

Supporting Information

Exploiting Photooxygenations Mediated by Porphyrinoid Photocatalysts under Continuous Flow Conditions

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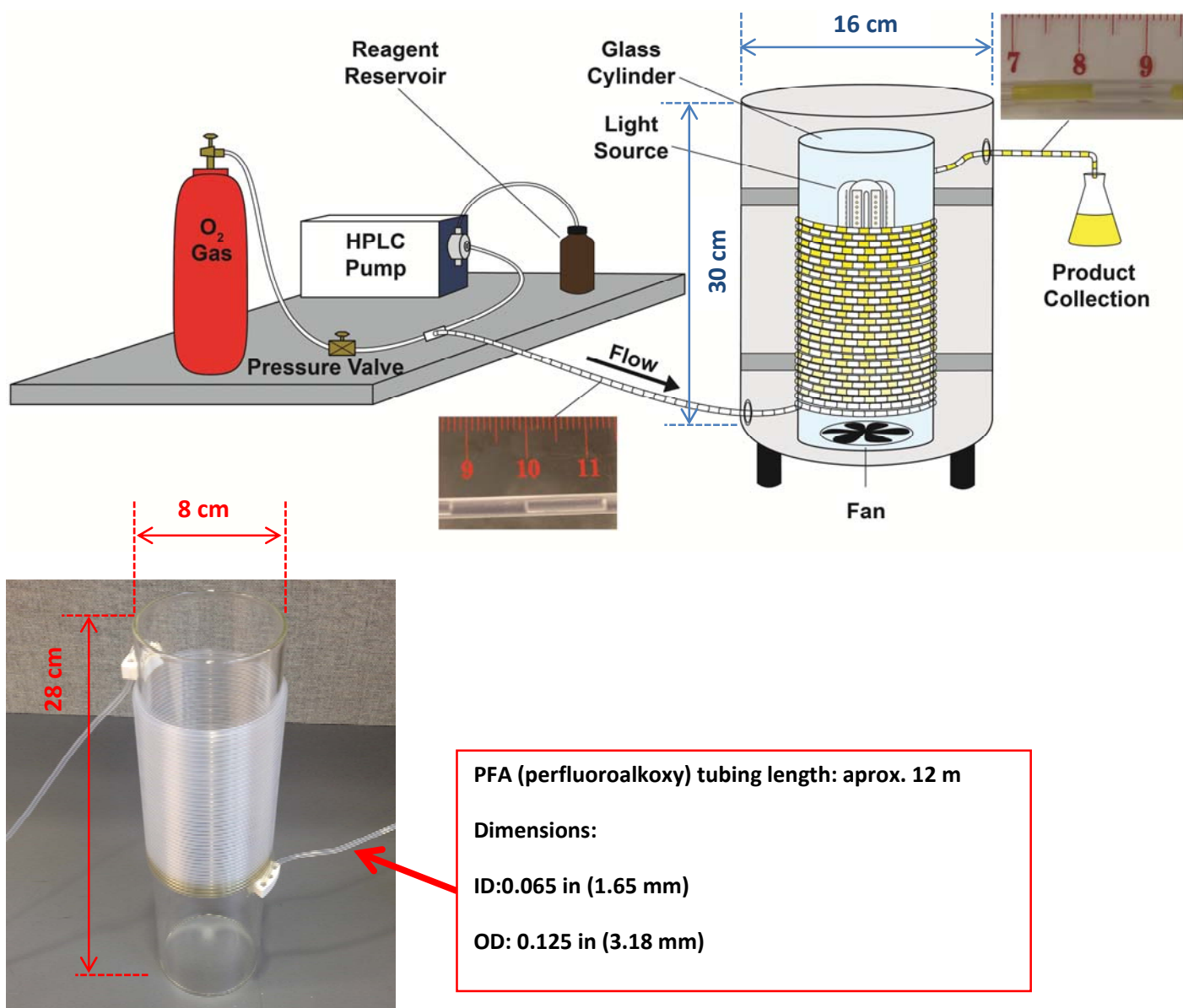
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LED Lamp



FLC Lamp



Figure S1: Detailed flow-chemistry setup.

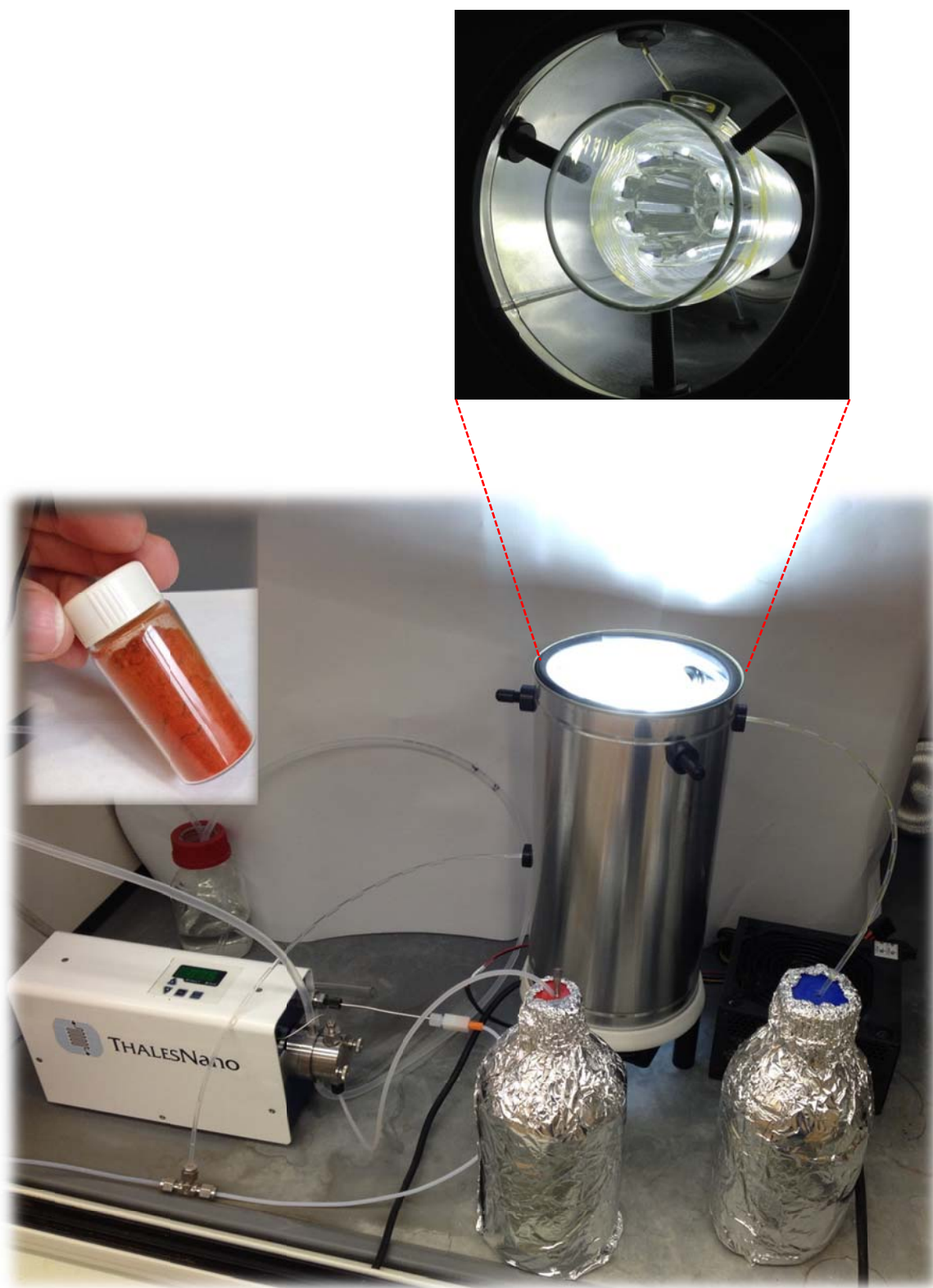


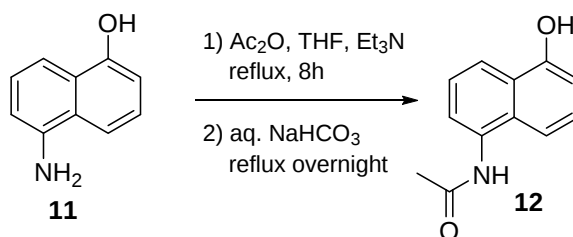
Figure S2: Actual flow-chemistry setup.



Figure S3: Batch reaction setup.

Experimental Procedures

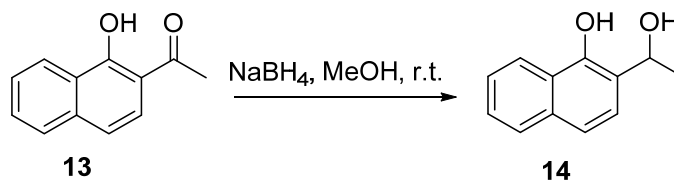
N-(5-hydroxynaphthalen-1-yl)acetamide (**12**):^{S1}



To a suspension of compound **12** (318.4 mg, 2.00 mmol) in dry THF (30 mL), acetic anhydride (4 eqv., 416 mg, 0.76 mL) was added and the reaction was maintained at room temperature for 5 min. After this period, Et₃N (6 eqv., 1.22g, 1.70 mL) was added and the reaction was then refluxed for 8 h. A saturated solution of NaHCO₃ (50 mL) was added and the reaction mixture refluxed overnight. The THF was evaporated and the pH was adjusted to 4 using concentrated HCl, and the desired reaction product was extracted with CH₂Cl₂ (2 x 100 mL). The solvent was distilled off and the product recrystallized from CH₂Cl₂ and MeOH. Yield: 240.5 mg (1.19 mmol, 60%). ¹H NMR (600 MHz, CDCl₃) δ (ppm): 2.16 (s, 3H); 6.89 (d, *J* = 7.4 Hz, 1H); 7.32 (t, *J* = 8.0 Hz, 1H); 7.39 (t, *J* = 7.4 Hz, 1H); 7.50 (d, *J* = 8.0 Hz, 1H); 7.65 (d, *J* = 7.4 Hz, 1H); 7.97 (d, *J* = 8.0 Hz, 1H); 9.81 (br.s, 1H); 10.18 (br.s, 1H). ¹³C NMR (150 MHz, CDCl₃) δ (ppm): 23.4; 108.1; 113.2; 119.1; 121.8; 124.0; 126.1; 129.1; 133.4; 153.3; 168.8.

^{S1} E. E. Coyle, K. Joyce, K. Nolan, M. Oelgemöller, *Green Chem.*, 2010, **12**, 1544.

2-(1-hydroxyethyl)naphthalen-1-ol (14**):**^{S2}



To a solution of compound **13** (1.87 g, 10.0 mmol) in MeOH (100 mL), NaBH₄ (0.38 g, 10 mmol) was added at room temperature. After 10 min the reaction was quenched by addition of 200 g of ice and HCl (1M) was added until the pH = 3. The reaction was filtered, washed with water, and dried yielding very pure crystals of compound **14** without further purification. Yield: 1.68 g (8.92 mmol, 89%). ¹H NMR (600 MHz, CDCl₃) δ (ppm): 1.61 (d, *J* = 6.6 Hz, 3H); 2.62 (br.s, 1H); 5.17 (q, *J* = 6.6 Hz, 1H); 6.99 (d, *J* = 8.3 Hz, 1H); 7.30 (d, *J* = 8.3 Hz, 1H); 7.44-7.47 (m, 2H); 7.72-7.76 (m, 1H); 8.24-8.27 (m, 1H); 8.89 (br.s, 1H). ¹³C NMR (150 MHz, CDCl₃) δ (ppm): 23.8; 72.7; 119.2; 120.7; 122.0; 124.3; 125.2; 125.4; 126.2; 127.2; 133.8; 151.0.

^{S2} A. Kisić, M. Stephan, B. Mohara, *Adv. Synth. Catal.* 2014, **356**, 3193.

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Spectrum Offset (Hz)	15084.1045	Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59
				Temperature (degree C)	22.298

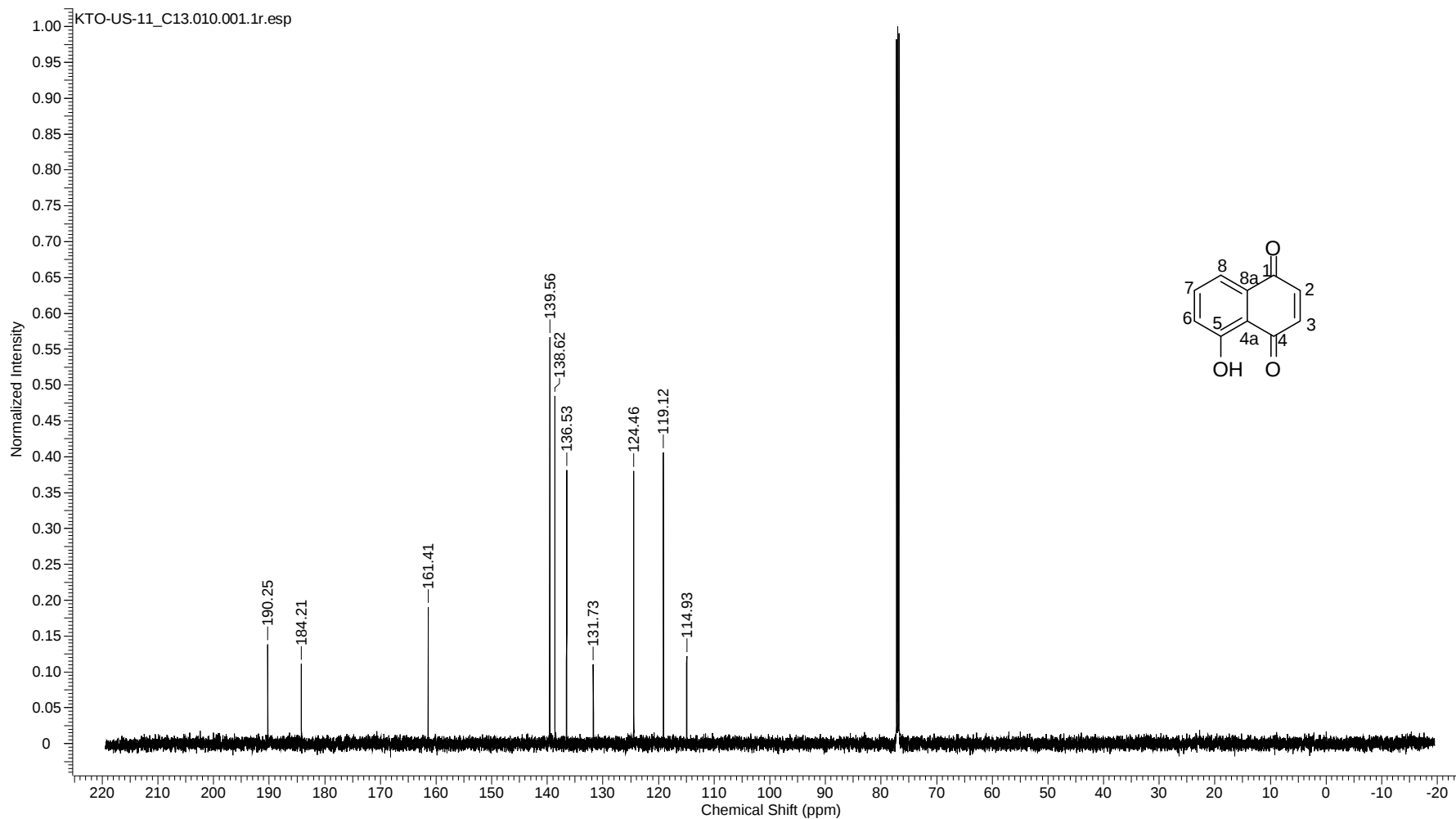


Figure S5

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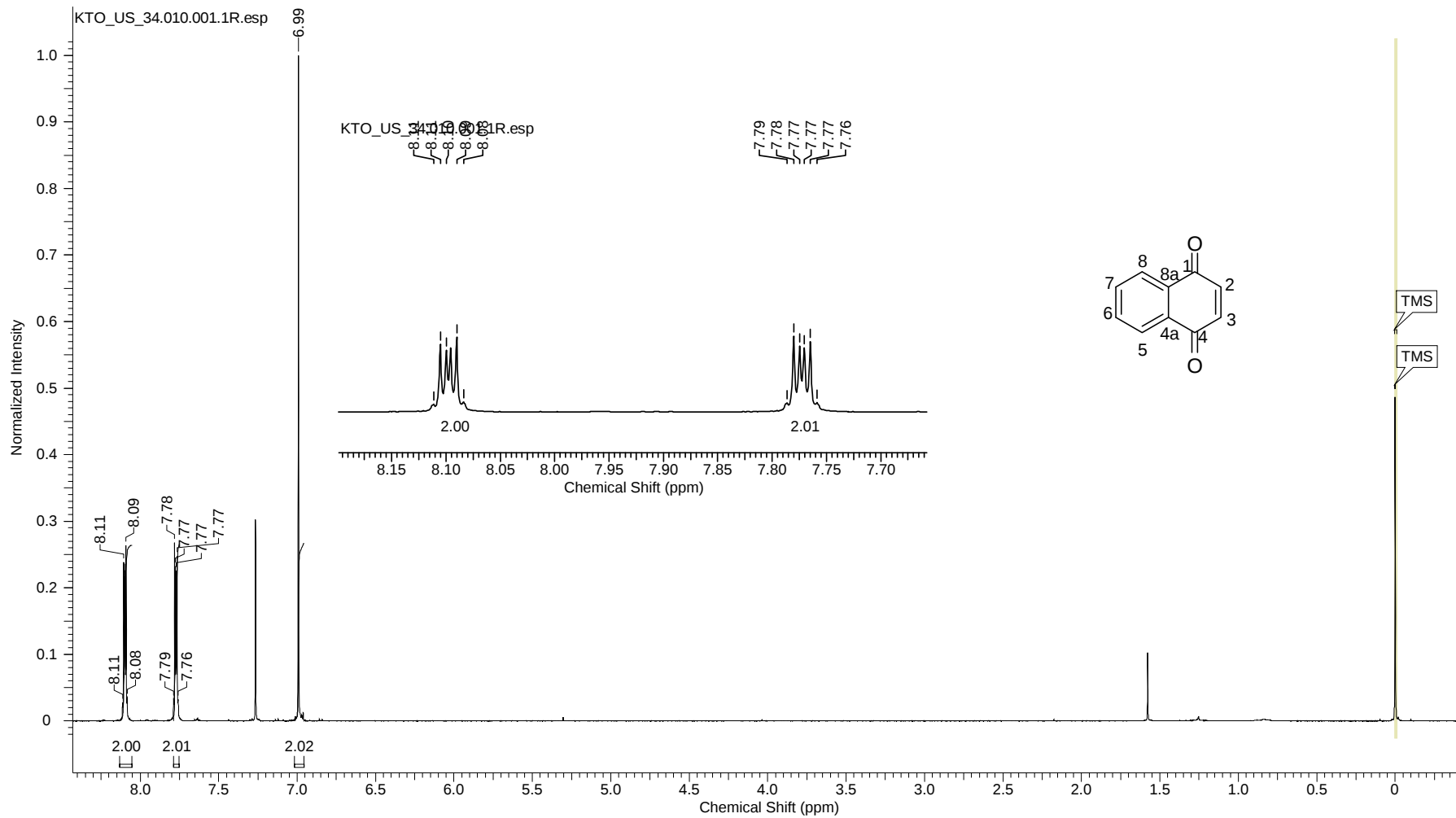


Figure S6

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Original Points Count	32768	Owner	mcquade	Points Count	32768	Pulse Sequence	zgpg30
Receiver Gain	203.00	SW(cyclical) (Hz)	36057.69	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	15087.4053	Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59	Temperature (degree C)	23.987

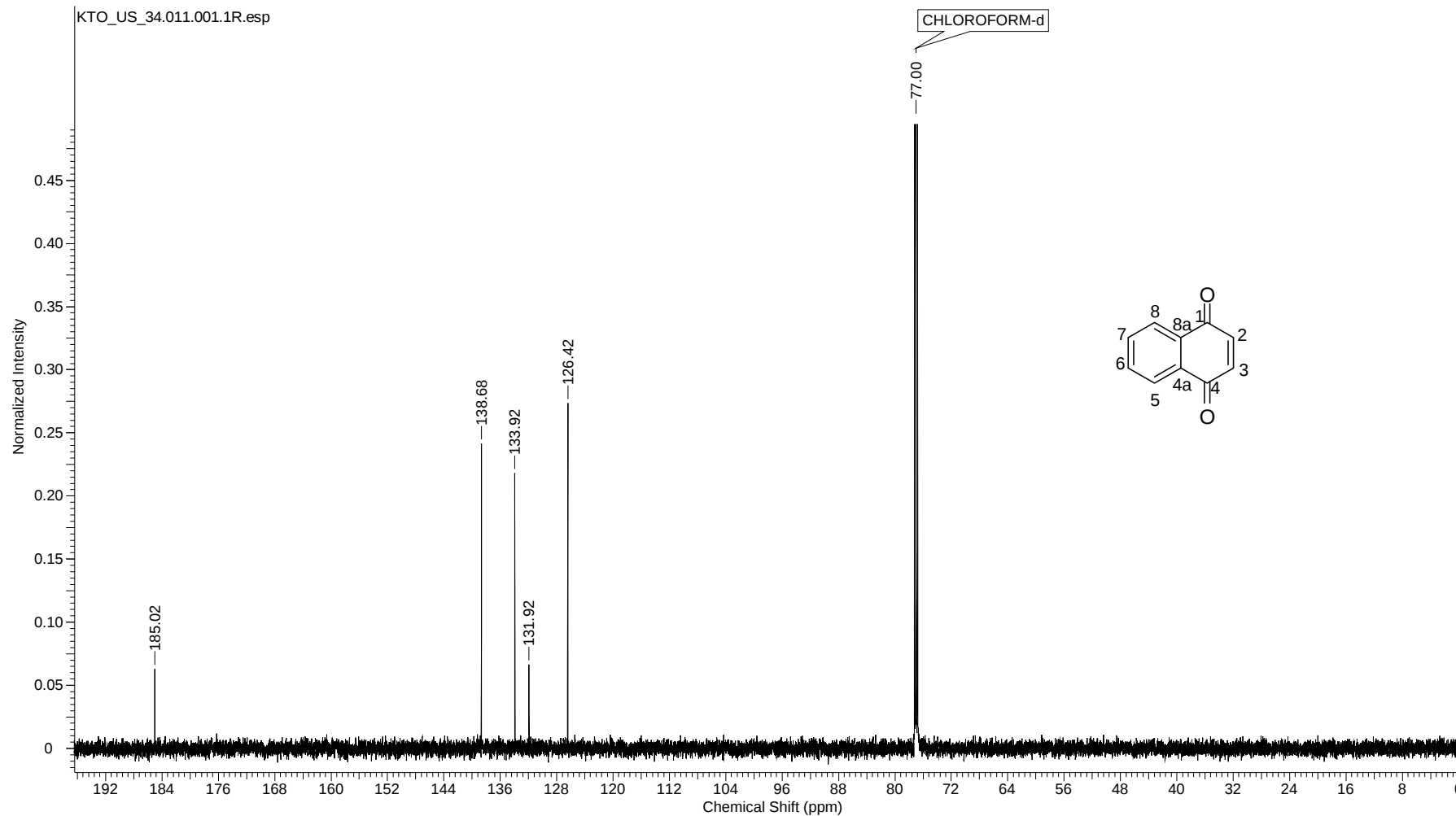


Figure S7

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Original Points Count	32768	Owner	mcquade	Points Count	32768	Pulse Sequence	zg30
Receiver Gain	203.00	SW(cyclical) (Hz)	12335.53	Solvent	DMSO-d6	Spectrum Offset (Hz)	3699.7366
Spectrum Type	STANDARD	Sweep Width (Hz)	12335.15	Temperature (degree C)	20.006		

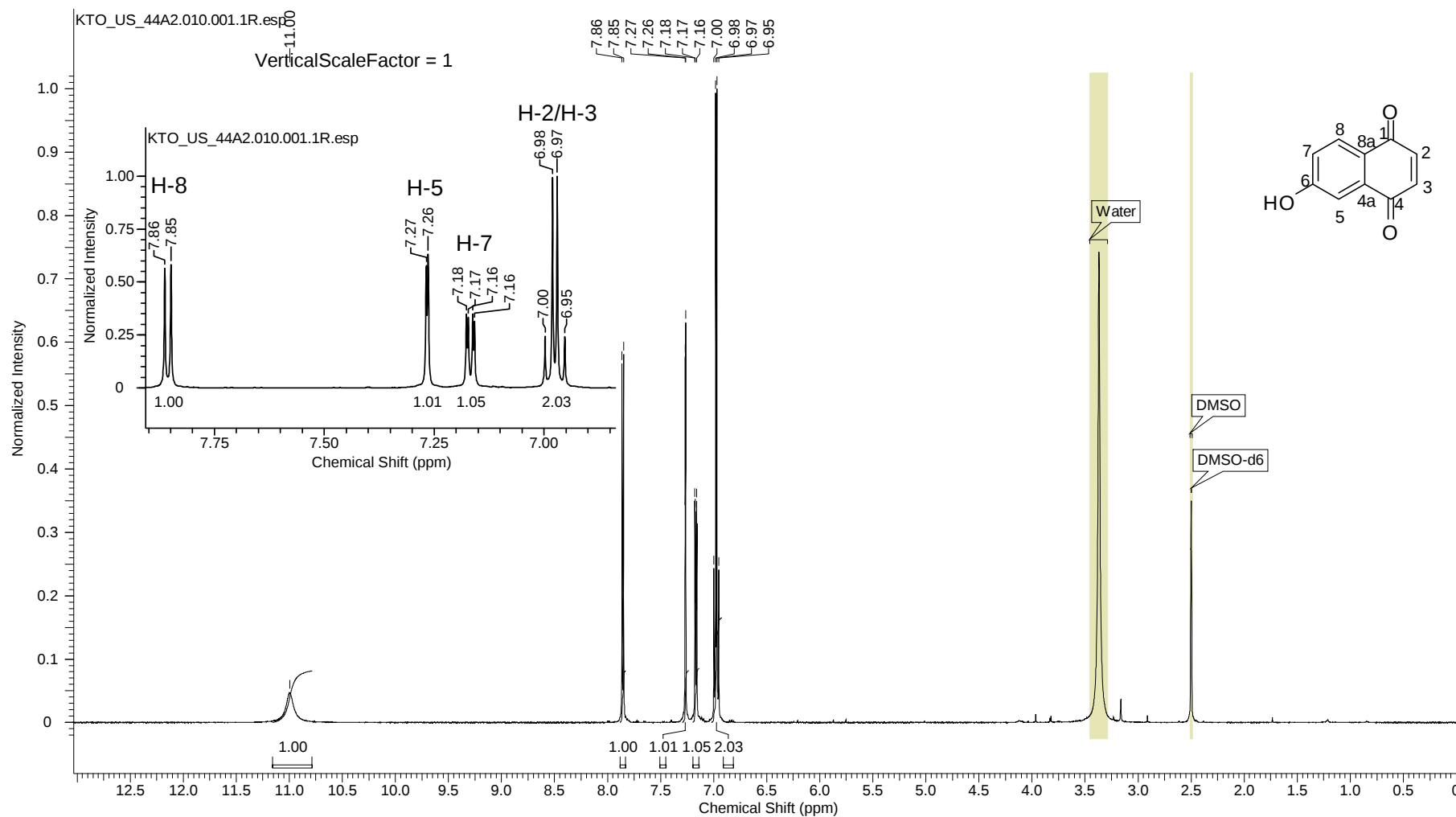


Figure S8

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Original Points Count	32768	Owner	mcquade	Points Count	32768	Pulse Sequence	zgpg30
Receiver Gain	203.00	SW(cyclical) (Hz)	36057.69	Solvent	DMSO-d6	Spectrum Offset (Hz)	15017.8604
Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59	Temperature (degree C)	23.867		

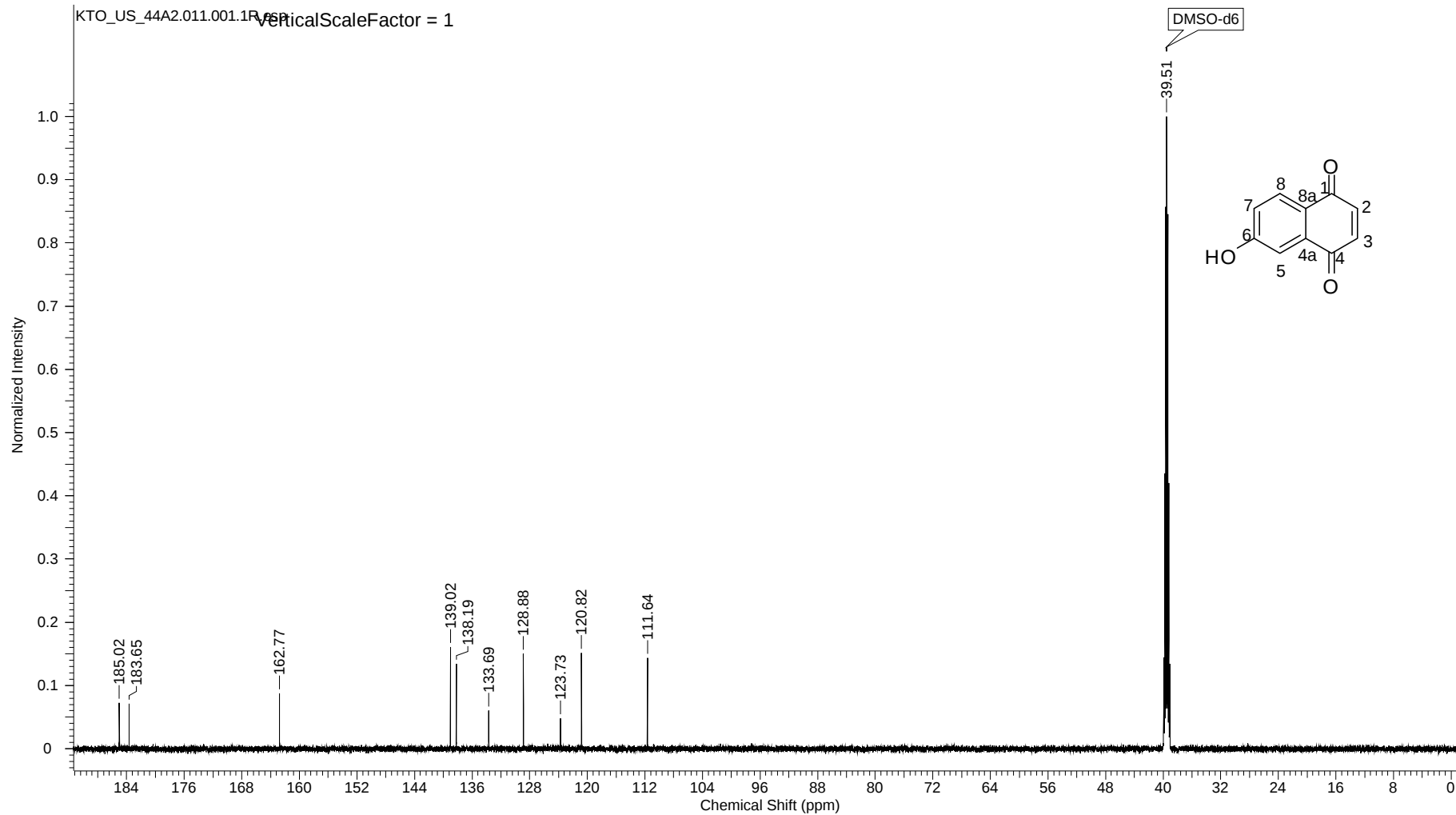


Figure S9

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Frequency (MHz)	150.90	Nucleus	13C	Number of Transients	1024	Origin spect
Owner	mcquade	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain 203.00
Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	15086.3047	SW(cyclical) (Hz) 36057.69
Temperature (degree C)	24.591					Sweep Width (Hz) 36056.59

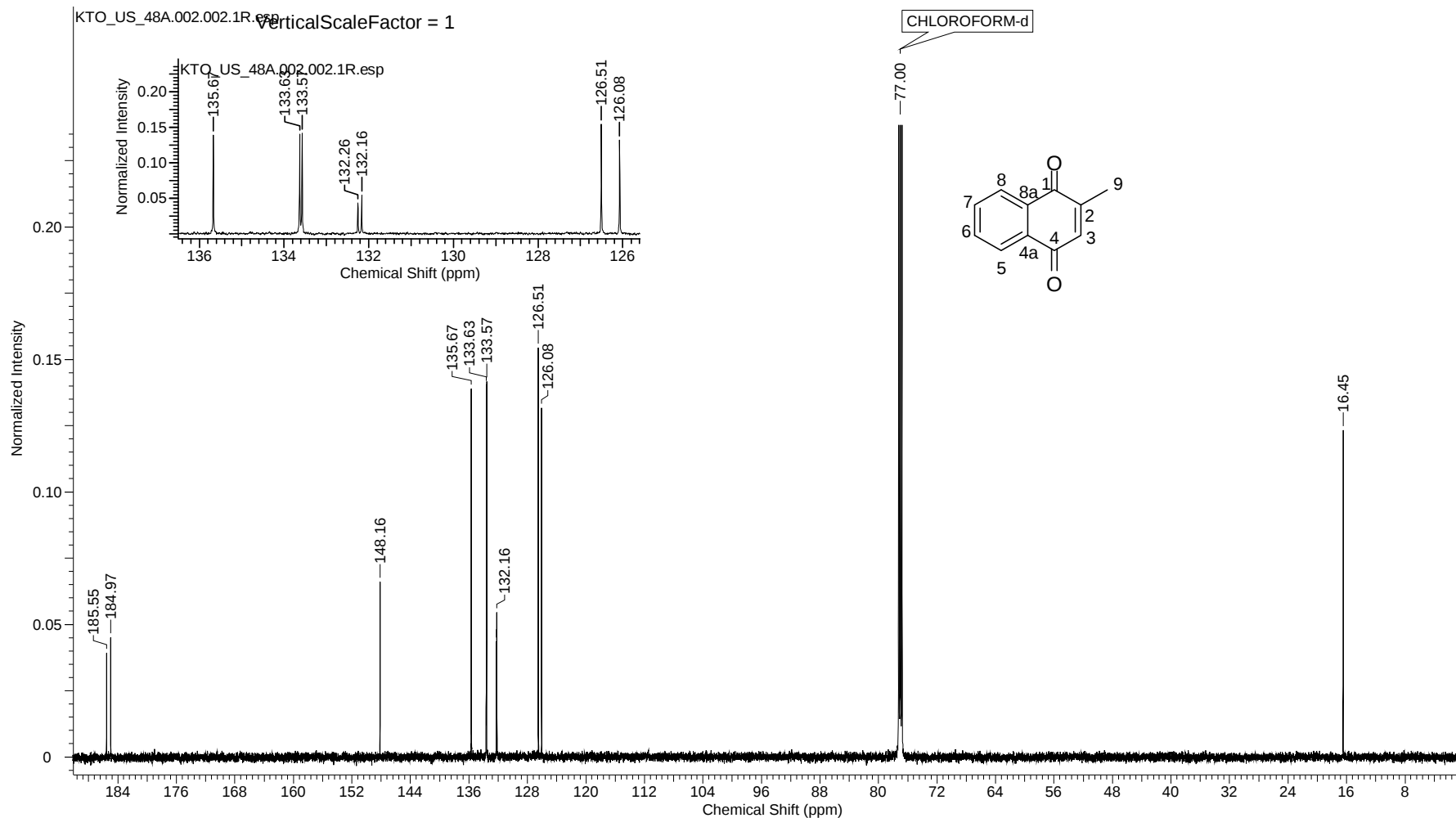


Figure S11

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Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	16	Origin spect
Owner	mcquade	Points Count	32768	Pulse Sequence	zg30	Receiver Gain 203.00
Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	3692.7903	SW(cyclical) (Hz) 12335.53
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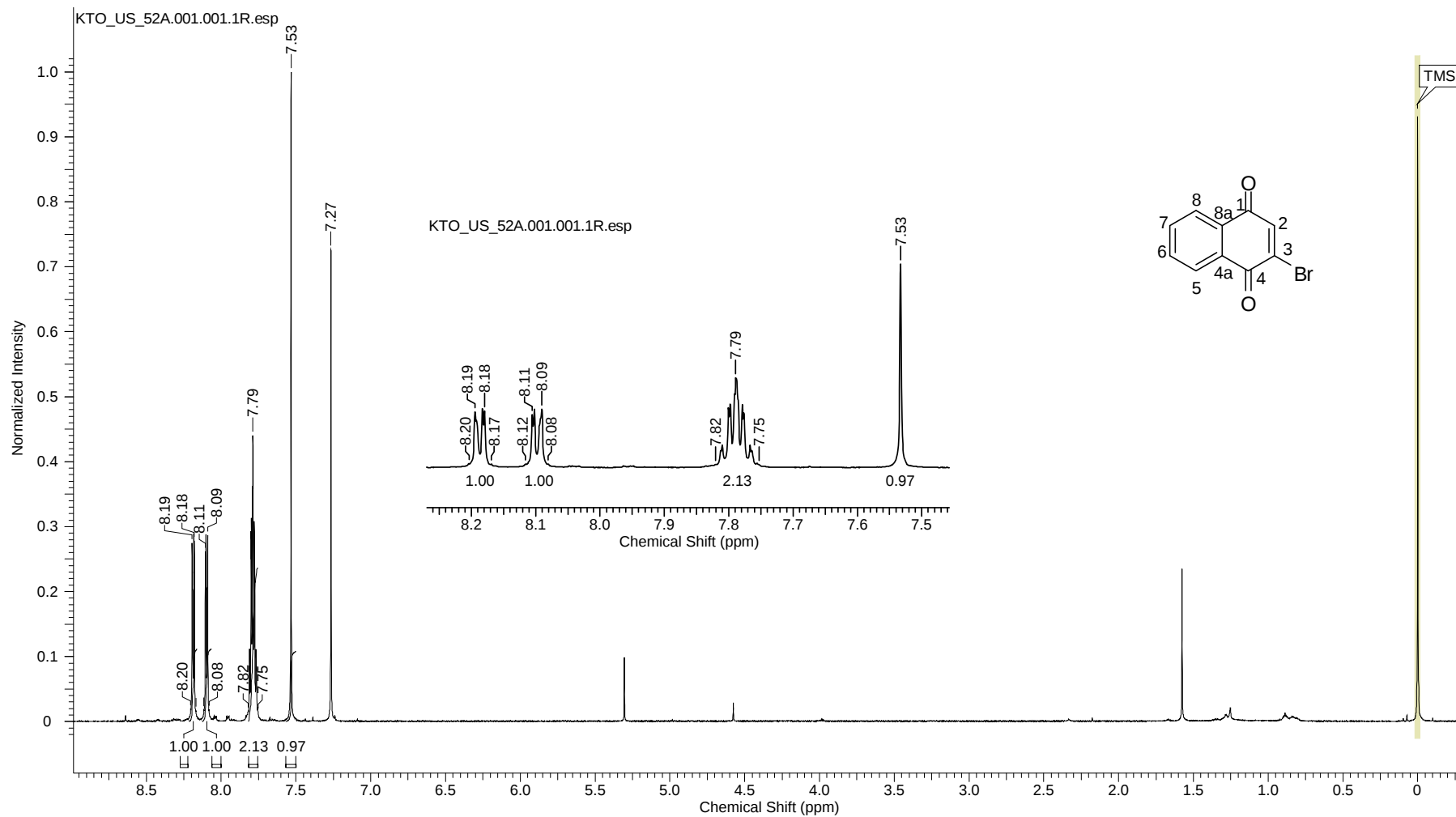


Figure S12

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Date Stamp	14 Sep 2015 09:54:08	File Name	C:\USERS\SONY\DESKTOP\NMR US\KTOUS-52-A\11\PDATA\11\1R		
Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	512
Original Points Count	32768	Owner	mcquade	Points Count	32768
Receiver Gain	203.00	SW(cyclical) (Hz)	36057.69	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15086.3047	Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59
				Temperature (degree C)	23.987

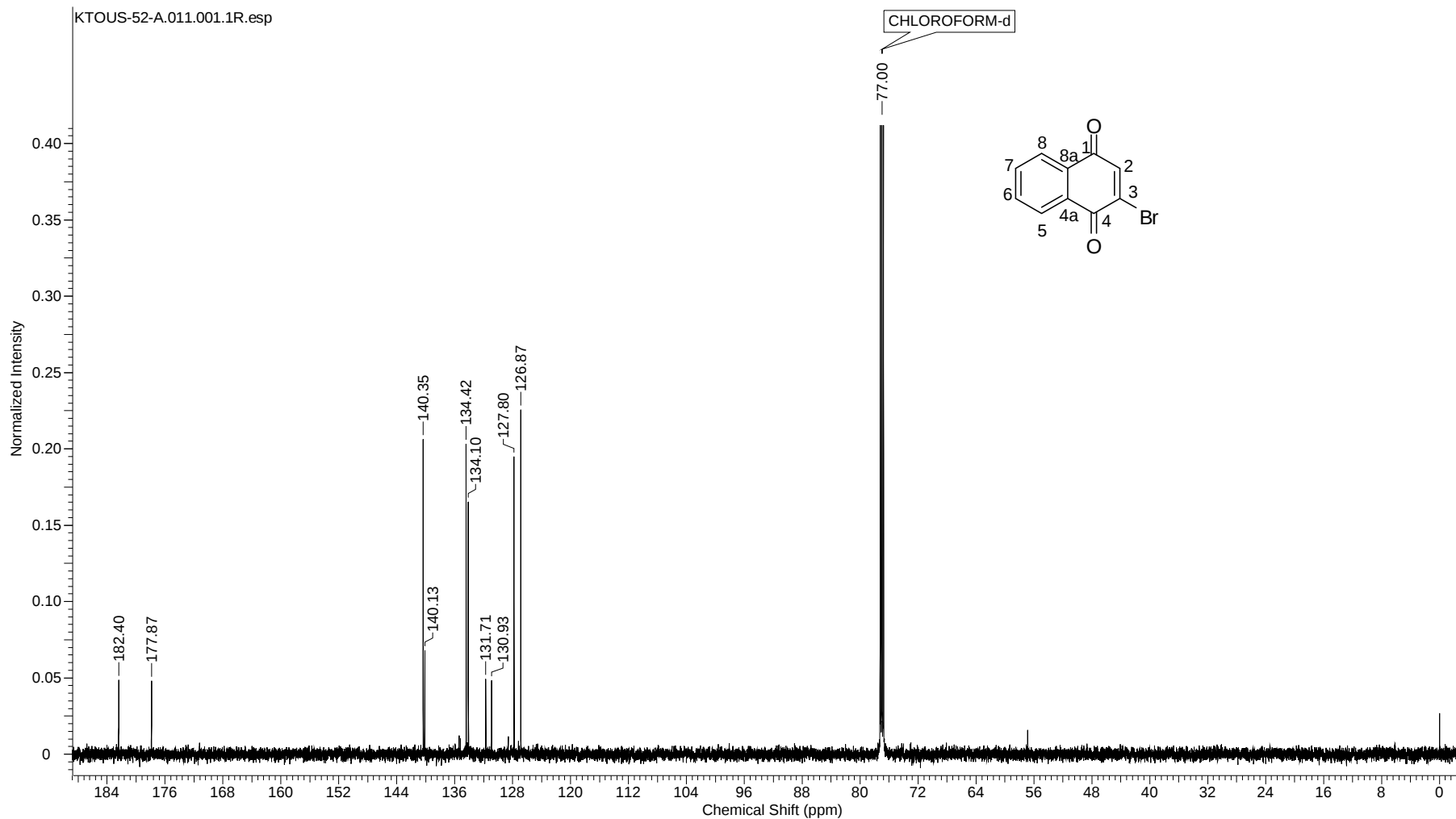


Figure S13

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Owner	mcquade	Points Count	32768	Pulse Sequence	zg30	Receiver Gain	203.00
Solvent	DMSO-d6	Spectrum Offset (Hz)	3700.1130	Spectrum Type	STANDARD	SW(cyclical) (Hz)	12335.53
						Temperature (degree C)	20.006

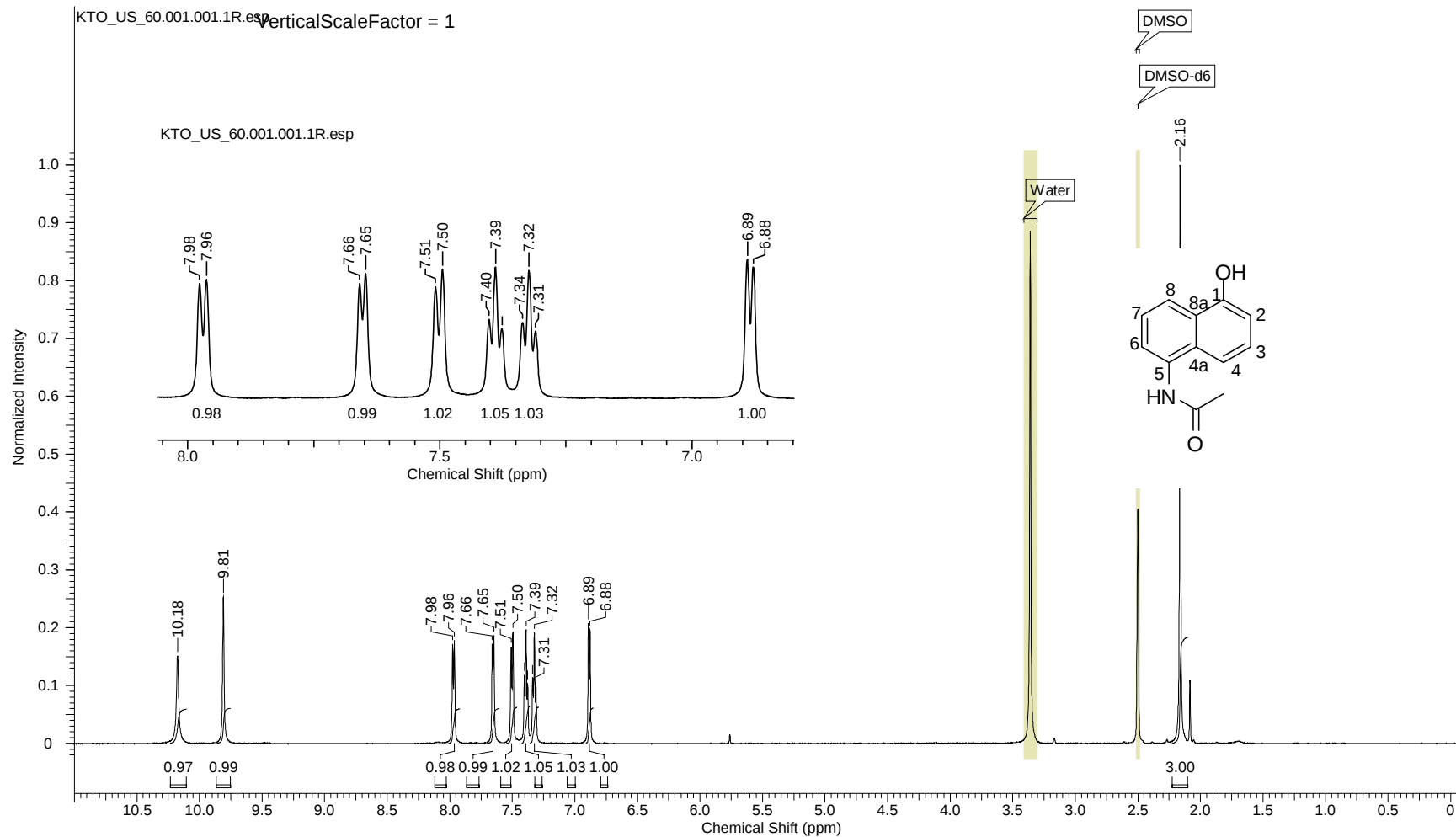


Figure S14

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Owner	mcquade	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain 203.00
Solvent	DMSO-d6	Spectrum Offset (Hz)	15015.6563	Spectrum Type	STANDARD	Sweep Width (Hz) 36056.59
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						Temperature (degree C) 21.333

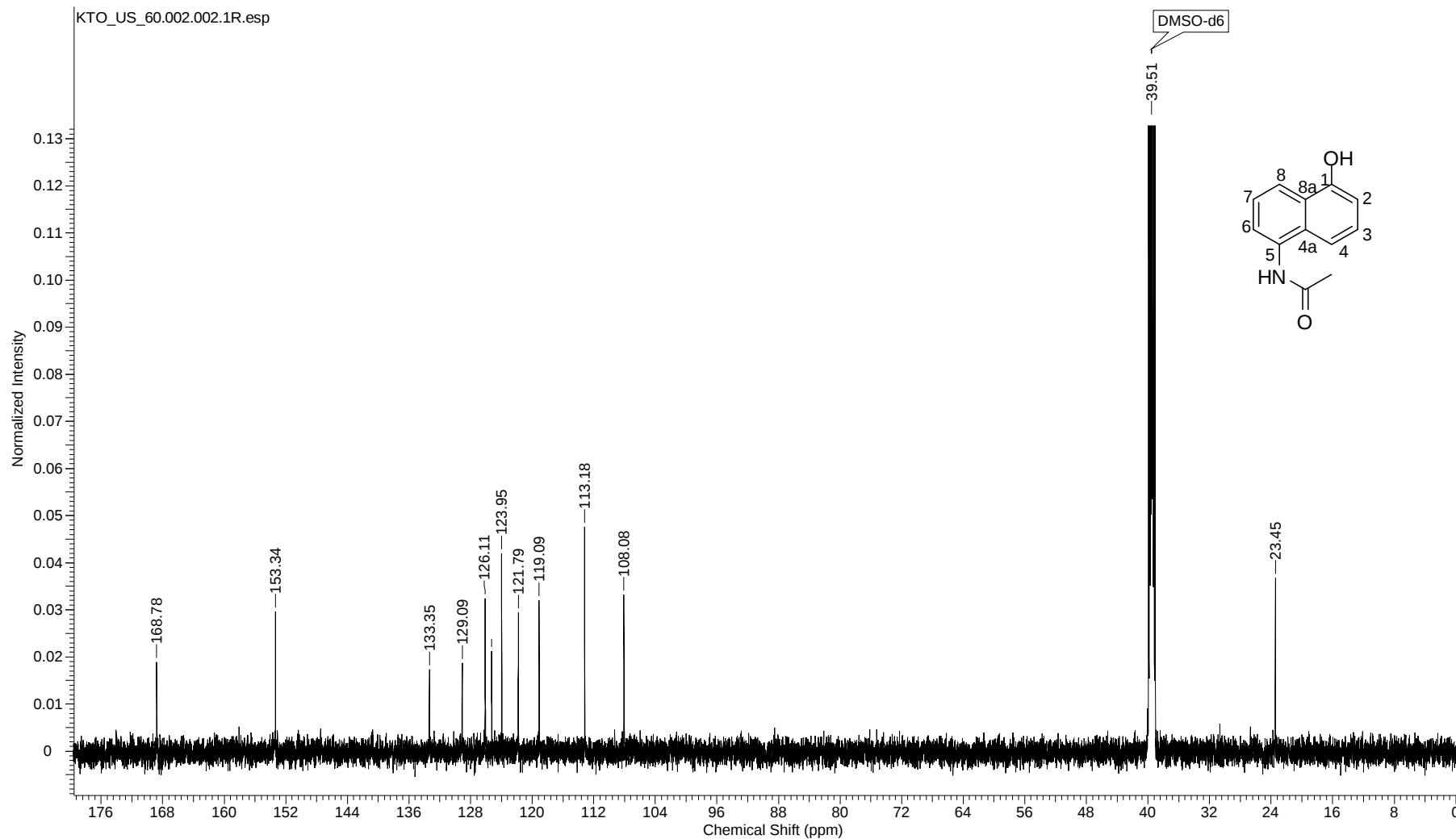


Figure S15

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Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	16
Original Points Count	32768	Owner	mcquade	Points Count	32768
Receiver Gain	203.00	SW(cyclical) (Hz)	12335.53	Solvent	CHLOROFORM-d
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				Temperature (degree C)	20.127

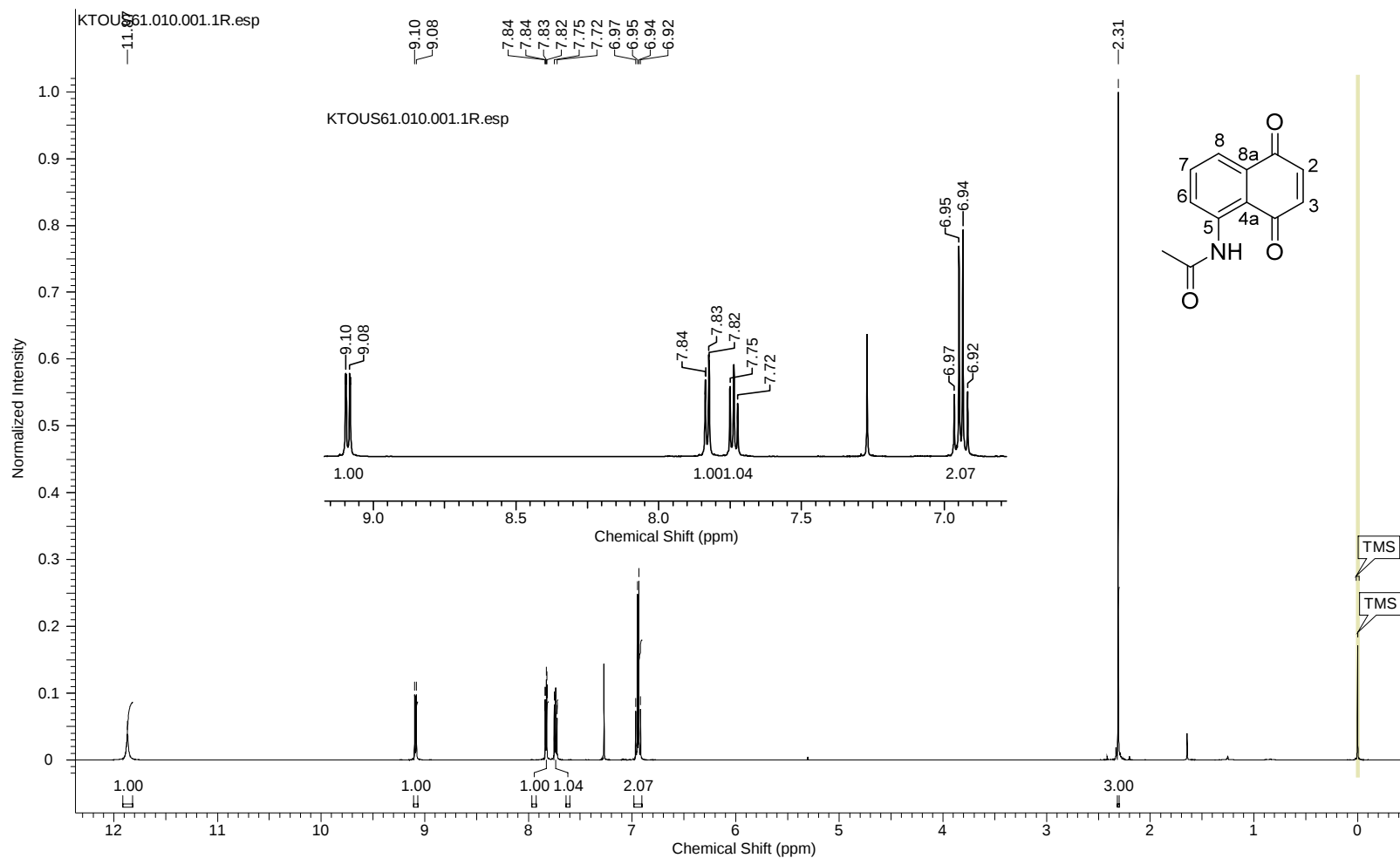


Figure S16

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Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	512
Original Points Count	32768	Owner	mcquade	Points Count	32768
Receiver Gain	203.00	SW(cyclical) (Hz)	36057.69	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15086.3047	Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59
				Temperature (degree C)	21.092

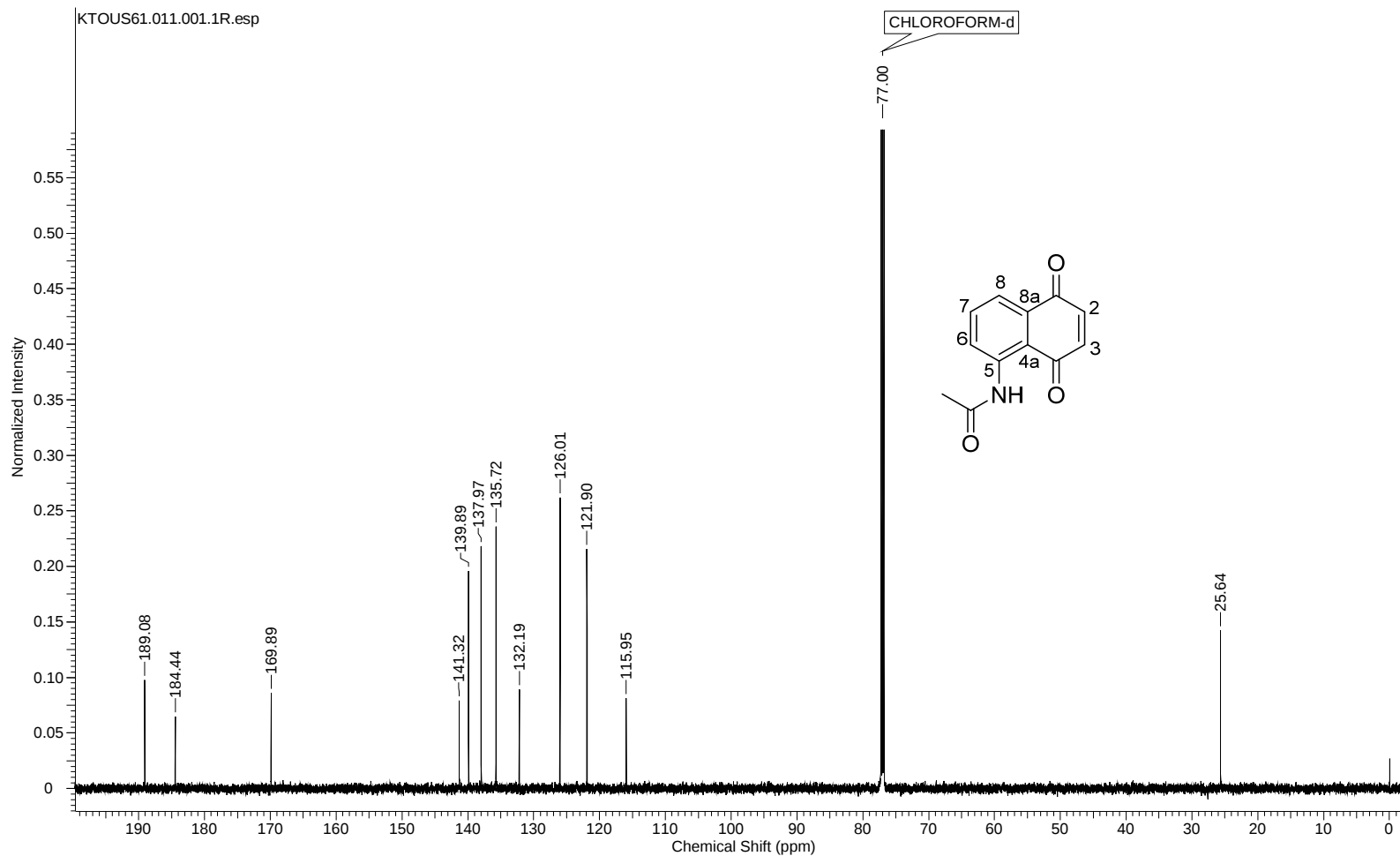


Figure S17

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				Sweep Width (Hz)	12335.15
				Temperature (degree C)	19.886

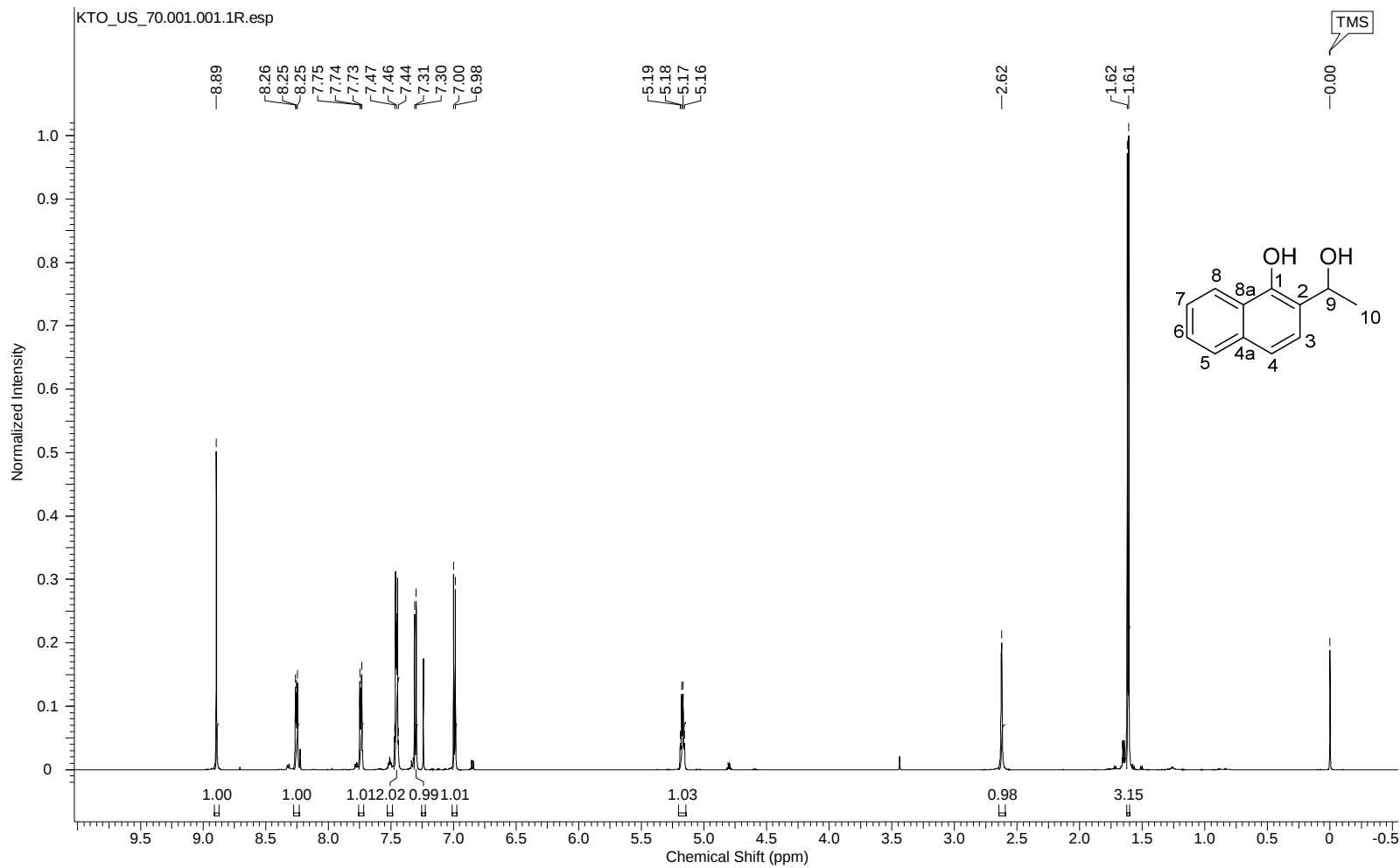


Figure S18

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				Temperature (degree C)	21.213

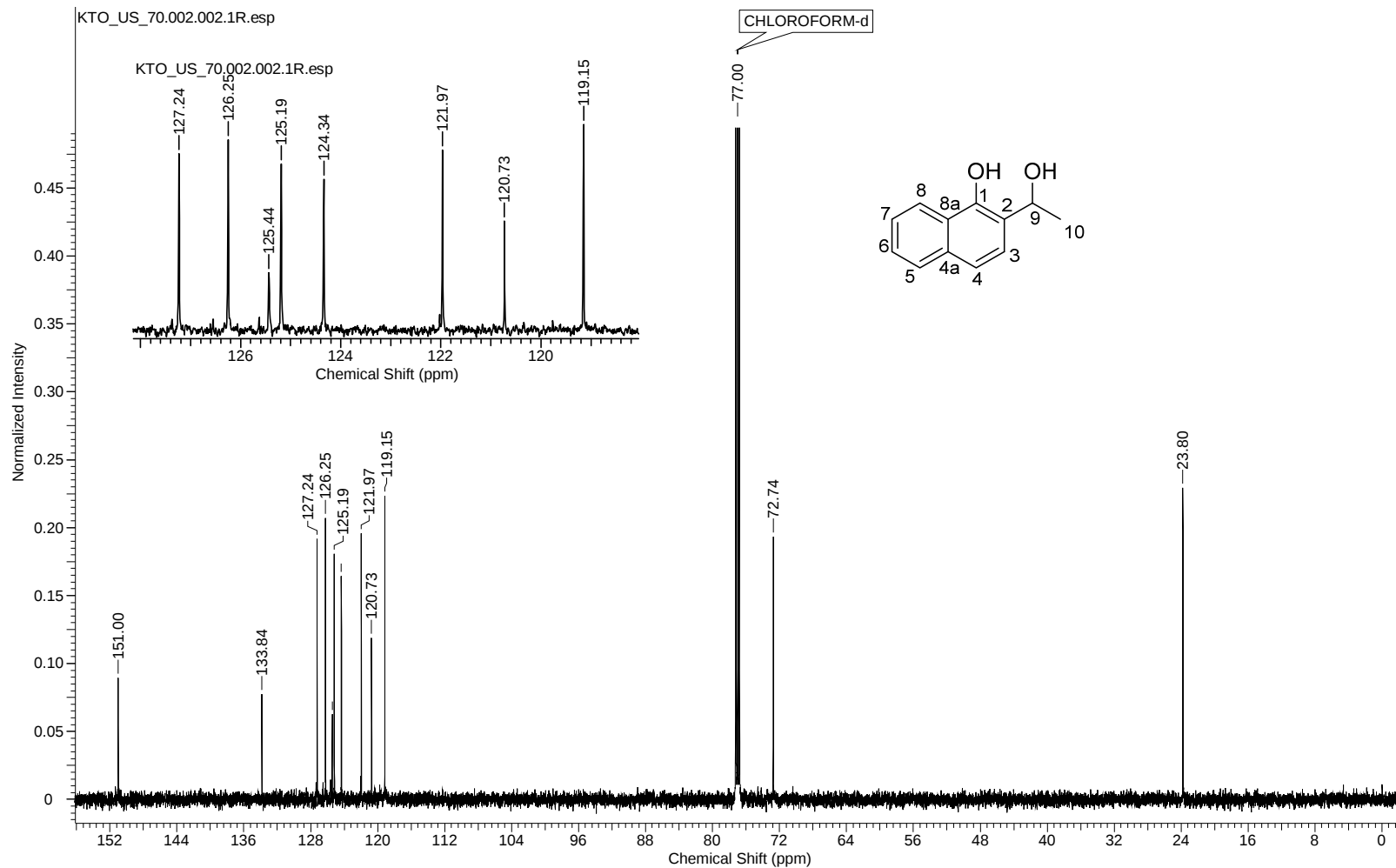


Figure S19

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Original Points Count	32768	Owner	mcquade	Points Count	32768	Pulse Sequence	zg30
Receiver Gain	203.00	SW(cyclical) (Hz)	12335.53	Solvent	CHLOROFORM-d		
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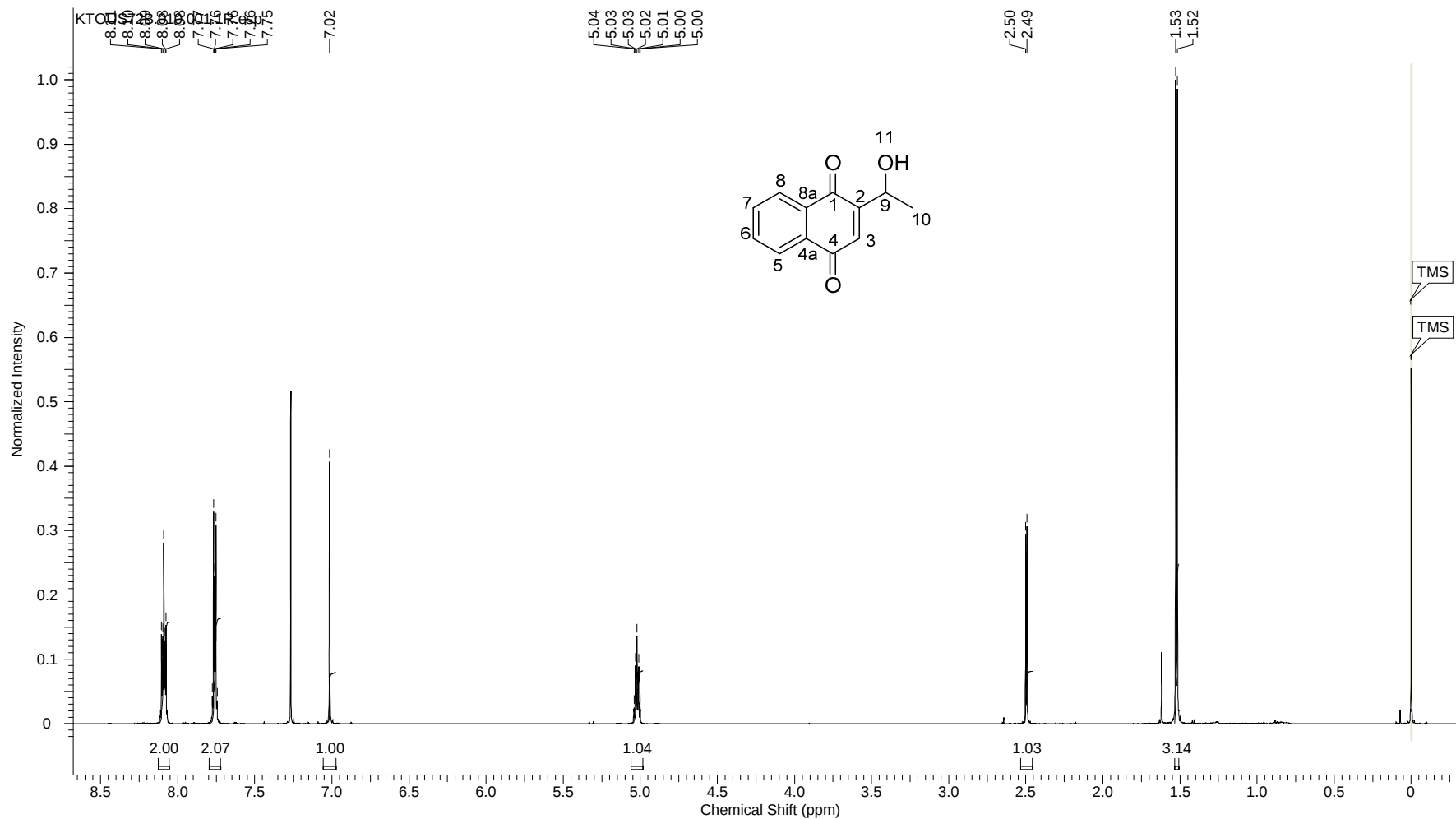


Figure S20

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Nucleus	¹³ C	Number of Transients	256	Origin	spect
Owner	mcquade	Points Count	32768	Pulse Sequence	zgpg30
SW(cyclical) (Hz)	36057.69	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15086.3047
Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59	Temperature (degree C)	23.867

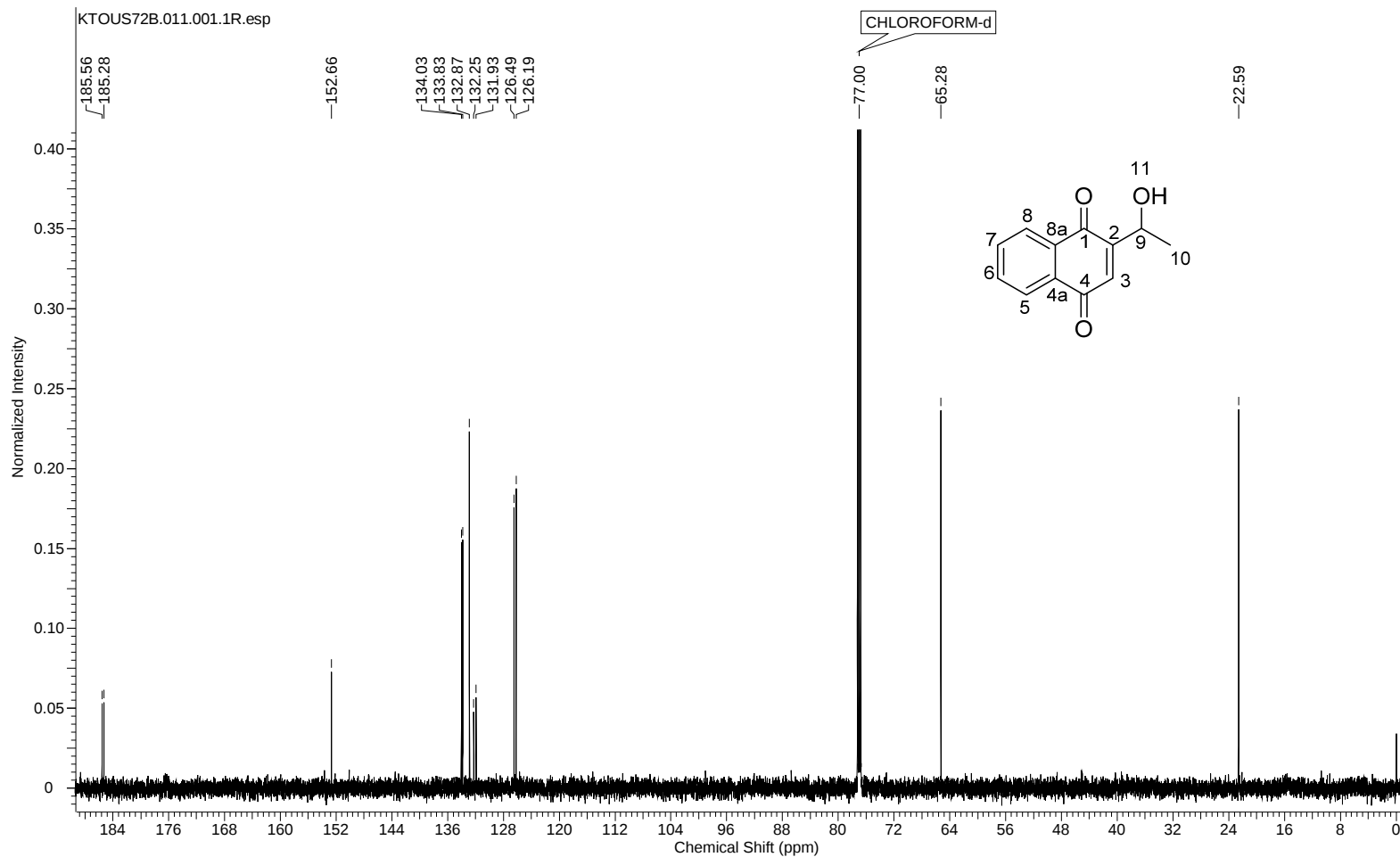


Figure S21