

Return of the lysergamides. Part VI: Analytical and behavioural characterization of 1-cyclopropanoyl-*D*-lysergic acid diethylamide (1CP-LSD)

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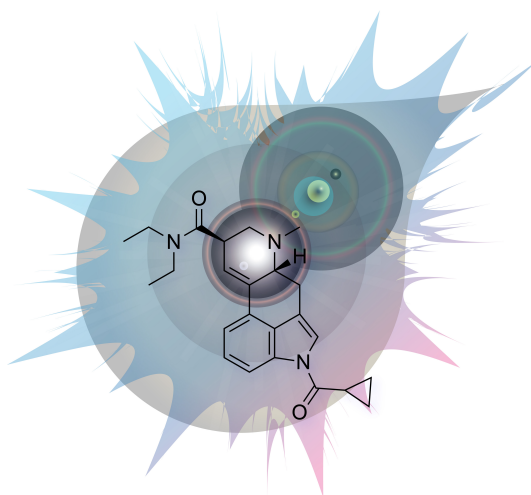
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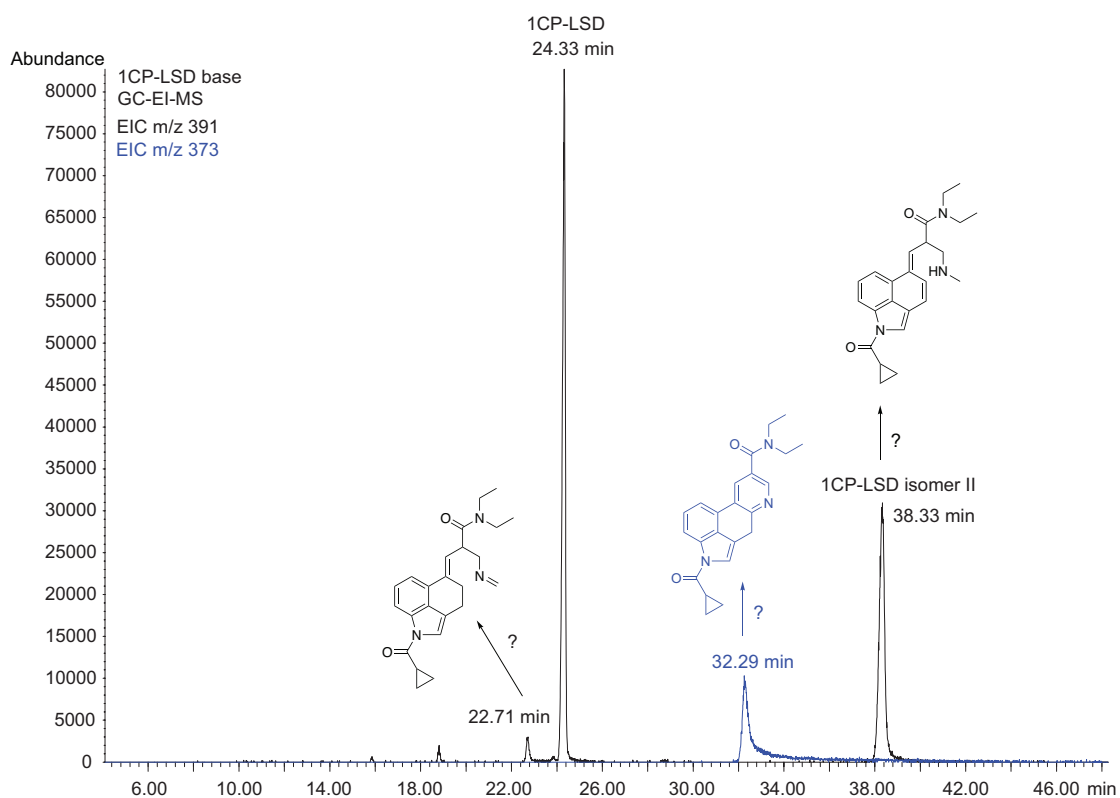
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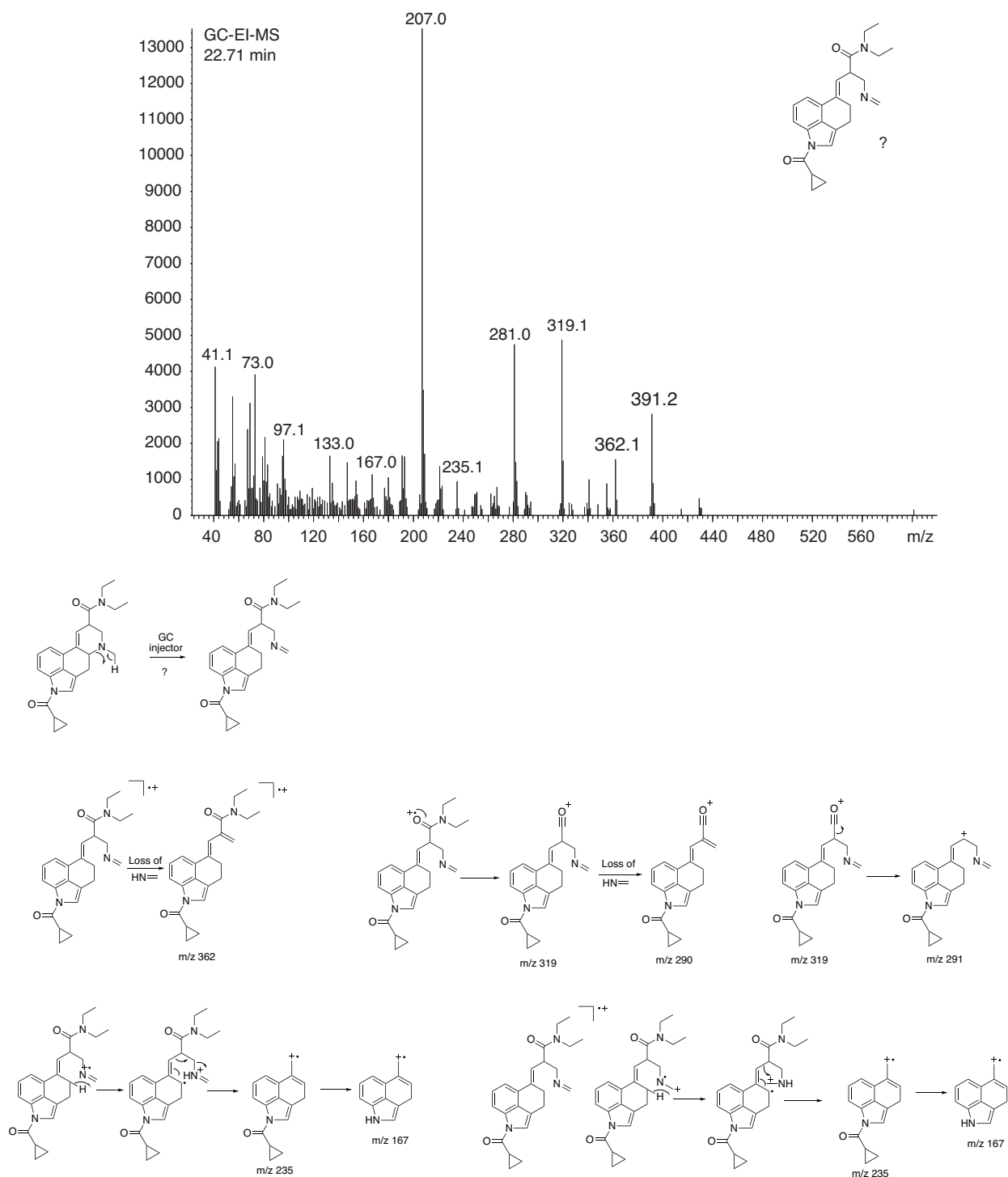
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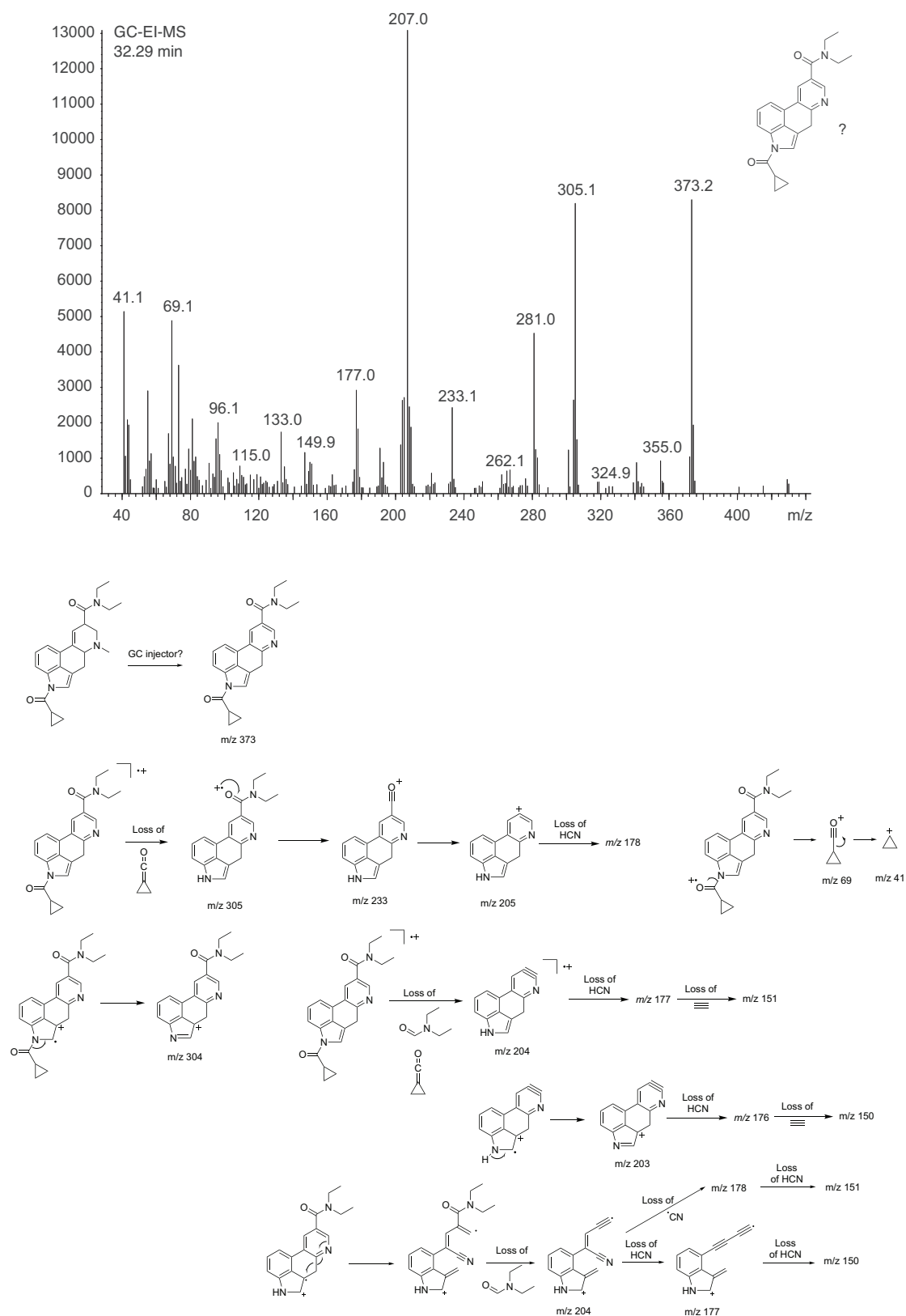


GC-induced formation of degradation products (GC-MS method 1; merged EIC traces)
(EI mass spectra next page; mass spectrum of 1CP-LSD in main text)

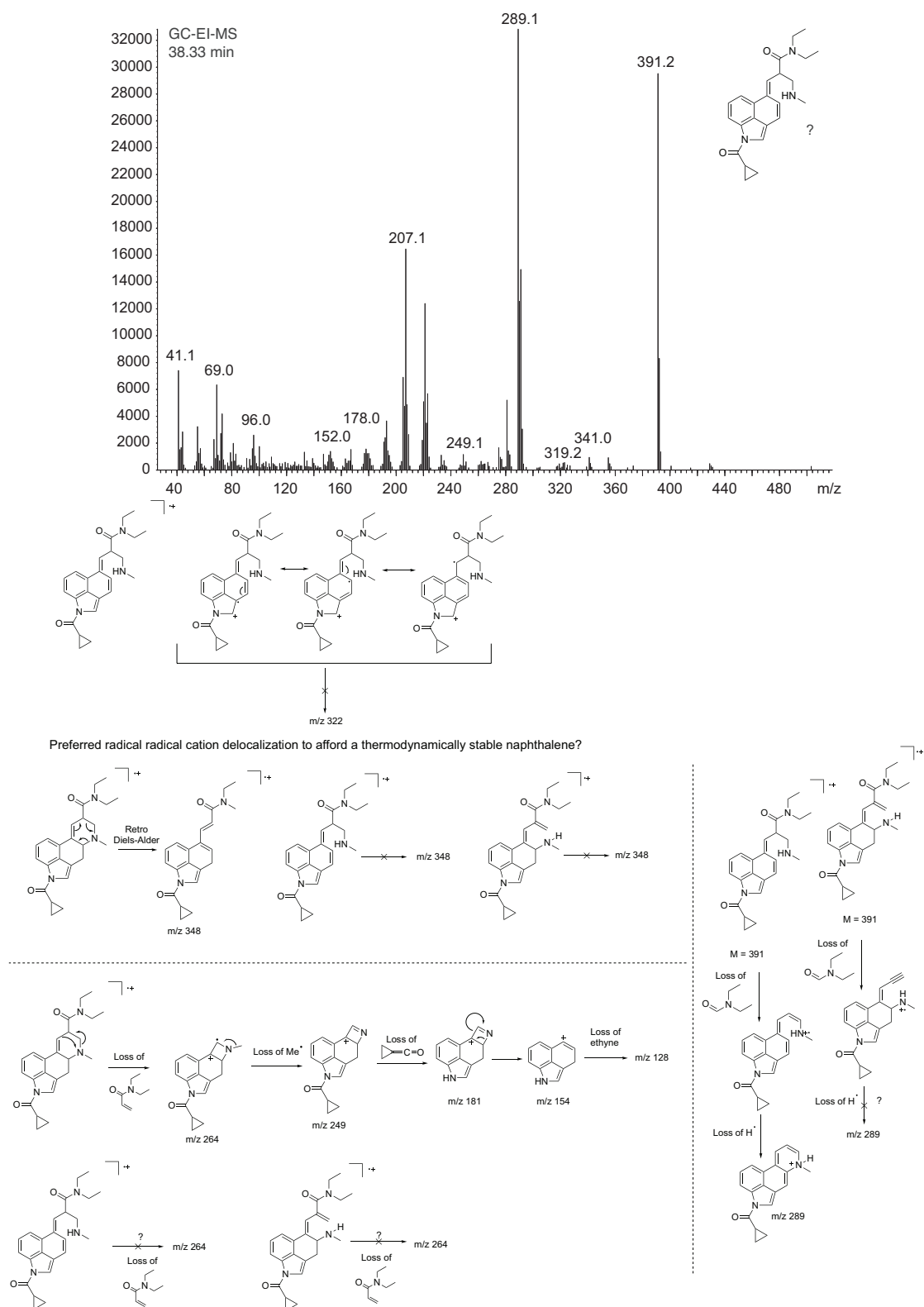
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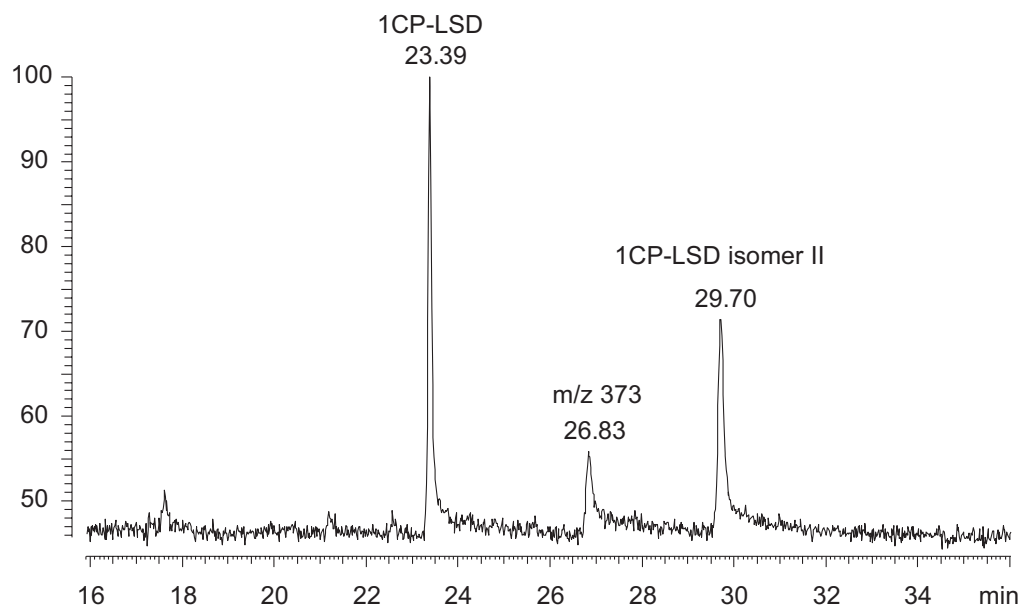


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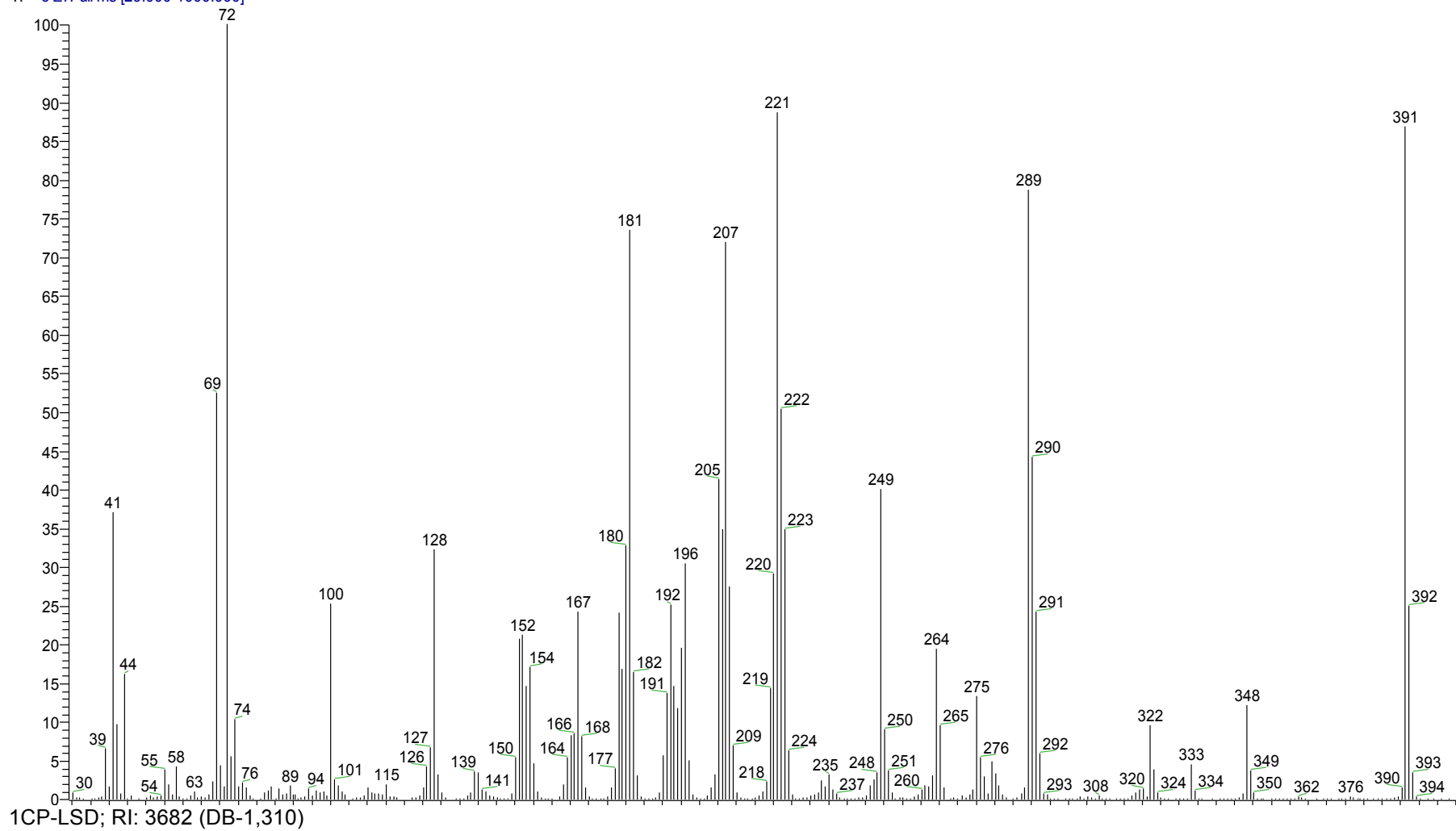
GC-induced formation of degradation products (GC-EI-MS method 2)

Electron ionization (EI) mass spectra (70 eV) were recorded using a Finnigan TSQ 8000 EVO triple stage quadrupole mass spectrometer coupled to a gas chromatograph (Trace 1310, Thermo Electron, Dreieich, Germany). Sample introduction was carried out using a Triplus RSH autosampler. The emission current was 200 μA and the scan time was 1 s spanning a scan range between m/z 29–600. The ion source temperature was maintained at 220°C. Samples were introduced via gas chromatography with splitless injection using a fused silica capillary DB-1 column (30 m \times 0.25 mm, film thickness 0.25 μm). The temperature program consisted of an initial temperature of 80°C, held for 2 min, followed by a ramp to 310°C at 20°C/min. The final temperature was held for 23 min. The injector temperature and the transfer line temperature were 280°C and 300°C and the carrier gas was helium in constant flow mode at a flow rate of 1.2 mL/min. Approximately 2 mg were dissolved in 1.5 mL methanol. For analysis, 1 μL of sample solution was injected into the GC-MS system. Kovats retention indices (RI) were calculated from measurement of an *n*-alkane mixture analyzed with the above-mentioned temperature program.

Samples were also analyzed using a GC-solid phase-IR-system (GC-sIR) that consisted of an Agilent GC 7890B (Waldbronn, Germany) with probe sampler Agilent G4567A and a DiscovIR-GC™ (Spectra Analysis, Marlborough, MA, USA). The column eluent was cryogenically accumulated on a spirally rotating ZnSe disk cooled by liquid nitrogen. IR spectra were recorded through the IR-transparent ZnSe disk using a nitrogen-cooled MCT detector. GC parameters: injection in splitless mode with the injection port temperature set at 240°C and a DB-1 fused silica capillary column (30 m \times 0.32 mm i.d., 0.25 μm film thickness). The carrier gas was helium with a flow rate of 2.5 mL/min and the oven temperature program was as follows: 80°C for 2 min, ramped to 300°C at 20 °C/min, and held at for 22 min. The transfer line was heated at 280°C. Infrared conditions: oven temperature, restrictor temperature, disc temperature, and Dewar cap temperatures were 280°C, 280°C, -40°C, and 35°C, respectively. The vacuum was 0.2 mTorr, disc speed 3 mm/s, spiral separation was 1 mm, wavelength resolution 4 cm^{-1} and IR range 650–4000 cm^{-1} . Acquisition time was 0.6 s/file with 64 scans/spectrum. Data were processed using GRAMS/AI Ver. 9.1 (Grams Spectroscopy Software Suite, Thermo Fisher Scientific, Dreieich, Germany) followed by implementation of the OMNIC Software, Ver. 7.4.127 (Thermo Electron Corporation, Dreieich, Germany).

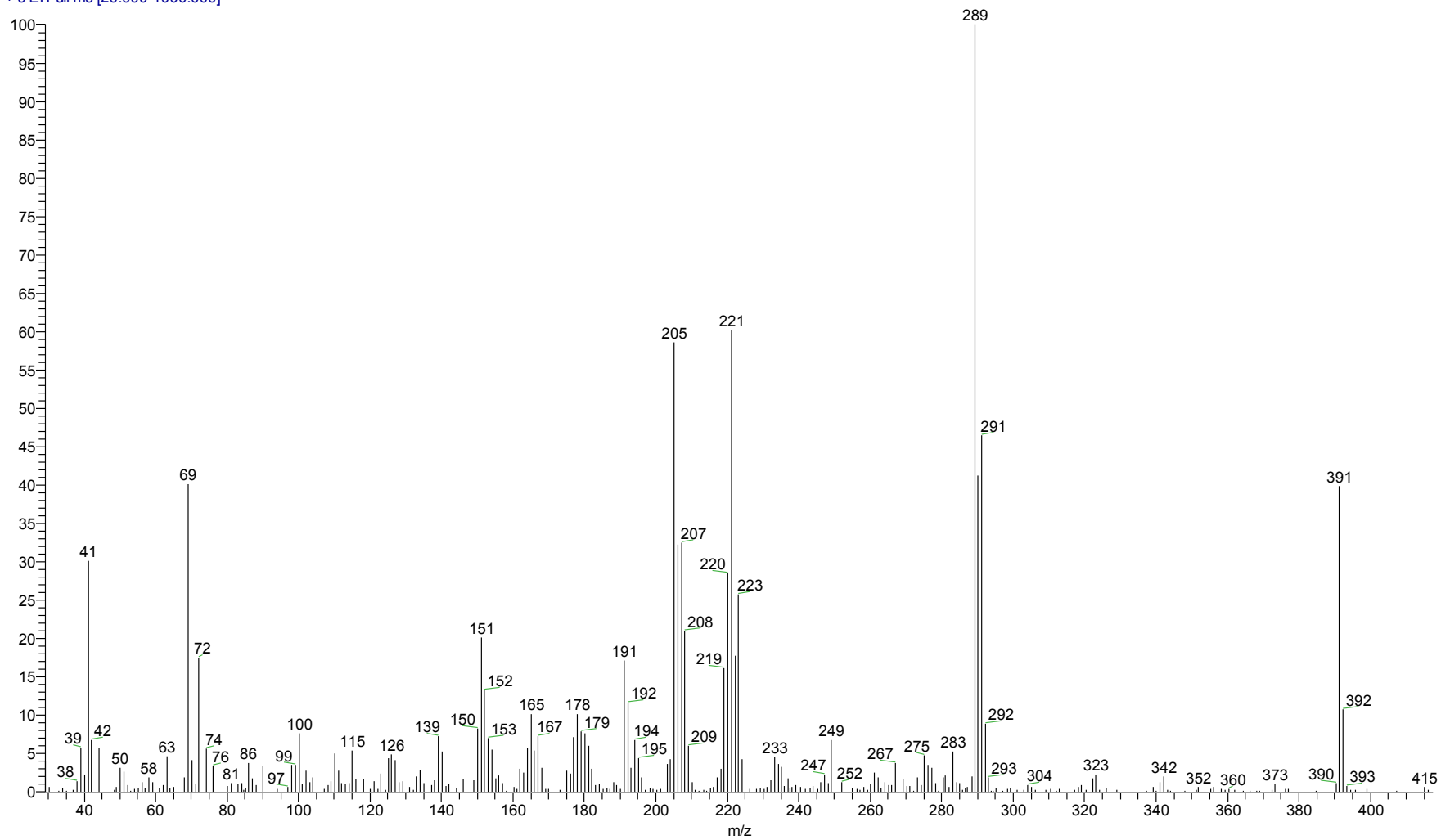
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T: + c EI Full ms [29.000-1000.000]



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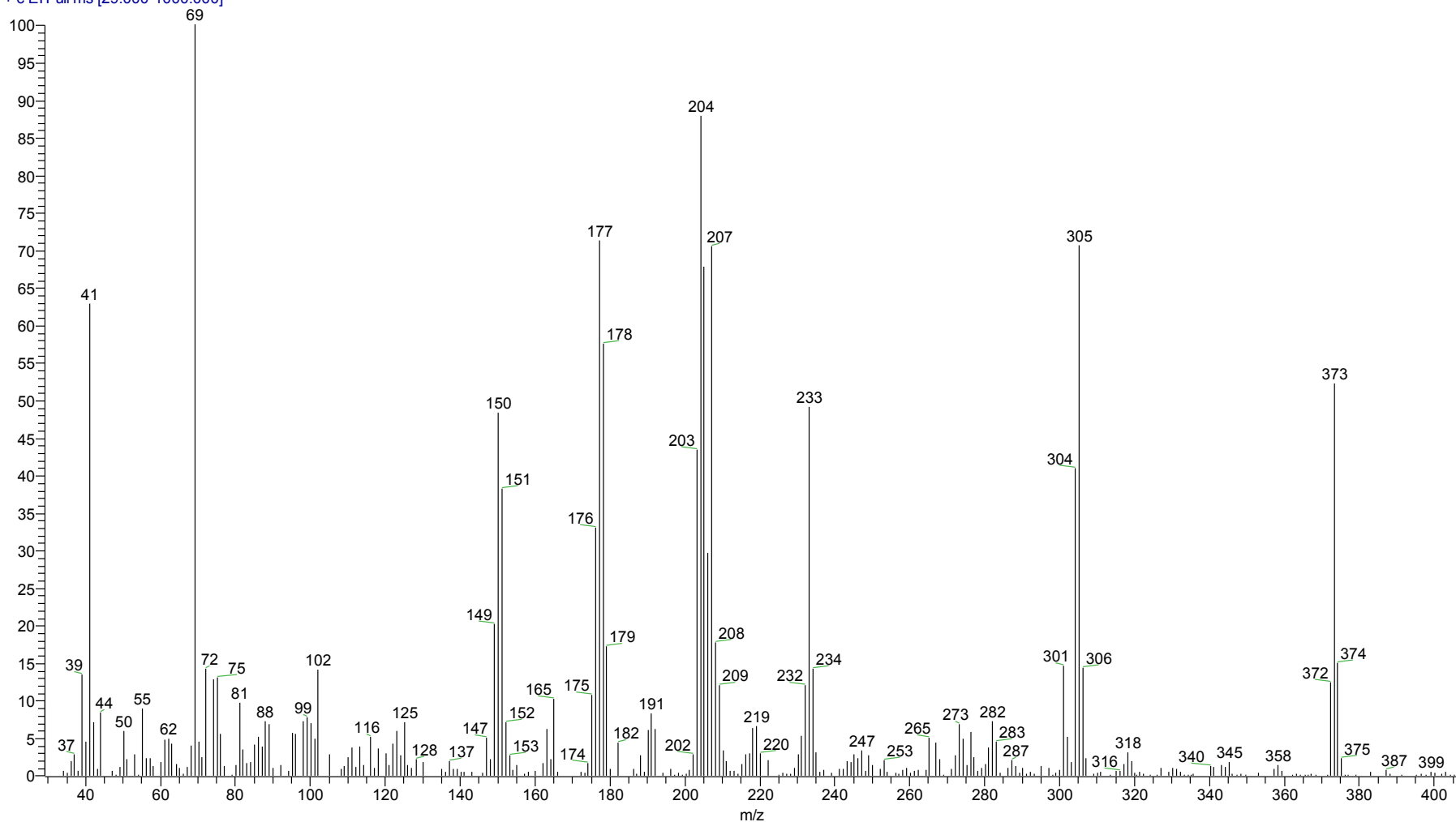
SBXIV_20191024 #1600 RT: 29.68 AV: 1 SB: 2 29.38, 30.47 NL: 3
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1CP-LSD Isomer II; RI: 3918 (DB-1,310)

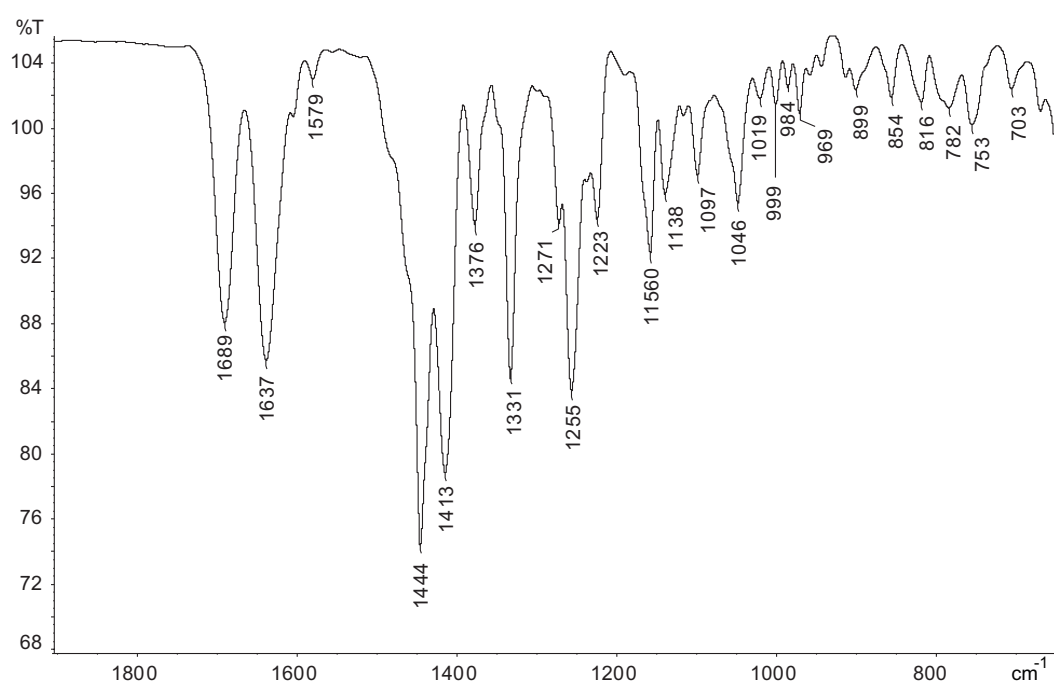
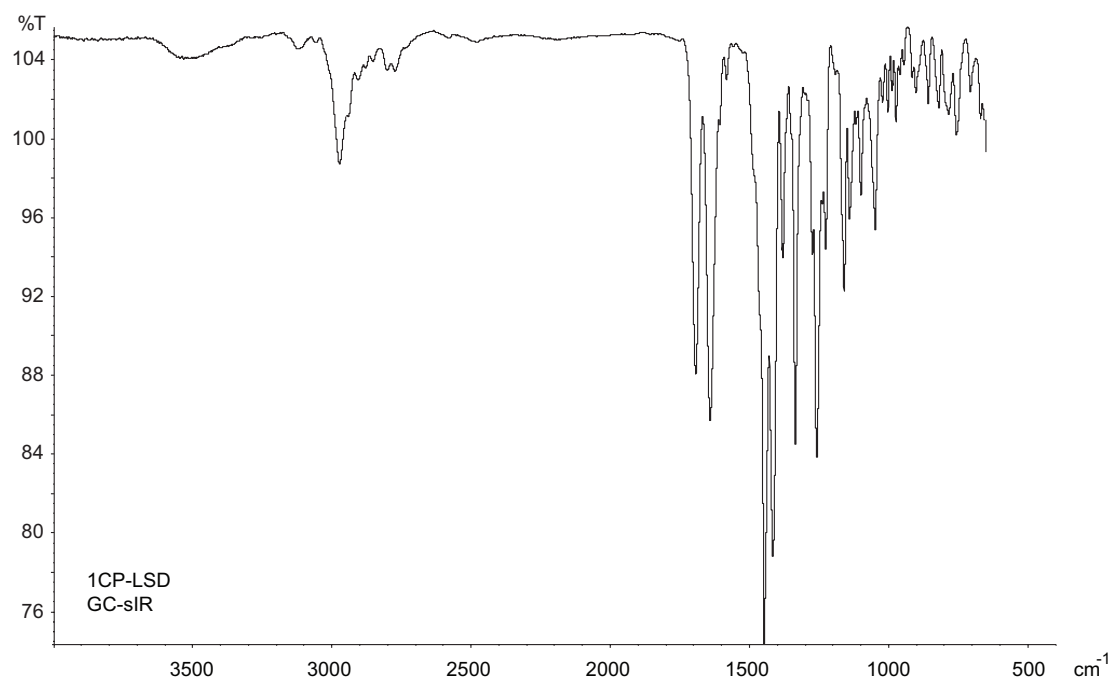
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SBXIV_20191024 #1423-1439 RT: 26.73-27.00 AV: 17 SB: 37 26. J, 27.25-27.66 NL: 5.21E5
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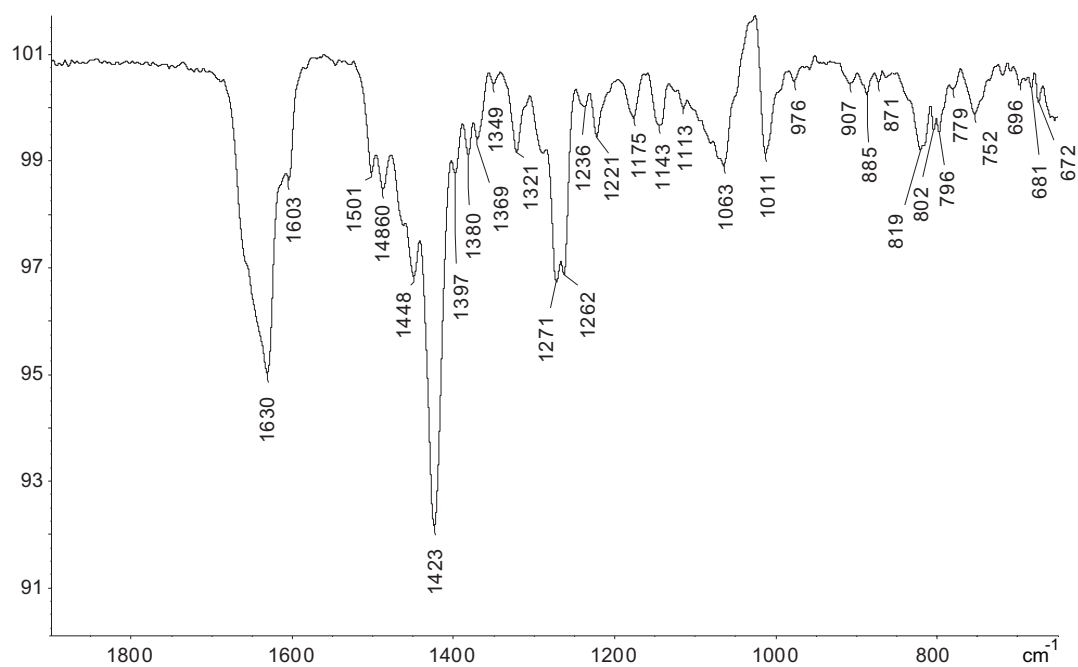
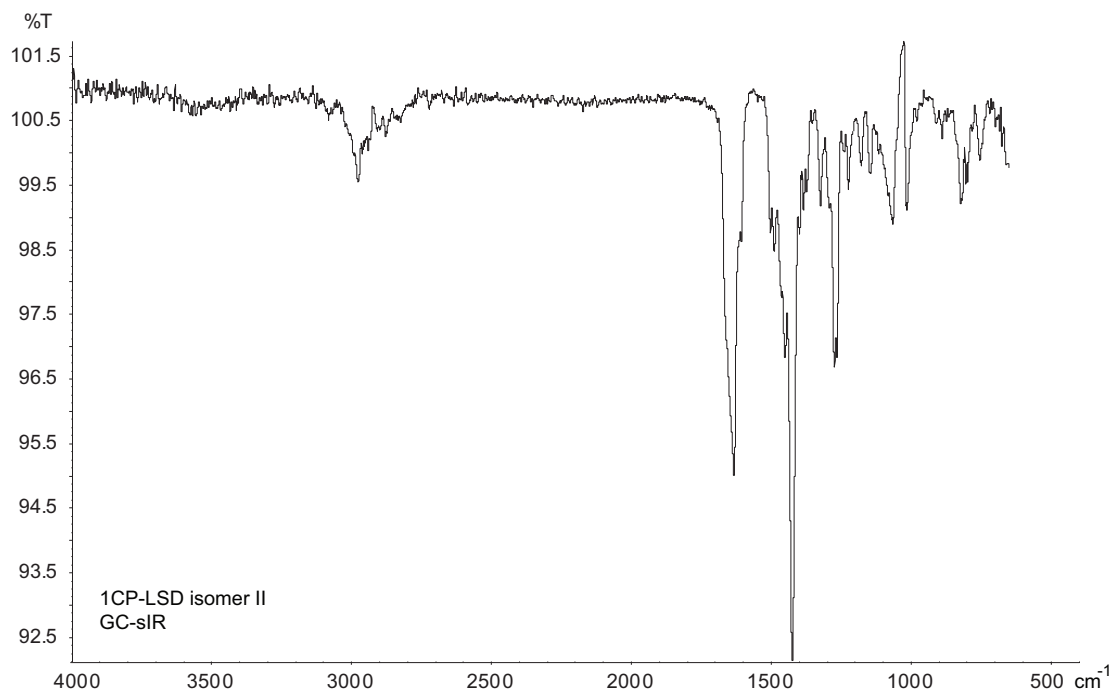


Pyridine derivative degradation product ("m/z 373 species")? RI: 3834 (DB-1,310)

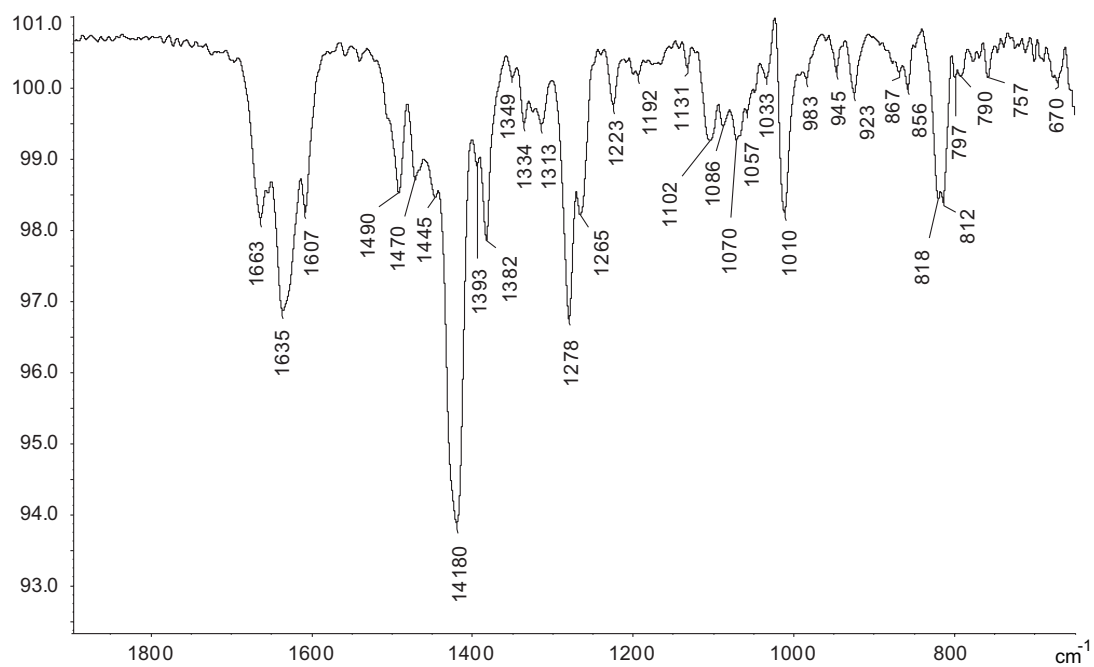
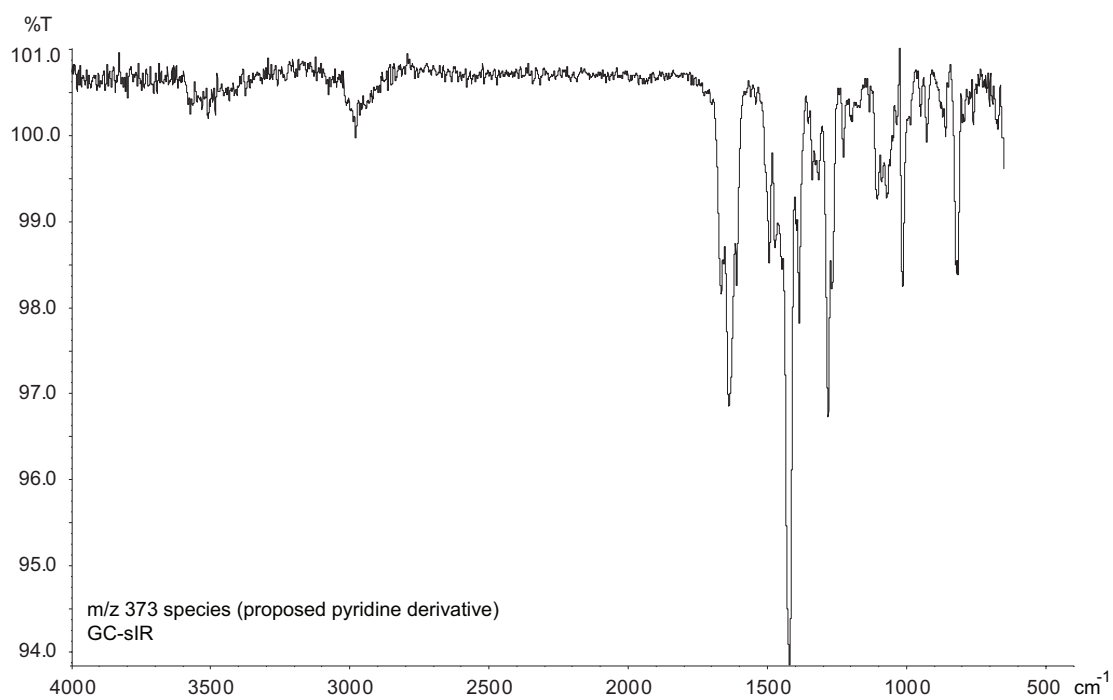
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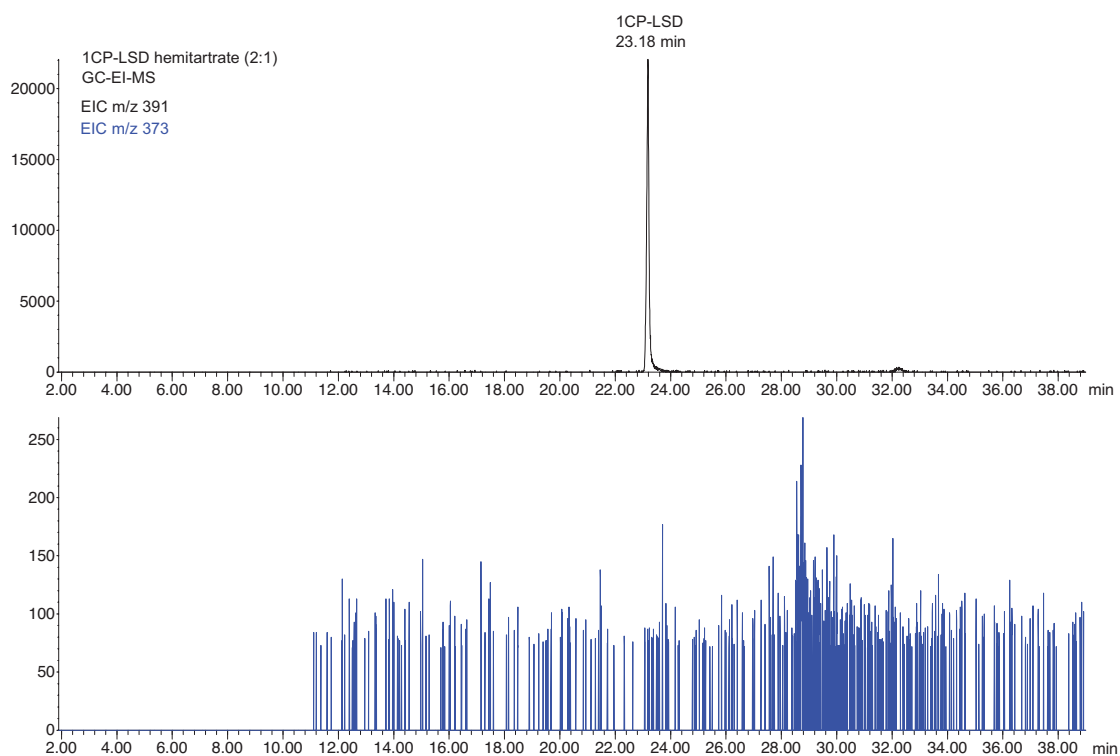
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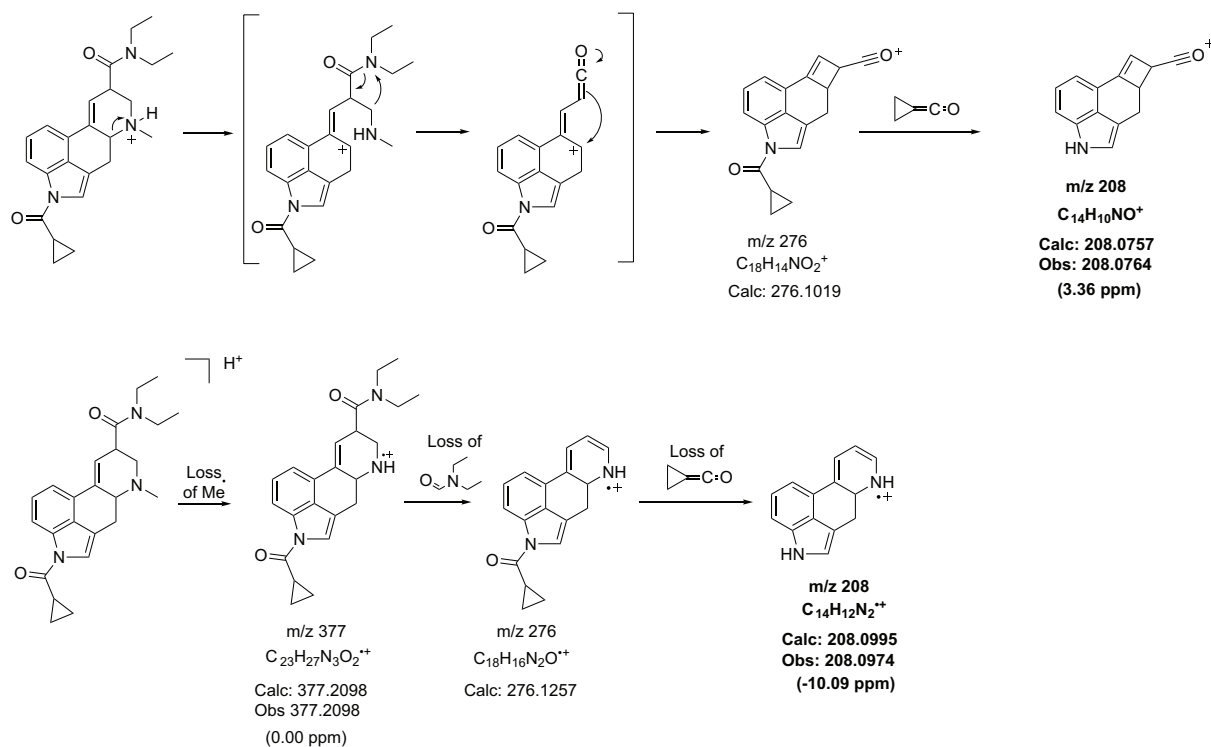
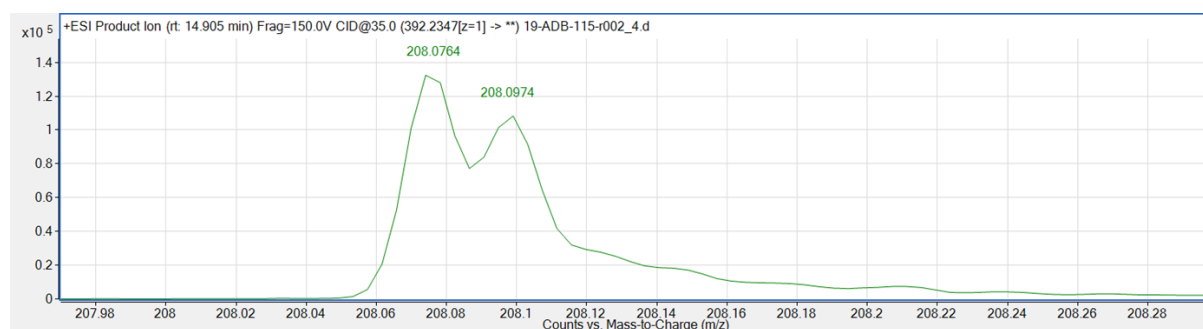
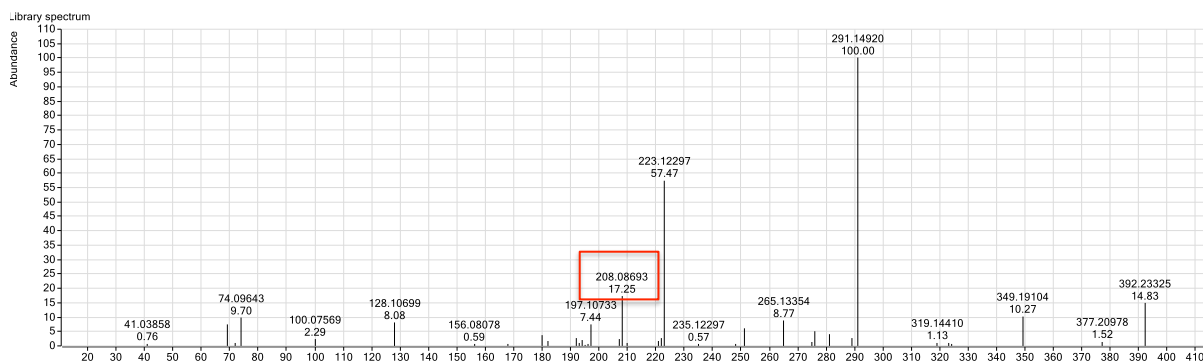


Samples were analyzed on an Agilent 7890A gas chromatograph coupled to a 5977A MSD. An AgilentHP-5MS UI column (30 m × 0.25 mm × 0.25 µm; Agilent Technologies, Stockport, UK) was used in split mode (1:50) with helium carrier gas at a constant flow of 1.0 mL/min. The injection port and transfer line temperatures were set at 275°C and 275°C, respectively. The initial oven temperature was 100°C, held for 1 min, ramped at 20 °C/min to 300°C and held at for 28 min (runtime 39 min). The ionization energy was set at 70 eV, the quadrupole at 150 °C, the ion source at 230°C and the mass range was set at m/z 29–500. The sample injection volume was 1 µL and a 1 mg/mL solution in methanol was analyzed.



GC-EI-MS analysis (method 3; merged EIC traces)

Repeated mass measurements of the m/z 208.0869 species and detection of two distinct product ions (Agilent 6530 QTOF system) in profile mode.



Dependence of both m/z 208 ions on collision energy

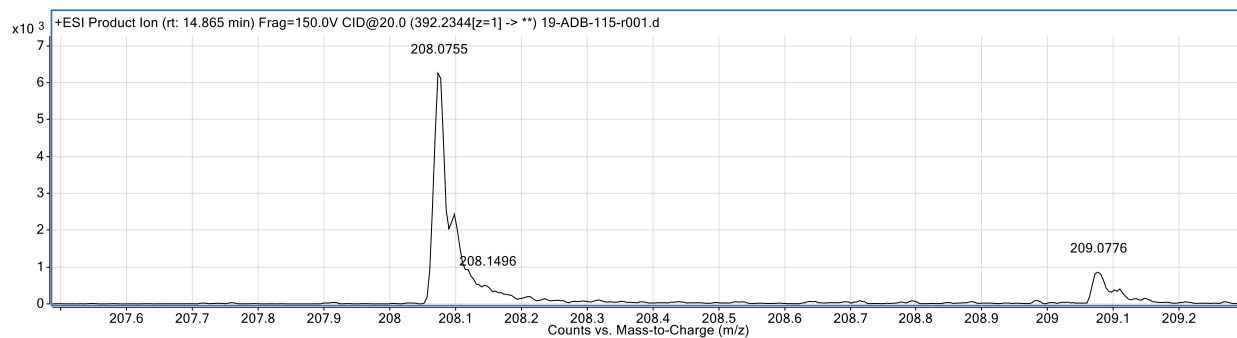
10V



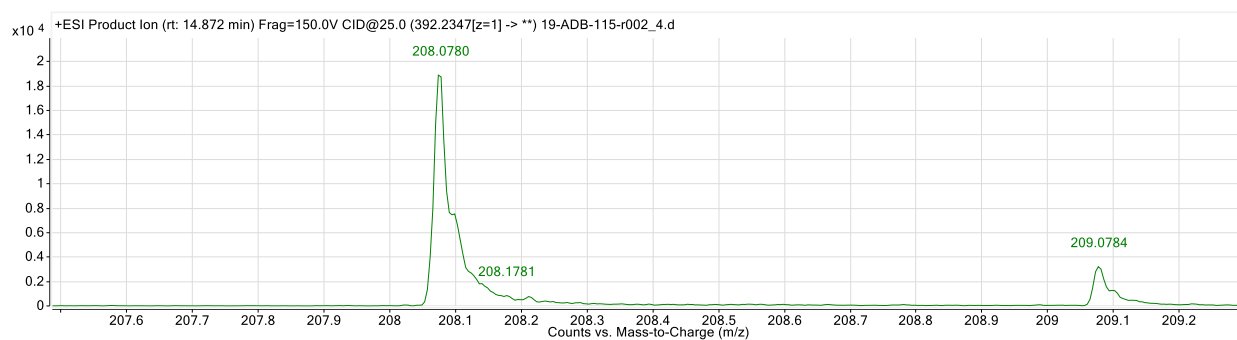
15V



20V

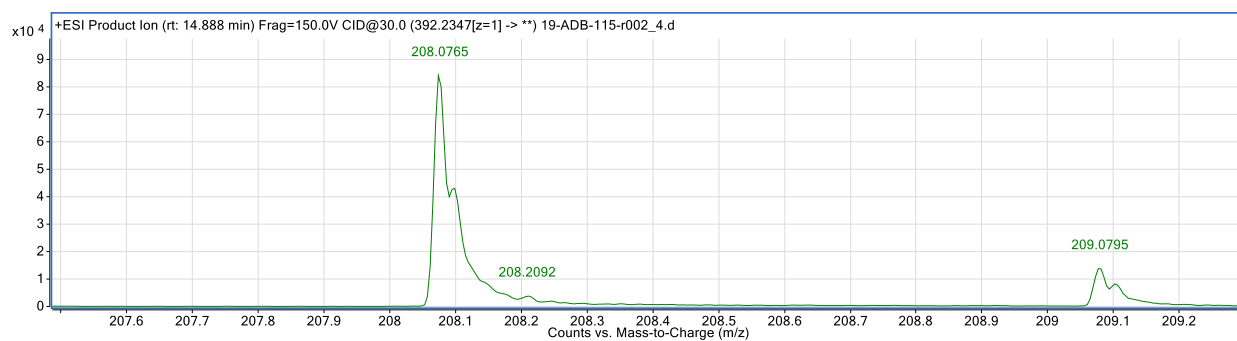


25V

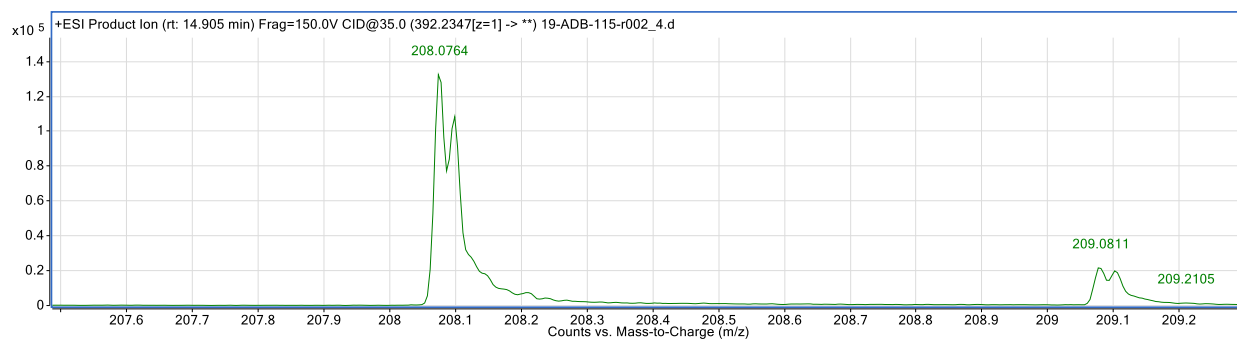


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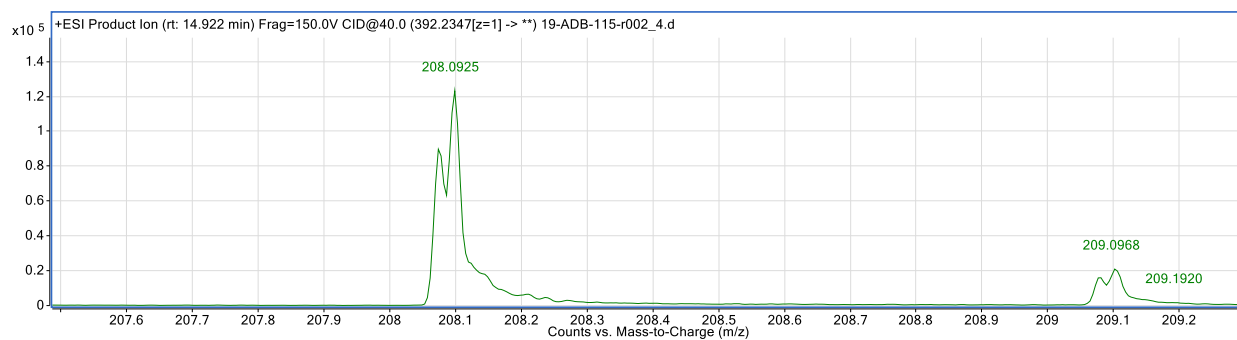
30V



35V



40V



Thermolysis method

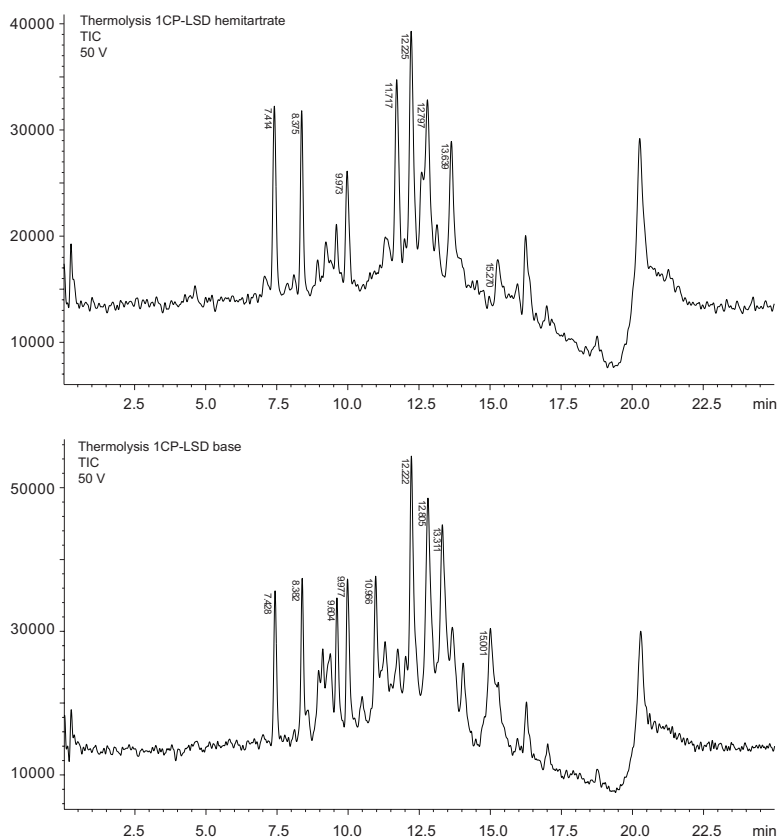
A sample (1 mg) in a glass vial (1.5 mL, purged with nitrogen and plugged with glass wool) was heated at 280°C for 3 min. This was allowed to cool to room temperature and the residue (dark brown for both the tartrate and freebase) was dissolved as much as possible (some insoluble material present) in methanol (500 µL). The mixture was centrifuged at 18,000 rpm (25,350 g) for 3 min and the supernatant was collected.

LC-ESI-MS

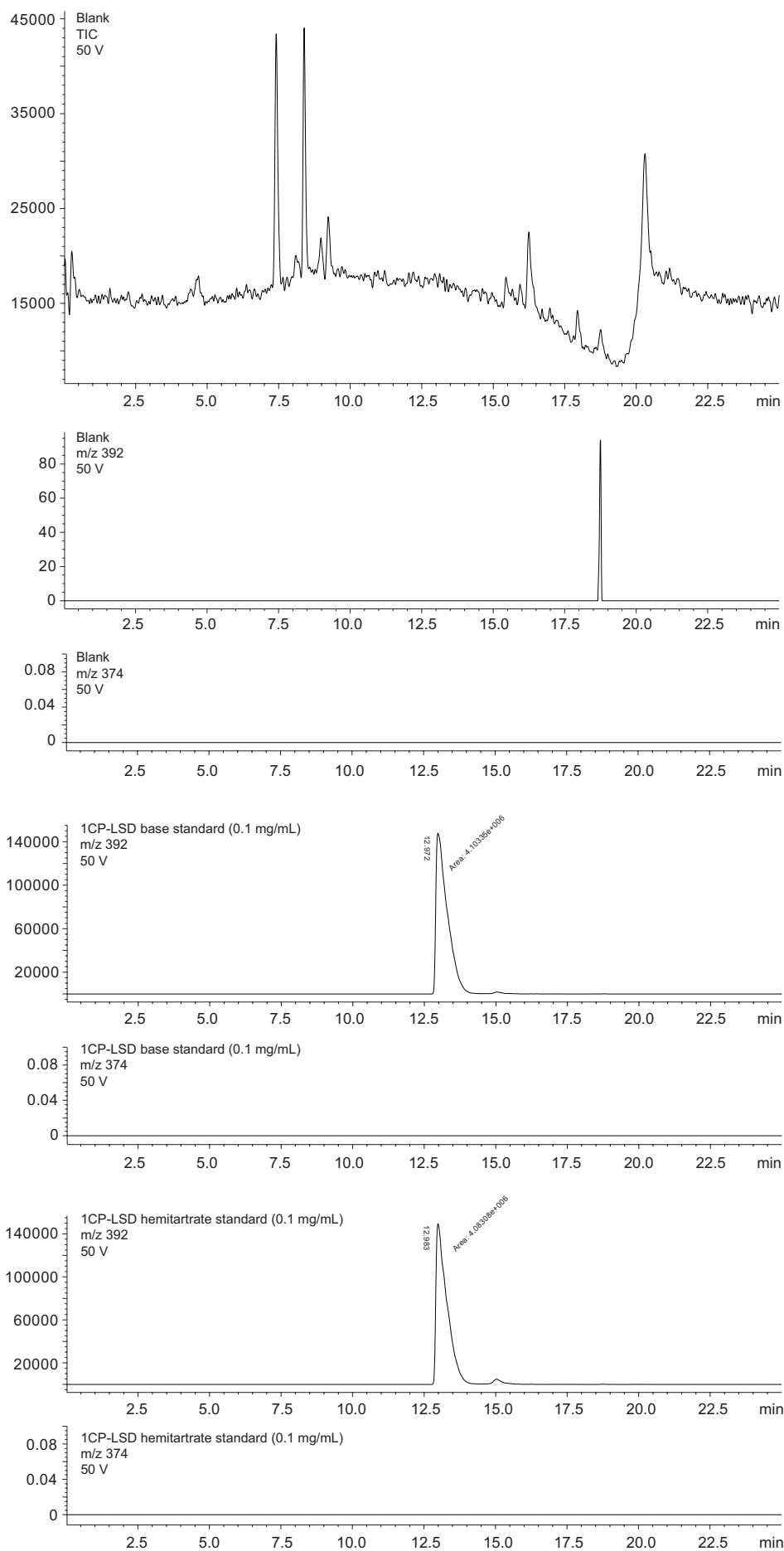
An aliquot (100 µL) was diluted with acetonitrile (150 µL, containing 0.1 % formic acid) and water (250 µL, containing 0.1 % formic acid). The mixture was passed through a spin filter (Costar Spin-X, nylon membrane, pore size 0.22 µm) and the filtrate was analyzed.

LC-MS was performed on Agilent 1100 LC system using an Allure® PFP Propyl (3 µm, pore size 60 Å; 50 mm x 2.1 mm) (Thames Restek, High Wycombe, UK). Mobile phase A: acetonitrile containing 0.1% formic acid; mobile phase B: water containing 0.1% formic acid). Elution profile: two % A (0–2 min), then linear gradient to 60 % A at 15 min; 60 % linear gradient to 80 % A at 18 min, and linear gradient down to 2 % A at 20 min and 2 % A for 5 min (run time 25 min); flow rate, 800 µL/min, 10 µL injected.

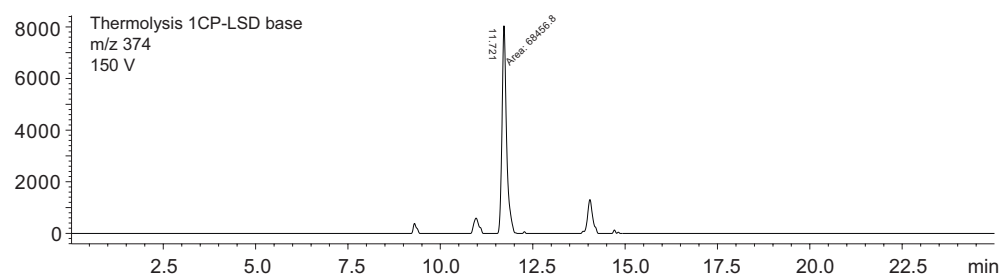
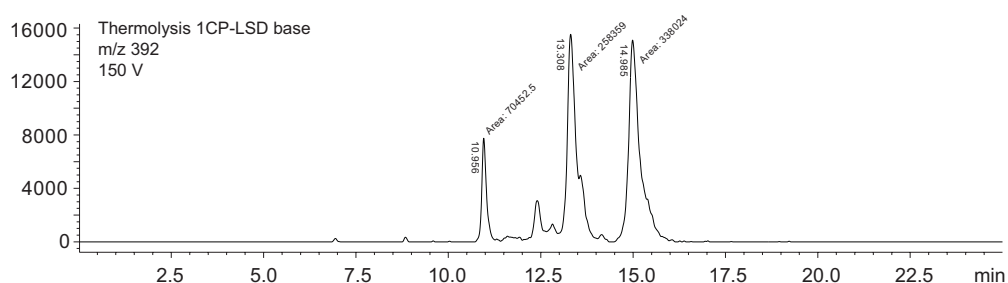
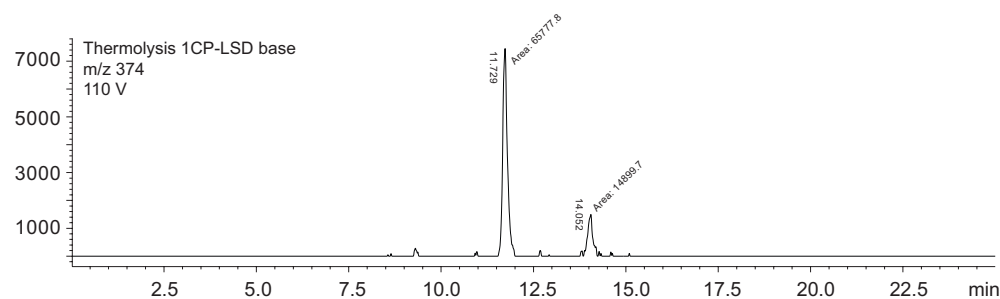
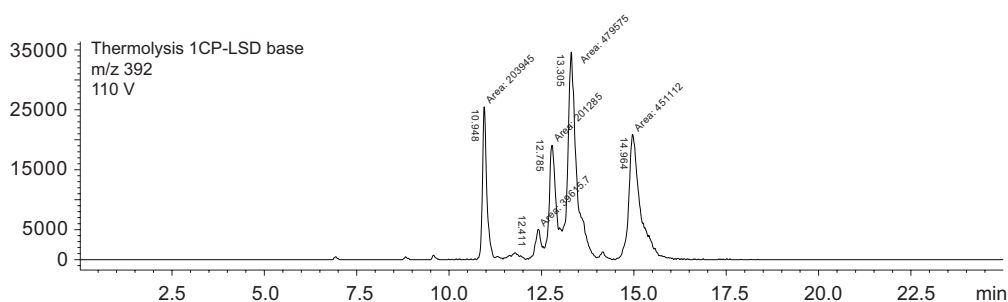
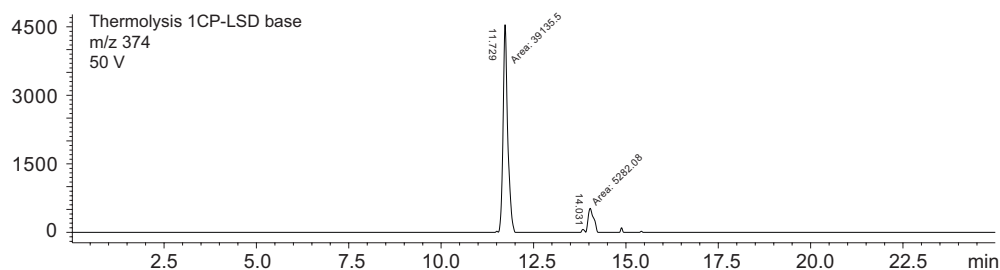
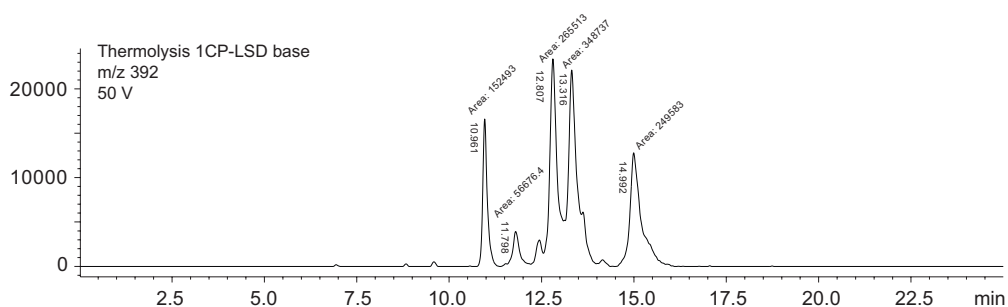
The LC system was coupled to a Hewlett Packard/Agilent 1100 MSD (Santa Clara, CA, USA) using the following conditions: positive ESI mode (full scan, m/z 70–500) with a fragmentor voltage as stated for in-source CID, capillary voltage 3500 V, drying gas (N₂) 12 L/min at 350 °C and nebulizer (N₂) pressure 50 psig. The mass spectrometer was tuned according to the manufacturer's instructions using ESI Tuning Mix G2421A (Agilent Technologies).



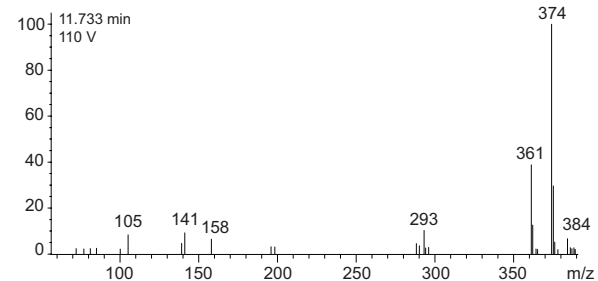
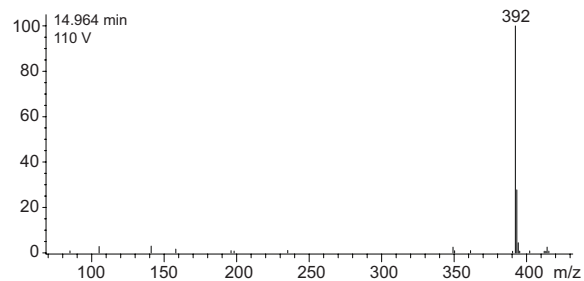
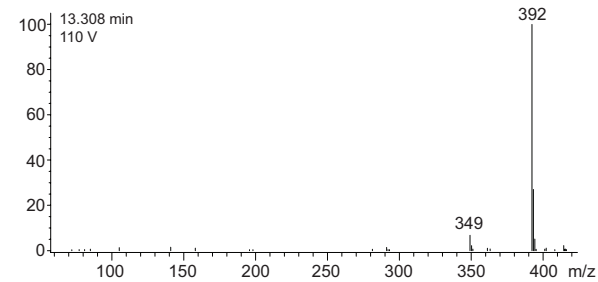
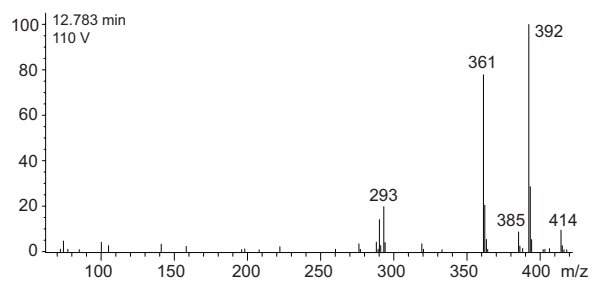
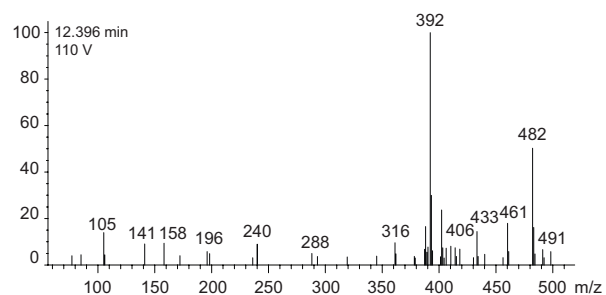
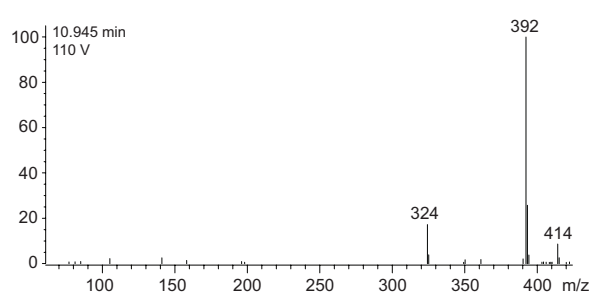
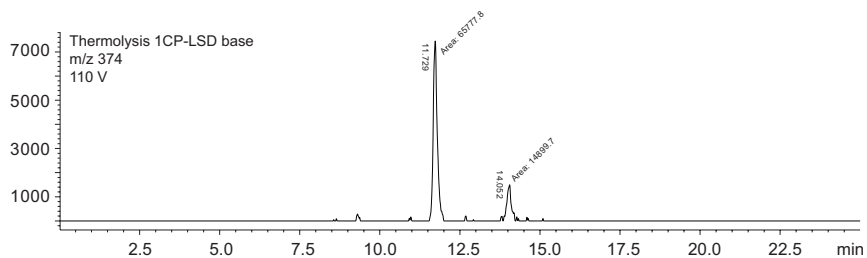
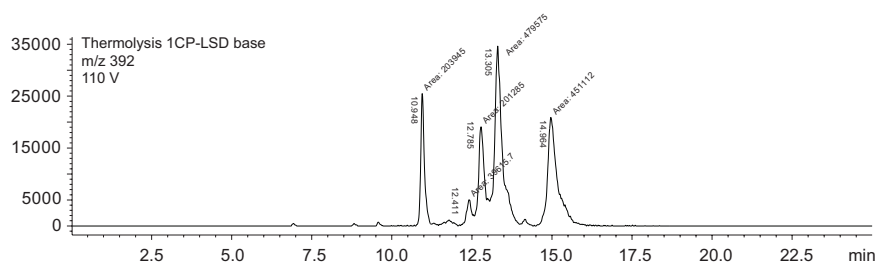
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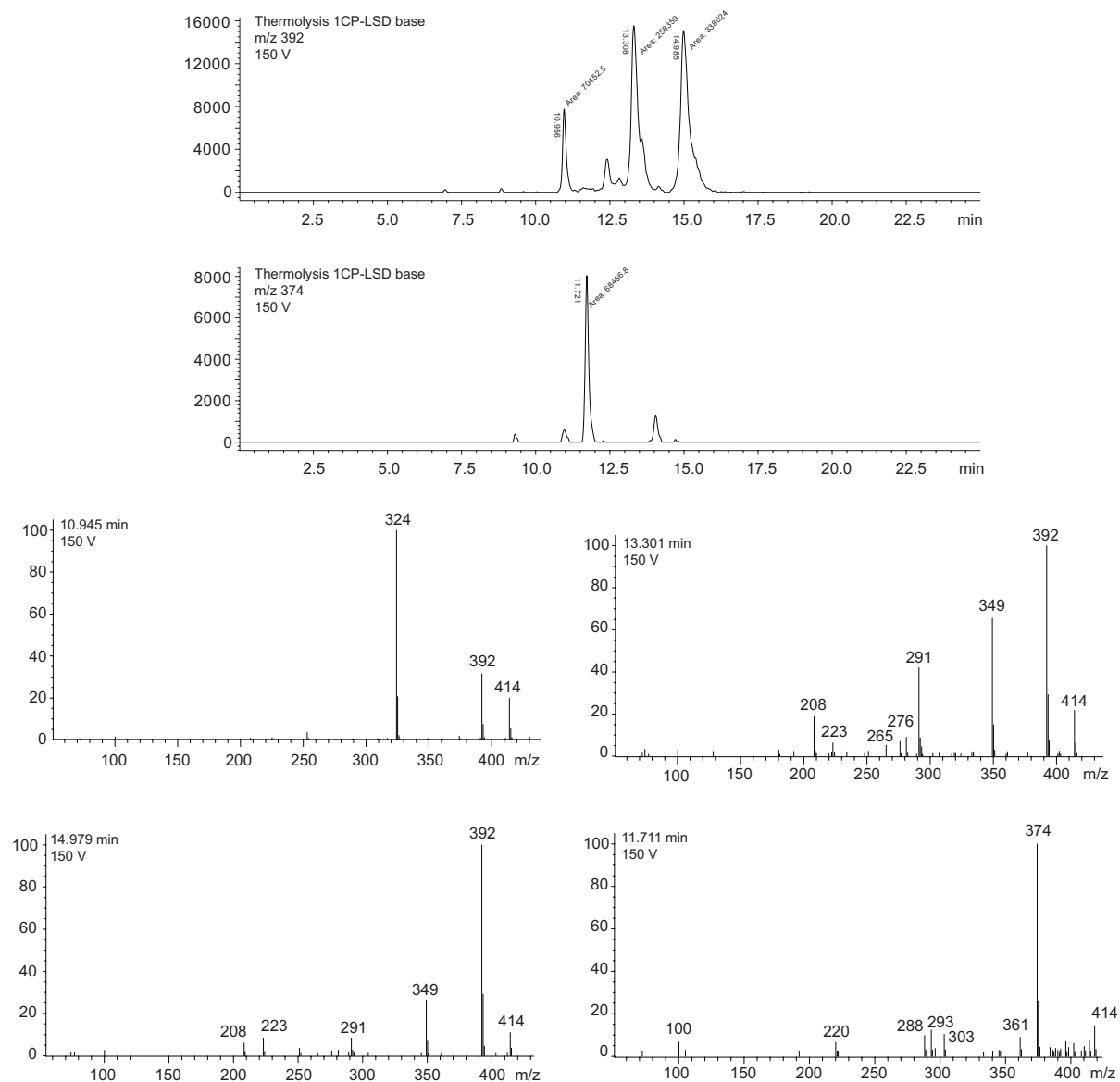
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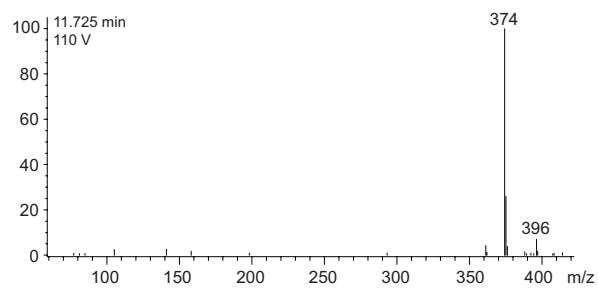
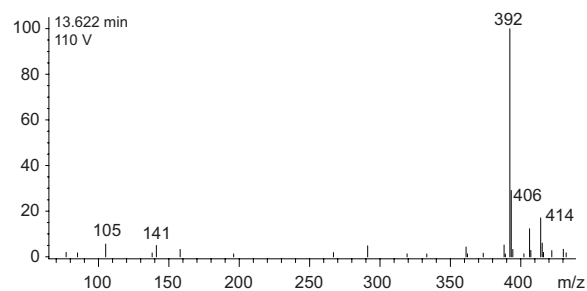
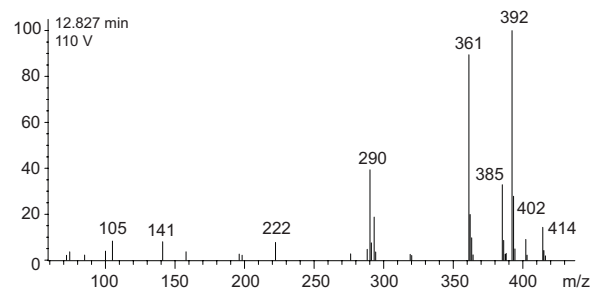
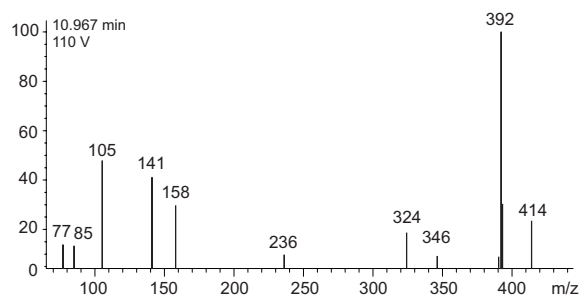
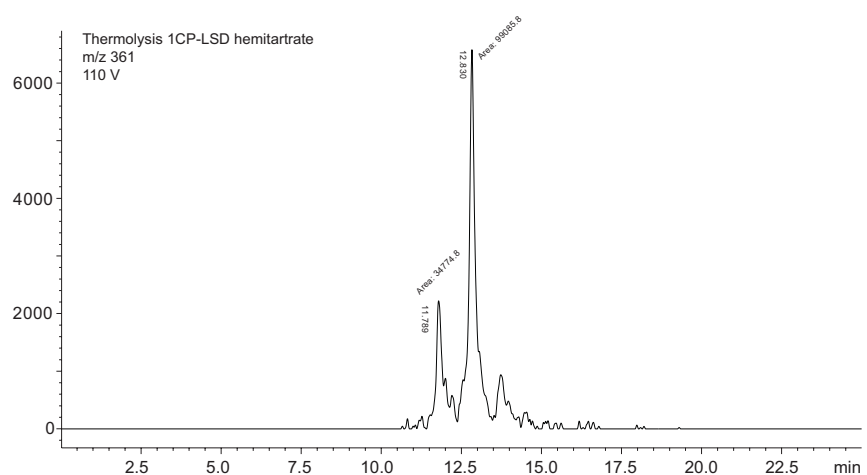
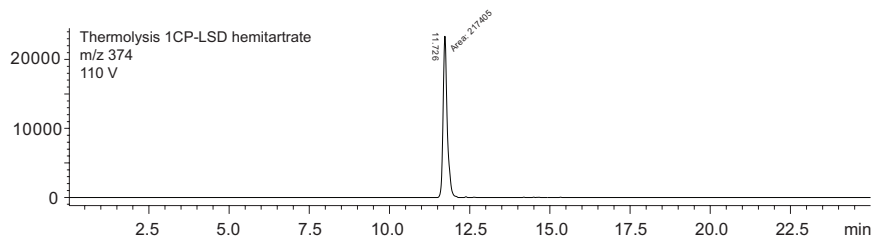
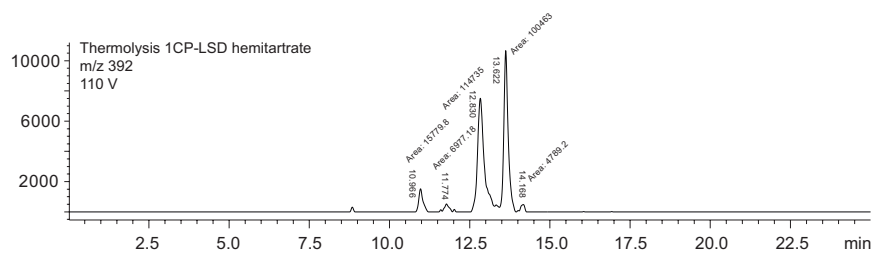
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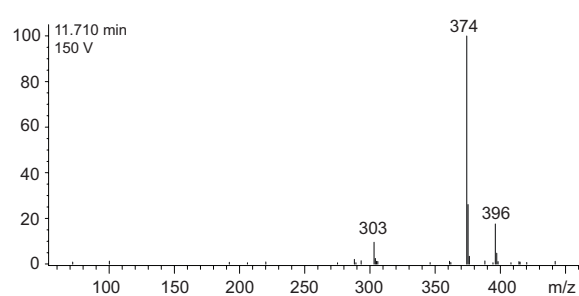
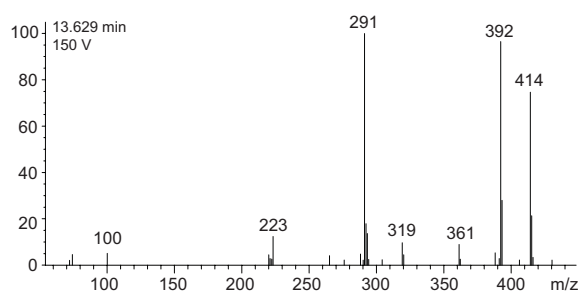
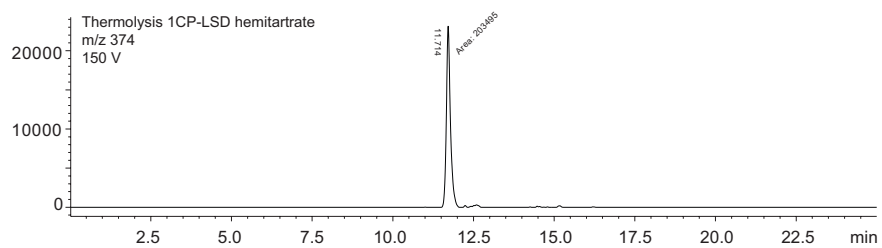
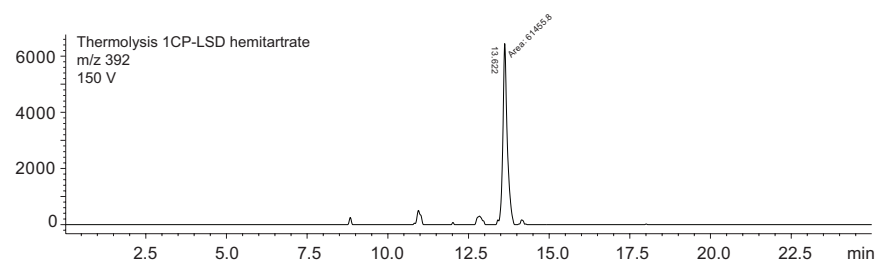
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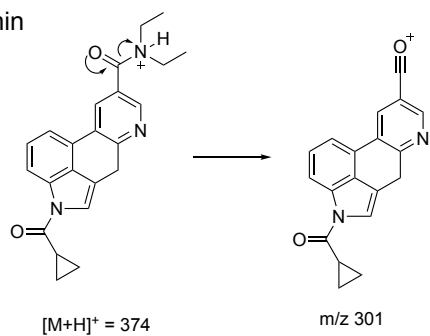
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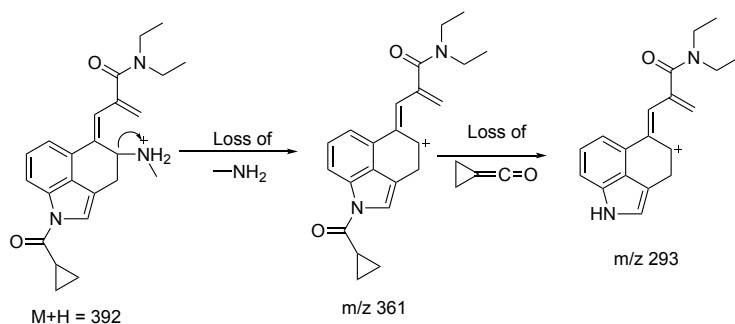
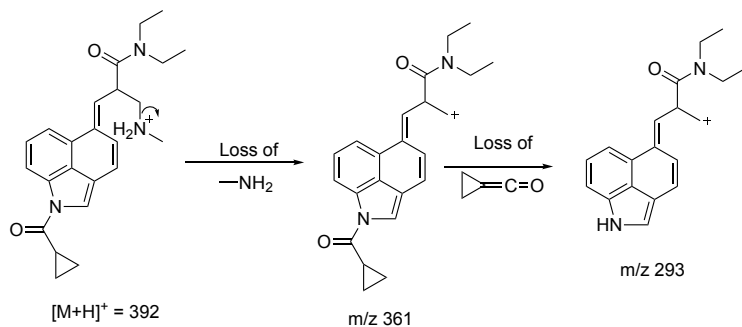
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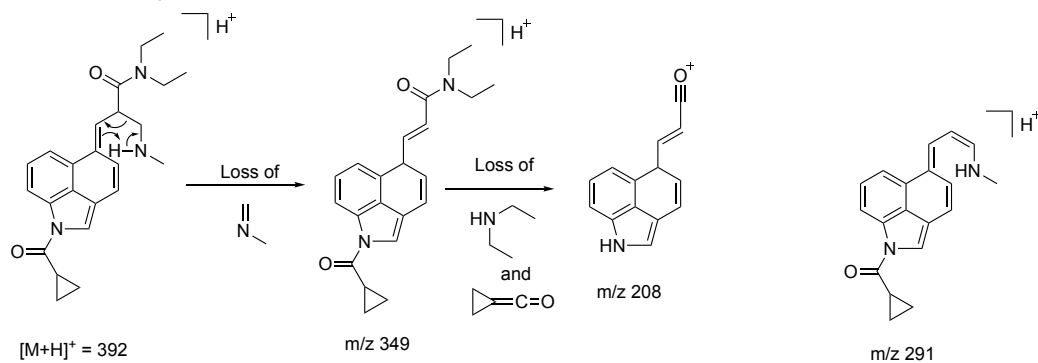
~11.7 min



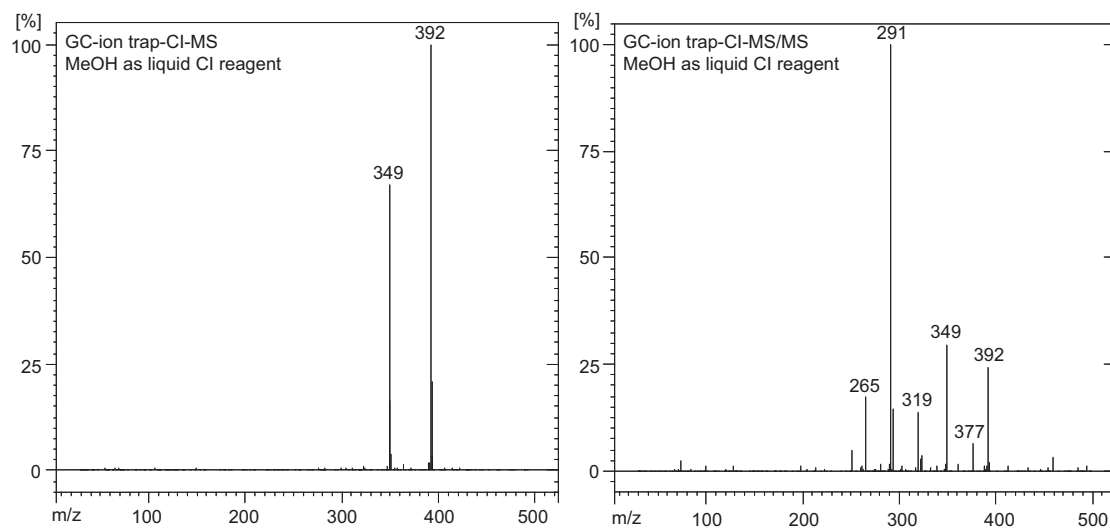
~12.8 min - peak with m/z 361



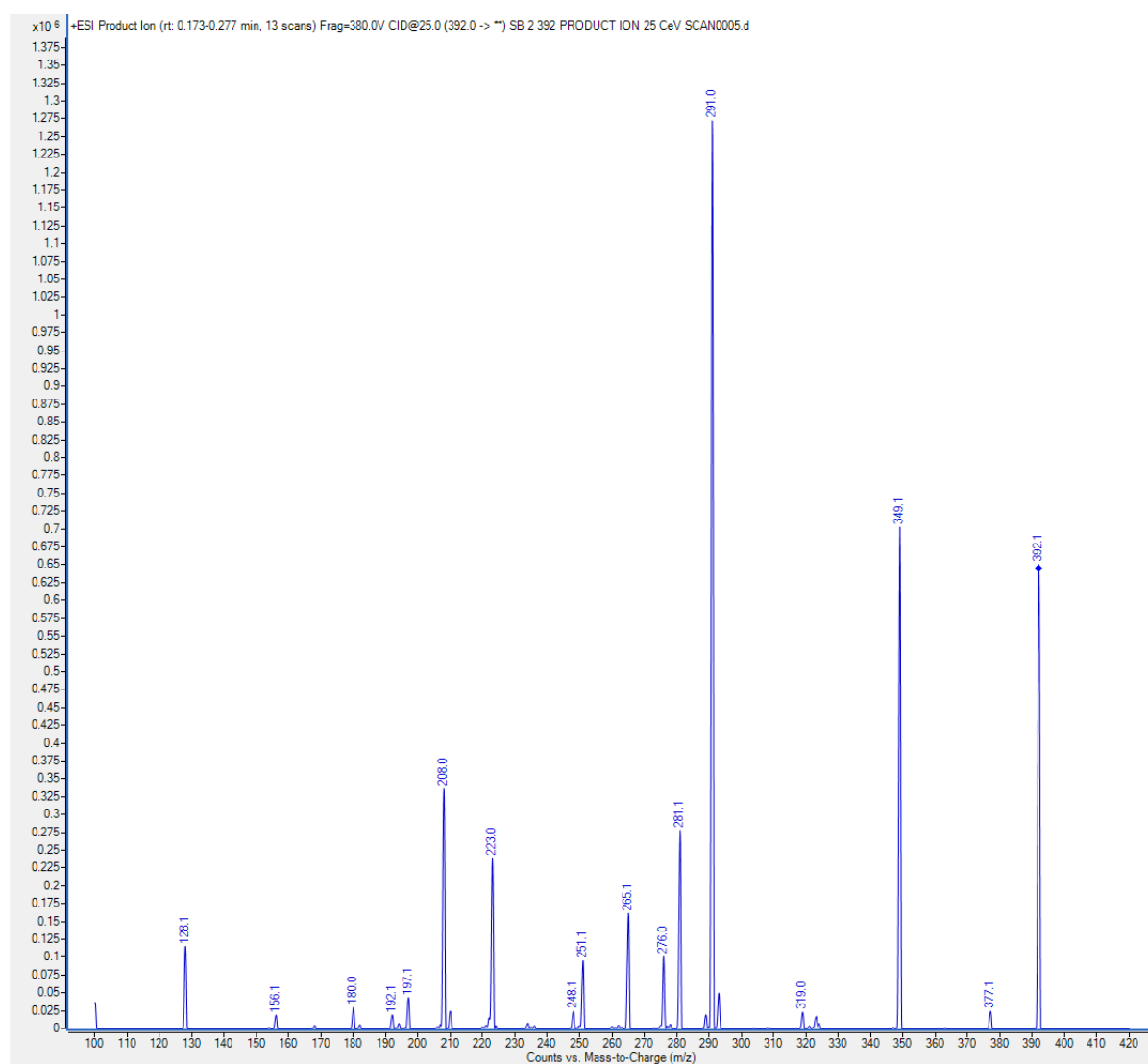
or ~13.3 min - peak with m/z 349



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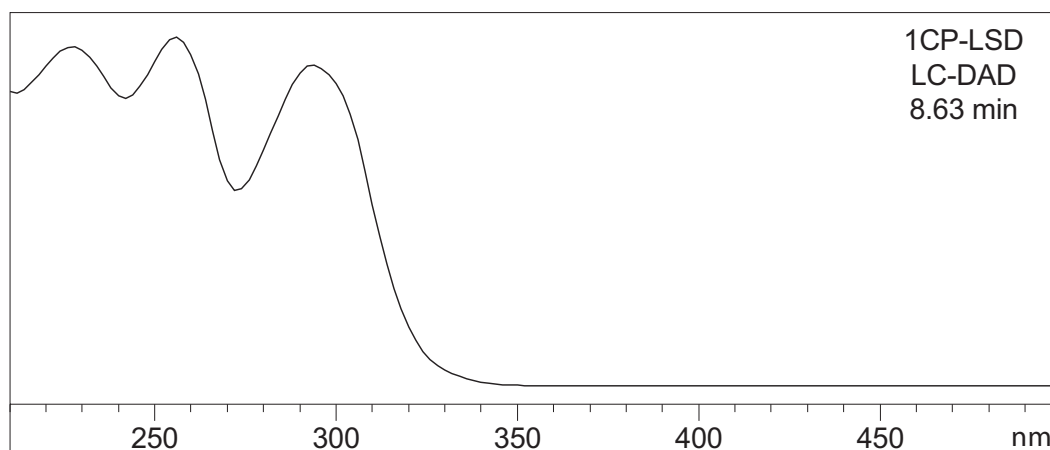


ESI-QqQ-MS/MS of 1CP-LSD



HPLC-DAD method

An Agilent 1100/1200 liquid chromatography system (quaternary pump G1311A, degasser G1322A, autosampler G1313A, column oven G1316A) coupled to a UV diode array detector (G1315D) was used using a Zorbax Eclipse XDB-C18 column (150 mm × 4.6 mm, 5 µm, Agilent). The column oven was set to 25°C. The mobile phases were 0.1% formic acid in water (A) and 0.1% formic acid in methanol (B). The gradient elution commenced with 100% A and ramped to 100% B in 10 min and held for 2 min, followed by a return to starting conditions at the 20 min time point. The flow rate was 1.0 mL/min. The injection volume was 10 µL. The diode array detection window was set at 195–500 nm (2 nm steps).



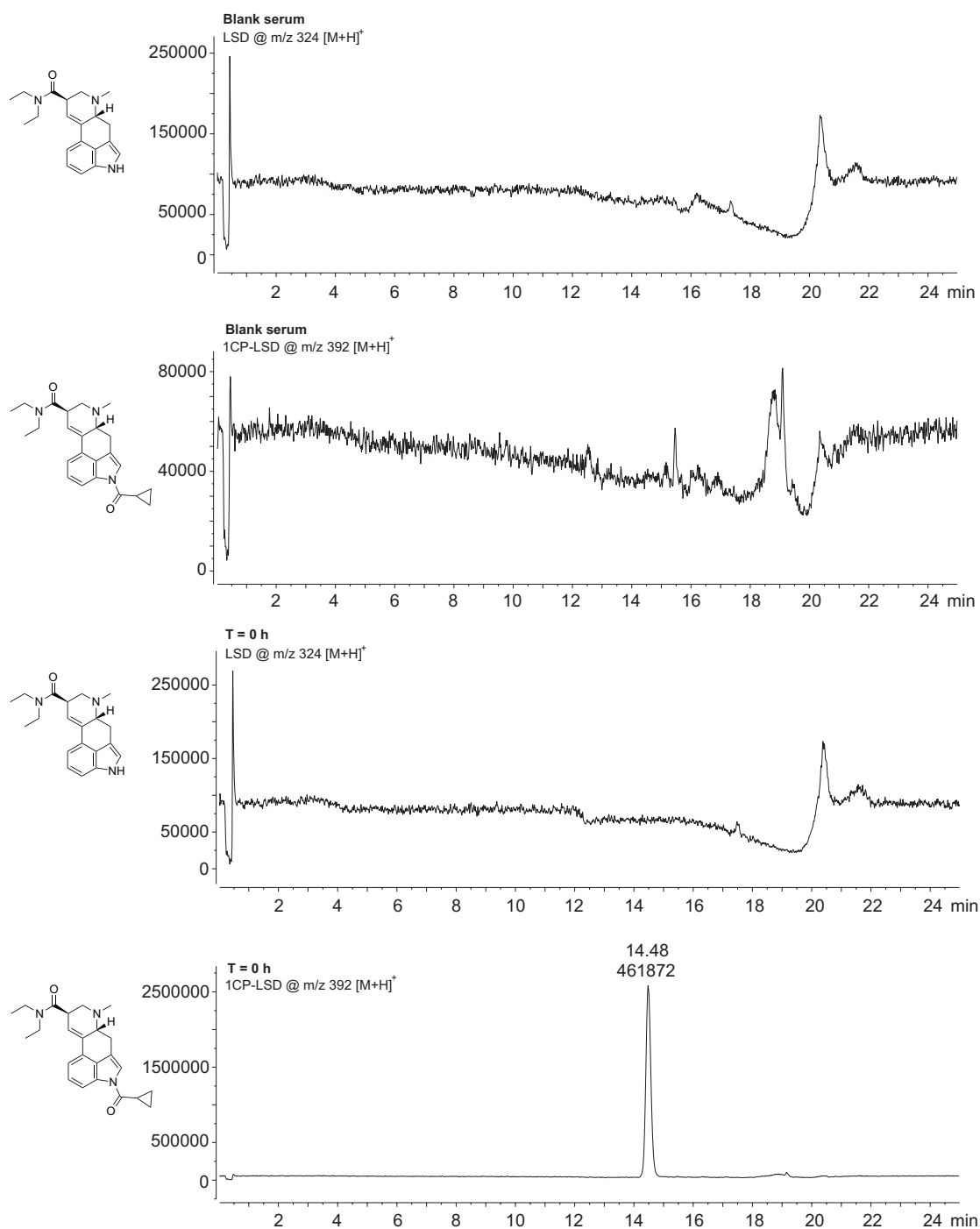
Serum containing 1CP-LSD or 1P-LSD (10 µg/mL, both as hemitartrates, 2:1) was incubated at 37°C and samples (10 µL) were removed at various times. These were diluted with acetonitrile/water (990 µL, 1:1, containing 0.1 % formic acid) and centrifuged at 18,000 rpm for 3 min. The supernatant was then passed through a Nylon filter (0.22 µm, Costar® Spin-X® centrifuge tube filter) and the filtrate was analysed by LC-ESI-MS.

LC-MS was performed on Agilent 1100 LC system using an Allure® PFP Propyl (3 µm, pore size 60 Å; 50 x 2.1 mm; part no. 9169552) (Thames Restek, High Wycombe HP14 4HW, UK): eluent A – acetonitrile containing 0.1% formic acid, eluent B – water containing 0.1% formic acid; 2 % A (0 - 2 min.) a linear gradient up to 60 % A at 15 min, 60 %, a linear gradient up to 80 % A at 18 min, a linear gradient down to 2 % A at 20 min and 2 % A for 5 min (run-time 25 min); flow rate, 800 µL/min, 10 µL injected.

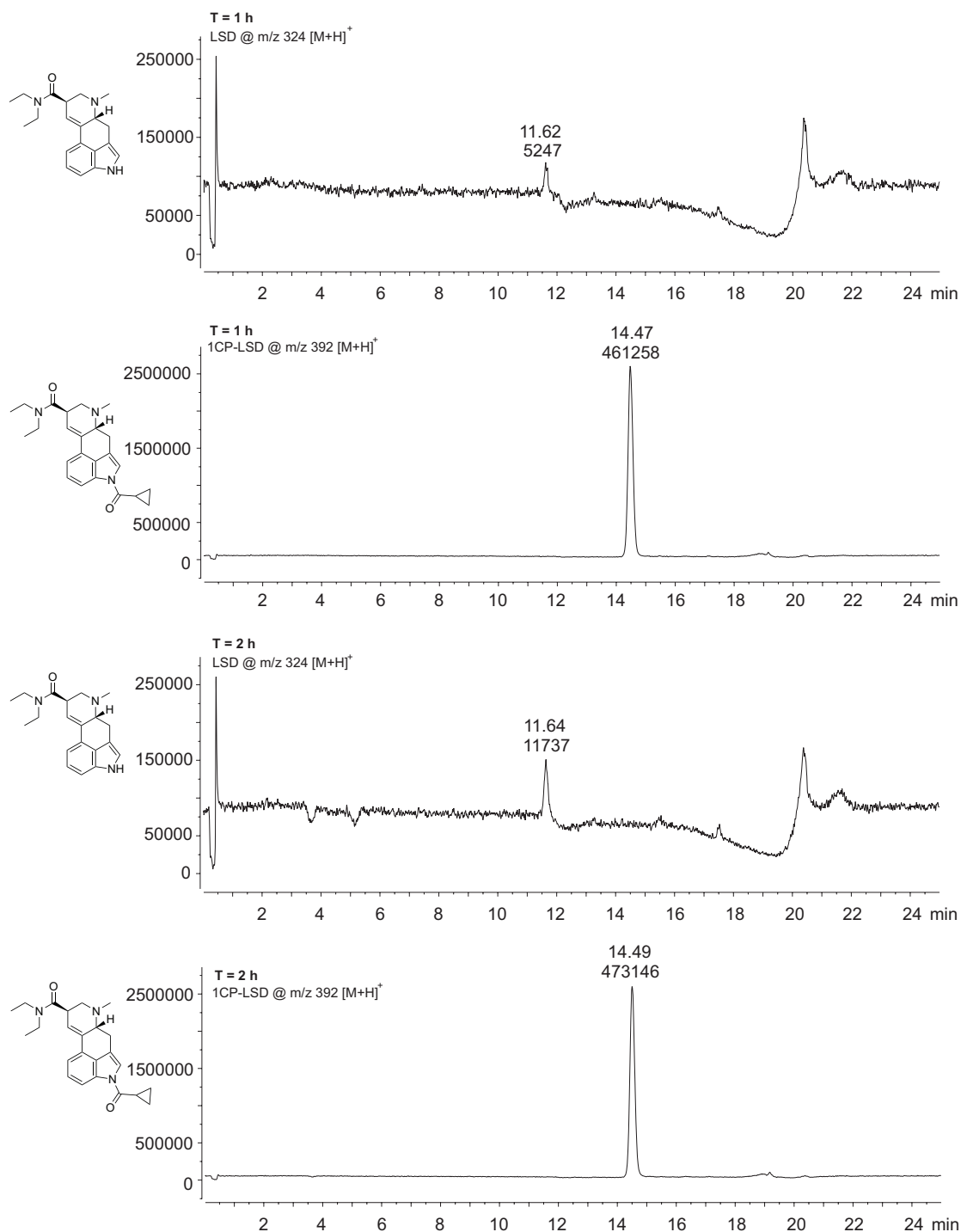
The LC system was coupled to a Hewlett Packard/Agilent 1100 MSD (Santa Clara, CA, USA) using the following conditions: positive ESI mode with a fragmentor voltage of 50 V for in-source CID), capillary voltage 3500 V, drying gas (N₂) 12 L/min at 350 °C and nebulizer (N₂) pressure 50 psig. The mass spectrometer was tuned according to the manufacturer's instructions using ESI Tuning Mix G2421A (Agilent Technologies). For SIM, m/z's 392, 380 and 324 were used for 1CP-LSD, 1P-LSD and LSD respectively. For full scan, a range of m/z 70-500 was used.

Supporting Information – Drug Testing and Analysis

1CP-LSD (10 µg/mL) incubation in human serum (LC-ESI-SIM-MS)

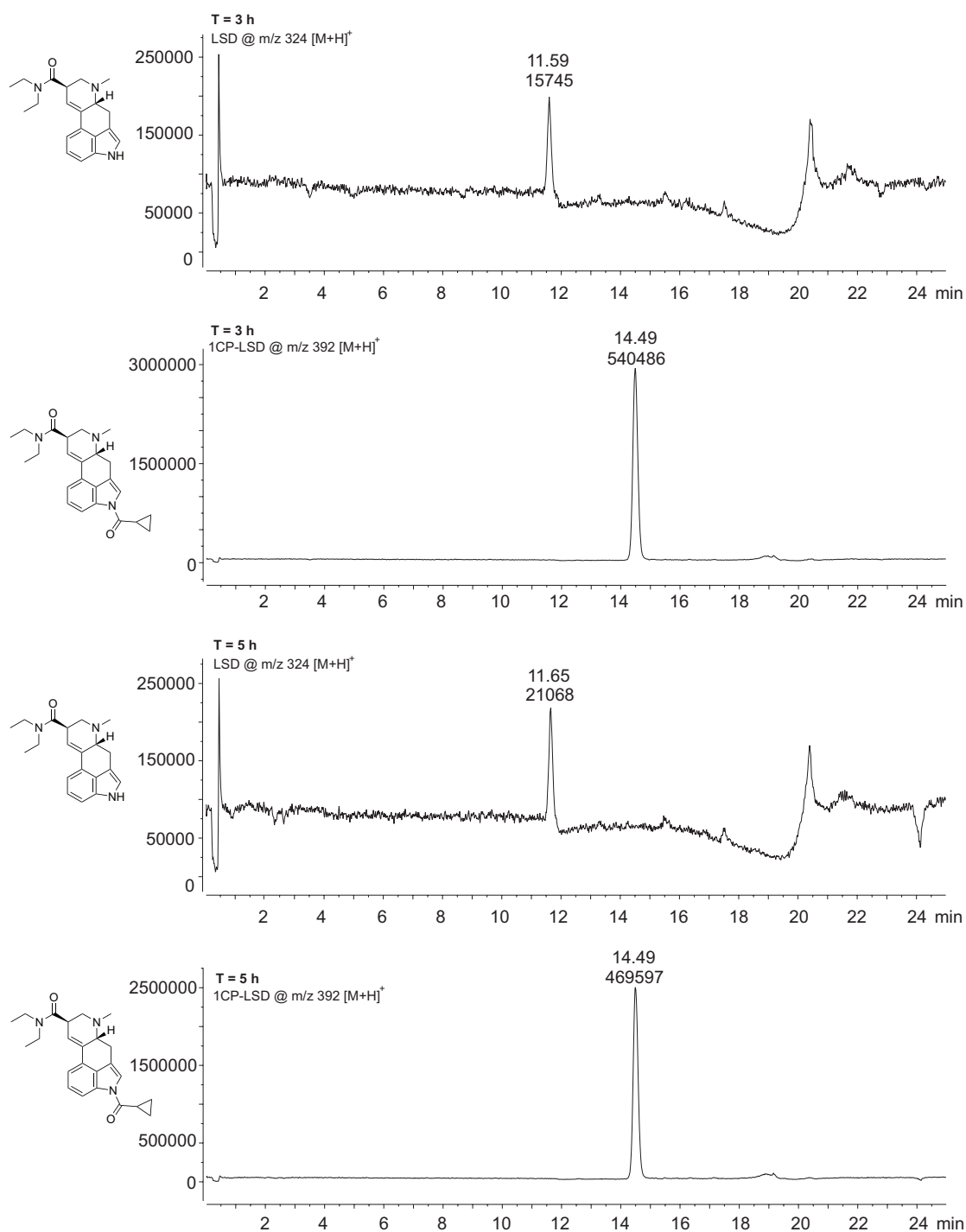


1CP-LSD (10 µg/mL) incubation in human serum (LC-ESI-SIM-MS)

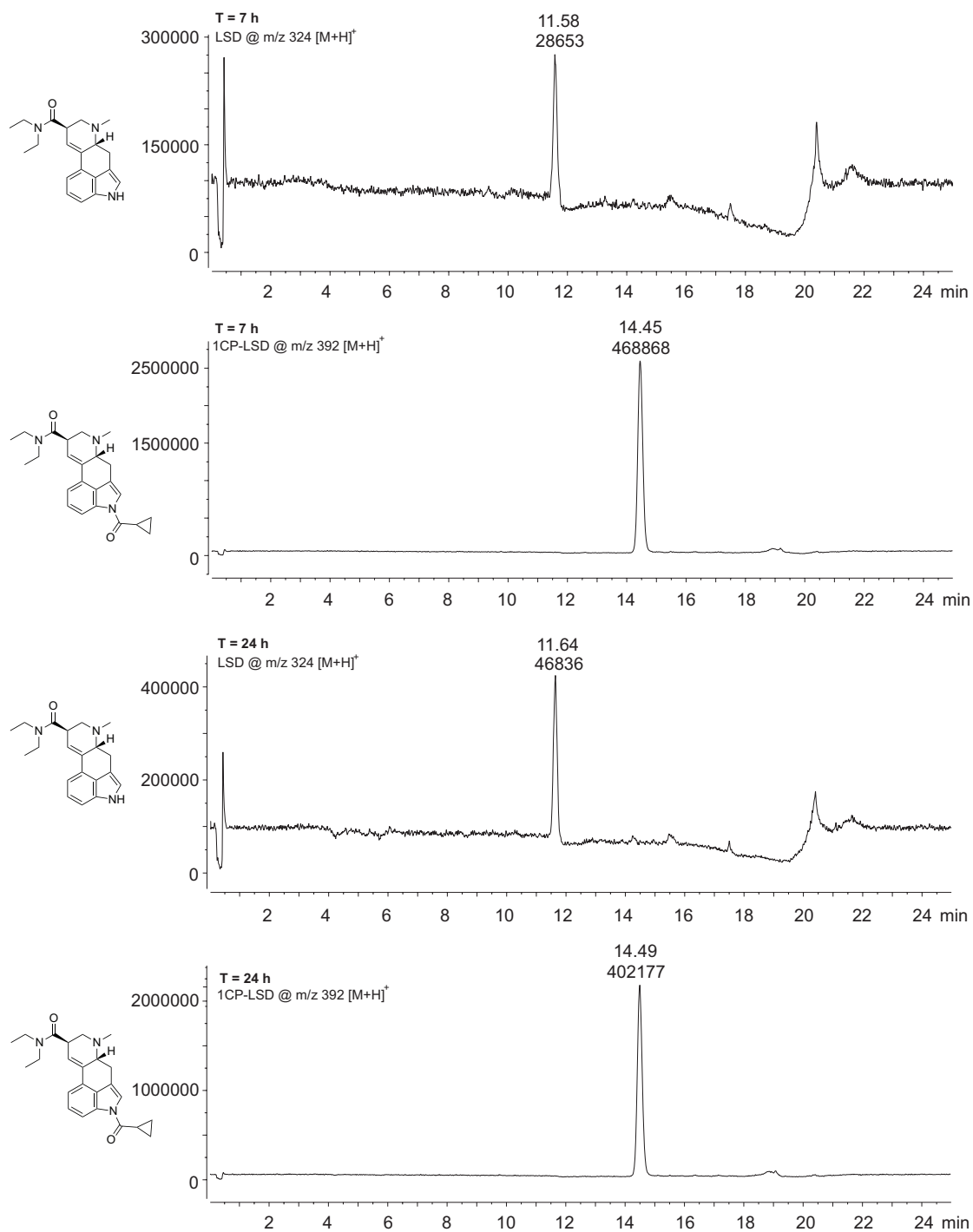


Supporting Information – Drug Testing and Analysis

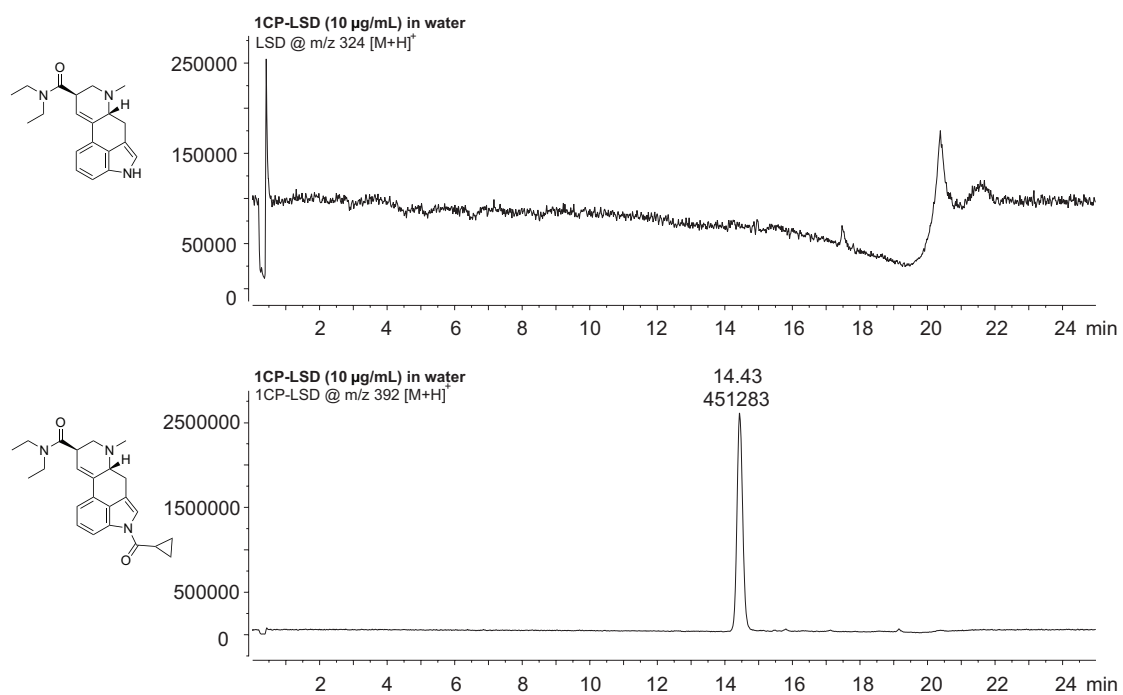
1CP-LSD (10 µg/mL) incubation in human serum (LC-ESI-SIM-MS)



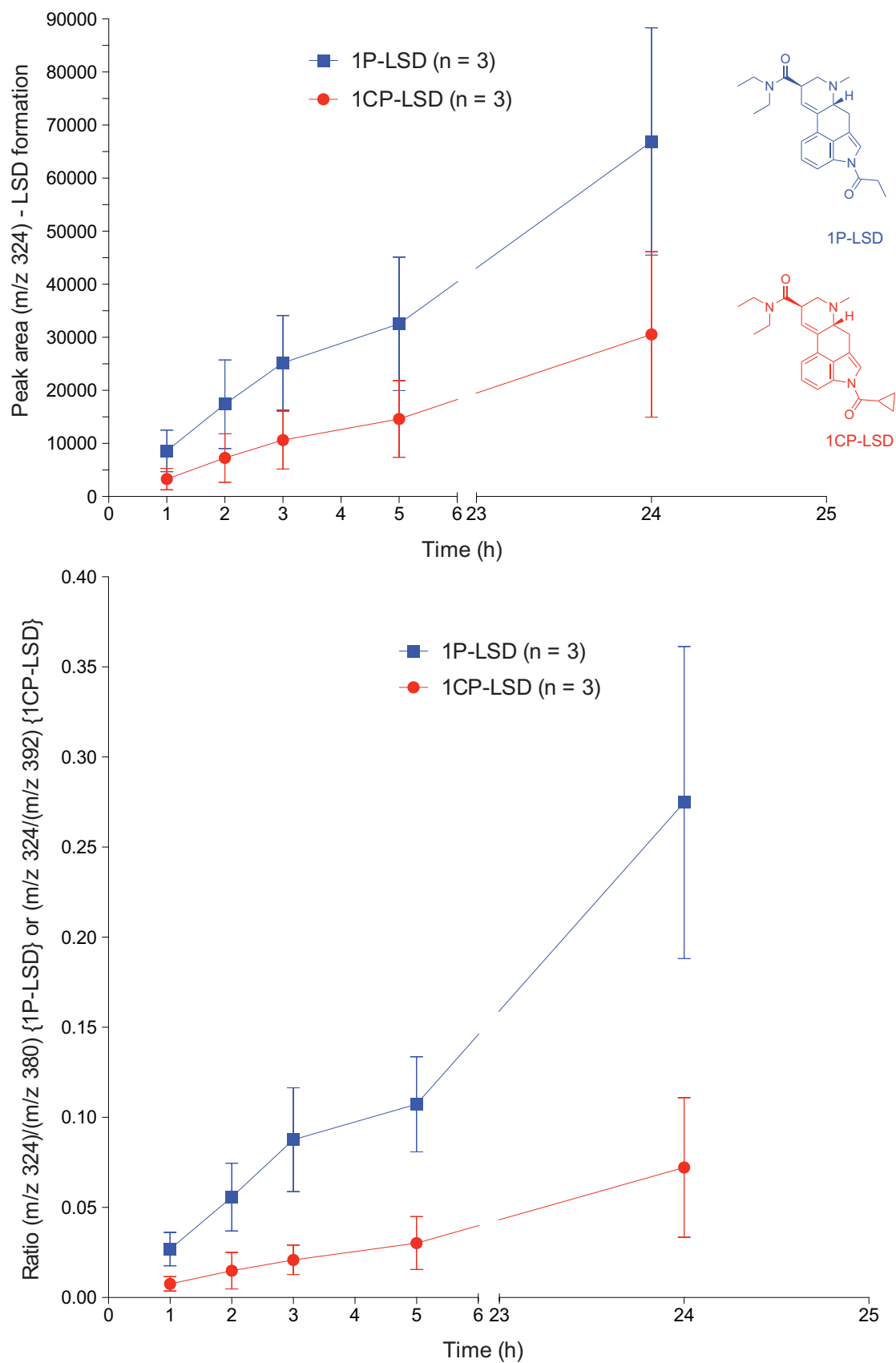
1CP-LSD (10 µg/mL) incubation in human serum (LC-ESI-SIM-MS)



Supporting Information – Drug Testing and Analysis



LSD formation after incubation of 1P-LSD and 1CP-LSD with human serum



Pellet and blotter extraction

The sample (pellet - labeled to contain 150 µg 1CP-LSD hemitartrate or blotter – labeled to contain 100 µg 1CP-LSD freebase) was sonicated with acetonitrile/water (1:1, containing 0.1 % formic acid (AWFA); 4 x 2 mL for 5 min each time), centrifuged (5,000 rpm for 3 min.) and the supernatant was collected. The supernatants were combined and centrifuged (5,000 rpm for 5 min). The supernatant was collected and made up to 10 mL with AWFA. An aliquot (100 µL) was diluted to 10 mL with AWFA. A 1CP-LSD standard curve (0.3750, 0.1875, 0.0938, 0.0469, 0.0234 and 0.0117 µg/per mL) was prepared in AWFA.

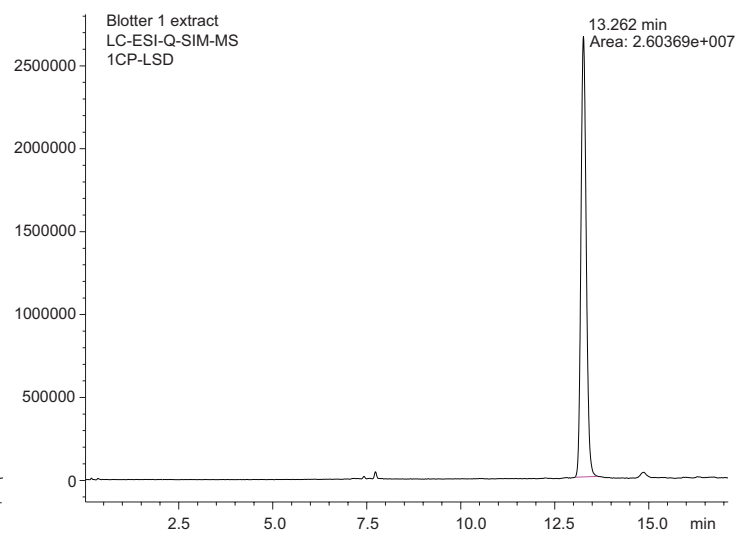
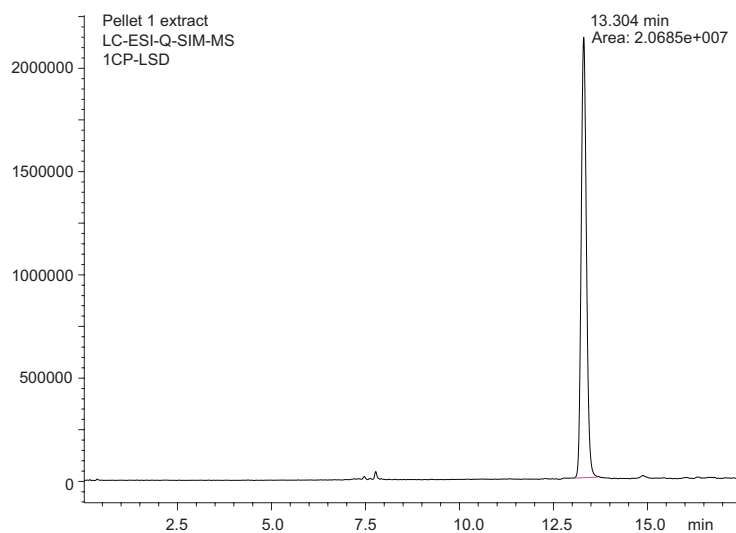
Results:

Pellet 1: 97 µg freebase (116 µg hemitartrate equivalent; pellet weight 14.9 mg)

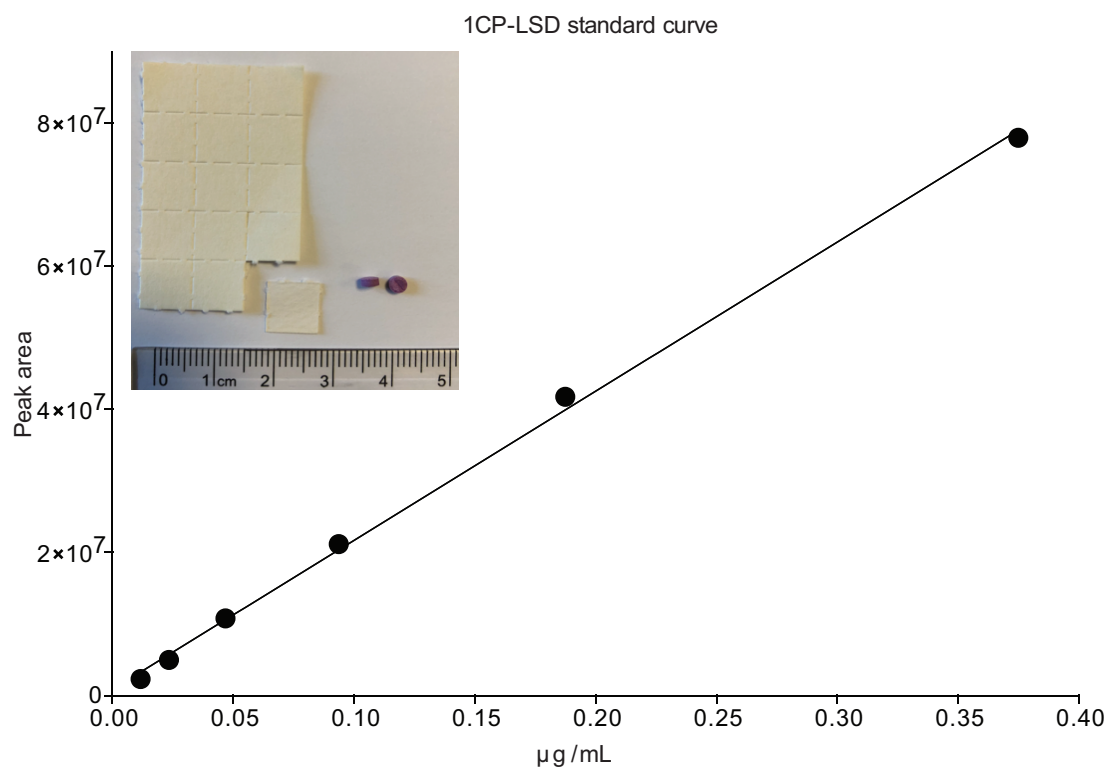
Pellet 2: 105 µg freebase (125 µg hemitartrate equivalent; pellet weight 14.2 mg)

Blotter 1: 120 µg freebase (blotter weight 16.0 mg)

Blotter 2: 112 µg freebase (blotter weight 15.5 mg)



Supporting Information – Drug Testing and Analysis



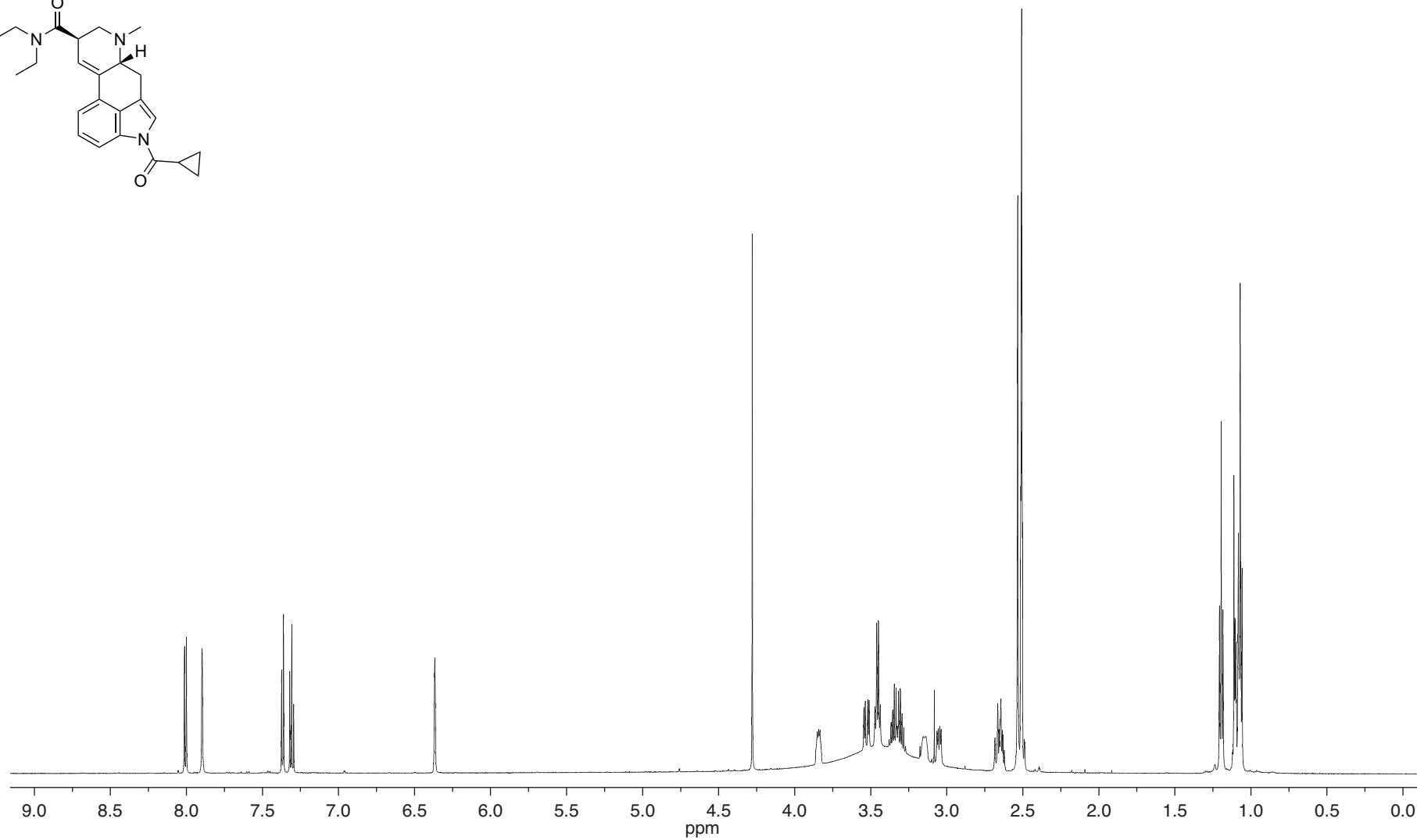
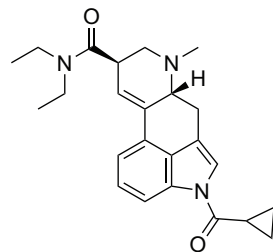
	$\mu\text{g/mL}$		Peak area		
	X	Err. Bar	Y1	Y2	Y3
	0.011719		2311890.00000	2343940.00000	2307770.00000
	0.023438		4941250.00000	5029580.00000	5006670.00000
	0.046875		1.05214e+007	1.14813e+007	1.04113e+007
	0.093750		2.10664e+007	2.12056e+007	2.12797e+007
	0.187500		4.17822e+007	4.19014e+007	4.15572e+007
	0.375000		7.87946e+007	7.75800e+007	7.74465e+007
Pellet 1			2.06850e+007	2.12446e+007	2.12866e+007
Pellet 2			2.25967e+007	2.27479e+007	2.31372e+007
Blotter 1			2.60369e+007	2.59435e+007	2.54800e+007
Blotter 2			2.41236e+007	2.41803e+007	2.41447e+007

	Peak area
Slope	$2.083\text{e}+008 \pm 2.163\text{e}+006$
Y-intercept when X=0.0	861720 ± 382333
X-intercept when Y=0.0	-0.004136
1/slope	4.800e-009
95% Confidence Intervals	
Slope	2.038e+008 to 2.129e+008
Y-intercept when X=0.0	51174 to 1.672e+006
X-intercept when Y=0.0	-0.008153 to -0.0002419
Goodness of Fit	
R square	0.9983

	$\mu\text{g/mL}$	Peak area
Pellet 1	0.097	2.107e+007
Pellet 2	0.105	2.283e+007
Blotter 1	0.120	2.582e+007
Blotter 2	0.112	2.415e+007

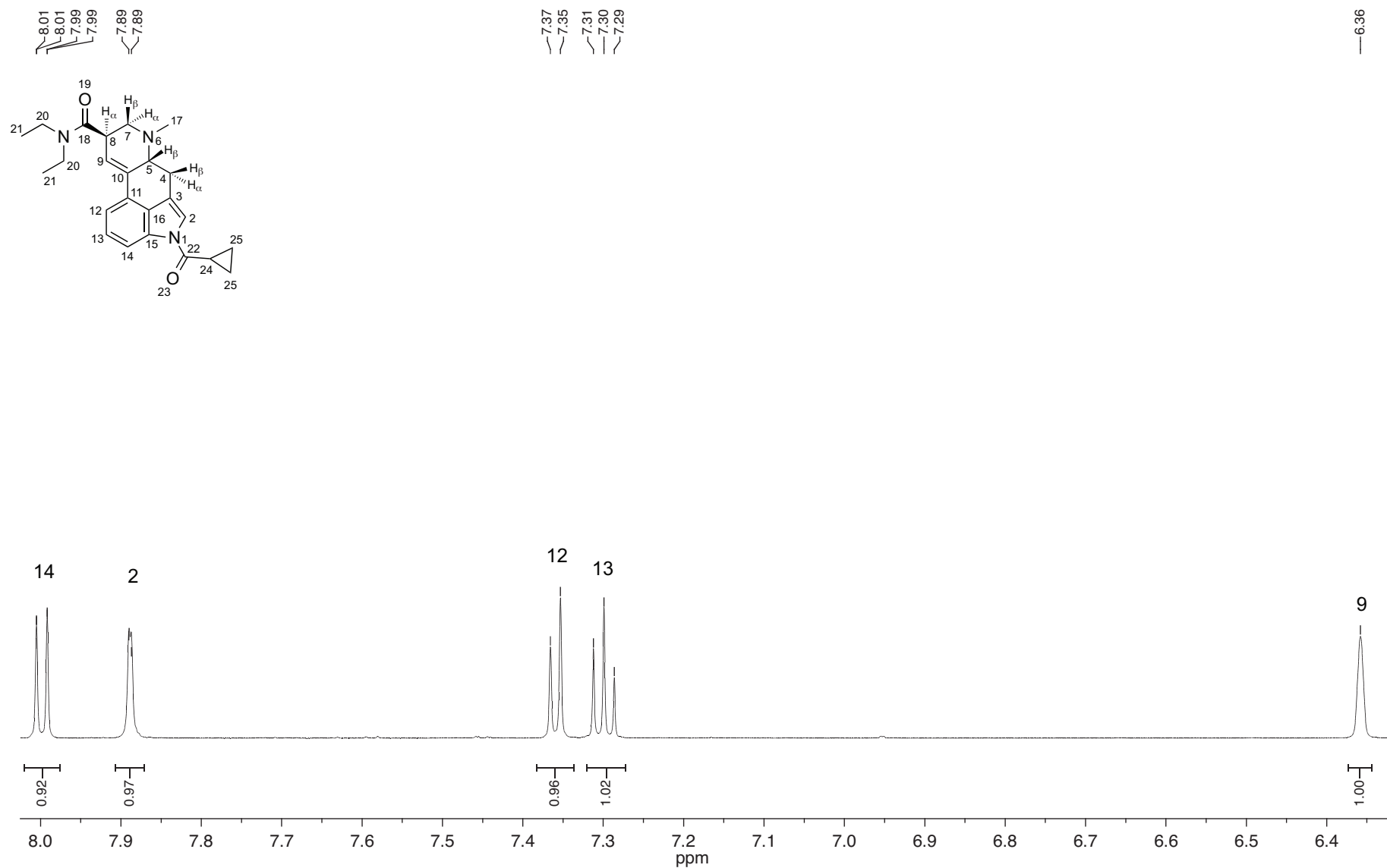
Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
 ^1H -NMR (600 MHz)
 d_6 -DMSO



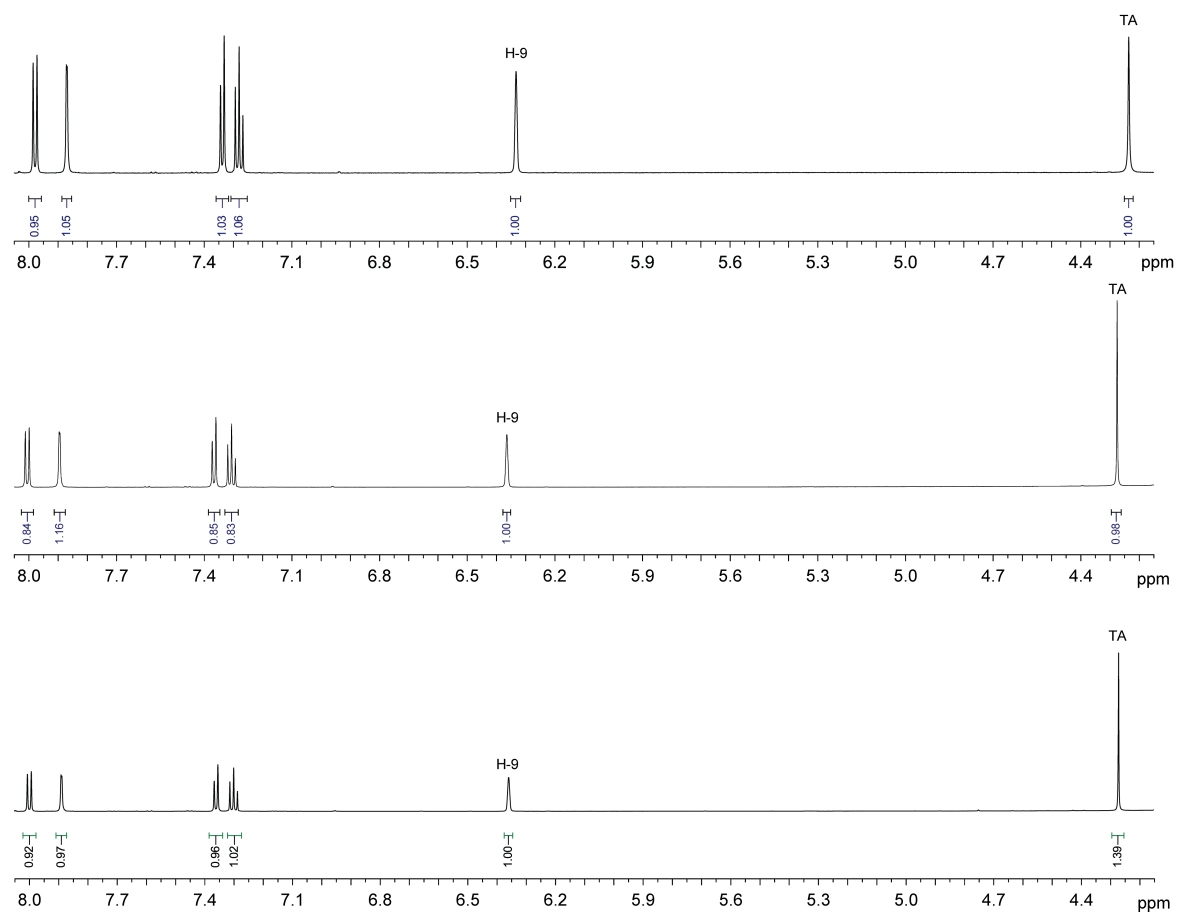
Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
¹H-NMR (600 MHz)
d₆-DMSO



Supporting Information – Drug Testing and Analysis

1CP-LSD:tartaric acid (TA) integration ratios



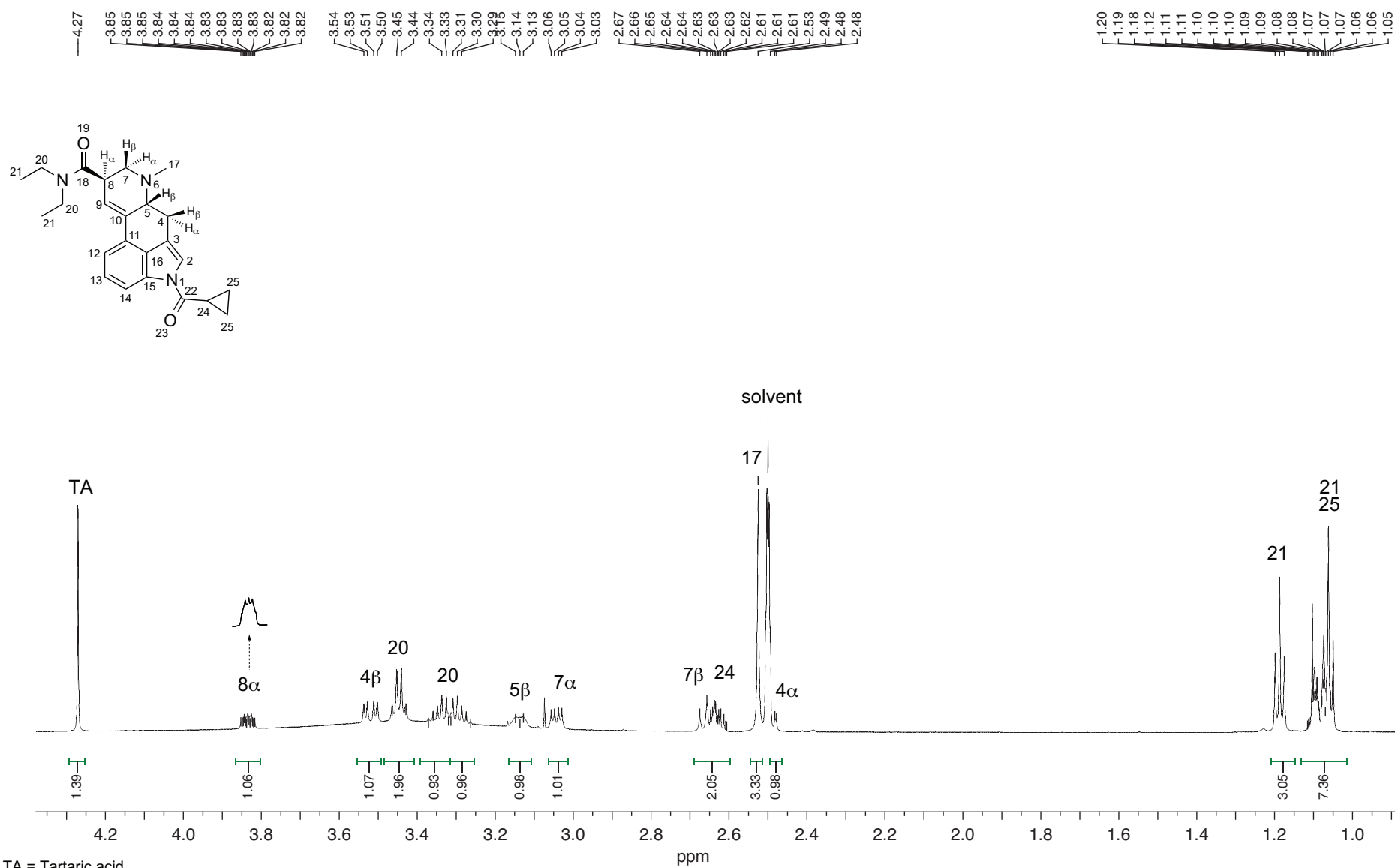
	1CPL:TA int. ratio	1CPL:TA mole ratio	MW	% 1CP-LSD
Analysis 1	1.00:1.00	1.00:0.50	466.57	83.92
Analysis 2	1.00:0.98	1.00:0.49	465.06	84.19
Analysis 3	1.00:1.39	1:0.69(5)	495.83	78.96
Mean			475.82	82.35

Supporting Information – Drug Testing and Analysis

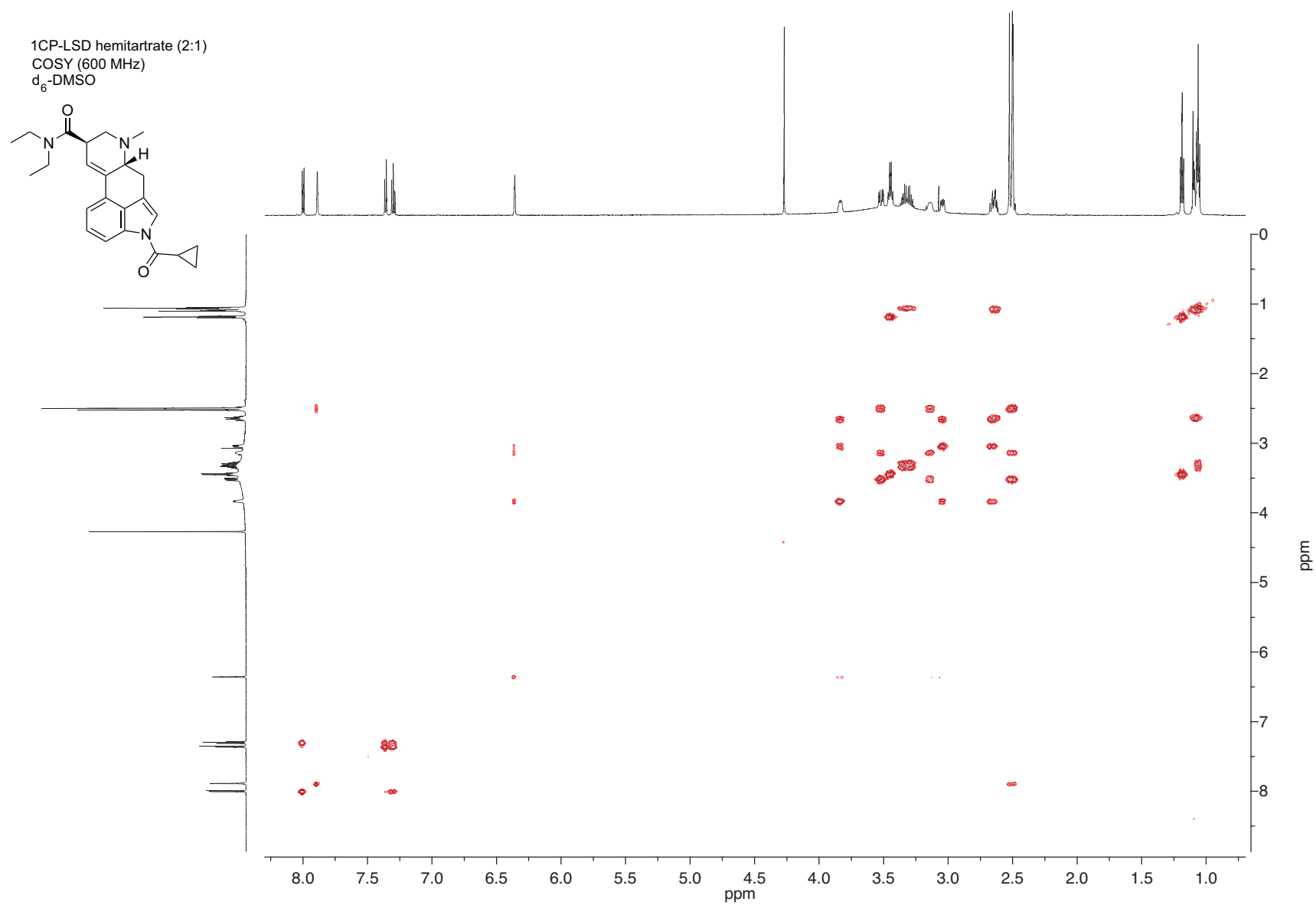
1CP-LSD hemitartrate (2:1)

¹H-NMR (600 MHz)

d₆-DMSO

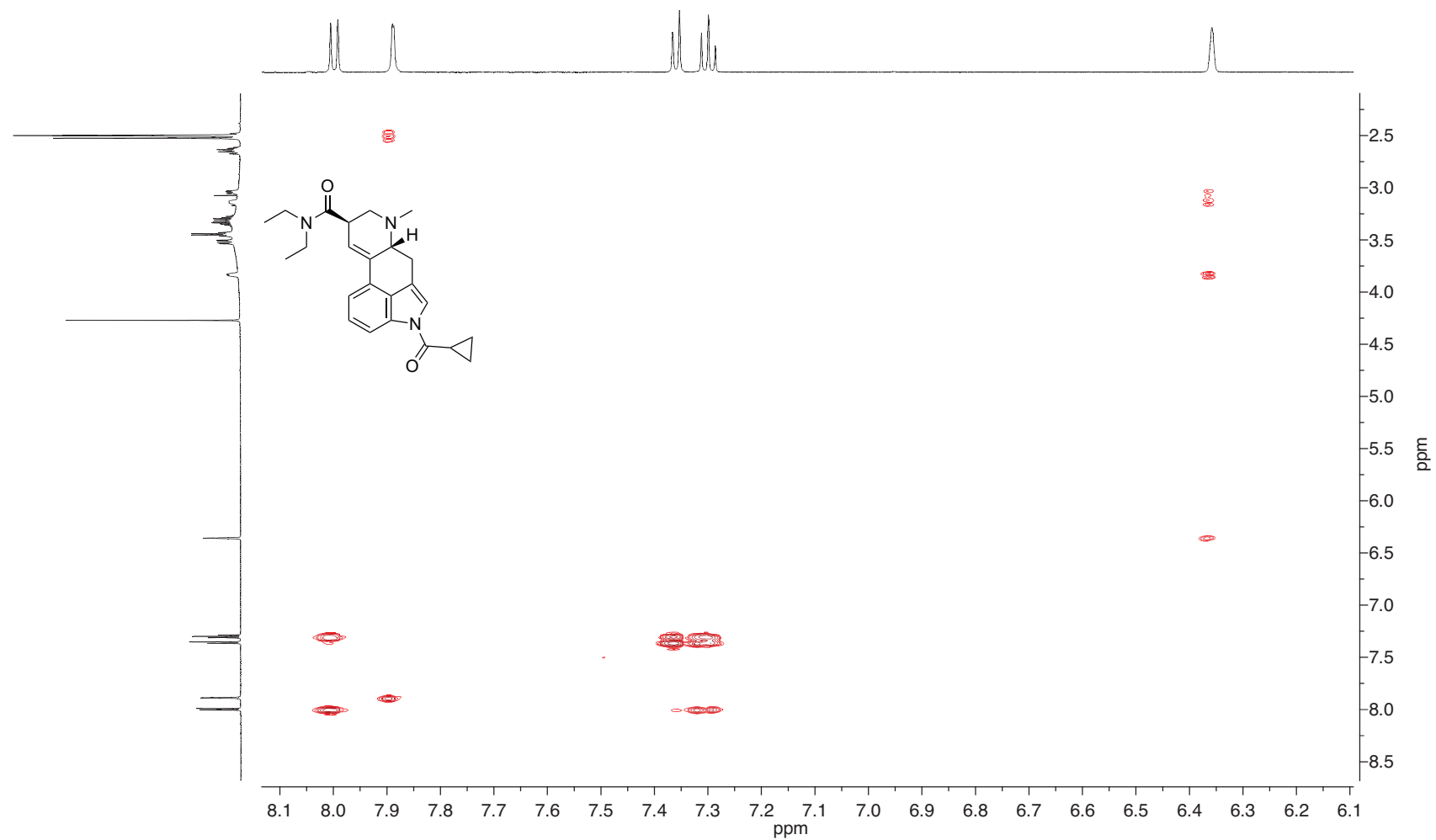


Supporting Information – Drug Testing and Analysis



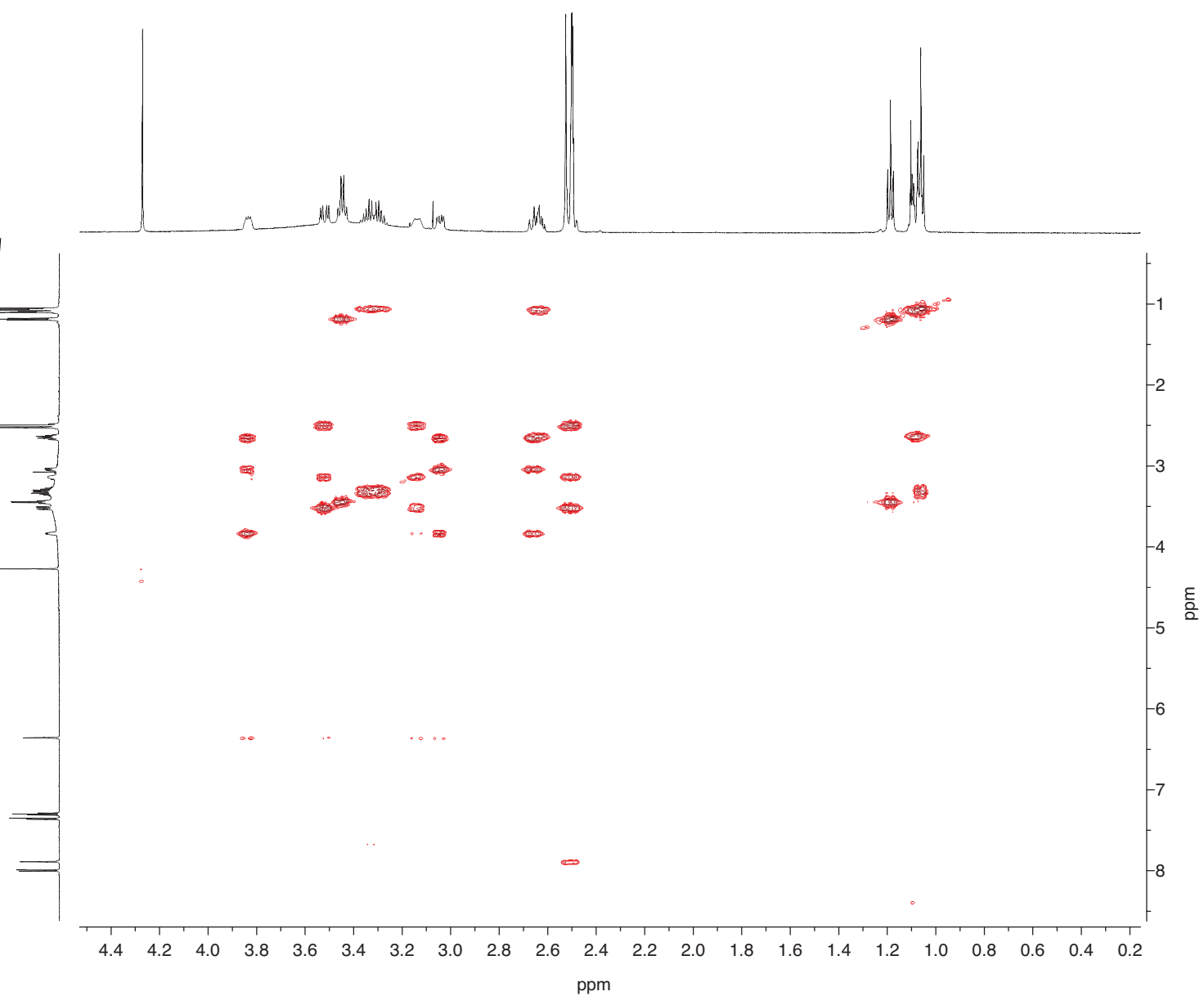
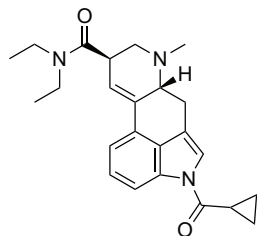
Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
COSY (600 MHz)
d₆-DMSO



Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
COSY (600 MHz)
d₆-DMSO

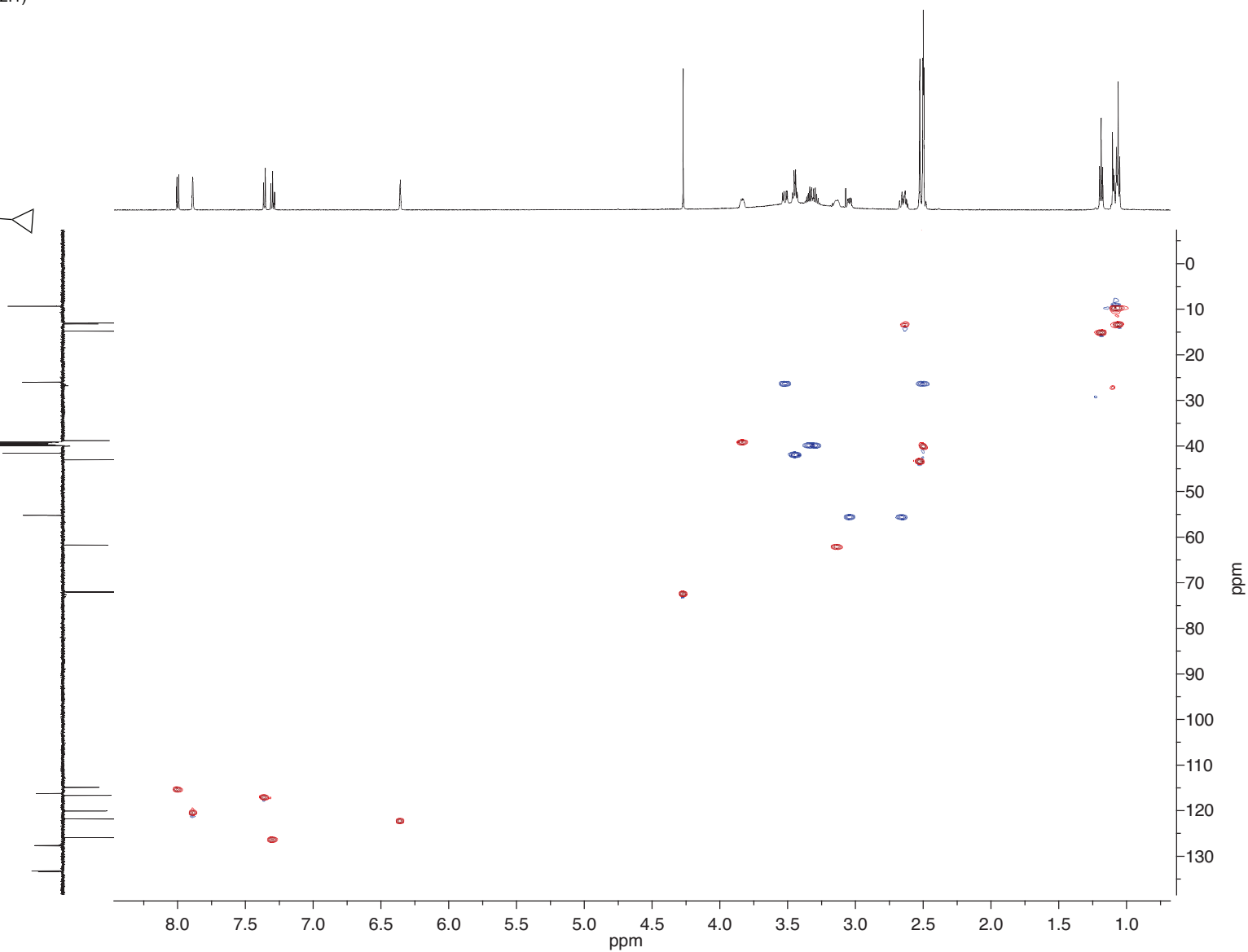
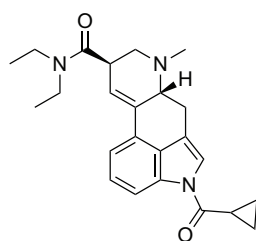


1CP-LSD hemitartrate (2:1)
¹³C DEPTQ (150 MHz)
d₆-DMSO



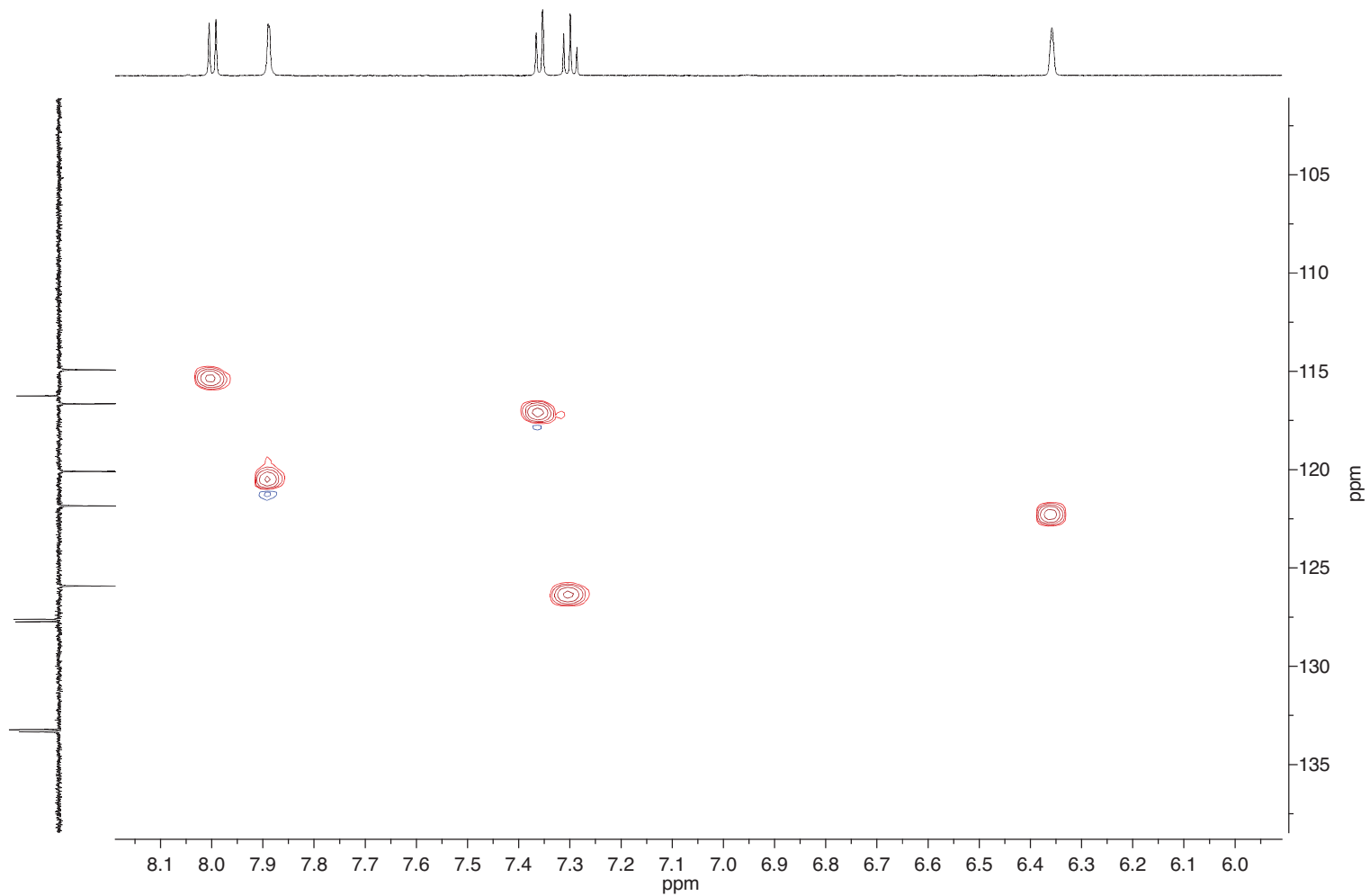
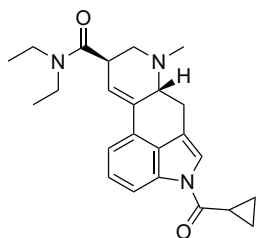
Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
HSQC
d₆-DMSO



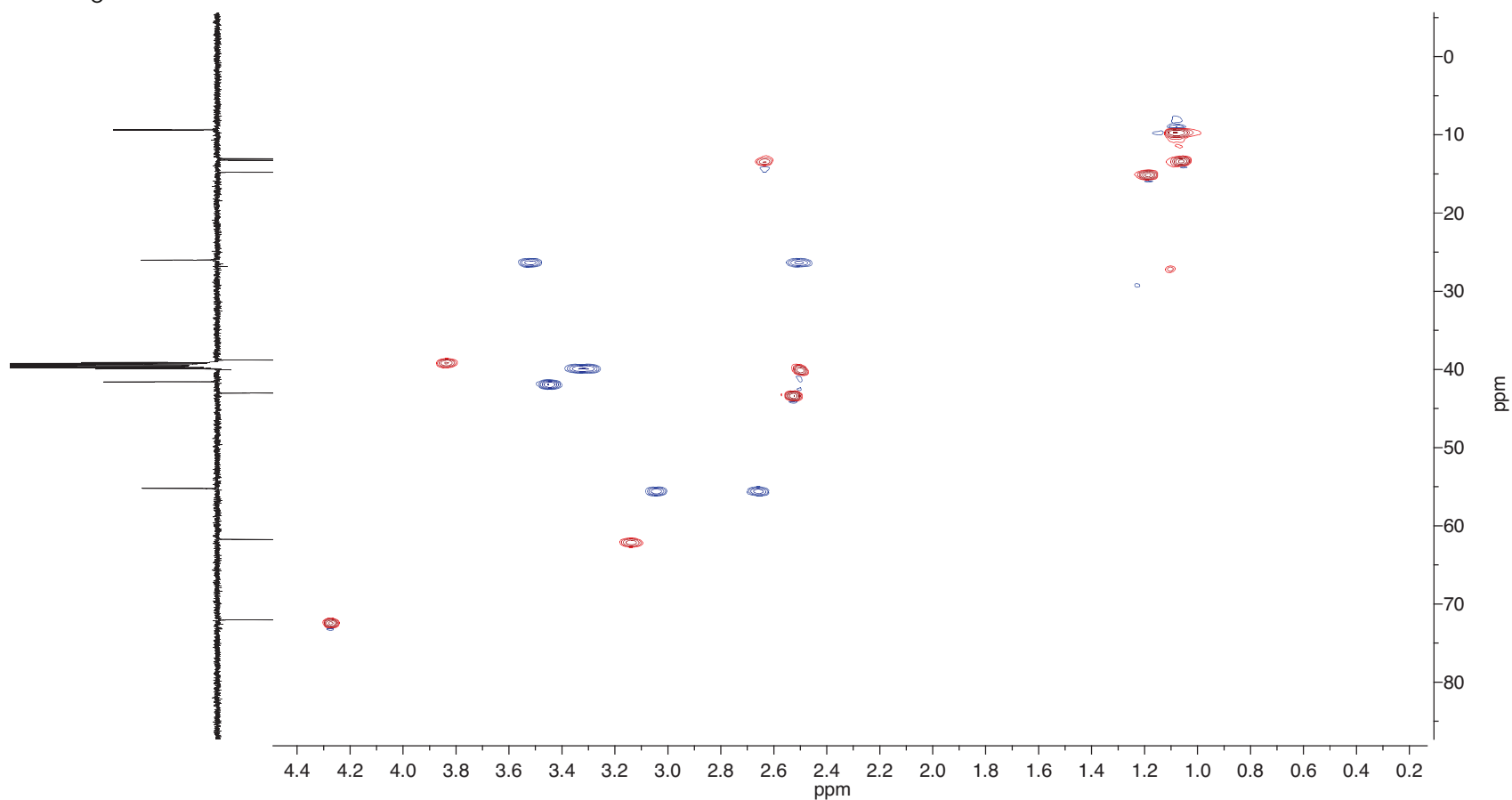
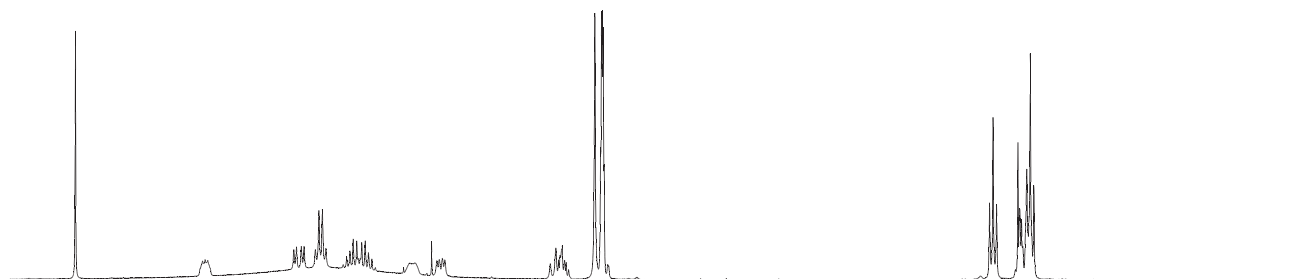
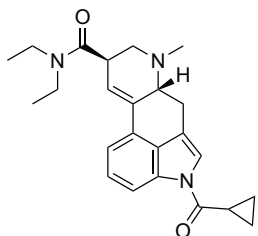
Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
HSQC
d₆-DMSO



Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)
HSQC
d₆-DMSO

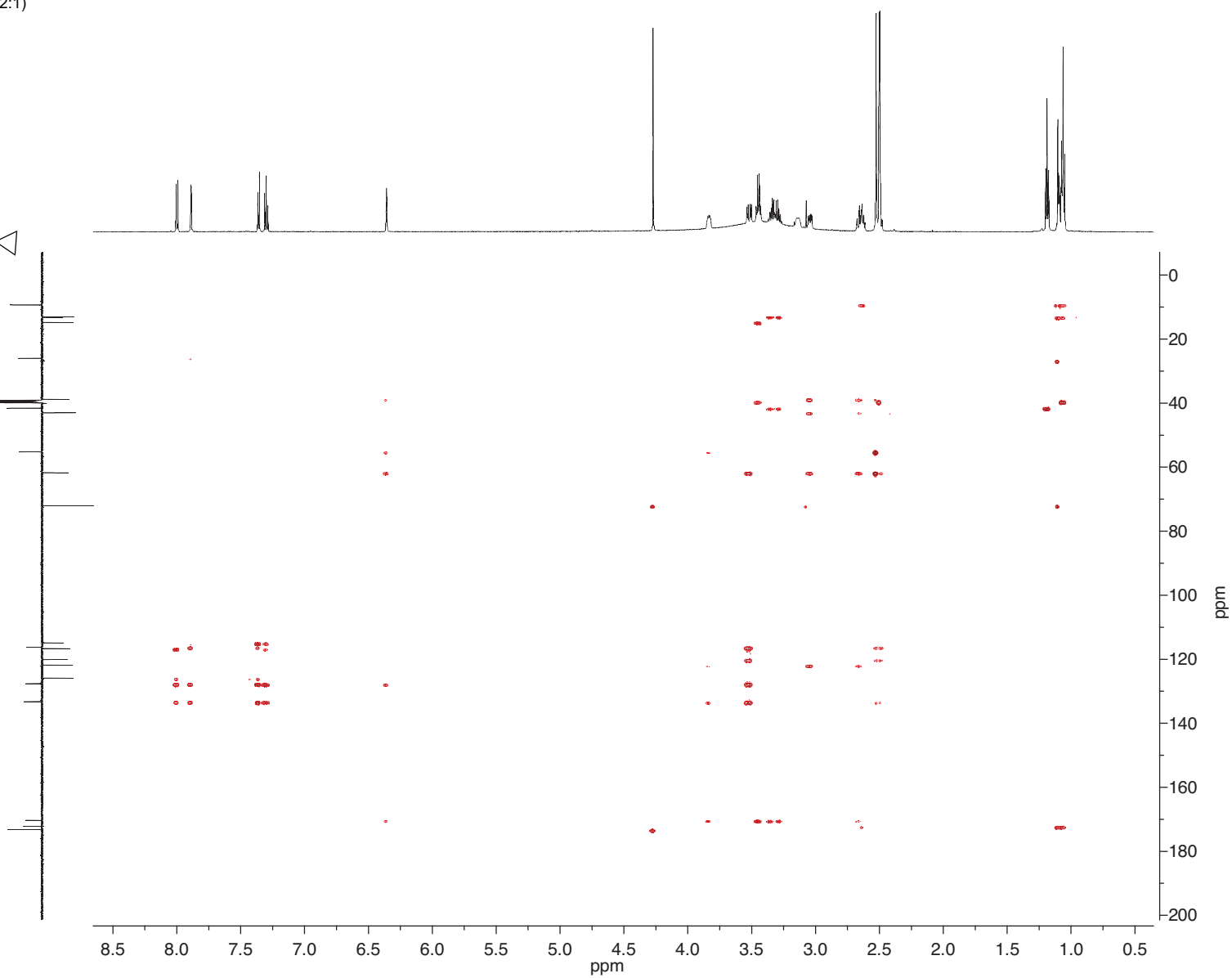
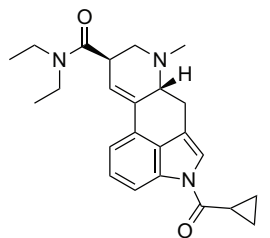


Supporting Information – Drug Testing and Analysis

1CP-LSD hemitartrate (2:1)

HMBC

d₆-DMSO

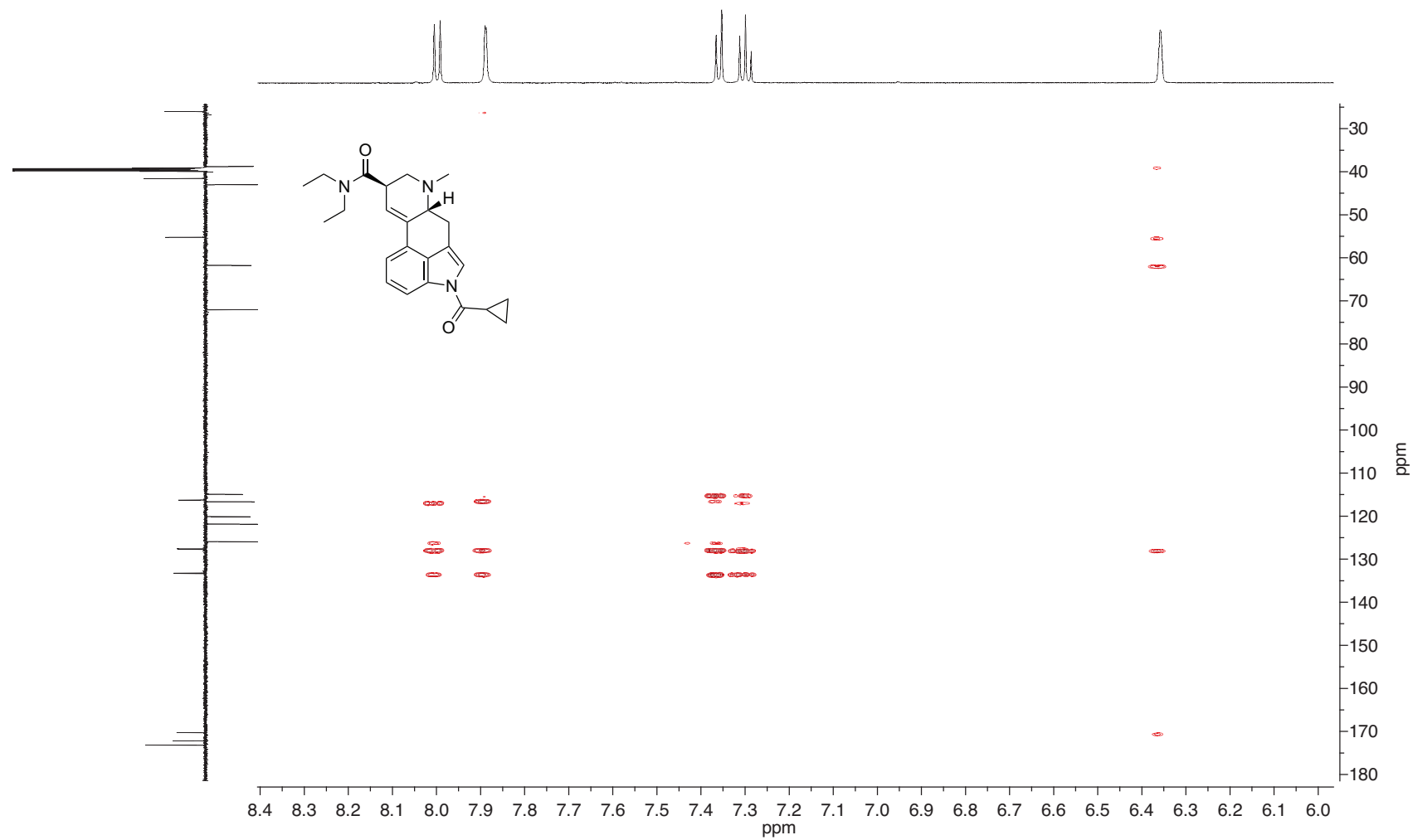


Supporting Information – Drug Testing and Analysis

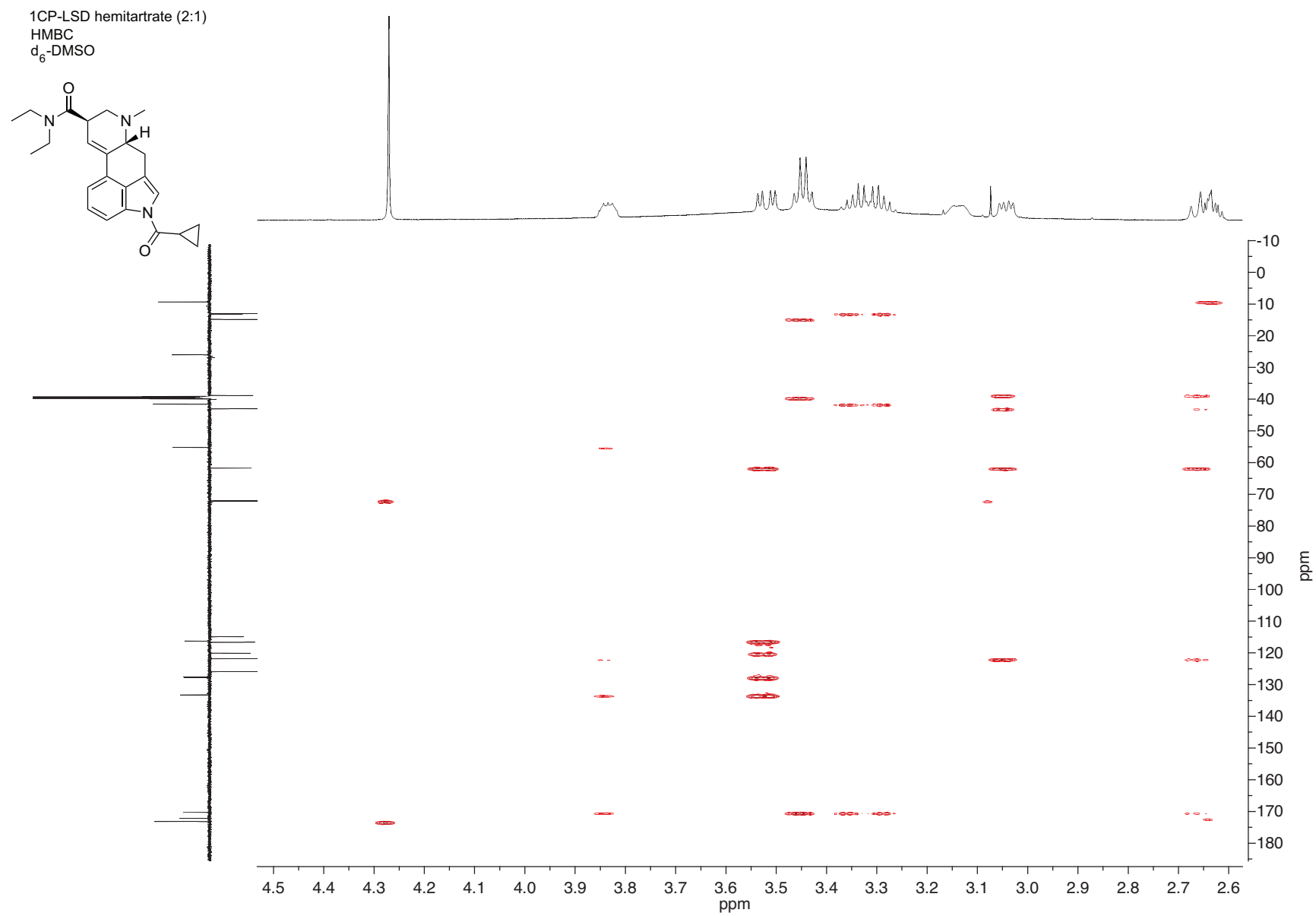
1CP-LSD hemitartrate (2:1)

HMBC

d_6 -DMSO

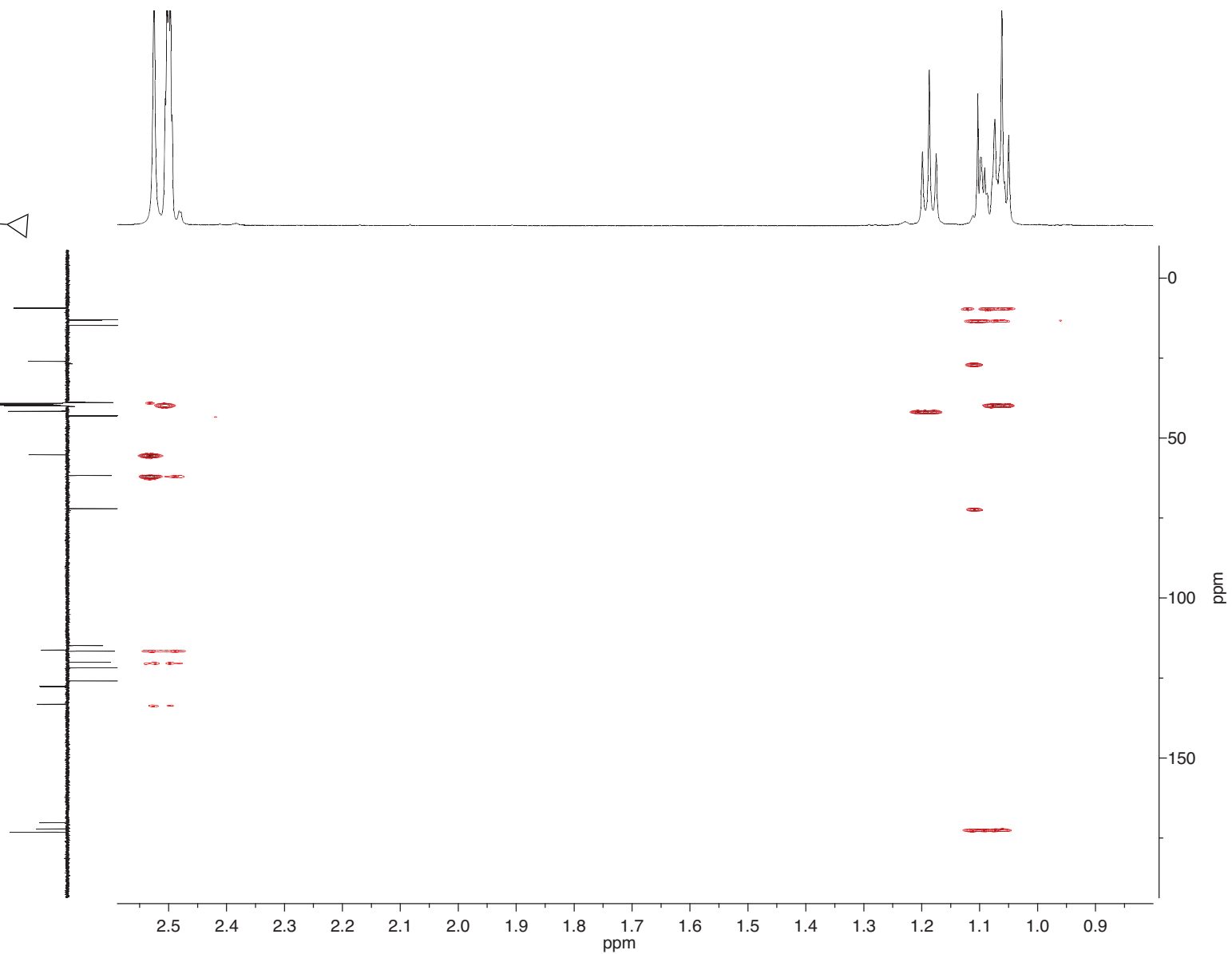
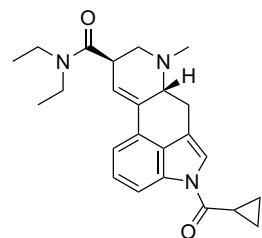


Supporting Information – Drug Testing and Analysis

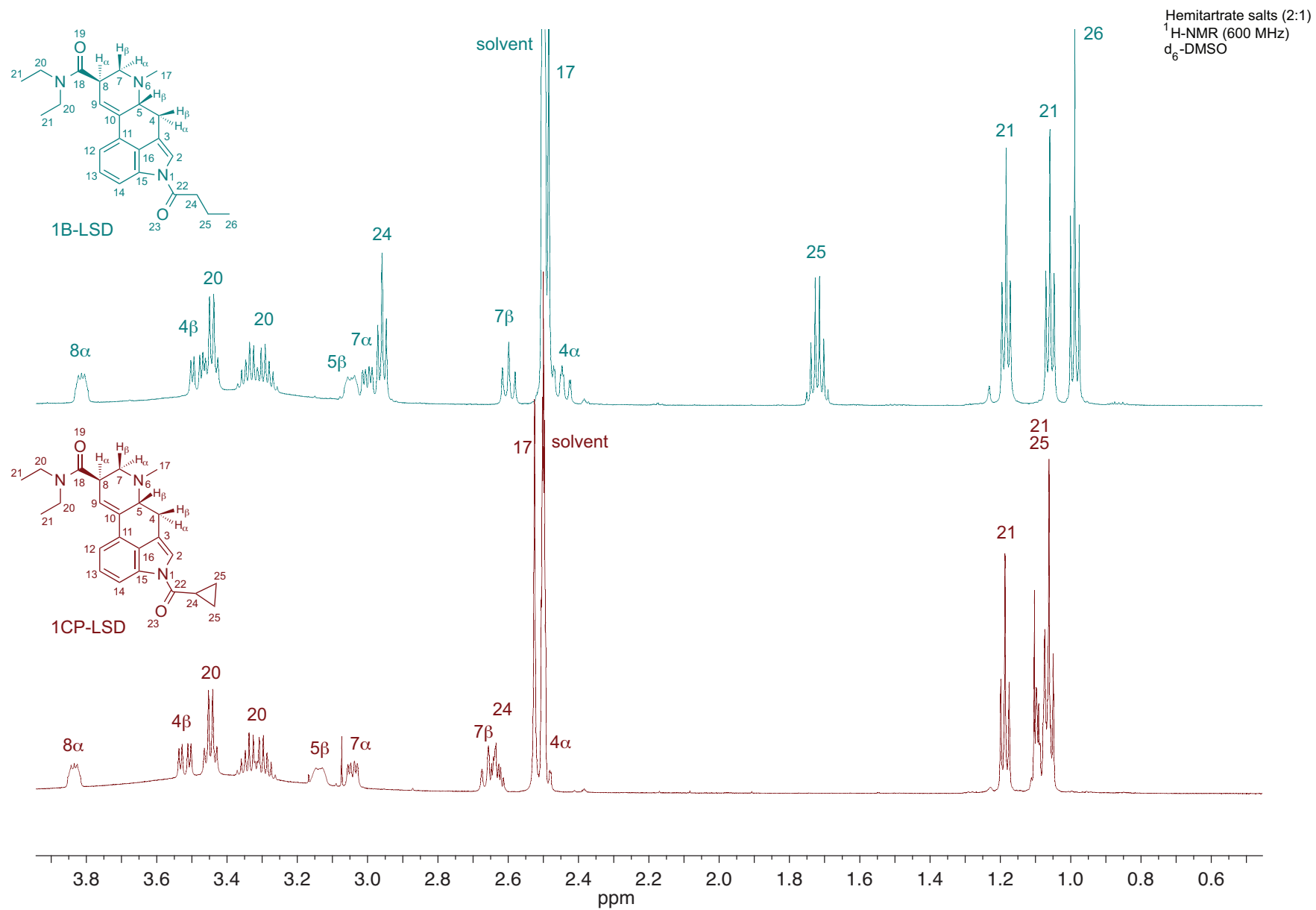


Supporting Information – Drug Testing and Analysis

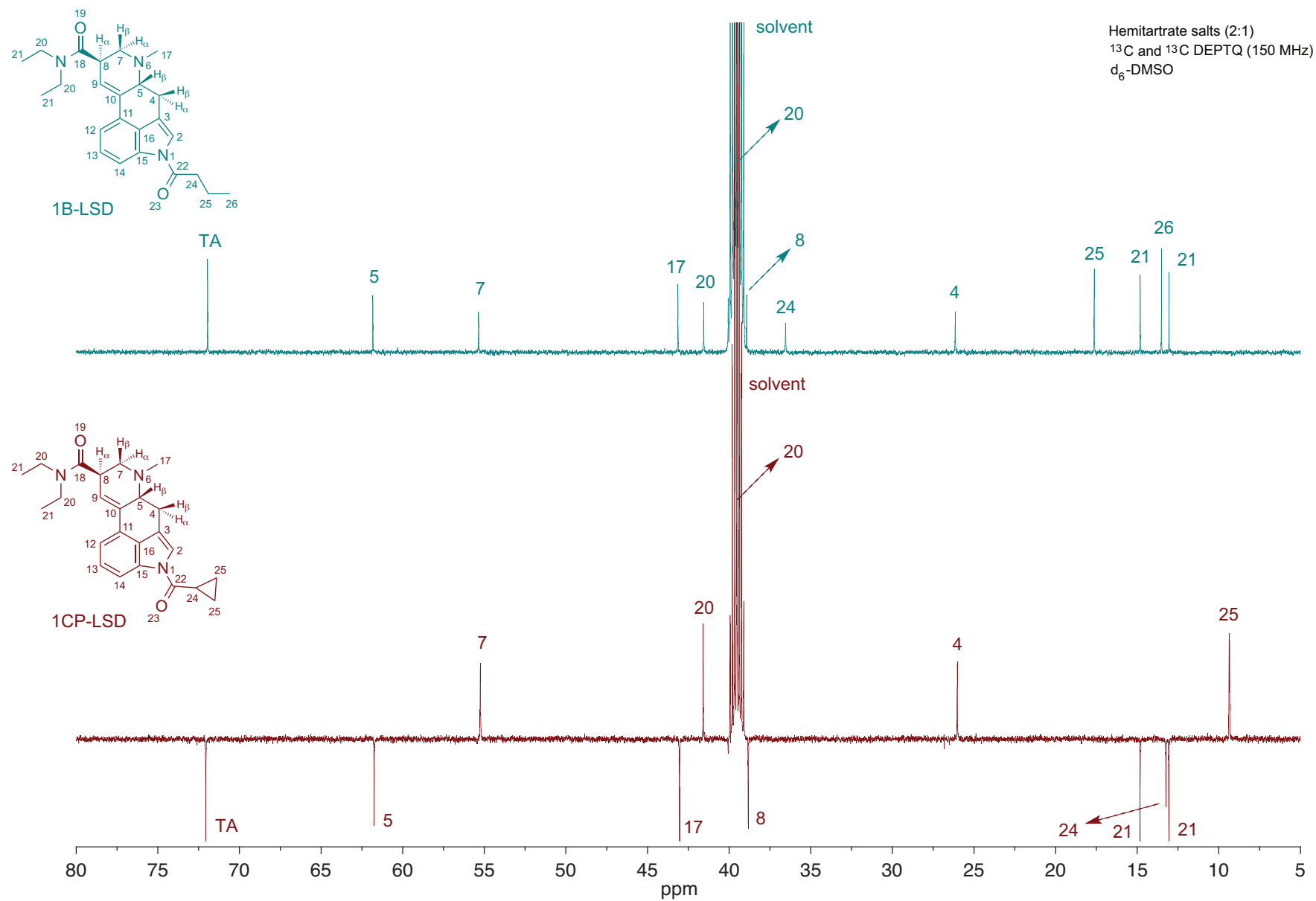
1CP-LSD hemitartrate (2:1)
HMBC
d₆-DMSO



Supporting Information – Drug Testing and Analysis

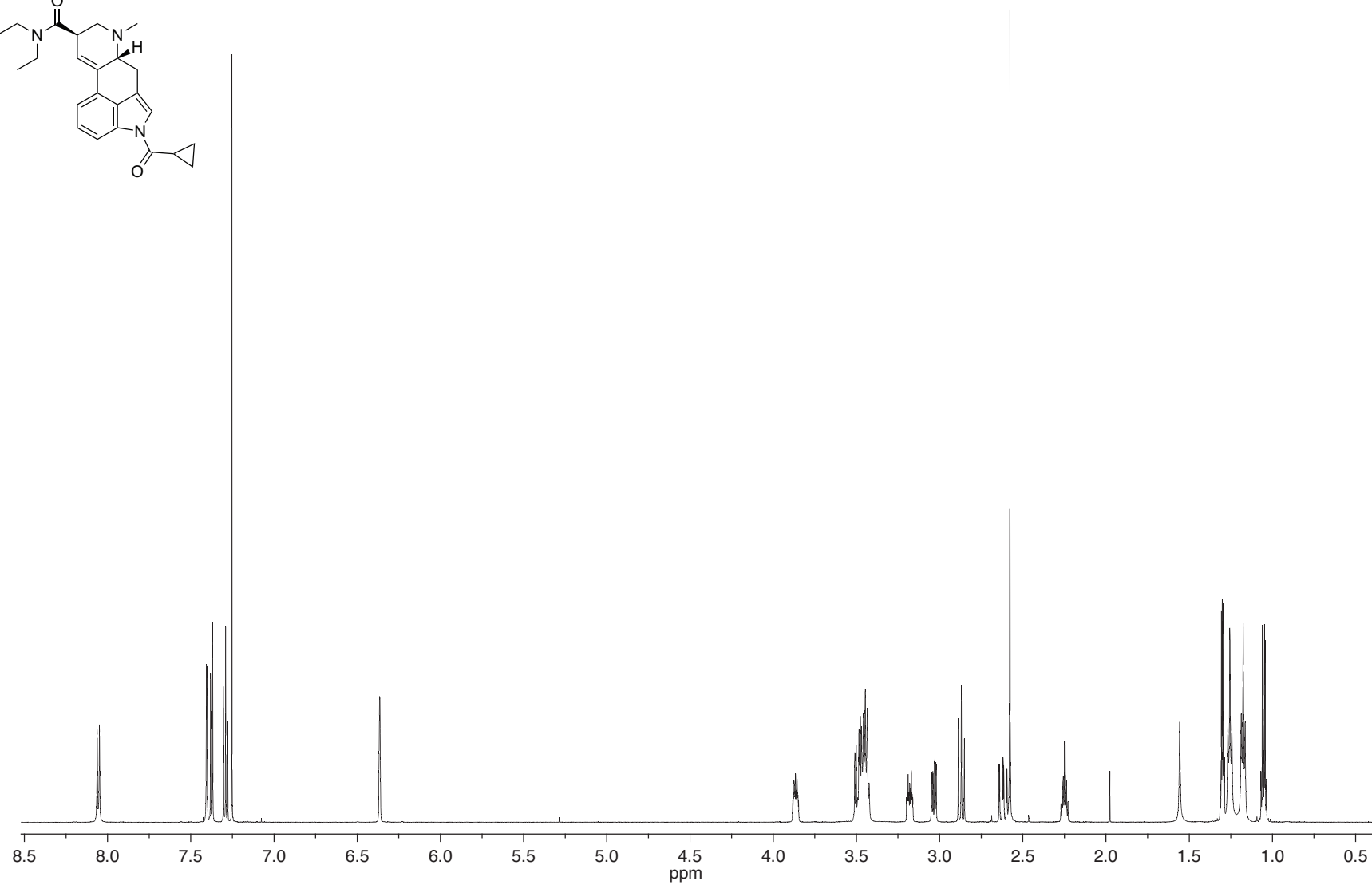
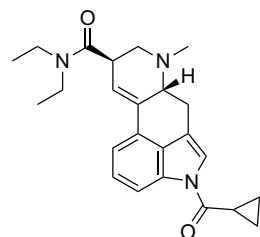


Supporting Information – Drug Testing and Analysis

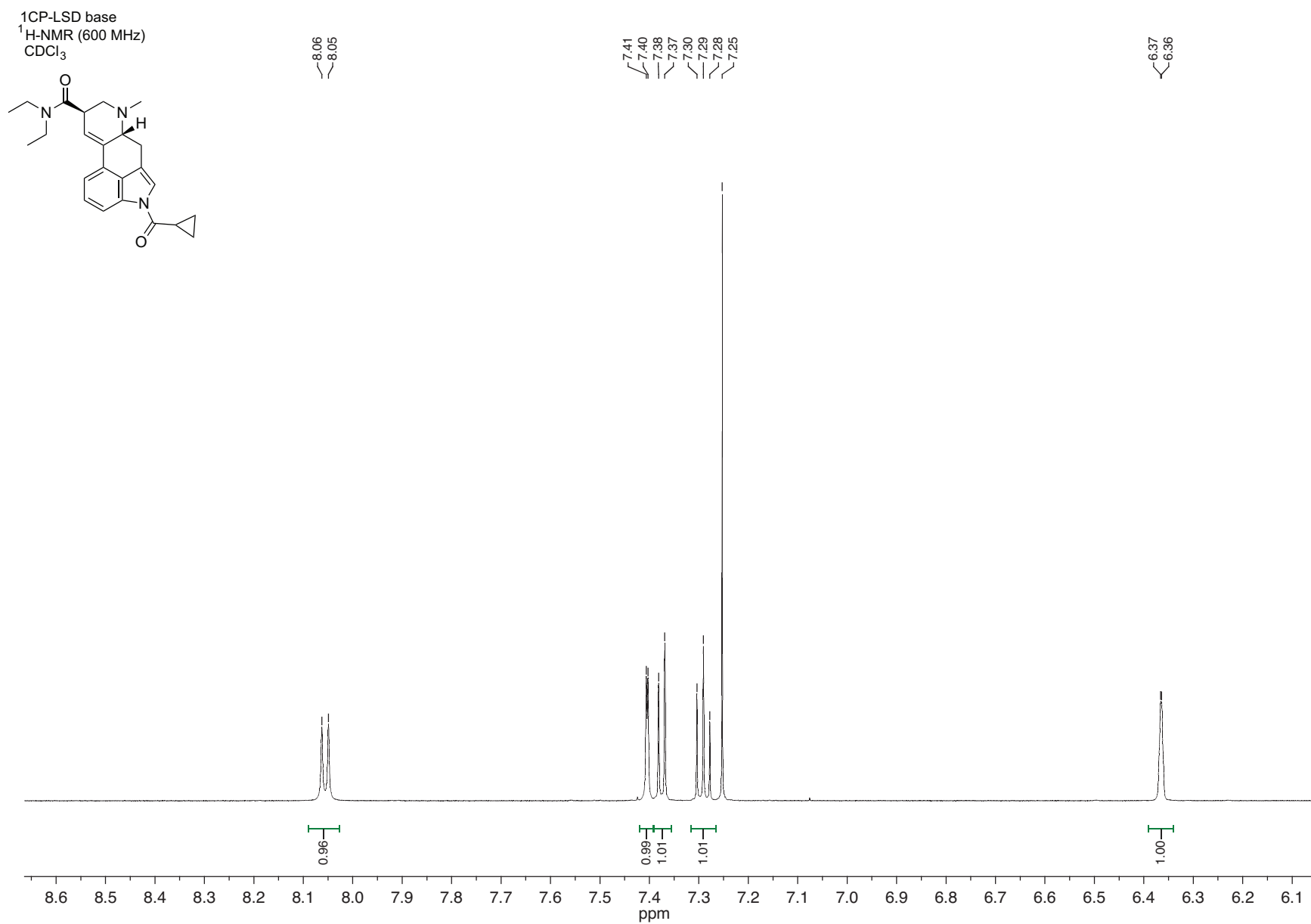


Supporting Information – Drug Testing and Analysis

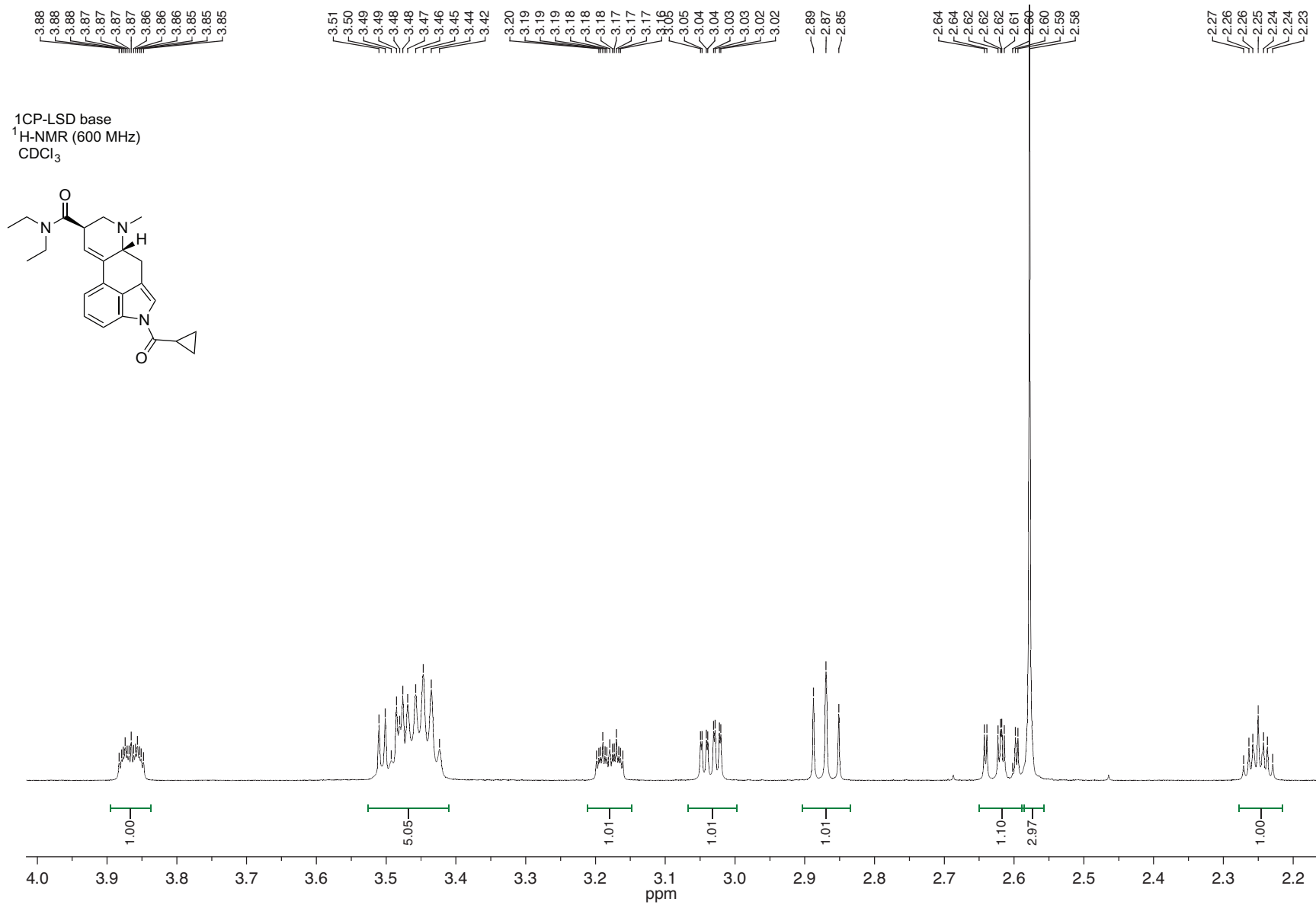
1CP-LSD base
¹H-NMR (600 MHz)
CDCl₃



Supporting Information – Drug Testing and Analysis

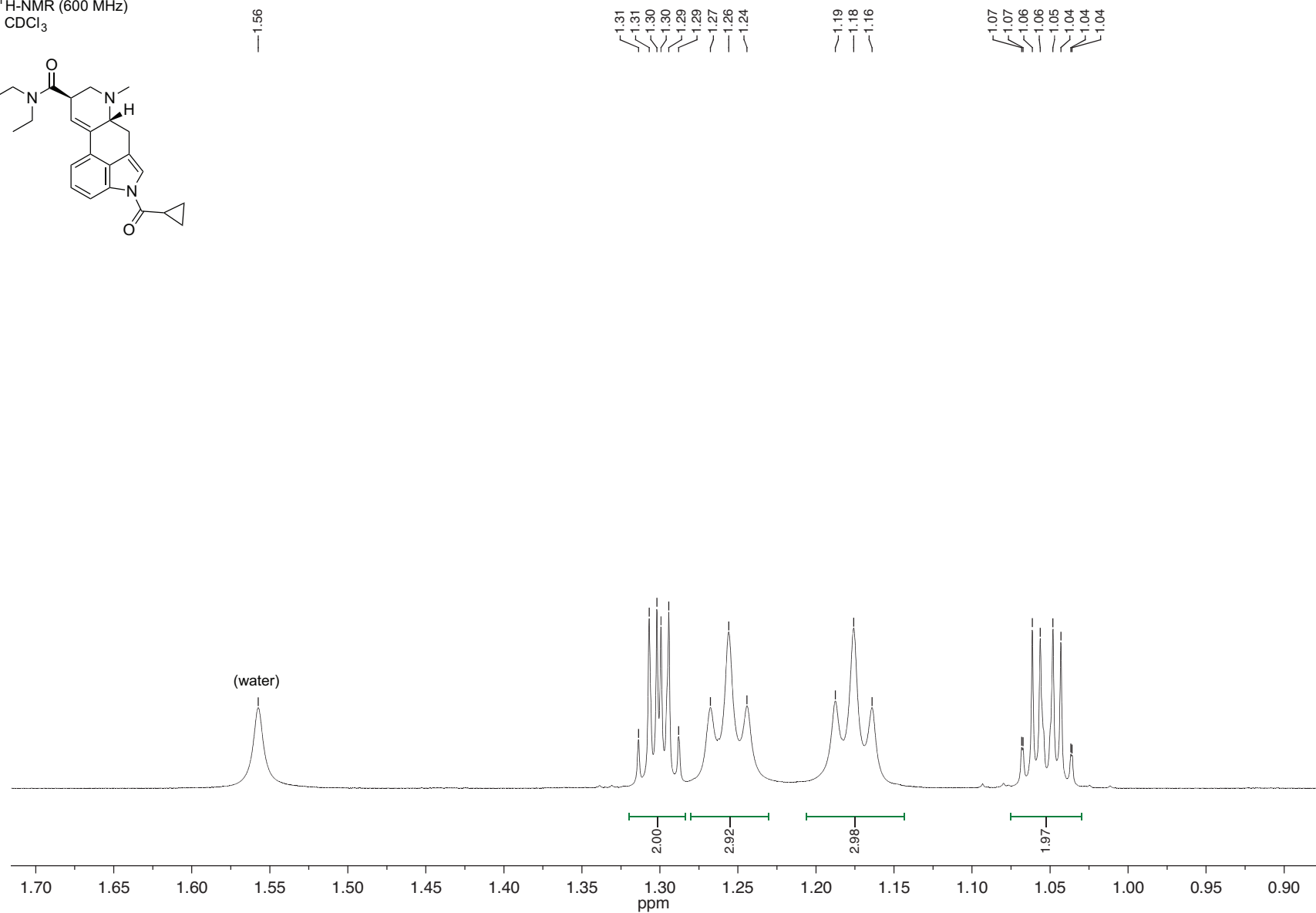
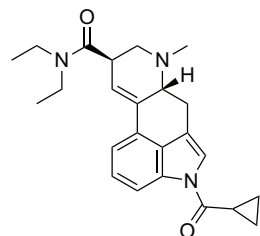


Supporting Information – Drug Testing and Analysis



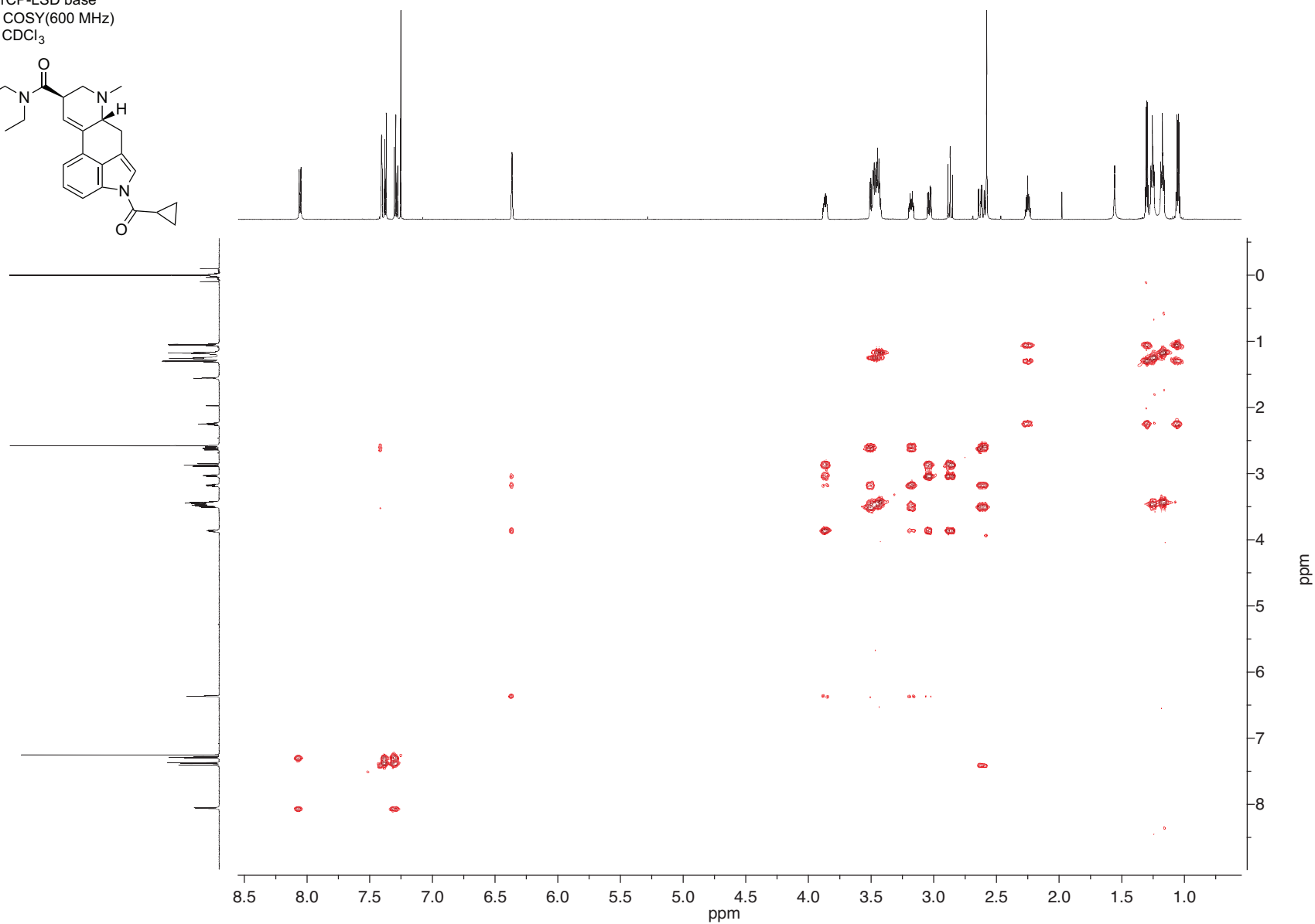
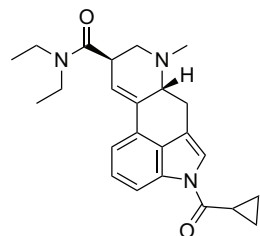
Supporting Information – Drug Testing and Analysis

1CP-LSD base
¹H-NMR (600 MHz)
 CDCl₃

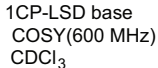


Supporting Information – Drug Testing and Analysis

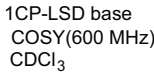
1CP-LSD base
COSY(600 MHz)
CDCl₃



Supporting Information – Drug Testing and Analysis

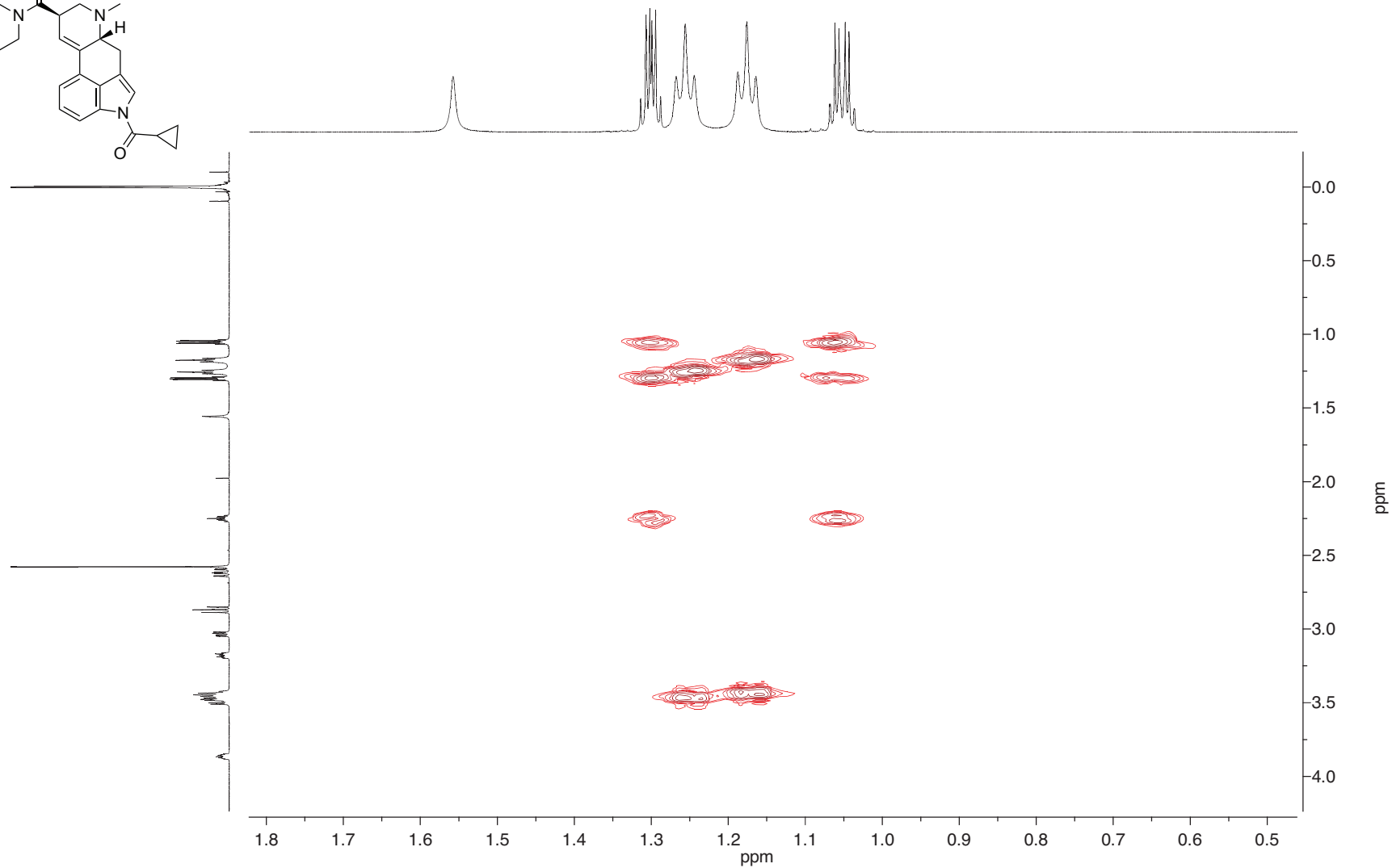
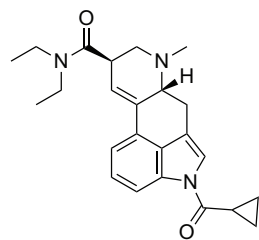


Supporting Information – Drug Testing and Analysis



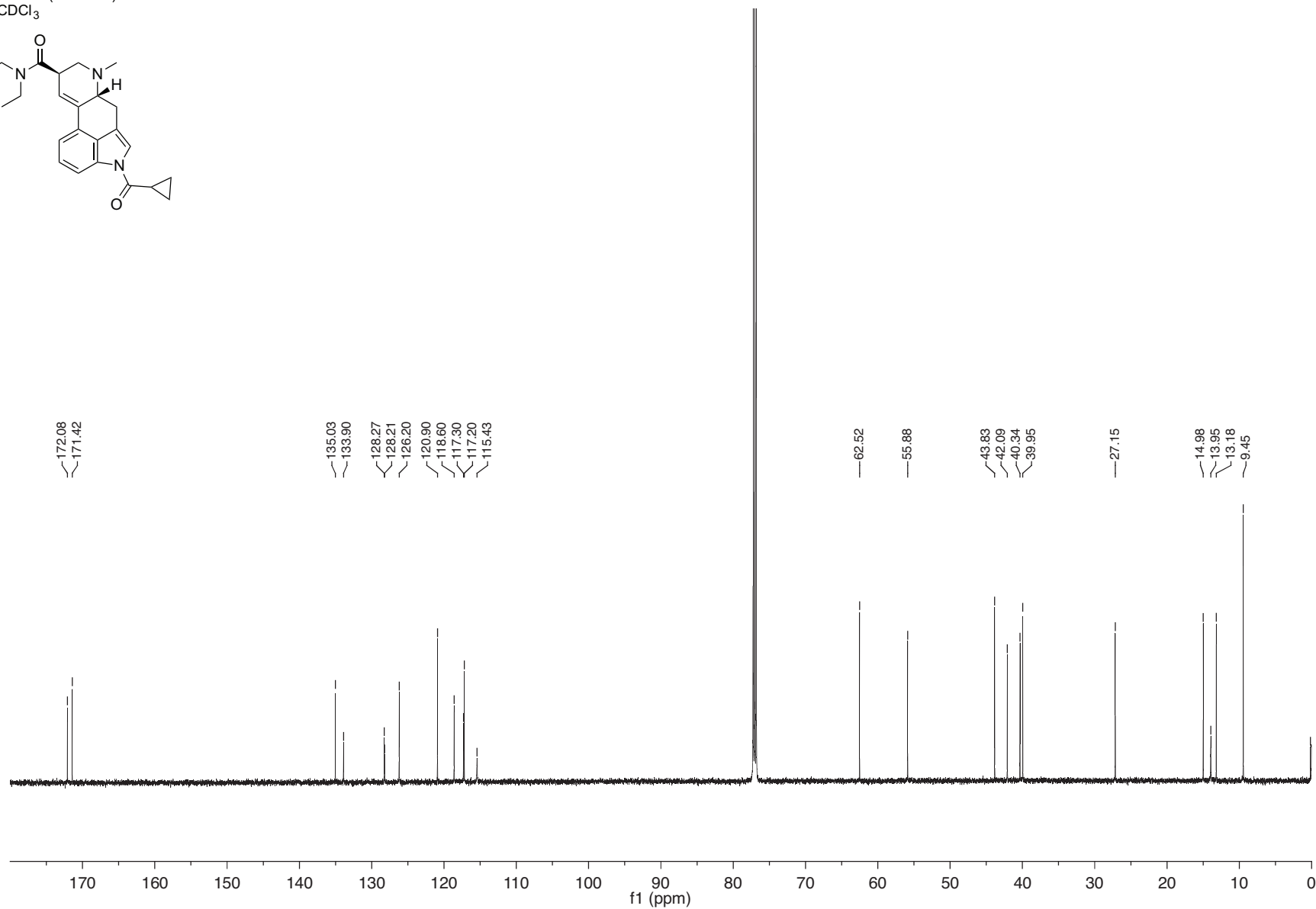
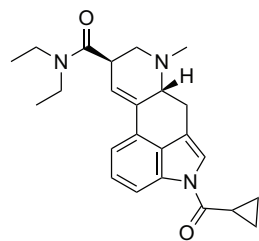
Supporting Information – Drug Testing and Analysis

1CP-LSD base
COSY(600 MHz)
CDCl₃



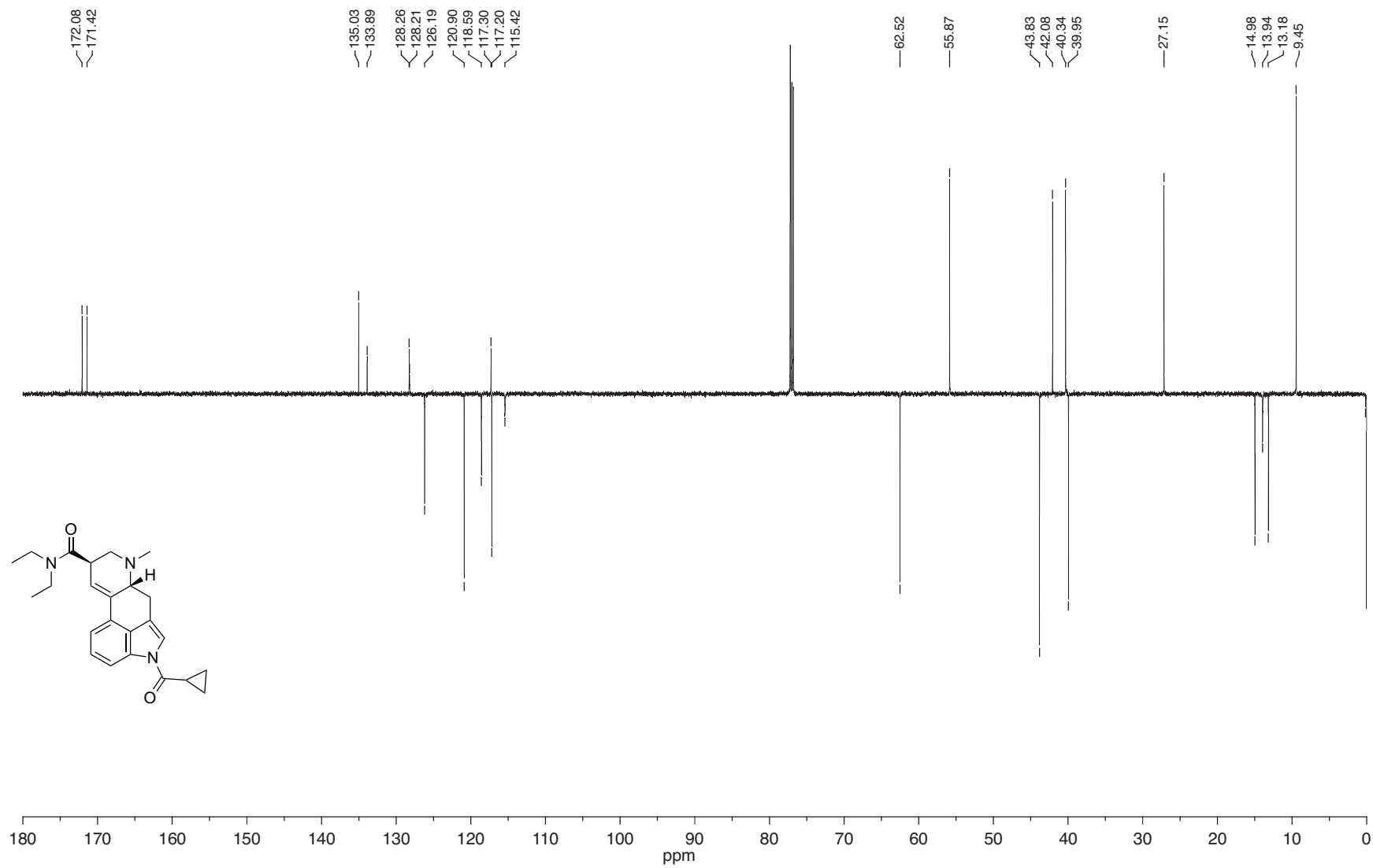
Supporting Information – Drug Testing and Analysis

1CP-LSD base
¹³C NMR (150 MHz)
 CDCl₃



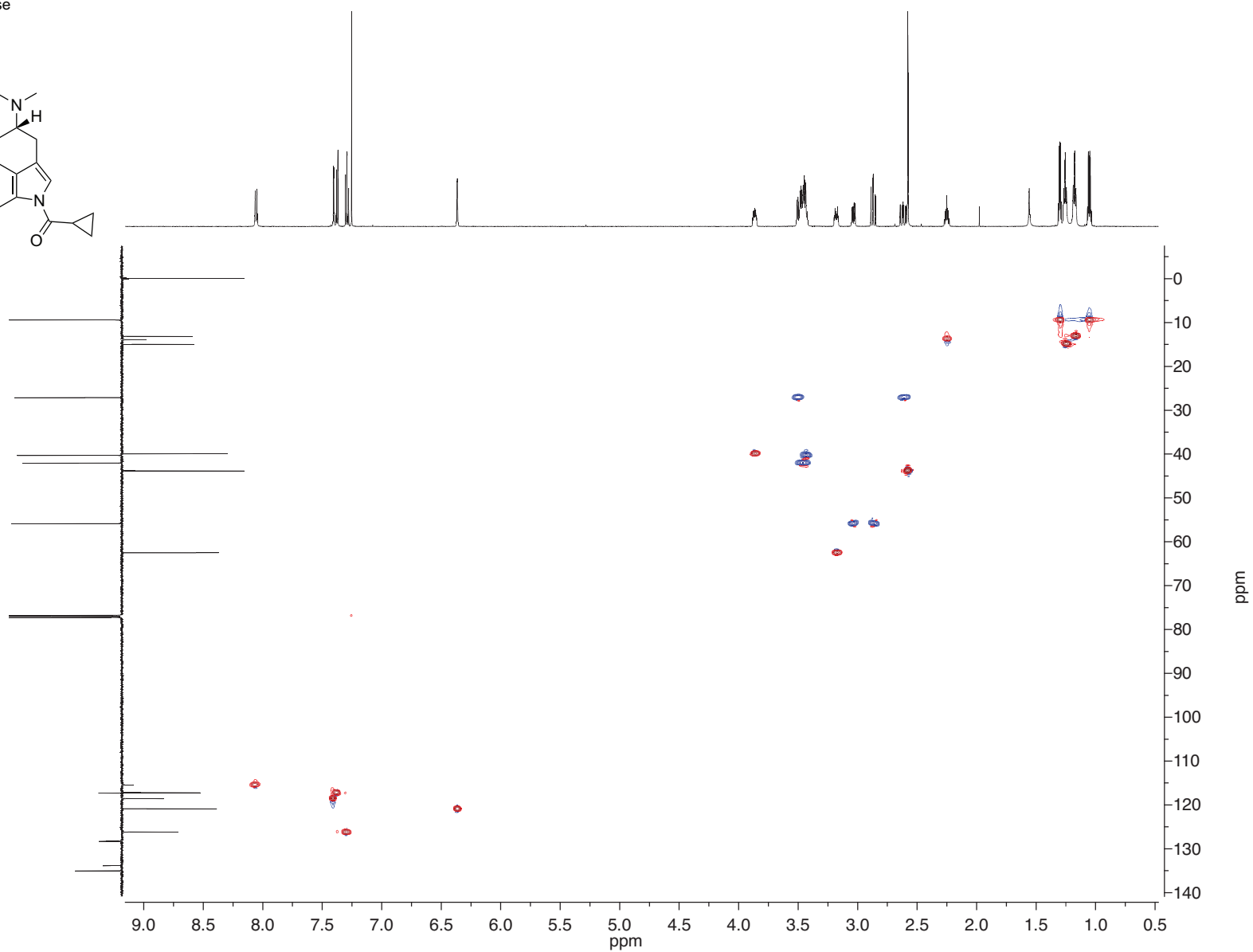
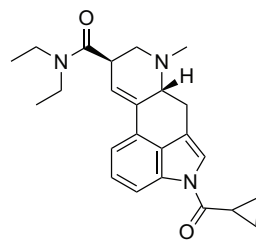
Supporting Information – Drug Testing and Analysis

1CP-LSD base
¹³C DEPTQ (150 MHz)
 CDCl₃



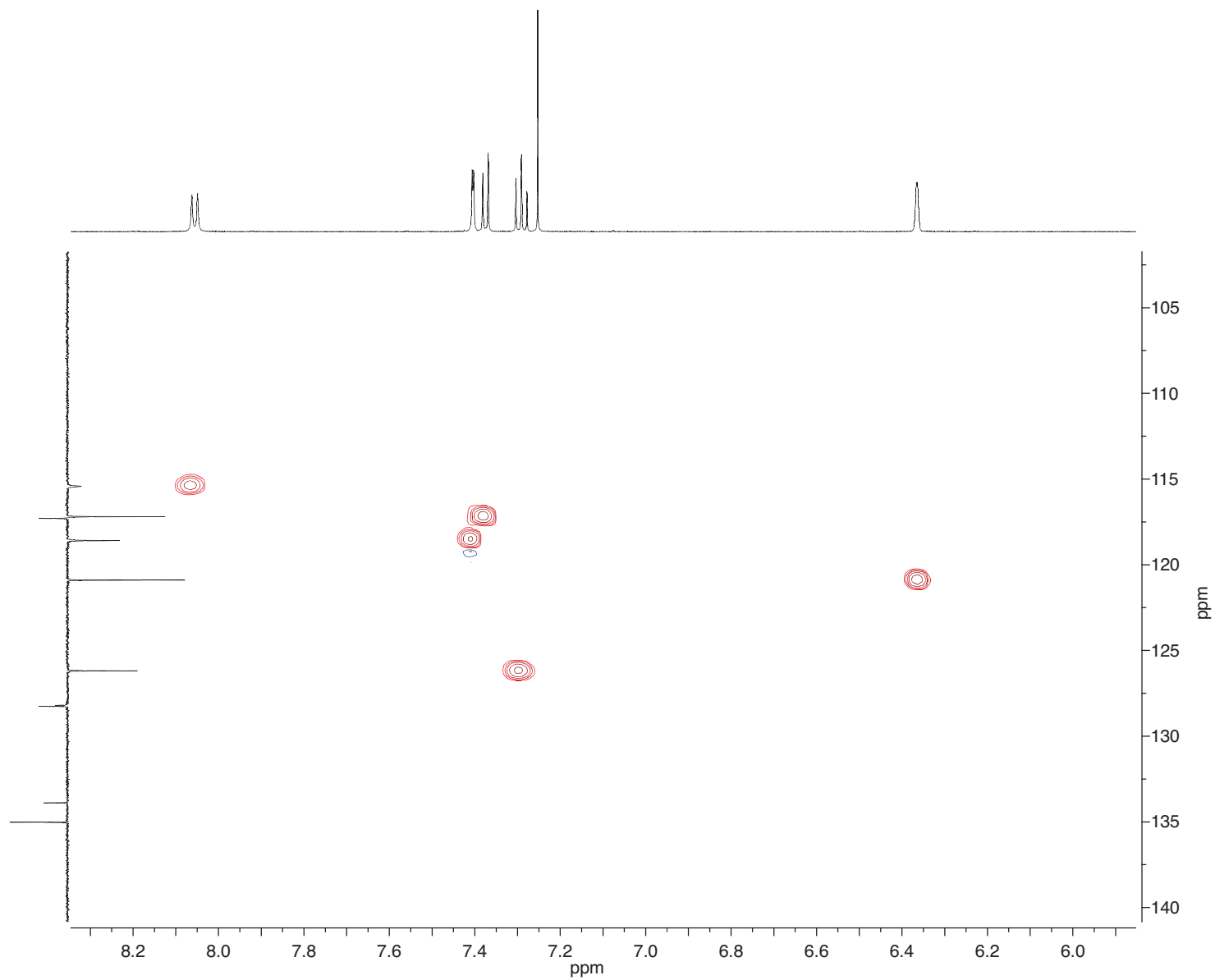
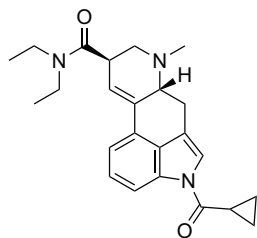
Supporting Information – Drug Testing and Analysis

1CP-LSD base
HSCQ
CDCl₃



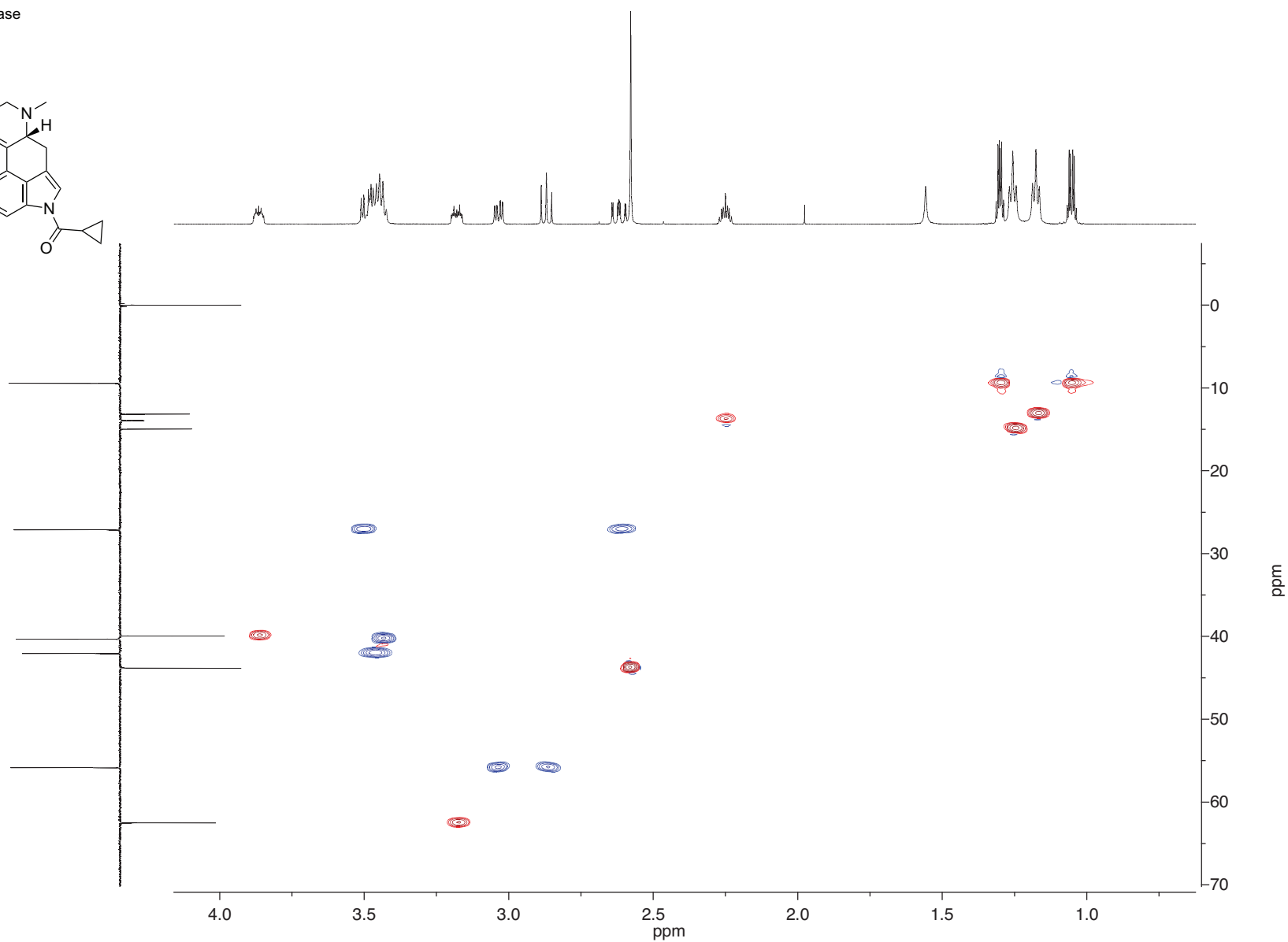
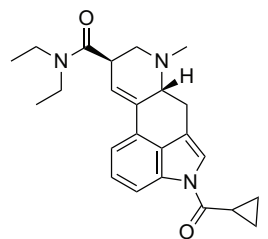
Supporting Information – Drug Testing and Analysis

1CP-LSD base
HSCQ
CDCl₃



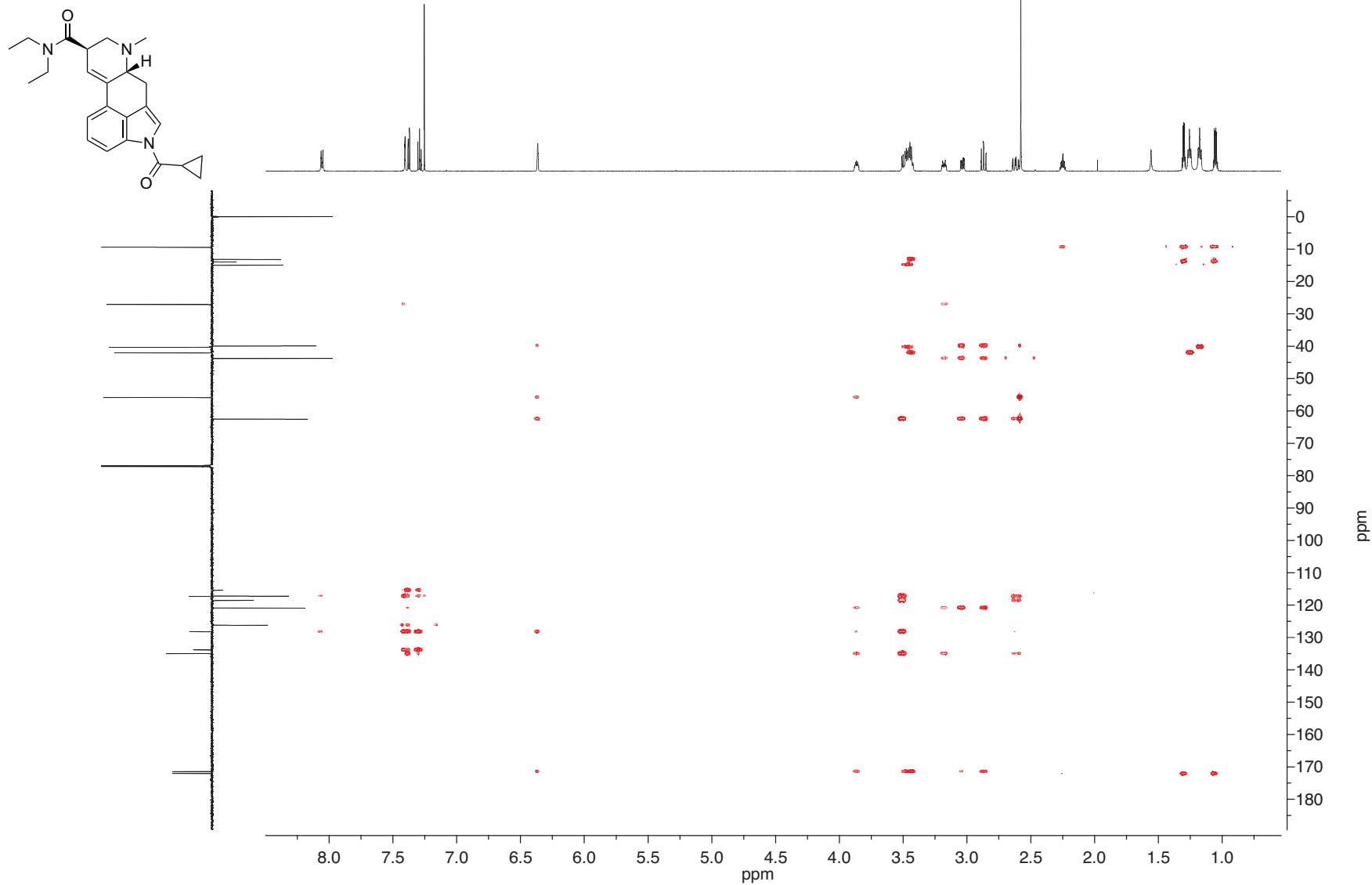
Supporting Information – Drug Testing and Analysis

1CP-LSD base
HSCQ
CDCl₃



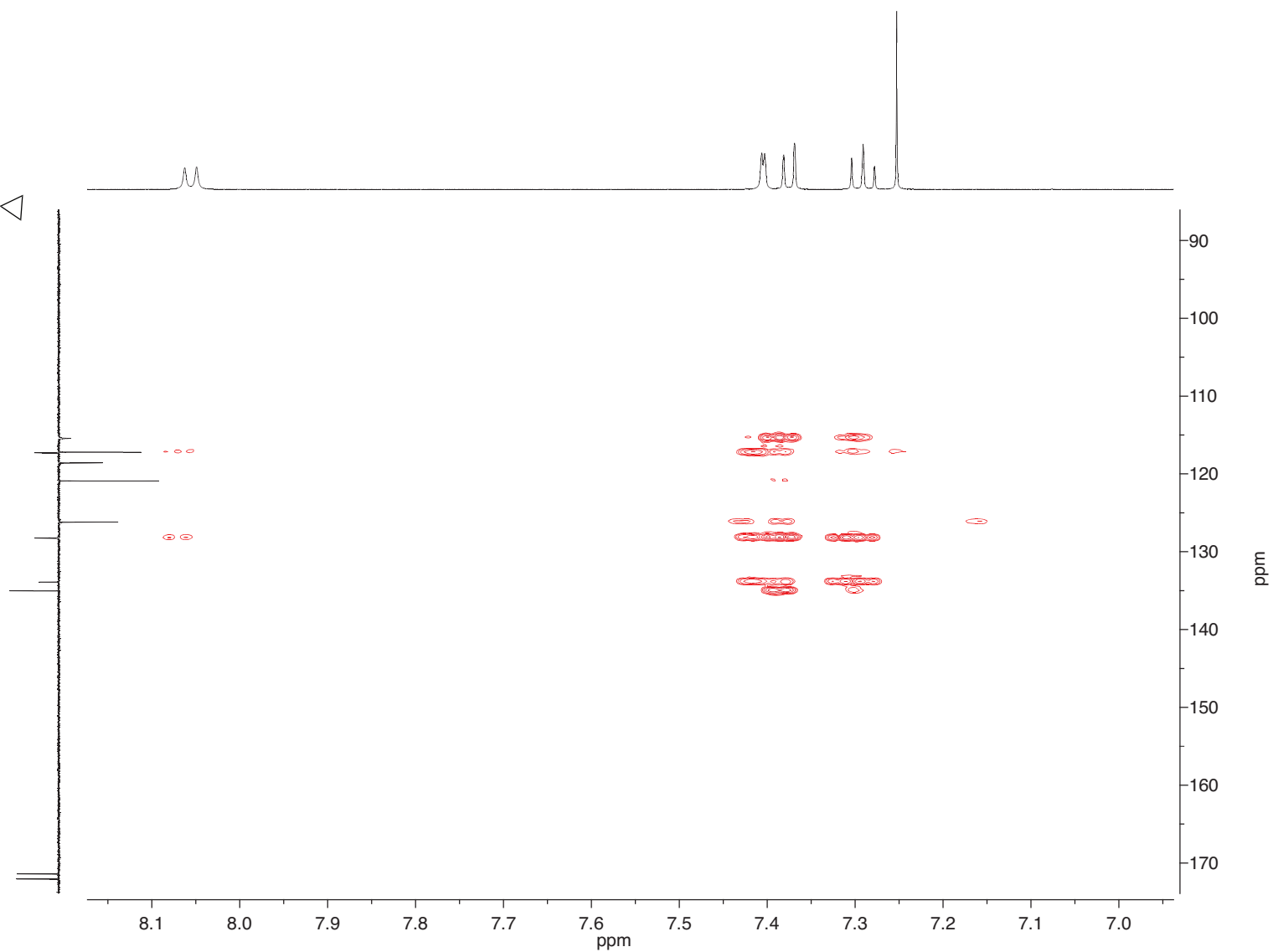
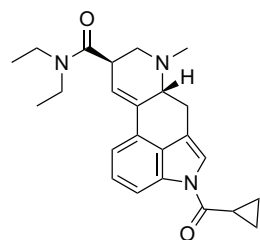
Supporting Information – Drug Testing and Analysis

1CP-LSD base
HMBC
CDCl₃

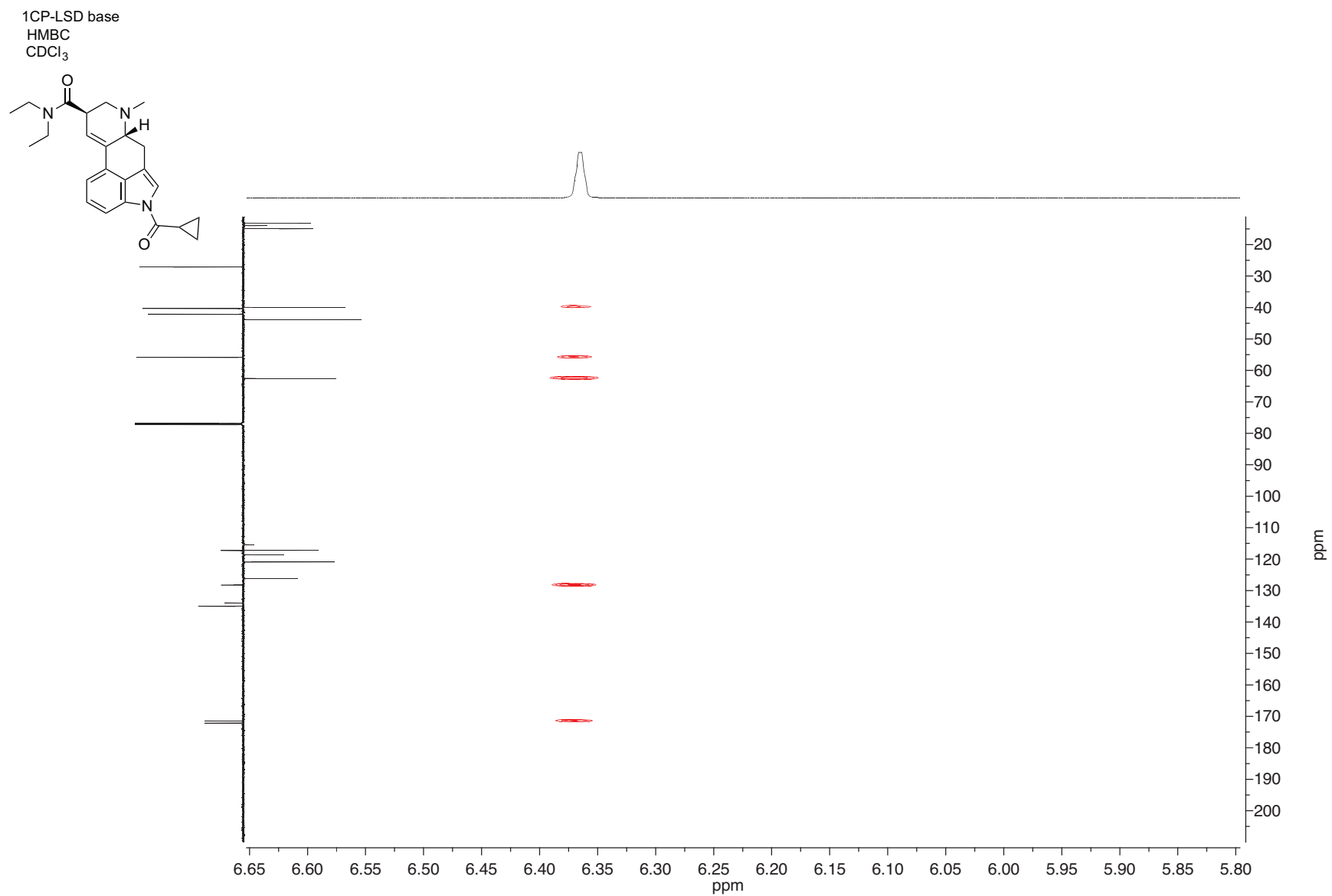


Supporting Information – Drug Testing and Analysis

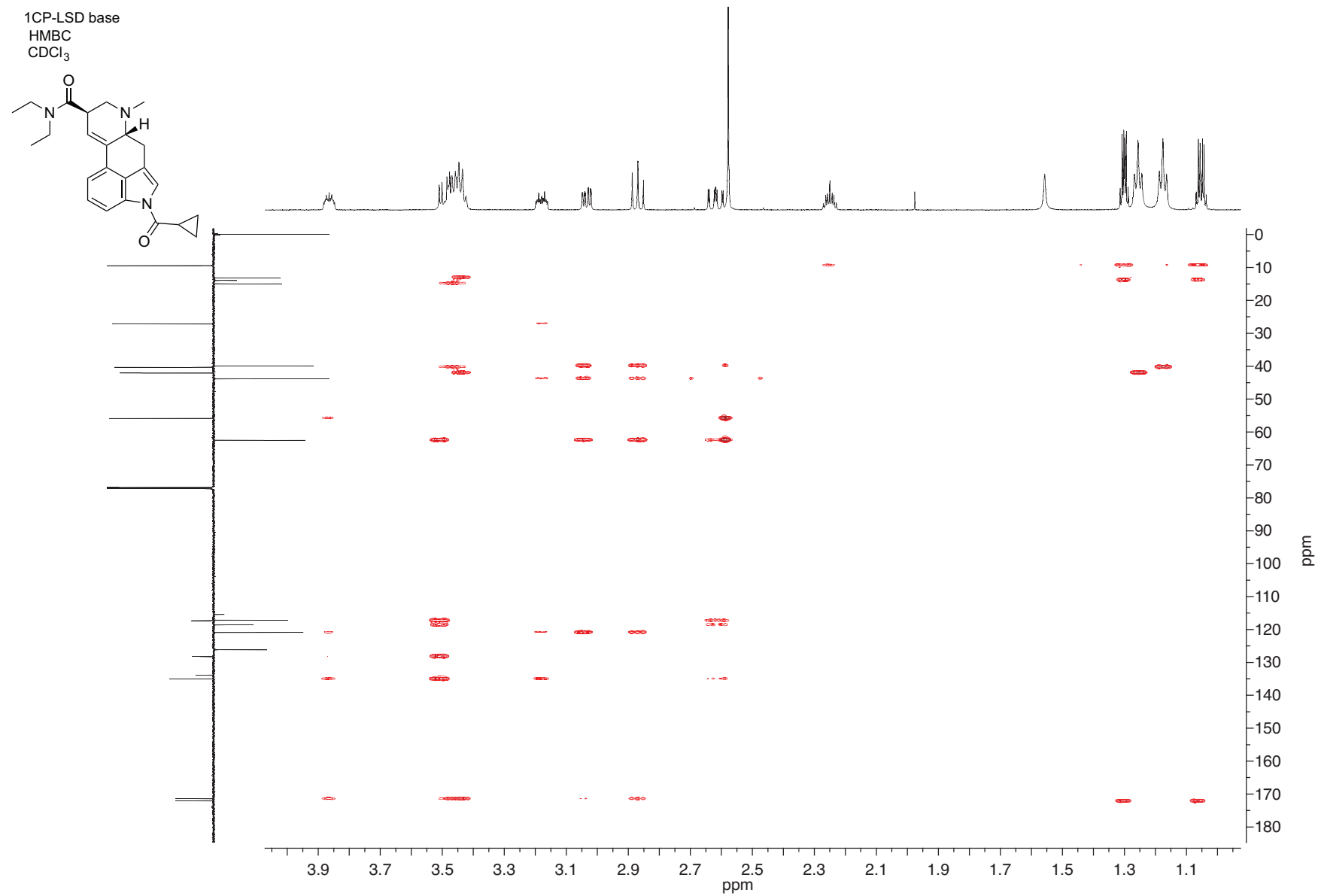
1CP-LSD base
HMBC
CDCl₃

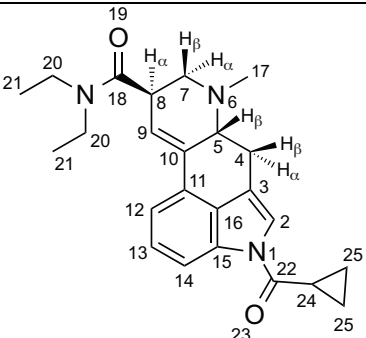


Supporting Information – Drug Testing and Analysis



Supporting Information – Drug Testing and Analysis



¹ H and ¹³ C DEPTQ NMR data for 1CP-LSD base in CDCl ₃ at 600/150 MHz		
		
No.	¹³ C [δ / ppm]	¹ H [δ / ppm]
1	–	–
2	118.57	7.41 (d, <i>J</i> = 2.0 Hz, 1H)
3	117.28	–
4	27.13	2.62 (ddd, <i>J</i> = 13.8, 11.8, 2.8 Hz, 4α-H, 1H) 3.50 (dd, <i>J</i> = 13.7, 5.4 Hz, 4β-H, 1 H) *overlap with H-20
5	62.50	3.19 (dddd, <i>J</i> = 11.2, 5.4, 3.5, 2.0 Hz, H-5β, 1H)
6	–	–
7	55.85	3.04 (ddd, <i>J</i> = 11.3, 5.0, 1.2 Hz, H-7α, 1H) 2.87 (t, <i>J</i> = 10.8 Hz, H-7β, 1H)
8	39.93	3.87 (dddd, <i>J</i> = 10.5, 4.9, 3.3, 2.1 Hz, H-8α, 1H)
9	120.88	6.38–6.35 (m, 1H)
10	135.01	–
11	128.24	–
12	117.18	7.38 (d, <i>J</i> = 7.4 Hz, 1H)
13	126.17	7.30 (t, <i>J</i> = 7.8 Hz, 1H)
14	115.40	8.06 (d, <i>J</i> = 8.1 Hz, 1H)
15	133.87	–
16	128.19	–
17	43.81	2.58 (s, 3H)
18	171.40	–
19	–	–
20	42.06	3.53–3.42 (m, 4H)
20	40.31	*coalescing with 4β-H
21	14.96	1.26 (t, <i>J</i> = 7.0 Hz, 3H)
21	13.16	1.18 (t, <i>J</i> = 7.0 Hz, 3H)
22	172.06	–
23	–	–
24	13.92	2.26 (tt, <i>J</i> = 8.0, 4.5 Hz, 1H)
25	9.42	1.32–1.29 (m, 2H) 1.08–1.04 (m, 2H)