Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Electronic Supplementary Information

Autocatalytic aerobic *ipso*-hydroxylation of arylboronic acid with Hantzsch ester and Hantzsch pyridine

Chi-Hang Fan, Tianyue Xu, Zhihai Ke,* Ying-Yeung Yeung*

Department of Chemistry and State Key Laboratory of Synthetic Chemistry, The Chinese University of Hong Kong

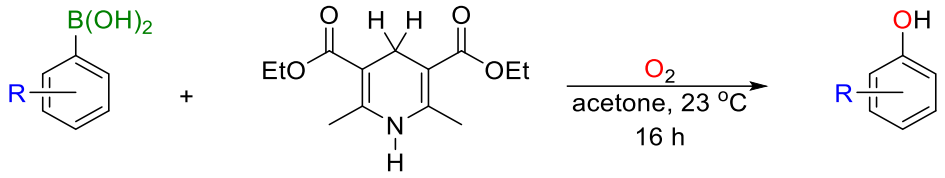
Shatin, NT, Hong Kong, China

E-mail: yyyeung@cuhk.edu.hk; Tel: +852-3943-6377

(A) General information

All reactions were carried out by standard procedures under air condition at room temperature unless stated otherwise. Commercially available reagents from Sigma Aldrich and J&K were used as received unless stated otherwise. The solvents were dried over a solvent purification system from Innovative Technology. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AMX400 (400 MHz) spectrometer or a Bruker Avance III 400MHz Spectrometer. Chemical shifts (δ) are reported in parts per million (ppm) relative to TMS (δ 0.00) for the ^{1}H NMR and referenced to residual signals in NMR solvents ($CDCl_3$ at δ 77.16) for the ^{13}C NMR measurements. Coupling constant (*J*) are quoted in Hz. High resolution mass spectra were obtained on a Finnigan MAT 95XL Mass Spectrometer. Analytical thin layer chromatography (TLC) was performed with Merck pre-coated TLC plates, silica gel 60F-254, layer thickness 0.25 mm. Flash chromatography separations were performed on Merck 60 (0.040-0.063 mm) mesh silica gel.

(B) General procedure for the *ipso*-hydroxylation of arylboronic acids

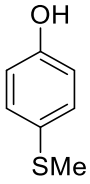


1 **HE1** **2**

To a Schlenk flask was charged with arylboronic acid **1** (0.1 mmol) and Hantzsch ester **HE1** (1.5 equiv, 0.15 mmol). The flask was sealed with a rubber septum, evacuated under vacuum, and refilled with pure oxygen using a balloon. Then, 2 mL of acetone was added. The reaction mixture was stirred at room temperature for 16 hours. The solution was concentrated under reduced pressure and the residue was purified by flash column chromatography using silica gel as the stationary phase and *n*-hexane/ethyl acetate as the gradient eluent to afford the desired phenol products **2**.

(C) Characterization of products

4-(methylthio)-phenol (**2a**)[1]



Appearance: pale yellow solid

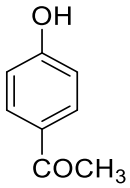
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.24 – 7.20 (dt, $^3J_{ortho}$ = 8.65 Hz, $^4J_{meta}$ = 2.56 Hz, 2H), 6.81 – 6.77 (dt, $^3J_{ortho}$ = 8.70 Hz, $^4J_{meta}$ = 2.58 Hz, 2H), 4.78 (s, 1H), 2.44 (s, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 154.20, 130.49, 129.08, 116.17, 18.20

QEFMS (ESI): m/z calcd for C_7H_8SO $[M–H]^-$: 139.02231; found: 139.02229

Column chromatography: R_f = 0.3 (H/EA 5:1)

4'-hydroxyacetophenone (**2b**)[1]



Appearance: pale yellow solid

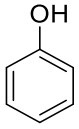
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 – 7.89 (dt, $^3J_{ortho}$ = 8.75 Hz, $^4J_{meta}$ = 2.50 Hz, 2H), 6.94 – 6.90 (dt, $^3J_{ortho}$ = 8.85 Hz, $^4J_{meta}$ = 2.50 Hz, 2H), 2.57 (s, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 197.88, 160.84, 131.12, 129.95, 115.45, 26.36

QEFMS (ESI): m/z calcd for $C_8H_8O_2$ $[M–H]^-$: 135.04515; found: 135.04501

Column chromatography: R_f = 0.1 (H/EA 5:1)

Phenol (**2c**)[1]



Appearance: white solid

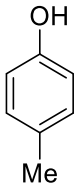
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28 – 7.23 (tt, $^3J_{ortho}$ = 8.14 Hz, $^4J_{meta}$ = 2.09 Hz, 2H), 6.96 – 6.92 (tt, $^3J_{ortho}$ = 7.61 Hz, 1H), 6.85 – 6.82 (dt, $^3J_{ortho}$ = 7.65 Hz, $^4J_{meta}$ = 2.83 Hz, 2H), 4.88 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 155.56, 129.81, 120.93, 115.41

QEFMS (ESI): m/z calcd for C_6H_6O $[M–H]^-$: 93.03459; found: 93.03447

Column chromatography: R_f = 0.3 (H/EA 5:1)

4-methylphenol (**2d**)[2]



Appearance: white solid

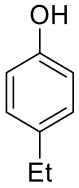
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.05 – 7.03 (d, *J* = 8.10 Hz, 2H), 6.75 – 6.72 (d, *J* = 8.60 Hz, 2H), 4.66 (bs, 1H), 2.28 (s, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 153.33, 130.20, 130.11, 115.19, 20.60

QEFMS (ESI): m/z calcd for C_7H_8O $[M–H]^-$:107.05024 ; found: 107.05022

Column chromatography: R_f = 0.5 (H/EA 5:1)

4-ethylphenol (**2e**)[3]



Appearance: pale yellow solid

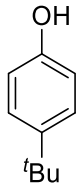
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.08 – 7.06 (d, *J* = 8.05 Hz, 2H), 6.77 – 6.75 (d, *J* = 8.30 Hz, 2H), 4.74 (s, 1H), 2.61 – 2.55 (q, *J* = 7.57 Hz, 2H), 1.22 – 1.19 (t, *J* = 7.63 Hz, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 153.51, 136.68, 129.03, 115.23, 28.10, 16.03

QEFMS (ESI): m/z calcd for $C_8H_{10}O$ $[M–H]^-$: 121.06589; found: 121.06574

Column chromatography: R_f = 0.5 (H/EA 5:1)

4-*tert*-butylphenol (**2f**)[4]



Appearance: white solid

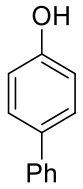
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28 – 7.24 (d, *J* = 8.65 Hz, 2H), 6.79 – 6.75 (d, *J* = 8.70 Hz, 2H), 4.60 (s, 1H), 1.29 (s, 9H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 153.25, 143.67, 126.58, 114.85, 34.21, 31.66

QEFMS (ESI): m/z calcd for $C_{10}H_{14}O$ $[M–H]^-$: 149.09719; found: 149.09696

Column chromatography: R_f = 0.4 (H/EA 5:1)

[1,1'-biphenyl]-4-ol (**2g**)[3]



Appearance: while solid

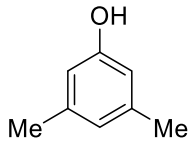
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 – 7.53 (d, *J* = 7.25 Hz, 2H), 7.50 – 7.46 (d, *J* = 8.60 Hz, 2H), 7.44 – 7.40 (t, *J* = 7.73 Hz, 2H), 7.33 – 7.29 (t, *J* = 7.38 Hz, 1H), 6.93 – 6.89 (dt, $^3J_{ortho}$ = 9.55 Hz, $^4J_{meta}$ = 2.55 Hz, 2H), 4.80 (s, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 155.18, 140.88, 134.18, 128.87, 128.54, 126.86, 115.76

QEFMS (ESI): m/z calcd for $C_{12}H_{10}O$ $[M–H]^-$: 169.06589; found: 169.06584

Column chromatography: R_f = 0.4 (H/EA 5:1)

3,5-dimethylphenol (**2h**)[4]



Appearance: pale yellow solid

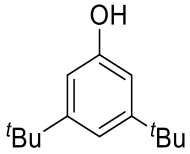
^{1}H NMR (500 MHz, $CDCl_3$): δ 6.58 (s, 1H), 6.46 (s, 2H), 4.60 (s, 1H), 2.27 (s, 6H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 155.51, 139.69, 122.69, 131.13, 21.39

QEFMS (ESI): m/z calcd for $C_8H_{10}O$ $[M–H]^-$: 121.06589; found: 121.06575

Column chromatography: R_f = 0.5 (H/EA 5:1)

3,5-di-*tert*-butylphenol (**2i**)[5]



Appearance: pale yellow solid

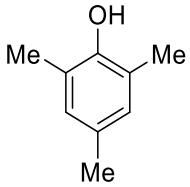
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.01 – 6.99 (t, *J* = 1.60 Hz, 1H), 6.70 – 6.68 (d, *J* = 1.55 Hz, 2H), 4.64 (s, 1H), 1.30 (s, 18H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 155.01, 152.75, 115.08, 109.77, 34.99, 31.52

QEFMS (ESI): m/z calcd for $C_{14}H_{22}O$ $[M–H]^-$: 205.15979; found: 205.15960

Column chromatography: R_f = 0.5 (H/EA 5:1)

2,4,6-trimethylphenol (**2j**)[6]



Appearance: white solid

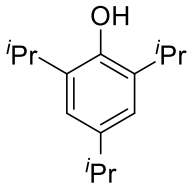
^{1}H NMR (500 MHz, $CDCl_3$): δ 6.79 (s, 2H), 4.44 (s, 1H), 2.21 (s, 9H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 150.00, 129.42, 129.24, 122.88, 20.52, 15.97

QEFMS (ESI): m/z calcd for $C_9H_{12}O$ $[M–H]^-$: 135.08154; found: 135.08145

Column chromatography: R_f = 0.6 (H/EA 5:1)

2,4,6-triisopropylphenol (**2k**)[7]



Appearance: yellow oil

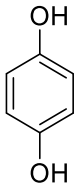
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.93 (s, 2H), 4.65 (s, 1H), 3.22 – 3.11 (sept, *J* = 6.87 Hz, 2H), 2.92 – 2.80 (sept, *J* = 6.92 Hz, 1H), 1.31 – 1.28 (d, *J* = 6.84 Hz, 12H), 1.27 – 1.24 (d, *J* = 6.96 Hz, 6H),

^{13}C NMR (125 MHz, $CDCl_3$): δ 148.05, 140.86, 133.42, 121.42, 33.99, 27.48, 24.50, 22.84

QEFMS (ESI): m/z calcd for $C_{15}H_{24}O$ $[M–H]^-$: 219.17544; found: 219.17542

Column chromatography: R_f = 0.7 (H/EA 5:1)

4-hydroxylphenol (**2l**)[2]



Appearance: white solid

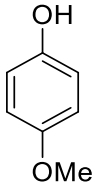
^{1}H NMR (500 MHz, DMSO-*d6*): δ 8.63 (s, 2H), 6.55 – 6.54 (d, 4H)

^{13}C NMR (125 MHz, DMSO-*d6*): δ 149.76, 115.68

QEFMS (ESI): m/z calcd for $C_6H_6O_2$ $[M–H]^-$: 109.02950; found: 109.02941

Column chromatography: R_f = 0.4 (H/EA 2:1)

4-methoxyphenol (**2m**)[1]



Appearance: while solid

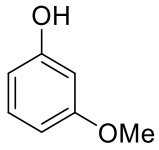
^{1}H NMR (500 MHz, $CDCl_3$): δ 6.81 – 6.75 (m, 4H), 4.74 (s, 1H), 3.77 (s, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 153.86, 149.58, 116.17, 114.97, 55.93

QEFMS (ESI): m/z calcd for $C_7H_8O_2$ $[M–H]^-$: 123.04515; found: 123.04504

Column chromatography: R_f = 0.3 (H/EA 5:1)

3-methoxyphenol (**2n**)[6]



Appearance: pale yellow oil

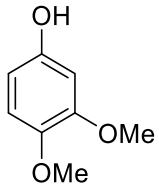
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 – 7.11 (t, *J* = 8.12 Hz, 1H), 6.52 – 6.50 (dt, $^3J_{ortho}$ = 8.09 Hz, $^4J_{meta}$ = 1.32 Hz, 1H), 6.45 – 6.44 (m, 1H), 6.43 – 6.41 (m, 1H), 5.17 (s, 1H), 3.78 (s, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 161.10, 156.83, 130.28, 107.86, 106.58, 101.62, 55.41

QEFMS (ESI): m/z calcd for $C_7H_8O_2$ $[M–H]^–$: 123.04515; found: 123.04517

Column chromatography: R_f = 0. 3 (H/EA 5:1)

3,4-dimethoxyphenol (**2o**)[2]



Appearance: pale red oil

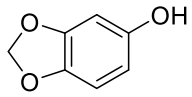
^{1}H NMR (500 MHz, $CDCl_3$): δ 6.74 – 6.71 (d, $^3J_{ortho}$ = 8.60 Hz, 1H), 6.48 – 6.46 (d, $^4J_{meta}$ = 2.80 Hz, 1H), 6.36 – 6.32 (dd, $^3J_{ortho}$ = 8.58 Hz, $^4J_{meta}$ = 2.83 Hz, 1H), 4.73 (bs, 1H), 3.84 (s, 1H), 3.82 (s, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 150.12, 150.04, 143.39, 112.36, 105.84, 100.66, 56.65, 55.97

QEFMS (ESI): m/z calcd for $C_8H_{10}O_3$ $[M+Na]^+$: 177.05222; found: 177.05209

Column chromatography: R_f = 0.2 (H/EA 5:1)

3,4-(methylenedioxy)phenol (**2p**)[4]



Appearance: pale red oil

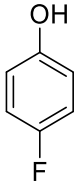
^{1}H NMR (500 MHz, $CDCl_3$): δ 6.66 – 6.64 (d, $^3J_{ortho}$ = 8.30 Hz, 1H), 6.43 – 6.42 (d, $^4J_{meta}$ = 2.50 Hz, 1H), 6.26 – 6.23 (dd, $^3J_{ortho}$ = 8.30 Hz, $^4J_{meta}$ = 2.50 Hz, 1H), 5.91 (s, 1H), 4.58 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 150.79, 148.43, 141.71, 108.26, 106.75, 101.32, 98.41

QEFMS (ESI): m/z calcd for $C_7H_6O_3$ $[M–H]^-$: 137.02442; found: 137.02428

Column chromatography: R_f = 0.3 (H/EA 5:1)

4-fluorophenol (**2q**)[2]



Appearance: while solid

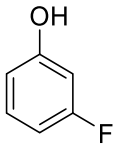
^{1}H NMR (500 MHz, $CDCl_3$): δ 6.96 – 6.990 (m, 2H), 6.80 – 6.75 (m, 2H), 4.80 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.35, 156.46, 151.57, 116.37 – 116.03 (dd)

QEFMS (ESI): m/z calcd for C_6H_5OF $[M–H]^-$: 111.02517; found: 111.02503

Column chromatography: R_f = 0.3 (H/EA 5:1)

3-fluorophenol (**2r**)[8]



Appearance: colorless oil

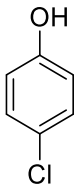
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.21 – 7.15 (m, 1H), 6.67 – 6.57 (m, 3H), 5.34 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 164.72, 162.77, 156.96 – 156.87 (d), 130.67 – 130.59 (d), 111.28 – 111.26 (d), 107.97 – 107.80 (d), 103.16 – 103.26 (d)

QEFMS (ESI): m/z calcd for C_6H_5OF $[M–H]^-$: 111.02517; found: 111.02520

Column chromatography: R_f = 0.4 (H/EA 5:1)

4-chlorophenol (**2s**)[3]



Appearance: white solid

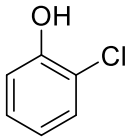
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.21 – 7.17 (dt, $^3J_{ortho}$ = 8.75 Hz, $^4J_{meta}$ = 2.69 Hz, 2H), 6.79 – 6.75 (dt, $^3J_{ortho}$ = 8.85 Hz, $^4J_{meta}$ = 2.66 Hz, 2H), 4.92 (s, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 154.15, 129.67, 125.85, 116.78

QEFMS (ESI): m/z calcd for C_6H_5OCl $[M–H]^-$: 126.99562; found: 126.99561

Column chromatography: R_f = 0.4 (H/EA 5:1)

2-chlorophenol (**2t**)[6]



Appearance: colorless oil

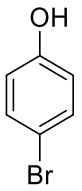
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 – 7.31 (dd, $^3J_{ortho}$ = 8.00 Hz, $^4J_{meta}$ = 1.30 Hz, 1H), 7.21 – 7.16 (td, $^3J_{ortho}$ = 7.76 Hz, $^4J_{meta}$ = 1.12 Hz, 1H), 7.04 – 7.01 (dd, $^3J_{ortho}$ = 8.18 Hz, $^4J_{meta}$ = 1.23 Hz, 1H), 6.90 – 6.85 (td, $^3J_{ortho}$ = 7.70 Hz, $^4J_{meta}$ = 1.12 Hz, 1H), 5.60 (s, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 151.46, 129.13, 128.54, 121.50, 120.00, 116.39

QEFMS (ESI): m/z calcd for C_6H_5OCl $[M–H]^-$: 126.99562; found: 126.99567

Column chromatography: R_f = 0.4 (H/EA 5:1)

4-bromophenol (**2u**)[4]



Appearance: yellow oil

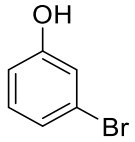
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 – 7.31 (dt, $^3J_{ortho}$ = 8.90 Hz, $^4J_{meta}$ = 2.78 Hz, 2H), 6.74 – 6.70 (dt, $^3J_{ortho}$ = 8.85 Hz, $^4J_{meta}$ = 2.75 Hz, 2H), 4.83 (s, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 154.77, 132.61, 117.32, 113.01

QEFMS (ESI): m/z calcd for C_6H_5OBr $[M–H]^-$: 170.94510; found: 170.94518

Column chromatography: R_f = 0.4 (H/EA 5:1)

3-bromophenol (**2v**)[6]



Appearance: yellow oil

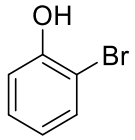
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.12 – 7.06 (m ,2H), 7.02 – 7.01 (m, 1H), 6.78 – 6.75 (m, 1H), 5.03 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.46, 130.93, 124.12, 122.93, 118.93, 114.36

QEFMS (ESI): m/z calcd for C_6H_5OBr $[M–H]^-$: 170.94510; found: 170.94521

Column chromatography: R_f = 0.4 (H/EA 5:1)

2-bromophenol (**2w**)[1]



Appearance: pale yellow oil

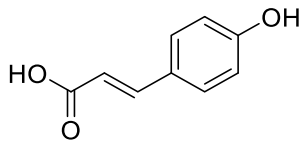
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48 – 7.45 (dd, $^3J_{ortho}$ = 8.00 Hz, $^4J_{meta}$ = 1.20 Hz, 1H), 7.24 – 7.20 (td, $^3J_{ortho}$ = 7.73 Hz, $^4J_{meta}$ = 1.10 Hz, 1H), 7.04 – 7.02 (dd, $^3J_{ortho}$ = 8.13 Hz, $^4J_{meta}$ = 1.18 Hz, 1H), 6.83 – 6.79 (td, $^3J_{ortho}$ = 7.68 Hz, $^4J_{meta}$ = 1.15 Hz, 1H), 5.57 (s, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 152.36, 132.16, 129.31, 121.94, 116.27, 110.37

QEFMS (ESI): m/z calcd for C_6H_5OBr $[M–H]^-$: 170.94510; found: 170.94531

Column chromatography: R_f = 0.5 (H/EA 5:1)

(*E*)-4-hydroxycinnamic acid (**2x**)[9]



The arylboronic acid substrate **1x** was prepared according to K. Khaldoun et al. *Synthesis* **2019**, *51*, 3891–3900.

Appearance: white solid

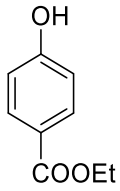
^{1}H NMR (500 MHz, DMSO-*d6*): δ ~10 (bs, 1H), 7.52 – 7.49 (d, *J* = 8.49 Hz, 2H), 7.49 – 7.45 (d, *J* = 16.10 Hz, 1H), 6.79 – 6.77 (d, *J* = 8.24 Hz, 2H), 6.30 – 6.26 (d, *J* = 15.89 Hz, 2H)

^{13}C NMR (125 MHz, DMSO-*d6*): δ 168.10, 159.61, 144.02, 130.11, 125.34, 115.78, 115.64

QEFMS (ESI): m/z calcd for $C_9H_8O_3$ $[M–H]^-$: 163.04007; found: 163.04009

Column chromatography: R_f = 0.3 (H/EA 1:1)

Ethyl 4-hydroxybenzoate (**2y**)[10]



Appearance: white solid

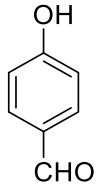
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 – 7.94 (dt, $^3J_{ortho}$ = 8.80 Hz, $^4J_{meta}$ = 2.38 Hz, 2H), 6.89 – 6.85 (dt, $^3J_{ortho}$ = 8.80 Hz, $^4J_{meta}$ = 2.38 Hz, 2H), 6.17 (s, 1H), 4.38 – 4.32 (q, *J* = 7.12 Hz, 2H), 1.40 – 1.36 (t, *J* = 7.08 Hz, 3H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 166.91, 160.16, 132.02, 122.91, 115.32, 61.01, 14.48

QEFMS (ESI): m/z calcd for $C_9H_{10}O_3$ $[M–H]^-$: 165.05572; found: 165.05544

Column chromatography: R_f = 0.2 (H/EA 5:1)

4-hydroxybenaldehyde (**2z**)[8]



Appearance: pale yellow solid

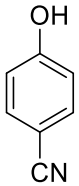
^{1}H NMR (500 MHz, $CDCl_3$): δ 9.86 (s, 1H), 7.84 – 7.80 (d, *J* = 8.55 Hz, 2H), 6.99 – 6.96 (d, *J* = 8.55 Hz, 2H), 6.39 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 191.36, 161.73, 132.66, 129.98, 116.14

QEFMS (ESI): m/z calcd for $C_7H_6O_2$ $[M–H]^-$: 121.02950; found: 121.02930

Column chromatography: R_f = 0.1 (H/EA 5:1)

4-cyanophenol (**2aa**)[3]



Appearance: white solid

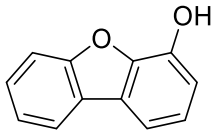
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 – 7.53 (dt, $^3J_{ortho}$ = 8.79 Hz, $^4J_{meta}$ = 2.14 Hz, 2H), 6.92 – 6.89 (dt, $^3J_{ortho}$ = 8.85 Hz, $^4J_{meta}$ = 2.08 Hz, 2H), 6.25 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 159.75, 134.31, 119.21, 116.37, 103.73

QEFMS (ESI): m/z calcd for C_7H_5NO $[M–H]^-$: 118.02984; found: 118.02970

Column chromatography: R_f = 0.5 (H/EA 2:1)

Dibenzo[b,d]furan-4-ol (**2ab**)[11]



Appearance: white solid

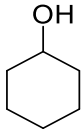
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 – 7.93 (d, *J* = 7.67 Hz, 1H), 7.59 – 7.56 (d, *J* = 8.25 Hz, 1H), 7.54 – 7.51 (d, *J* = 7.70 Hz, 1H), 7.49 – 7.45 (t, *J* = 7.73 Hz, 1H), 7.38 – 7.34 (t, *J* = 7.47 Hz, 1H), 7.25 – 7.21 (t, *J* = 7.81 Hz, 1H), 7.07 – 7.04 (d, *J* = 7.91 Hz, 1H), 5.83 (bs, 1H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.11, 144.16, 141.21, 127.34, 125.85, 124.66, 123.77, 123.06, 121.10, 113.75, 112.89, 111.87

QEFMS (ESI): m/z calcd for $C_{12}H_8O_2$ $[M–H]^-$: 183.04515; found: 183.04503

Column chromatography: R_f = 0.4 (H/EA 4:1)

Cyclohexanol (**2ac**)[12]



Appearance: colorless oil

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.64 – 3.57 (dq, *J* = 8.8, 4.1 Hz, 1H), 1.92 – 1.86 (m, 2H), 1.76 – 1.71 (m, 2H), 1.57 – 1.50 (m, 2H), 1.31 – 1.23 (m, 4H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 70.48, 35.68, 25.58, 24.26

Column chromatography: R_f = 0.3 (H/EA 4:1)

(D) Studies on oxidant-sensitive substrates

Ipso-hydroxylation of 4-(methylthio)phenylboronic acid **1a**

Using substrate **1a**, the mild **HE1**/O_2 protocol exclusively oxidized the boronic acid, keeping the thioether group intact (Scheme S1a). However, using H_2O_2 as the oxidant showed unsatisfactory selectivity. Under the literature conditions,[6] low conversion was attained and trace a small amount of sulfoxide **2a'** was detected (Scheme S1b-i). In prolonged reaction time, the reaction progress was monitored (Figure S1) and a higher conversion was achieved but more sulfoxide **2a'** was formed (Scheme S1b-ii). When the oxidant was added in far excess according to another literature method (Scheme S1b-iii),[13] the over oxidized product sulfone **2a''** was formed quantitative. When reduced amount of H_2O_2 (1.0 or 1.5 equiv) was used, the efficiency dropped and sulfoxides were still generated (Scheme S1b-iv and v).

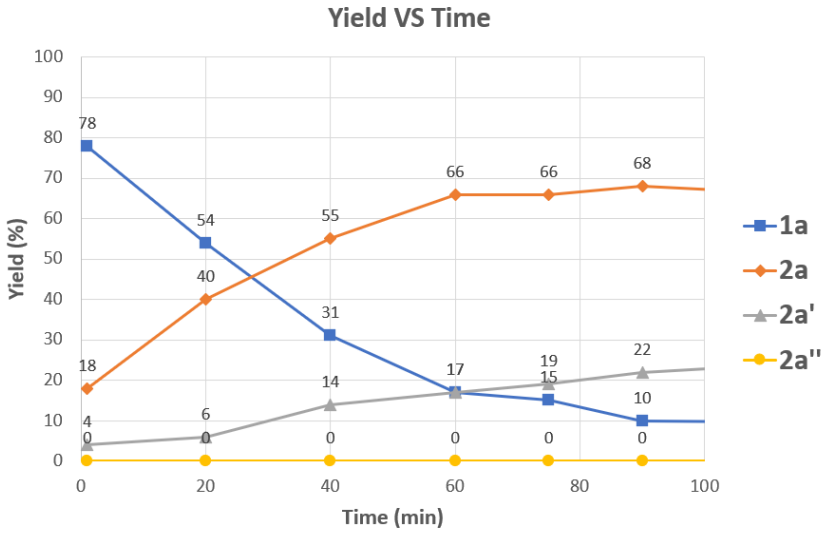
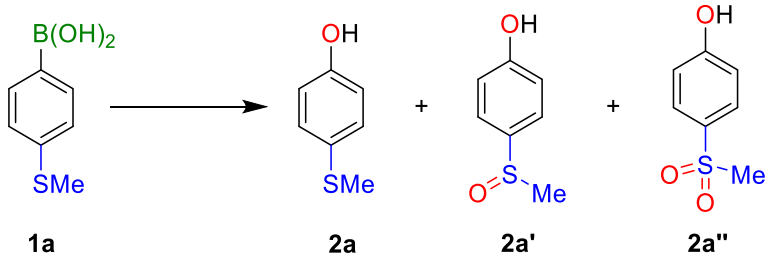


Figure S1 Reaction progress with substrate **1a** and oxidant H_2O_2 using the conditions in Scheme S1b-ii.

Scheme S1 Chemoselectivity performance in the oxidation of substrate **1a**



1a **2a** **2a'** **2a''**

(a) This work[a]

1a —(**HE1** (1.5 equiv), O_2, acetone, 16 h)→ **2a:** 95% **2a':** 0% **2a'':** 0%

(b) Oxidation using H_2O_2[b]

	1a		**2a**	**2a'**	**2a''**
(i)	78%	30% H_2O_2 (3.0 equiv), EtOH, 1 min	18%	4%	0%
(ii)	10%	30% H_2O_2 (3.0 equiv), EtOH, 90 min	68%	22%	0%
(iii)	0%	30% H_2O_2 (40.0 equiv), EtOH, 16 h	0%	0%	100%
(iv)	50%	30% H_2O_2 (1.0 equiv), EtOH, 16 h	38%	12%	0%
(v)	32%	30% H_2O_2 (1.5 equiv), EtOH, 16 h	48%	20%	0%

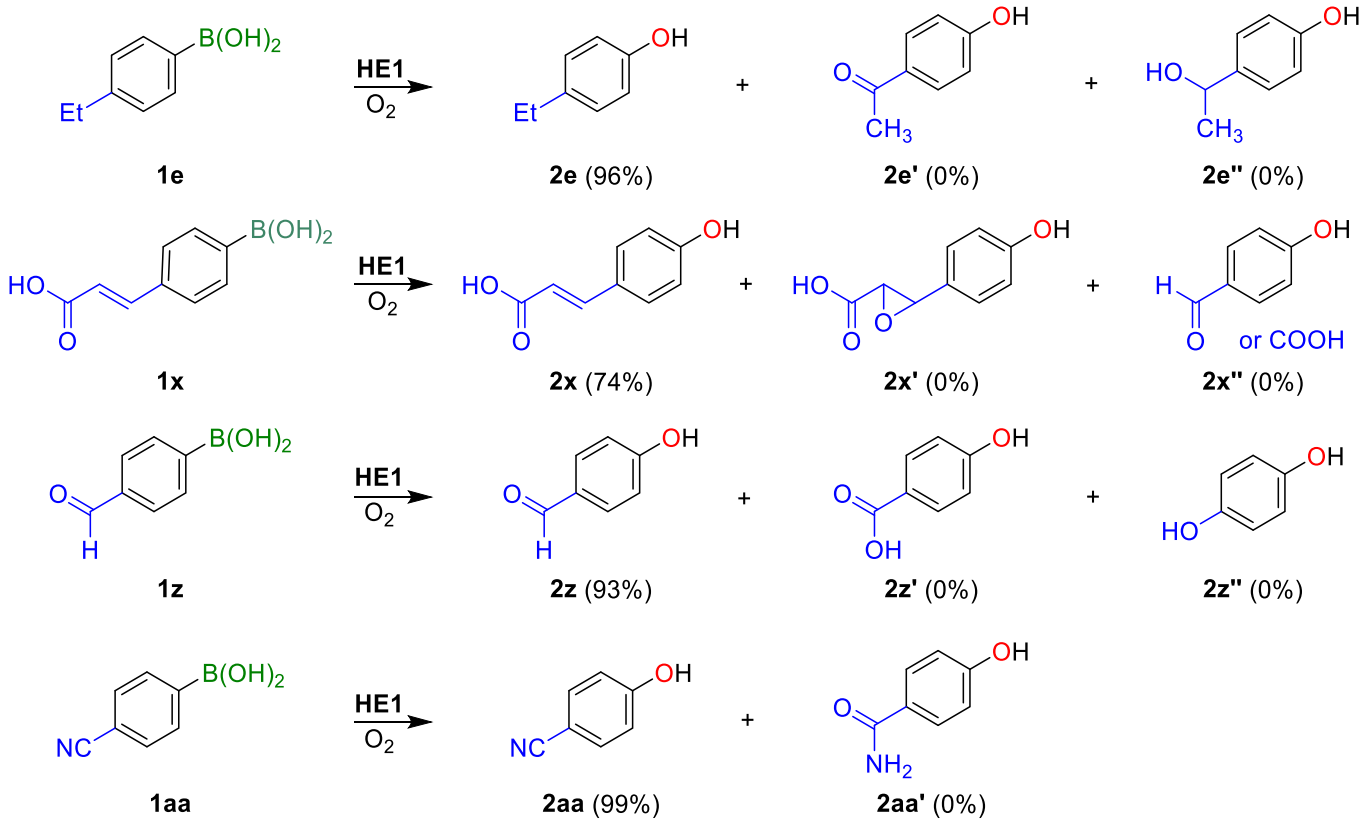
[a] Standard reaction conditions. Isolated yield.

[b] Reaction conditions: 4-(methylthio)phenylboronic acid **1a** (0.5 mmol), H_2O_2, EtOH (0.33 M), r.t. NMR yields using dibromomethane as the internal standard.

Ipso-hydroxylation of phenylboronic acids **1e, 1x, 1z** and **1aa**

Substrates bearing benzylic ethyl (**1e**), propenoic acid (**1x**), aldehyde (**1z**), and cyano (**1aa**) groups could be over oxidized using some literature protocols such as ascorbate/quinone.[1] In sharp contrast, these substituents survived using the **HE1**/O_2 protocol (Scheme S2). For instance, boronic acid **2e** was obtained and no benzylic oxidation product **2e'** was detected. For the case with substrate **1x**, product **2x** was obtained in 74% yield and no epoxide **2x'** was formed. Boronic acid **2z** was obtained in excellent yield and the aldehyde remained intact. The cyano group in **1aa** also well-survived and boronic **2aa** was obtained quantitative and no amide **2aa'** was detected.

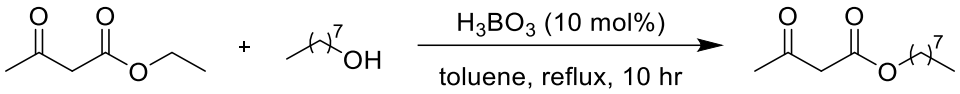
Scheme S2 Chemoselectivity of the oxidation of substrates **1e**, **1x**, **1z**, and **1aa** under optimized conditions[a]



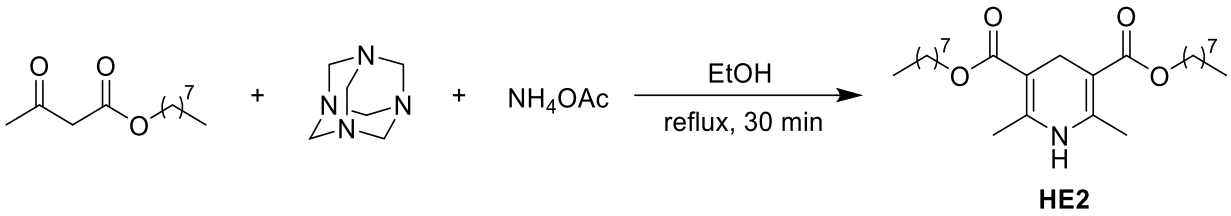
[a] Standard reaction conditions.

(E) Studies on the initial rate of reaction

Synthesis of dioctyl 2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate (**HE2**) and dioctyl 2,6-dimethylpyridine-3,5-dicarboxylate (**HP2**)



Octyl acetoacetate was prepared according to the modified literature procedure.[14] To a round-bottom flask was charged with ethyl acetoacetate (5.7 mL, 45 mmol), *n*-octanol (10.6 mL, 67.5 mmol) and toluene (200 mL), followed by the addition of boric acid catalyst (0.2 mL, 10 mol%). A pressure-equalized addition funnel containing a cotton plug and 5 Å molecular sieves (pellets) was directly attached above the round-bottom flask to remove ethanol, a water condenser was subsequently connected to the addition funnel. The resultant solution mixture was heated at reflux for 10 hours with continuous removal of ethanol. The solution was concentrated under reduced pressure. The residue was purified by flash column chromatography using silica gel as the stationary phase and *n*-hexane/ethyl acetate as the gradient eluent to afford octyl acetoacetate.



HE2

HE2 was prepared according to the modified literature procedure.[15] To a Schlenk flask was charged with octyl acetoacetate (4.5 mL, 20 mmol), hexamethylenetetramine (0.4 g, 3 mmol), ammonium acetate (0.4 g, 5 mmol) and ethanol (5 mL) under nitrogen atmosphere. The resultant mixture was heated at reflux for 30 minutes and subsequently cooled to room temperature. The light-yellow precipitate was filtered and recrystallized from ethanol to afford **HE2**.

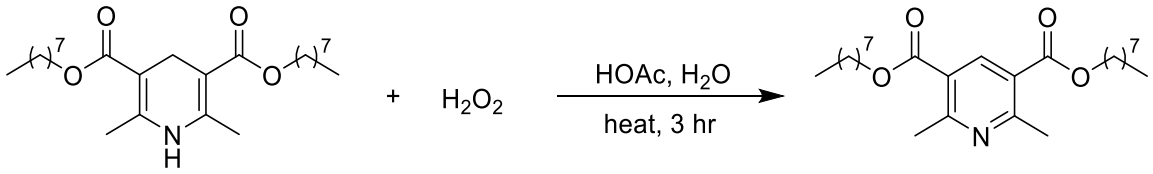
Dioctyl 2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate (**HE2**)[14]

Appearance: light yellow solid

^{1}H NMR (500 MHz, $CDCl_3$): δ 5.15 (s, 1H), 4.11 – 4.07 (t, *J* = 6.68 Hz, 4H), 3.27 (s, 2H), 2.19 (s, 6H), 1.68 – 1.61 (quin, *J* = 7.03 Hz, 4H), 1.38 – 1.26 (m, 20H), 0.89 – 0.86 (t, *J* = 6.88 Hz, 6H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 168.23, 144.84, 99.77, 64.02, 31.95, 29.39, 29.37, 28.93, 26.23, 24.95, 22.79, 19.31, 14.24

QEFMS (ESI): m/z calcd for $C_{25}H_{43}NO_4$ $[M+Na]^+$: 444.30843; found: 444.30806



HE2 **HP2**

The **HP2** was prepared according to the literature procedure.[16]

Dioctyl 2,6-dimethylpyridine-3,5-dicarboxylate (**HP2**)[14]

Appearance: pale yellow liquid

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.66 (s, 1H), 4.32 – 4.29 (t, *J* = 6.73 Hz, 4H), 2.83 (s, 6H), 1.79 – 1.73 (quin, *J* = 7.14 Hz, 4H), 1.45 – 1.24 (m, 20H), 0.88 – 0.85 (t, *J* = 6.85 Hz, 6H)

^{13}C NMR (125 MHz, $CDCl_3$): δ 166.12, 162.35, 141.11, 123.19, 65.69, 31.90, 29.33, 29.31, 28.74, 26.16, 15.10, 22.75, 14.20

QEFMS (ESI): m/z calcd for $C_{25}H_{41}NO_4$ $[M+H]^+$: 420.31084; found: 420.31007

Reaction progress monitored by NMR

To an NMR tube was charged with phenylboronic acid (**1c**) (3.0 mg, 0.025 mmol), **HE2** (10.5 mg, 0.025 mmol) and dibromomethane (7 μL, 0.1 mmol). Then, 500 μL of acetone-*d6* was added. The tube was filled with pure oxygen. The reaction progress was monitored by NMR with data collected automatically at stated time interval. **HP2** was added at the initial stage of reaction (orange line: 10 mol%; grey line: 0 mol%). The amounts of each component were used with respect to 1 equivalence of dibromomethane as the internal standard (Figure S2).

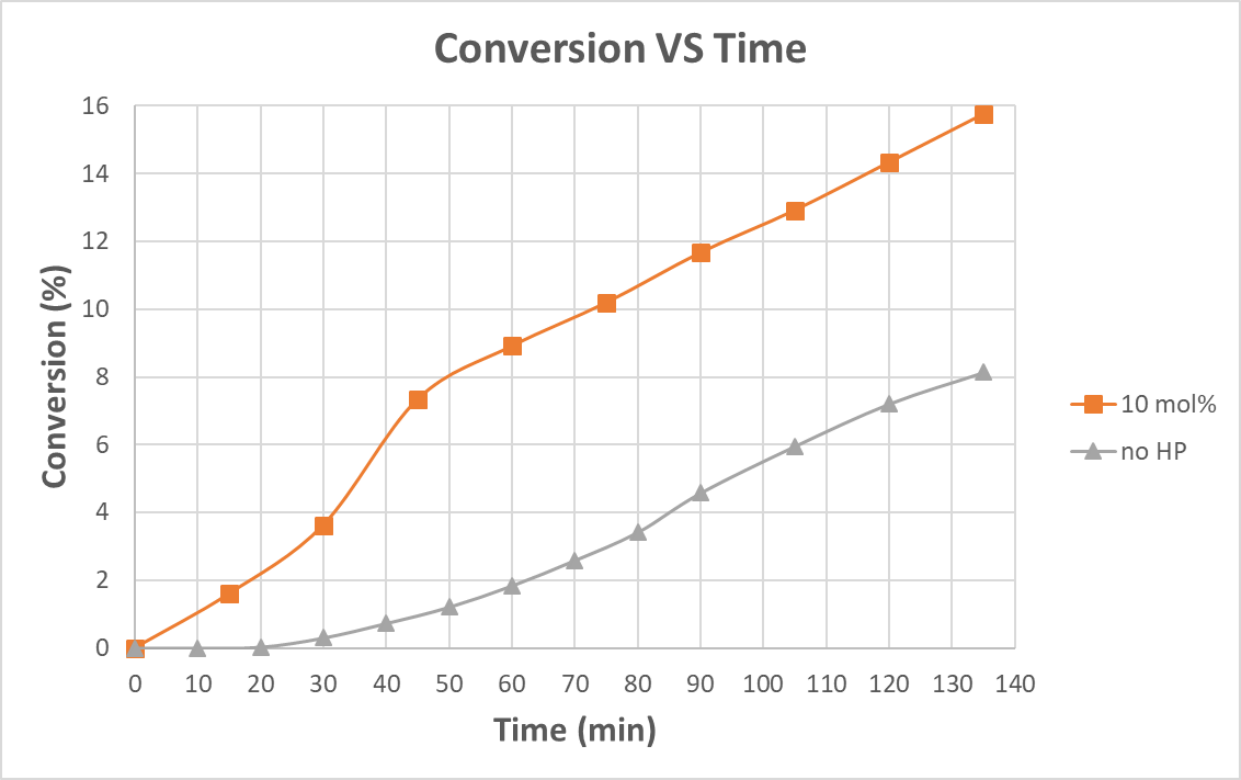


Figure S2 Reaction progress with **1c** and **HE2** monitored by NMR.

(F) Hantzsch pyridine impurity in Hantzsch ester

NMR study on the commercial sample of **HE1**

^{1}H NMR study was performed on the commercial bottle of **HE1**. It was found that c.a. 8% of **HP1** existed in the sample (Figure S3).

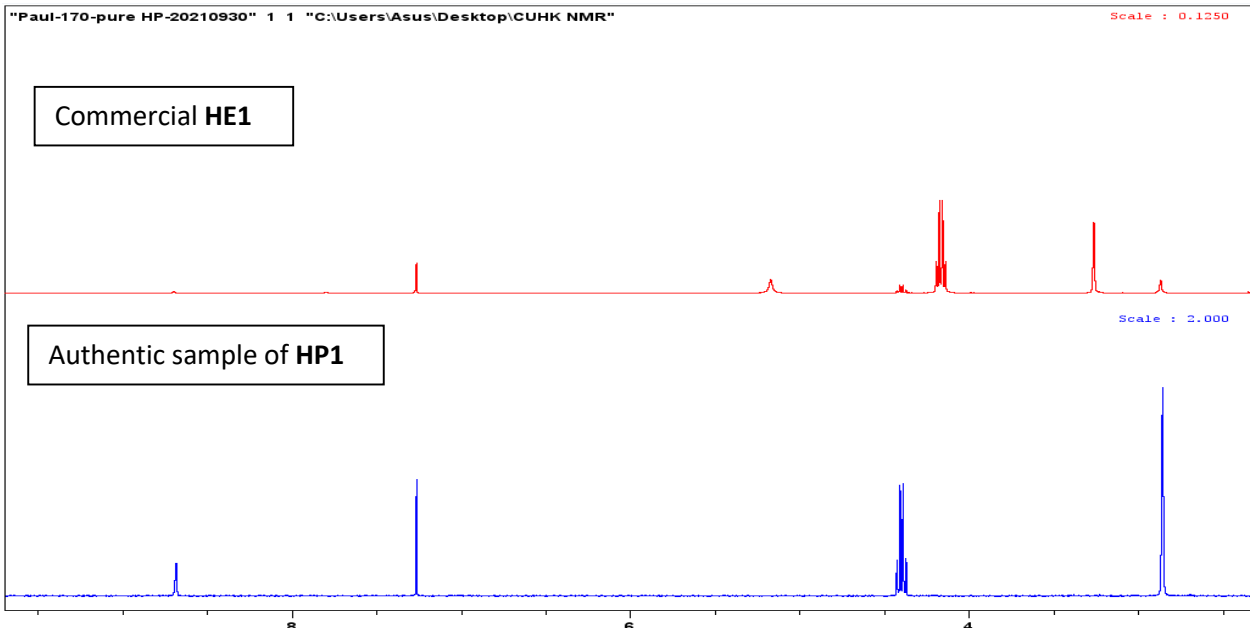


Figure S3 ^{1}H NMR study on the commercial **HE1**.

NMR study on the commercial sample of **HE1** after exposure under air

A solid sample of commercial **HE1** was exposed to air for one week (Figure S4). The amount of **HP1** increased by c.a. 2%, attributed to the air oxidation of **HE1**. So, we believe that oxygen might oxidize **HE1** to **HP1** during the reaction. The **HP1** would then enhance the reaction rate of boronic oxidation by promoting the generation of hydrogen peroxy radical. This might explain why the reaction rate could increase eventually even when **HP** was not added initially.

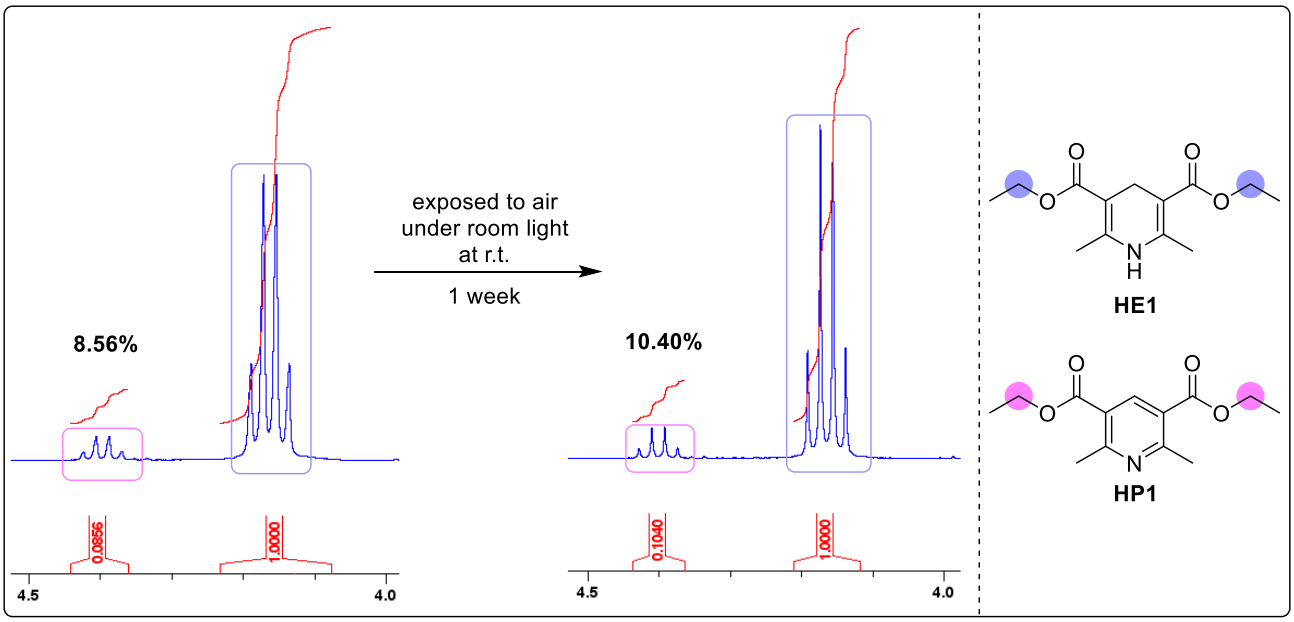


Figure S4 ^{1}H NMR study on the commercial **HE1** sample after 1 week of exposure under air.

(G) References

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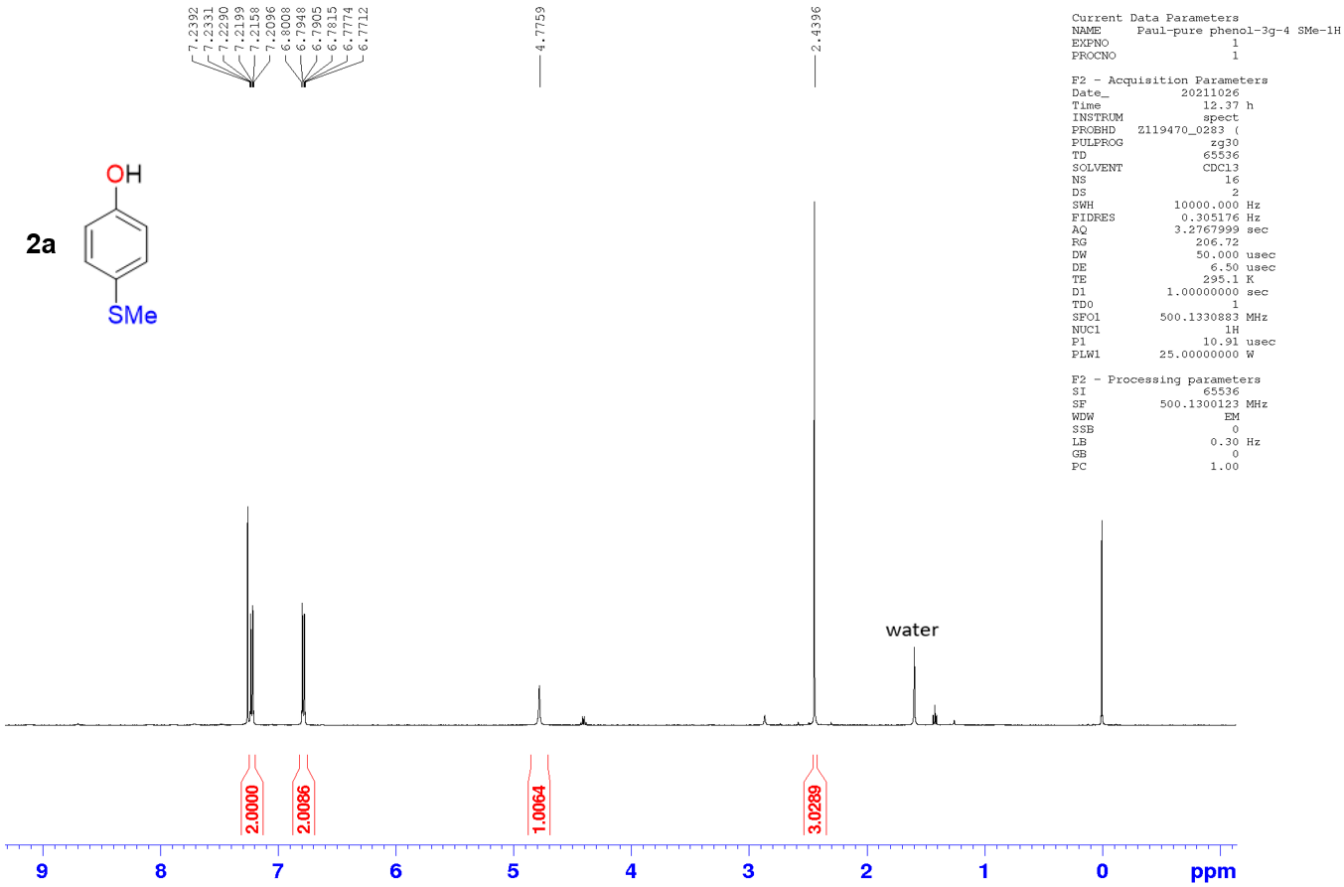
(H) NMR spectra

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PROCNO                  1

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DE                  6.50 usec
TE                 295.1 K
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PC                  1.00
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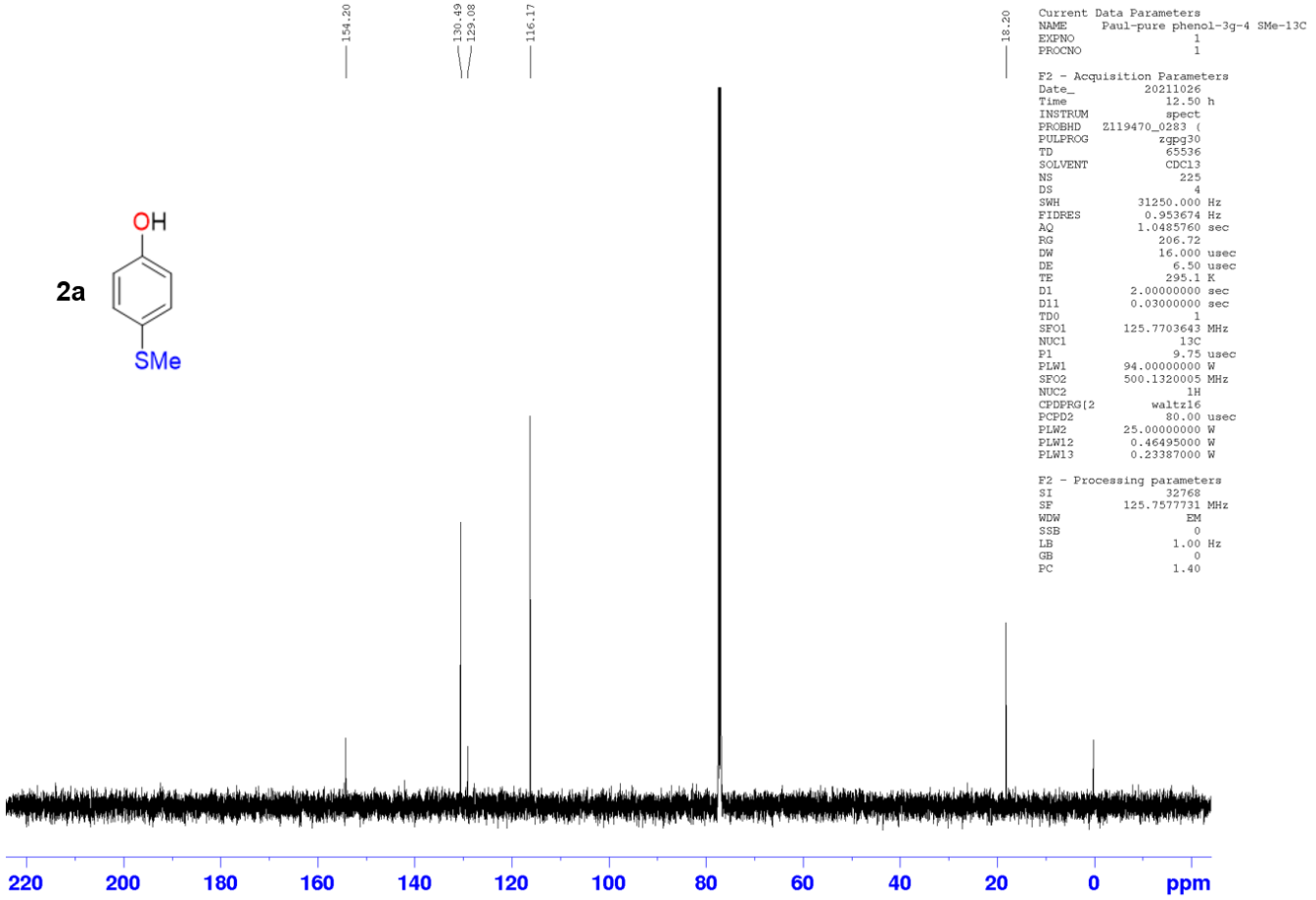
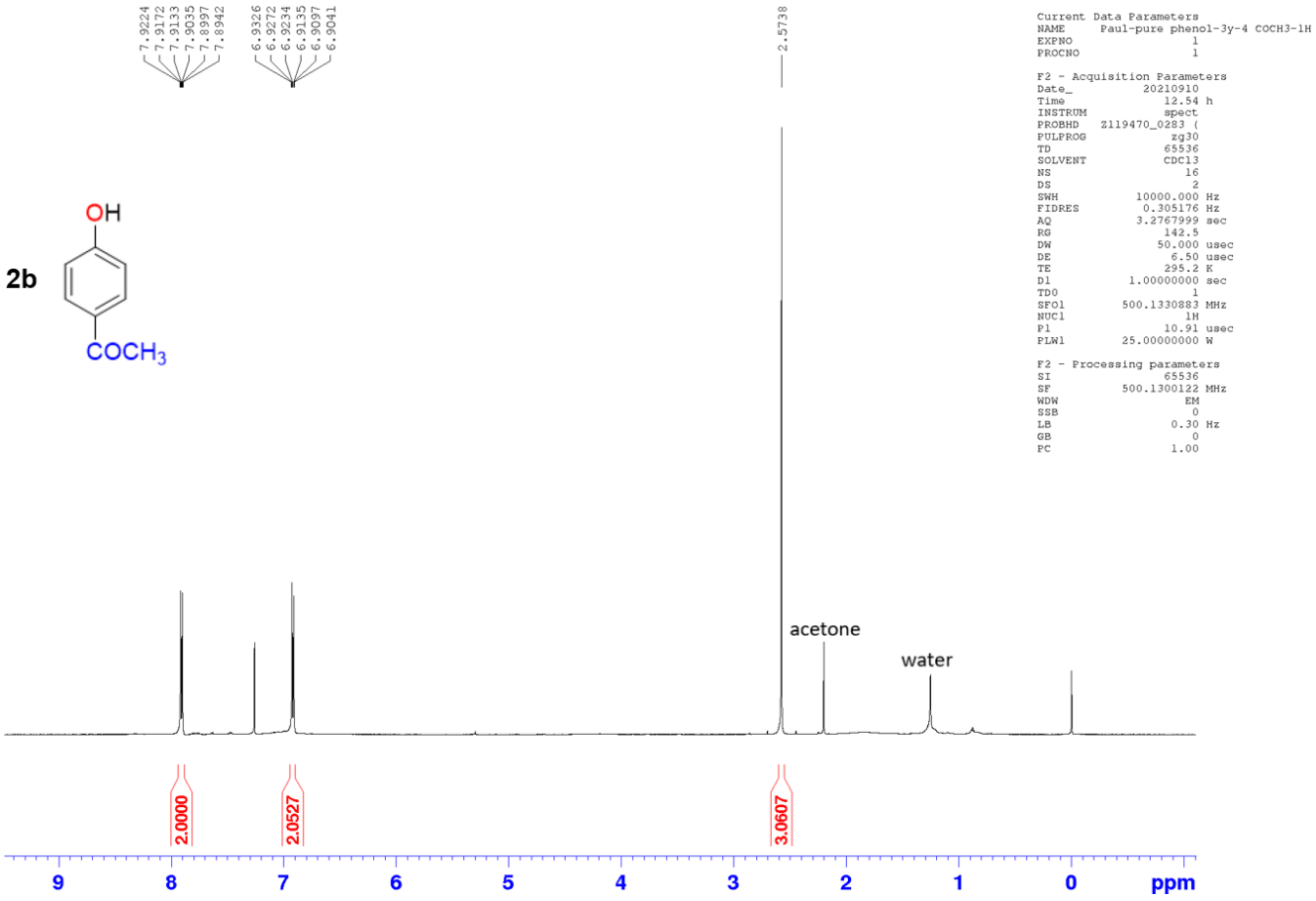
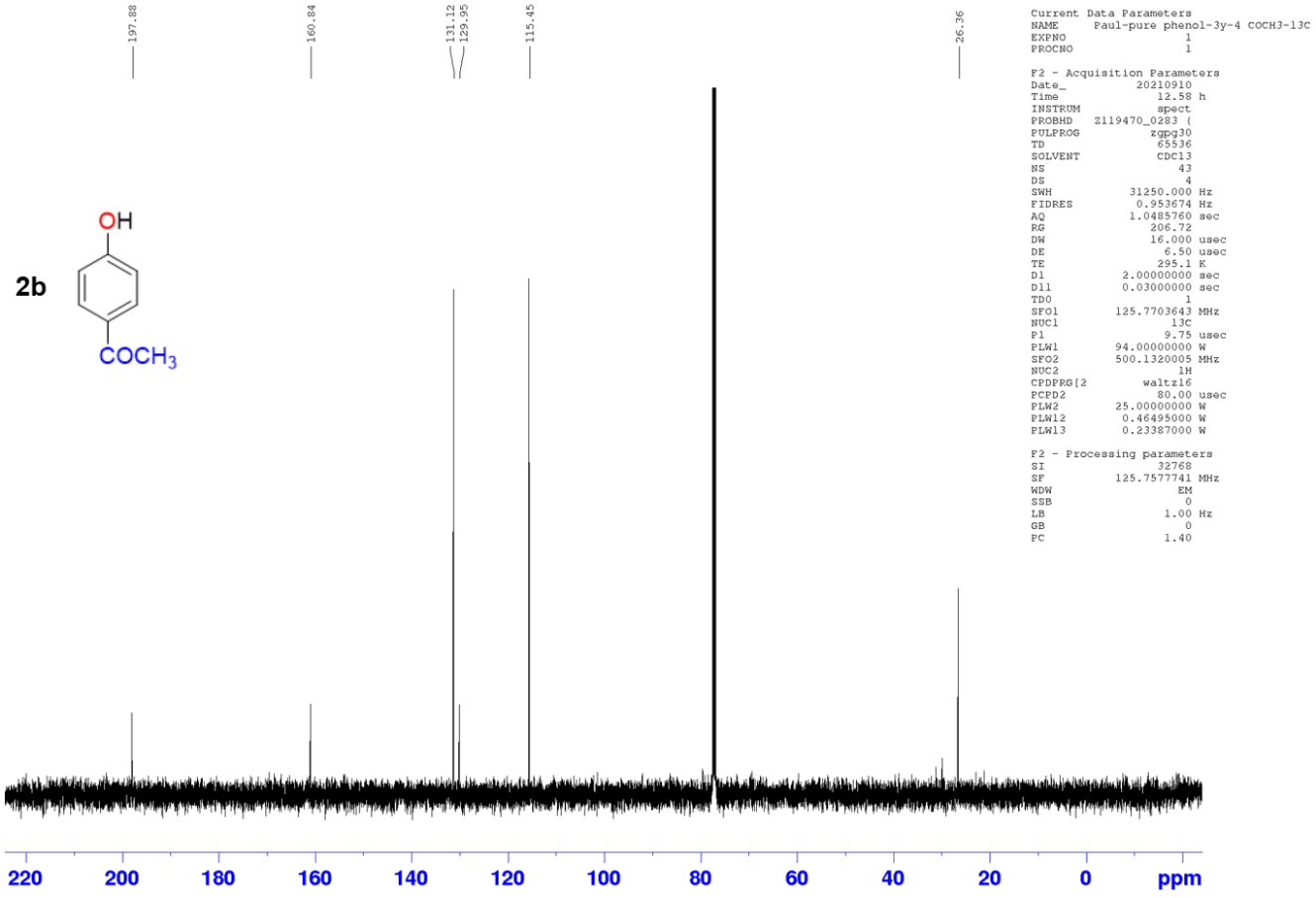


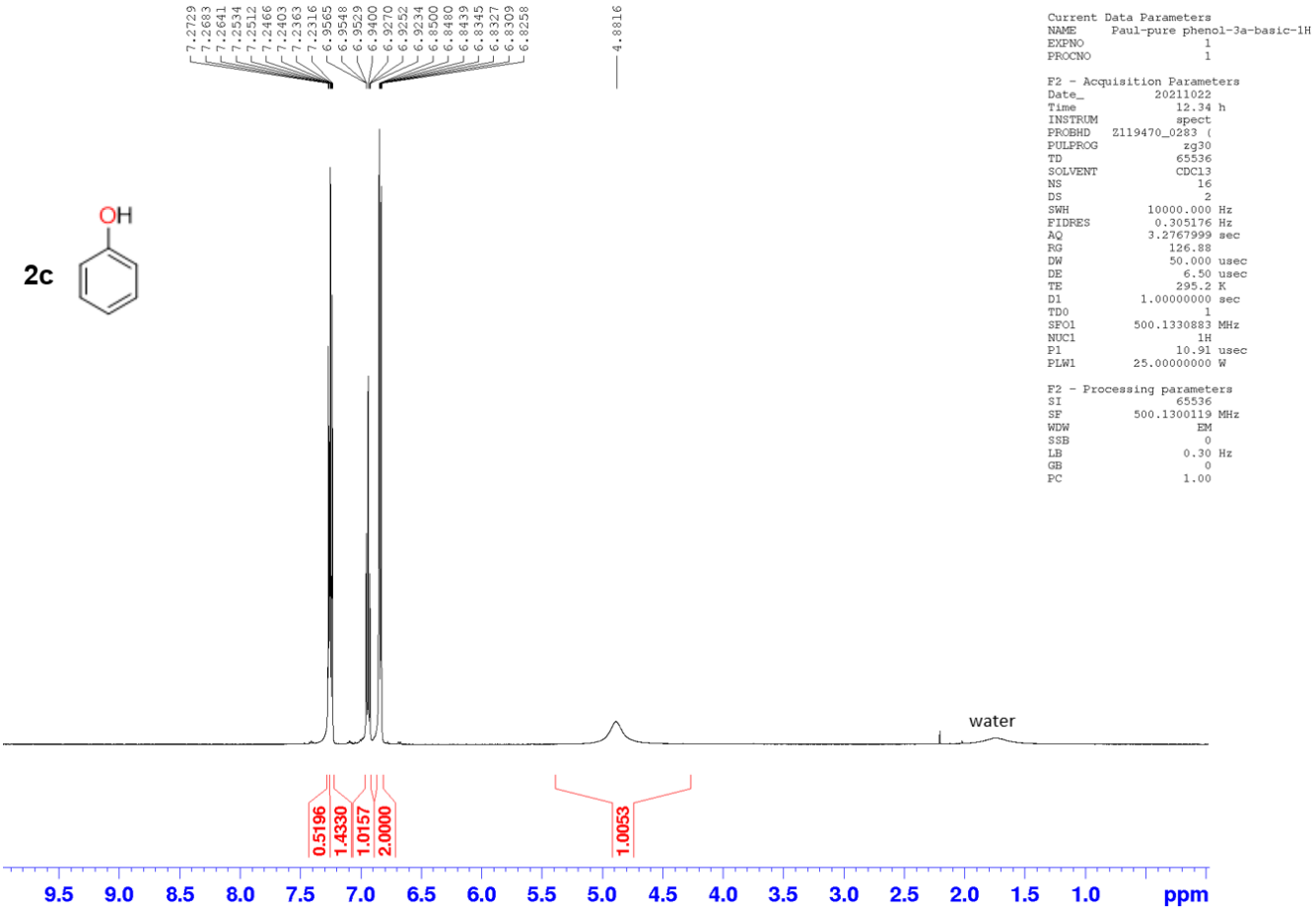

```
Current Data Parameters
NAME      Paul-pure phenol-3g-4 SMe-13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20211026
Time              12.50 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   225
DS                     4
SWH           31250.000 Hz
FIDRES            0.953674 Hz
AQ              1.0485760 sec
RG                 206.72
DW                 16.000 usec
DE                  6.50 usec
TE                 295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                  9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577731 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



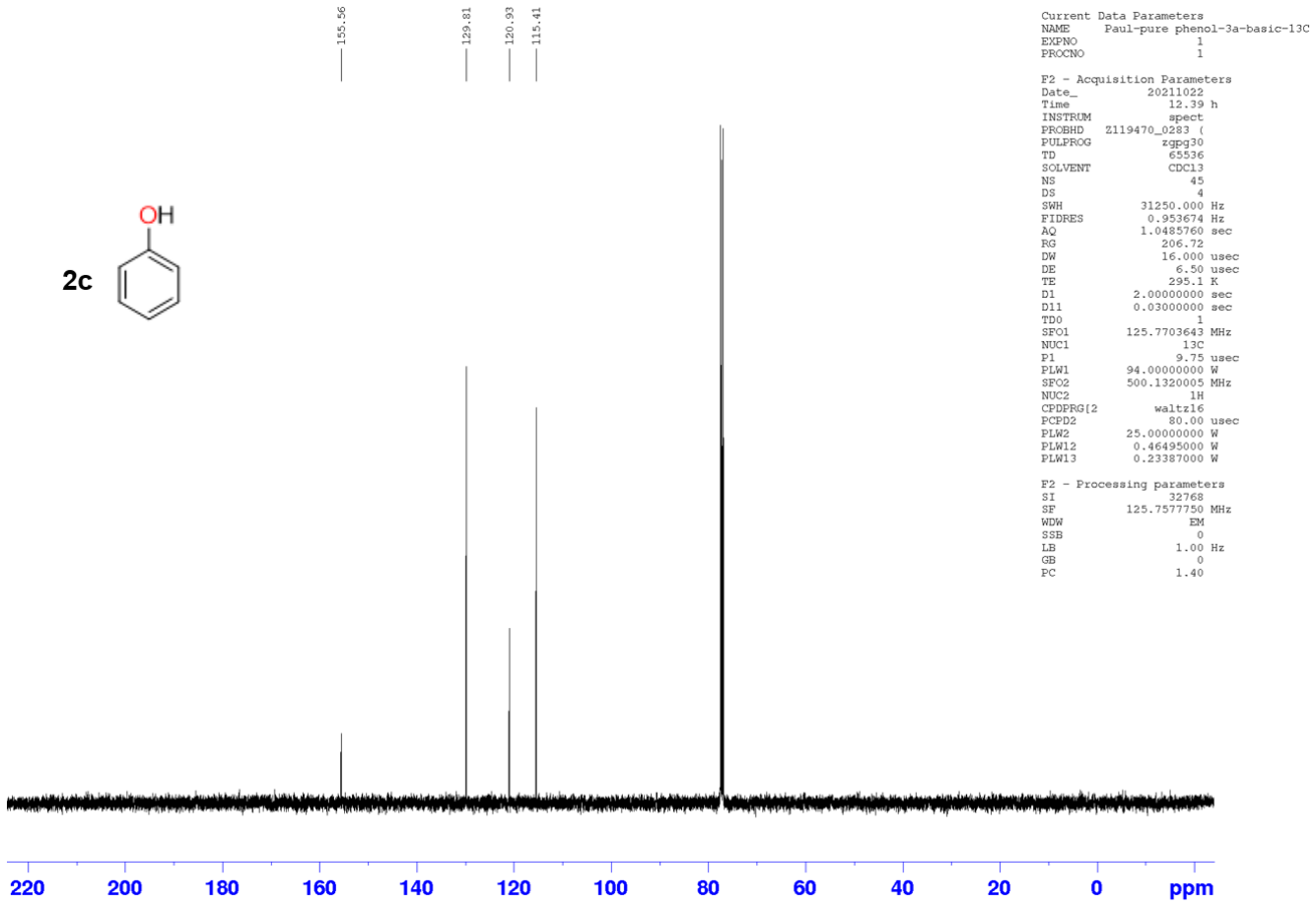

```
Current Data Parameters
NAME      Paul-pure phenol-3y-4 COCH3-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_               20210910
Time                  12.54 h
INSTRUM               spect
PROBHD    Z119470_0283 (
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10000.000 Hz
FIDRES              0.305176 Hz
AQ                3.2767999 sec
RG                    142.5
DW                   50.000 usec
DE                    6.50 usec
TE                   295.2 K
D1              1.00000000 sec
TD0                      1
SFO1           500.1330883 MHz
NUC1                    1H
P1                   10.91 usec
PLW1           25.00000000 W

F2 - Processing parameters
SI                   65536
SF              500.1300122 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



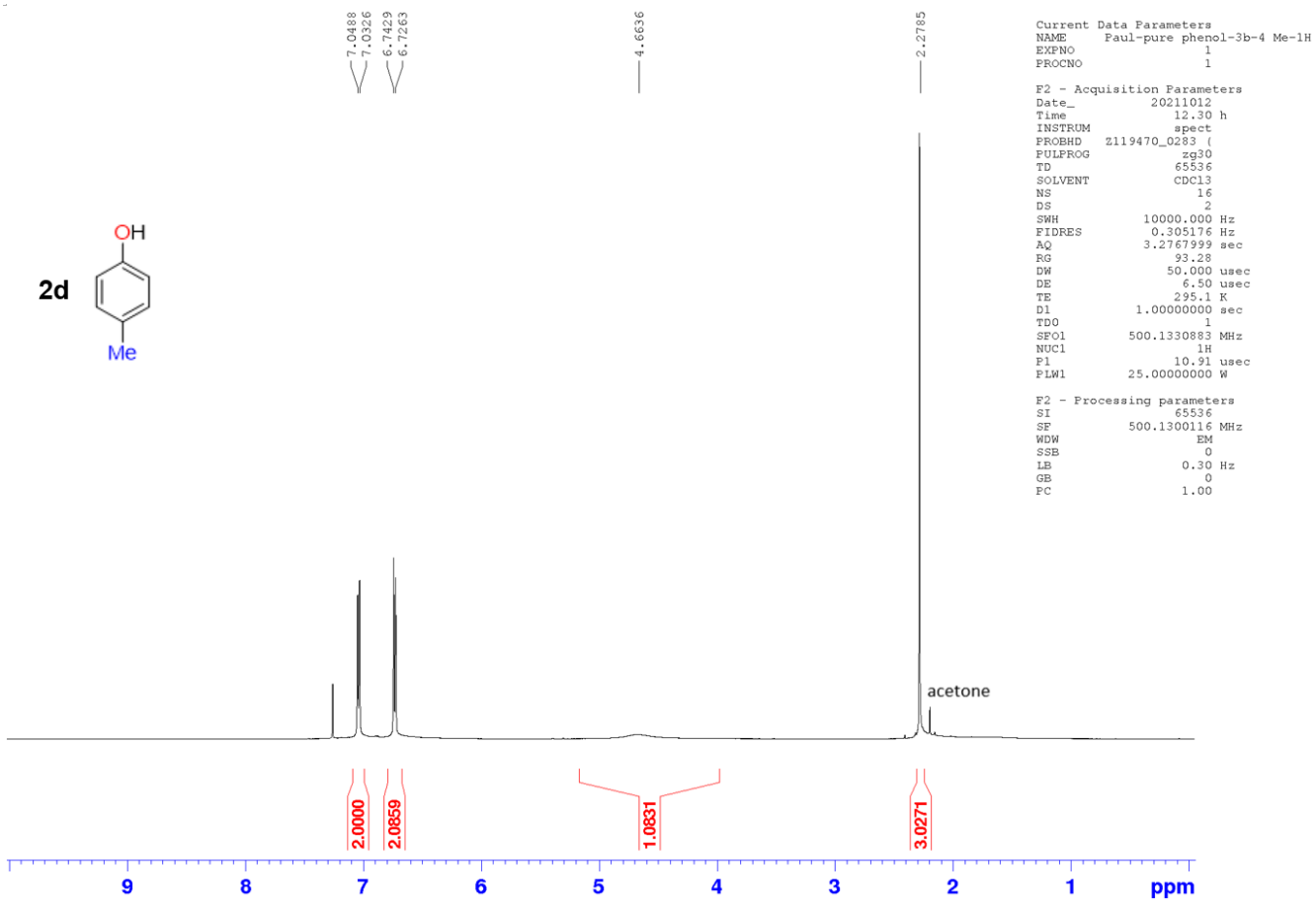

```
Current Data Parameters
NAME      Paul-pure phenol-3y-4 COCH3-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_               20210910
Time                  12.58 h
INSTRUM               spect
PROBHD    Z119470_0283 (
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      43
DS                       4
SWH             31250.000 Hz
FIDRES              0.953674 Hz
AQ                1.0485760 sec
RG                   206.72
DW                   16.000 usec
DE                    6.50 usec
TE                   295.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           125.7703643 MHz
NUC1                    13C
P1                    9.75 usec
PLW1           94.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           25.00000000 W
PLW12           0.46495000 W
PLW13           0.23387000 W

F2 - Processing parameters
SI                   32768
SF              125.7577741 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

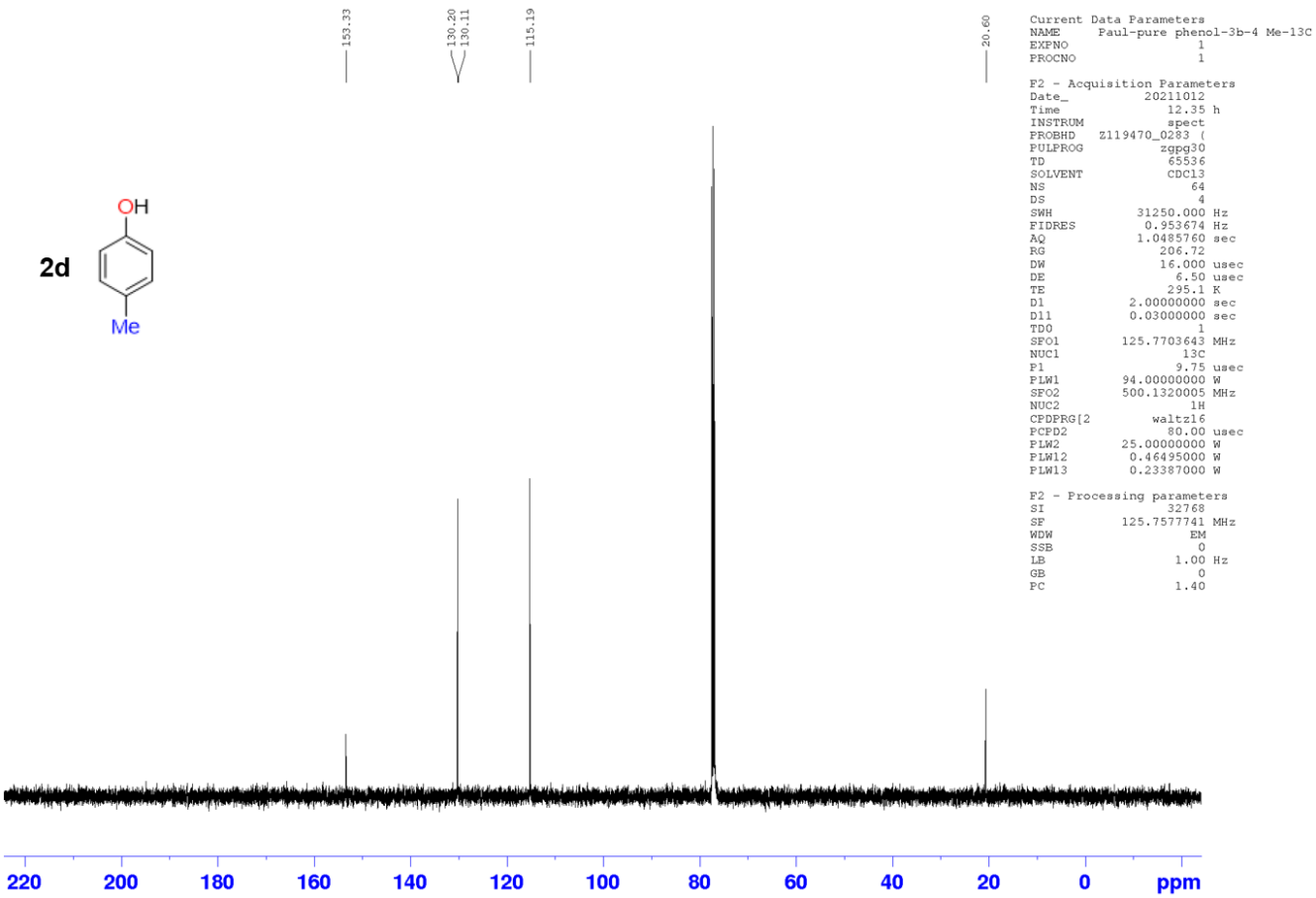


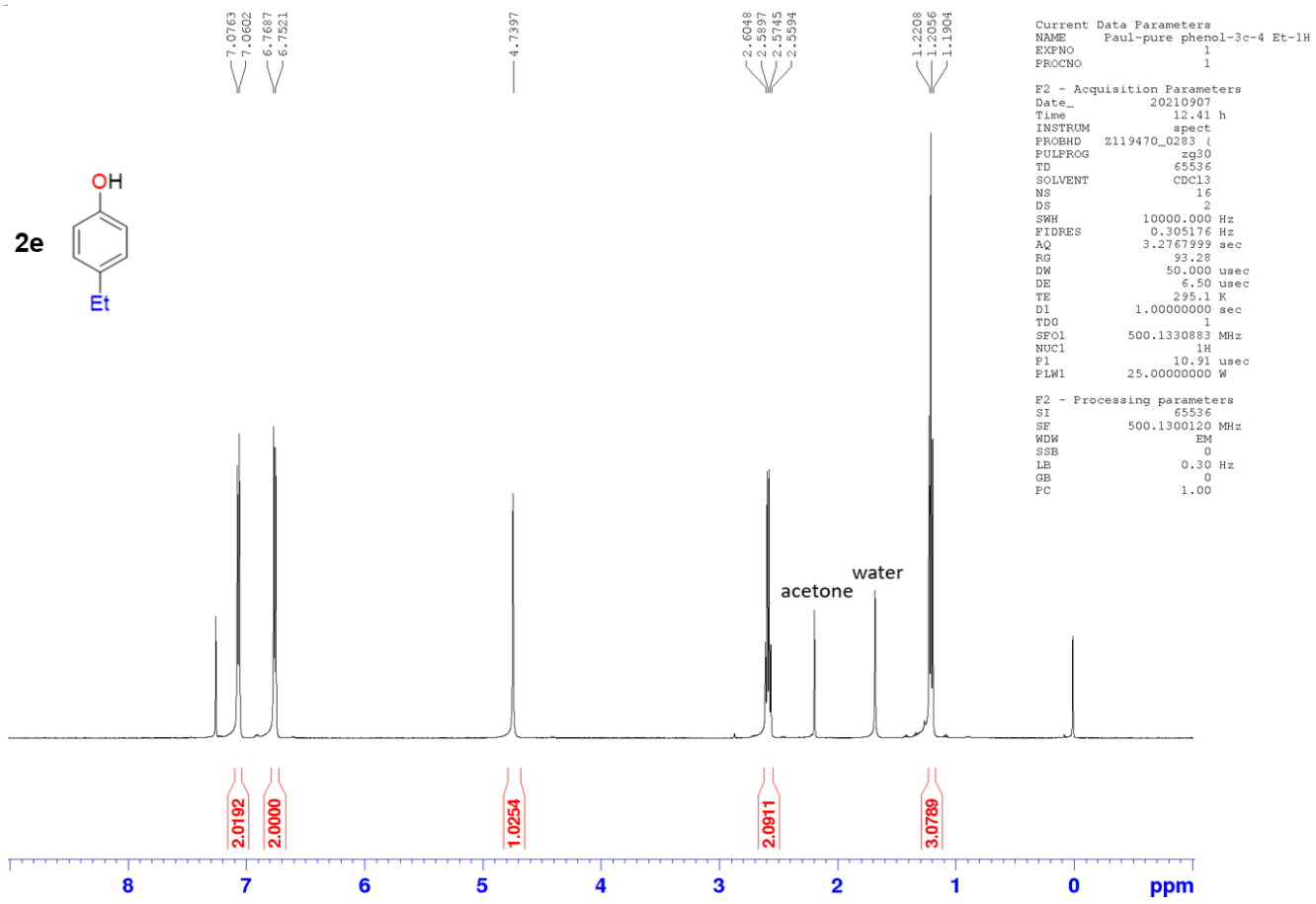

```
Current Data Parameters
NAME      Paul-pure phenol-3a-basic-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20211022
Time              12.34 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ              3.2767999 sec
RG                126.88
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                   1
SFO1          500.1330883 MHz
NUC1                 1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                65536
SF            500.1300119 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

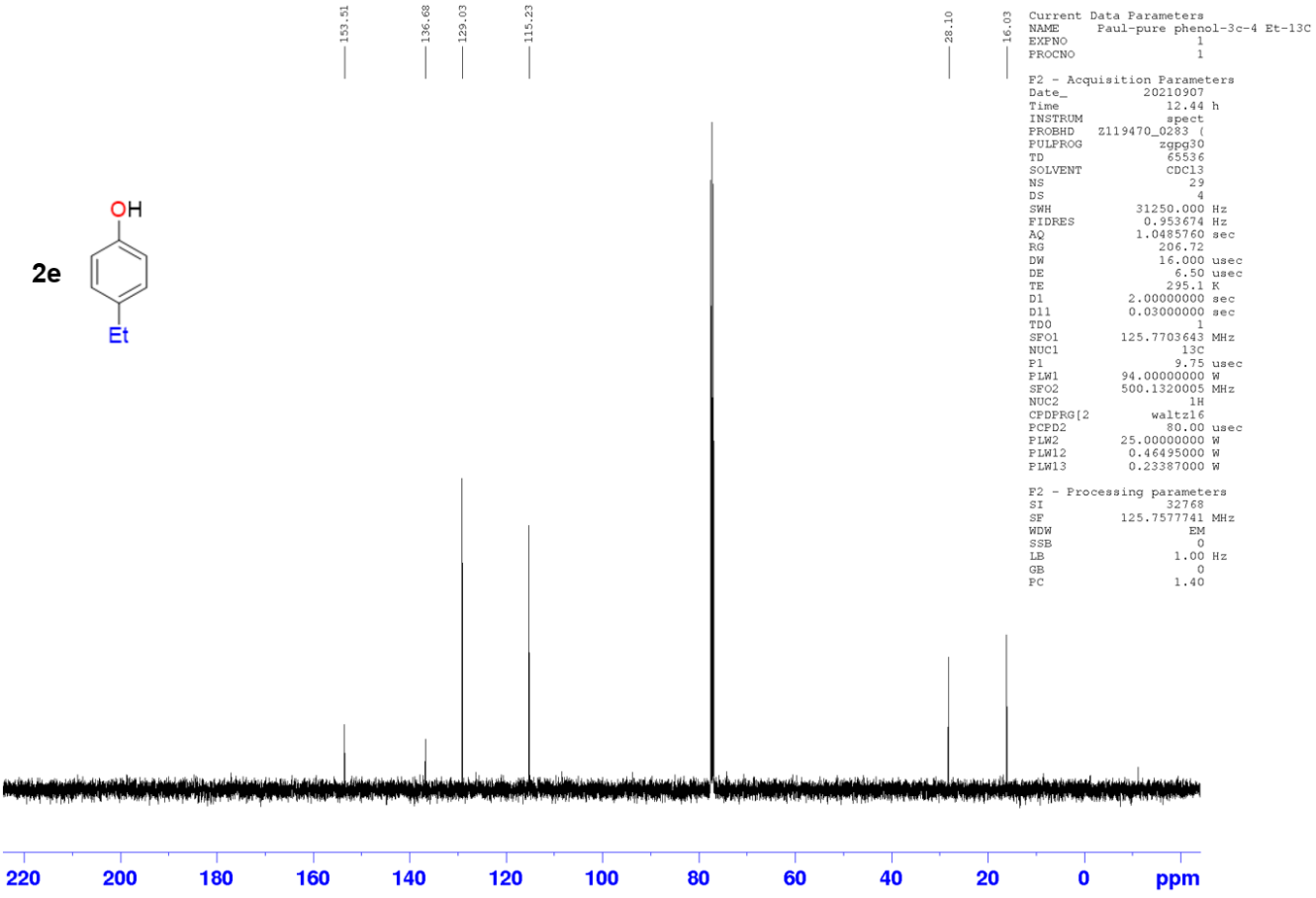


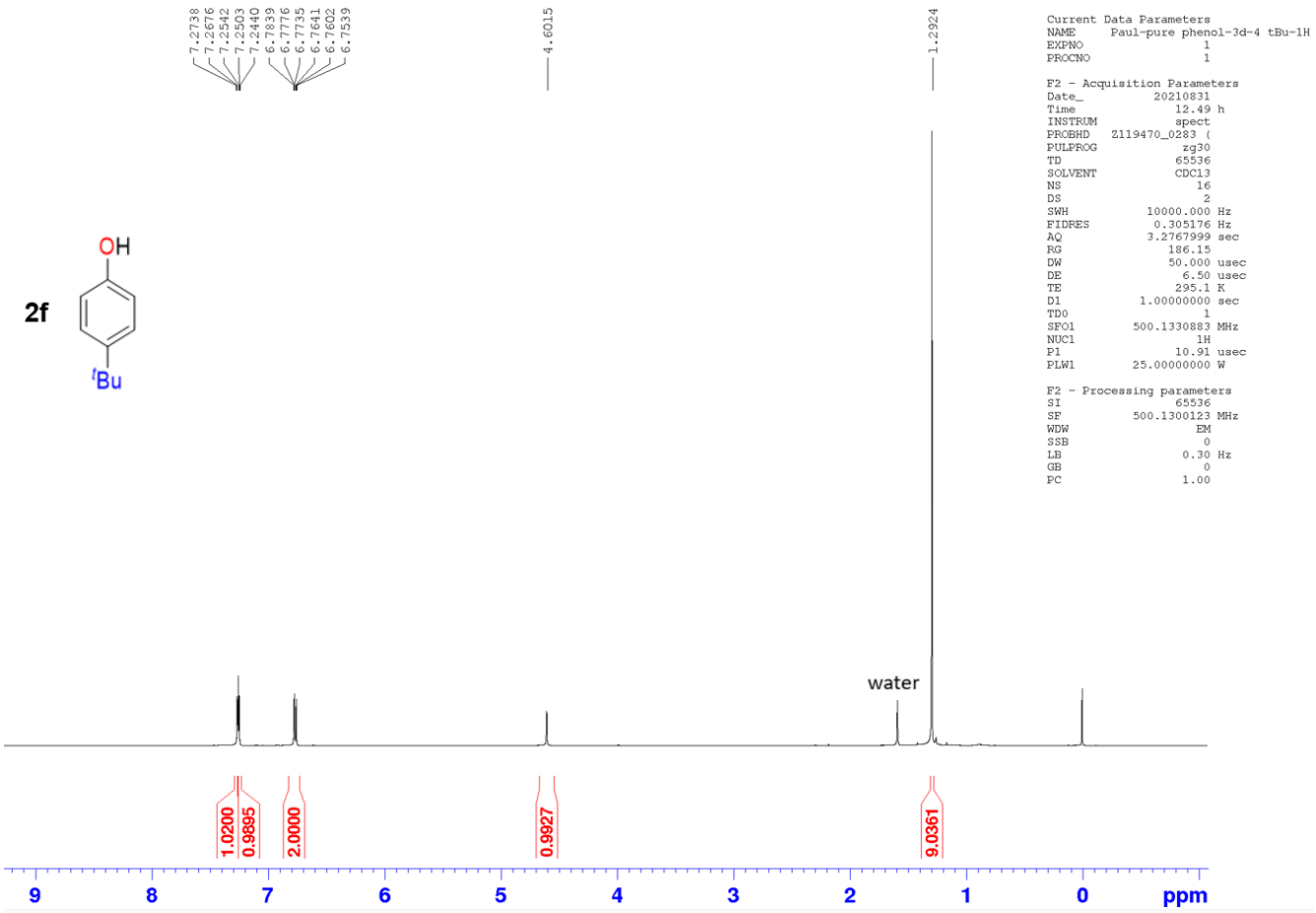

```
Current Data Parameters
NAME      Paul-pure phenol-3a-basic-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20211022
Time              12.39 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   45
DS                    4
SWH           31250.000 Hz
FIDRES            0.953674 Hz
AQ              1.0485760 sec
RG                206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1          125.7703643 MHz
NUC1                 13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                32768
SF            125.7577750 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


7.0488
7.0326
6.7429
6.7263
4.6636
2.2785
OH
2d
Me
acetone
2.0000
2.0859
1.0831
3.0271
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME Paul-pure phenol-3b-4 Me-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211012
Time 12.30 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 93.28
DW 50.000 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 10.91 usec
PLW1 25.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

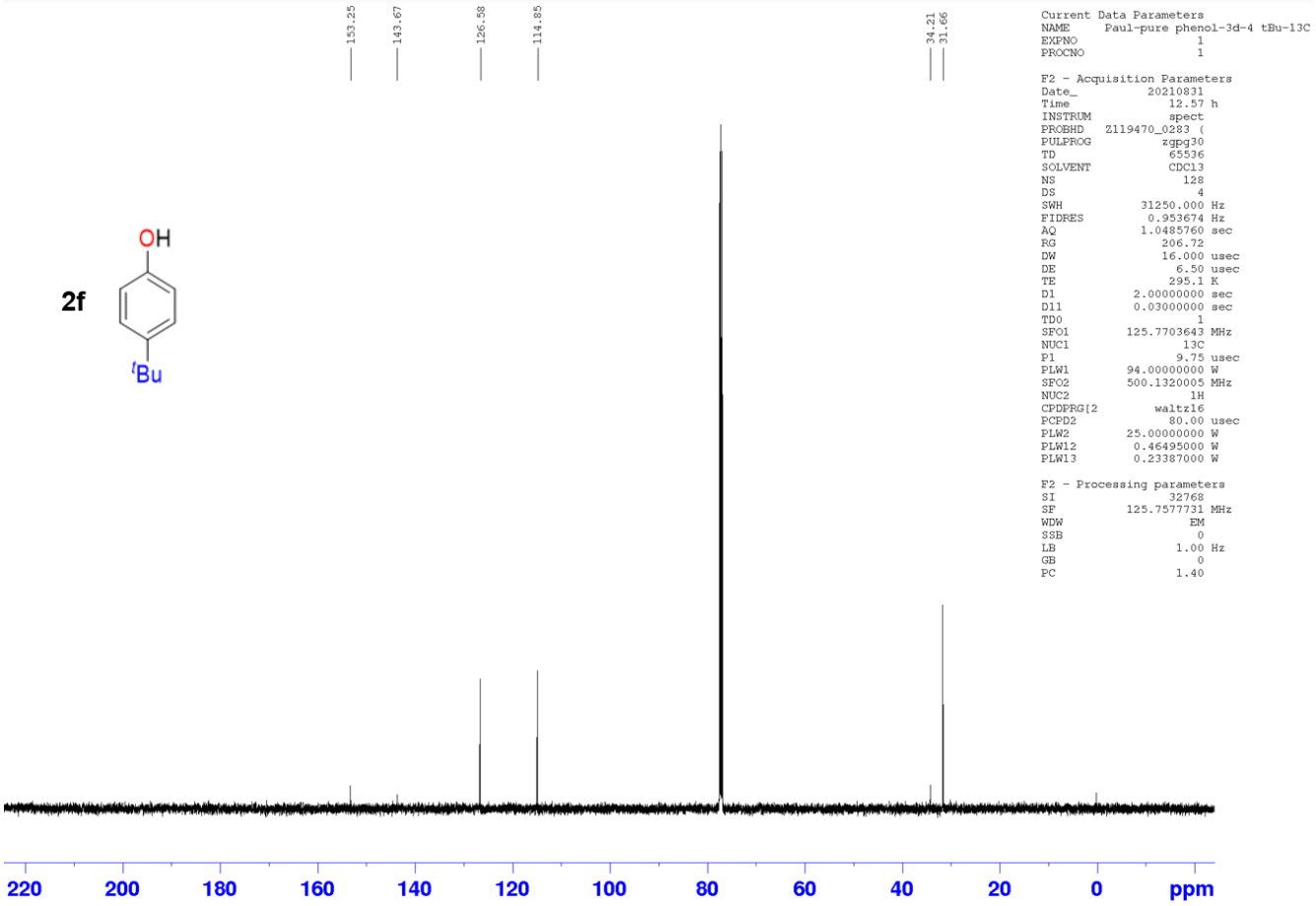

153.33
130.20
130.11
115.19
20.60
OH
2d
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME Paul-pure phenol-3b-4 Me-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211012
Time 12.35 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 206.72
DW 16.000 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.75 usec
PLW1 94.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 25.00000000 W
PLW12 0.46495000 W
PLW13 0.23387000 W
F2 - Processing parameters
SI 32768
SF 125.7577741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.0763
7.0602
6.7687
6.7521
4.7397
2.6048
2.5897
2.5745
2.5594
1.2208
1.2056
1.1904
OH
2e
Et
acetone
water
2.0192
2.0000
1.0254
2.0911
3.0789
8
7
6
5
4
3
2
1
0
ppm
Current Data Parameters
NAME Paul-pure phenol-3c-4 Et-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210907
Time 12.41 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 93.28
DW 50.000 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 10.91 usec
PLW1 25.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

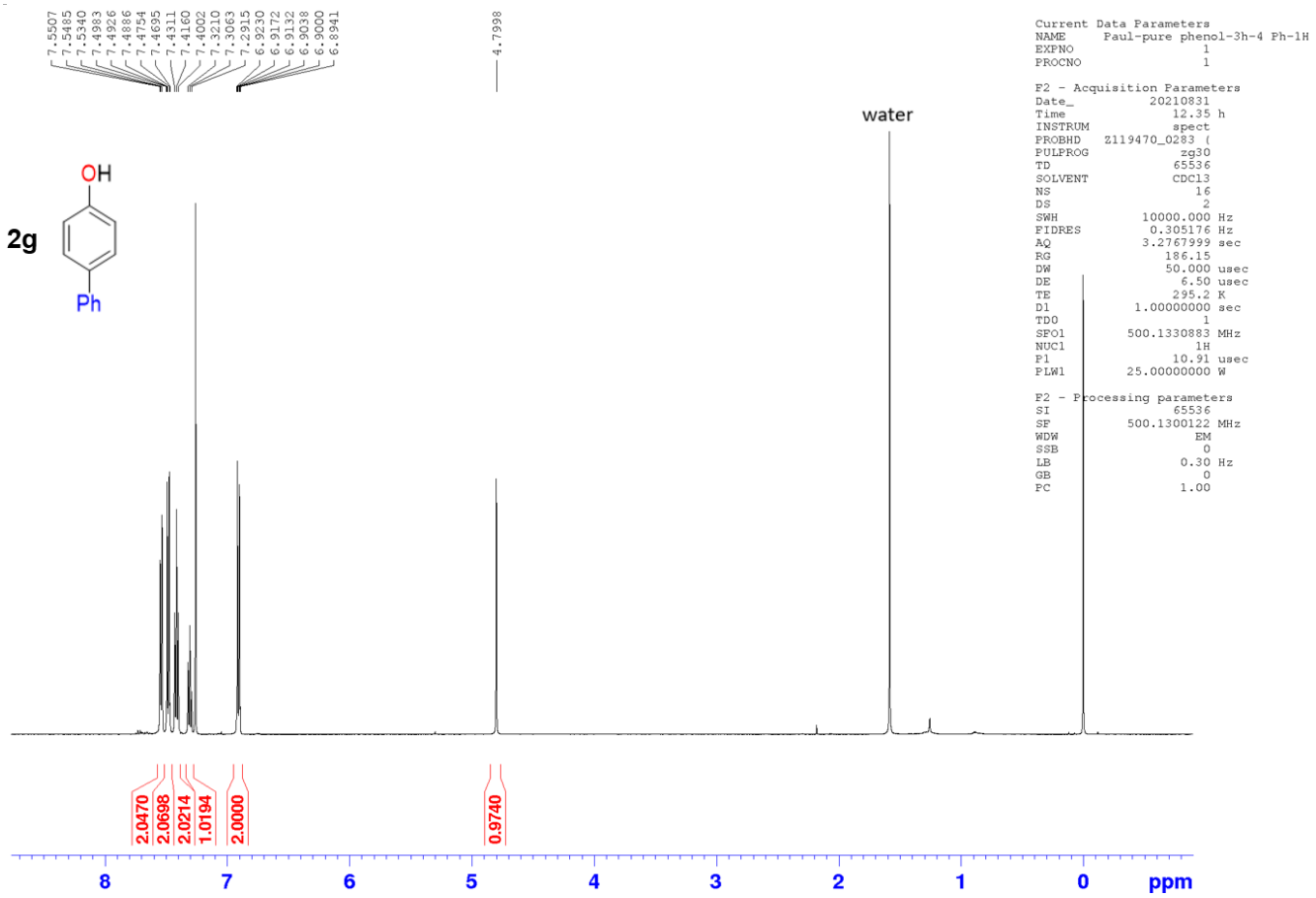

153.51
136.68
129.03
115.23
28.10
16.03
OH
2e
Et
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME Paul-pure phenol-3c-4 Et-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210907
Time 12.44 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 29
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 206.72
DW 16.000 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.75 usec
PLW1 94.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 25.00000000 W
PLW12 0.46495000 W
PLW13 0.23387000 W
F2 - Processing parameters
SI 32768
SF 125.7577741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



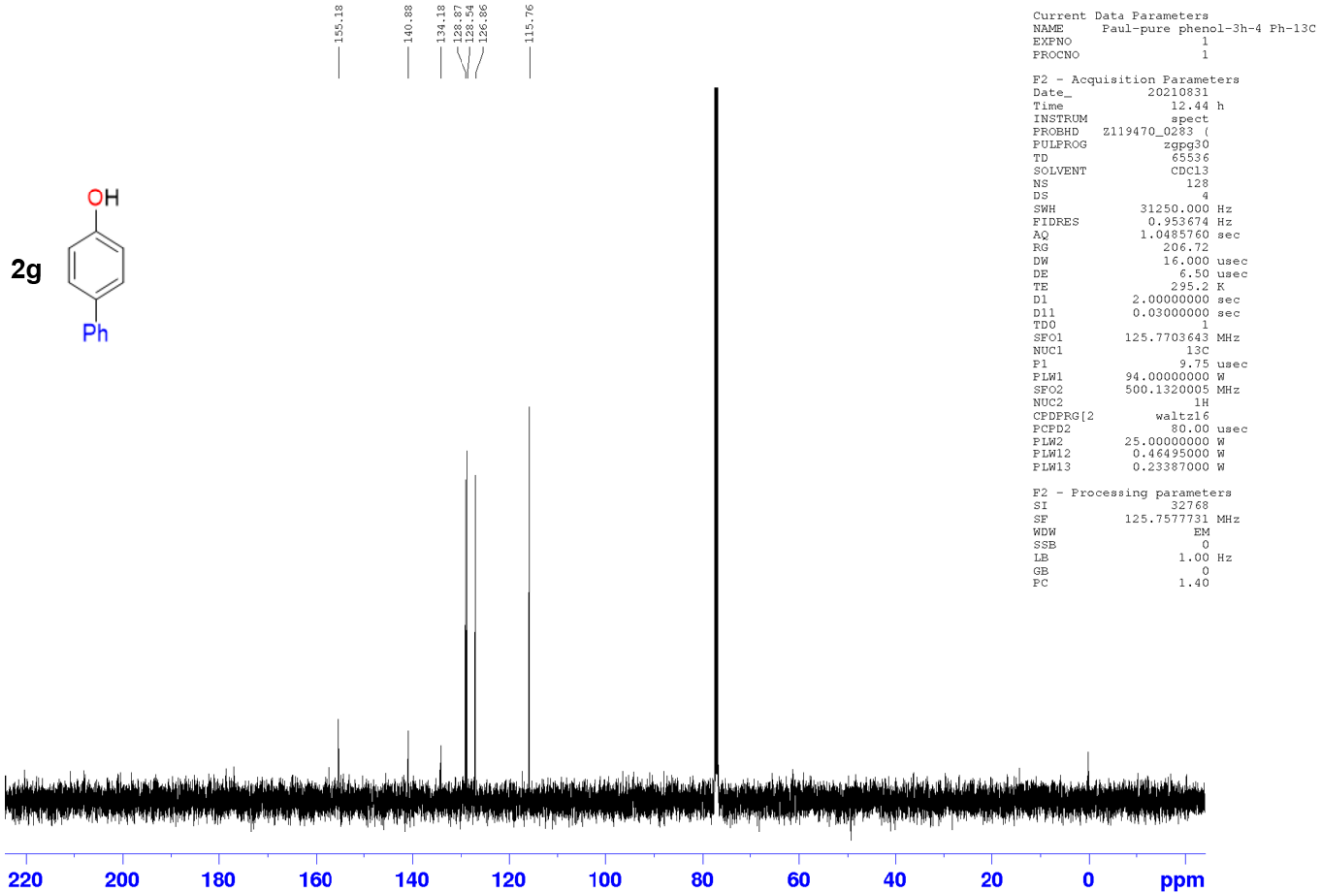

```
Current Data Parameters
NAME      Paul-pure phenol-3d-4 tBu-1H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20210831
Time              12.49 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                186.15
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                  1
SFO1          500.1330883 MHz
NUC1                 1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                65536
SF           500.1300123 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



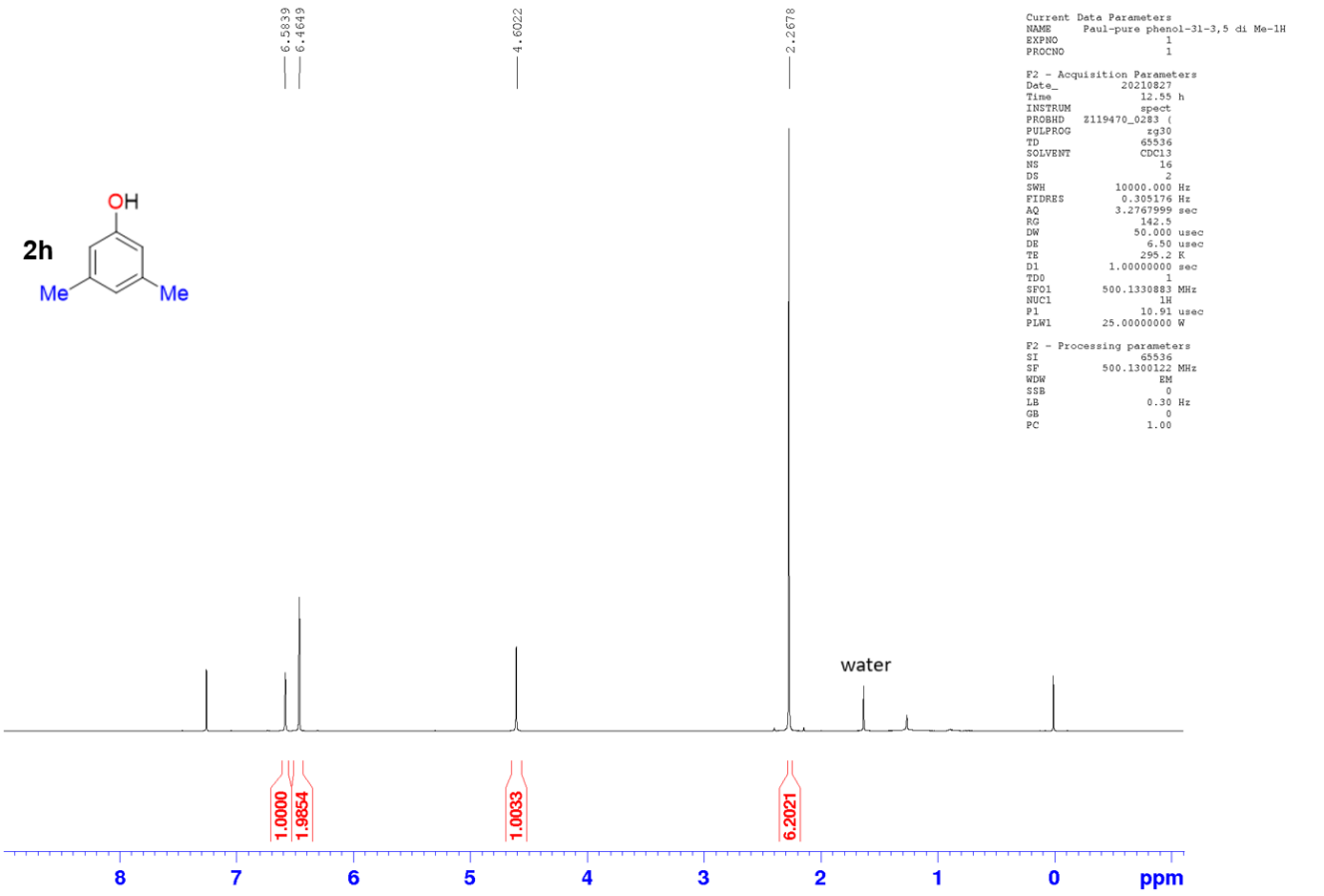

```
Current Data Parameters
NAME      Paul-pure phenol-3d-4 tBu-13C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20210831
Time              12.57 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 128
DS                   4
SWH           31250.000 Hz
FIDRES            0.953674 Hz
AQ             1.0485760 sec
RG                206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                  1
SFO1          125.7703643 MHz
NUC1                 13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                32768
SF           125.7577731 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



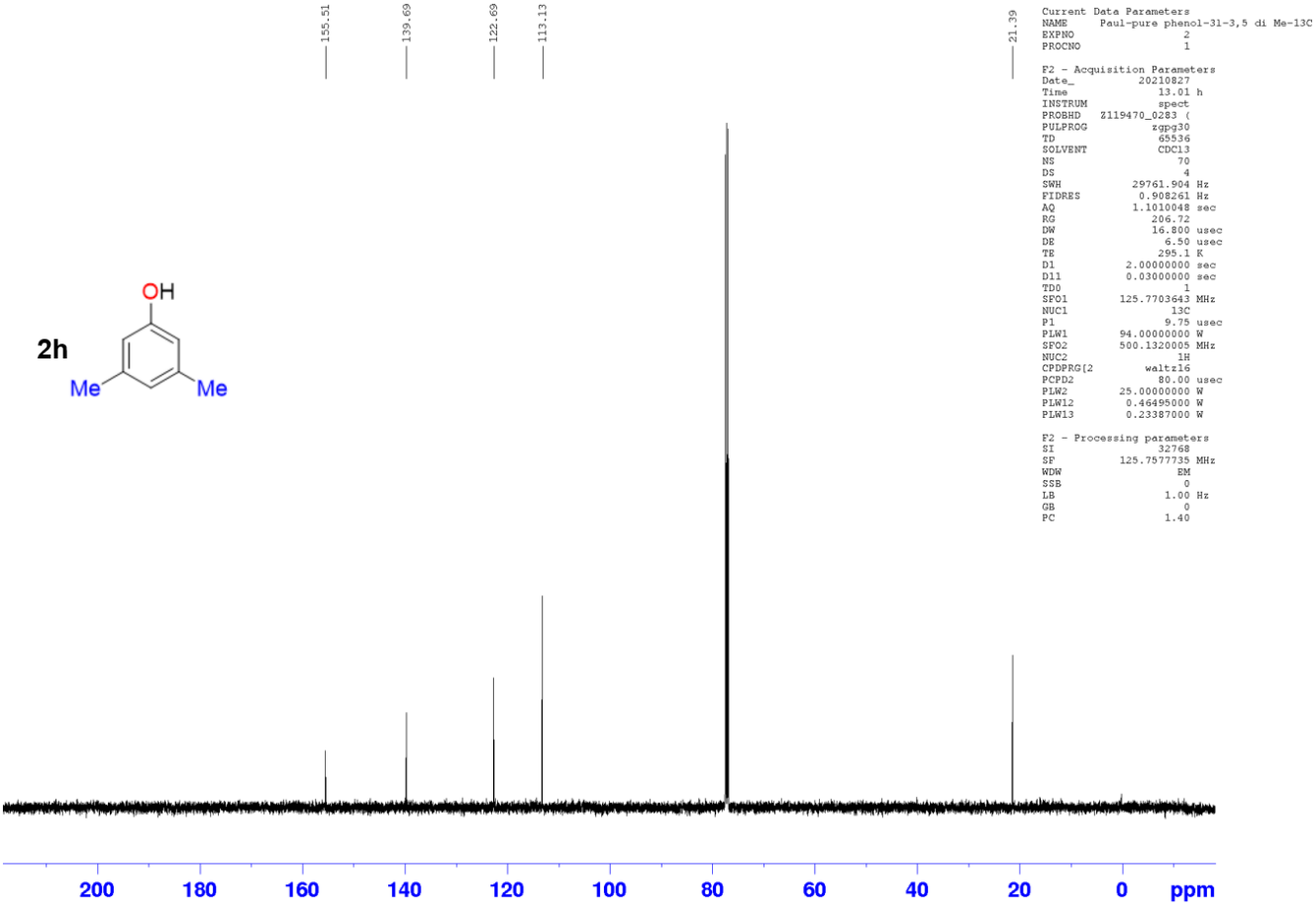

```
Current Data Parameters
NAME      Paul-pure phenol-3h-4 Ph-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210831
Time              12.35 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                186.15
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1            1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



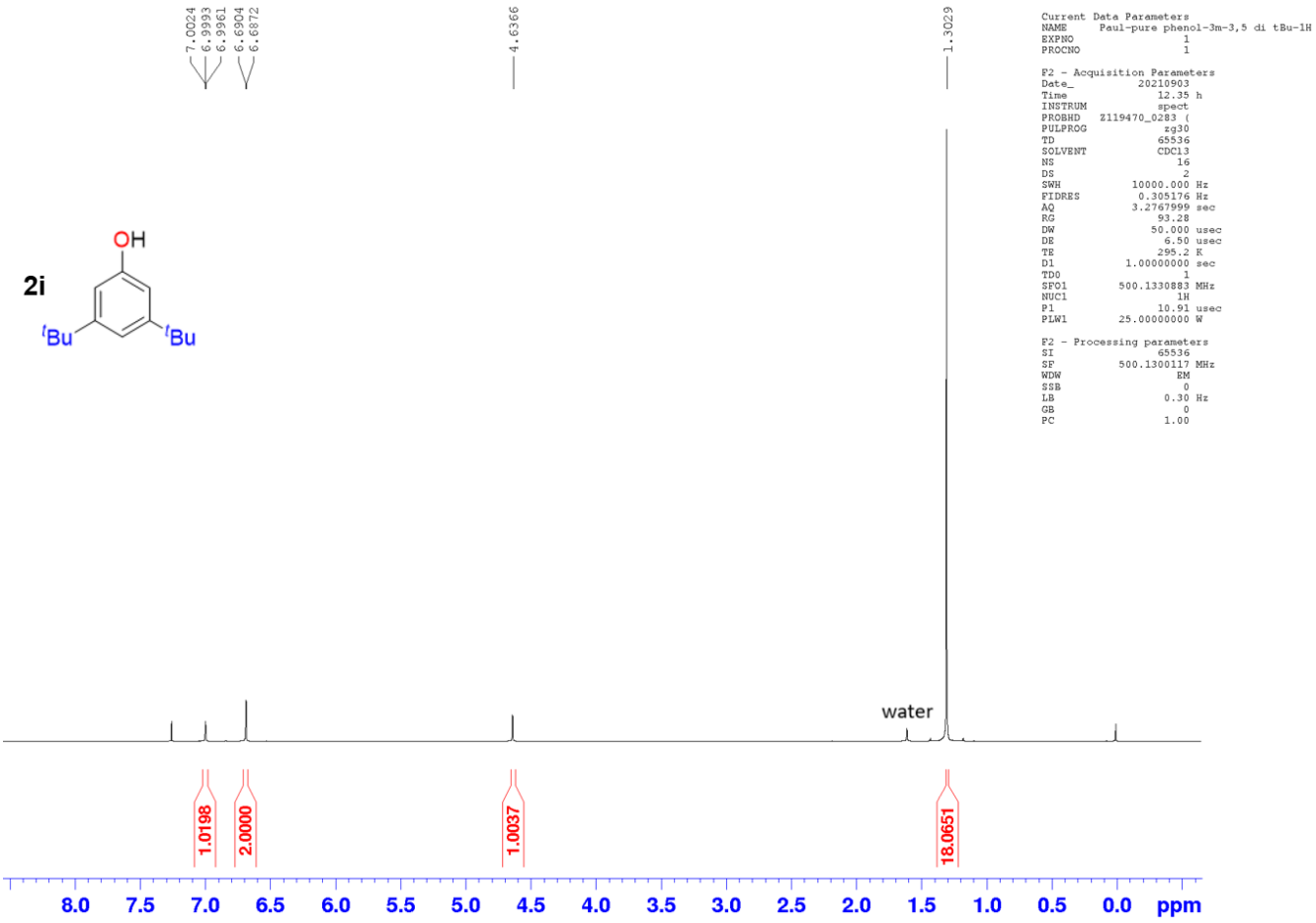

```
Current Data Parameters
NAME      Paul-pure phenol-3h-4 Ph-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210831
Time              12.44 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     4
SWH           31250.000 Hz
FIDRES            0.953674 Hz
AQ             1.0485760 sec
RG                206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577731 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



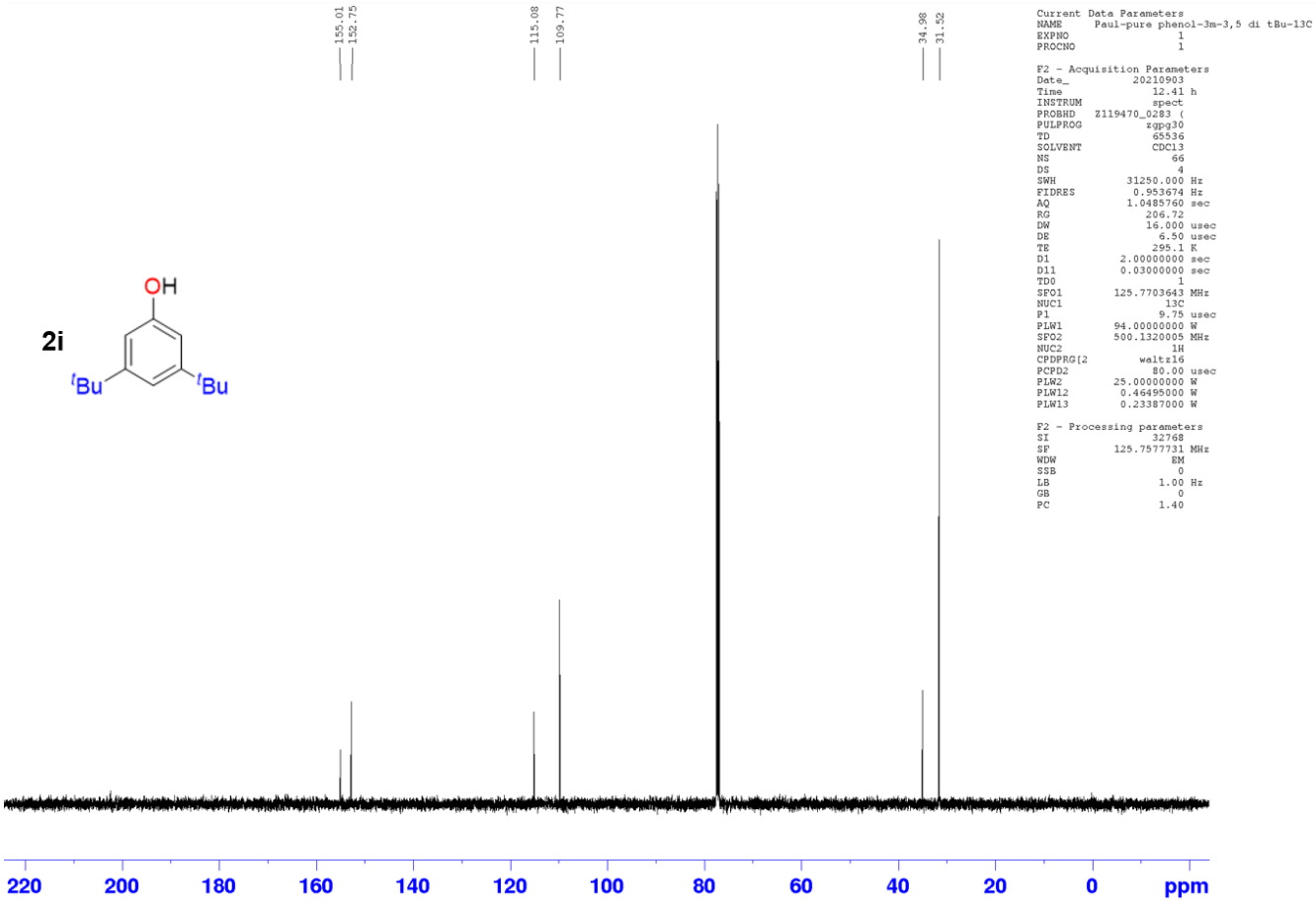

```
Current Data Parameters
NAME       Paul-pure phenol-31-3,5 di Me-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210827
Time              12.55 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ                3.2767999 sec
RG                   142.5
DW                  50.000 usec
DE                   6.50 usec
TE                 295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



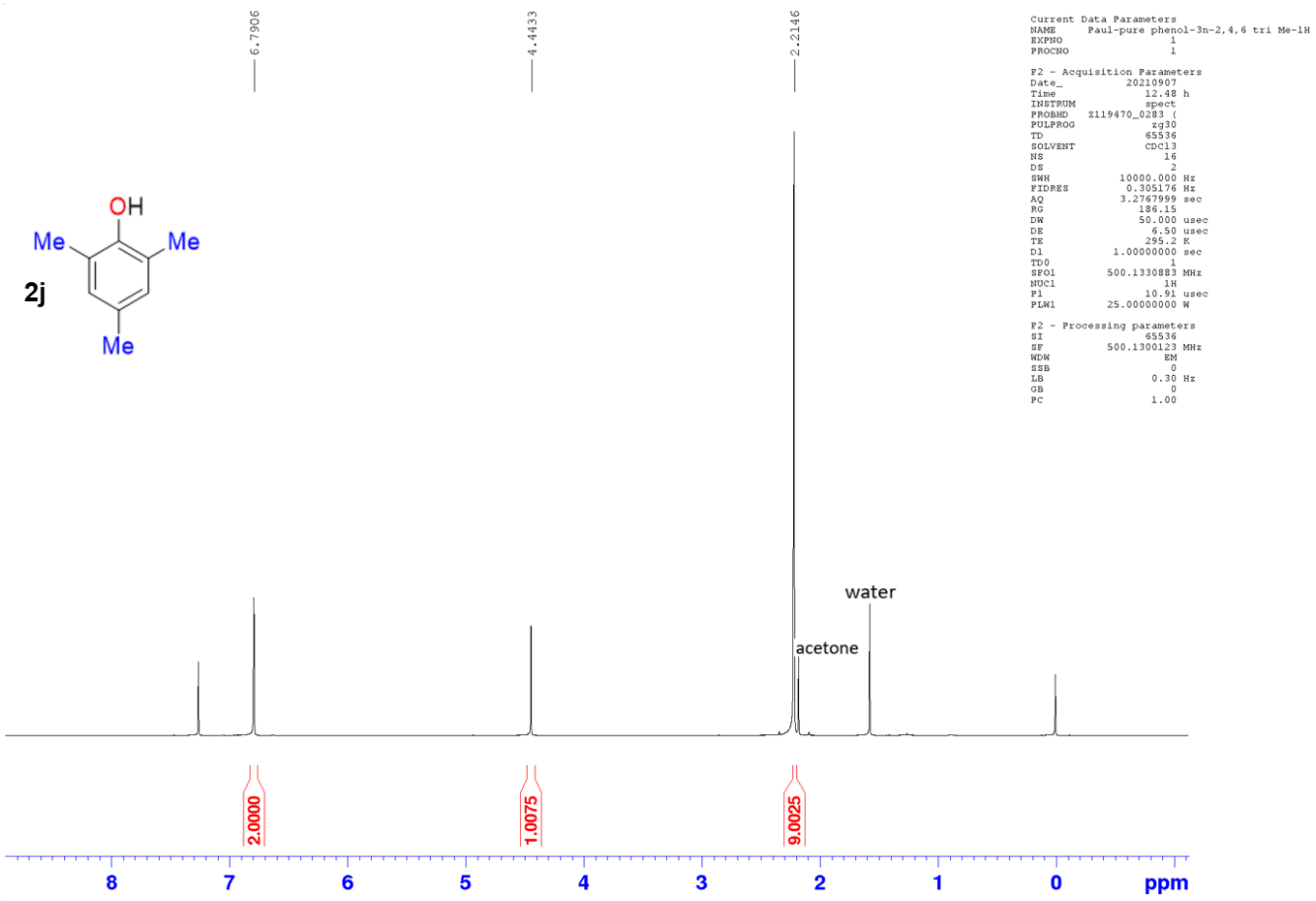

```
Current Data Parameters
NAME       Paul-pure phenol-31-3,5 di Me-13C
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20210827
Time              13.01 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    70
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ                1.1010048 sec
RG                  206.72
DW                  16.800 usec
DE                   6.50 usec
TE                 295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                  9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2               80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577735 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
Current Data Parameters
NAME      Paul-pure phenol-3m-3,5 di tBu-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20210903
Time              12.35 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                93.28
DW               50.000 usec
DE                6.50 usec
TE               295.2 K
D1            1.00000000 sec
TD0                   1
SFO1        500.1330883 MHz
NUC1                 1H
P1               10.91 usec
PLW1         25.00000000 W

F2 - Processing parameters
SI                65536
SF          500.1300117 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



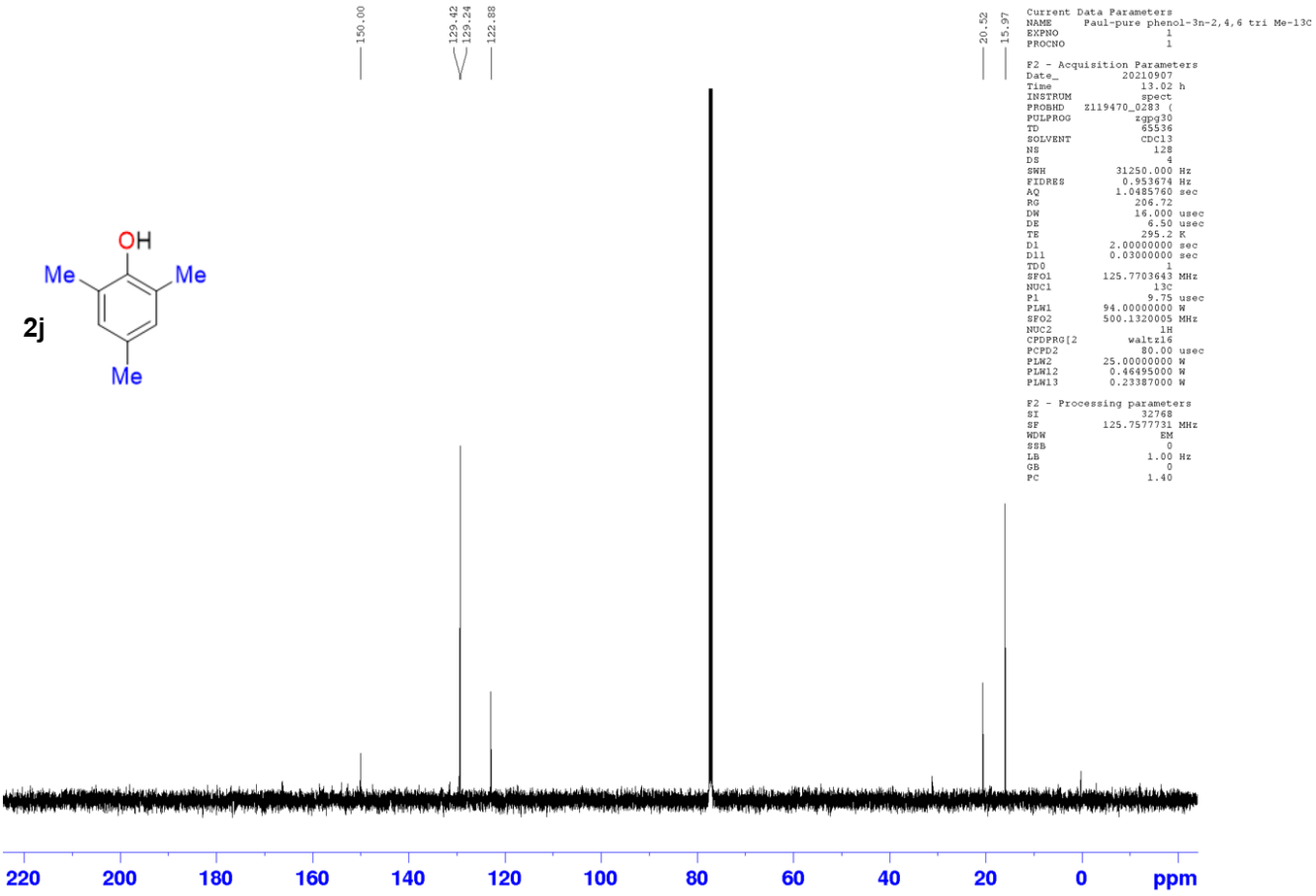

```
Current Data Parameters
NAME      Paul-pure phenol-3m-3,5 di tBu-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20210903
Time              12.41 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   66
DS                    4
SWH           31250.000 Hz
FIDRES        0.953674 Hz
AQ            1.0485760 sec
RG               206.72
DW               16.000 usec
DE                6.50 usec
TE               295.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                 13C
P1                9.75 usec
PLW1         94.00000000 W
SFO2        500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                32768
SF          125.7577731 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



Current Data Parameters
NAME Paul-pure phenol-3n-2,4,6 tri Me-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210907
Time 12.48 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 186.15
DW 50.000 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 10.91 usec
PLW1 25.00000000 W

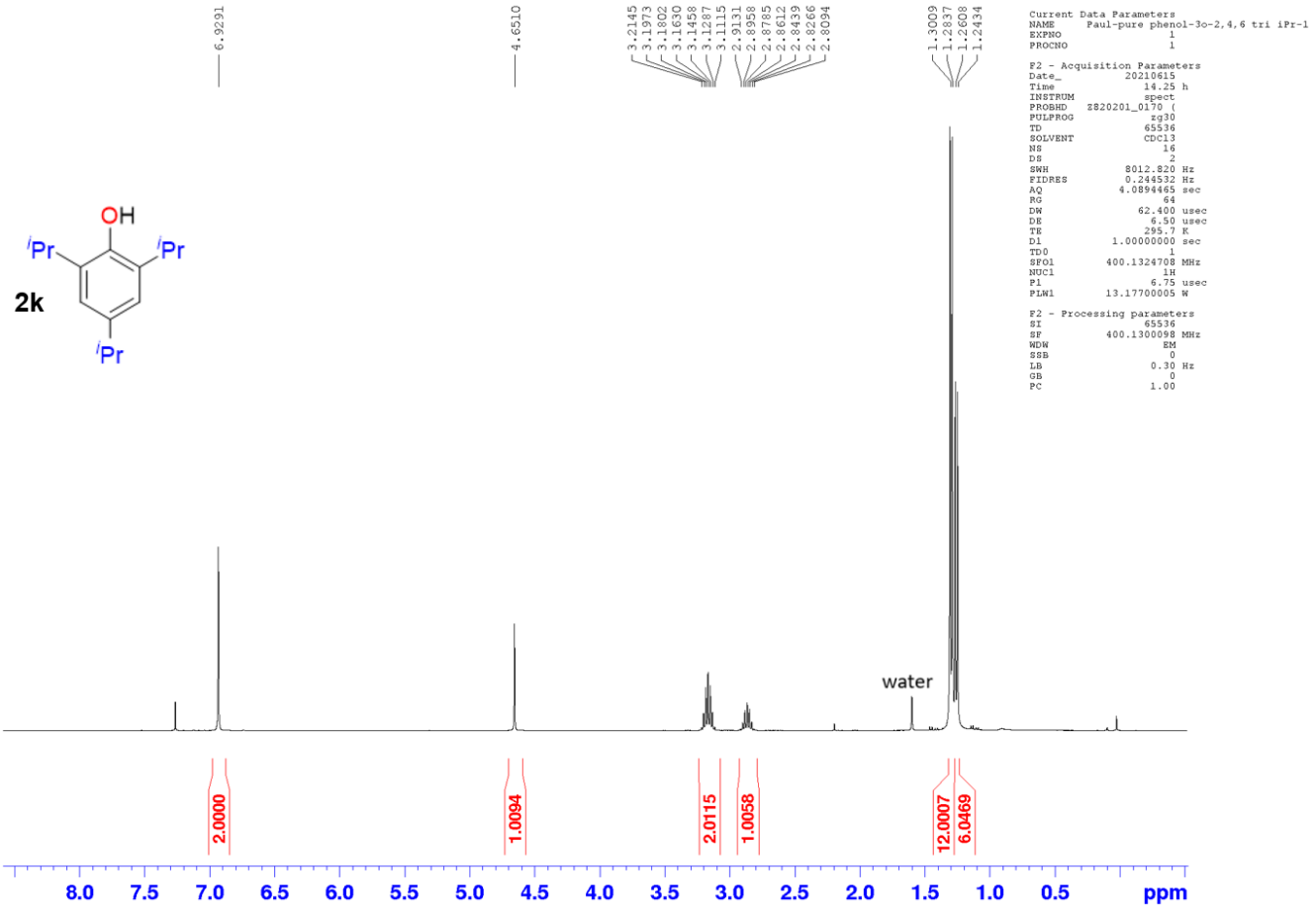
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



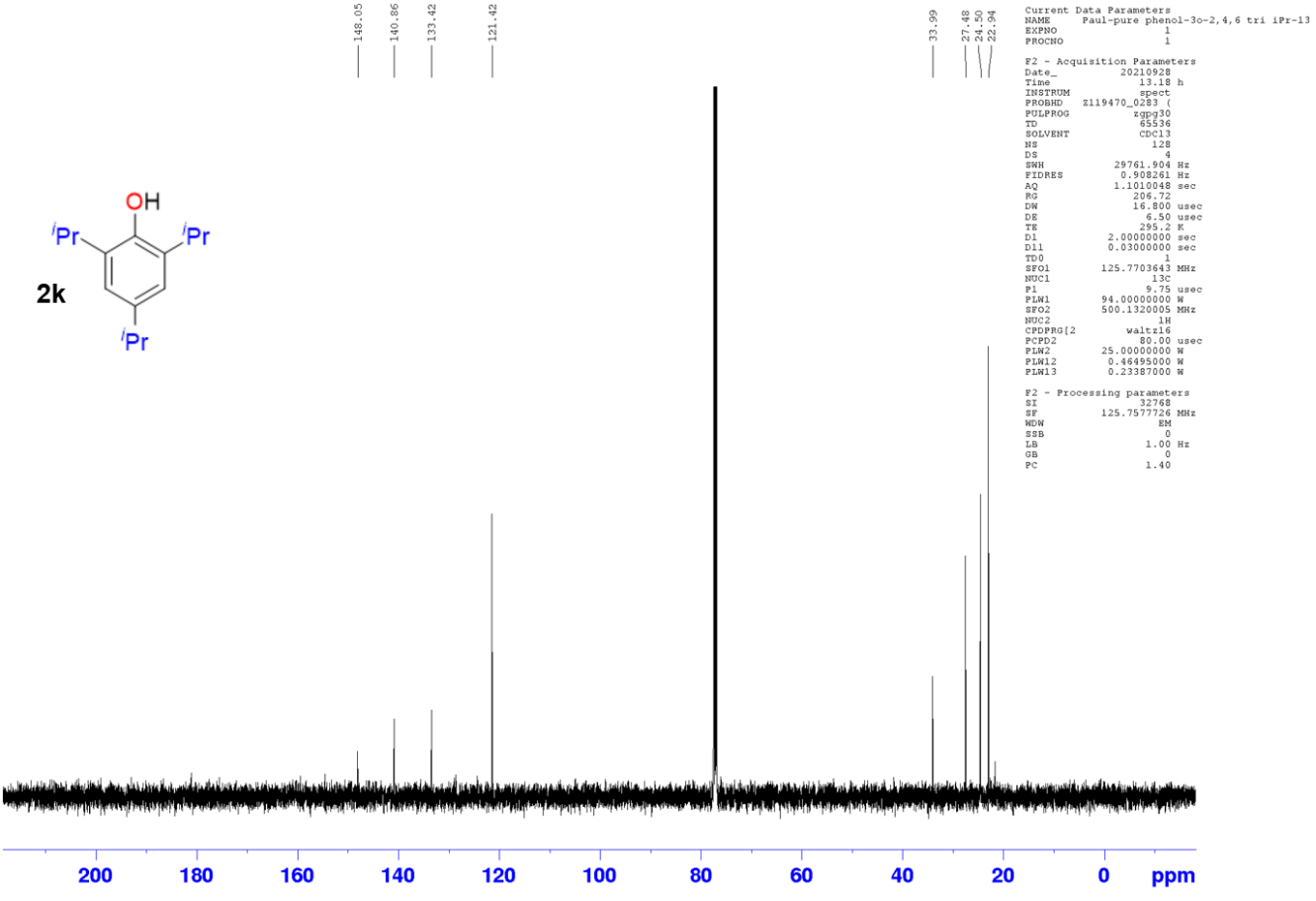
Current Data Parameters
NAME Paul-pure phenol-3n-2,4,6 tri Me-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210907
Time 13.02 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 206.72
DW 16.000 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.75 usec
PLW1 94.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 25.00000000 W
PLW12 0.46495000 W
PLW13 0.23387000 W

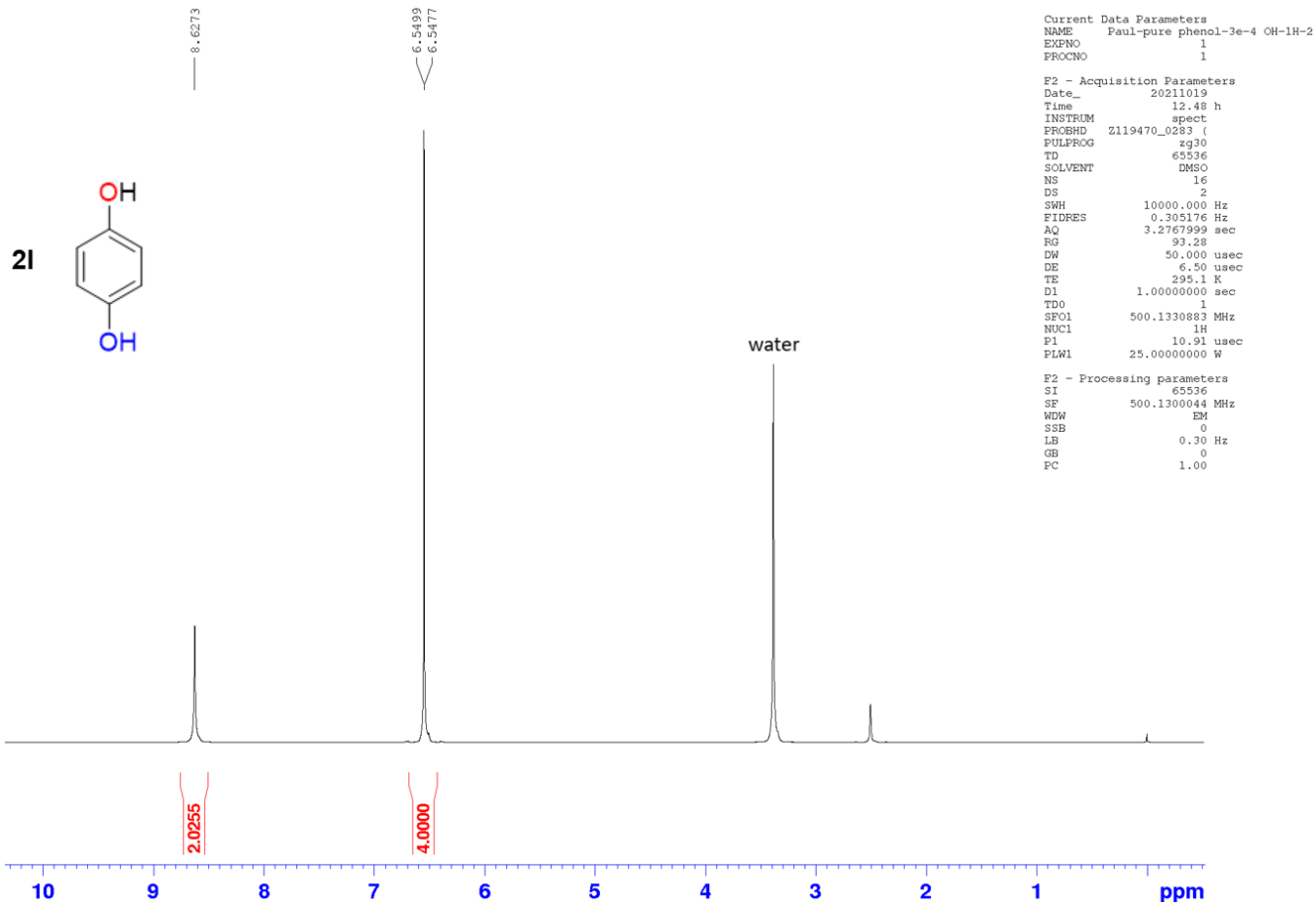
F2 - Processing parameters
SI 32768
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME      Paul-pure phenol-3o-2,4,6 tri iPr-1
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_             20210615
Time                14.25 h
INSTRUM              spect
PROBHD    Z820201_0170 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                     64
DW                  62.400 usec
DE                   6.50 usec
TE                  295.7 K
D1                1.00000000 sec
TD0                     1
SFO1            400.1324708 MHz
NUC1                   1H
P1                   6.75 usec
PLW1            13.17700005 W

F2 - Processing parameters
SI                  65536
SF              400.1300098 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

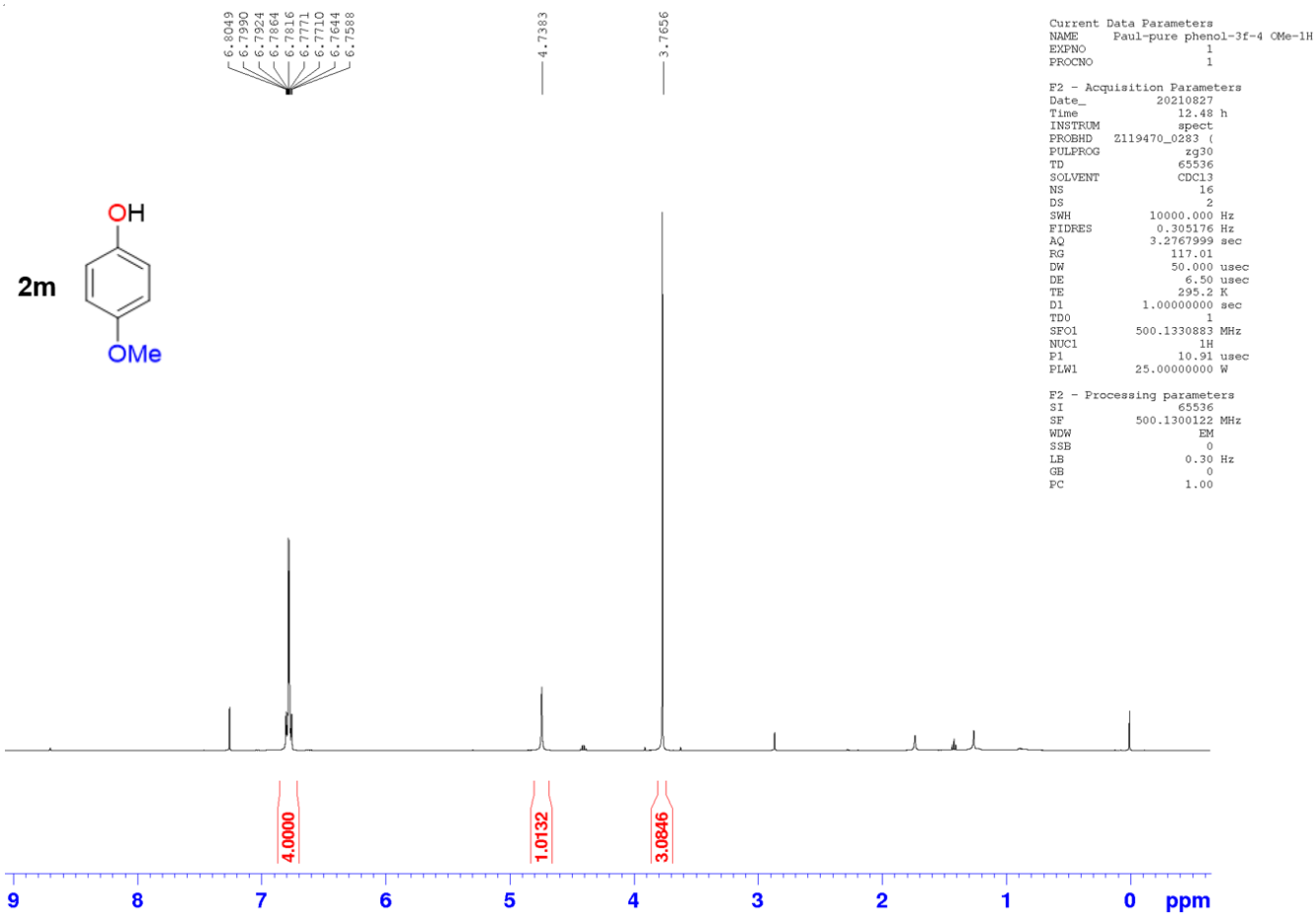



```
Current Data Parameters
NAME      Paul-pure phenol-3o-2,4,6 tri iPr-13
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_             20210928
Time                13.18 h
INSTRUM              spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ                1.1010048 sec
RG                   206.72
DW                  16.800 usec
DE                   6.50 usec
TE                  295.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                     1
SFO1            125.7703643 MHz
NUC1                   13C
P1                   9.75 usec
PLW1            94.00000000 W
SFO2            500.1320005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2            25.00000000 W
PLW12            0.46495000 W
PLW13            0.23387000 W

F2 - Processing parameters
SI                  32768
SF              125.7577726 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

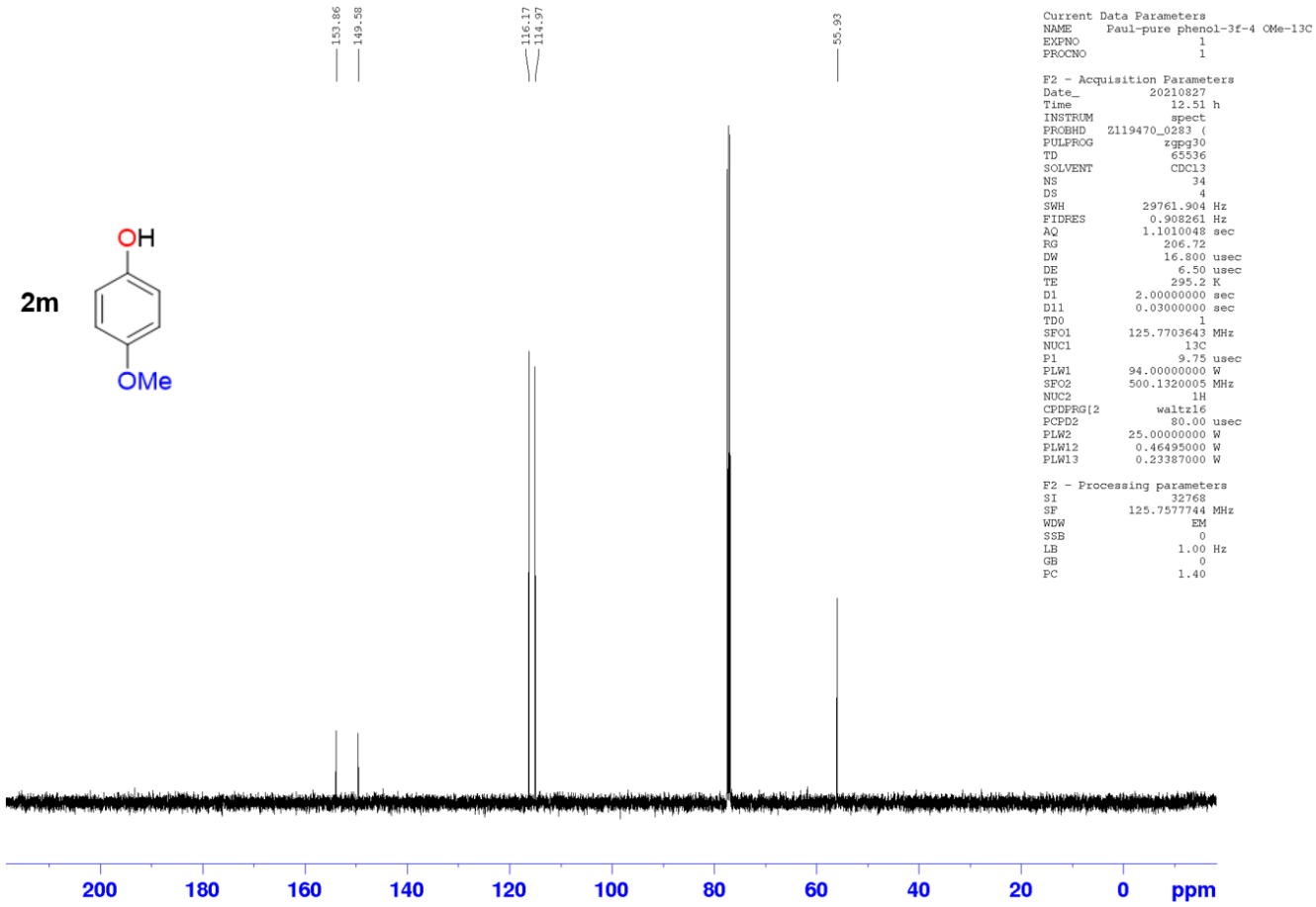



```
Current Data Parameters
NAME      Paul-pure phenol-3e-4 OH-1H-2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20211019
Time              12.48 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                 93.28
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1         25.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300044 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

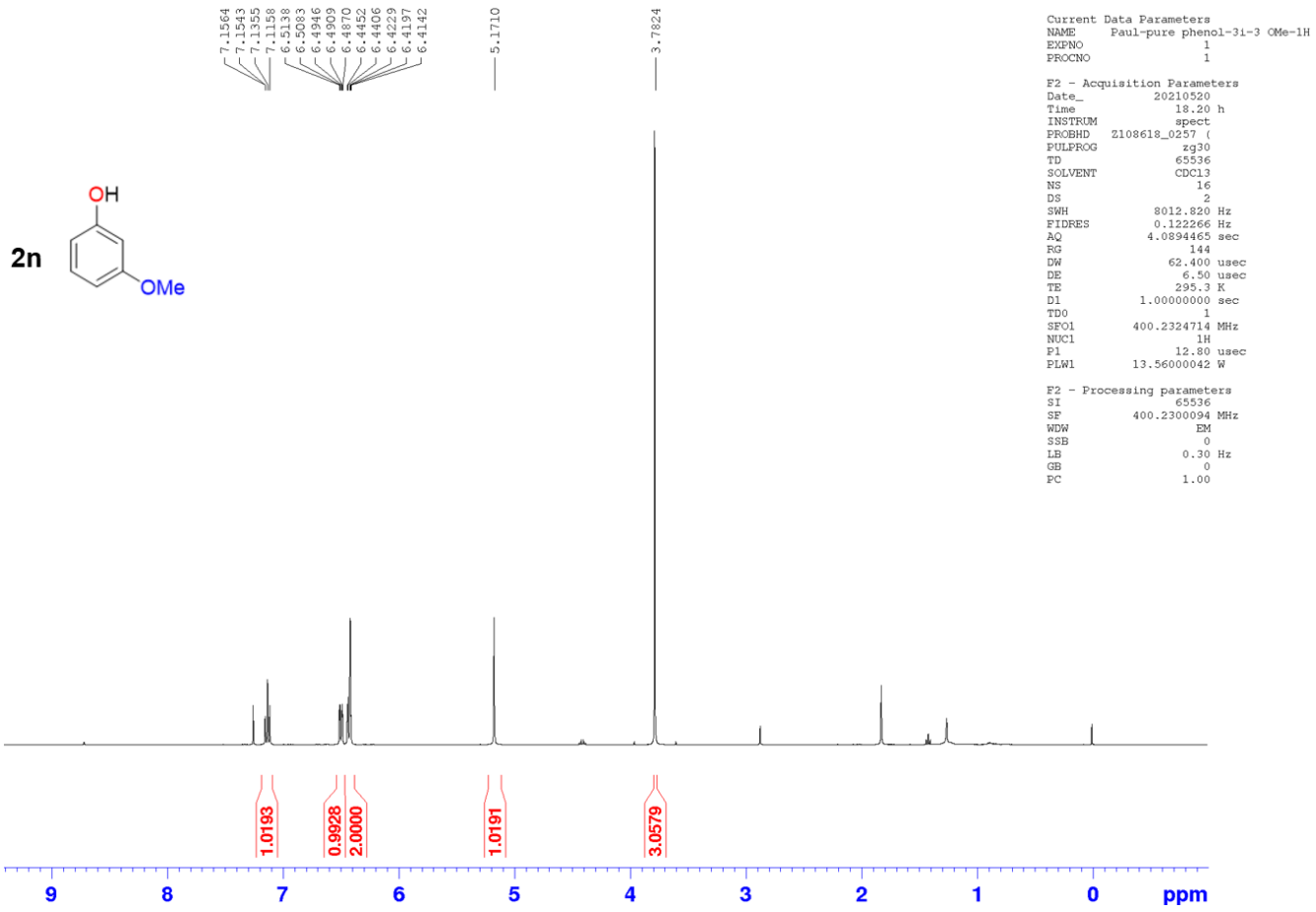

```
Current Data Parameters
NAME      Paul-pure phenol-3e-4 OH-13C-2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20211019
Time              12.52 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                    55
DS                     4
SWH           31250.000 Hz
FIDRES            0.953674 Hz
AQ             1.0485760 sec
RG                206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         125.7703643 MHz
NUC1                 13C
P1                 9.75 usec
PLW1         94.00000000 W
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                 32768
SF           125.7578413 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

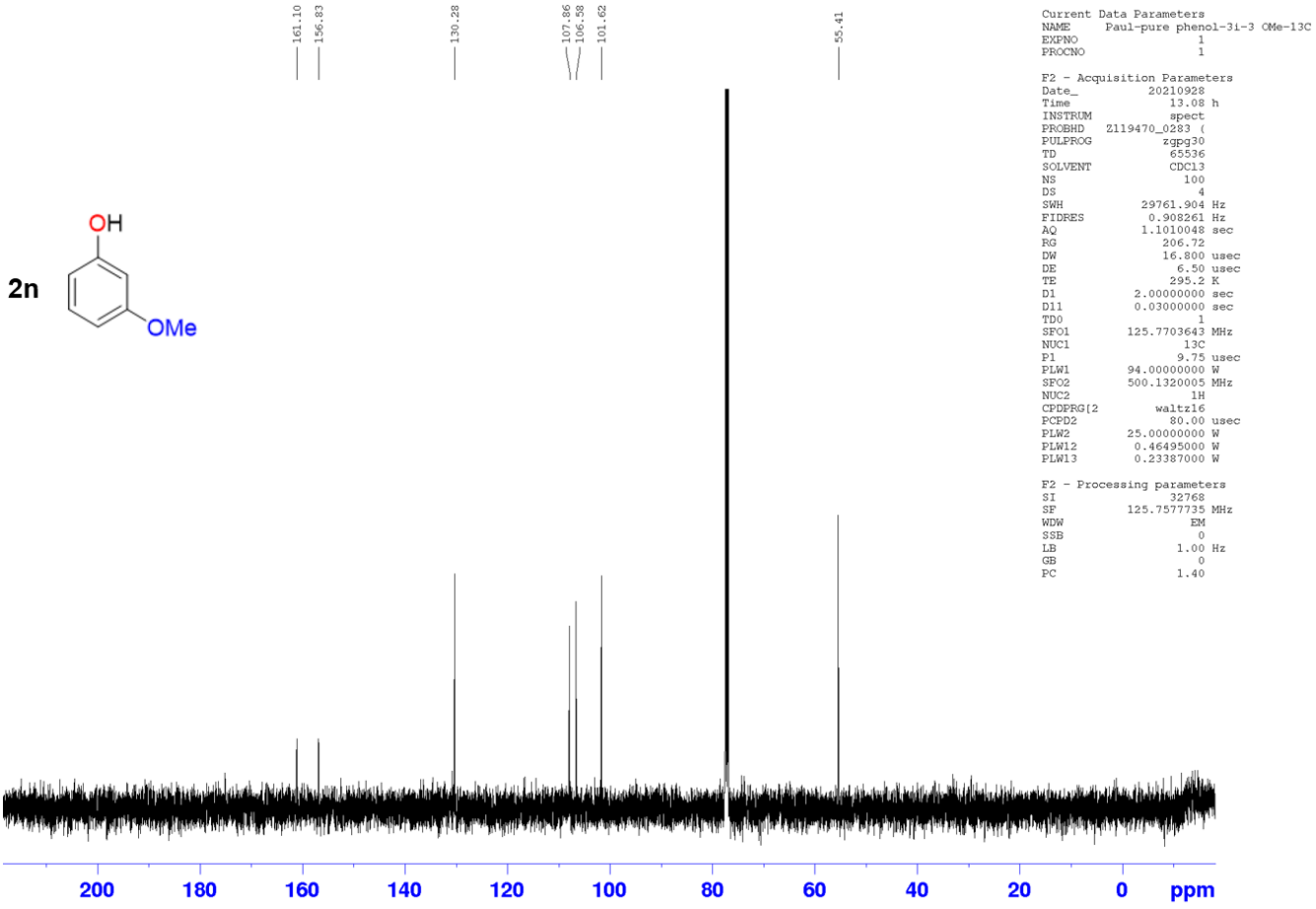


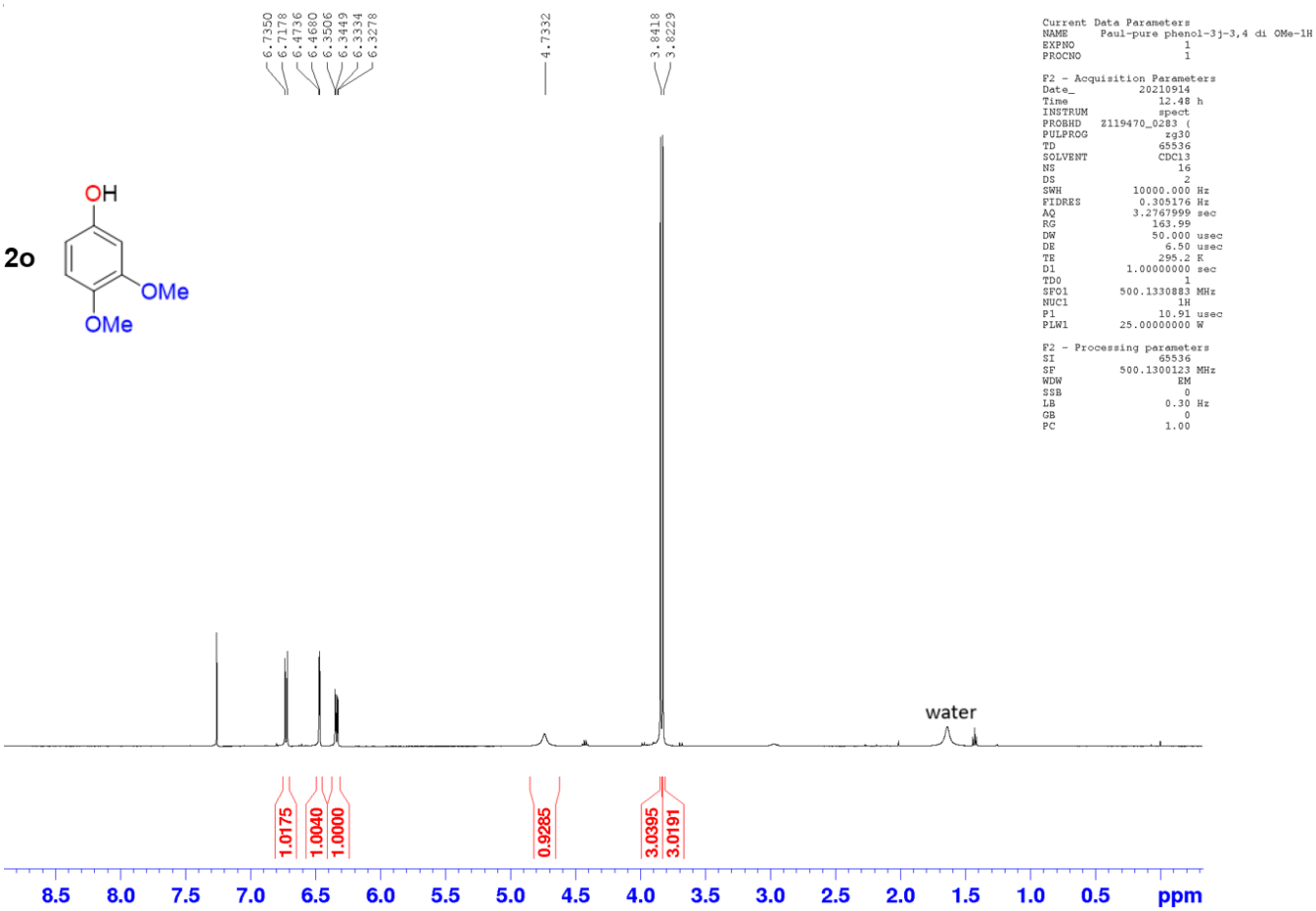

```
Current Data Parameters
NAME      Paul-pure phenol-3f-4 OMe-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210827
Time              12.48 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                117.01
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



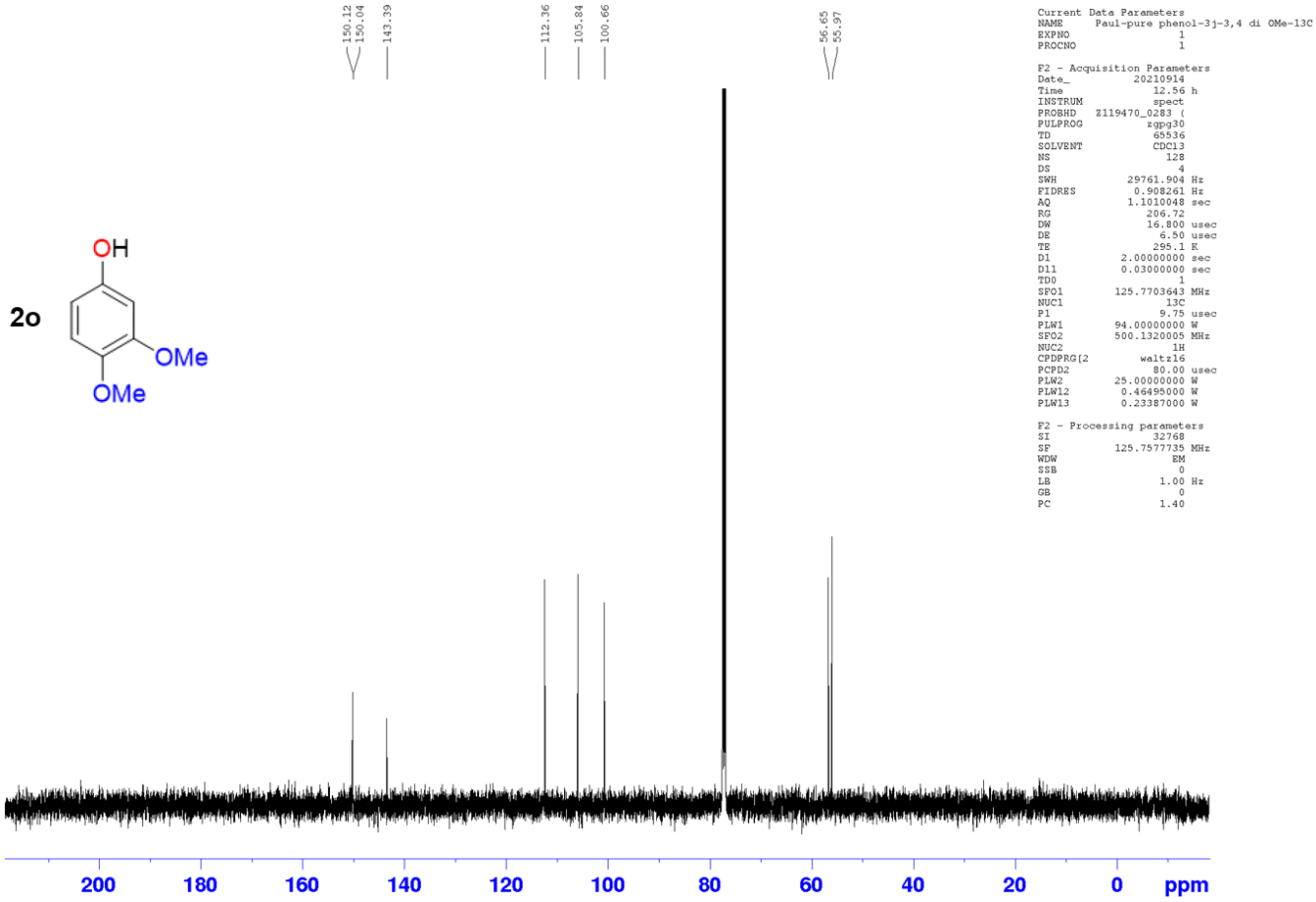

```
Current Data Parameters
NAME      Paul-pure phenol-3f-4 OMe-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210827
Time              12.51 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    34
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         125.7703643 MHz
NUC1                  13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF           125.7577744 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


7.1564
7.1543
7.1355
7.1158
6.5138
6.5083
6.4946
6.4909
6.4870
6.4452
6.4406
6.4229
6.4197
6.4142
5.1710
3.7824
2n
OH
OMe
Current Data Parameters
NAME Paul-pure phenol-3i-3 OMe-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210520
Time 18.20 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 144
DW 62.400 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W
F2 - Processing parameters
SI 65536
SF 400.2300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.0193
0.9928
2.0000
1.0191
3.0579
9
8
7
6
5
4
3
2
1
0
ppm

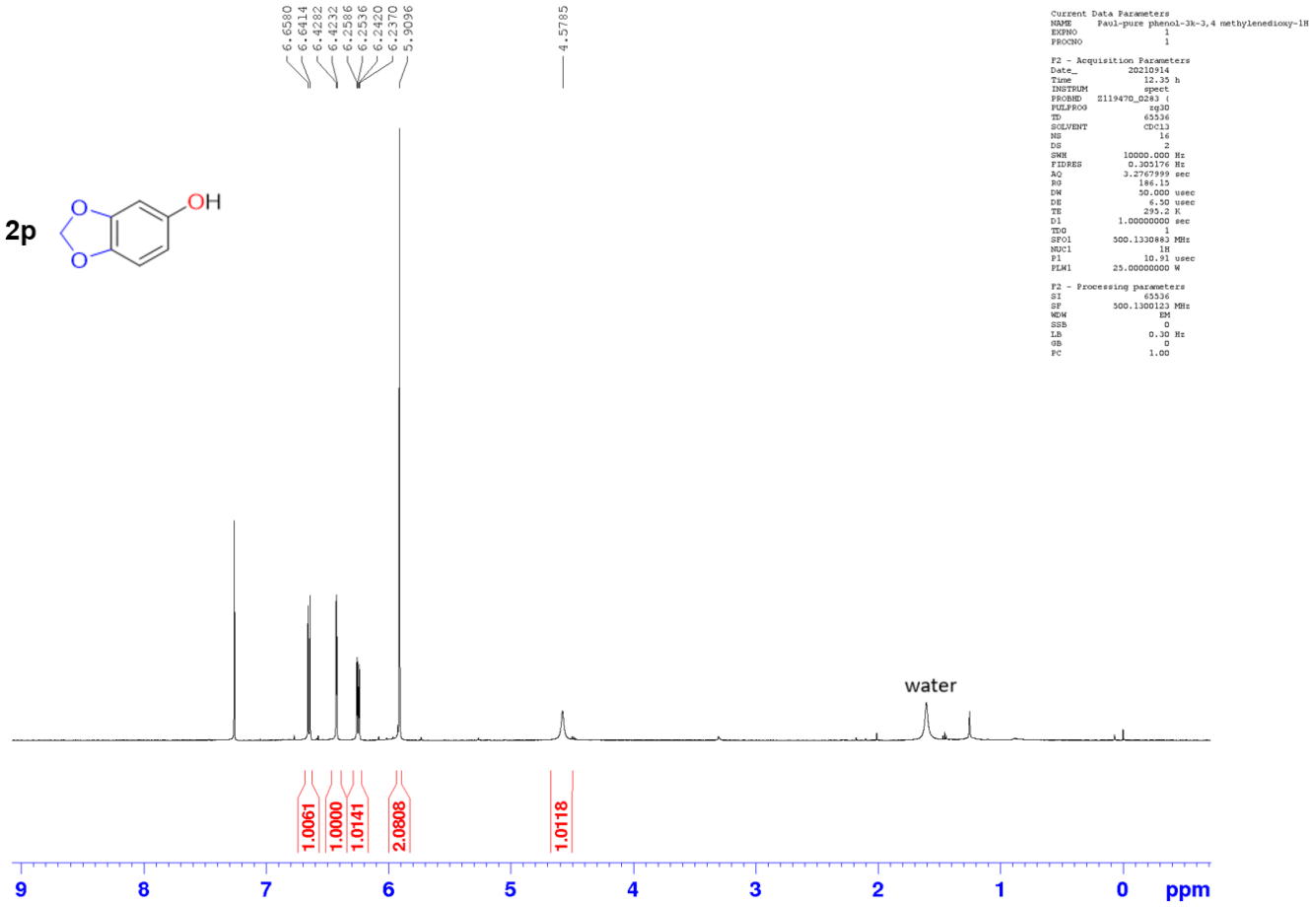

161.10
156.83
130.28
107.86
106.58
101.62
55.41
2n
OH
OMe
Current Data Parameters
NAME Paul-pure phenol-3i-3 OMe-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210928
Time 13.08 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 206.72
DW 16.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.75 usec
PLW1 94.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 25.00000000 W
PLW12 0.46495000 W
PLW13 0.23387000 W
F2 - Processing parameters
SI 32768
SF 125.7577735 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

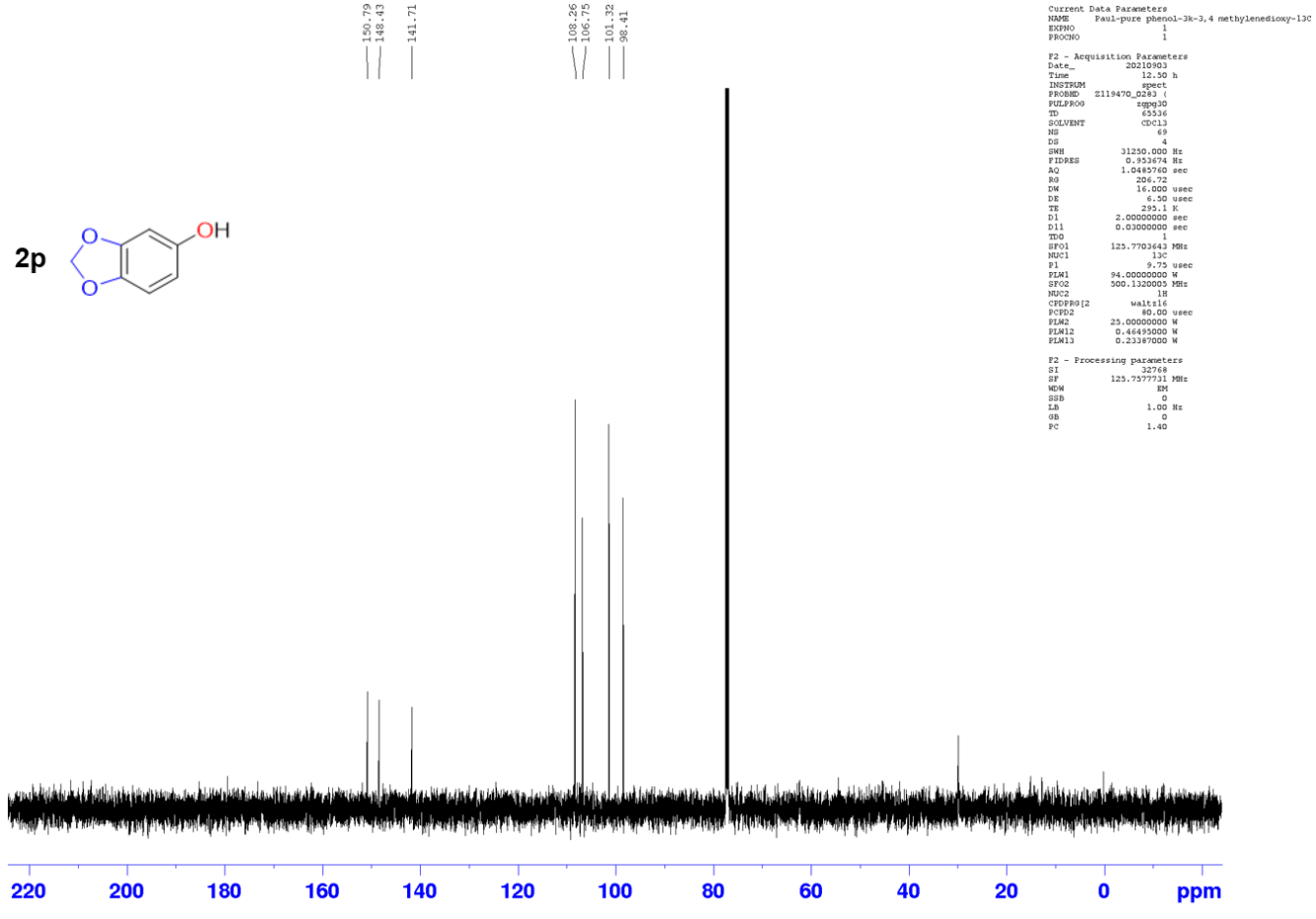


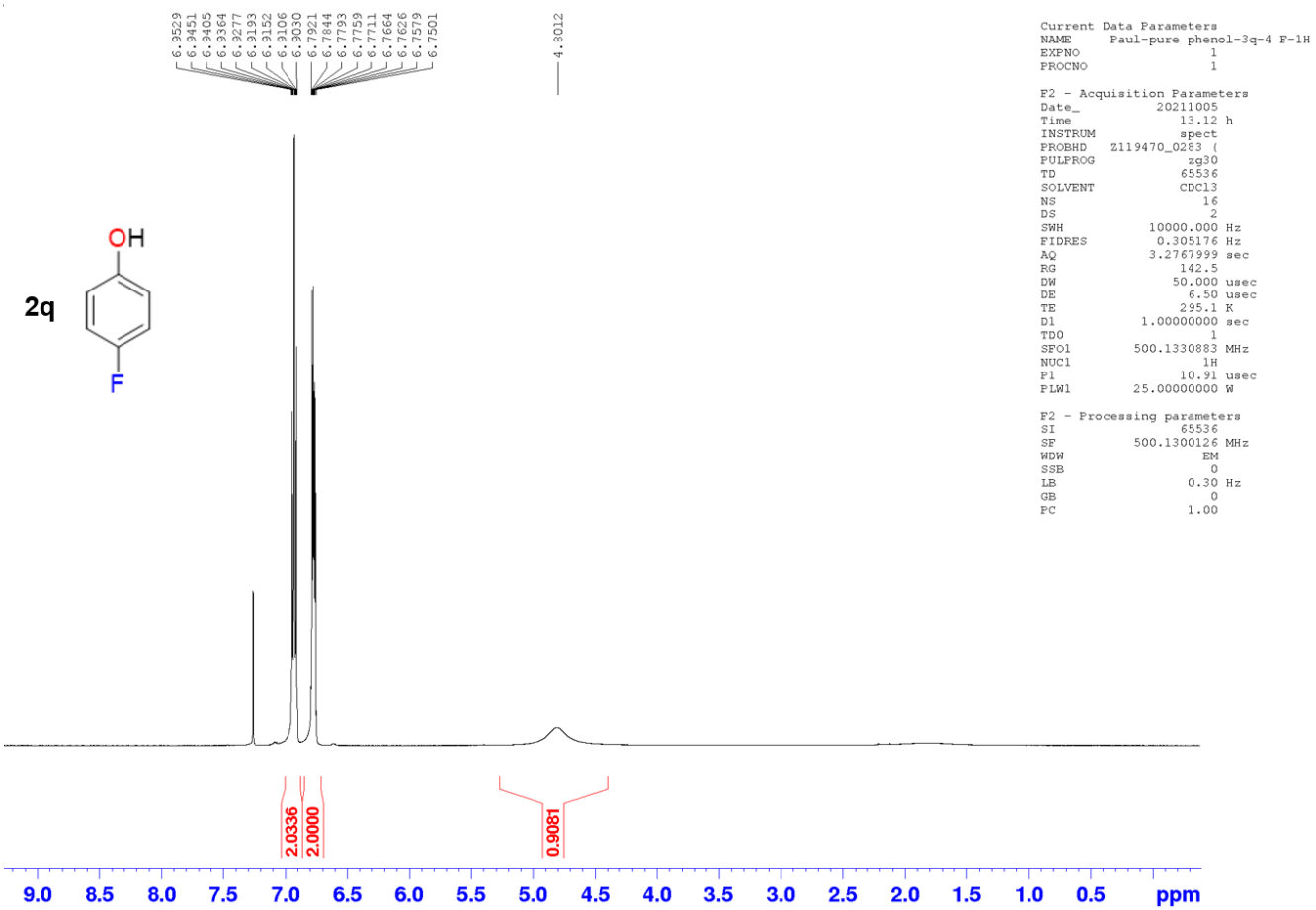

```
Current Data Parameters
NAME      Paul-pure phenol-3j-3,4 di OMe-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210914
Time              12.48 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                 163.99
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1        500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1        25.00000000 W

F2 - Processing parameters
SI                 65536
SF          500.1300123 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



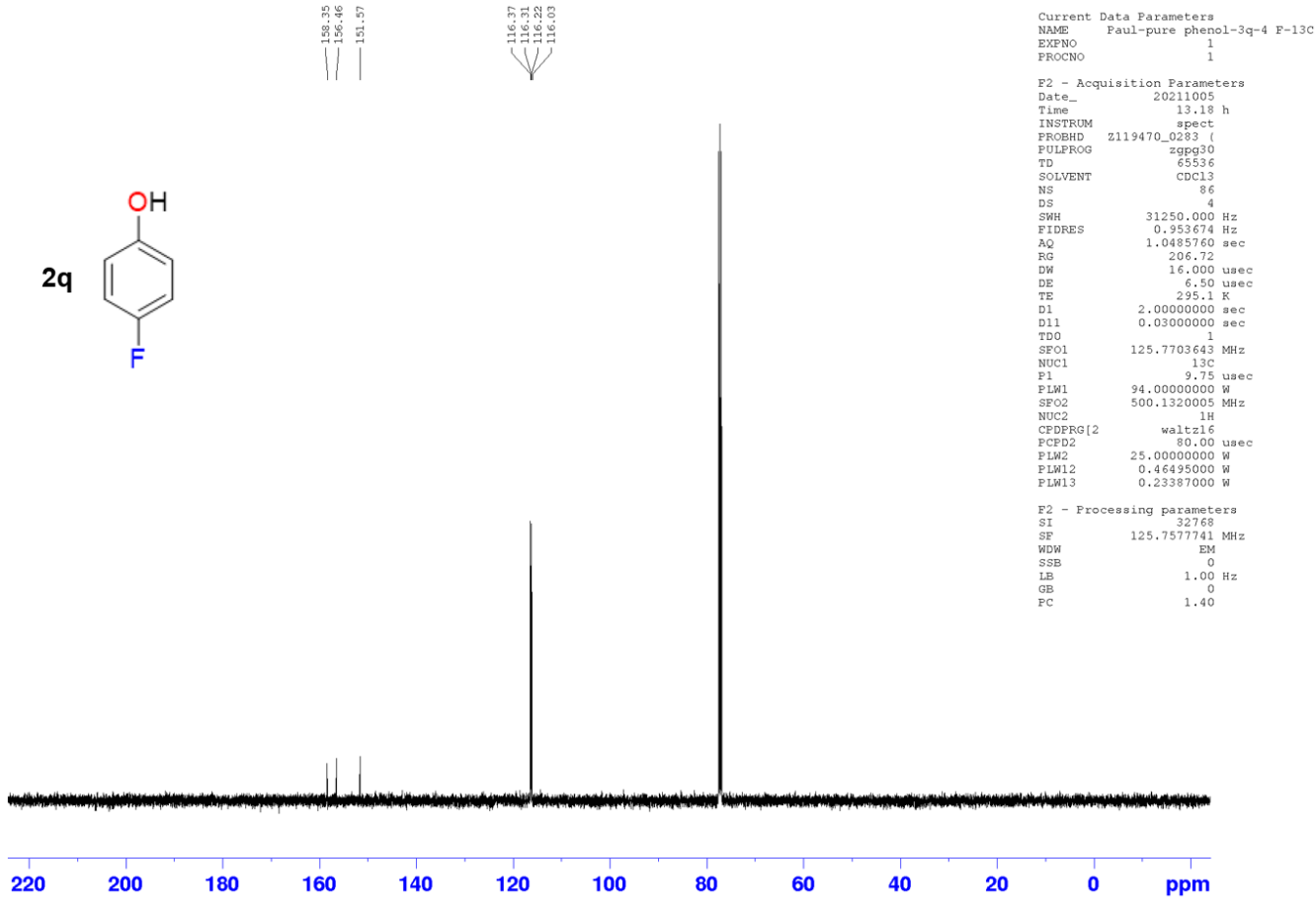

```
Current Data Parameters
NAME      Paul-pure phenol-3j-3,4 di OMe-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210914
Time              12.56 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ             1.1010048 sec
RG                 206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1        125.7703643 MHz
NUC1                 13C
P1                 9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                 32768
SF          125.7577735 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


6.6580
6.6414
6.4282
6.4232
6.2586
6.2536
6.2420
6.2370
5.9096
4.5785
2p
OH
water
1.0061
1.0000
1.0141
2.0808
1.0118
9
8
7
6
5
4
3
2
1
0
ppm

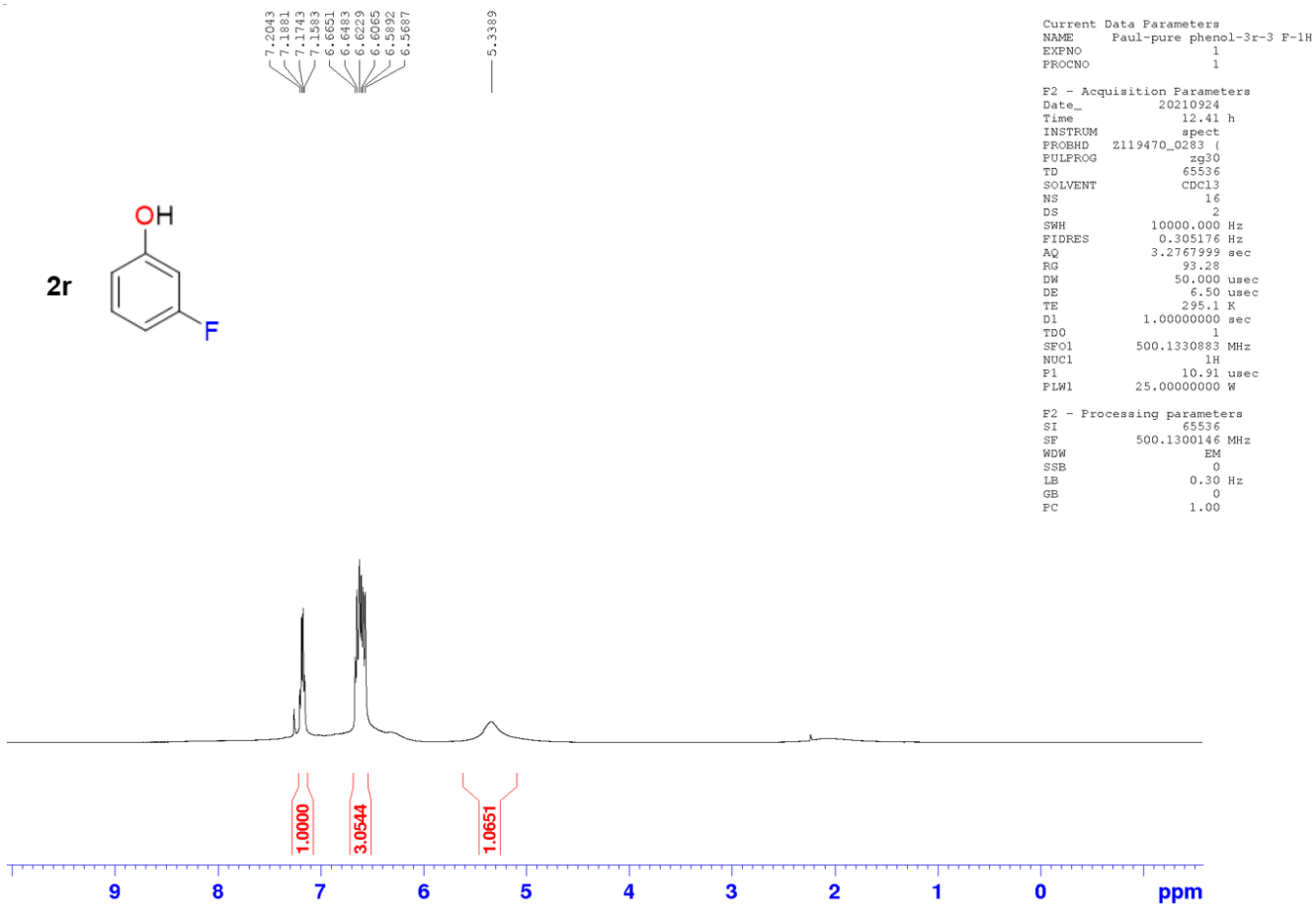

150.79
148.43
141.71
108.26
106.75
101.32
98.41
2p
OH
220
200
180
160
140
120
100
80
60
40
20
0
ppm



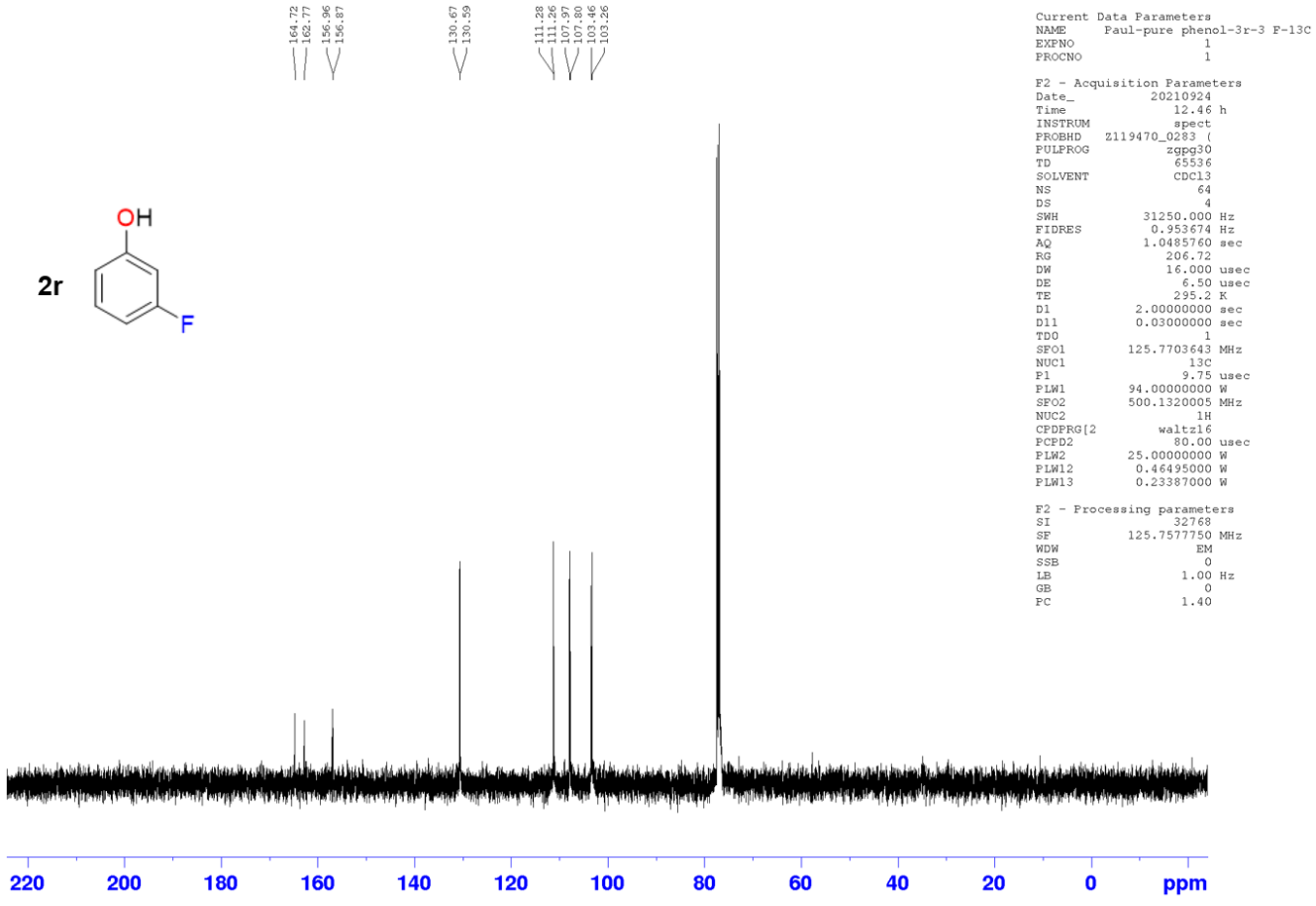

```
Current Data Parameters
NAME      Paul-pure phenol-3q-4 F-1H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20211005
Time              13.12 h
INSTRUM           spect
PROBHD    Z119470_0283 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10000.000 Hz
FIDRES           0.305176 Hz
AQ             3.2767999 sec
RG                142.5
DW               50.000 usec
DE                6.50 usec
TE               295.1 K
D1            1.00000000 sec
TD0                  1
SFO1         500.1330883 MHz
NUC1                 1H
P1               10.91 usec
PLW1         25.00000000 W

F2 - Processing parameters
SI               65536
SF           500.1300126 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



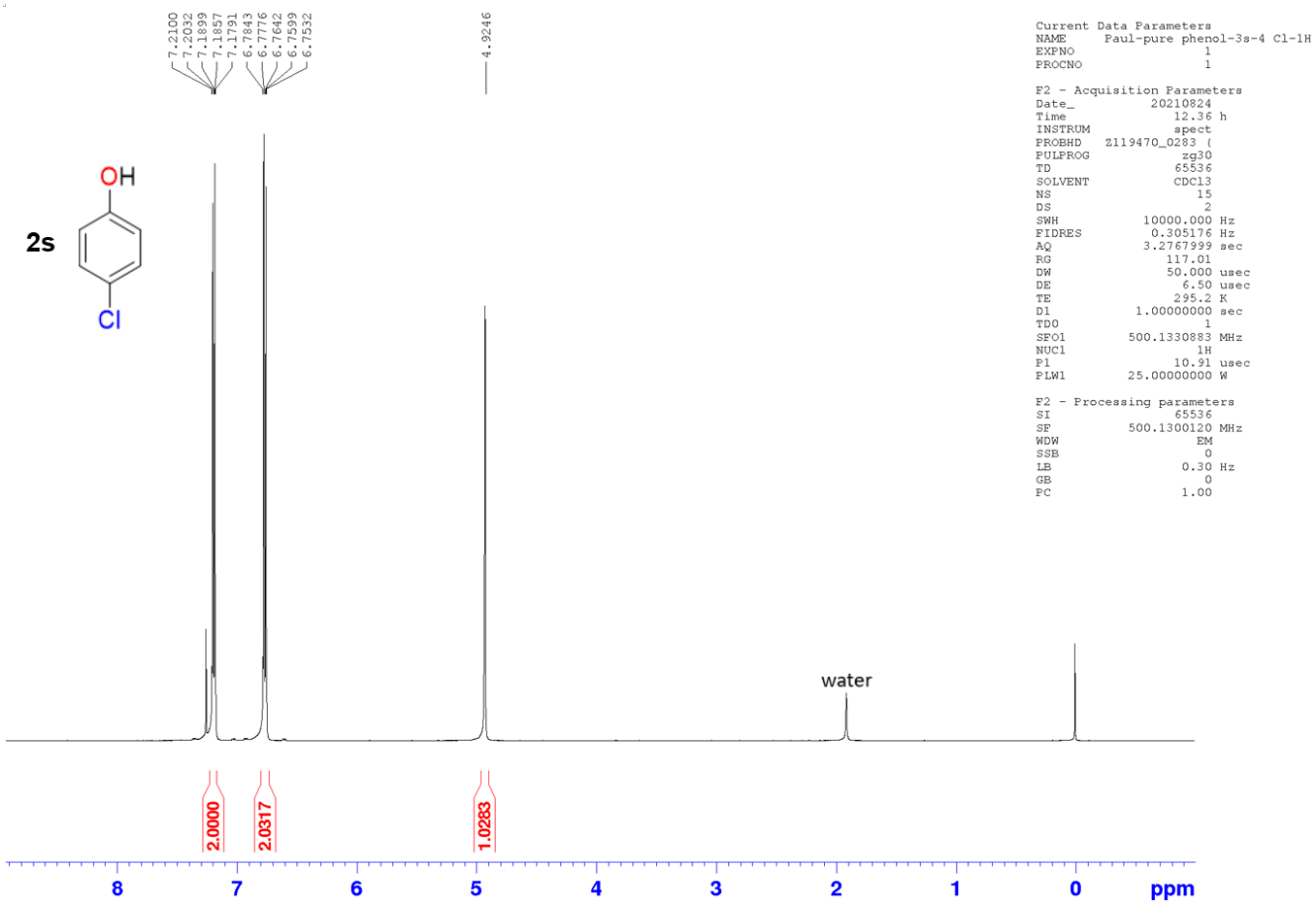

```
Current Data Parameters
NAME      Paul-pure phenol-3q-4 F-13C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20211005
Time              13.18 h
INSTRUM           spect
PROBHD    Z119470_0283 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  86
DS                   4
SWH          31250.000 Hz
FIDRES           0.953674 Hz
AQ             1.0485760 sec
RG                206.72
DW               16.000 usec
DE                6.50 usec
TE               295.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1        125.7703643 MHz
NUC1                 13C
P1                9.75 usec
PLW1         94.00000000 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI               32768
SF           125.7577741 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



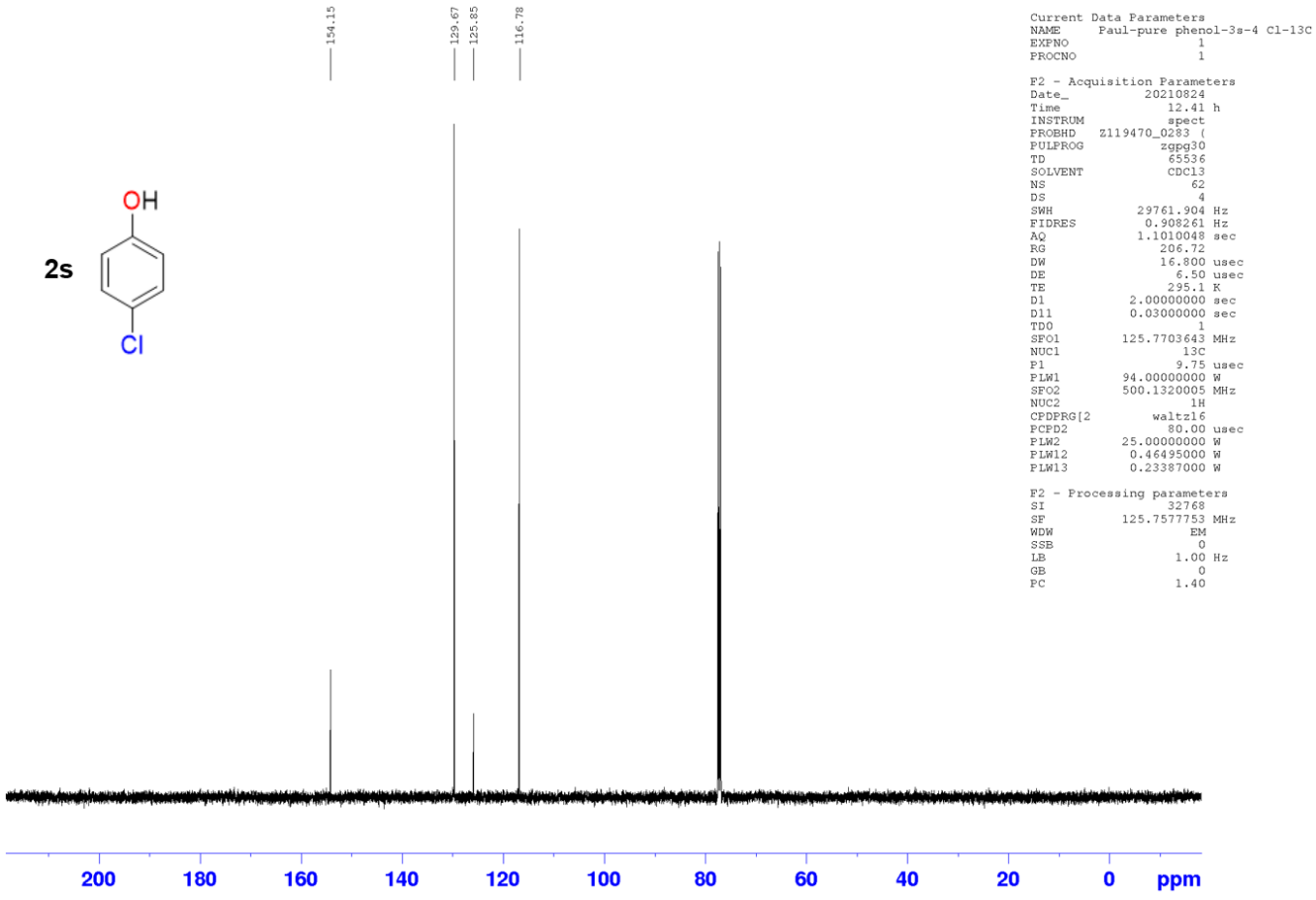

```
Current Data Parameters
NAME      Paul-pure phenol-3r-3 F-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210924
Time              12.41 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                 93.28
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
TD0                    1
SFO1        500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1        25.00000000 W

F2 - Processing parameters
SI                 65536
SF          500.1300146 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



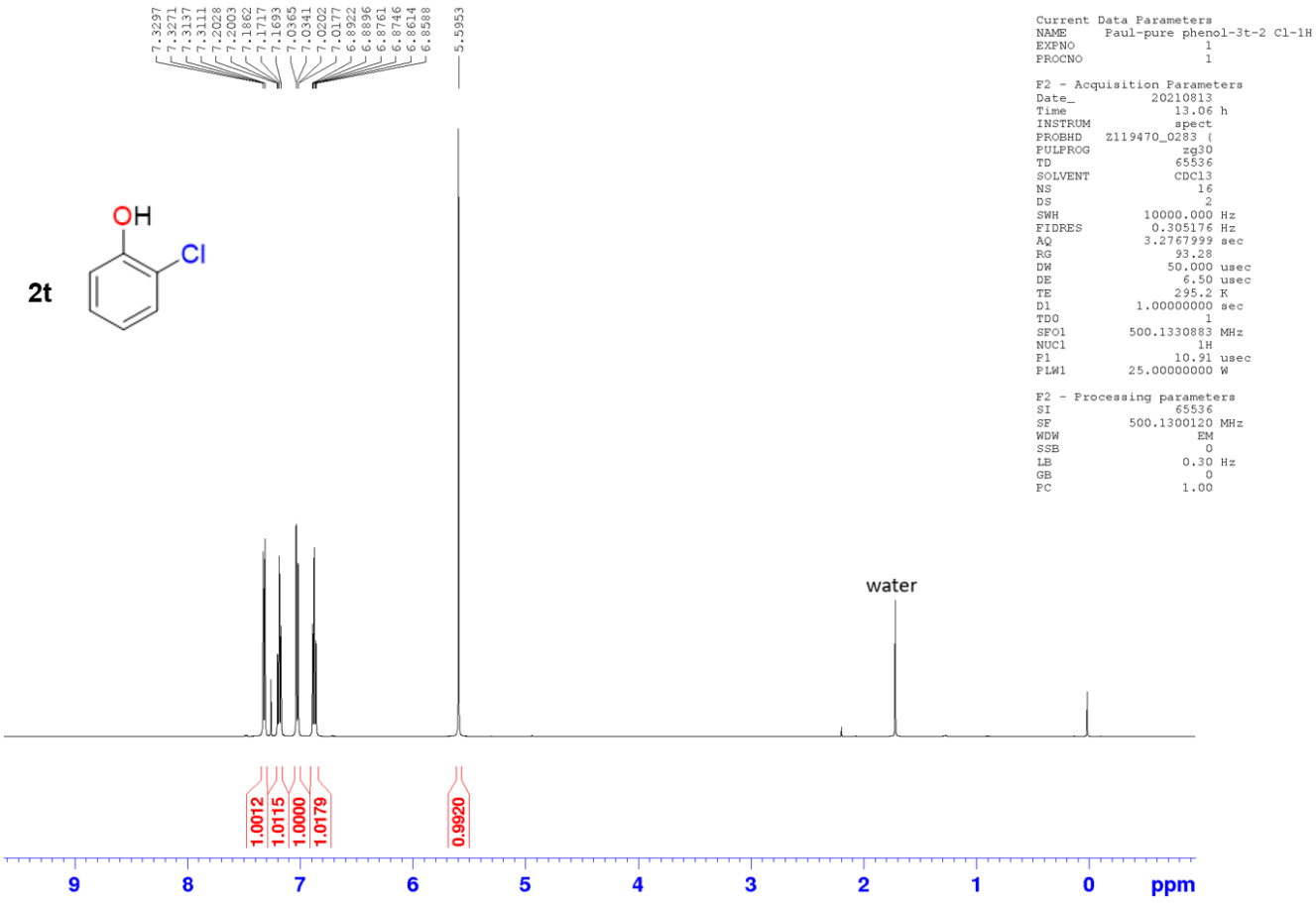

```
Current Data Parameters
NAME      Paul-pure phenol-3r-3 F-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210924
Time              12.46 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    64
DS                     4
SWH           31250.000 Hz
FIDRES         0.953674 Hz
AQ            1.0485760 sec
RG                206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        125.7703643 MHz
NUC1                  13C
P1                 9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                 32768
SF          125.7577750 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



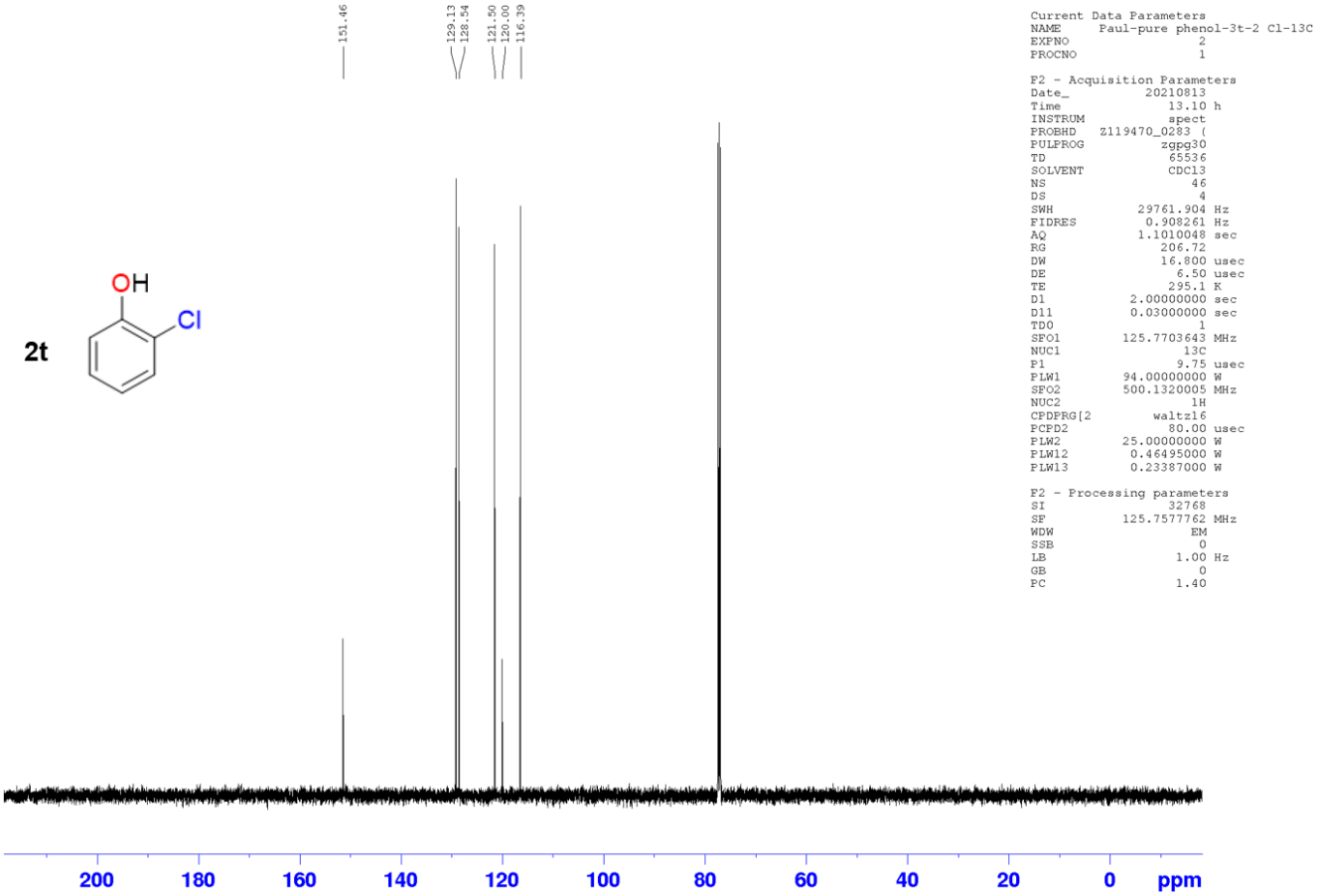

```
Current Data Parameters
NAME      Paul-pure phenol-3s-4 Cl-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210824
Time              12.36 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    15
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                117.01
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300120 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



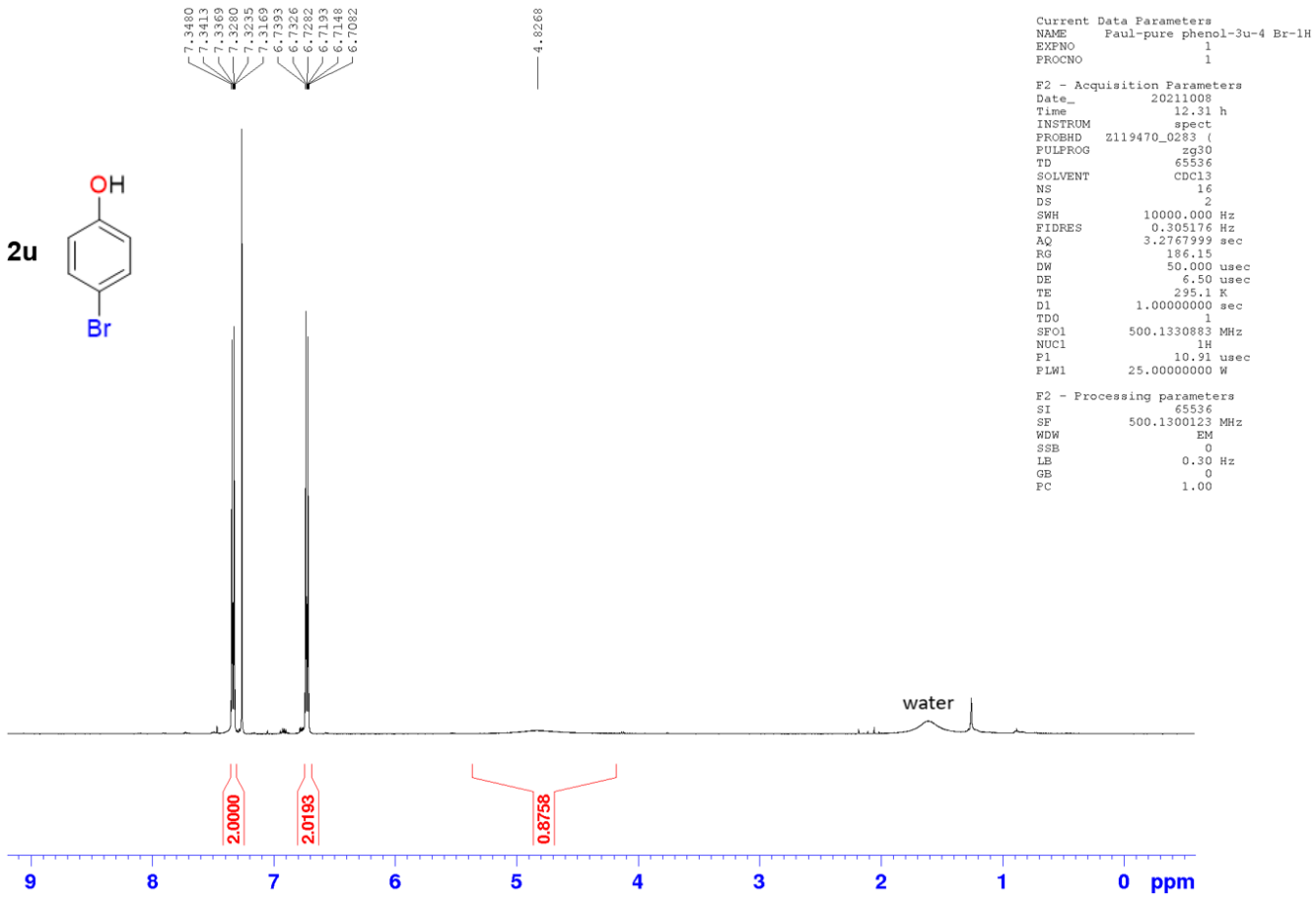

```
Current Data Parameters
NAME      Paul-pure phenol-3s-4 Cl-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210824
Time              12.41 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    62
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                 13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577753 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



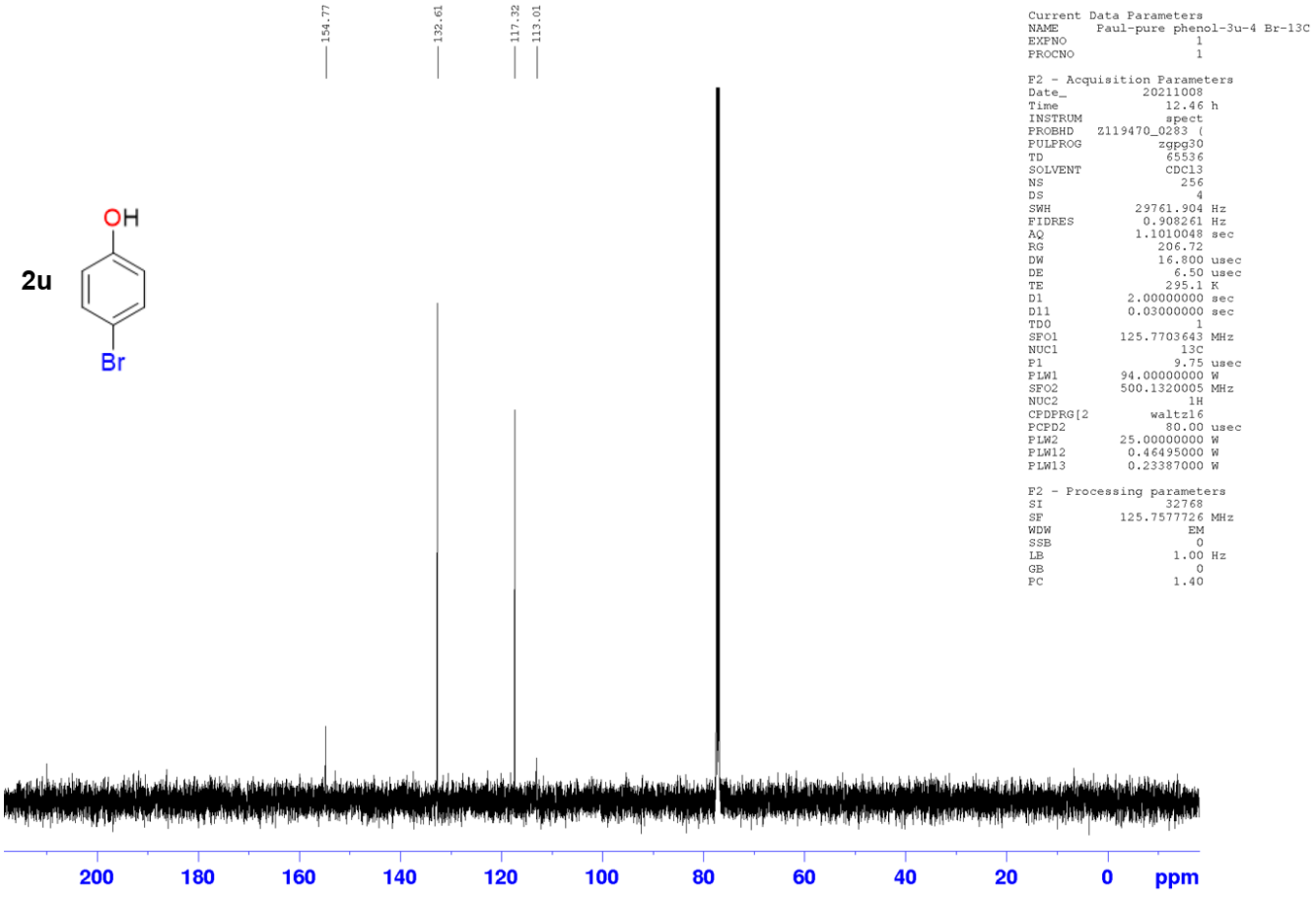

```
Current Data Parameters
NAME      Paul-pure phenol-3t-2 Cl-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210813
Time              13.06 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ                3.2767999 sec
RG                   93.28
DW                 50.000 usec
DE                  6.50 usec
TE                 295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300120 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



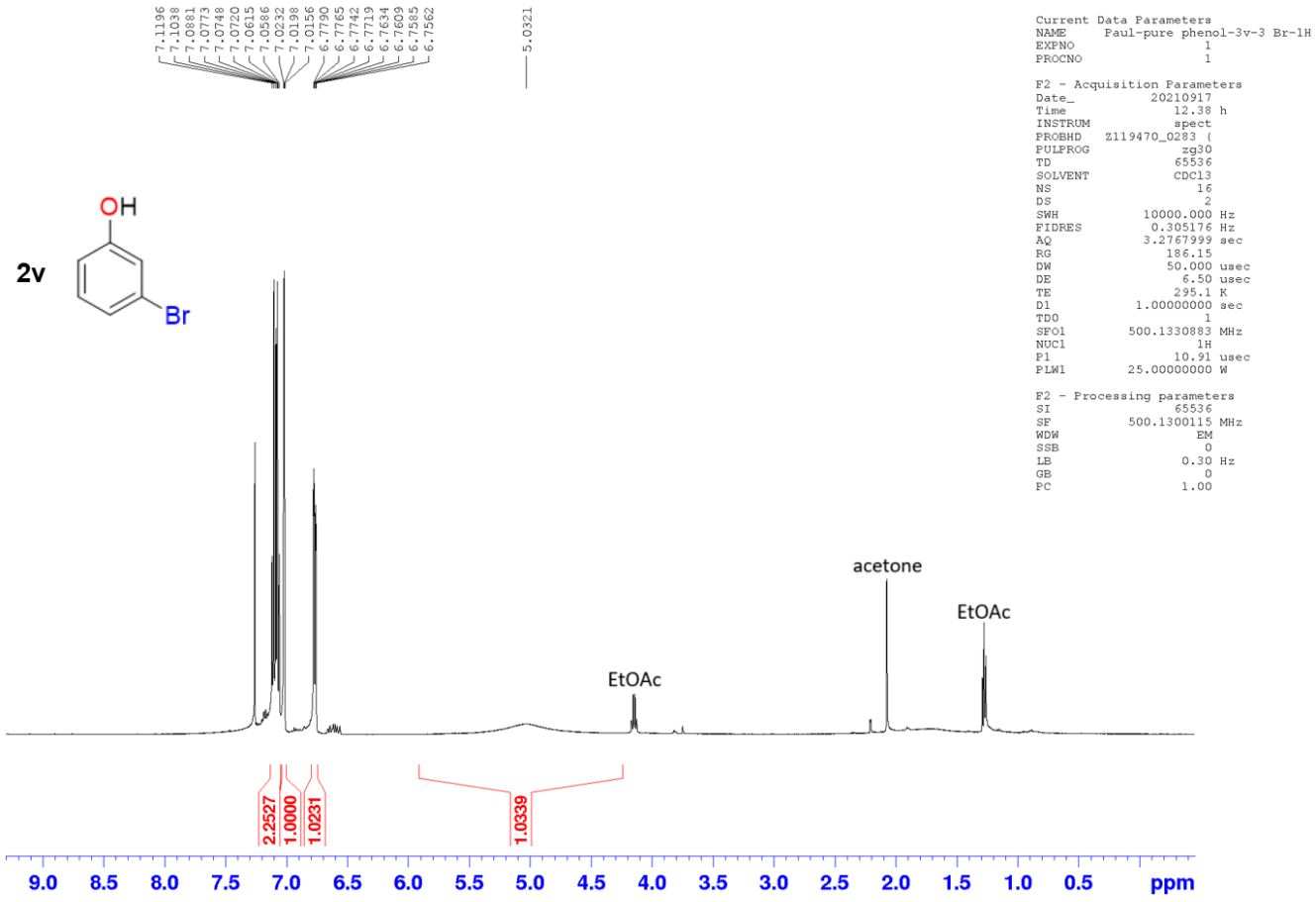

```
Current Data Parameters
NAME      Paul-pure phenol-3t-2 Cl-13C
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20210813
Time              13.10 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    46
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ                1.1010048 sec
RG                  206.72
DW                 16.800 usec
DE                  6.50 usec
TE                 295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                  9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577762 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



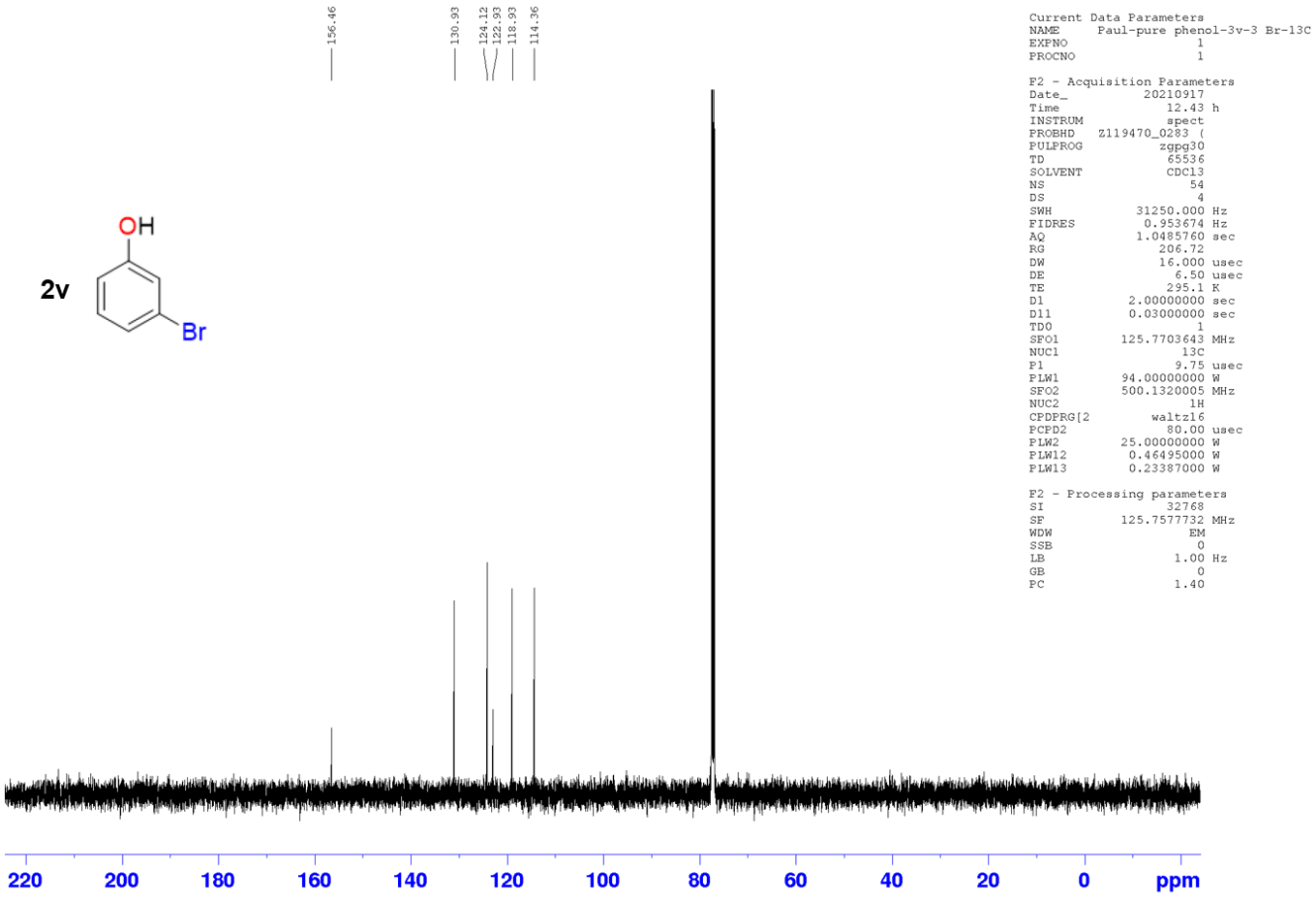

```
Current Data Parameters
NAME      Paul-pure phenol-3u-4 Br-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211008
Time              12.31 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                186.15
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1         25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300123 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



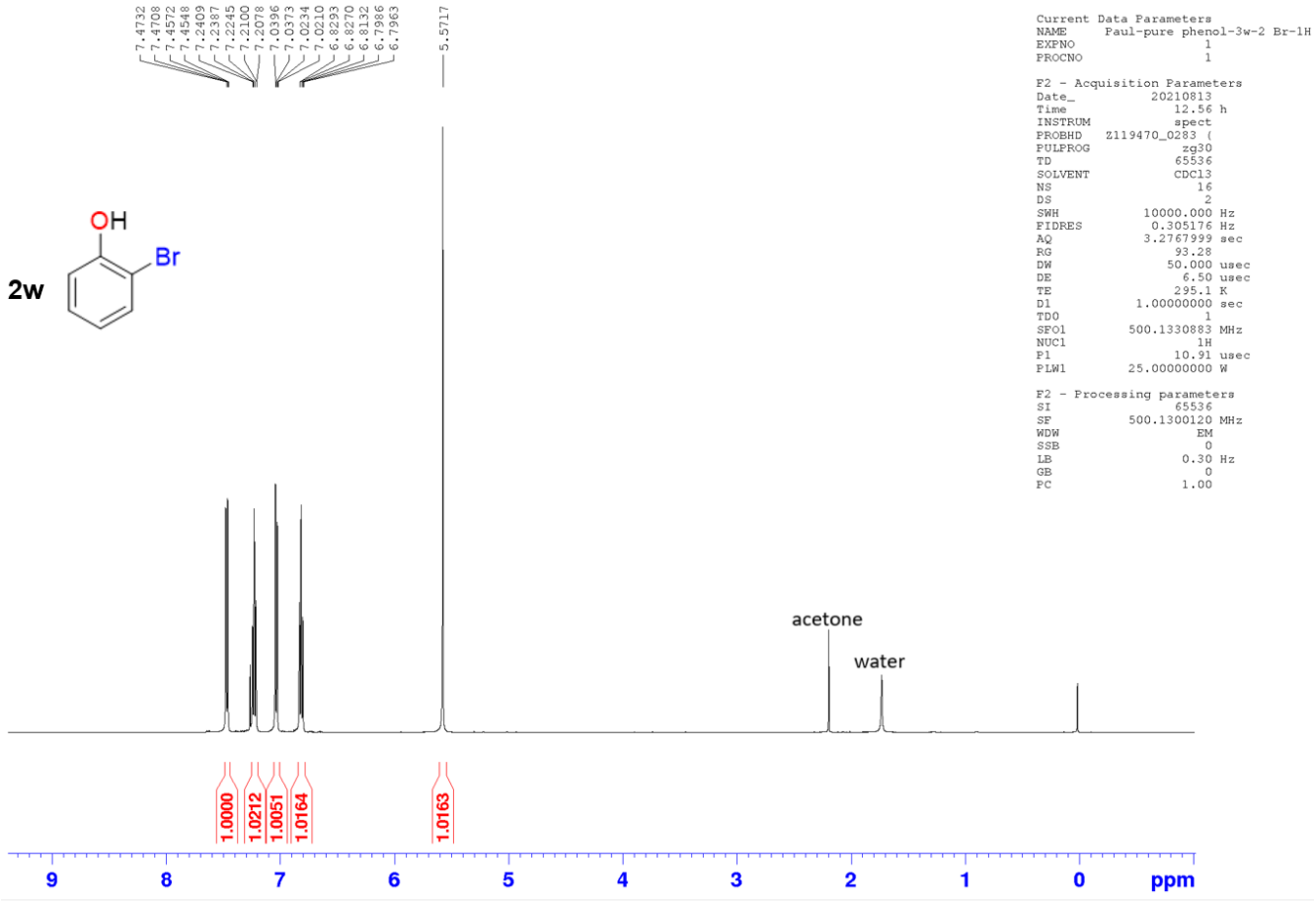

```
Current Data Parameters
NAME      Paul-pure phenol-3u-4 Br-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211008
Time              12.46 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                 13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577726 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



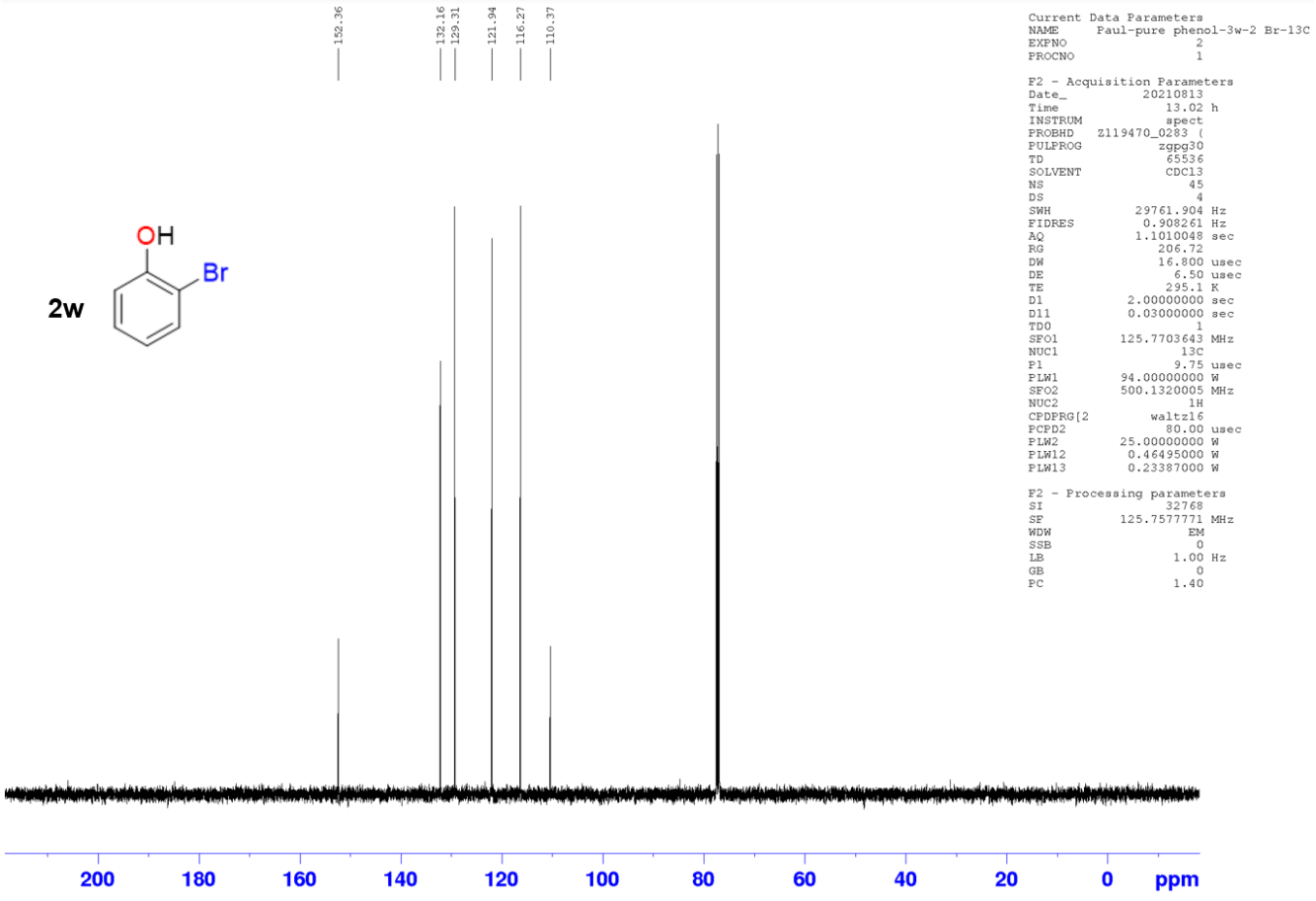

```
Current Data Parameters
NAME      Paul-pure phenol-3v-3 Br-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210917
Time              12.38 h
INSTRUM            spect
PROBHD    Z119470_0283 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ              3.2767999 sec
RG                186.15
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                65536
SF          500.1300115 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



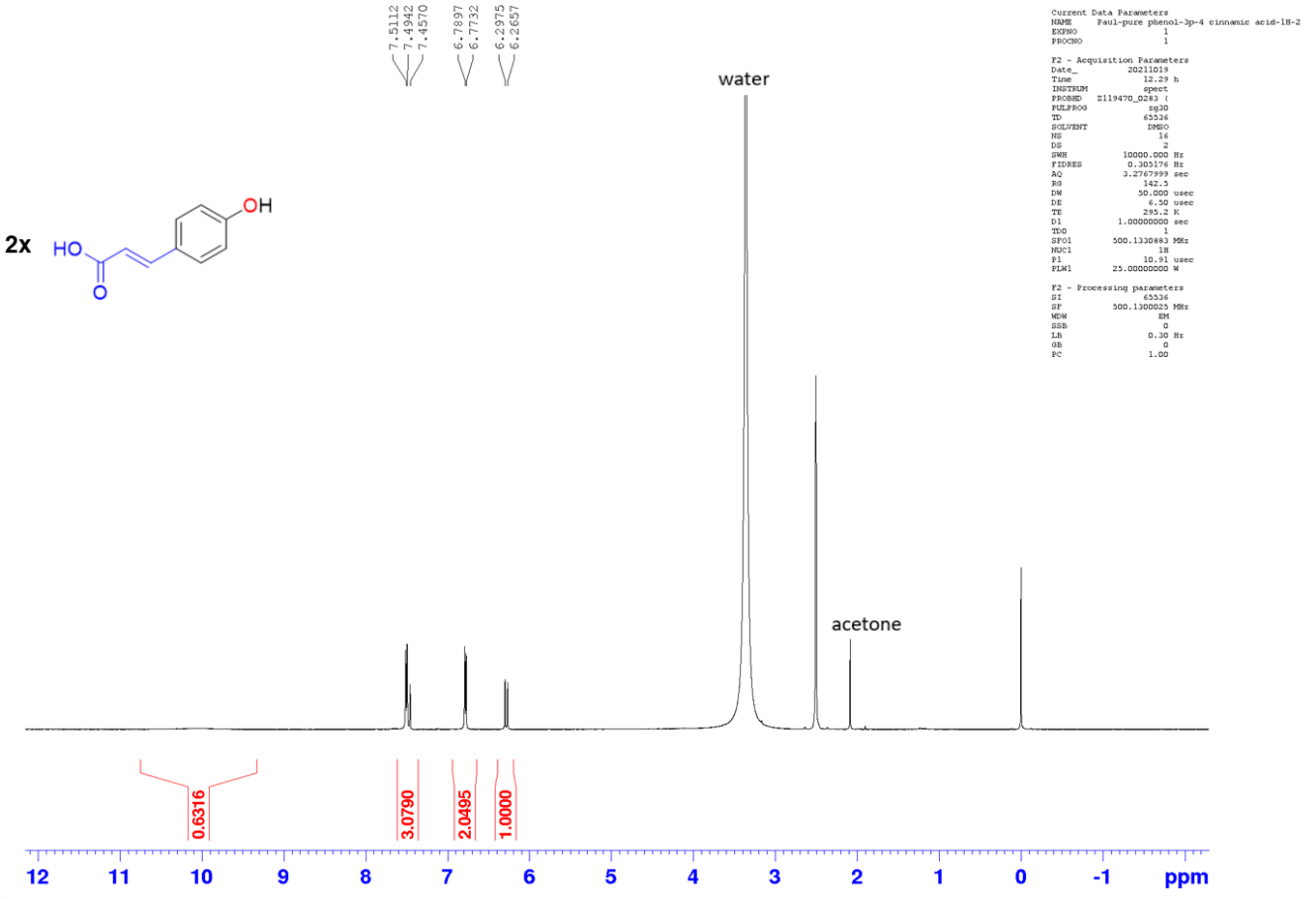

```
Current Data Parameters
NAME      Paul-pure phenol-3v-3 Br-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210917
Time              12.43 h
INSTRUM            spect
PROBHD    Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   54
DS                    4
SWH           31250.000 Hz
FIDRES            0.953674 Hz
AQ              1.0485760 sec
RG                206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                  9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                32768
SF          125.7577732 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

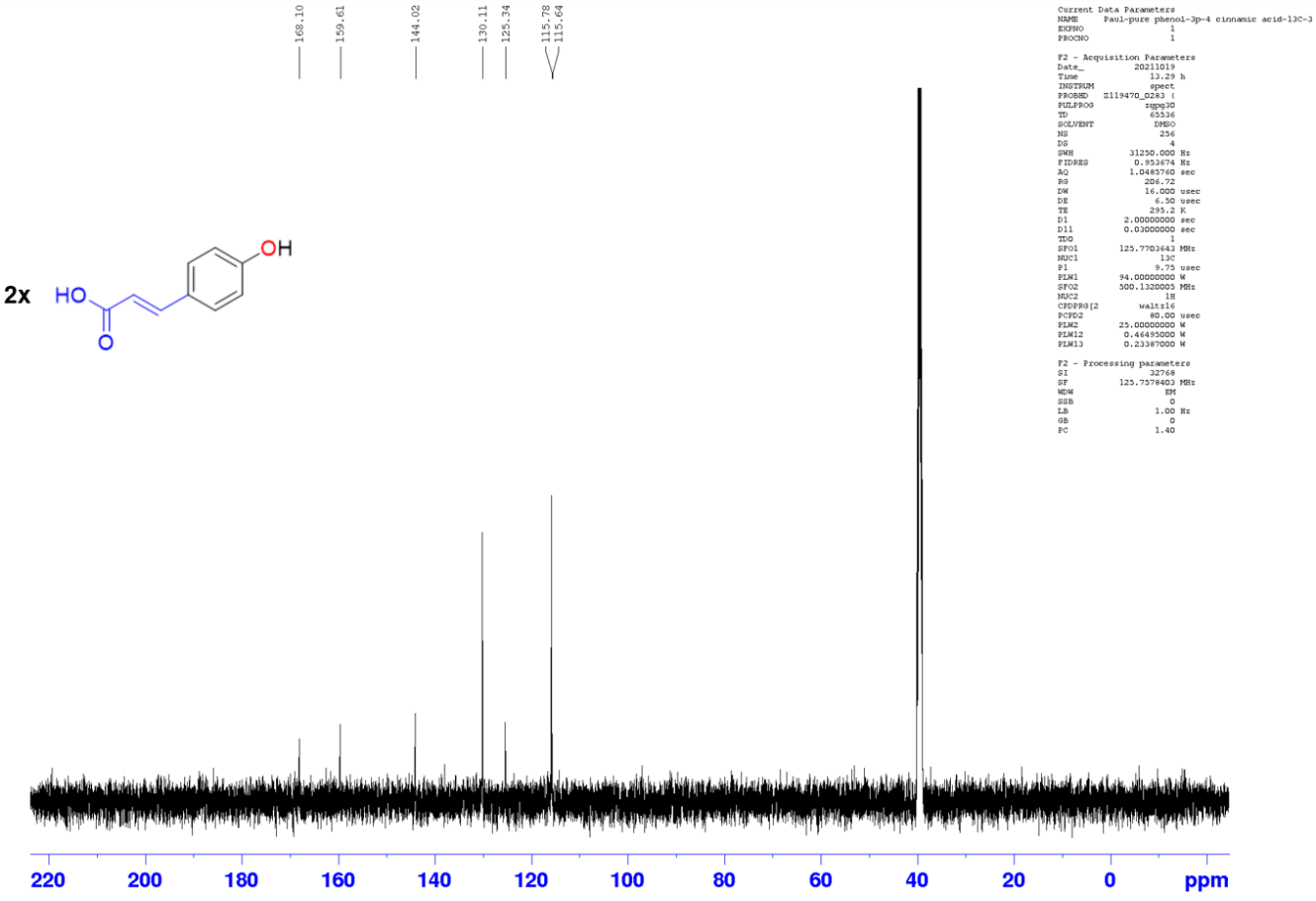


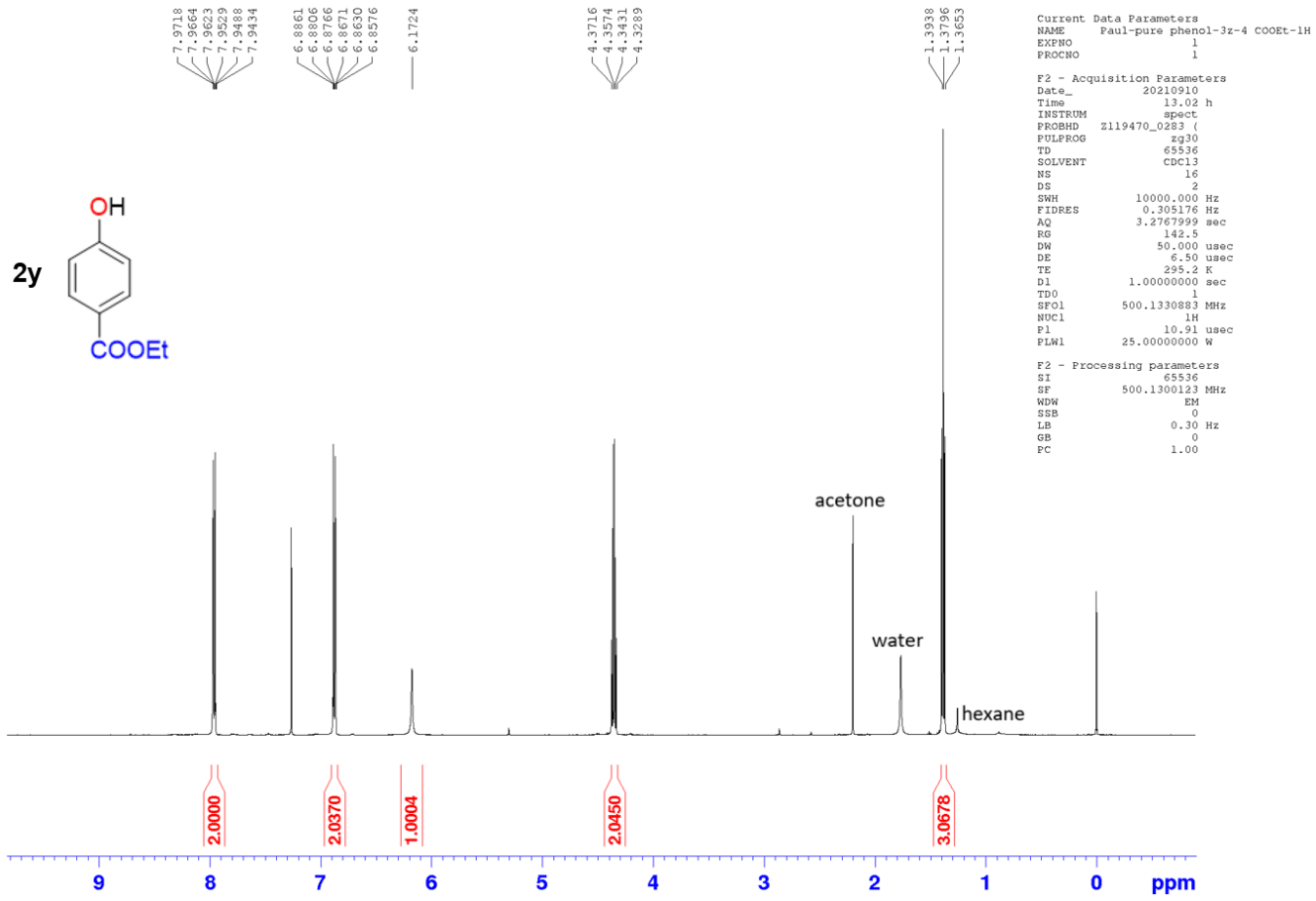

```
Current Data Parameters
NAME      Paul-pure phenol-3w-2 Br-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210813
Time              12.56 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                 93.28
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300120 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



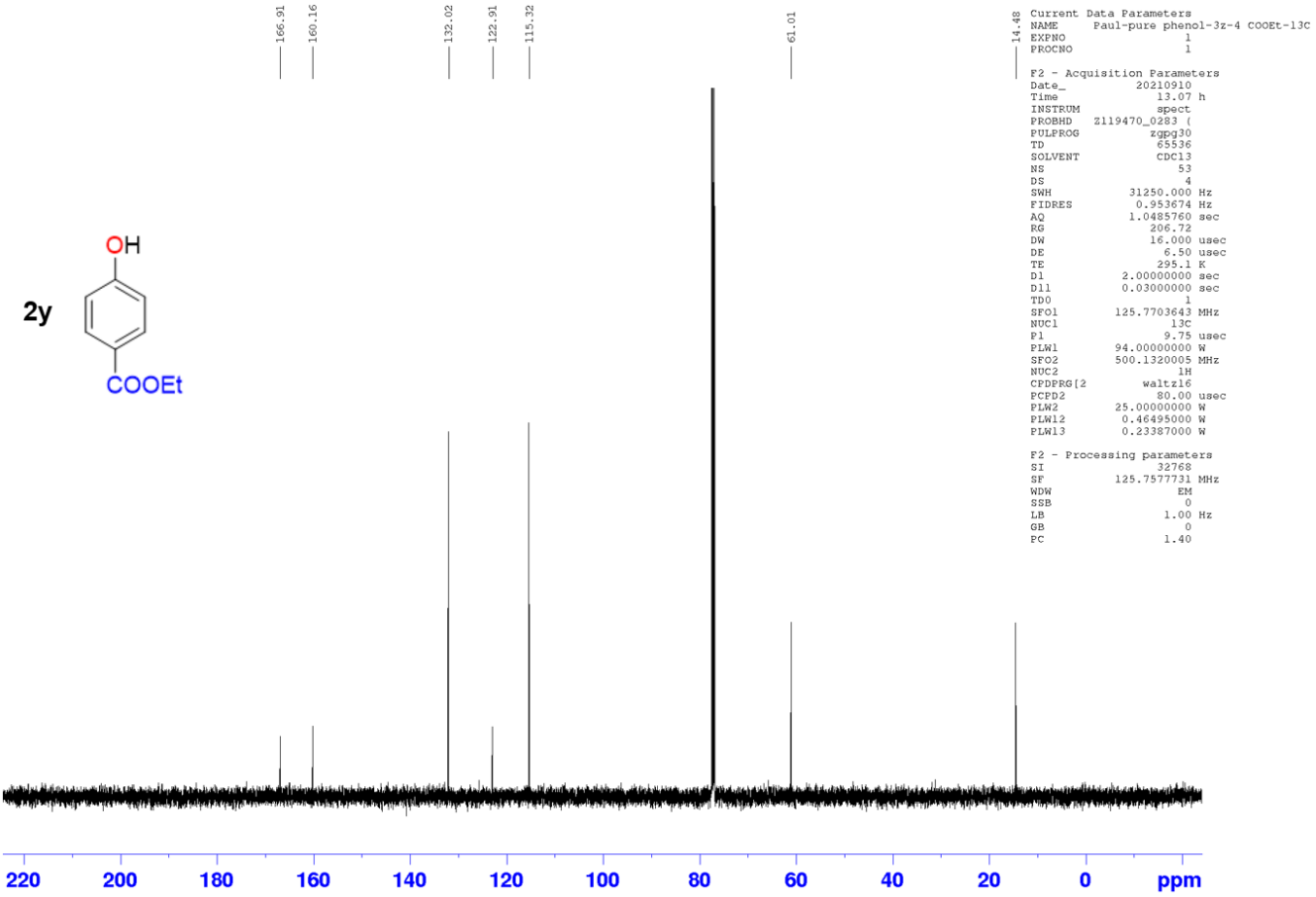

```
Current Data Parameters
NAME      Paul-pure phenol-3w-2 Br-13C
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20210813
Time              13.02 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    45
DS                     4
SWH           29761.904 Hz
FIDRES            0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                 9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577771 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


7.5112
7.4942
7.4570
6.7897
6.7732
6.2975
6.2657
water
acetone
2x
HO
OH
O
0.6316
3.0790
2.0495
1.0000
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

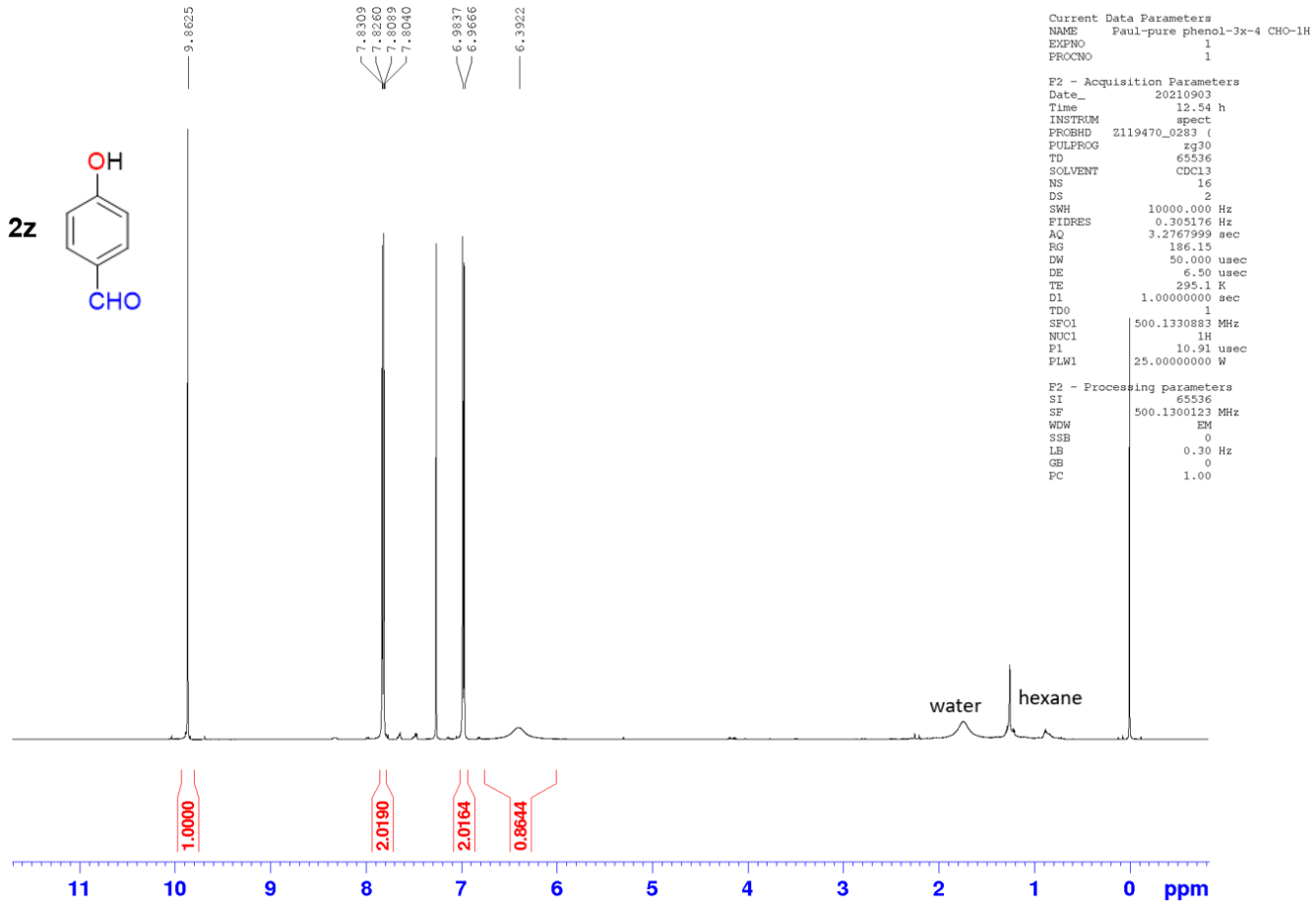

168.10
159.61
144.02
130.11
125.34
115.78
115.64
2x
HO
OH
O
220
200
180
160
140
120
100
80
60
40
20
0
ppm



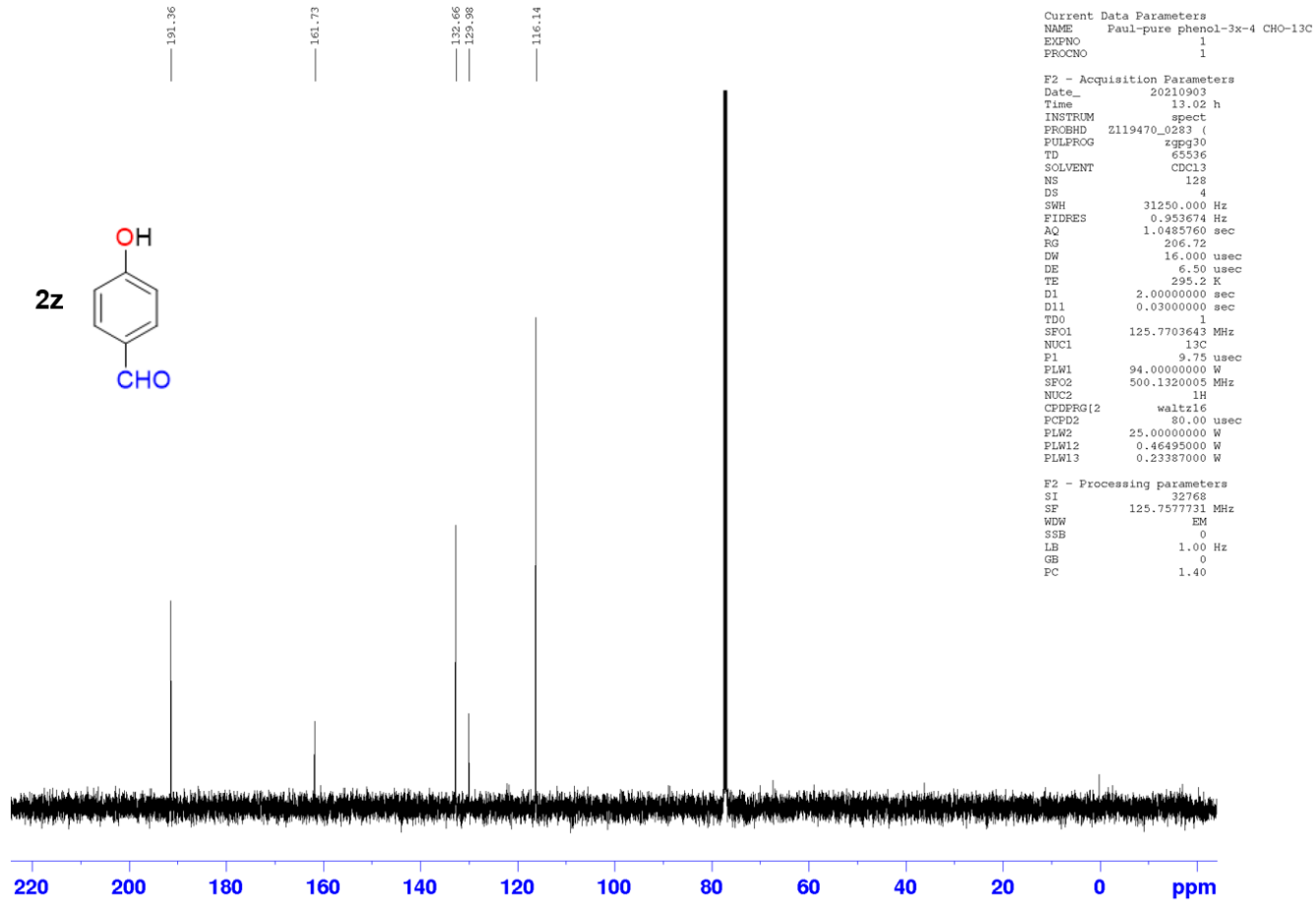

```
Current Data Parameters
NAME      Paul-pure phenol-3z-4 COOEt-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_              20210910
Time                 13.02 h
INSTRUM              spect
PROBHD    Z119470_0283 (
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10000.000 Hz
FIDRES              0.305176 Hz
AQ                  3.2767999 sec
RG                      142.5
DW                   50.000 usec
DE                    6.50 usec
TE                   295.2 K
D1              1.00000000 sec
TD0                      1
SFO1           500.1330883 MHz
NUC1                    1H
P1                   10.91 usec
PLW1           25.00000000 W

F2 - Processing parameters
SI                   65536
SF              500.1300123 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



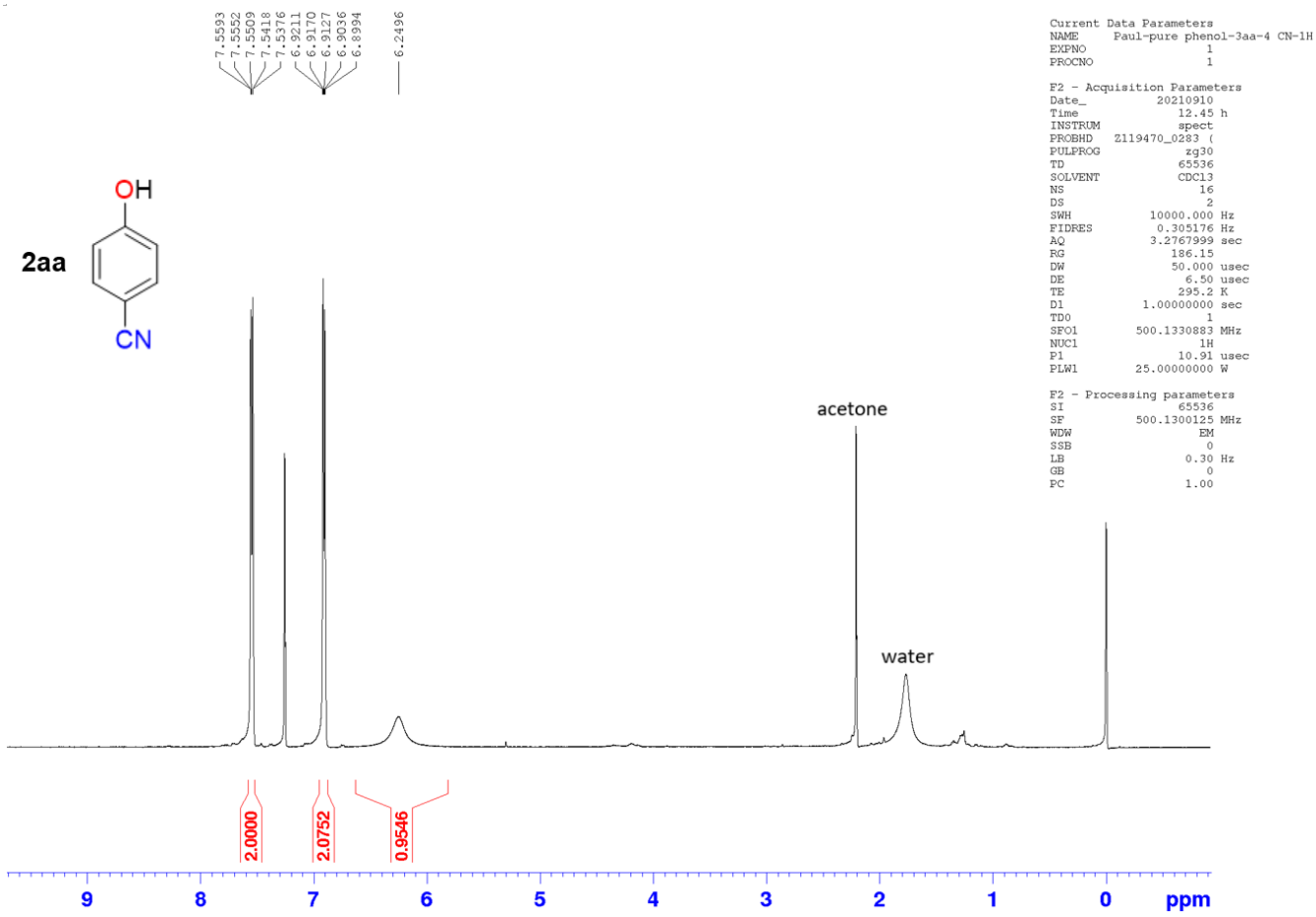

```
Current Data Parameters
NAME      Paul-pure phenol-3z-4 COOEt-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_              20210910
Time                 13.07 h
INSTRUM              spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                  65536
SOLVENT              CDCl3
NS                      53
DS                       4
SWH             31250.000 Hz
FIDRES              0.953674 Hz
AQ                  1.0485760 sec
RG                     206.72
DW                   16.000 usec
DE                    6.50 usec
TE                   295.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           125.7703643 MHz
NUC1                    13C
P1                    9.75 usec
PLW1           94.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2                80.00 usec
PLW2           25.00000000 W
PLW12           0.46495000 W
PLW13           0.23387000 W

F2 - Processing parameters
SI                   32768
SF              125.7577731 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

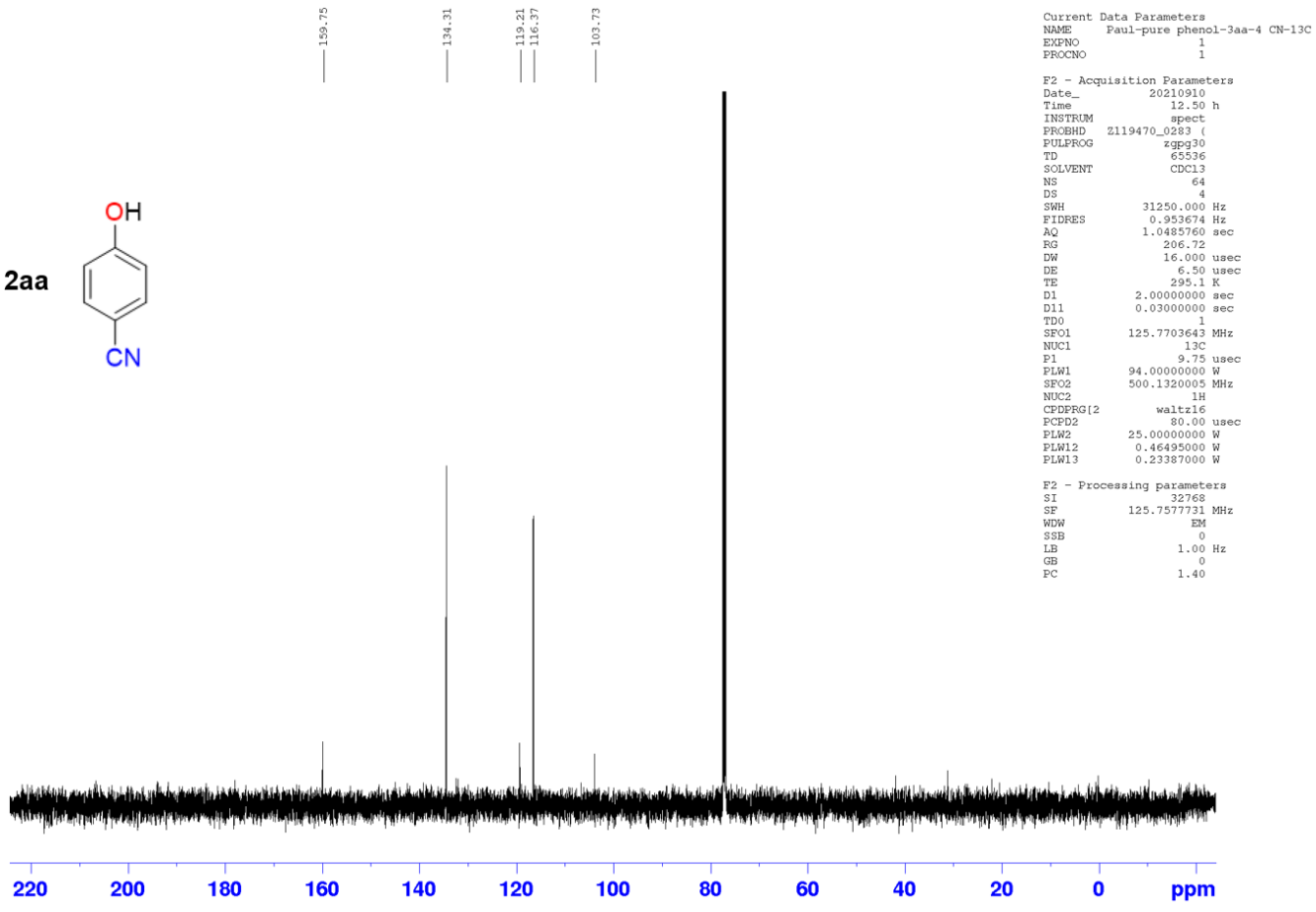


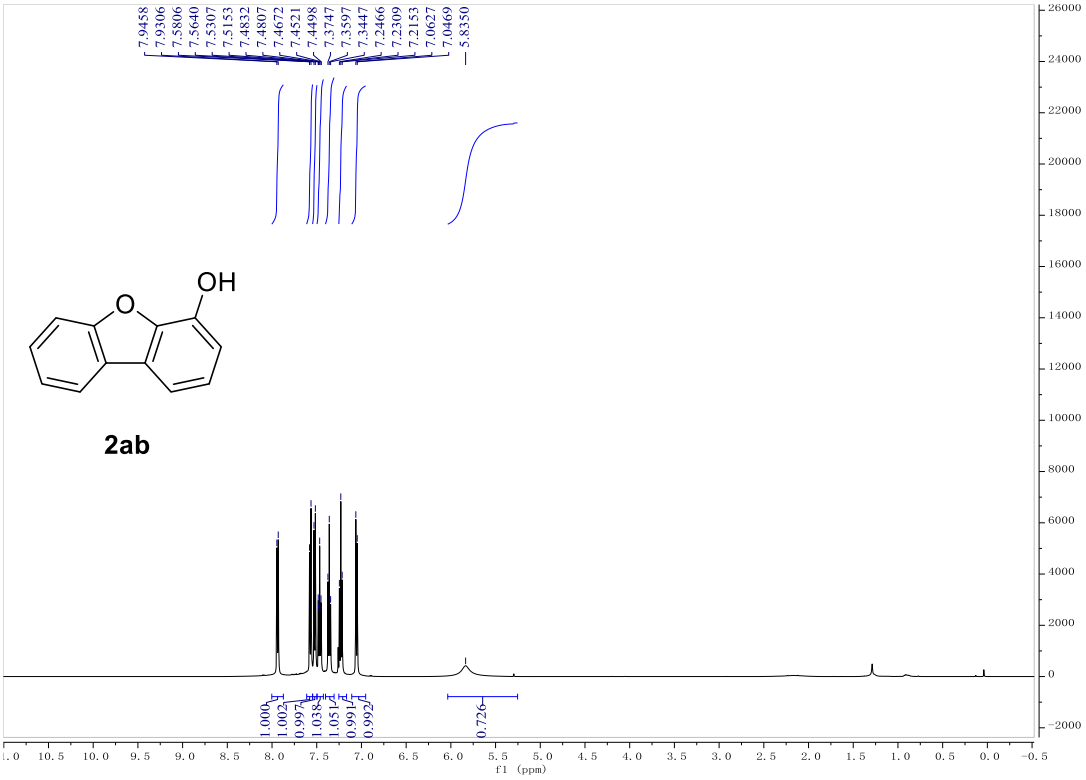

```
Current Data Parameters
NAME      Paul-pure phenol-3x-4 CHO-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210903
Time              12.54 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG               186.15
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                  1
SFO1         500.1330883 MHz
NUC1                 1H
P1                10.91 usec
PLW1         25.00000000 W

F2 - Processing parameters
SI                65536
SF           500.1300123 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

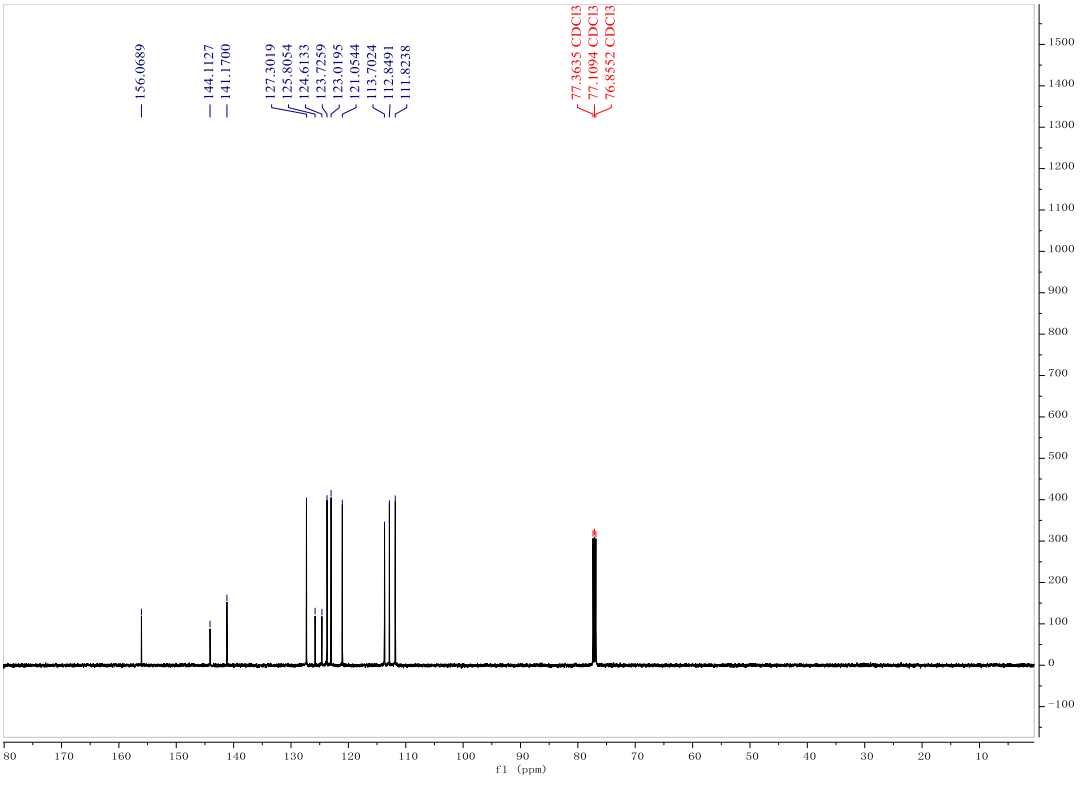


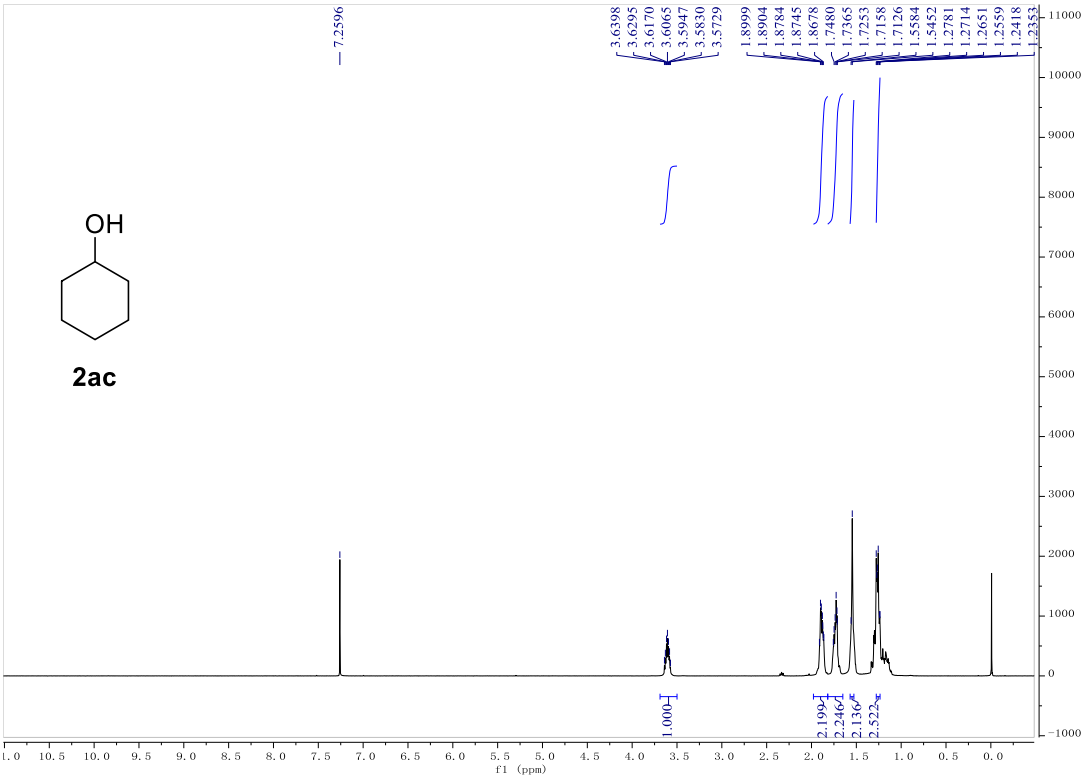

```
Current Data Parameters
NAME      Paul-pure phenol-3x-4 CHO-13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20210903
Time              13.02 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 128
DS                   4
SWH           31250.000 Hz
FIDRES         0.953674 Hz
AQ             1.0485760 sec
RG               206.72
DW                16.000 usec
DE                 6.50 usec
TE                295.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                  1
SFO1         125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1         94.00000000 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                32768
SF           125.7577731 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

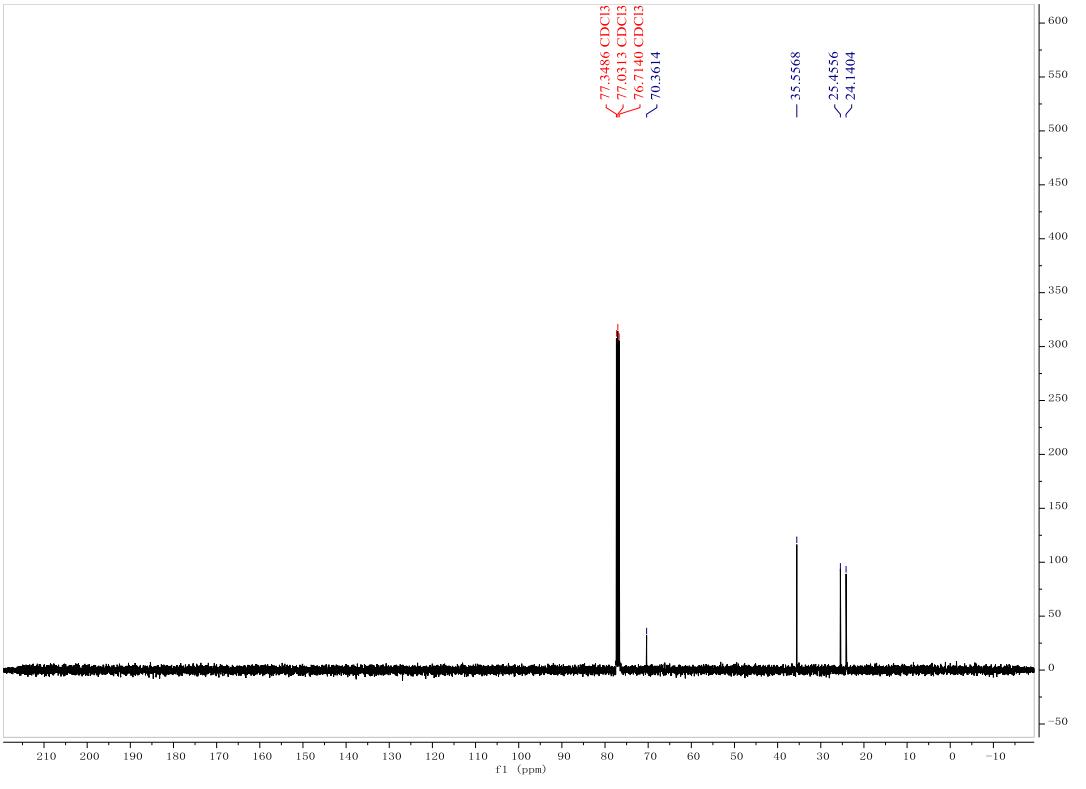

7.5593
7.5552
7.5509
7.5418
7.5376
6.9211
6.9170
6.9127
6.9036
6.8994
6.2496
OH
2aa
CN
acetone
water
2.0000
2.0752
0.9546
9
8
7
6
5
4
3
2
1
0
ppm
Current Data Parameters
NAME Paul-pure phenol-3aa-4 CN-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210910
Time 12.45 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 186.15
DW 50.000 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 10.91 usec
PLW1 25.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

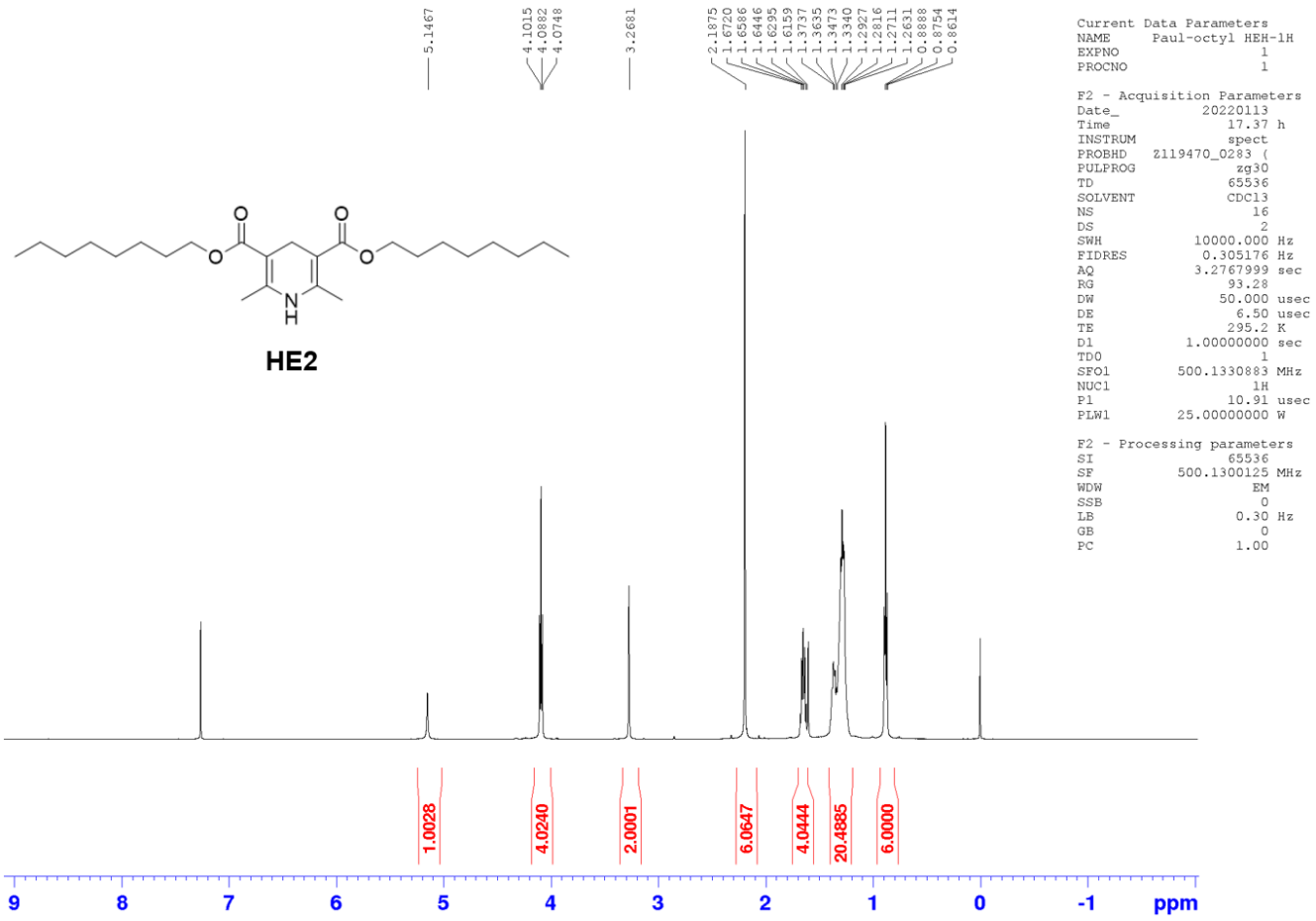

159.75
134.31
119.21
116.37
103.73
OH
2aa
CN
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME Paul-pure phenol-3aa-4 CN-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210910
Time 12.50 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 206.72
DW 16.000 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.75 usec
PLW1 94.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 25.00000000 W
PLW12 0.46495000 W
PLW13 0.23387000 W
F2 - Processing parameters
SI 32768
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.9458
7.9306
7.5806
7.5640
7.5307
7.5153
7.4832
7.4807
7.4672
7.4521
7.4498
7.3747
7.3597
7.3447
7.2466
7.2309
7.2153
7.0627
7.0469
5.8350
OH
O
2ab
1.000
1.002
0.997
1.038
1.051
0.991
0.992
0.726
f1 (ppm)


156.0689
144.1127
141.1700
127.3019
125.8054
124.6133
123.7259
123.0195
121.0544
113.7024
112.8491
111.8238
77.3635 CDCl3
77.1094 CDCl3
76.8552 CDCl3
f1 (ppm)

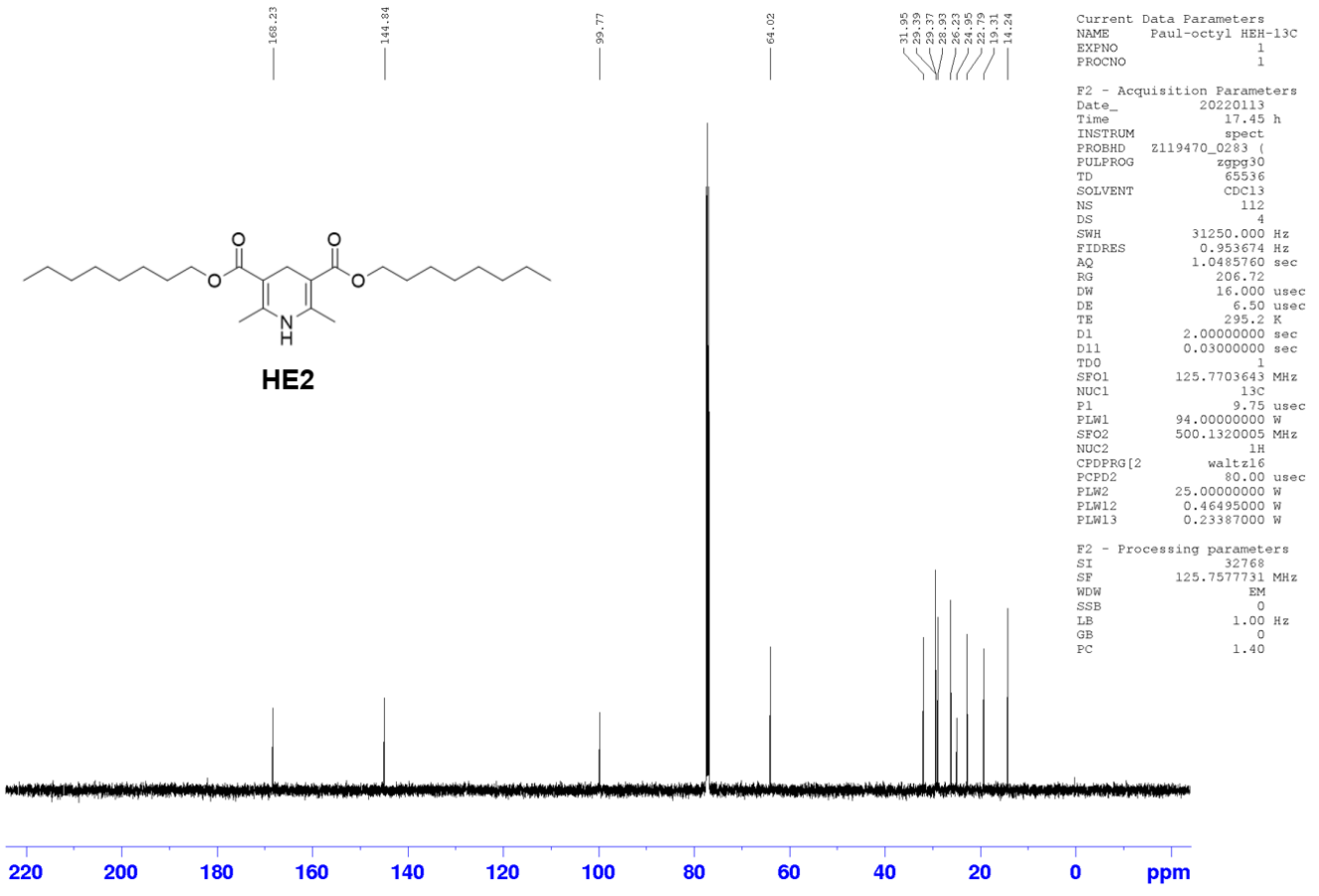

OH
2ac
7.2596
3.6398
3.6295
3.6170
3.6065
3.5947
3.5830
3.5729
1.8999
1.8904
1.8784
1.8745
1.8678
1.7480
1.7365
1.7253
1.7158
1.7126
1.5584
1.5452
1.2781
1.2714
1.2651
1.2559
1.2418
1.2353
1.000
2.199
2.246
2.136
2.522
f1 (ppm)


77.3486 CDCl3
77.0313 CDCl3
76.7140 CDCl3
70.3614
35.5568
25.4556
24.1404
f1 (ppm)



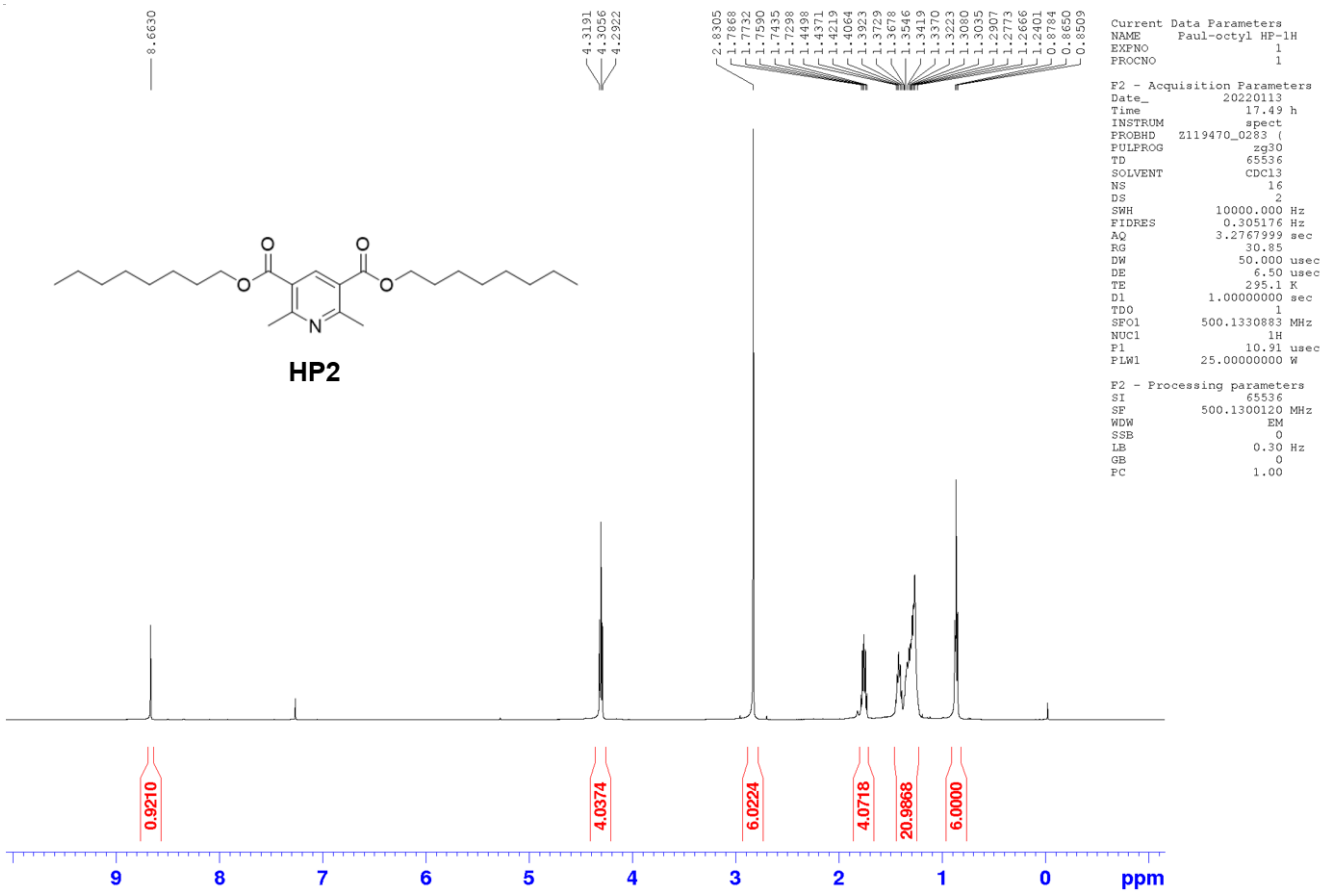
HE2

```
Current Data Parameters
NAME      Paul-octyl HEH-1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20220113
Time                 17.37 h
INSTRUM              spect
PROBHD    Z119470_0283 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10000.000 Hz
FIDRES              0.305176 Hz
AQ                3.2767999 sec
RG                    93.28
DW                   50.000 usec
DE                    6.50 usec
TE                   295.2 K
D1              1.00000000 sec
TD0                     1
SFO1           500.1330883 MHz
NUC1                   1H
P1                   10.91 usec
PLW1           25.00000000 W

F2 - Processing parameters
SI                  65536
SF             500.1300125 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

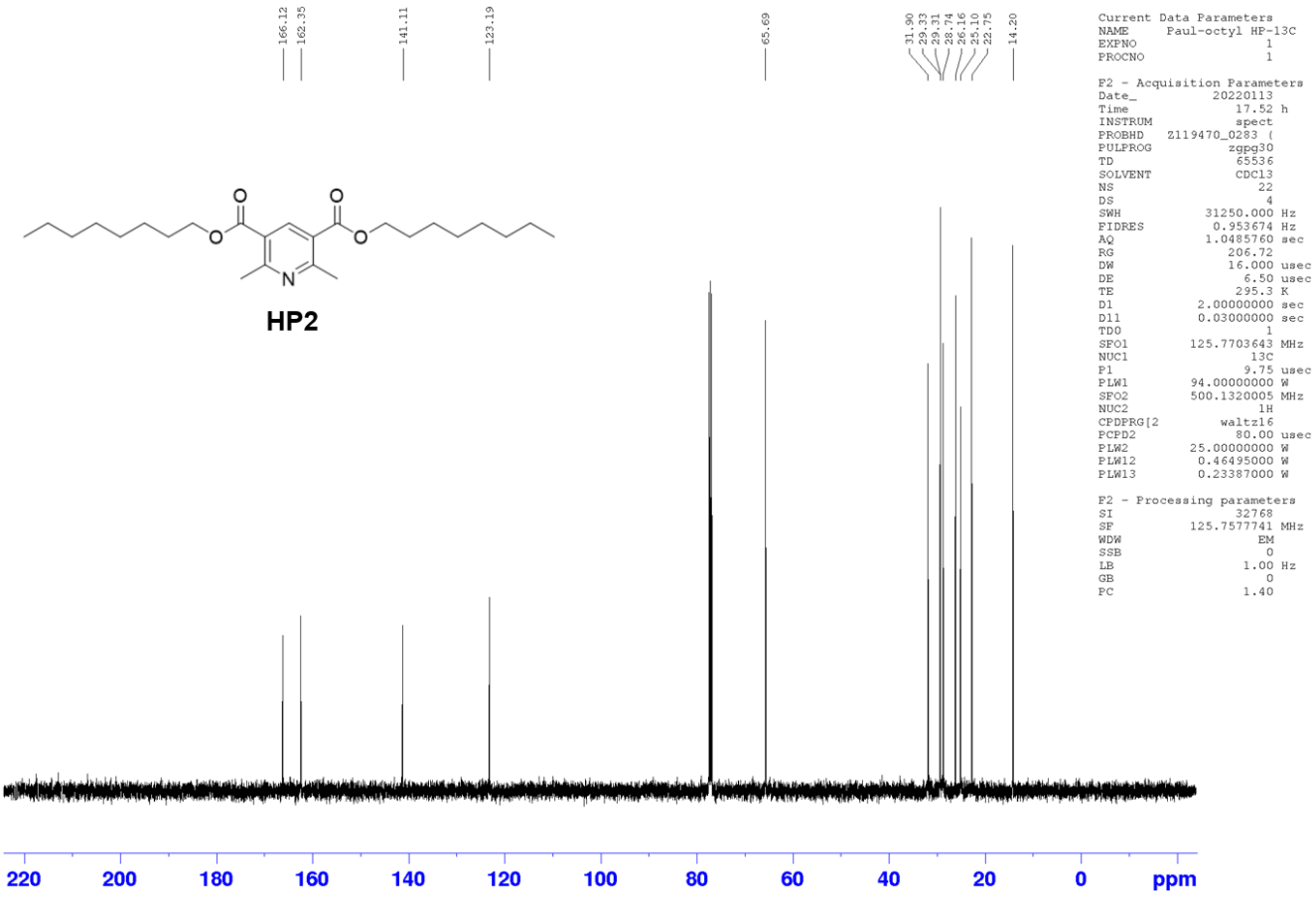


HE2

```
Current Data Parameters
NAME      Paul-octyl HEH-13C
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20220113
Time                 17.45 h
INSTRUM              spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    112
DS                      4
SWH            31250.000 Hz
FIDRES              0.953674 Hz
AQ                1.0485760 sec
RG                   206.72
DW                   16.000 usec
DE                    6.50 usec
TE                   295.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           125.7703643 MHz
NUC1                   13C
P1                    9.75 usec
PLW1           94.00000000 W
SFO2           500.1320005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                80.00 usec
PLW2           25.00000000 W
PLW12           0.46495000 W
PLW13           0.23387000 W

F2 - Processing parameters
SI                  32768
SF             125.7577731 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```




```
Current Data Parameters
NAME      Paul-octyl HP-1H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_             20220113
Time                17.49 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES             0.305176 Hz
AQ                 3.2767999 sec
RG                    30.85
DW                 50.000 usec
DE                  6.50 usec
TE                 295.1 K
D1              1.00000000 sec
TD0                    1
SFO1           500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1           25.00000000 W

F2 - Processing parameters
SI                 65536
SF             500.1300120 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME      Paul-octyl HP-13C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_             20220113
Time                17.52 h
INSTRUM             spect
PROBHD    Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    22
DS                     4
SWH            31250.000 Hz
FIDRES             0.953674 Hz
AQ                 1.0485760 sec
RG                   206.72
DW                 16.000 usec
DE                  6.50 usec
TE                 295.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1           125.7703643 MHz
NUC1                  13C
P1                  9.75 usec
PLW1           94.00000000 W
SFO2           500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           25.00000000 W
PLW12           0.46495000 W
PLW13           0.23387000 W

F2 - Processing parameters
SI                 32768
SF             125.7577741 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```