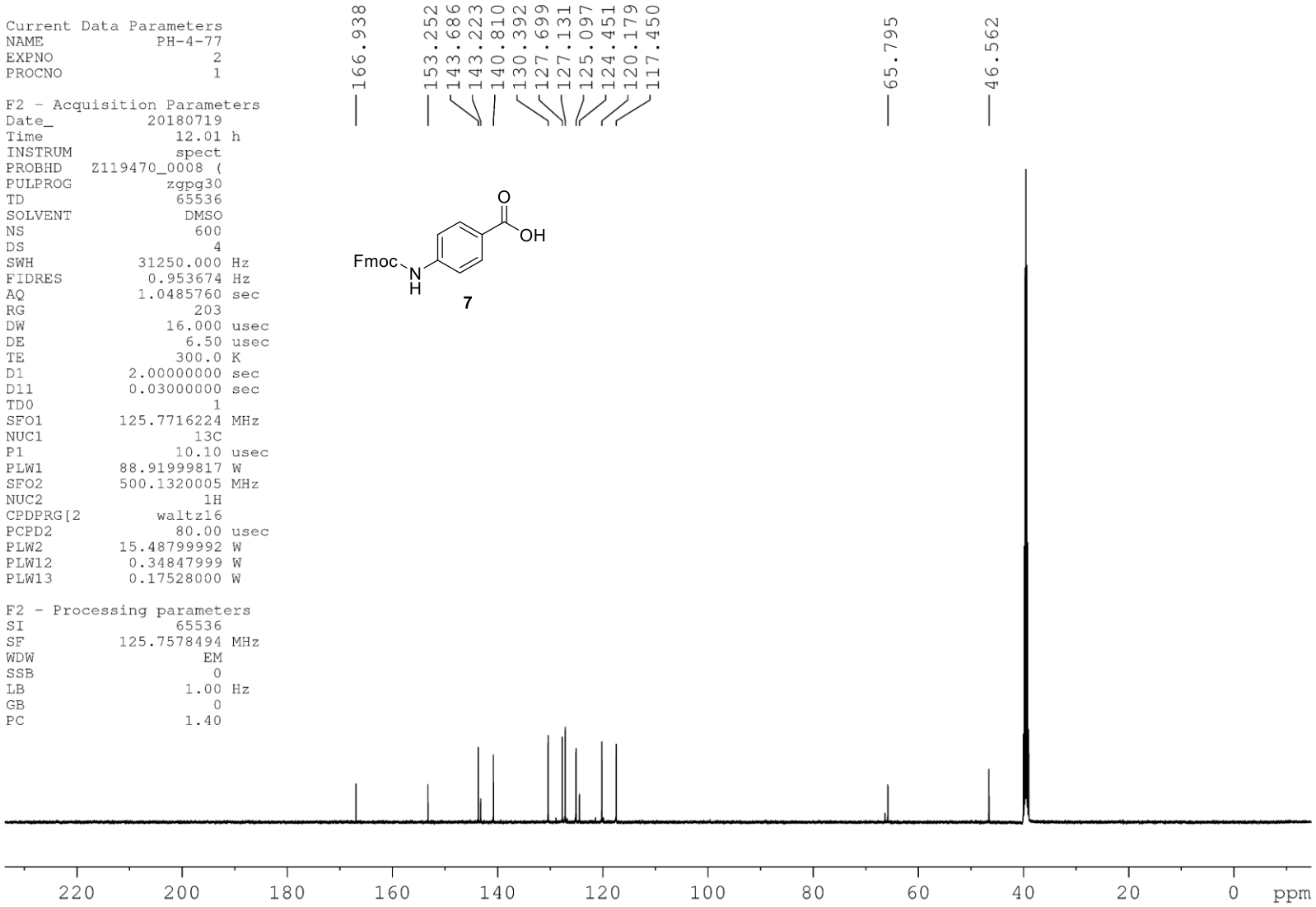
[3] Z. Zhang, Z. Wang, *J. Org. Chem.* **2006**, *71*, 7485.

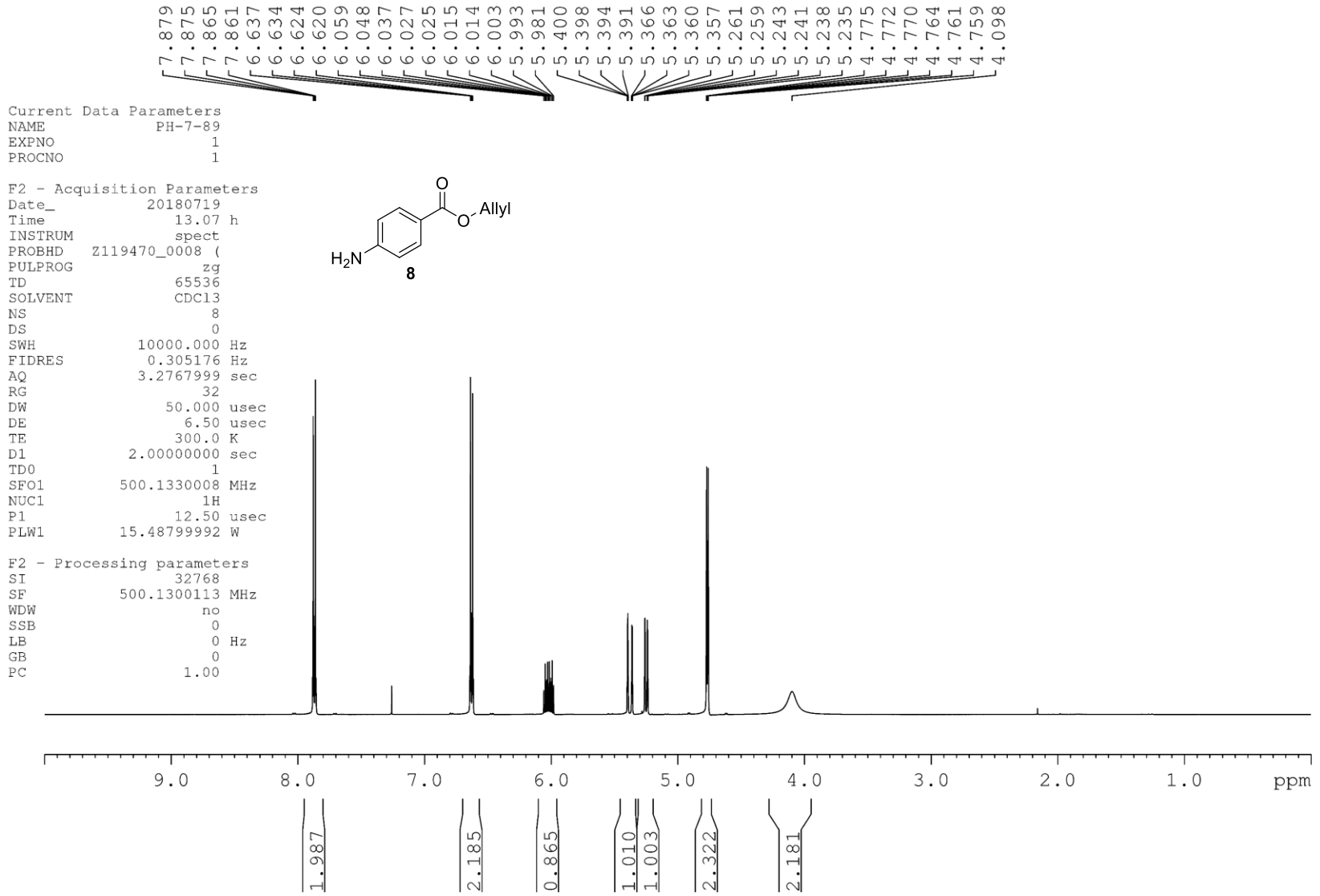
[4] R. K. Mishra, C. M. Coates, K. D. Revell, E. Turos, *Org. Lett.* **2007**, *9*, 575.

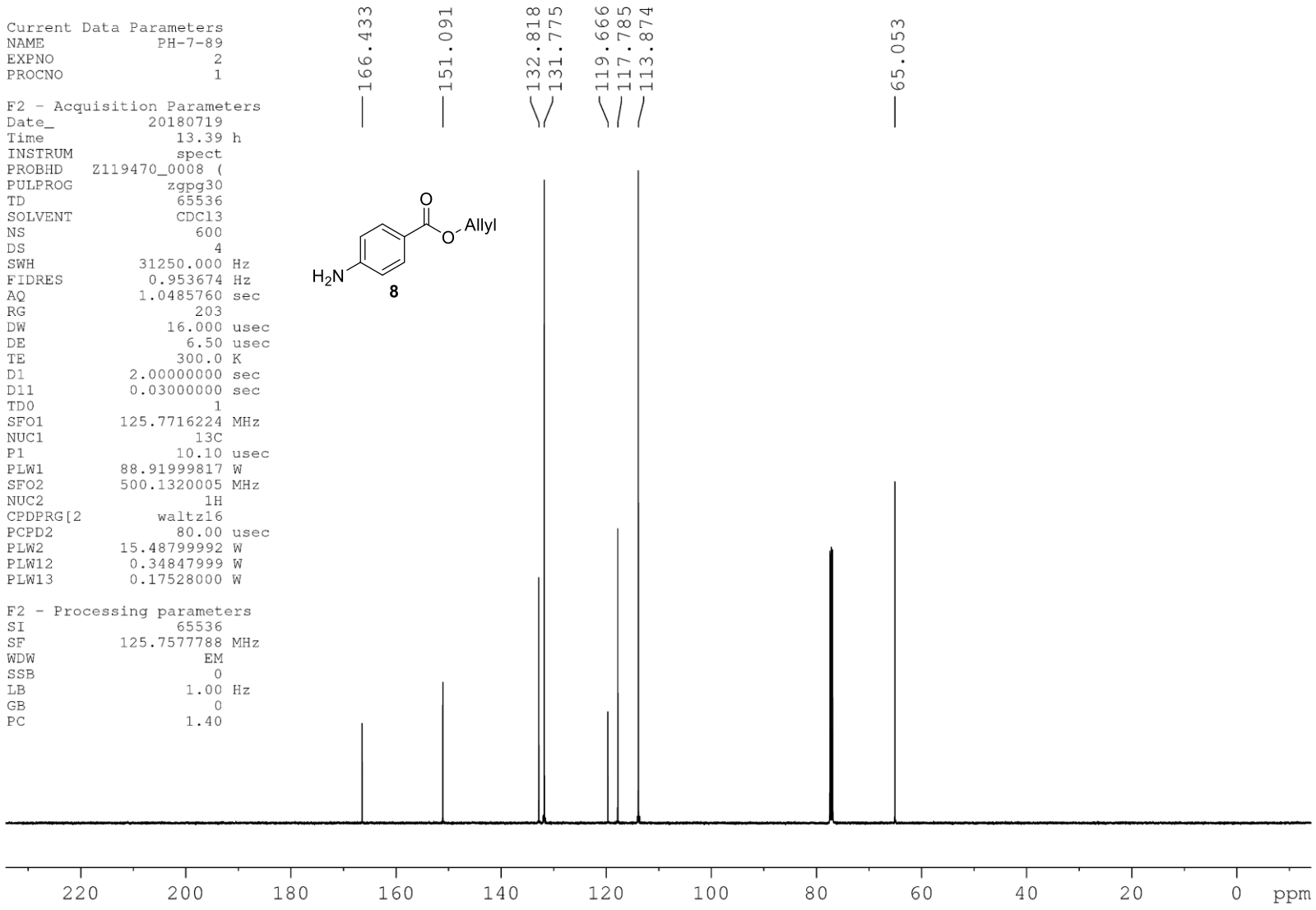
[5] Y. K. Kim, H.-J. Kim, G.-W. Kim, K. Cho, S. Takahashi, H. Koshino, W.-G. Kim, *J. Nat. Prod.* **2016**, *79*, 2223.

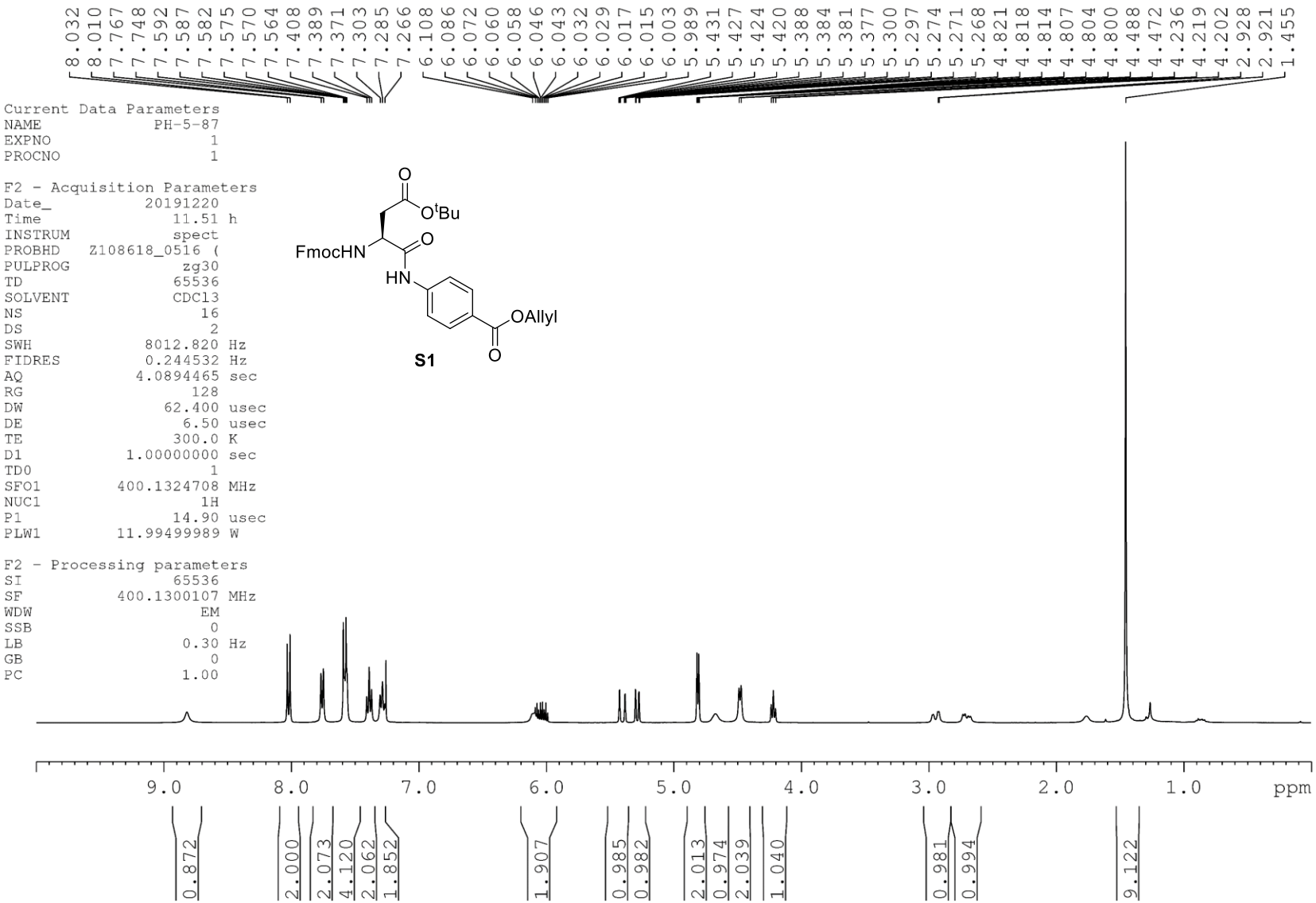
NMR Spectra

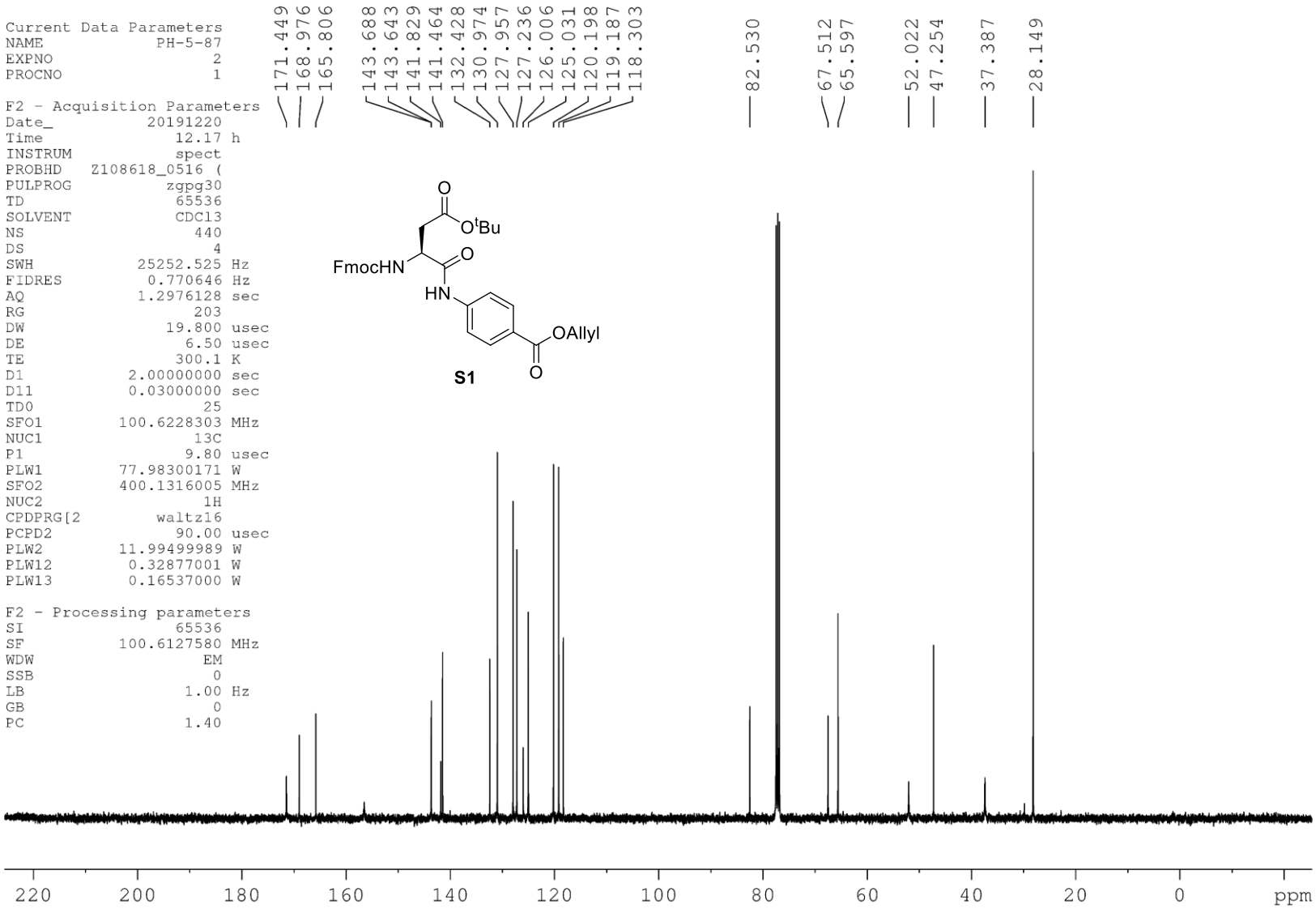

Current Data Parameters
NAME PH-4-77
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 11.29 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 45.2
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Fmoc
N
H
OH
O
7
10.082
7.920
7.905
7.891
7.866
7.849
7.834
7.771
7.756
7.697
7.681
7.666
7.632
7.629
7.615
7.565
7.446
7.431
7.416
7.399
7.398
7.373
7.358
7.356
7.343
7.330
7.318
7.316
7.303
7.301
7.288
6.564
6.547
6.277
4.543
4.530
4.344
4.331
4.318
4.298
4.284
3.753
3.739
3.567
3.348
3.227
3.213
3.201
3.177
2.598
2.509
2.505
2.502
2.085
1.988
1.173
0.890
0.913
1.984
1.953
2.000
1.750
2.093
2.110
1.997
1.035
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm

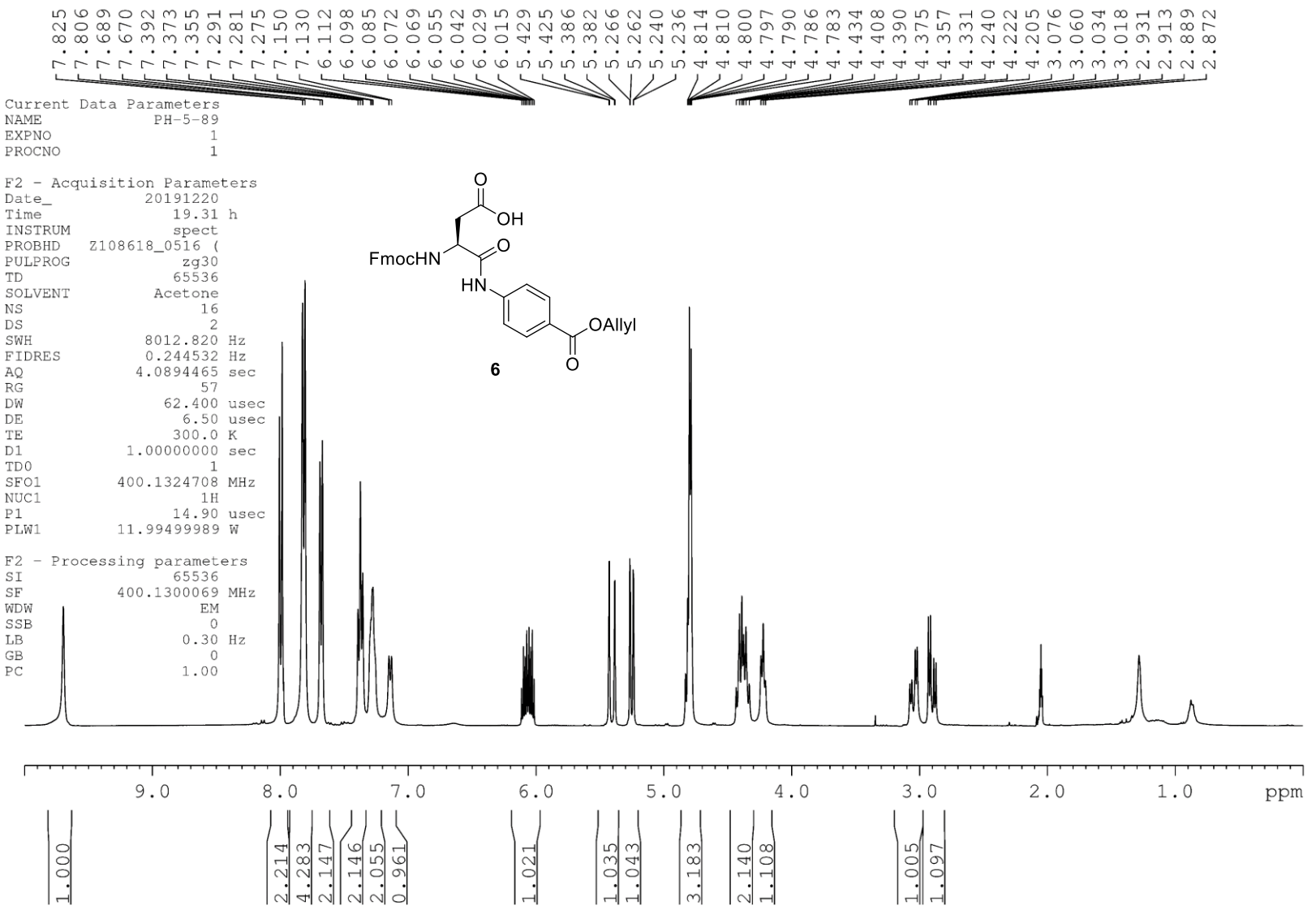

Current Data Parameters
NAME PH-4-77
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 12.01 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 600
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7578494 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
166.938
153.252
143.686
143.223
140.810
130.392
127.699
127.131
125.097
124.451
120.179
117.450
65.795
46.562
O
OH
Fmoc
N
H
7
220
200
180
160
140
120
100
80
60
40
20
0
ppm

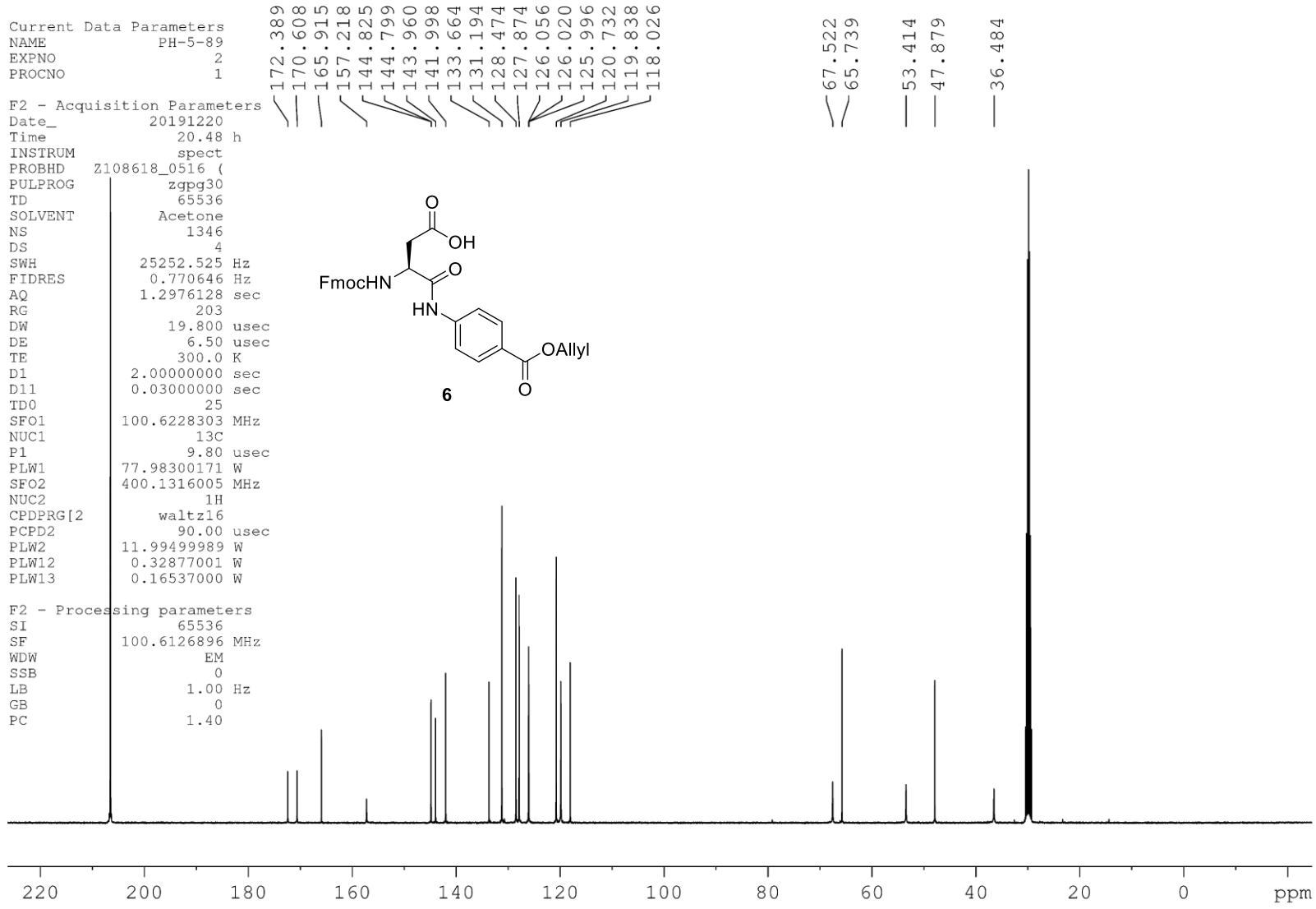

7.879
7.875
7.865
7.861
6.637
6.634
6.624
6.620
6.059
6.048
6.037
6.027
6.025
6.015
6.014
6.003
5.993
5.981
5.400
5.398
5.394
5.391
5.366
5.363
5.360
5.357
5.261
5.259
5.243
5.241
5.238
5.235
4.775
4.772
4.770
4.764
4.761
4.759
4.098
Current Data Parameters
NAME PH-7-89
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 13.07 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 32
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300113 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
O
Allyl
H2N
8
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
1.987
2.185
0.865
1.010
1.003
2.322
2.181

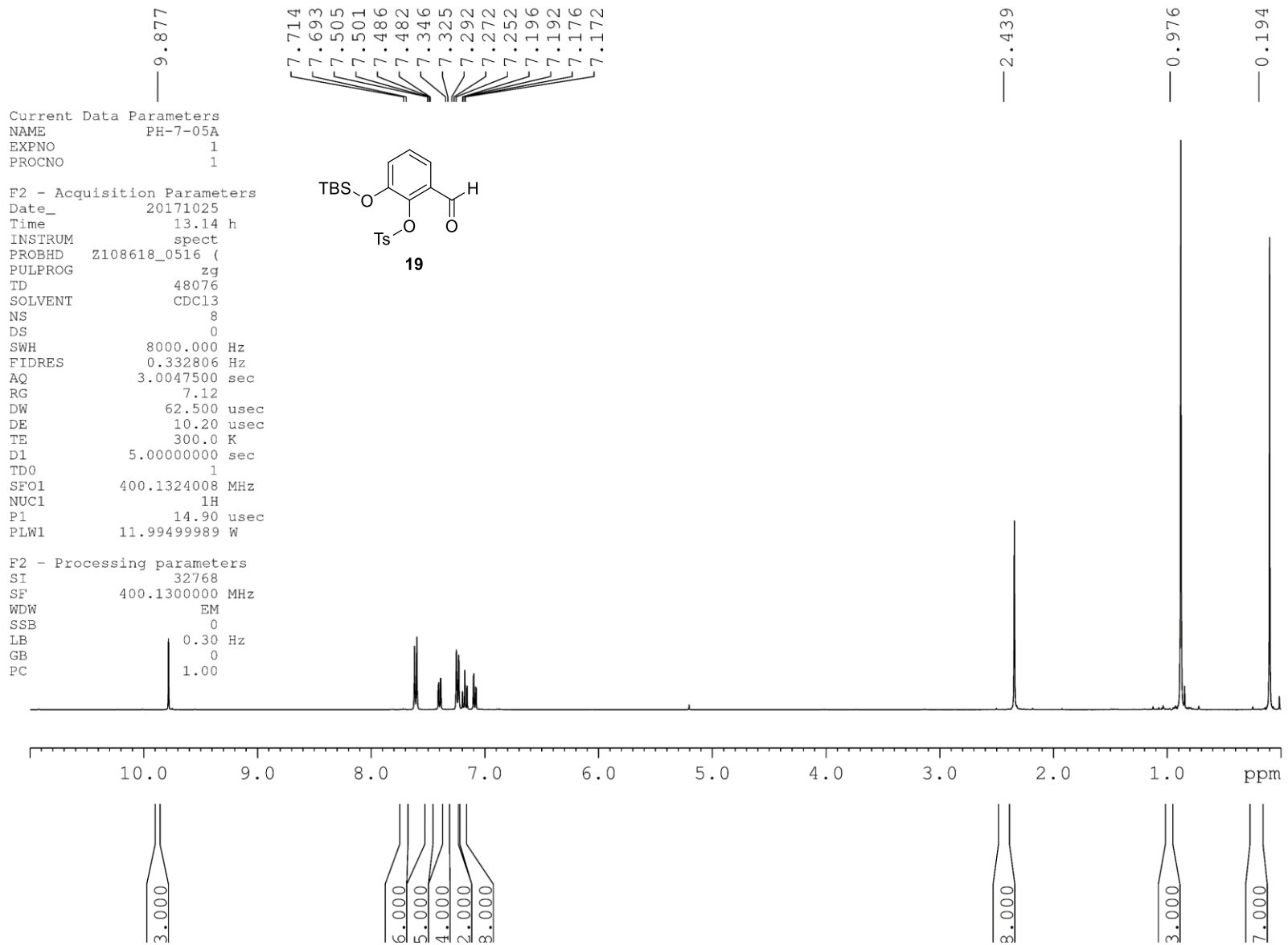

Current Data Parameters
NAME PH-7-89
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 13.39 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577788 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
166.433
151.091
132.818
131.775
119.666
117.785
113.874
65.053
O
O
Allyl
H2N
8
220
200
180
160
140
120
100
80
60
40
20
0
ppm

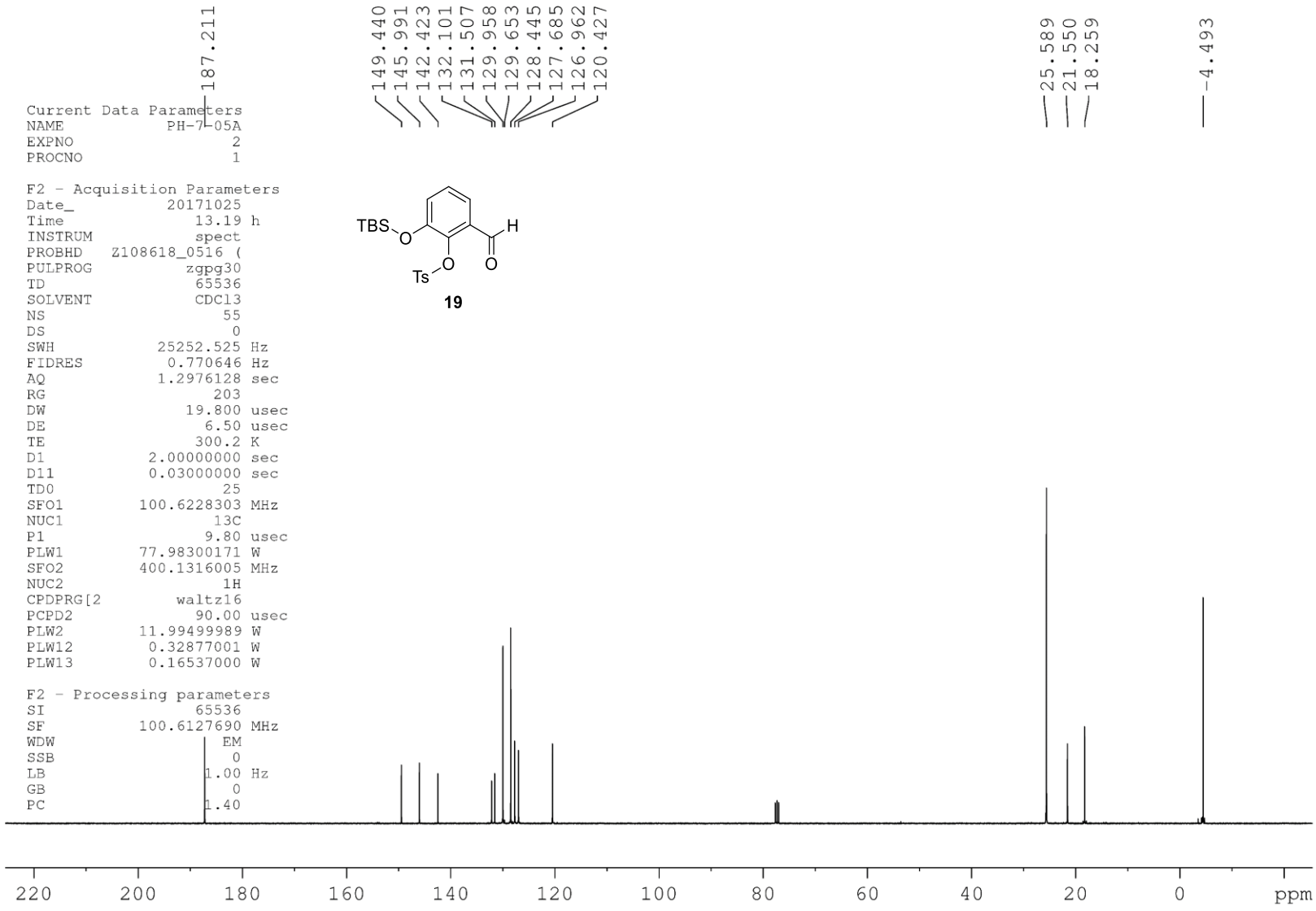

8.032
8.010
7.767
7.748
7.592
7.587
7.582
7.575
7.570
7.564
7.408
7.389
7.371
7.303
7.285
7.266
6.108
6.086
6.072
6.060
6.058
6.046
6.043
6.032
6.029
6.017
6.015
6.003
5.989
5.431
5.427
5.424
5.420
5.388
5.384
5.381
5.377
5.300
5.297
5.274
5.271
5.268
4.821
4.818
4.814
4.807
4.804
4.800
4.488
4.472
4.236
4.219
4.202
2.928
2.921
1.455
Current Data Parameters
NAME PH-5-87
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 11.51 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 128
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 65536
SF 400.1300107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OtBu
FmocHN
O
HN
OAllyl
O
S1
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
0.872
2.000
2.073
4.120
2.062
1.852
1.907
0.985
0.982
2.013
0.974
2.039
1.040
0.981
0.994
9.122

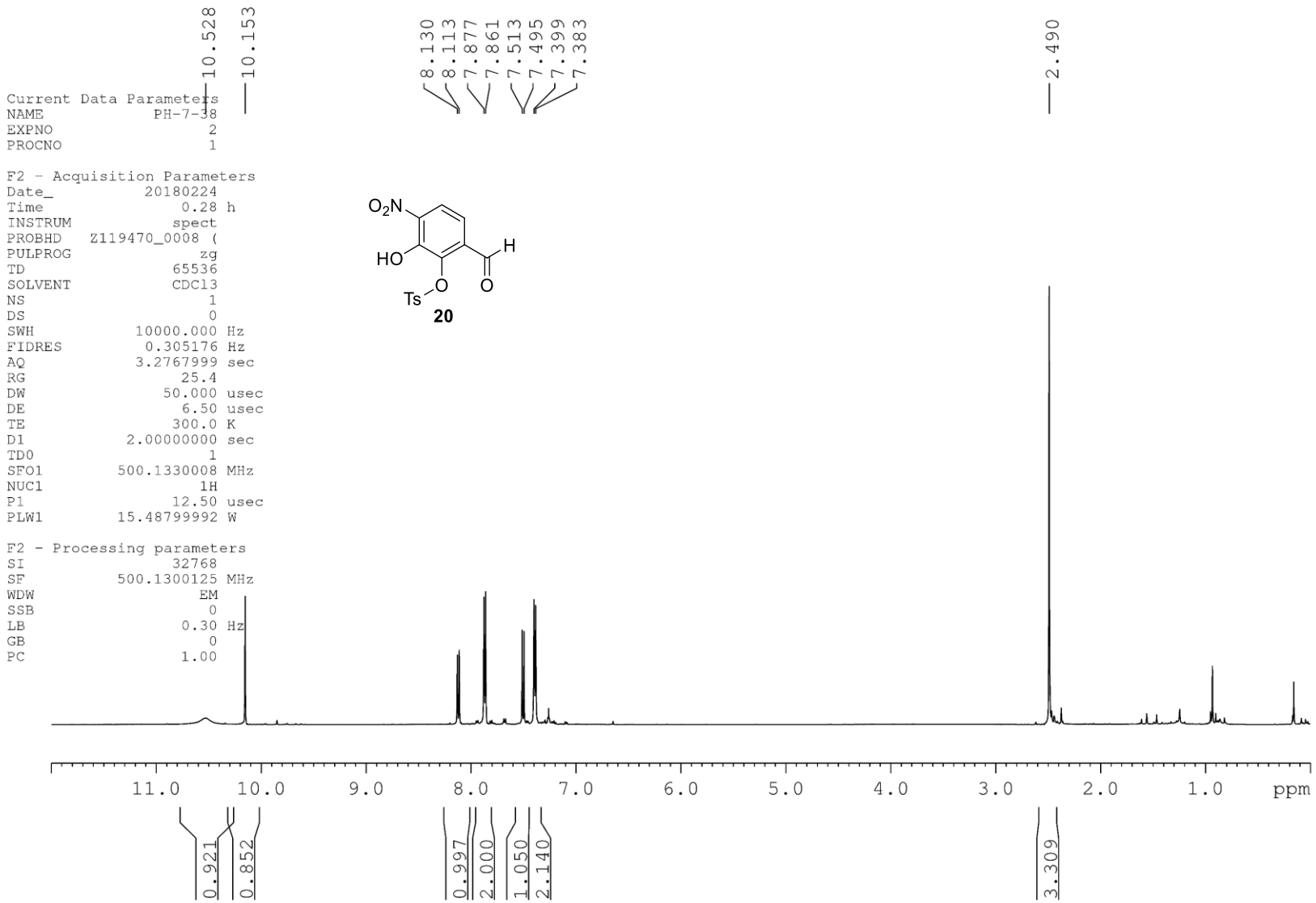

Current Data Parameters
NAME PH-5-87
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 12.17 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 440
DS 4
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6228303 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6127580 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
171.449
168.976
165.806
143.688
143.643
141.829
141.464
132.428
130.974
127.957
127.236
126.006
125.031
120.198
119.187
118.303
82.530
67.512
65.597
52.022
47.254
37.387
28.149
O
O^{t}Bu
FmocHN
O
HN
OAllyl
O
S1
220
200
180
160
140
120
100
80
60
40
20
0
ppm

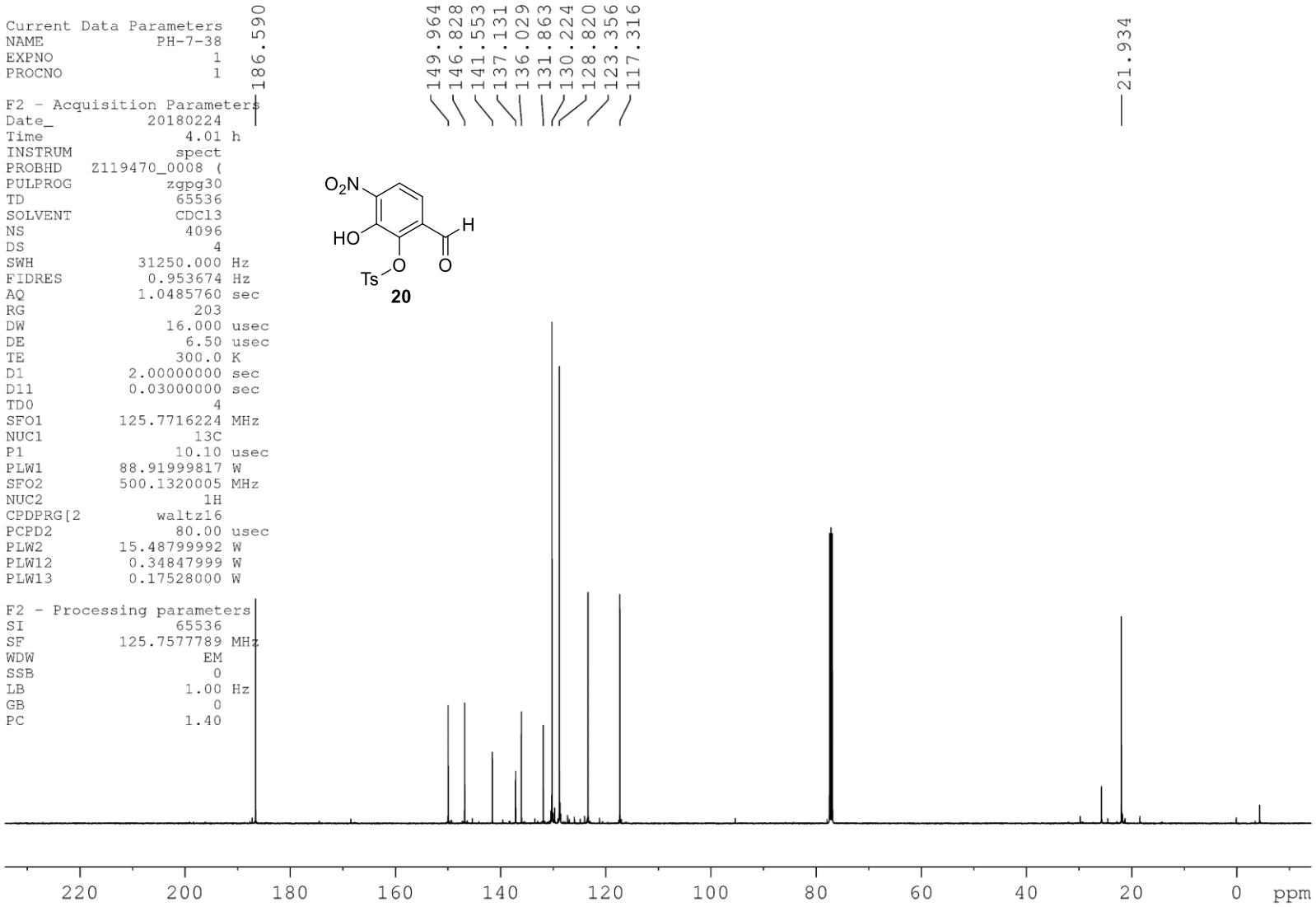

7.825 7.806 7.689 7.670 7.392 7.373 7.355 7.291 7.281 7.275 7.150 7.130 6.112 6.098 6.085 6.072 6.069 6.055 6.042 6.029 6.015 5.429 5.425 5.386 5.382 5.266 5.262 5.240 5.236 4.814 4.810 4.800 4.797 4.790 4.786 4.783 4.434 4.408 4.390 4.375 4.357 4.331 4.240 4.222 4.205 3.076 3.060 3.034 3.018 2.931 2.913 2.889 2.872
Current Data Parameters
NAME PH-5-89
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 19.31 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 57
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OH
FmocHN
O
HN
OAllyl
O
6
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 ppm
1.000 2.214 4.283 2.147 2.146 2.055 0.961 1.021 1.035 1.043 3.183 2.140 1.108 1.005 1.097

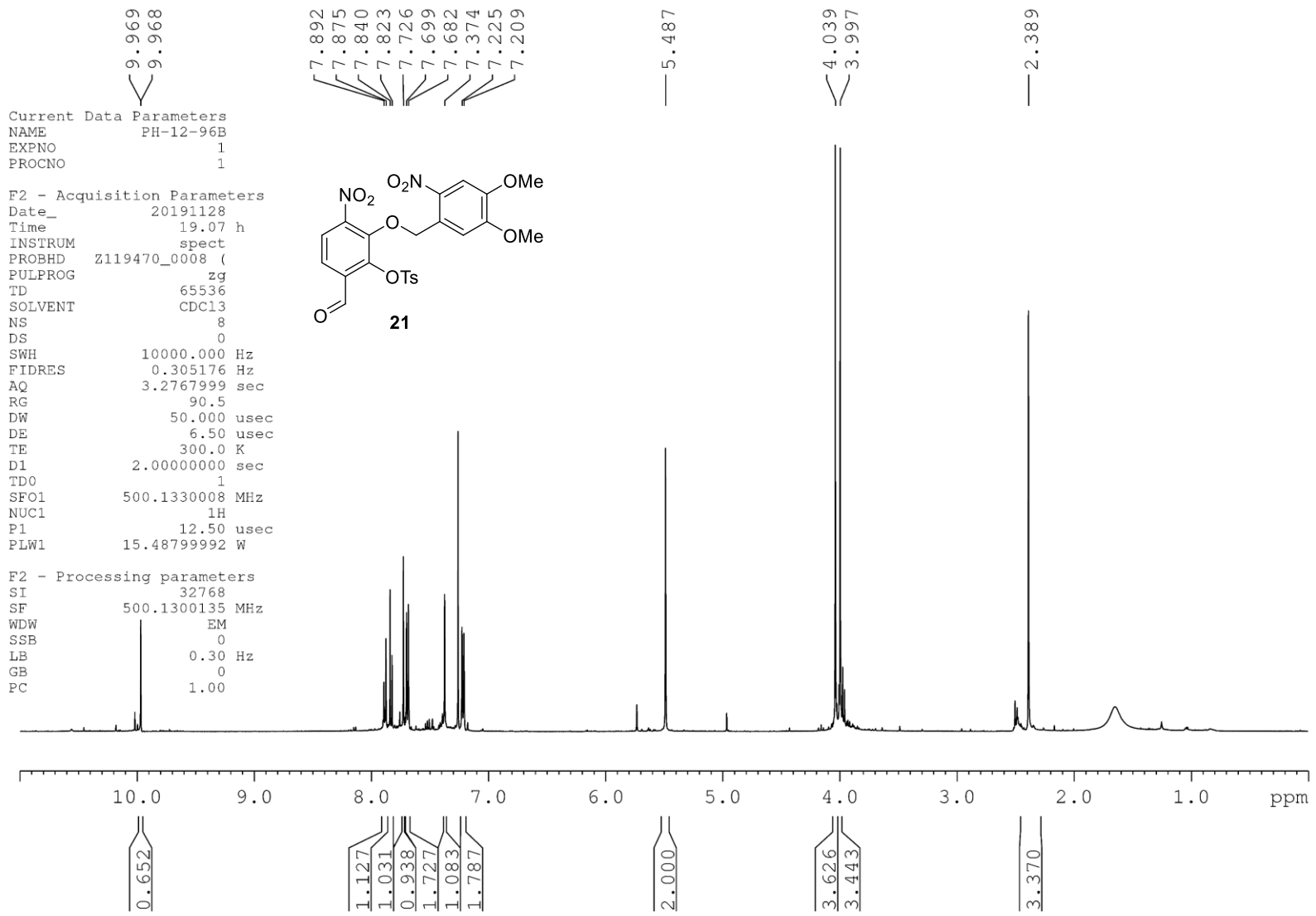

Current Data Parameters
NAME PH-5-89
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 20.48 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1346
DS 4
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6228303 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6126896 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
172.389
170.608
165.915
157.218
144.825
144.799
143.960
141.998
133.664
131.194
128.474
127.874
126.056
126.020
125.996
120.732
119.838
118.026
67.522
65.739
53.414
47.879
36.484
O
OH
O
FmocHN
HN
OAllyl
O
6
220
200
180
160
140
120
100
80
60
40
20
0
ppm

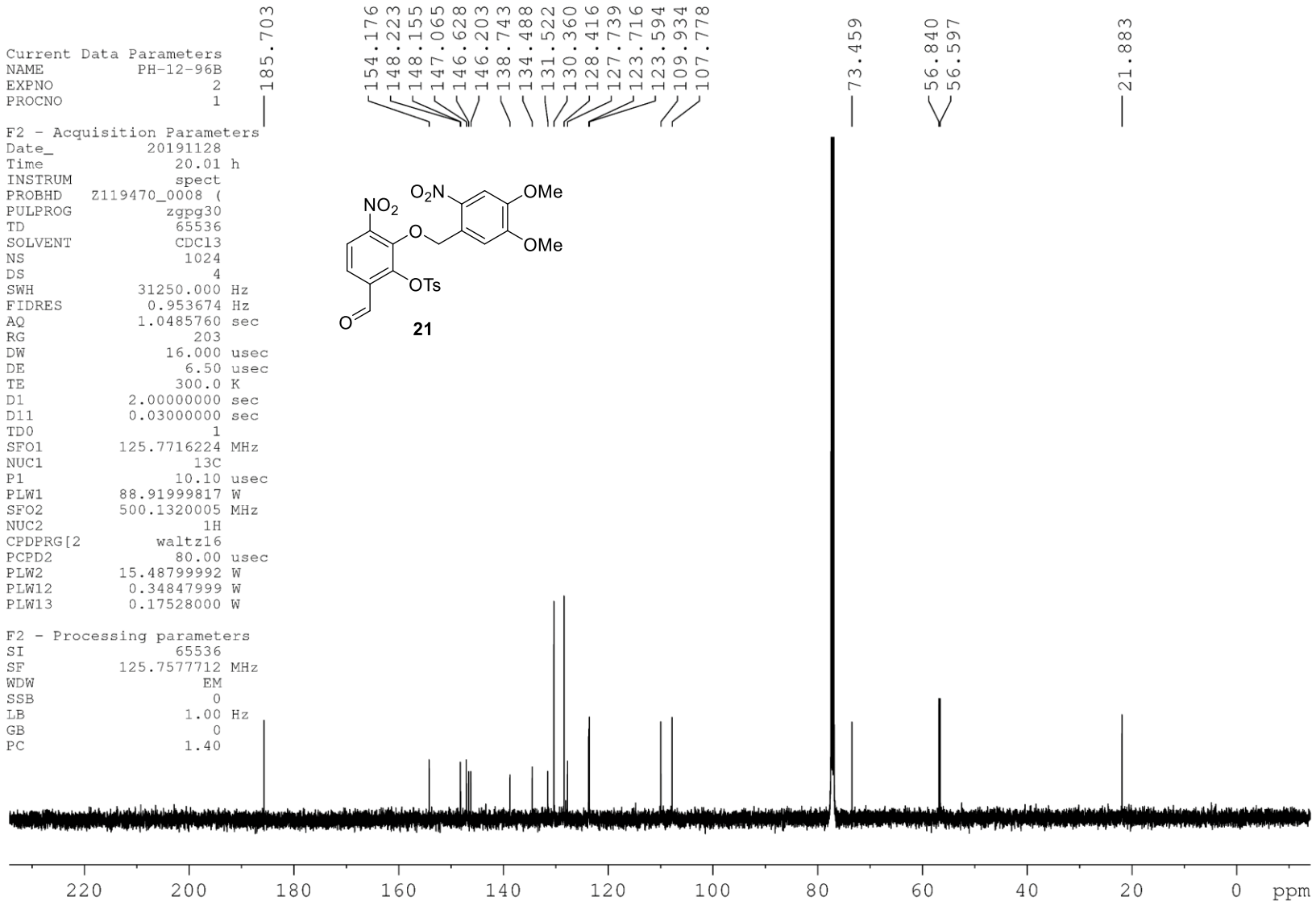

9.877
7.714
7.693
7.505
7.501
7.486
7.482
7.346
7.325
7.292
7.272
7.252
7.196
7.192
7.176
7.172
2.439
0.976
0.194
Current Data Parameters
NAME PH-7-05A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171025
Time 13.14 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 8
DS 0
SWH 8000.000 Hz
FIDRES 0.332806 Hz
AQ 3.0047500 sec
RG 7.12
DW 62.500 usec
DE 10.20 usec
TE 300.0 K
D1 5.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBS
O
H
O
Ts
O
O
19
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
3.000
6.000
5.000
4.000
2.000
8.000
3.000
3.000
7.000

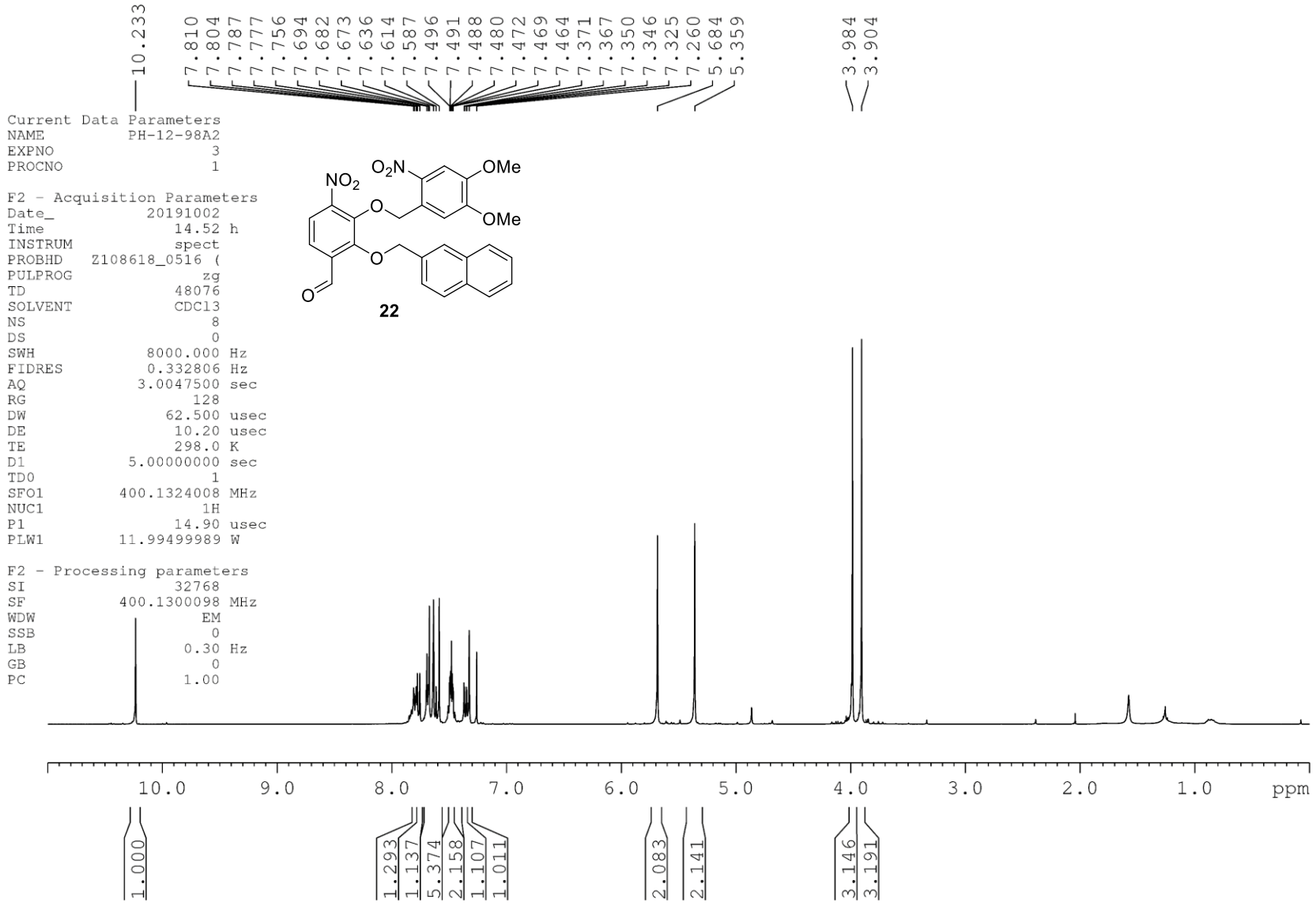

187.211
149.440
145.991
142.423
132.101
131.507
129.958
129.653
128.445
127.685
126.962
120.427
25.589
21.550
18.259
-4.493
Current Data Parameters
NAME PH-7-05A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171025
Time 13.19 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6228303 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TBS
O
H
O
Ts
O
O
19
220
200
180
160
140
120
100
80
60
40
20
0
ppm

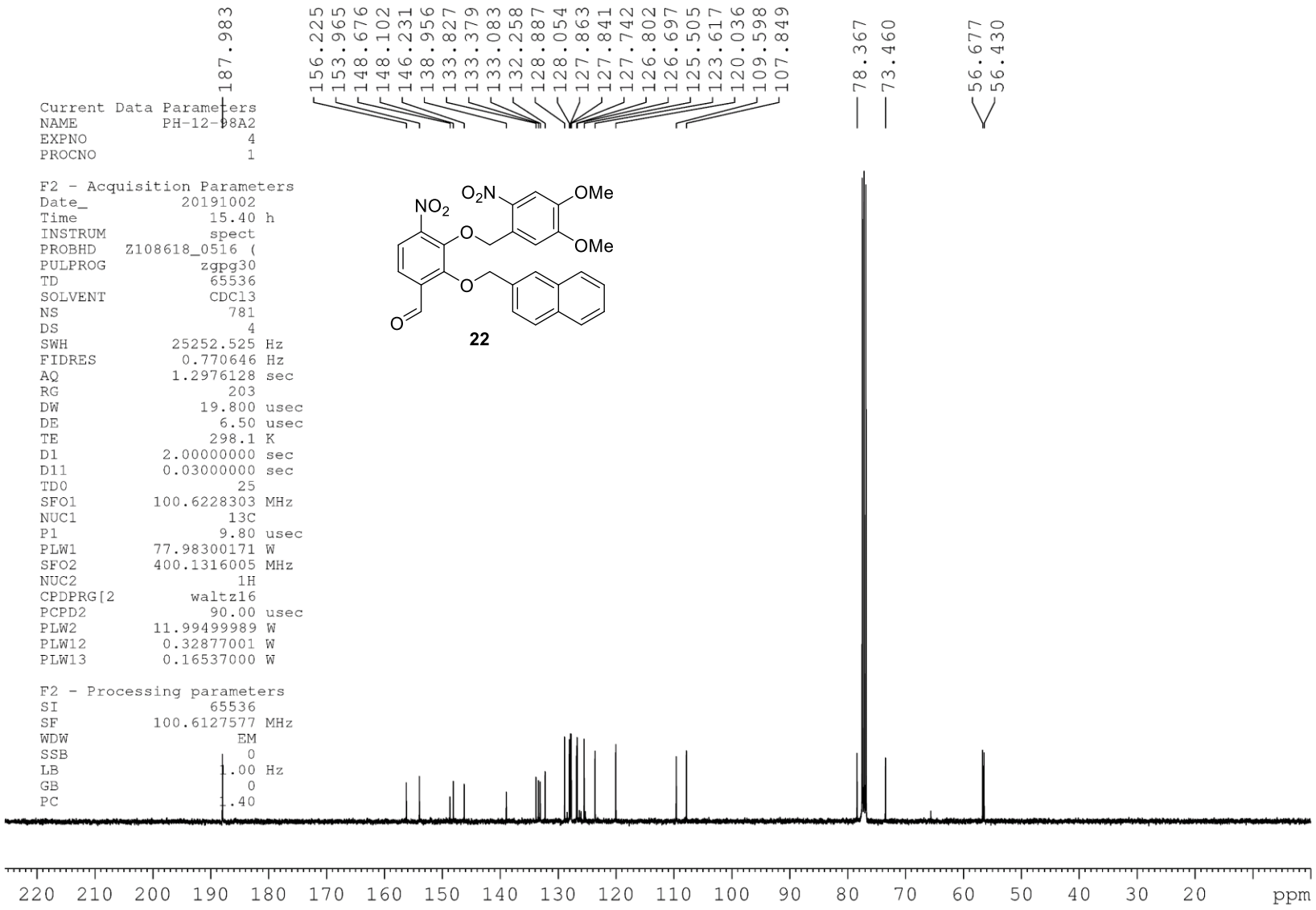

10.528
10.153
8.130
8.113
7.877
7.861
7.513
7.495
7.399
7.383
2.490
Current Data Parameters
NAME PH-7-38
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180224
Time 0.28 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 25.4
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O2N
HO
H
O
Ts
O
20
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
0.921
0.852
0.997
2.000
1.050
2.140
3.309

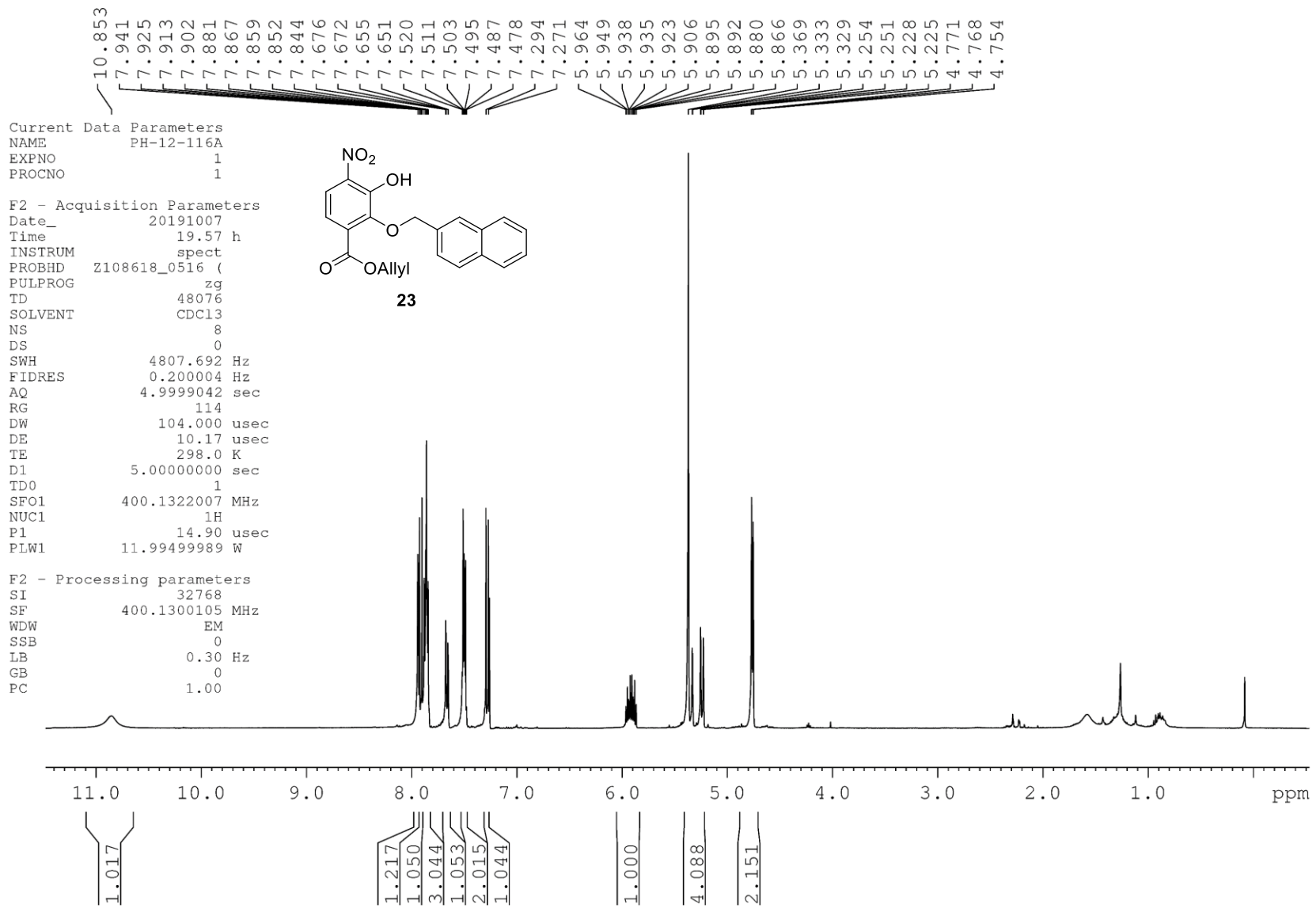

Current Data Parameters
NAME PH-7-38
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180224
Time 4.01 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 4096
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 4
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577789 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
186.590
149.964
146.828
141.553
137.131
136.029
131.863
130.224
128.820
123.356
117.316
21.934
O2N
HO
H
O
Ts
O
20
220
200
180
160
140
120
100
80
60
40
20
0
ppm

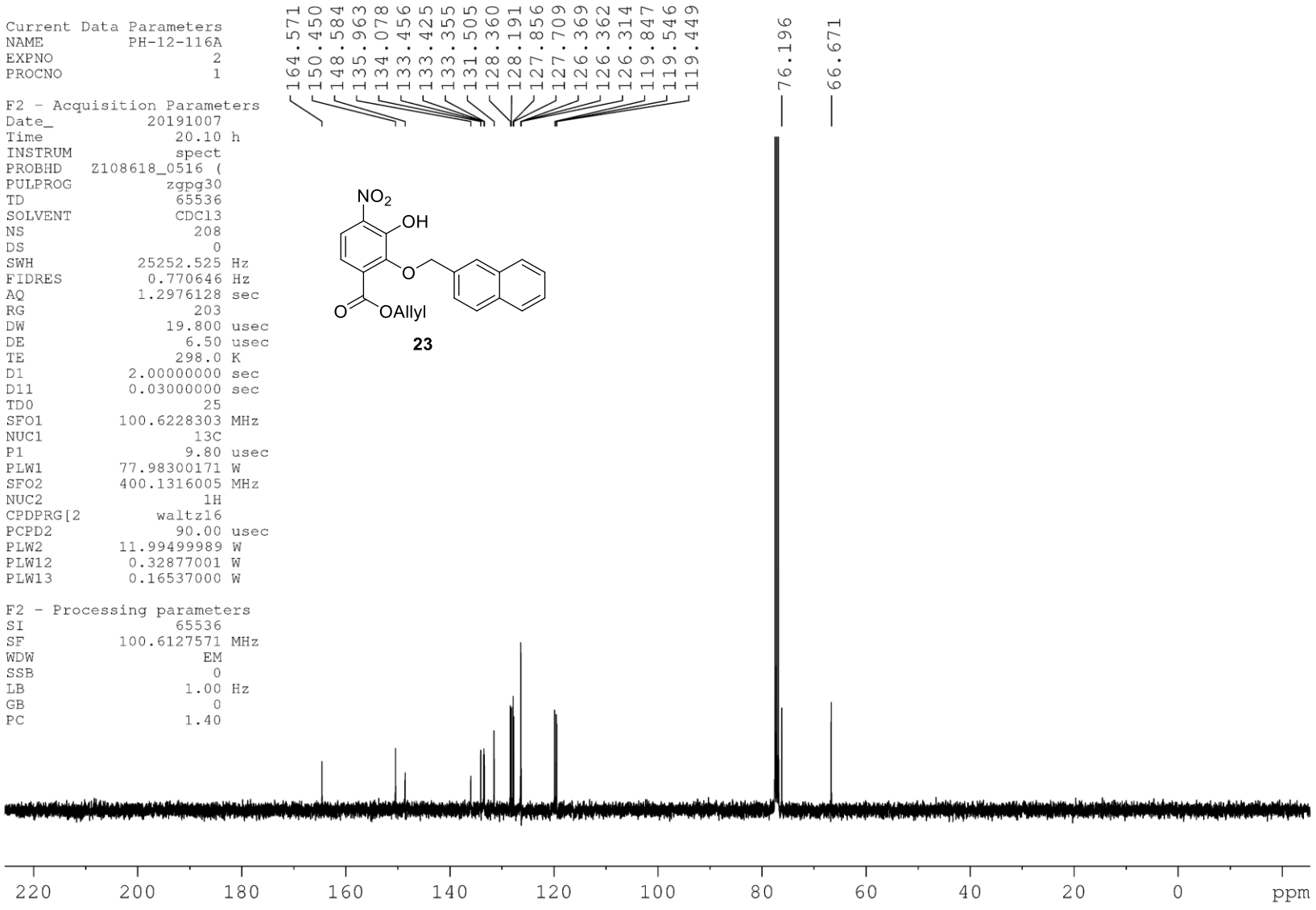

9.969
9.968
7.892
7.875
7.840
7.823
7.726
7.699
7.682
7.374
7.225
7.209
5.487
4.039
3.997
2.389
Current Data Parameters
NAME PH-12-96B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191128
Time 19.07 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 90.5
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
O2N
OMe
OMe
O
OTs
O
21
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
0.652
1.127
1.031
0.938
1.727
1.083
1.787
2.000
3.626
3.443
3.370

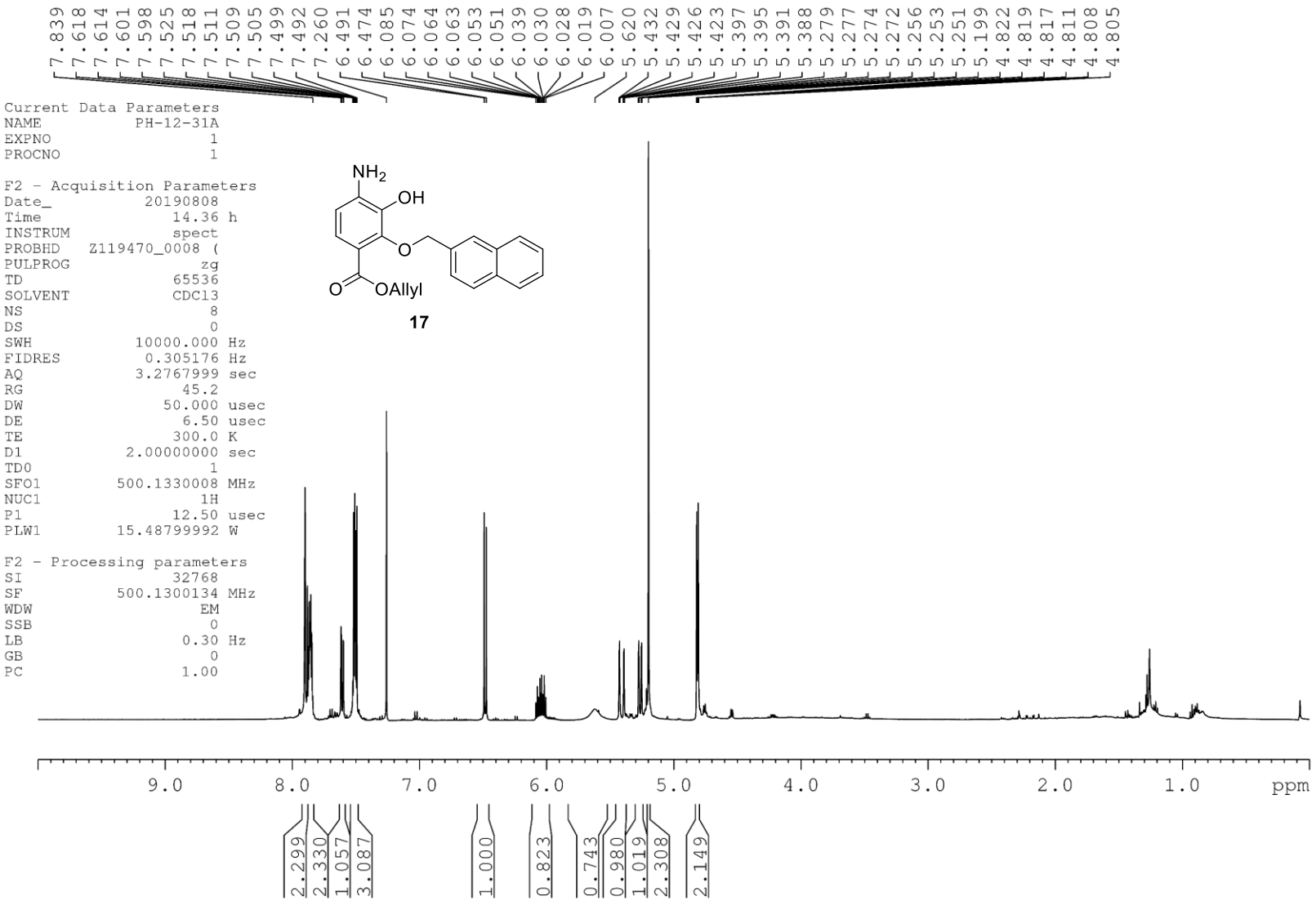

185.703
154.176
148.223
148.155
147.065
146.628
146.203
138.743
134.488
131.522
130.360
128.416
127.739
123.716
123.594
109.934
107.778
73.459
56.840
56.597
21.883
Current Data Parameters
NAME PH-12-96B
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191128
Time 20.01 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O2N
NO2
OMe
OMe
O
OTs
O
21
220
200
180
160
140
120
100
80
60
40
20
0
ppm


10.233
7.810
7.804
7.787
7.777
7.756
7.694
7.682
7.673
7.636
7.614
7.587
7.496
7.491
7.488
7.480
7.472
7.469
7.464
7.371
7.367
7.350
7.346
7.325
7.260
5.684
5.359
3.984
3.904
Current Data Parameters
NAME PH-12-98A2
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191002
Time 14.52 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 8
DS 0
SWH 8000.000 Hz
FIDRES 0.332806 Hz
AQ 3.0047500 sec
RG 128
DW 62.500 usec
DE 10.20 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
O2N
OMe
OMe
O
O
O
22
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
1.000
1.293
1.137
5.374
2.158
1.107
1.011
2.083
2.141
3.146
3.191


187.983
156.225
153.965
148.676
148.102
146.231
138.956
133.827
133.379
133.083
132.258
128.887
128.054
127.863
127.841
127.742
126.802
126.697
125.505
123.617
120.036
109.598
107.849
78.367
73.460
56.677
56.430
Current Data Parameters
NAME PH-12-08A2
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191002
Time 15.40 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 781
DS 4
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6228303 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6127577 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NO2
O2N
OMe
OMe
O
O
O
22
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

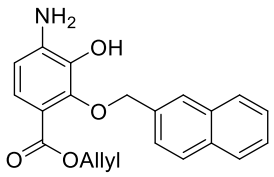

10.853
7.941
7.925
7.913
7.902
7.881
7.867
7.859
7.852
7.844
7.676
7.672
7.655
7.651
7.520
7.511
7.503
7.495
7.487
7.478
7.294
7.271
5.964
5.949
5.938
5.935
5.923
5.906
5.895
5.892
5.880
5.866
5.369
5.333
5.329
5.254
5.251
5.228
5.225
4.771
4.768
4.754
Current Data Parameters
NAME PH-12-116A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191007
Time 19.57 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 8
DS 0
SWH 4807.692 Hz
FIDRES 0.200004 Hz
AQ 4.9999042 sec
RG 114
DW 104.000 usec
DE 10.17 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1
SFO1 400.1322007 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 32768
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
OH
O
O
OAllyl
23
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
1.017
1.217
1.050
3.044
1.053
2.015
1.044
1.000
4.088
2.151


Current Data Parameters
NAME PH-12-116A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191007
Time 20.10 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 208
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6228303 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6127571 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
164.571
150.450
148.584
135.963
134.078
133.456
133.425
133.355
131.505
128.360
128.191
127.856
127.709
126.369
126.362
126.314
119.847
119.546
119.449
76.196
66.671
NO2
OH
O
O
OAllyl
23
220
200
180
160
140
120
100
80
60
40
20
0
ppm

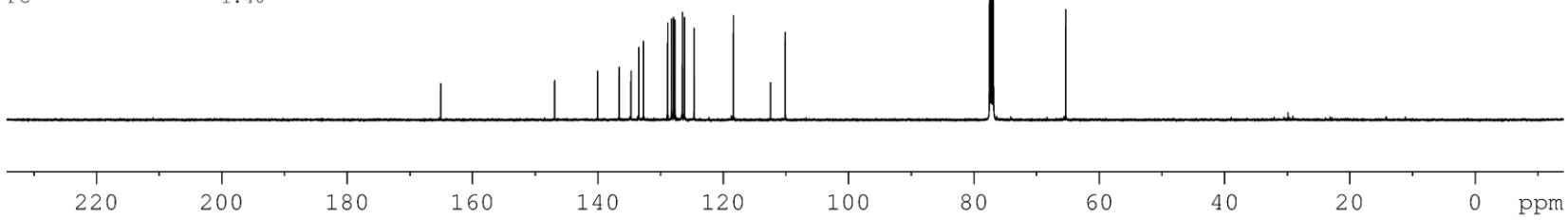

7.839
7.618
7.614
7.601
7.598
7.525
7.518
7.511
7.509
7.505
7.499
7.492
7.260
6.491
6.474
6.085
6.074
6.064
6.063
6.053
6.051
6.039
6.030
6.028
6.019
6.007
5.620
5.432
5.429
5.426
5.423
5.397
5.395
5.391
5.388
5.279
5.277
5.274
5.272
5.256
5.253
5.251
5.199
4.822
4.819
4.817
4.811
4.808
4.805
Current Data Parameters
NAME PH-12-31A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190808
Time 14.36 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 45.2
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH2
OH
O
O
OAllyl
17
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
2.299
2.330
1.057
3.087
1.000
0.823
0.743
0.980
1.019
2.308
2.149

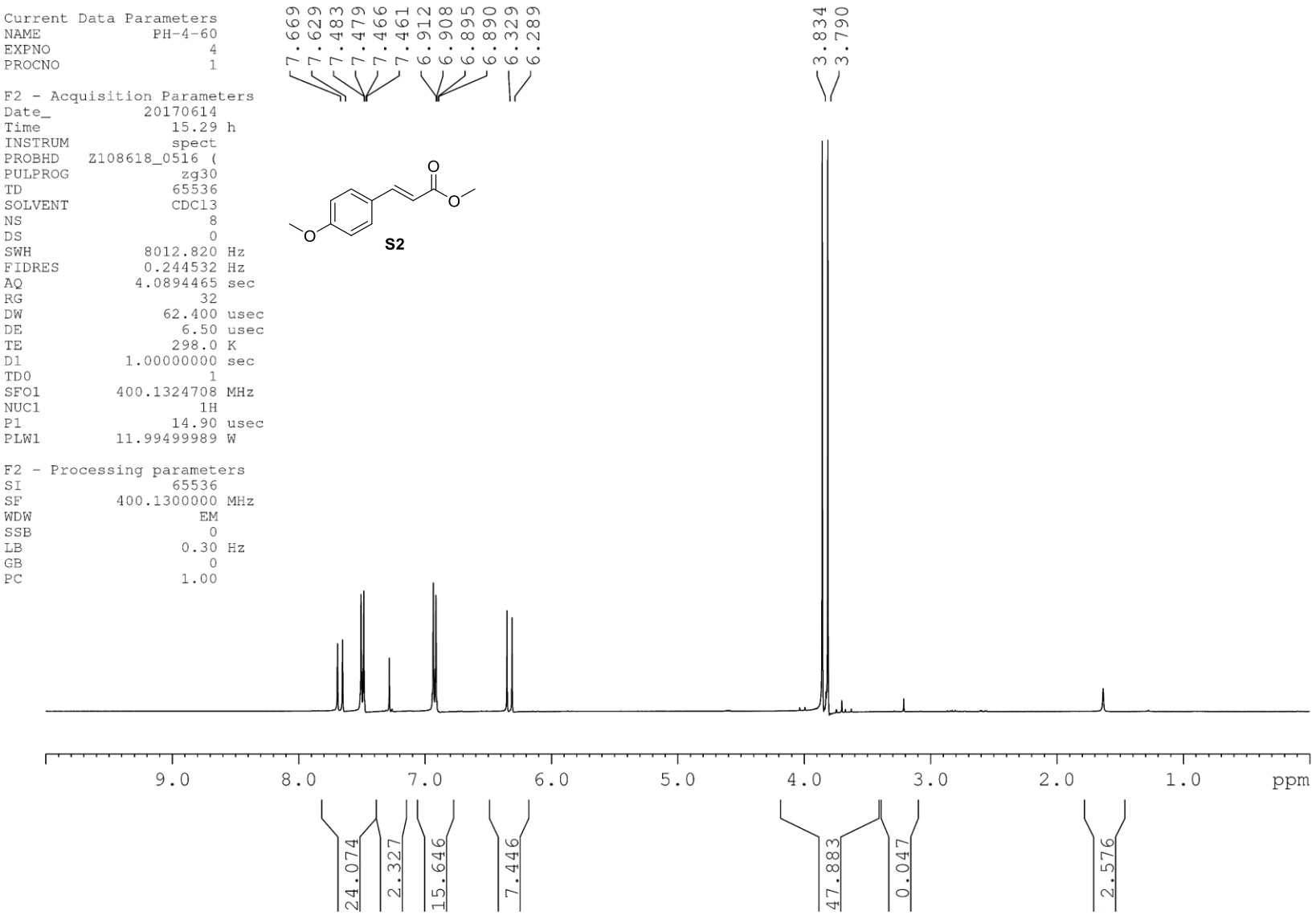
```
Current Data Parameters
NAME          PH-12-31A
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameters
Date_          20190809
Time               8.41 h
INSTRUM           spect
PROBHD   Z119470_0008 (
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                14398
DS                    4
SWH           31250.000 Hz
FIDRES         0.953674 Hz
AQ            1.0485760 sec
RG                  203
DW               16.000 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  25
SFO1        125.7716224 MHz
NUC1               13C
P1               10.10 usec
PLW1        88.91999817 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2        15.48799992 W
PLW12        0.34847999 W
PLW13        0.17528000 W

F2 - Processing parameters
SI                65536
SF          125.7577723 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

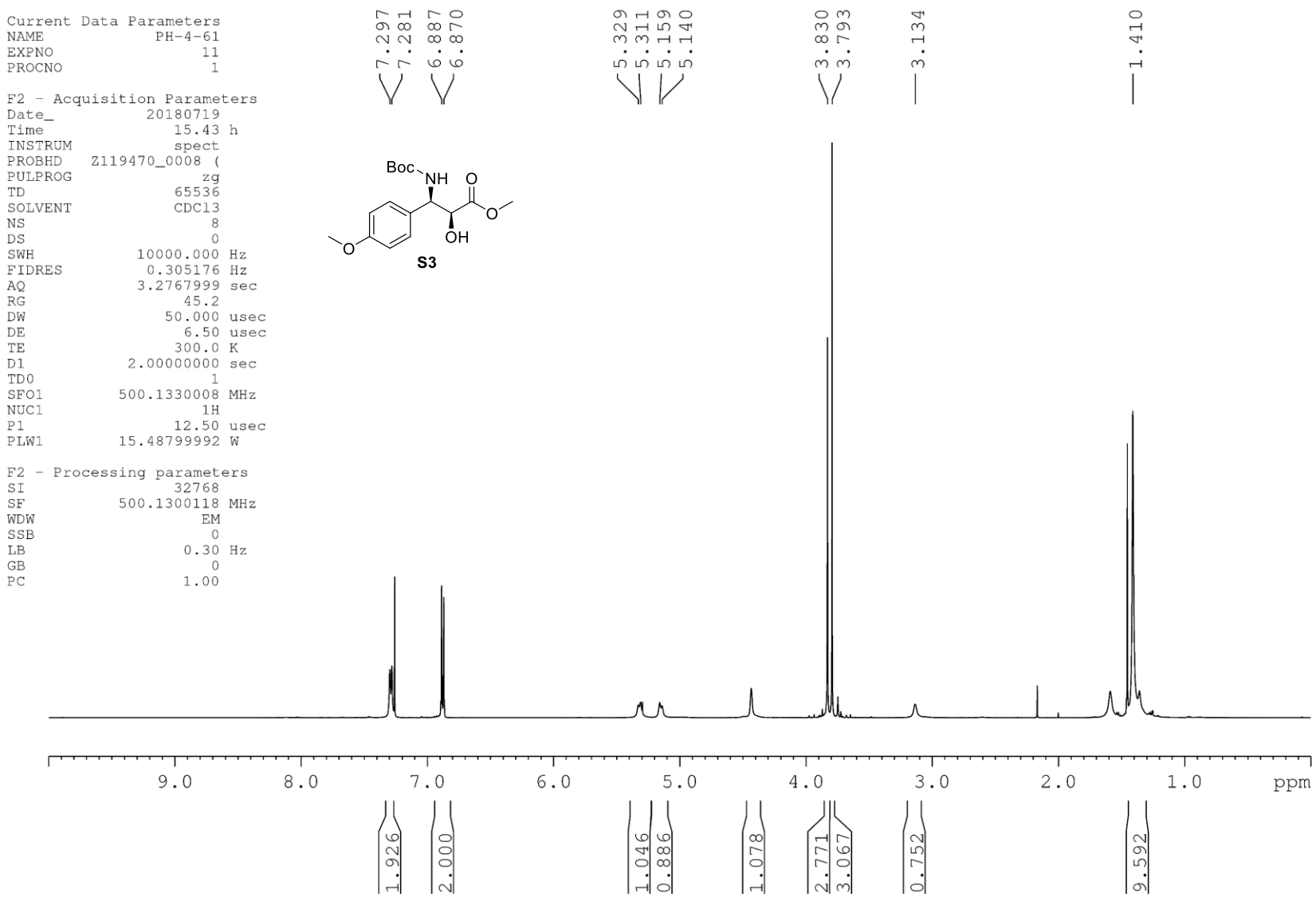
165.087
146.897
140.032
136.610
134.697
133.462
132.735
128.845
128.245
127.922
127.701
126.507
126.490
126.166
124.643
118.348
112.439
110.099
77.507
65.325

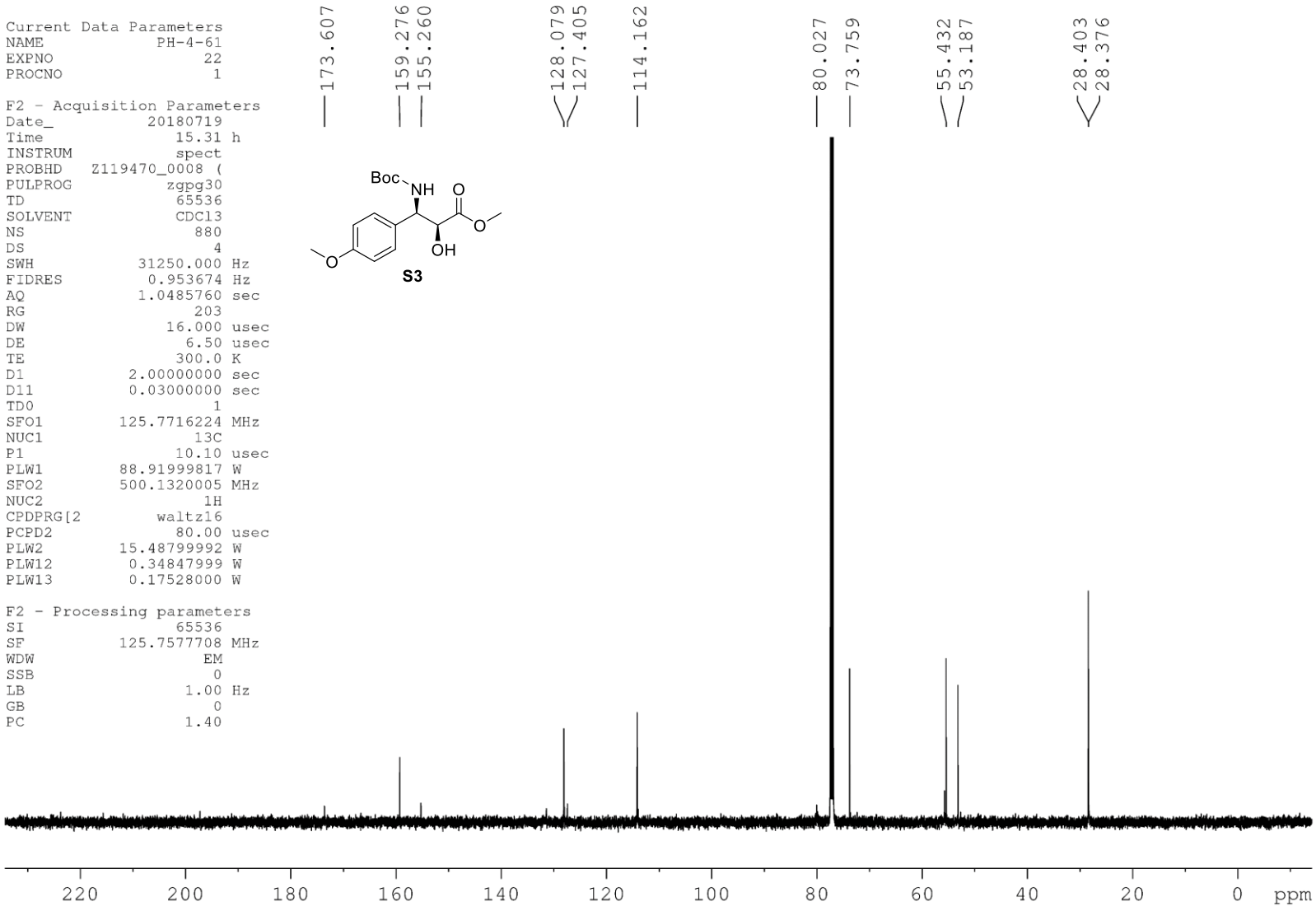


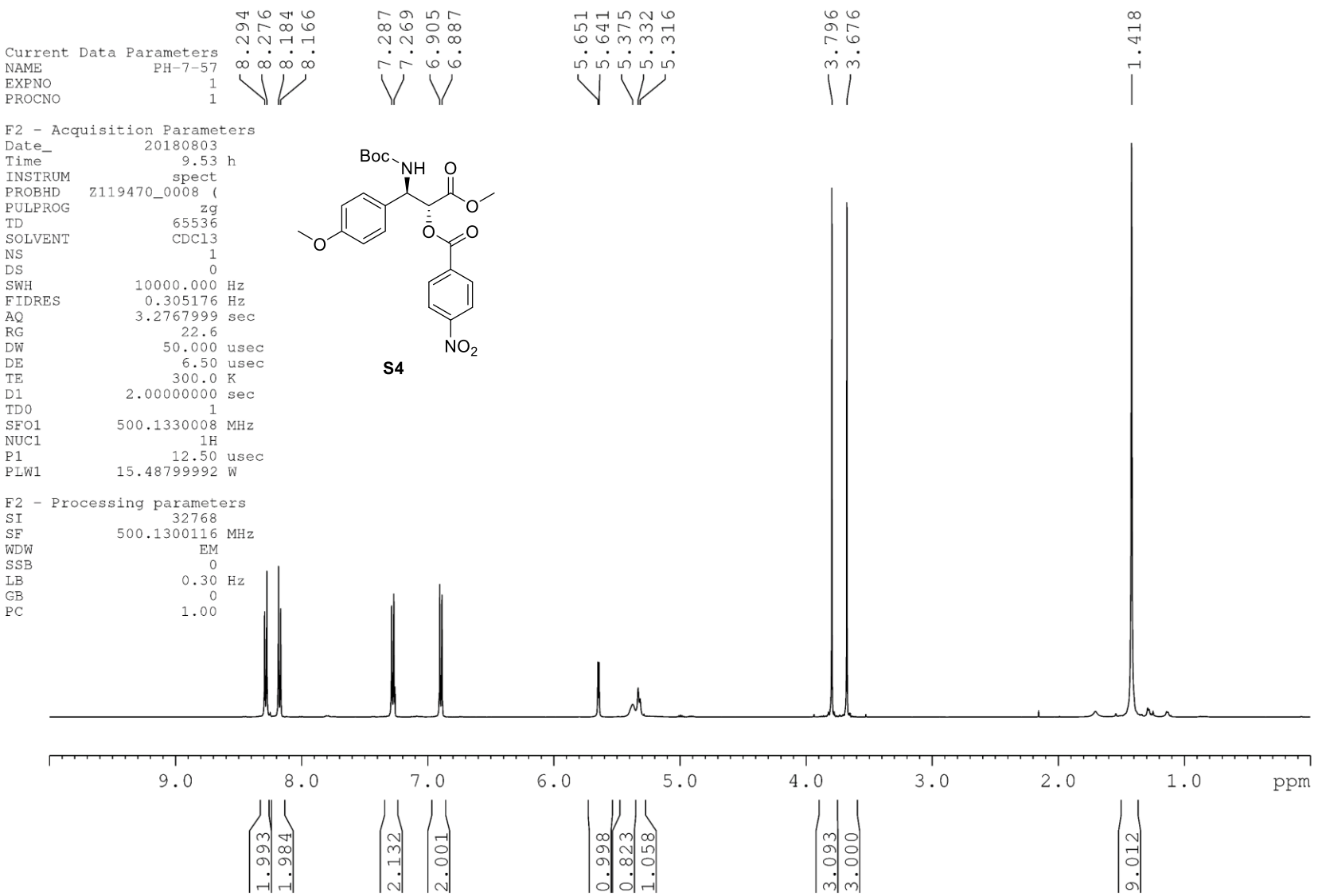
17

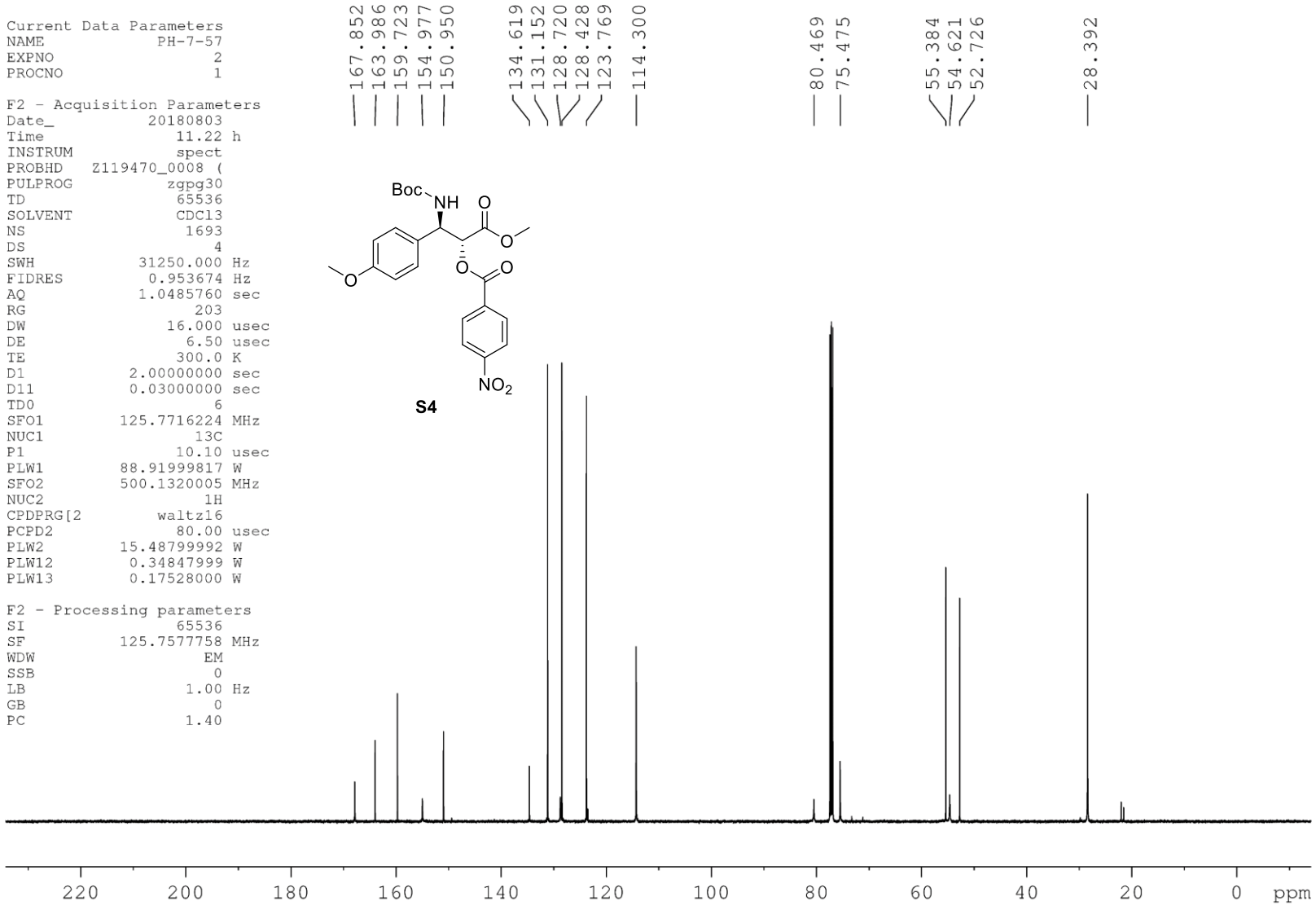


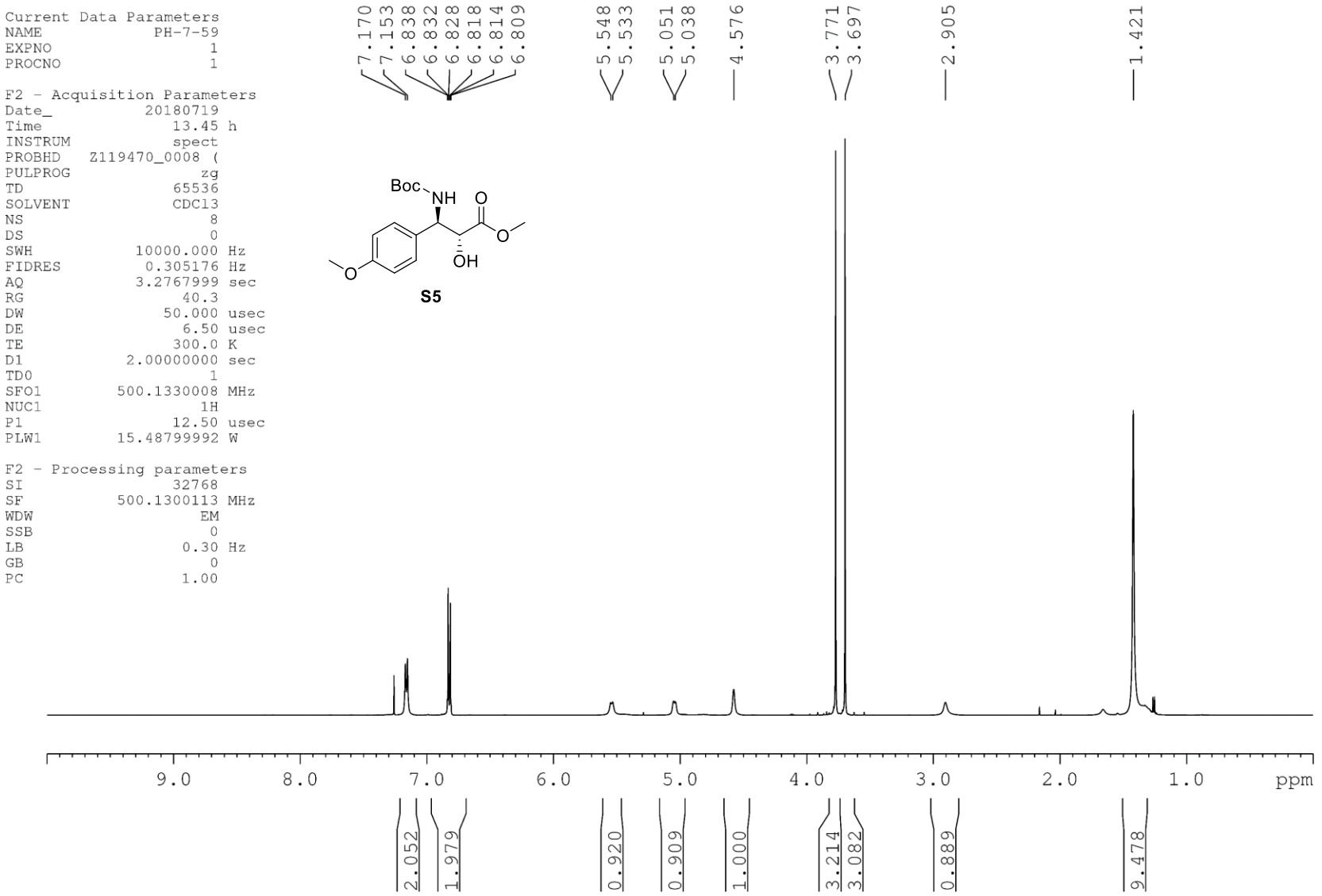

Current Data Parameters
NAME PH-4-60
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170614
Time 15.29 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.669
7.629
7.483
7.479
7.466
7.461
6.912
6.908
6.895
6.890
6.329
6.289
3.834
3.790
S2
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
24.074
2.327
15.646
7.446
47.883
0.047
2.576

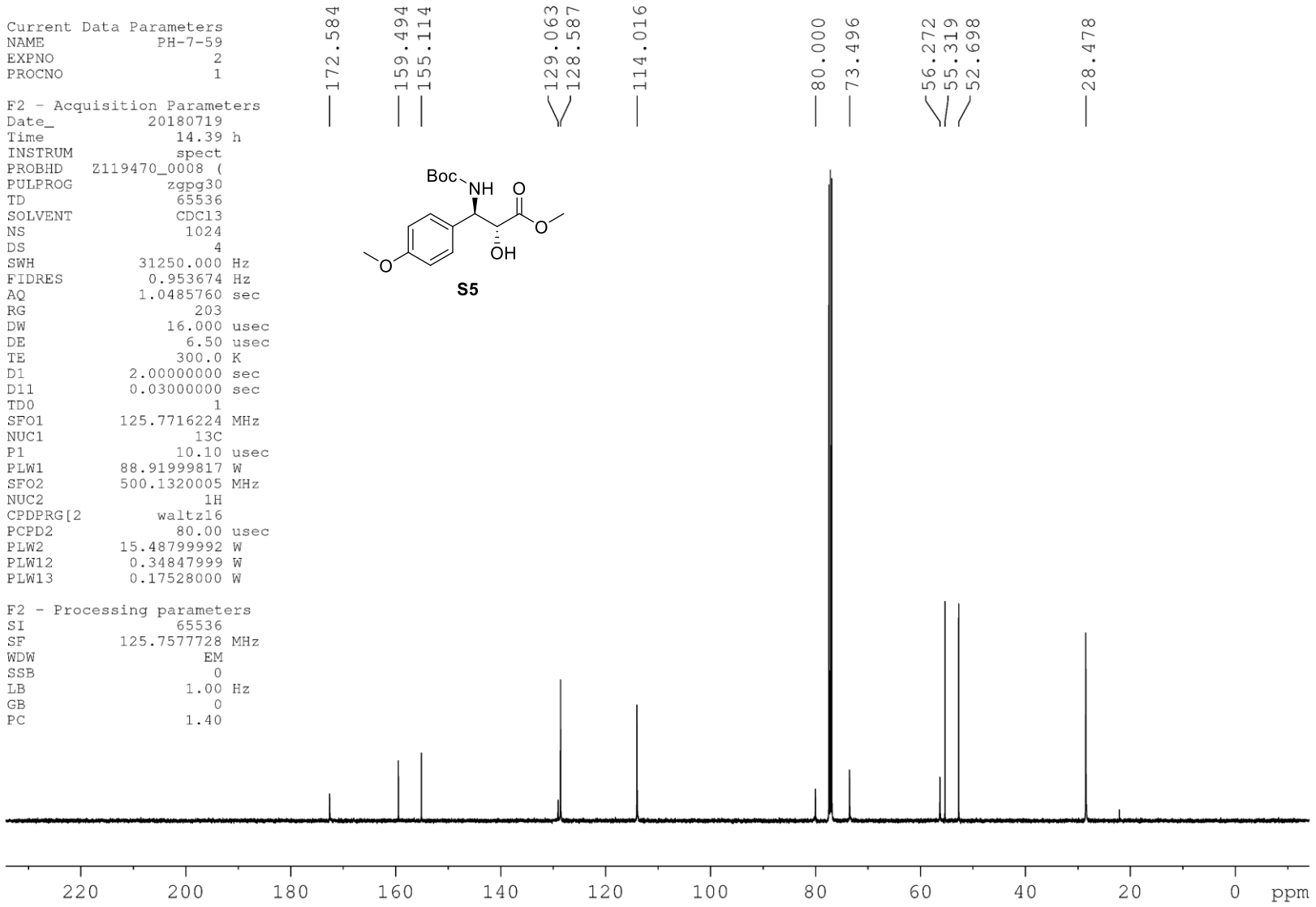

Current Data Parameters
NAME PH-4-61
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 15.43 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 45.2
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.297
7.281
6.887
6.870
5.329
5.311
5.159
5.140
3.830
3.793
3.134
1.410
Boc
NH
O
O
OH
O
S3
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
1.926
2.000
1.046
0.886
1.078
2.771
3.067
0.752
9.592

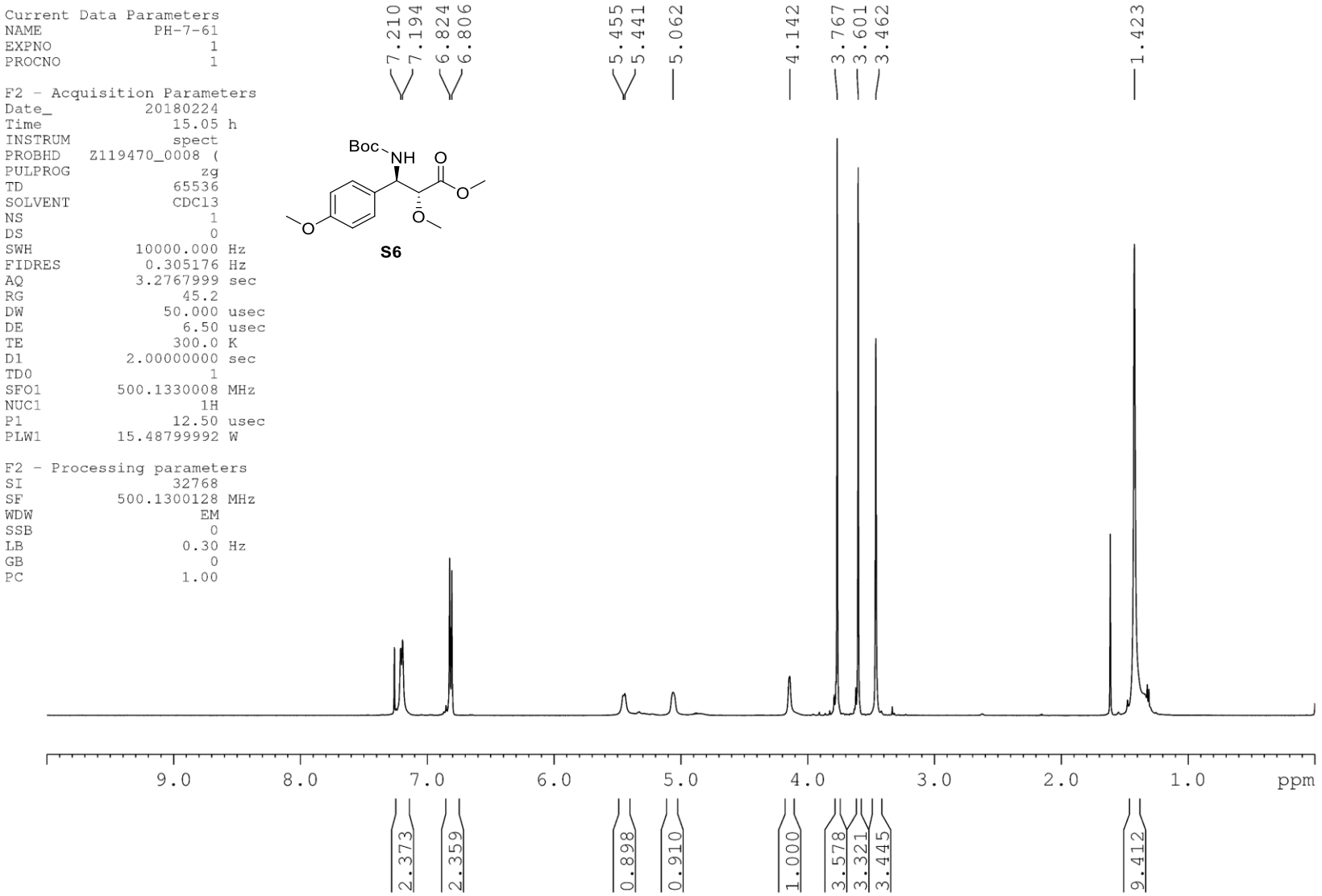

Current Data Parameters
NAME PH-4-61
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 15.31 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 880
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577708 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
173.607
159.276
155.260
128.079
127.405
114.162
80.027
73.759
55.432
53.187
28.403
28.376
Boc
NH
O
O
OH
O
S3
220
200
180
160
140
120
100
80
60
40
20
0
ppm

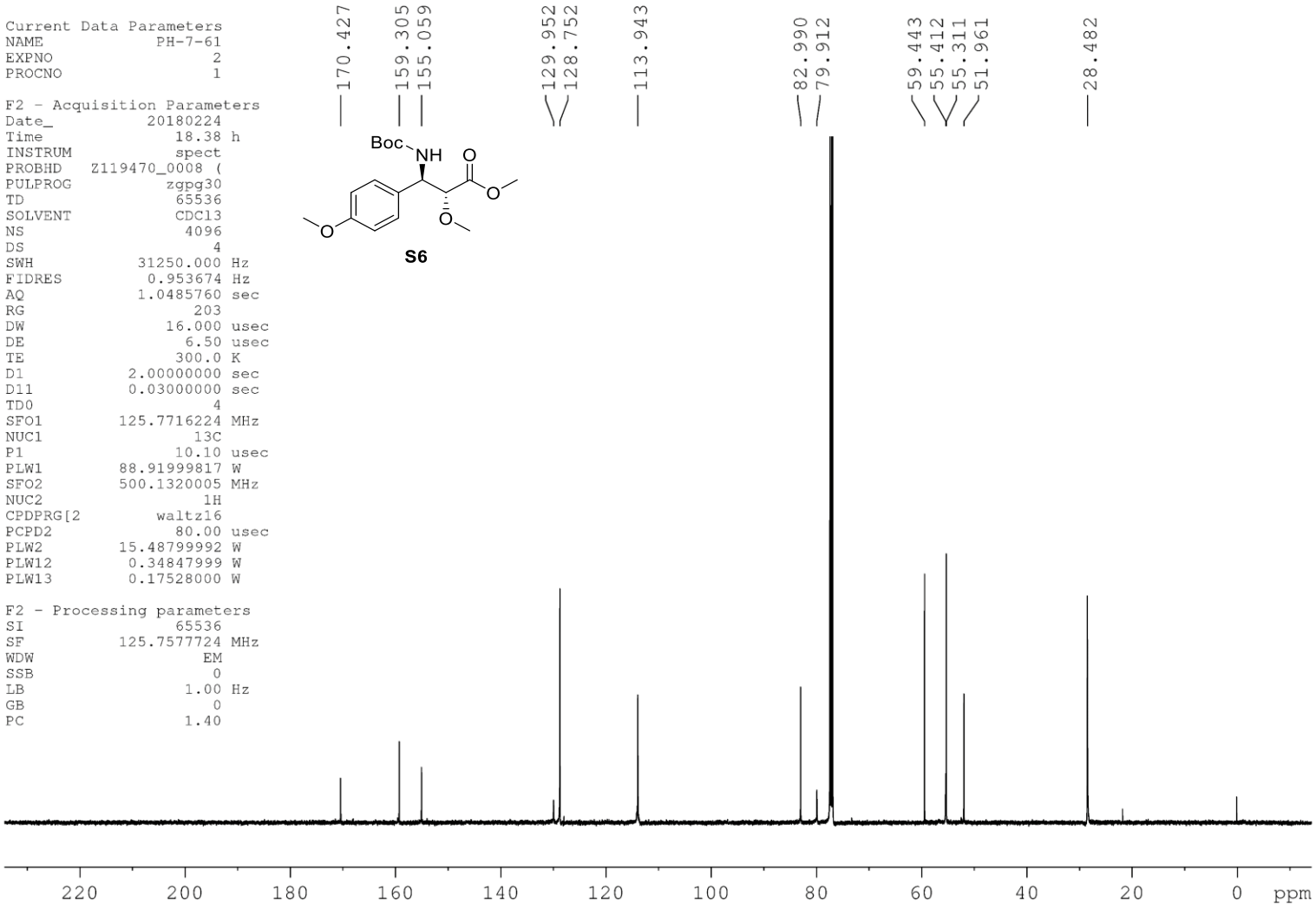

8.294
8.276
8.184
8.166
7.287
7.269
6.905
6.887
5.651
5.641
5.375
5.332
5.316
3.796
3.676
1.418
Current Data Parameters
NAME PH-7-57
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180803
Time 9.53 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 22.6
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
NH
O
O
O
O
O
NO2
S4
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
1.993
1.984
2.132
2.001
0.998
0.823
1.058
3.093
3.000
9.012

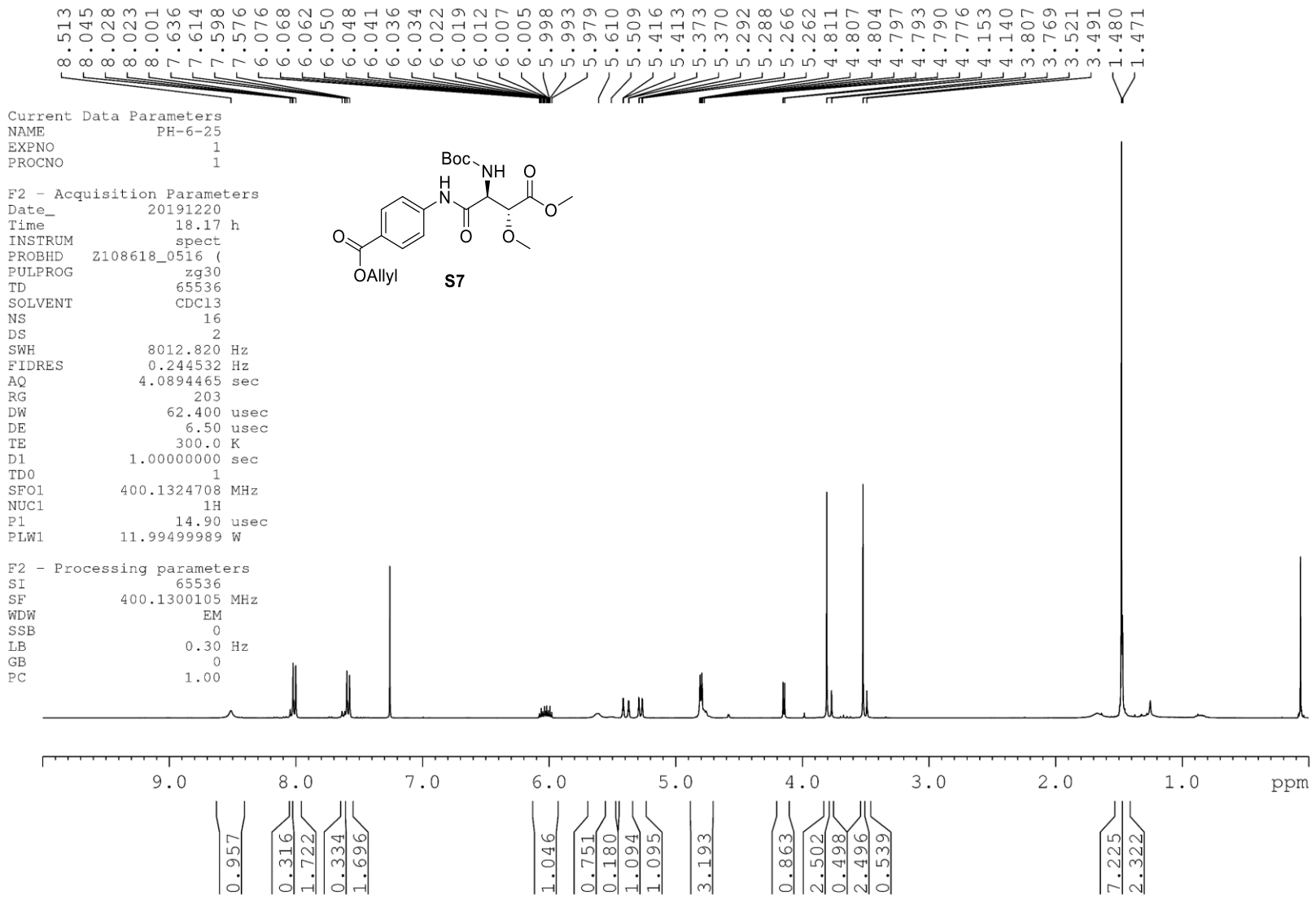

Current Data Parameters
NAME PH-7-57
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180803
Time 11.22 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1693
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 6
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577758 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
167.852
163.986
159.723
154.977
150.950
134.619
131.152
128.720
128.428
123.769
114.300
80.469
75.475
55.384
54.621
52.726
28.392
Boc
NH
O
O
O
O
O
O
NO2
S4
220
200
180
160
140
120
100
80
60
40
20
0
ppm

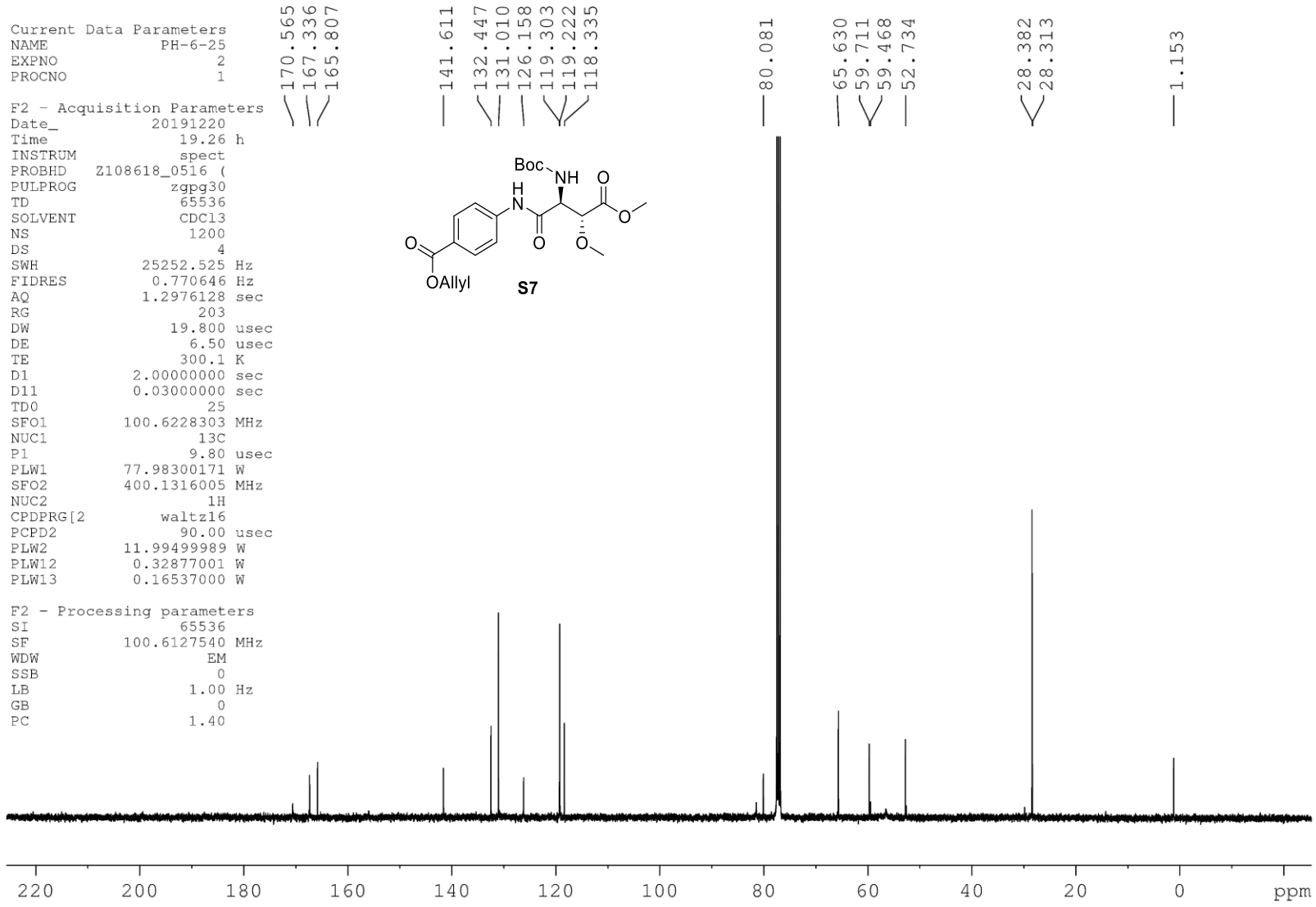

Current Data Parameters
NAME PH-7-59
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 13.45 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 40.3
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.170
7.153
6.838
6.832
6.828
6.818
6.814
6.809
5.548
5.533
5.051
5.038
4.576
3.771
3.697
2.905
1.421
Boc
NH
O
O
OH
O
S5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
2.052
1.979
0.920
0.909
1.000
3.214
3.082
0.889
9.478

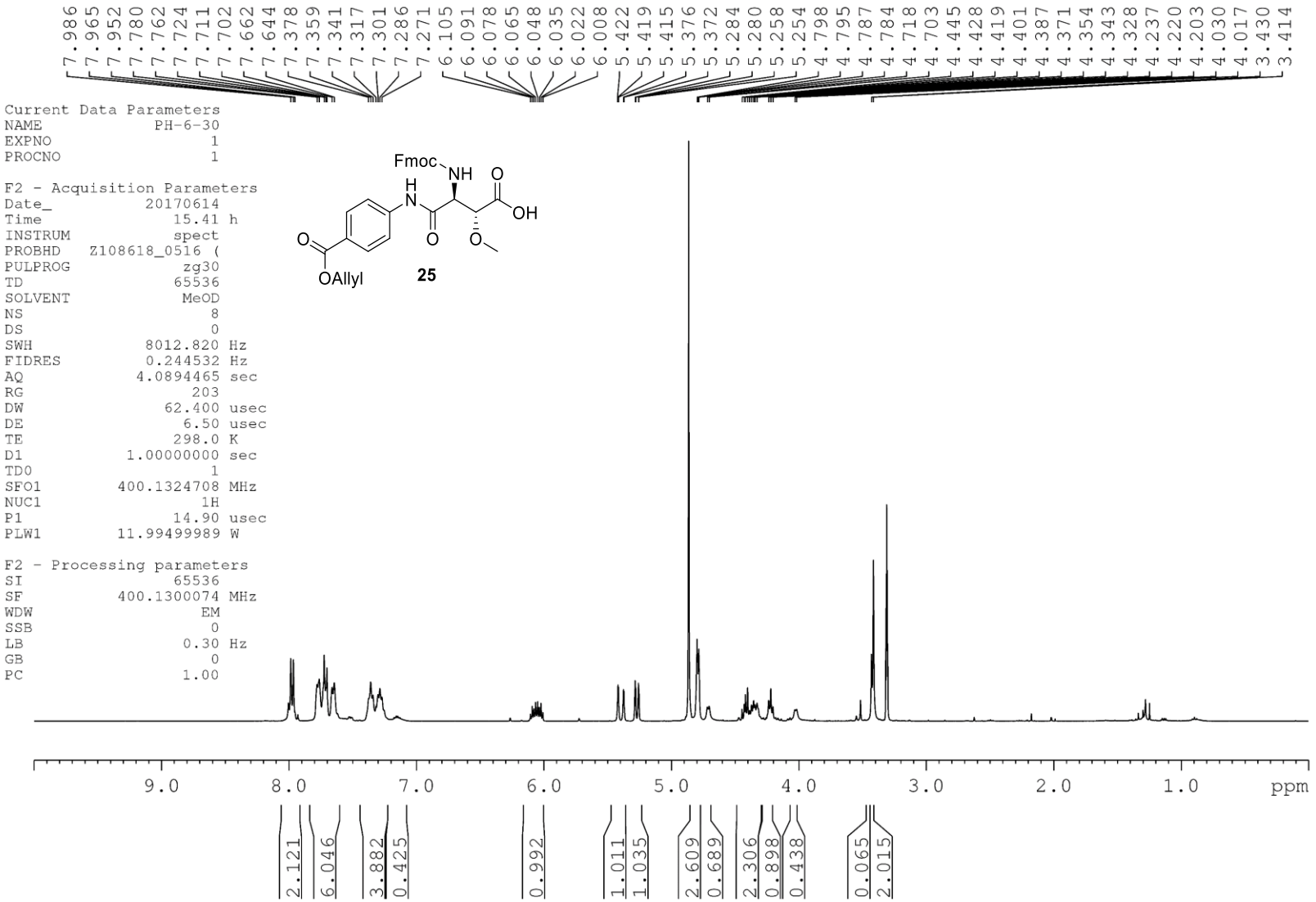

Current Data Parameters
NAME PH-7-59
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 14.39 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577728 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
172.584
159.494
155.114
129.063
128.587
114.016
80.000
73.496
56.272
55.319
52.698
28.478
Boc
NH
O
O
OH
O
S5
220
200
180
160
140
120
100
80
60
40
20
0
ppm

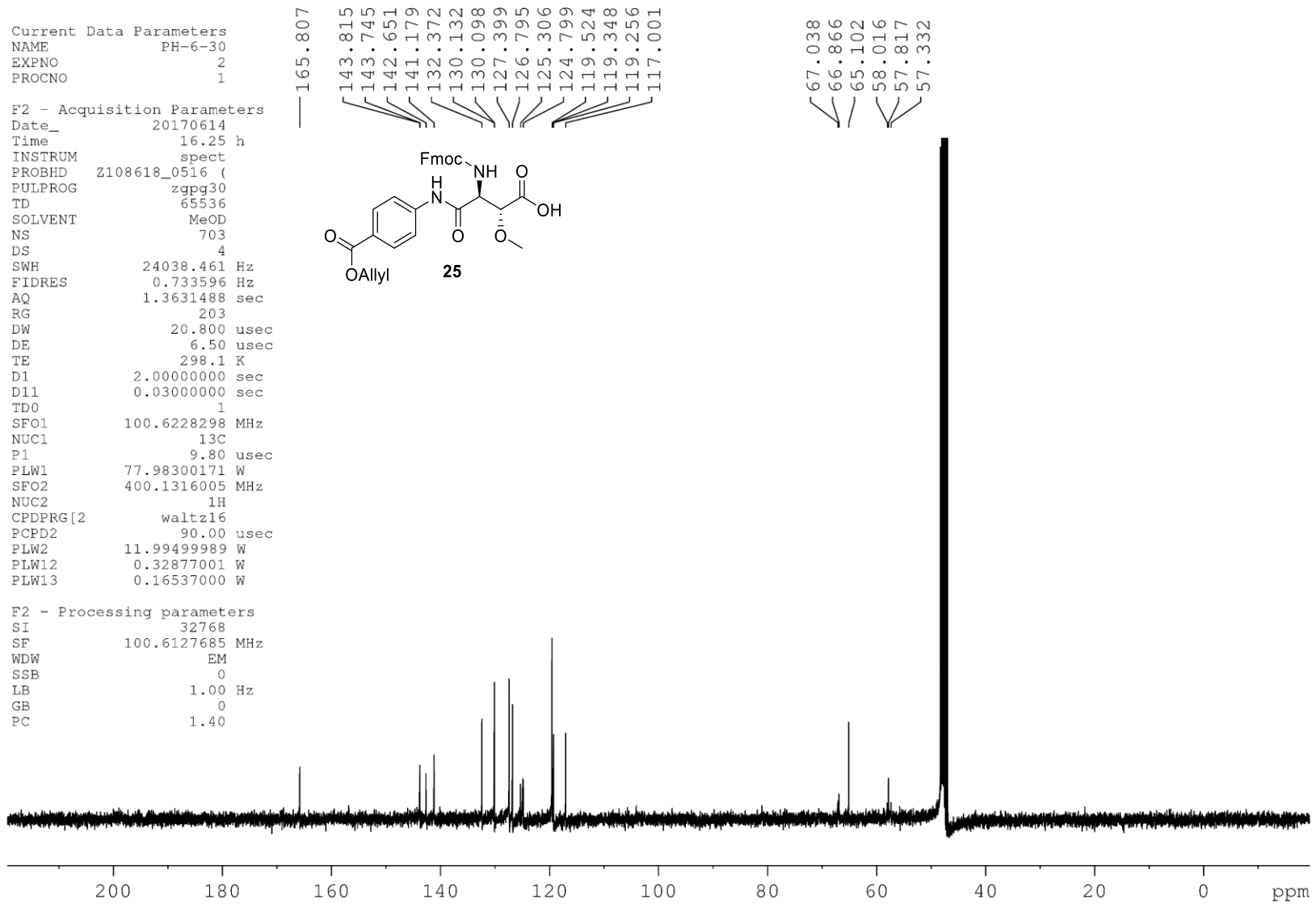

Current Data Parameters
NAME PH-7-61
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180224
Time 15.05 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 45.2
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 12.50 usec
PLW1 15.48799992 W
F2 - Processing parameters
SI 32768
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.210
7.194
6.824
6.806
5.455
5.441
5.062
4.142
3.767
3.601
3.462
1.423
Boc
NH
O
O
O
O
S6
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
2.373
2.359
0.898
0.910
1.000
3.578
3.321
3.445
9.412

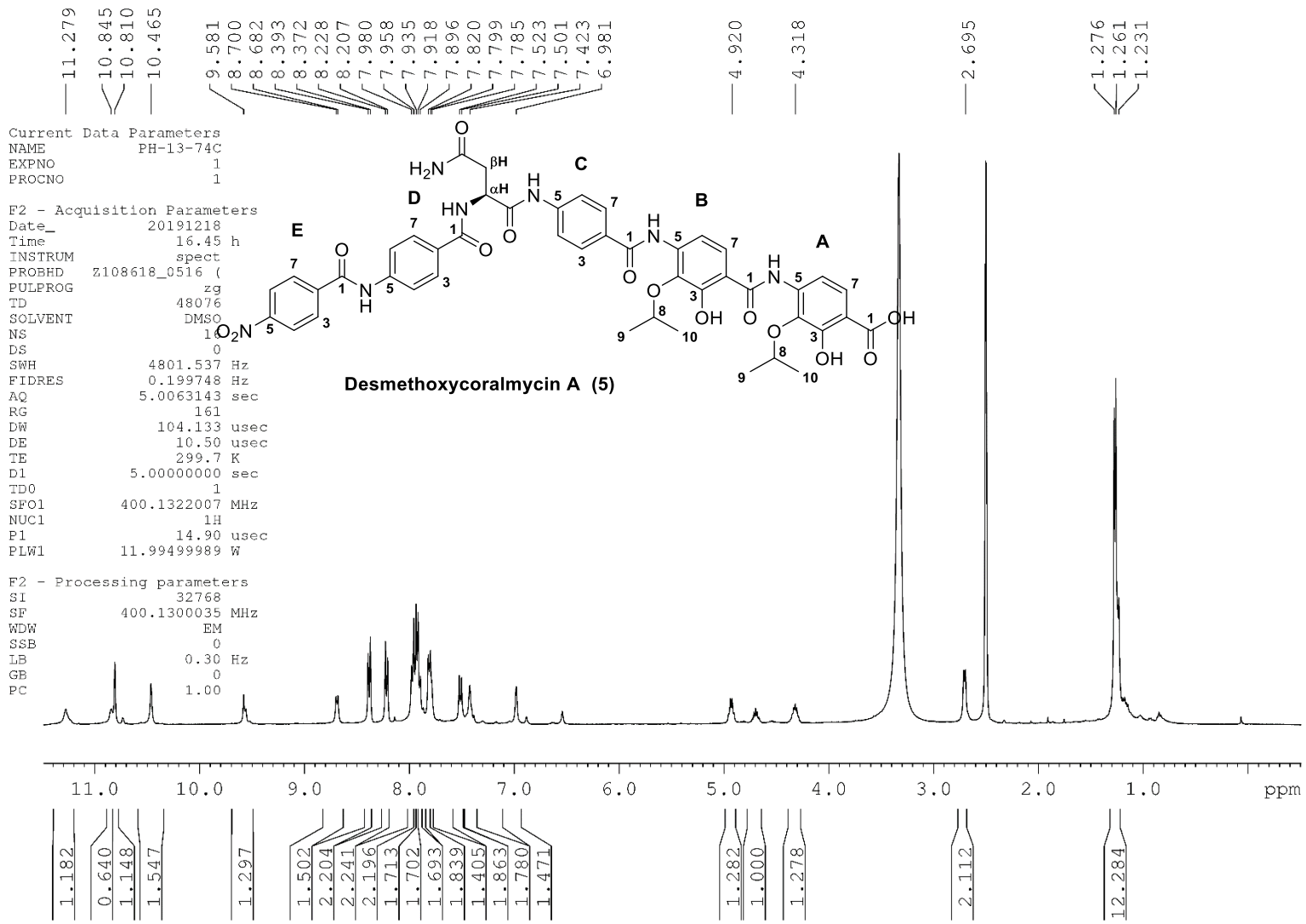

Current Data Parameters
NAME PH-7-61
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180224
Time 18.38 h
INSTRUM spect
PROBHD Z119470_0008 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 203
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 4
SFO1 125.7716224 MHz
NUC1 13C
P1 10.10 usec
PLW1 88.91999817 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.17528000 W
F2 - Processing parameters
SI 65536
SF 125.7577724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170.427
159.305
155.059
129.952
128.752
113.943
82.990
79.912
59.443
55.412
55.311
51.961
28.482
Boc
NH
O
O
O
O
S6
220
200
180
160
140
120
100
80
60
40
20
0
ppm

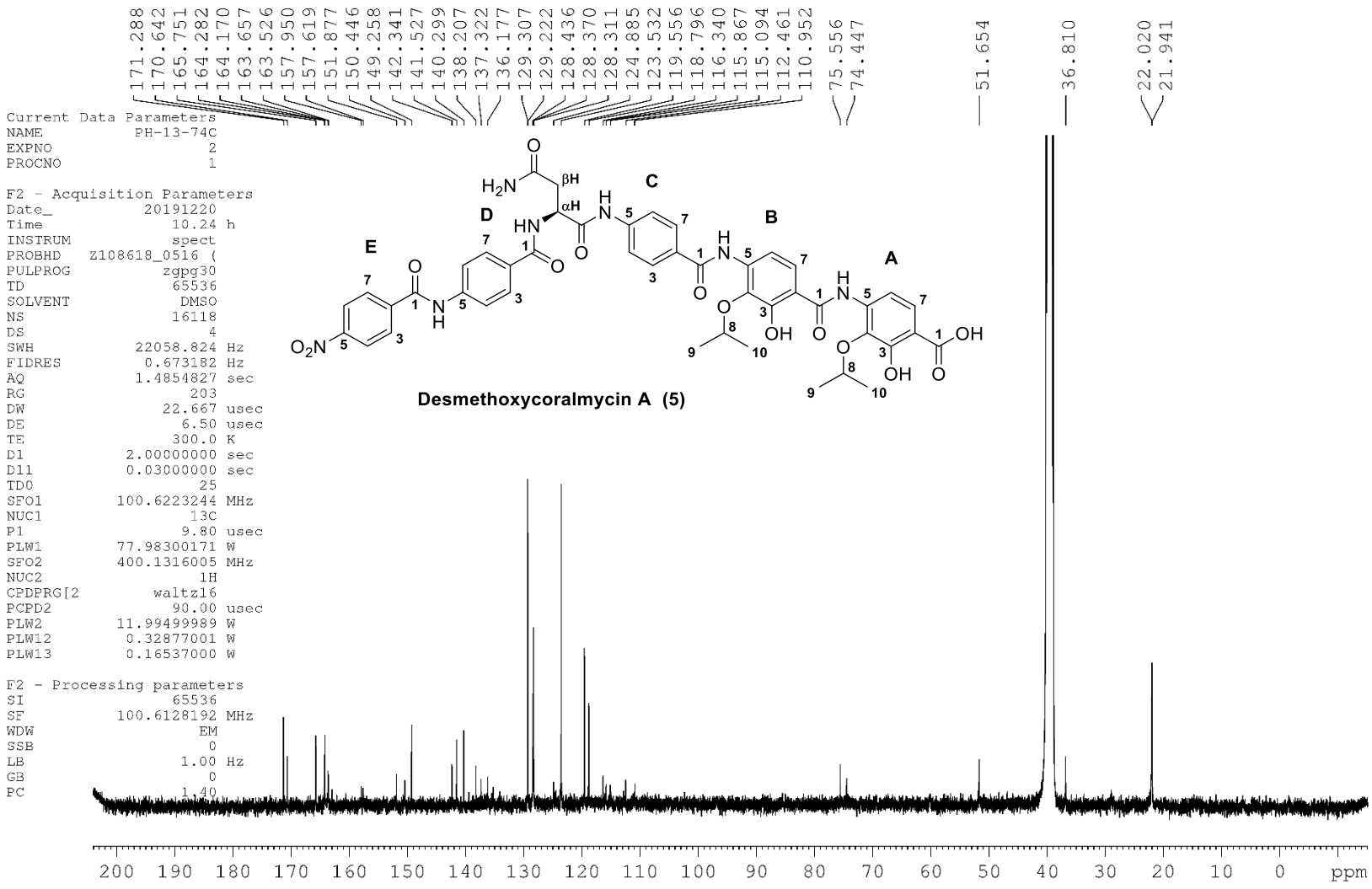

8.513
8.045
8.028
8.023
8.001
7.636
7.614
7.598
7.576
6.076
6.068
6.062
6.050
6.048
6.041
6.036
6.034
6.022
6.019
6.012
6.007
6.005
5.998
5.993
5.979
5.610
5.509
5.416
5.413
5.373
5.370
5.292
5.288
5.266
5.262
4.811
4.807
4.804
4.797
4.793
4.790
4.776
4.153
4.140
3.807
3.769
3.521
3.491
1.480
1.471
Current Data Parameters
NAME PH-6-25
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 18.17 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 65536
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
NH
H
N
O
O
O
O
O
OAllyl
S7
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
0.957
0.316
1.722
0.334
1.696
1.046
0.751
0.180
1.094
1.095
3.193
0.863
2.502
0.498
2.496
0.539
7.225
2.322

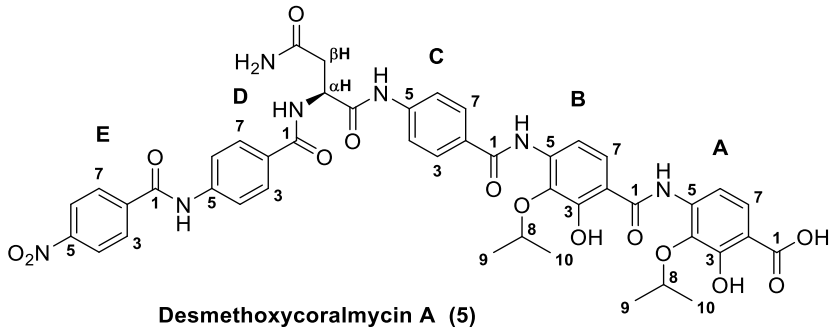

Current Data Parameters
NAME PH-6-25
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 19.26 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1200
DS 4
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6228303 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6127540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170.565
167.336
165.807
141.611
132.447
131.010
126.158
119.303
119.222
118.335
80.081
65.630
59.711
59.468
52.734
28.382
28.313
1.153
Boc
NH
H
N
O
O
O
O
O
OAllyl
S7
220
200
180
160
140
120
100
80
60
40
20
0
ppm


7.986
7.965
7.952
7.780
7.762
7.724
7.711
7.702
7.662
7.644
7.378
7.359
7.341
7.317
7.301
7.286
7.271
6.105
6.091
6.078
6.065
6.048
6.035
6.022
6.008
5.422
5.419
5.415
5.376
5.372
5.284
5.280
5.258
5.254
4.798
4.795
4.787
4.784
4.718
4.703
4.445
4.428
4.419
4.401
4.387
4.371
4.354
4.343
4.328
4.237
4.220
4.203
4.030
4.017
3.430
3.414
Current Data Parameters
NAME PH-6-30
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170614
Time 15.41 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 65536
SF 400.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Fmoc
NH
H
N
O
OH
O
O
OAllyl
25
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
2.121
6.046
3.882
0.425
0.992
1.011
1.035
2.609
0.689
2.306
0.898
0.438
0.065
2.015


Current Data Parameters
NAME PH-6-30
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170614
Time 16.25 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 703
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
165.807
143.815
143.745
142.651
141.179
132.372
130.132
130.098
127.399
126.795
125.306
124.799
119.524
119.348
119.256
117.001
67.038
66.866
65.102
58.016
57.817
57.332
Fmoc
NH
H
N
O
OH
O
O
O
OAllyl
25
200
180
160
140
120
100
80
60
40
20
0
ppm

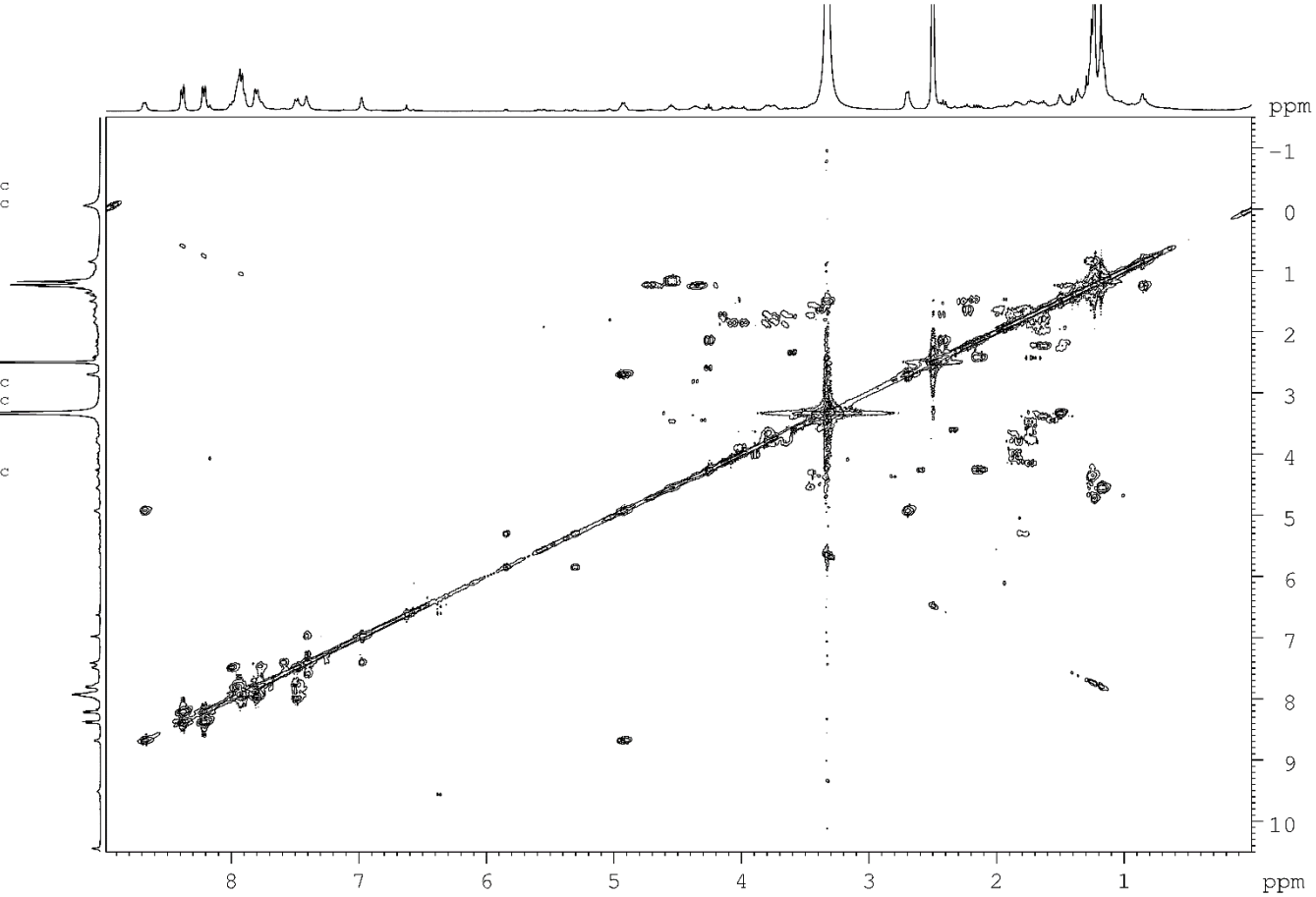

11.279
10.845
10.810
10.465
9.581
8.700
8.682
8.393
8.372
8.228
8.207
7.980
7.958
7.935
7.918
7.896
7.820
7.799
7.785
7.523
7.501
7.423
6.981
4.920
4.318
2.695
1.276
1.261
1.231
Current Data Parameters
NAME PH-13-74C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191218
Time 16.45 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zg
TD 48076
SOLVENT DMSO
NS 16
DS 0
SWH 4801.537 Hz
FIDRES 0.199748 Hz
AQ 5.0063143 sec
RG 161
DW 104.133 usec
DE 10.50 usec
TE 299.7 K
D1 5.00000000 sec
TD0 1
SFO1 400.1322007 MHz
NUC1 1H
P1 14.90 usec
PLW1 11.99499989 W
F2 - Processing parameters
SI 32768
SF 400.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Desmethoxycoralmycin A (5)
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm
1.182
0.640
1.148
1.547
1.297
1.502
2.204
2.241
2.196
1.713
1.702
1.693
1.839
1.405
1.863
1.780
1.471
1.282
1.000
1.278
2.112
12.284

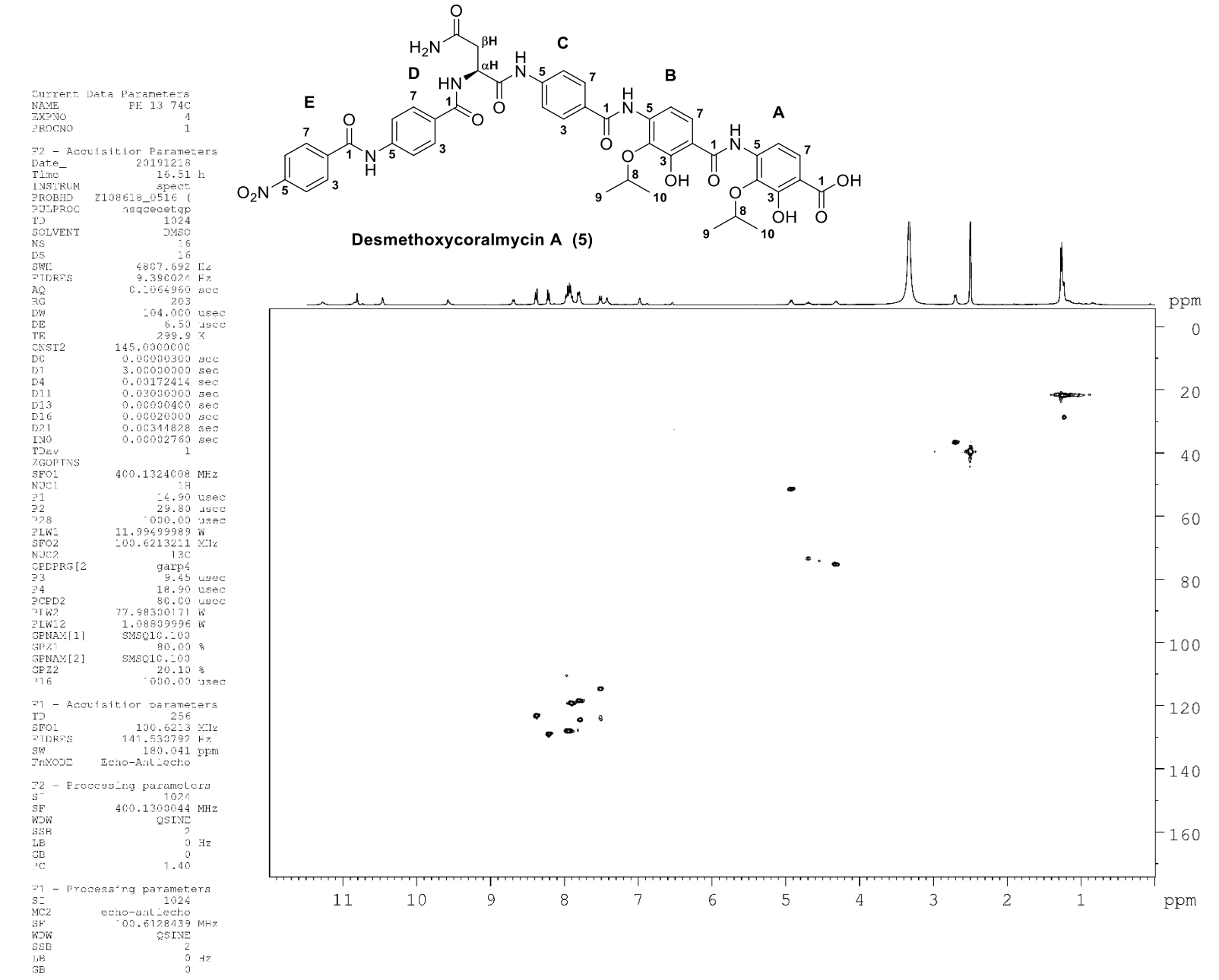

171.288
170.642
165.751
164.282
164.170
163.657
163.526
157.950
157.619
151.877
150.446
149.258
142.341
141.527
140.299
138.207
137.322
136.177
129.307
129.222
128.436
128.370
128.311
124.885
123.532
119.556
118.796
116.340
115.867
115.094
112.461
110.952
75.556
74.447
51.654
36.810
22.020
21.941
Current Data Parameters
NAME PH-13-74C
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191220
Time 10.24 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 16118
DS 4
SWH 22058.824 Hz
FIDRES 0.673182 Hz
AQ 1.4854827 sec
RG 203
DW 22.667 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 25
SFO1 100.6223244 MHz
NUC1 13C
P1 9.80 usec
PLW1 77.98300171 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.32877001 W
PLW13 0.16537000 W
F2 - Processing parameters
SI 65536
SF 100.6128192 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Desmethoxycoralmycin A (5)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



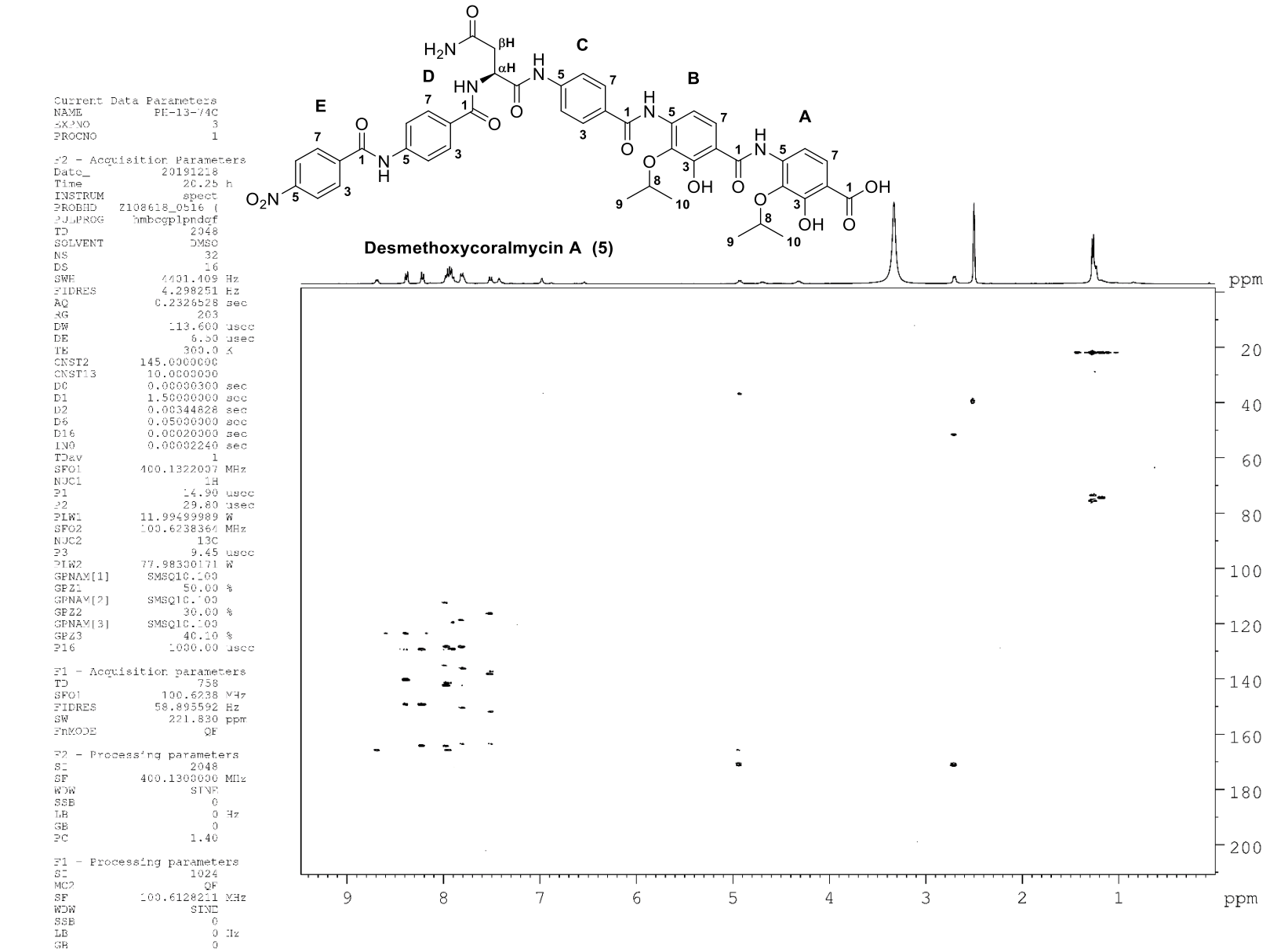
Desmethoxycoralmycin A (5)

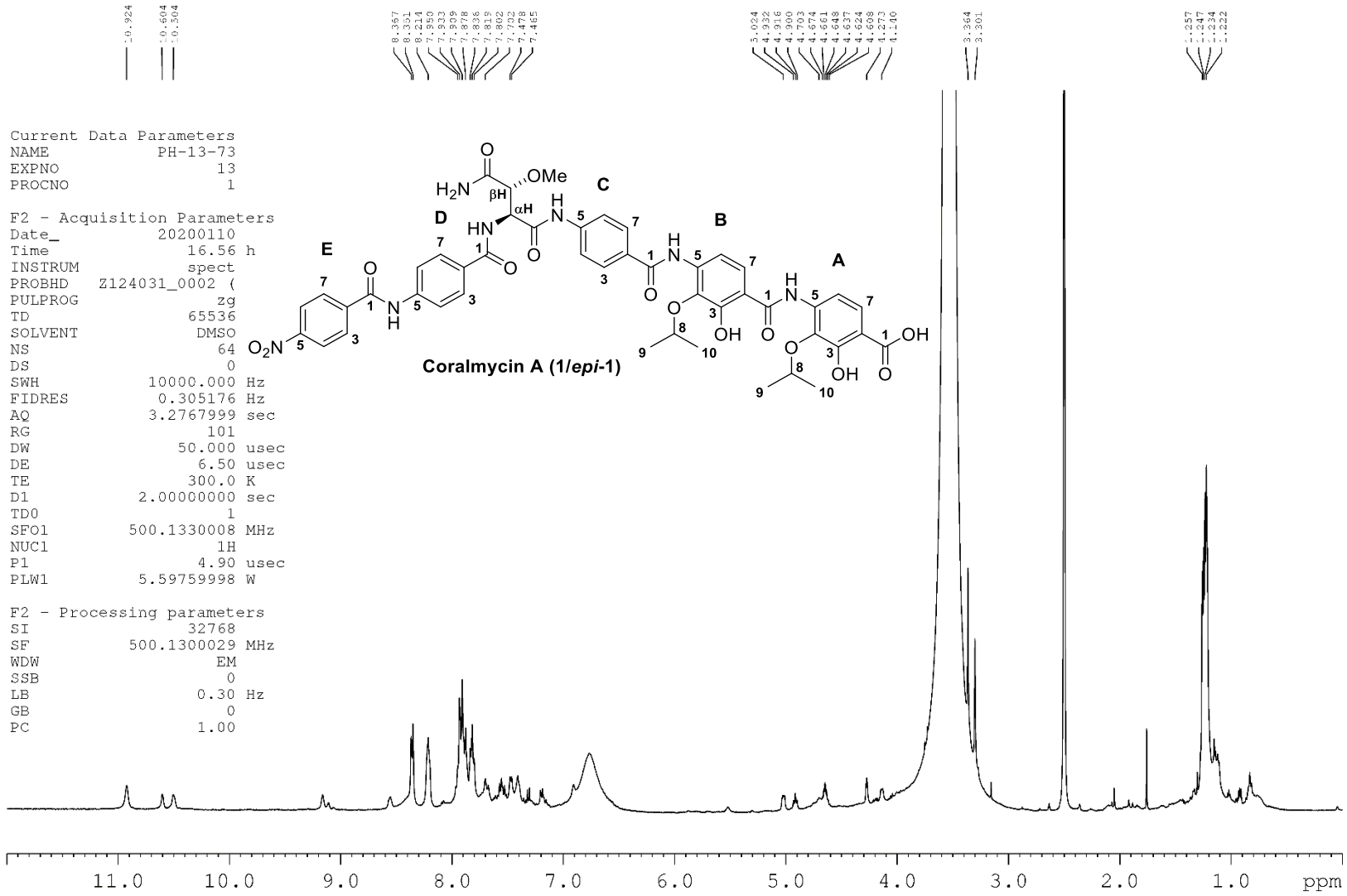
```
Current Data Parameters
NAME          PH-13-74C
EXPNO                 8
PROCNO                1

F2 - Acquisition Parameters
Date_          20200202
Time               2.17 h
INSTRUM           spect
PROBHD   Z108618_0516 (
PULPROG       cosygpqf
TD                2048
SOLVENT           DMSO
NS                  48
DS                  16
SWH           3597.122 Hz
FIDRES        3.512815 Hz
AQ            0.2846720 sec
RG                  203
DW              139.000 usec
DE                6.50 usec
TE               300.0 K
D0          0.00000300 sec
D1          1.95000005 sec
D13         0.00000400 sec
D16         0.00020000 sec
IN0         0.00020800 sec
TDav                1
SFO1       400.1318006 MHz
NUC1               1H
P0                8.02 usec
P1               16.05 usec
PLW1       11.99499989 W
GPNAM[1]    SMSQ10.100
GPZ1             10.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                 256
SFO1          400.1318 MHz
FIDRES       37.560097 Hz
SW              12.015 ppm
FnMODE              QF

F2 - Processing parameters
SI                1024
SF          400.1300054 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          400.1300029 MHz
WDW               SINE
SSB                  0
LB                   0 Hz
GB                   0
```

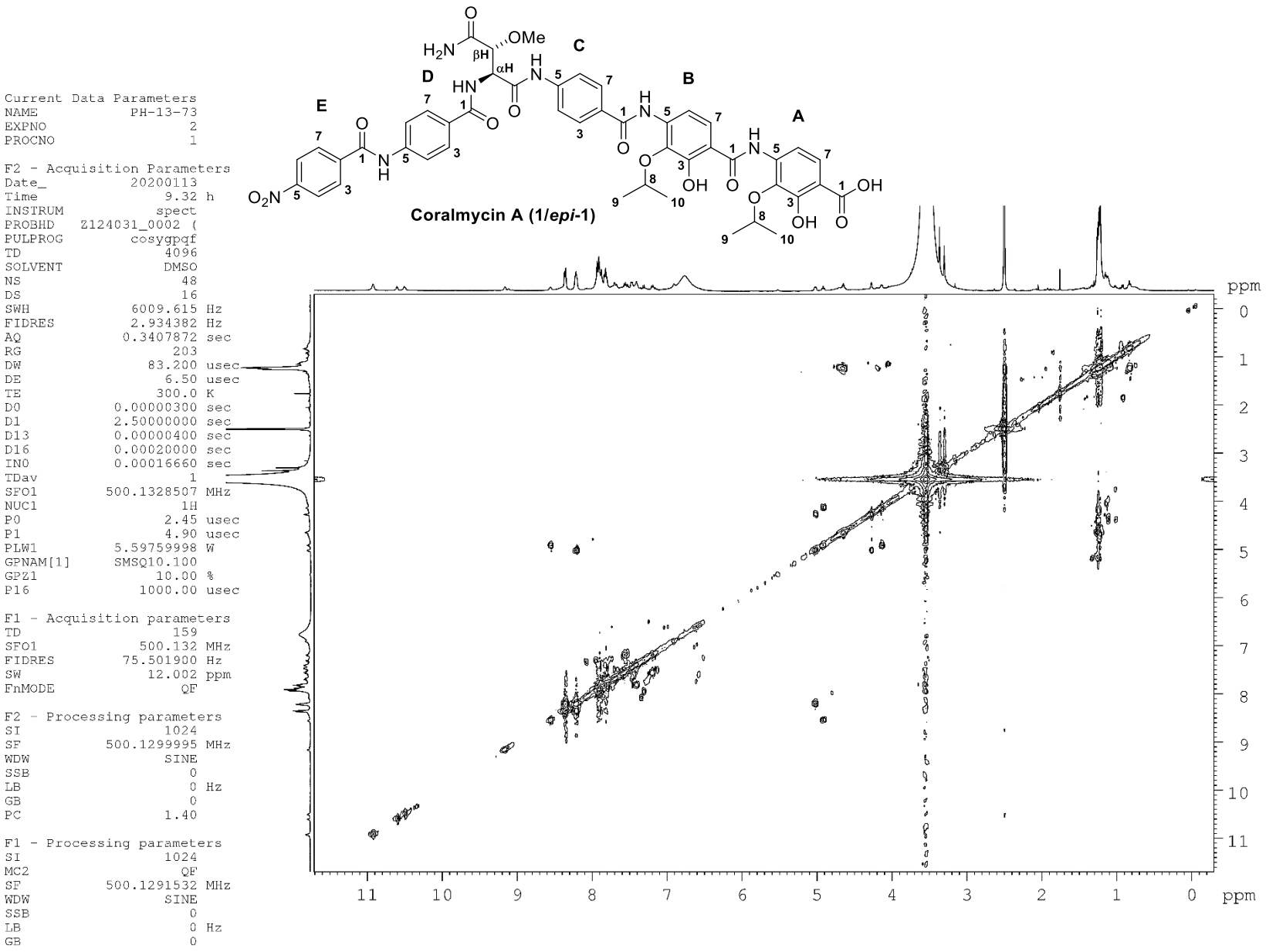


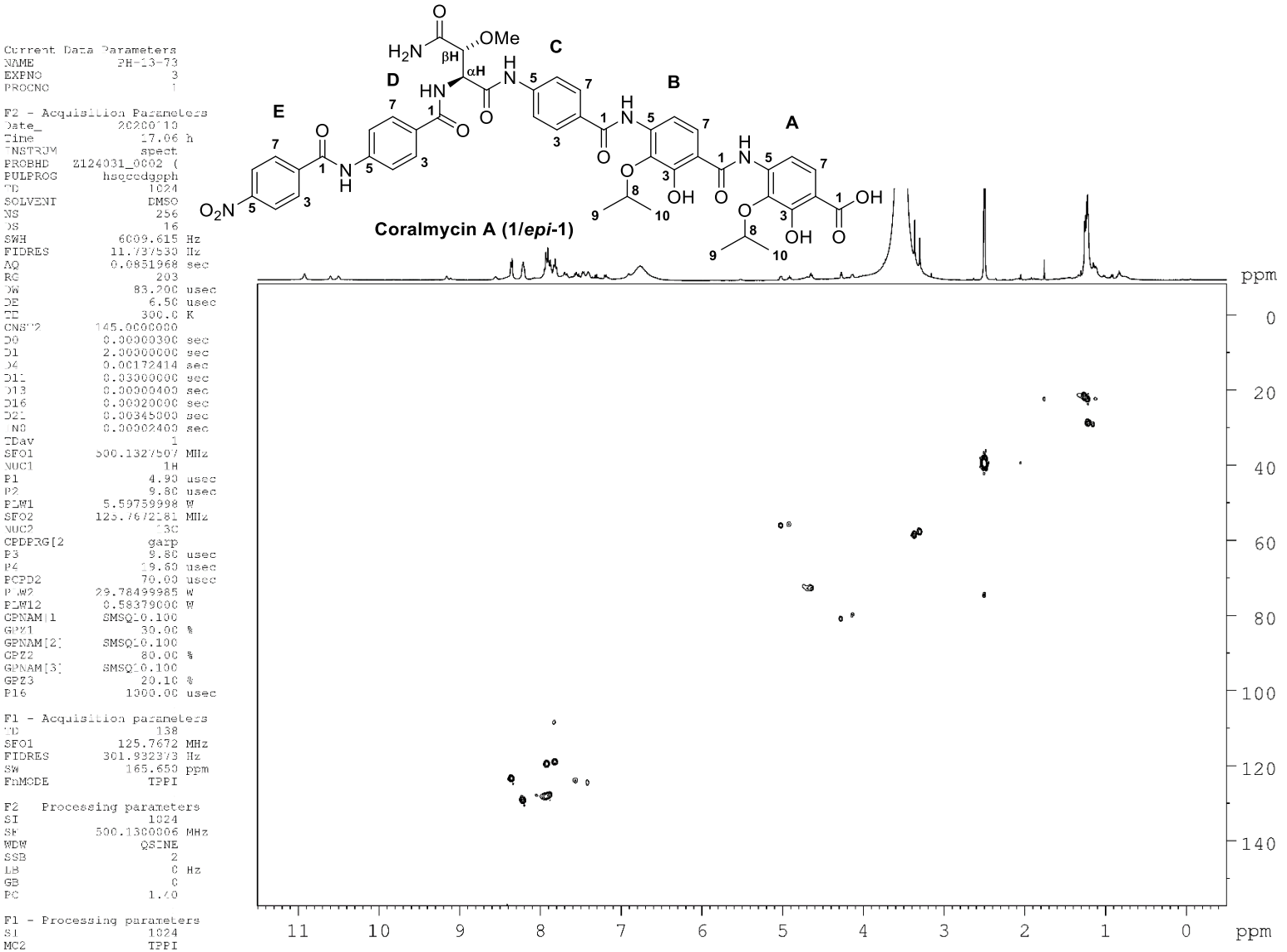


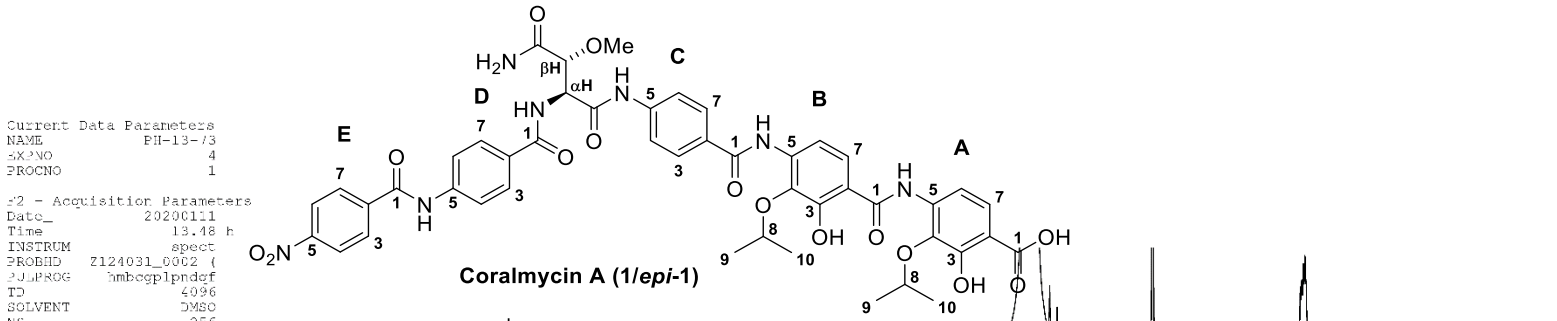

```
Current Data Parameters
NAME            PE 13 74C
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191218
Time                16.51 h
INSTRUM              spect
PROBHD    Z108618_0516 (
PULPROG       zgsqcecetgp
TD                   1024
SOLVENT              DMSO
NS                     16
DS                     16
SWH              4807.692 Hz
FIDRES           9.390024 Hz
AQ               0.1064960 sec
RG                     203
DW                104.000 usec
DE                  6.50 usec
TE                299.9 K
CNST2        145.0000000
D0            0.00000300 sec
D1            3.00000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
D21           0.00344828 sec
IN0           0.00002760 sec
TDav                   1
ZGOPTNS
SFO1         400.1324008 MHz
NUC1                  1H
P1                 14.90 usec
P2                 29.80 usec
P28              1000.00 usec
PLW1         11.99499989 W
SFO2         100.6213211 MHz
NUC2                 13C
CPDPRG[2            garp4
P3                  9.45 usec
P4                 18.90 usec
PCPD2              80.00 usec
PLW2         77.98300171 W
PLW12         1.08809996 W
GPNAM[1]      SMSQ10.100
GPZ1               80.00 %
GPNAM[2]      SMSQ10.100
GPZ2               20.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1          100.6213 MHz
FIDRES       141.530792 Hz
SW               180.041 ppm
FnMODE     Echo-AntiEcho

F2 - Processing parameters
SI                  1024
SF           400.1300044 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                  1024
MC2        echo-antiecho
SF           100.6128439 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```


Current Data Parameters
NAME PE-13-74C
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191218
Time 20.25 h
INSTRUM spect
PROBHD Z108618_0516 (
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 32
DS 16
SWH 4401.409 Hz
FIDRES 4.298251 Hz
AQ 0.2326528 sec
RG 203
DW 113.600 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST13 10.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.05000000 sec
D16 0.00020000 sec
IN0 0.00002240 sec
TDav 1
SFO1 400.1322007 MHz
NUC1 1H
P1 14.90 usec
P2 29.80 usec
PLW1 11.99499989 W
SFO2 100.6238364 MHz
NUC2 13C
P3 9.45 usec
PLW2 77.98330171 W
GPNAM[1] SMSQ10.100
GPZ1 50.00 %
GPNAM[2] SMSQ10.100
GPZ2 30.00 %
GPNAM[3] SMSQ10.100
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 758
SFO1 100.6238 MHz
FIDRES 58.895592 Hz
SW 221.830 ppm
FnMODE QF
F2 - Processing parameters
SI 2048
SF 400.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6128211 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
Desmethoxycoralmycin A (5)
ppm
20
40
60
80
100
120
140
160
180
200
9
8
7
6
5
4
3
2
1
ppm

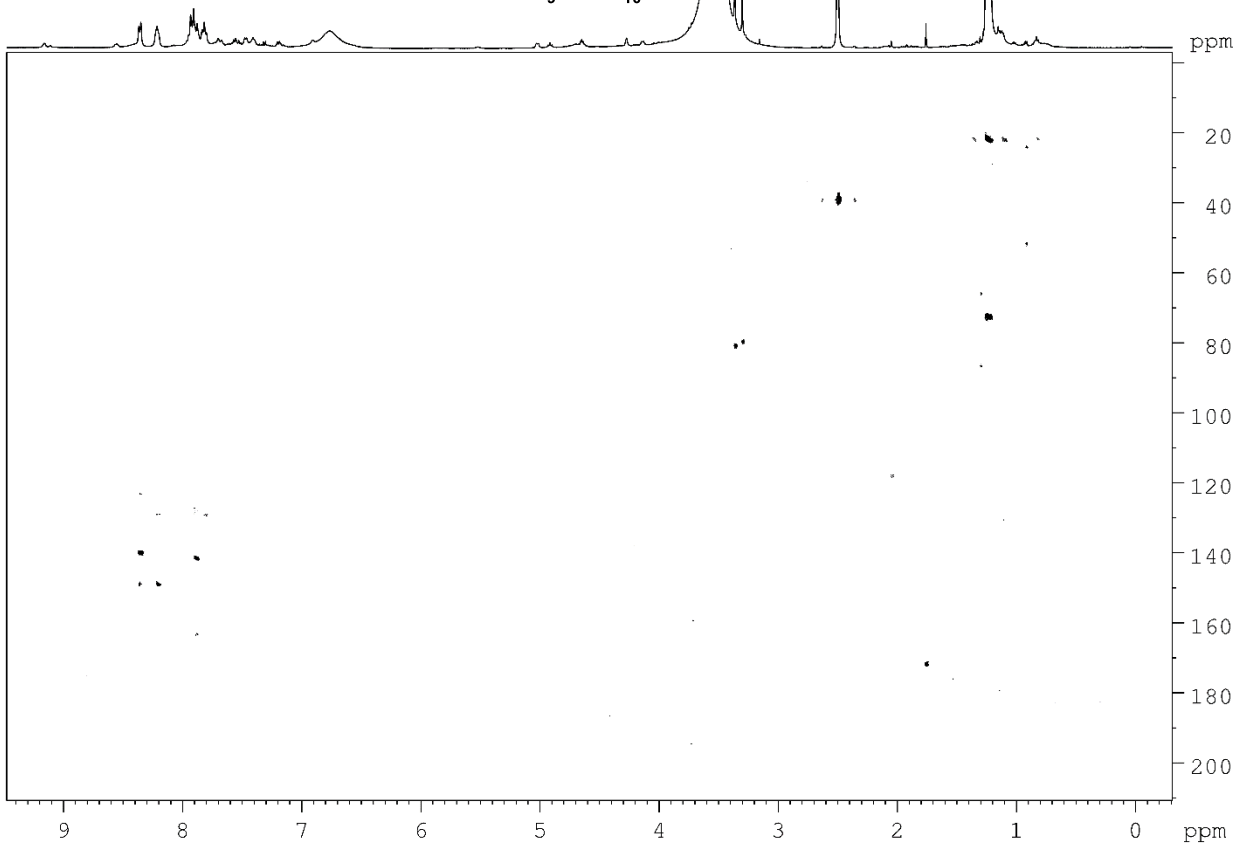

10.924
10.604
10.504
8.367
8.331
8.214
7.959
7.933
7.909
7.878
7.836
7.819
7.802
7.792
7.478
7.465
5.024
4.932
4.916
4.900
4.793
4.674
4.661
4.648
4.637
4.624
4.608
4.273
4.140
3.364
3.301
1.257
1.247
1.234
1.222
Current Data Parameters
NAME PH-13-73
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200110
Time 16.56 h
INSTRUM spect
PROBHD Z124031_0002 (
PULPROG zg
TD 65536
SOLVENT DMSO
NS 64
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 101
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 4.90 usec
PLW1 5.59759998 W
F2 - Processing parameters
SI 32768
SF 500.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Coralmycin A (1/epi-1)
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm


Current Data Parameters
NAME PH-13-73
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200113
Time 9.32 h
INSTRUM spect
PROBHD Z124031_0002 (
PULPROG cosygpqf
TD 4096
SOLVENT DMSO
NS 48
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.3407872 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 300.0 K
D0 0.00000300 sec
D1 2.50000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00016660 sec
TDav 1
SFO1 500.1328507 MHz
NUC1 1H
P0 2.45 usec
P1 4.90 usec
PLW1 5.59759998 W
GPNAM[1] SMSQ10.100
GPZ1 10.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 159
SFO1 500.132 MHz
FIDRES 75.501900 Hz
SW 12.002 ppm
FnMODE QF
F2 - Processing parameters
SI 1024
SF 500.1299995 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1291532 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
Coralmycin A (1/epi-1)
ppm
ppm


Current Data Parameters
NAME PH-13-73
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200710
Time 17.06 h
INSTRUM spect
PROBHD Z124031_0002 (
PULPROG hsqcedgpph
TD 1024
SOLVENT DMSO
NS 256
DS 16
SWH 6009.615 Hz
FIDRES 11.737530 Hz
AQ 0.0851968 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 2.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
TAU 0.00002400 sec
TDav 1
SFO1 500.1327507 MHz
NUC1 1H
P1 4.90 usec
P2 9.80 usec
PLW1 5.59759998 W
SFO2 125.7672181 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.80 usec
P4 19.60 usec
PCPD2 70.00 usec
PLW2 29.78499985 W
PLW12 0.58379000 W
GPNAM[1 SMSQ10.100
GPZ1 30.00 %
GPNAM[2 SMSQ10.100
GPZ2 80.00 %
GPNAM[3 SMSQ10.100
GPZ3 20.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 138
SFO1 125.7672 MHz
FIDRES 301.932373 Hz
SW 165.650 ppm
FnMODE TPPI
F2 Processing parameters
SI 1024
SF 500.1300006 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 TPPI
SF 125.7578545 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
Coralmycin A (1/epi-1)
ppm
0
20
40
60
80
100
120
140
11
10
9
8
7
6
5
4
3
2
1
0
ppm




```
Current Data Parameters
NAME                  PH-13-73
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_              20200111
Time                    13.48 h
INSTRUM                  spect
PROBHD     Z124031_0002 (
PULPROG          hmbcgplpndqf
TD                       4096
SOLVENT                  DMSO
NS                        256
DS                         16
SWH                  6009.615 Hz
FIDRES               2.934382 Hz
AQ                   0.3407872 sec
RG                         203
DW                      83.200 usec
DE                       6.50 usec
TE                      300.0 K
CNST2               145.0000000
CNST13               10.0000000
D0                   0.00000300 sec
D1                   2.00000000 sec
D2                   0.00344828 sec
D6                   0.05000000 sec
D16                  0.00020000 sec
IN0                  0.00001770 sec
TDav                         1
SFO1                500.1328507 MHz
NUC1                        1H
P1                       4.90 usec
P2                       9.80 usec
PLW1                 5.59759998 W
SFO2                125.7716224 MHz
NUC2                        13C
P3                       9.80 usec
PLW2                29.78799985 W
GPNAM[1]             SMSQ10.100
GPZ1                    50.00 %
GPNAM[2]             SMSQ10.100
GPZ2                    30.00 %
GPNAM[3]             SMSQ10.100
GPZ3                    40.10 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                          256
SFO1                125.7704 MHz
FIDRES               220.692093 Hz
SW                   224.634 ppm
FnMODE                      QF

F2 - Processing parameters
SI                         2048
SF                  500.1300000 MHz
WDW                        SINE
SSB                           0
LB                            0 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                         1024
MC2                         QF
SF                  125.7566068 MHz
WDW                        SINE
SSB                           0
LB                            0 Hz
GB                            0
```



Crystallographic Information File for 20

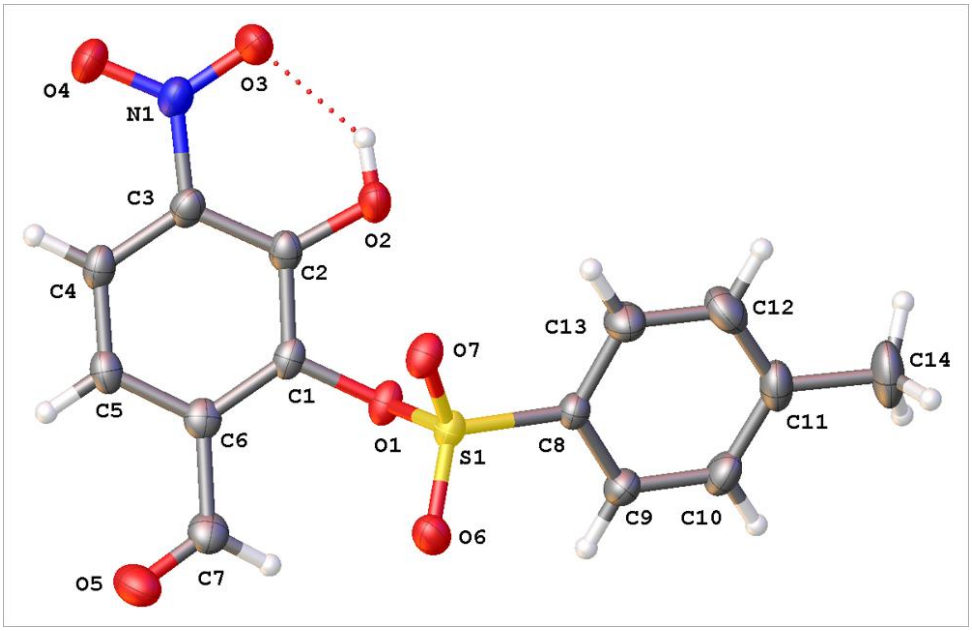


Table 1 Crystal data and structure refinement for rjp18pt2_s1634.

Identification code	rjp18pt2_s1634
Empirical formula	$C_{14}H_{11}NO_7S$
Formula weight	337.30
Temperature/K	100(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	10.61747(19)
b/Å	13.02984(15)
c/Å	10.52228(16)
α/°	90
β/°	101.8819(16)
γ/°	90
Volume/Å^3	1424.50(4)
Z	4
ρ_{calc}g/cm^3	1.573
μ/mm^{-1}	2.397
F(000)	696.0
Crystal size/mm^3	0.167 × 0.151 × 0.078
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.51 to 152.934
Index ranges	-13 ≤ h ≤ 13, -16 ≤ k ≤ 16, -13 ≤ l ≤ 13
Reflections collected	29696
Independent reflections	2981 [R_{int} = 0.0259, R_{sigma} = 0.0110]
Data/restraints/parameters	2981/0/212
Goodness-of-fit on F^2	1.076

Final R indexes [I>=2σ (I)]	R_1 = 0.0344, wR_2 = 0.0975
Final R indexes [all data]	R_1 = 0.0358, wR_2 = 0.0992
Largest diff. peak/hole / e Å^{-3}	0.41/-0.39

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for rjp18pt2_s1634. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	8732.8(3)	7080.0(2)	9276.9(3)	22.94(12)
O1	8743.1(10)	7471.6(7)	10739.7(9)	26.7(2)
O2	6737.1(10)	8710.5(8)	10341.6(11)	32.1(3)
O3	5956.9(11)	10604.7(9)	10420.5(14)	44.4(3)
O4	7259.2(12)	11823.0(9)	11222.6(13)	41.0(3)
O5	12425.1(11)	8544.6(10)	11702.8(13)	41.6(3)
O6	9990.0(10)	6687.2(8)	9295.4(11)	31.9(3)
O7	8253.4(11)	7887.3(7)	8394.8(10)	29.2(2)
N1	7042.7(13)	10928.2(10)	10908.4(14)	32.3(3)
C1	8960.7(14)	8519.4(10)	10985.1(13)	23.7(3)
C2	7872.8(14)	9157.7(11)	10793.1(13)	25.8(3)
C3	8105.1(14)	10200.3(11)	11104.5(14)	27.3(3)
C4	9339.8(14)	10578.2(11)	11579.5(14)	27.7(3)
C5	10377.4(14)	9934.7(11)	11735.0(13)	26.9(3)
C6	10195.7(14)	8887.2(11)	11431.3(13)	25.8(3)
C7	11347.1(15)	8206.8(13)	11579.4(15)	31.6(3)
C8	7619.8(13)	6080.0(10)	9147.5(13)	24.2(3)
C9	8001.5(15)	5129.6(11)	9688.4(15)	28.5(3)
C10	7118.8(16)	4331.4(12)	9506.2(17)	34.5(3)
C11	5877.7(17)	4468.3(13)	8785.4(17)	37.4(4)
C12	5519.0(17)	5430.9(15)	8269.2(18)	42.3(4)
C13	6380.4(16)	6245.3(13)	8444.6(17)	35.7(4)
C14	4953(2)	3574.9(17)	8544(2)	56.4(6)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for rjp18pt2_s1634. The Anisotropic displacement factor exponent takes the form: -2π^2[h^{2}a*^{2}U_{11}+2hka*b*U_{12}+…].

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	26.9(2)	19.35(19)	23.10(19)	-2.45(11)	6.42(13)	-2.49(11)
O1	39.0(6)	19.7(5)	22.9(5)	-1.4(4)	9.4(4)	-5.2(4)
O2	26.6(5)	27.0(5)	43.8(6)	-9.7(4)	9.6(5)	-6.5(4)
O3	25.2(6)	34.1(6)	73.7(9)	-12.1(6)	9.5(6)	-4.2(5)
O4	40.1(7)	25.9(5)	56.3(8)	-13.1(5)	8.2(5)	-1.8(5)
O5	30.2(6)	41.5(7)	50.2(7)	7.6(5)	1.7(5)	1.2(5)
O6	26.0(5)	33.7(6)	36.8(6)	-6.0(4)	8.4(4)	-2.5(4)
O7	41.4(6)	22.6(5)	24.0(5)	0.6(4)	7.2(4)	-3.2(4)
N1	30.8(7)	25.7(6)	42.4(7)	-8.2(5)	11.9(5)	-3.3(5)

C1	32.8(7)	19.9(6)	19.6(6)	-1.4(5)	8.2(5)	-4.6(5)
C2	27.7(7)	26.0(7)	25.5(6)	-3.9(5)	9.6(5)	-5.9(5)
C3	30.0(7)	23.8(7)	30.4(7)	-4.7(5)	11.3(6)	-2.0(5)
C4	34.3(8)	24.6(7)	25.5(7)	-5.2(5)	9.5(6)	-7.1(6)
C5	29.0(7)	28.9(7)	22.0(6)	-1.7(5)	3.9(5)	-8.3(6)
C6	30.6(7)	28.0(7)	18.5(6)	1.1(5)	4.2(5)	-2.1(6)
C7	33.8(8)	31.3(7)	26.6(7)	3.6(6)	-0.9(6)	0.3(6)
C8	25.6(7)	20.2(6)	26.3(6)	-2.3(5)	4.3(5)	-2.1(5)
C9	28.5(7)	22.6(7)	33.9(7)	0.0(6)	5.1(6)	0.5(5)
C10	41.0(9)	20.7(7)	43.3(9)	-1.1(6)	12.0(7)	-2.6(6)
C11	38.1(9)	33.5(8)	40.8(9)	-9.8(7)	8.9(7)	-13.4(7)
C12	28.8(8)	48.5(10)	44.7(9)	-0.6(8)	-4.0(7)	-6.4(7)
C13	31.6(8)	30.5(8)	41.6(8)	4.7(6)	-0.2(6)	1.8(6)
C14	58.7(13)	49.8(11)	61.2(13)	-18.0(10)	13.7(10)	-31.9(10)

Table 4 Bond Lengths for rjp18pt2_s1634.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	O1	1.6194(10)	C2	C3	1.407(2)
S1	O6	1.4259(11)	C3	C4	1.393(2)
S1	O7	1.4256(11)	C4	C5	1.367(2)
S1	C8	1.7454(14)	C5	C6	1.406(2)
O1	C1	1.3998(16)	C6	C7	1.492(2)
O2	C2	1.3354(17)	C8	C9	1.388(2)
O3	N1	1.2355(18)	C8	C13	1.387(2)
O4	N1	1.2208(17)	C9	C10	1.387(2)
O5	C7	1.208(2)	C10	C11	1.390(2)
N1	C3	1.4556(19)	C11	C12	1.388(3)
C1	C2	1.404(2)	C11	C14	1.510(2)
C1	C6	1.384(2)	C12	C13	1.388(2)

Table 5 Bond Angles for rjp18pt2_s1634.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	S1	C8	100.45(6)	C4	C3	C2	122.11(14)
O6	S1	O1	106.08(6)	C5	C4	C3	120.12(13)
O6	S1	C8	110.48(7)	C4	C5	C6	119.91(13)
O7	S1	O1	108.54(6)	C1	C6	C5	119.29(14)
O7	S1	O6	118.78(7)	C1	C6	C7	121.89(13)
O7	S1	C8	110.78(7)	C5	C6	C7	118.81(13)
C1	O1	S1	116.82(8)	O5	C7	C6	122.18(15)
O3	N1	C3	117.79(13)	C9	C8	S1	119.88(11)
O4	N1	O3	123.09(14)	C13	C8	S1	118.57(11)
O4	N1	C3	119.12(13)	C13	C8	C9	121.47(14)

O1	C1	C2	116.85(12)	C10	C9	C8	118.72(14)
C6	C1	O1	120.60(13)	C9	C10	C11	121.13(15)
C6	C1	C2	122.53(13)	C10	C11	C14	120.20(17)
O2	C2	C1	116.65(13)	C12	C11	C10	118.83(14)
O2	C2	C3	127.32(14)	C12	C11	C14	120.95(18)
C1	C2	C3	116.03(13)	C13	C12	C11	121.22(15)
C2	C3	N1	120.16(13)	C8	C13	C12	118.61(15)
C4	C3	N1	117.72(13)				

Table 6 Hydrogen Bonds for rjp18pt2_s1634.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O2	H2	O3	0.87(2)	1.85(2)	2.6103(16)	145(2)
O2	H2	O3[1]	0.87(2)	2.24(2)	2.9456(17)	138(2)

[1]1-X,2-Y,2-Z

Table 7 Torsion Angles for rjp18pt2_s1634.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
S1	O1	C1	C2	90.13(13)	N1	C3	C4	C5	-177.52(13)
S1	O1	C1	C6	-91.66(14)	C1	C2	C3	N1	178.48(12)
S1	C8	C9	C10	-176.25(12)	C1	C2	C3	C4	-0.2(2)
S1	C8	C13	C12	175.83(14)	C1	C6	C7	O5	164.24(15)
O1	S1	C8	C9	-80.33(13)	C2	C1	C6	C5	1.5(2)
O1	S1	C8	C13	102.78(13)	C2	C1	C6	C7	-177.47(13)
O1	C1	C2	O2	-2.74(18)	C2	C3	C4	C5	1.2(2)
O1	C1	C2	C3	177.04(12)	C3	C4	C5	C6	-0.9(2)
O1	C1	C6	C5	-176.63(12)	C4	C5	C6	C1	-0.4(2)
O1	C1	C6	C7	4.4(2)	C4	C5	C6	C7	178.53(13)
O2	C2	C3	N1	-1.8(2)	C5	C6	C7	O5	-14.7(2)
O2	C2	C3	C4	179.55(14)	C6	C1	C2	O2	179.08(13)
O3	N1	C3	C2	-2.6(2)	C6	C1	C2	C3	-1.1(2)
O3	N1	C3	C4	176.10(14)	C8	S1	O1	C1	-147.98(10)
O4	N1	C3	C2	177.73(14)	C8	C9	C10	C11	0.7(2)
O4	N1	C3	C4	-3.5(2)	C9	C8	C13	C12	-1.0(2)
O6	S1	O1	C1	96.97(11)	C9	C10	C11	C12	-1.5(3)
O6	S1	C8	C9	31.36(14)	C9	C10	C11	C14	176.99(17)
O6	S1	C8	C13	-145.53(12)	C10	C11	C12	C13	1.0(3)
O7	S1	O1	C1	-31.72(12)	C11	C12	C13	C8	0.2(3)
O7	S1	C8	C9	165.10(11)	C13	C8	C9	C10	0.5(2)
O7	S1	C8	C13	-11.79(15)	C14	C11	C12	C13	-177.45(18)

Table 8 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for rjp18pt2_s1634.

Atom	*x*	*y*	*z*	U(eq)
H2	6180(20)	9207(19)	10250(20)	48
H4	9461	11284.18	11795.62	33
H5	11220.03	10195.05	12047.86	32
H7	11228.51	7484.09	11576.53	38
H9	8851.01	5027.96	10174.11	34
H10	7366.63	3679.83	9880.52	41
H12	4668.38	5534.04	7787.02	51
H13	6126.46	6902.18	8090.74	43
H14A	5009.77	3242.45	7722.62	85
H14B	4074.03	3823.31	8498.8	85
H14C	5176.69	3079.17	9255.66	85

Experimental

Single crystals of $C_{14}H_{11}NO_7S$ **[rjp18pt2_s1634]** were **[crystallised from a mixture of ethyl acetate and petroleum benzine]**. A suitable crystal was selected and **[in paratone on a nylon loop]** on a **SuperNova, Dual, Cu at home/near, Atlas** diffractometer. The crystal was kept at 100(2) K during data collection. Using Olex2 [1], the structure was solved with the olex2.solve [2] structure solution program using Charge Flipping and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Bourhis, L.J., Dolomanov, O.V., Gildea, R.J., Howard, J.A.K., Puschmann, H. (2015). Acta Cryst. A71, 59-75.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [rjp18pt2_s1634]

Crystal Data for $C_{14}H_{11}NO_7S$ (M =337.30 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 10.61747(19) Å, b = 13.02984(15) Å, c = 10.52228(16) Å, β = 101.8819(16)°, V = 1424.50(4) Å^3, Z = 4, T = 100(2) K, μ(Cu Kα) = 2.397 mm^{-1}, *Dcalc* = 1.573 g/cm^3, 29696 reflections measured (8.51° ≤ 2Θ ≤ 152.934°), 2981 unique (R_{int} = 0.0259, R_{sigma} = 0.0110) which were used in all calculations. The final R_1 was 0.0344 (I > 2σ(I)) and wR_2 was 0.0992 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups

At 1.5 times of:

All C(H,H,H) groups, All O(H) groups

2.a Aromatic/amide H refined with riding coordinates:

C4(H4), C5(H5), C7(H7), C9(H9), C10(H10), C12(H12), C13(H13)

2.b Idealised Me refined as rotating group:

C14(H14A,H14B,H14C)