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Analytical and behavioral characterization of 1-dodecanoyl-LSD (1DD-LSD)

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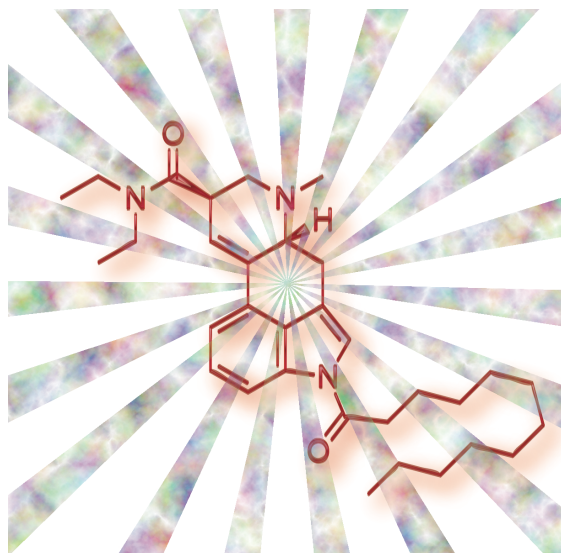
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⁸ *Research Service, VA San Diego Healthcare System, San Diego, USA*

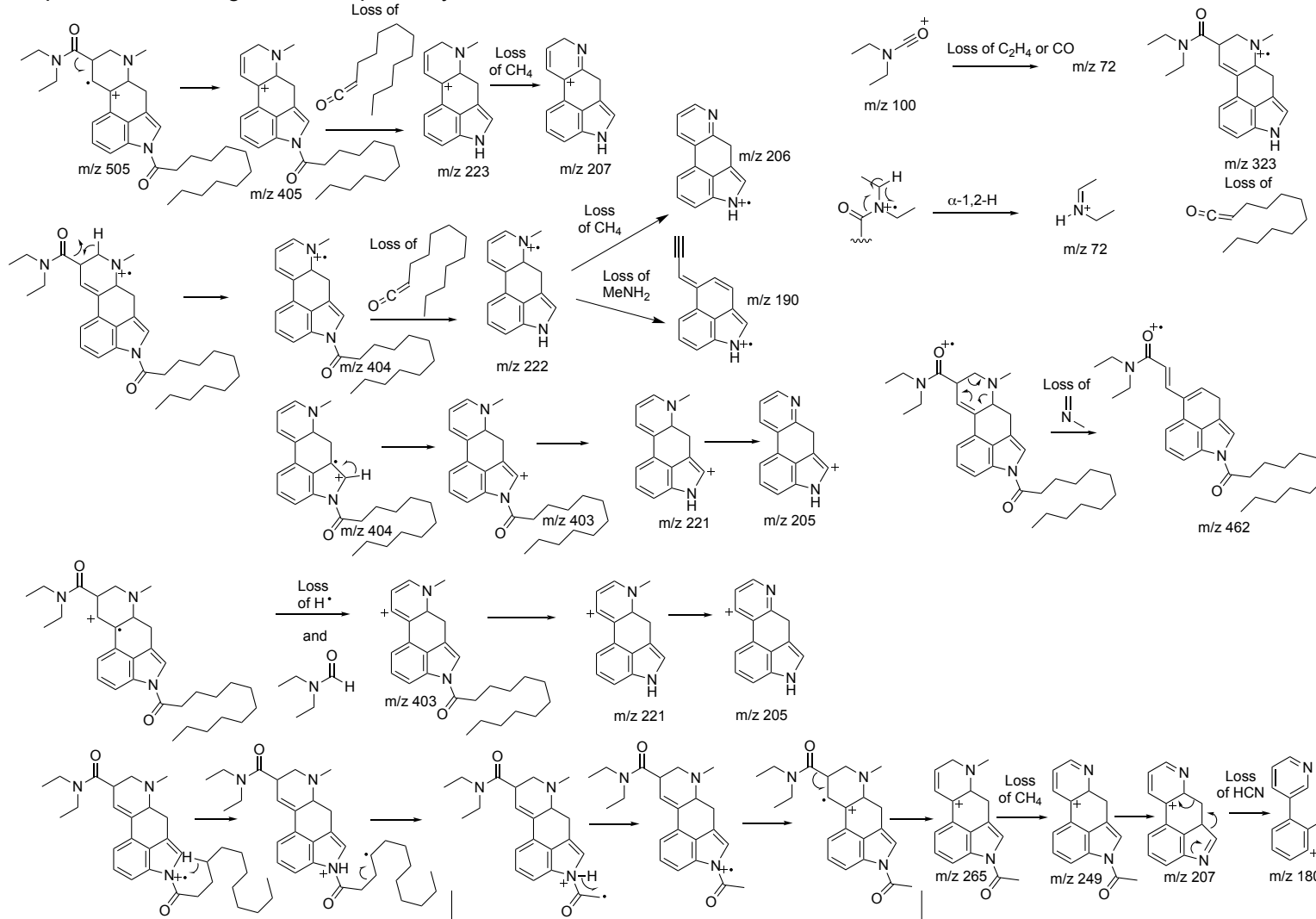
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* Correspondence to: Simon D. Brandt, School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK. E-Mail: s.brandt@ljmu.ac.uk

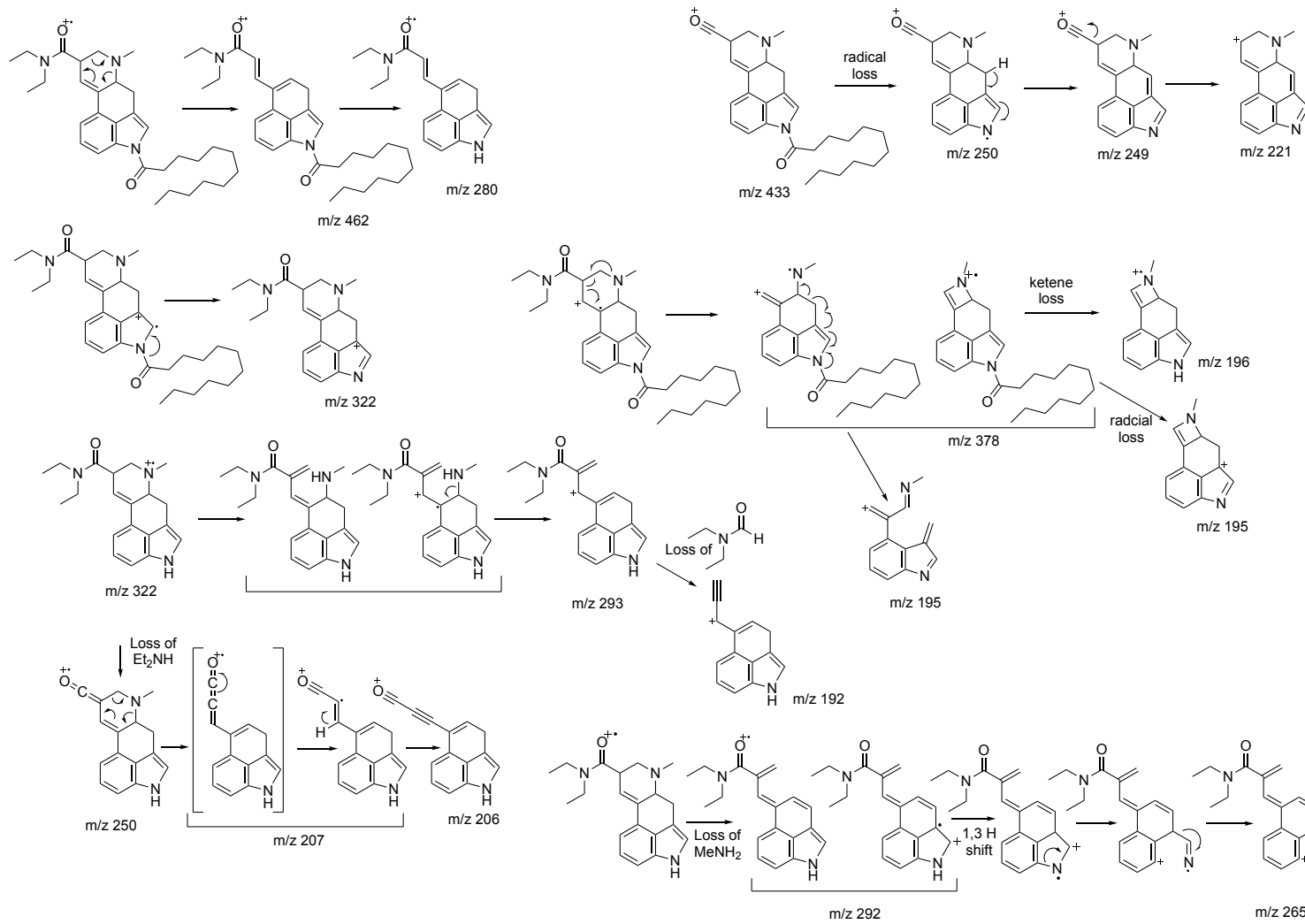


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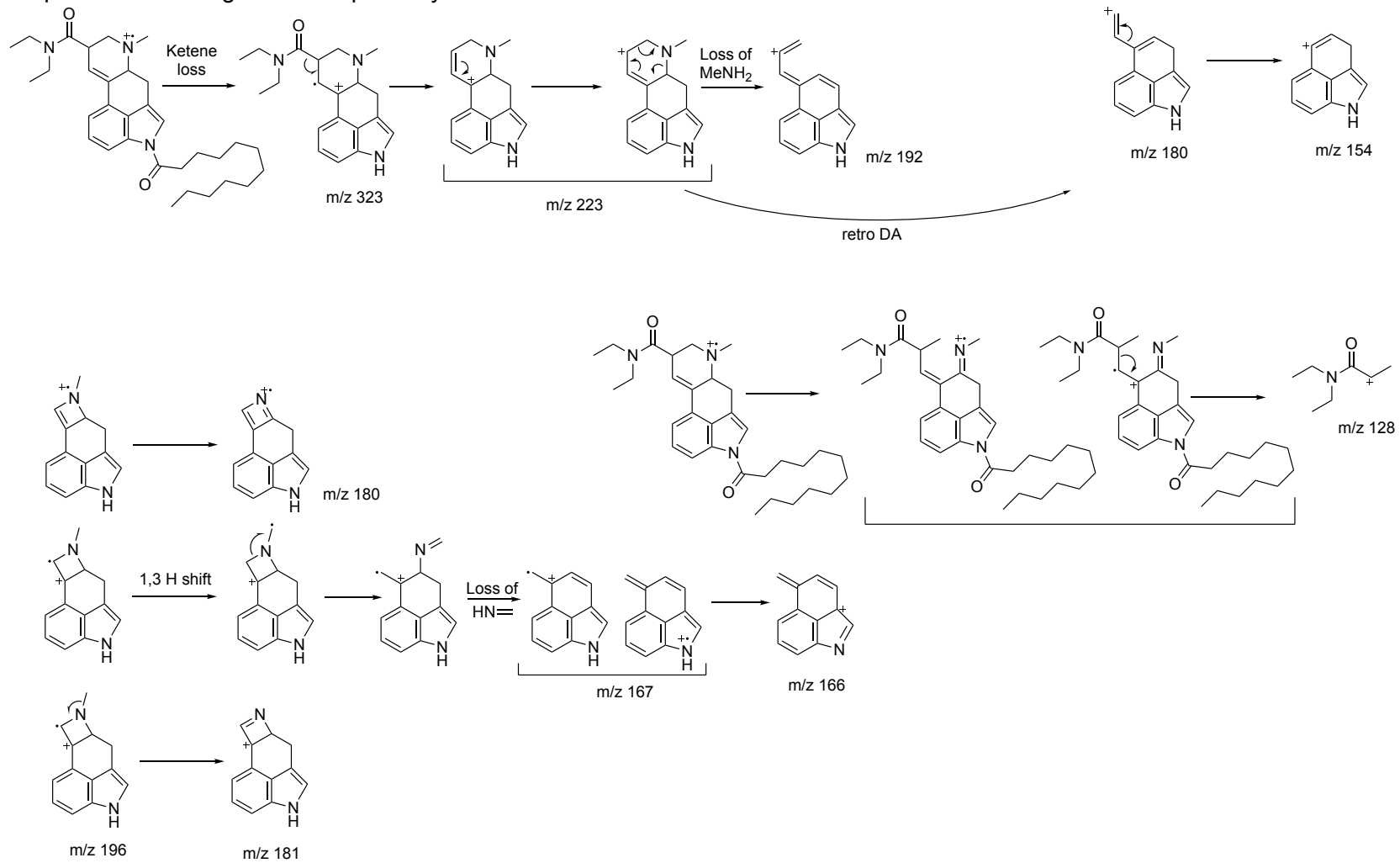
Proposed EI-MS fragmentation pathways for 1DD-LSD



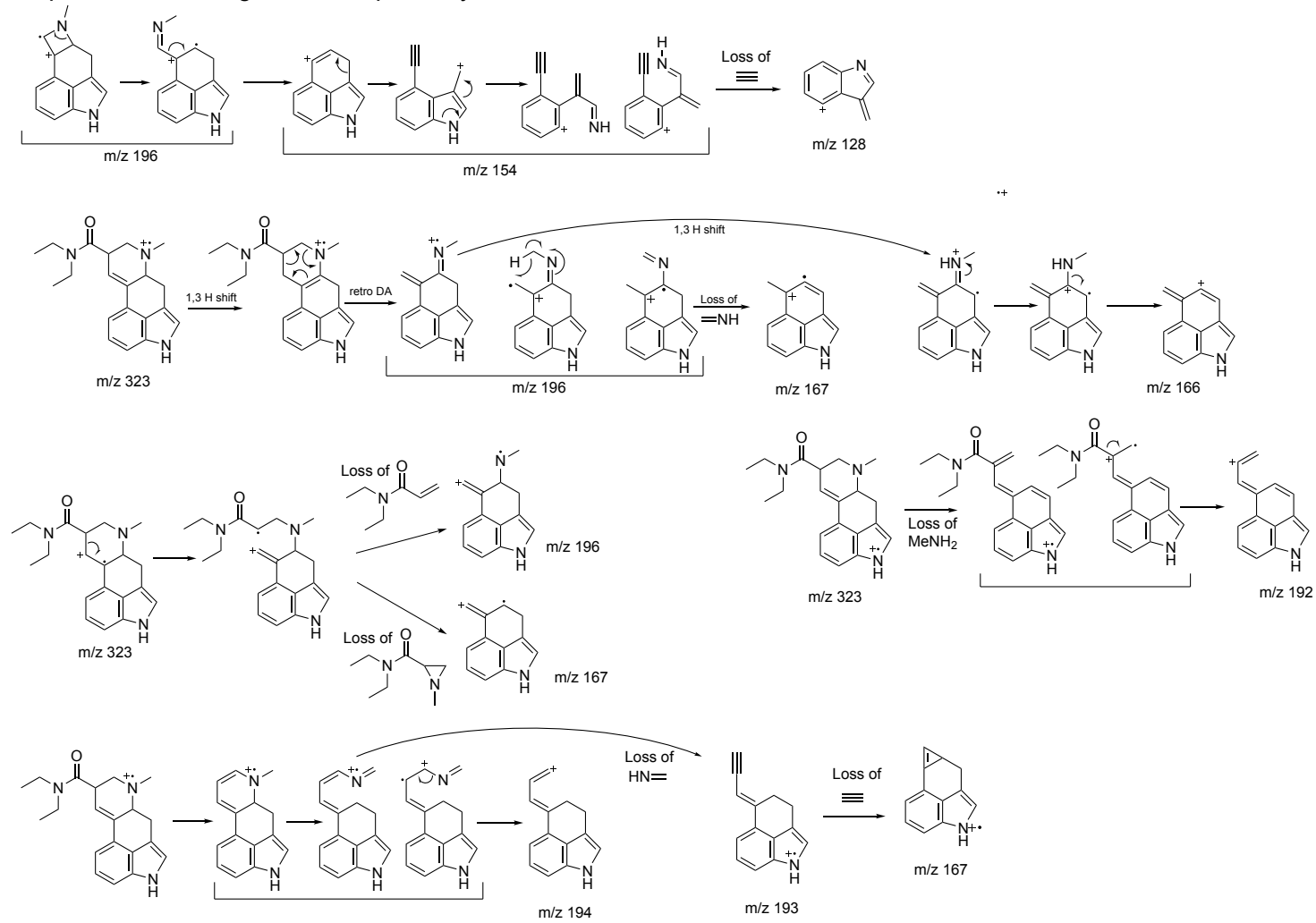
Proposed EI-MS fragmentation pathways for 1DD-LSD



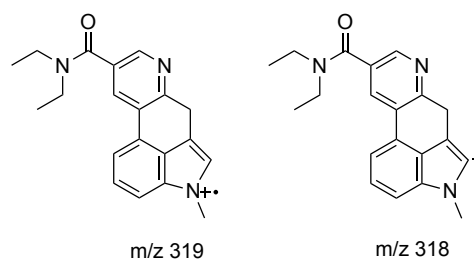
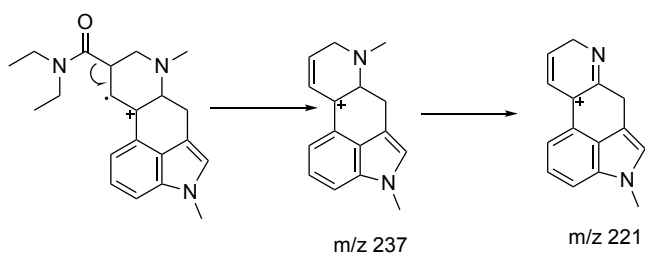
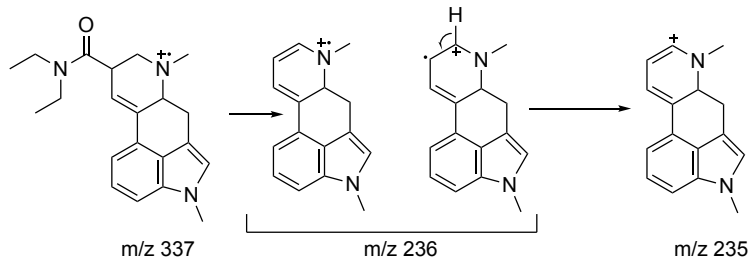
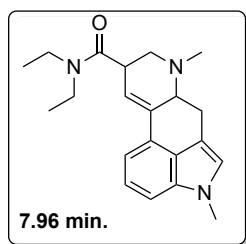
Proposed EI-MS fragmentation pathways for 1DD-LSD



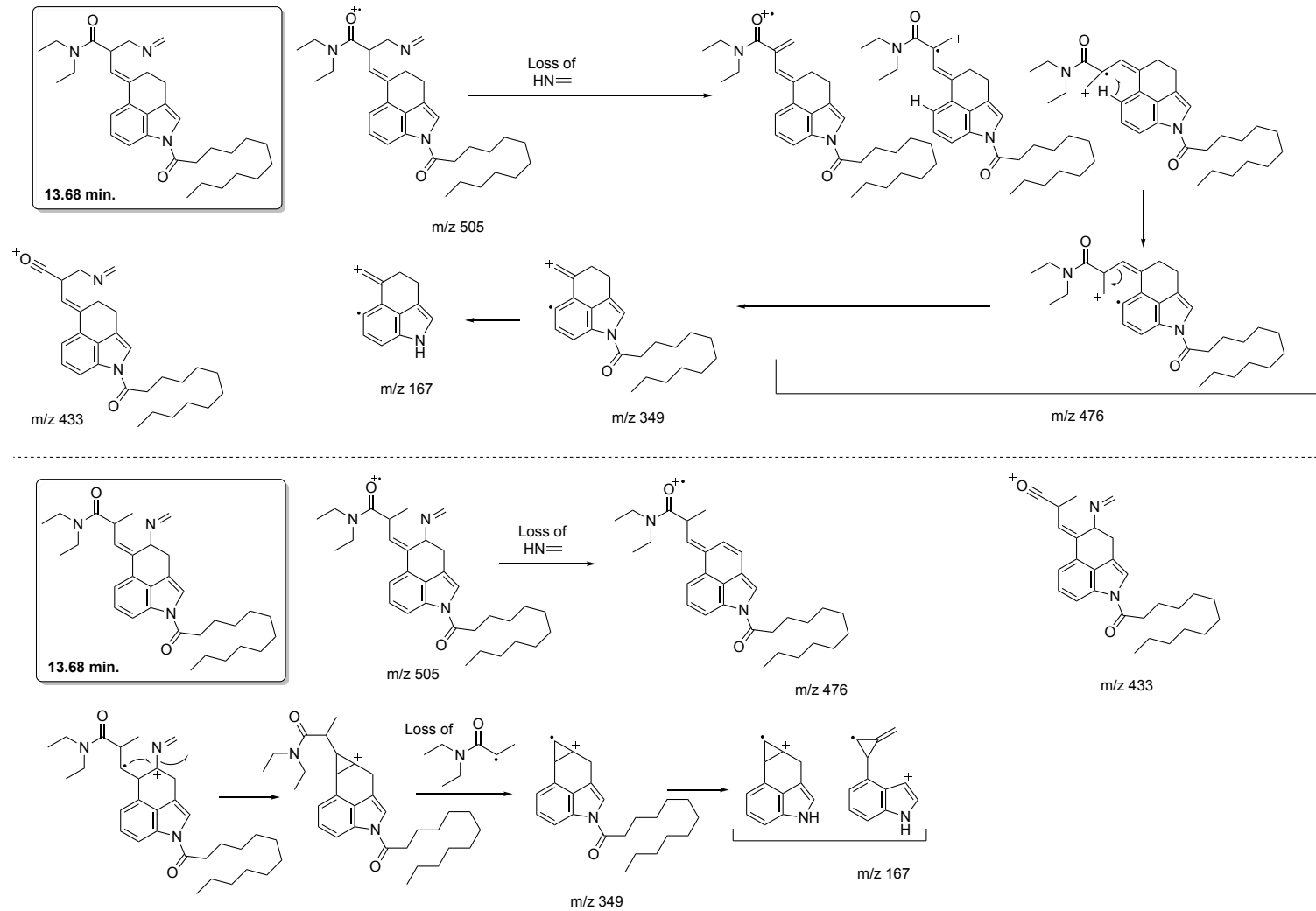
Proposed EI-MS fragmentation pathways for 1DD-LSD



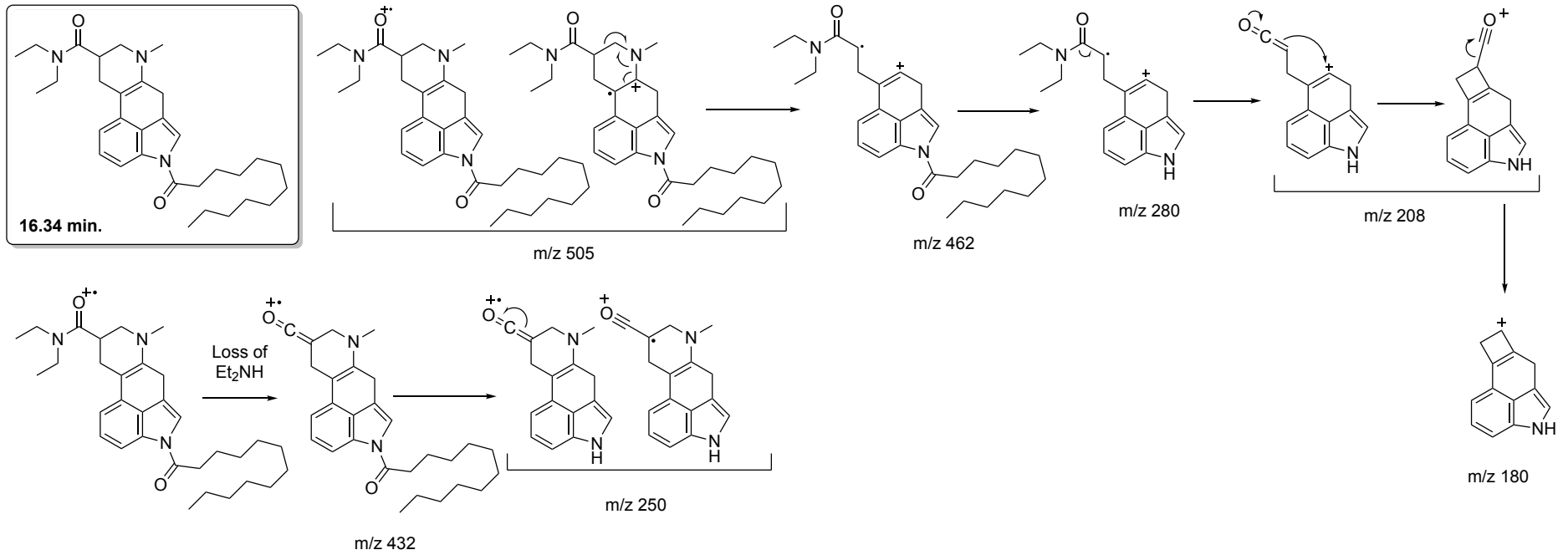
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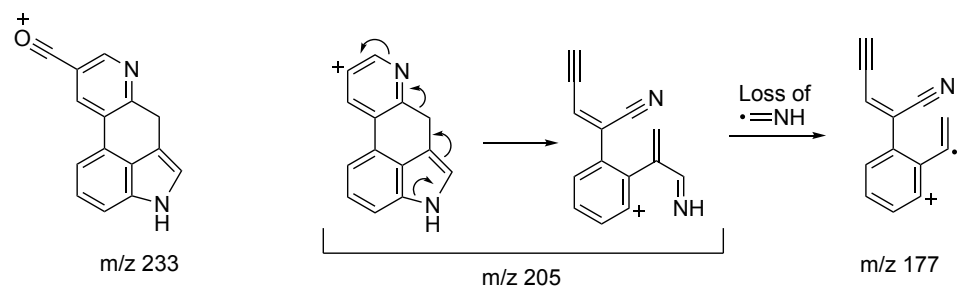
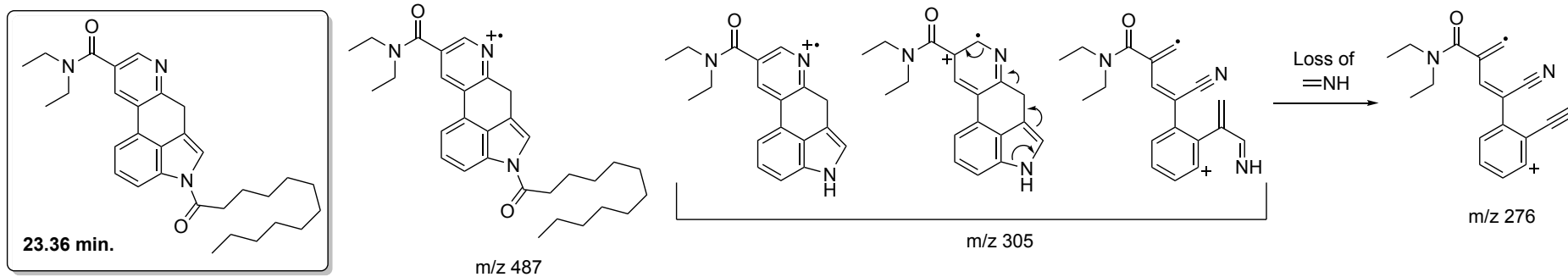
Tentative identification of GC-induced artifacts (GC-MS method 1)



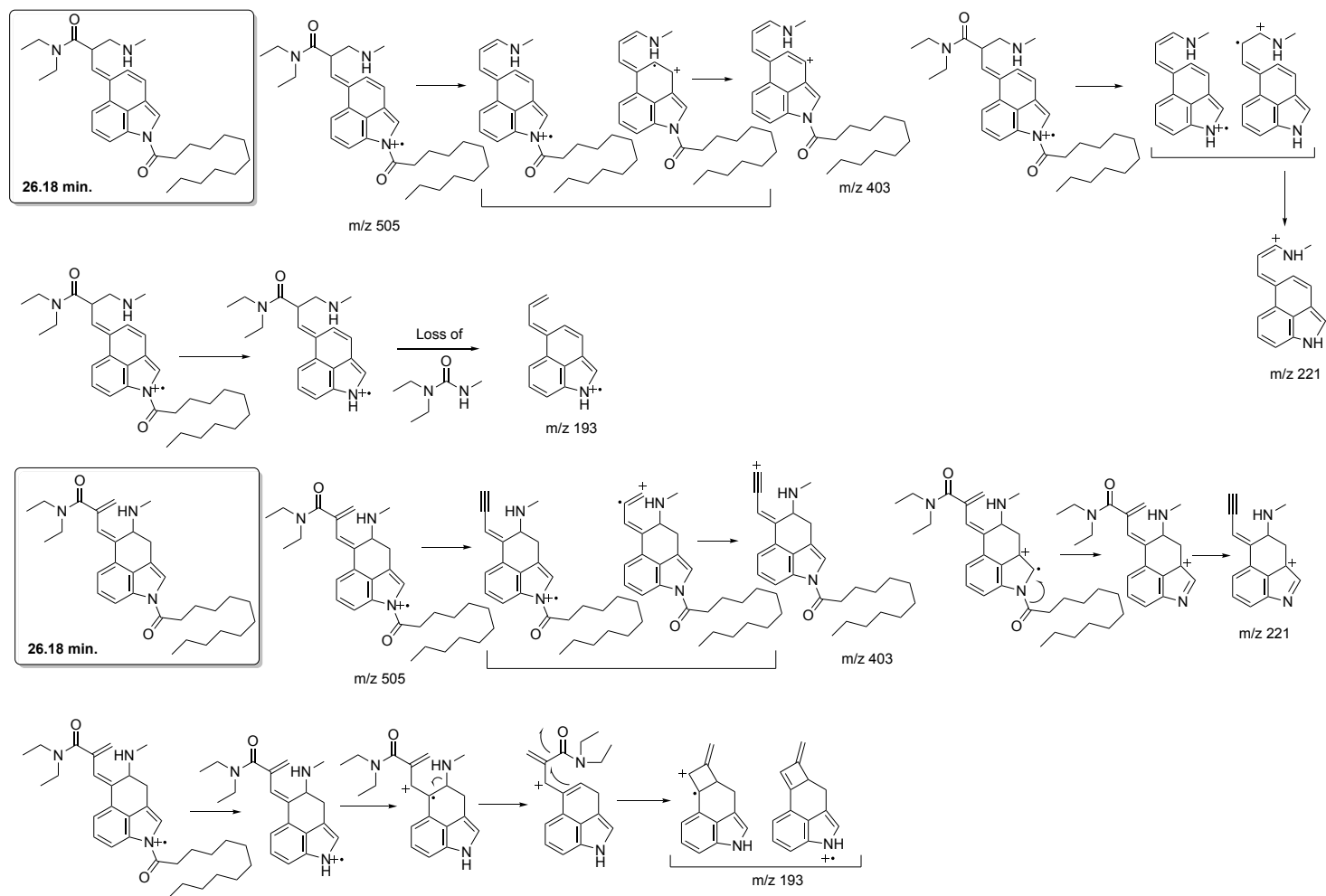
Tentative identification of GC-induced artifacts (GC-MS method 1)



Tentative identification of GC-induced artifacts (GC-MS method 1)



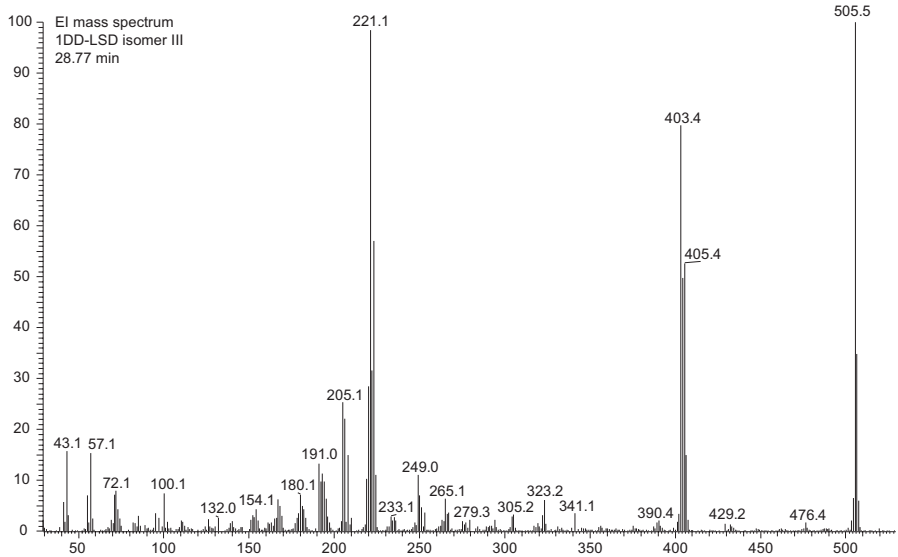
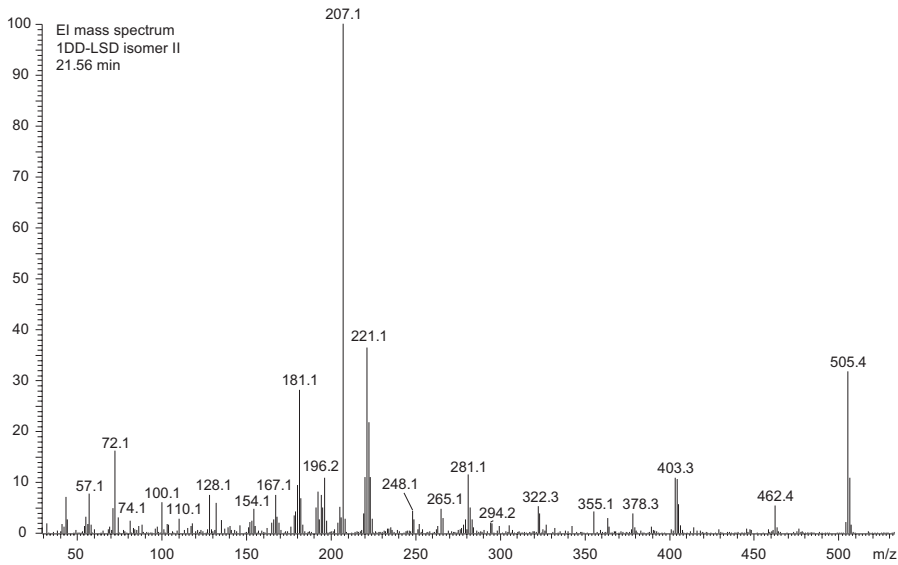
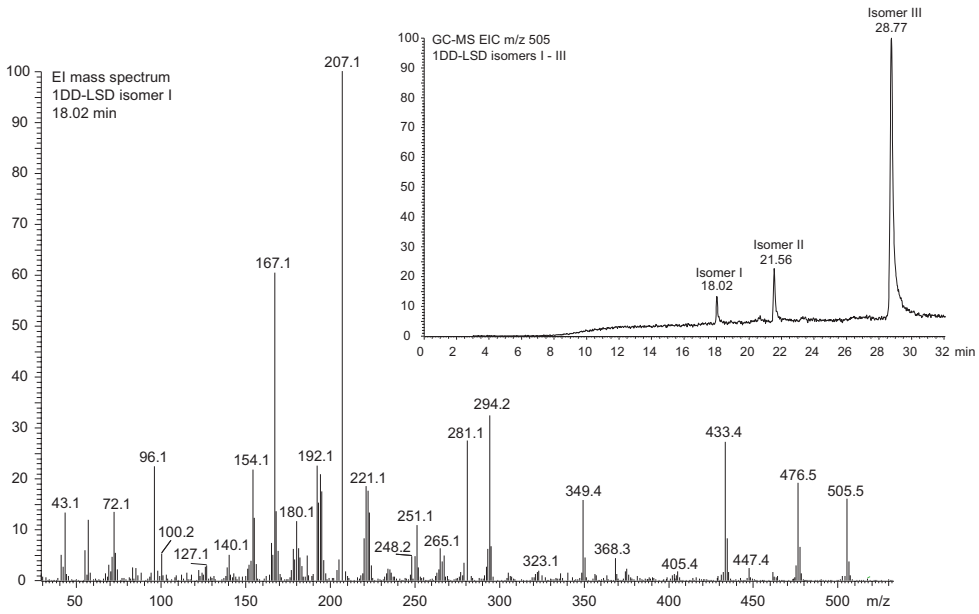
Tentative identification of GC-induced artifacts (GC-MS method 1)



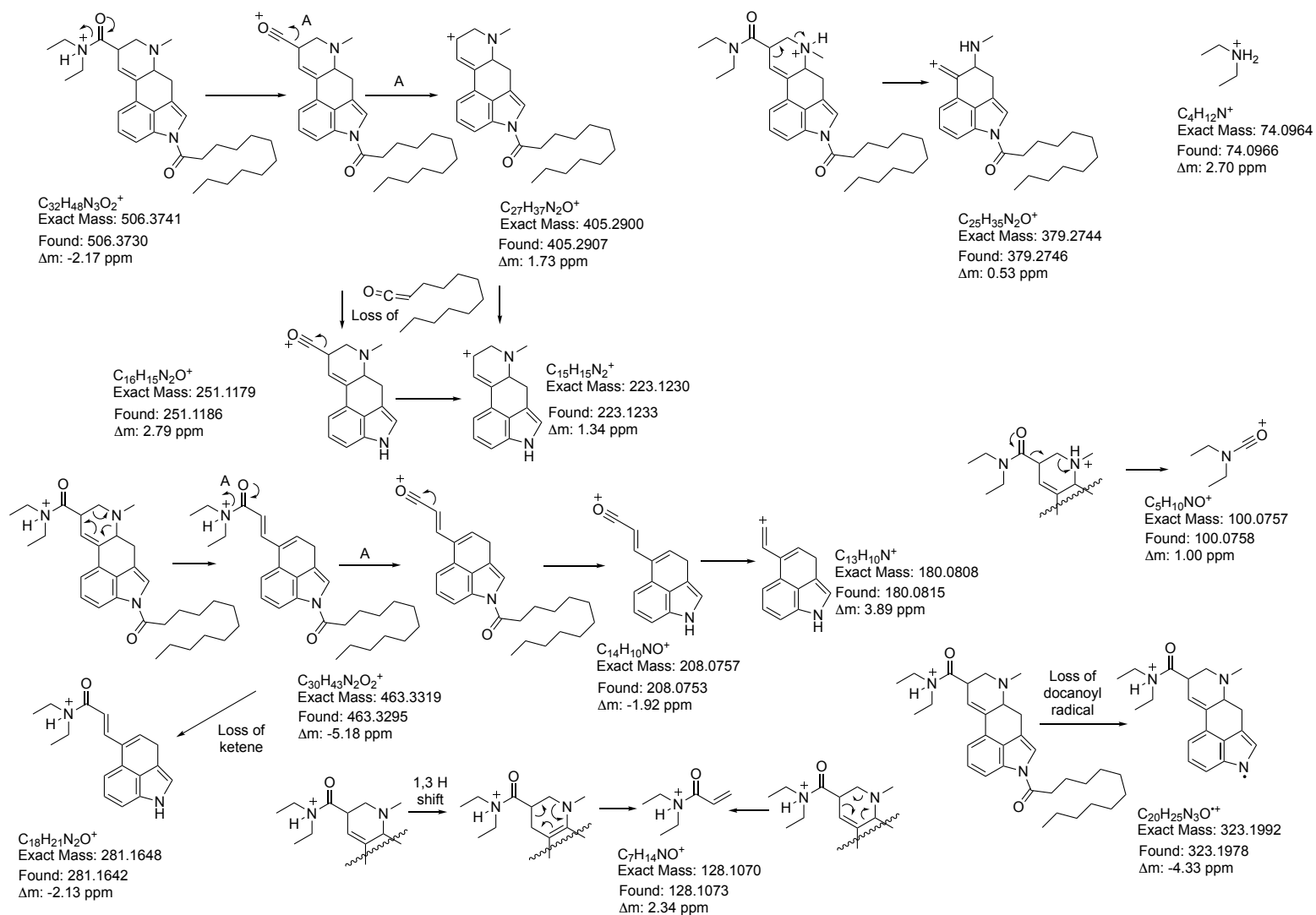
Gas chromatography-mass spectrometry (GC-MS) – method 2

For electron ionization mass spectrometry (EI-MS), a Finnigan TSQ 8000 Evo triple stage quadrupole mass spectrometer coupled to a gas chromatograph (Trace GC 1310, Thermo Electron, Dreieich, Germany) was used and a Triplus RSH (Thermo Scientific) autosampler was employed for sample introduction. Mass spectra were recorded using a 70 eV electron ionization energy. The ion source temperature was set at 175°C and the emission current was 50 µA. For recordings of EI mass spectra, the scan time was 1 s spanning a scan range between m/z 29–600 and samples were injected in splitless mode. For the analysis of 1DD-LSD base, the salt (2 mg) was dissolved in 2 mL demineralized water and made alkaline with one drop of NaOH (5% w/w). The solution was extracted with 2 ml diethyl ether, and the ethereal phase was transferred into a new vial and subjected to GC-MS analysis. Separation was achieved using a fused silica capillary DB-1 column (30 m × 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 340°C at 15°C/min. The final temperature was held for 20 min. The injector temperature was 280°C. The transfer line temperature was set at 280°C and the carrier gas was helium in constant flow mode at a flow rate of 1.2 mL/min. Mass spectra were treated as a sum of 6 spectra (AV:6), from 17.99 to 18.07 min. Background spectra were subtracted twice: from 17.57 and 17.72 min before and from 18.44 to 18.57 min after the peak in EIC mode. RI values could not be determined under these conditions when using a paraffin mixture at oven temperatures up to 340°C (RI > 4000).

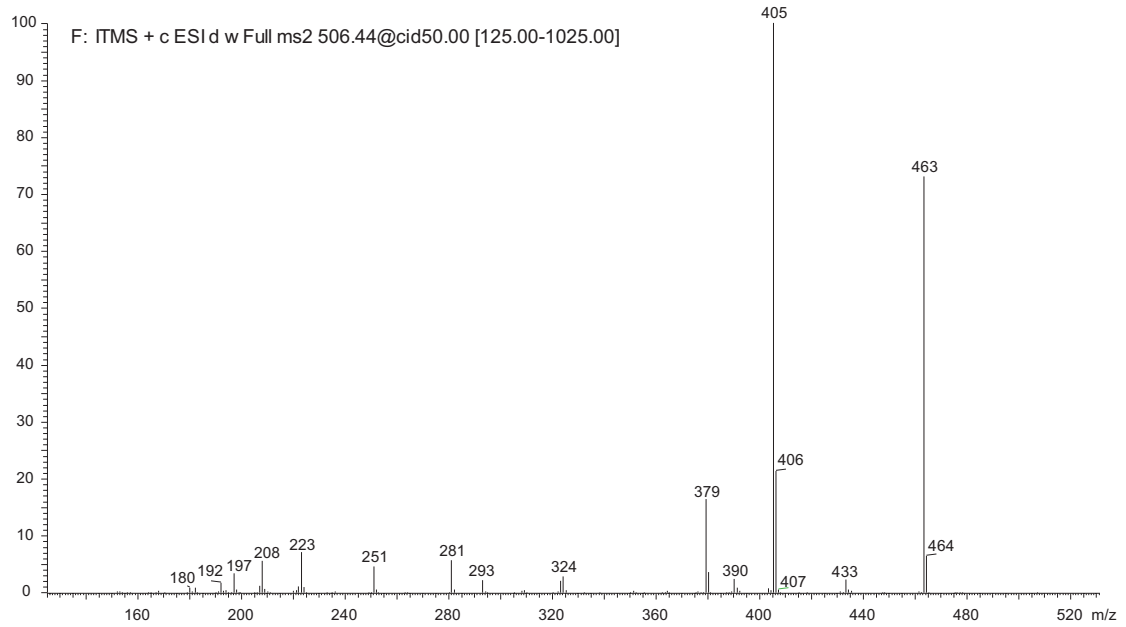
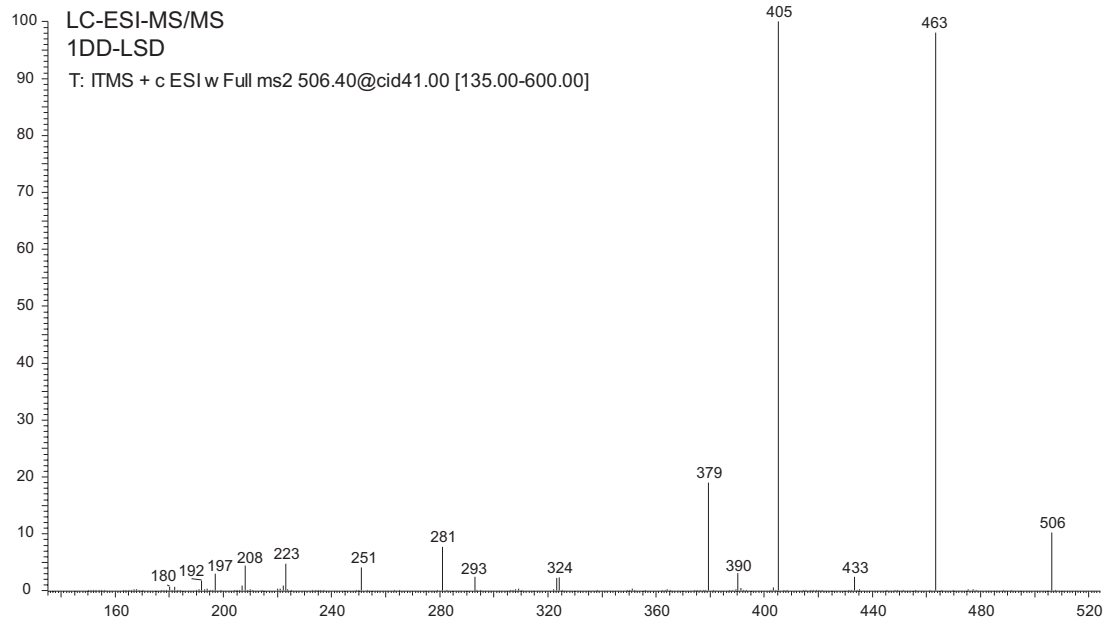
GC-MS data (method 2)



Proposed ESI-QTOF-MS/MS fragmentation pathways for 1DD-LSD

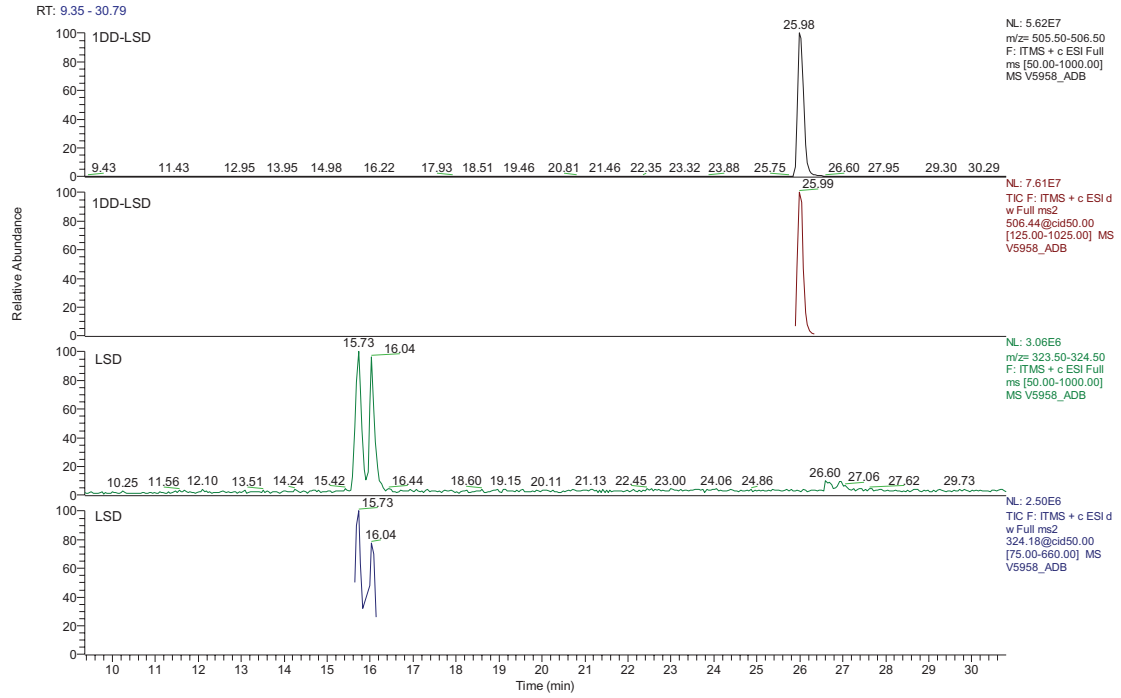


LC-ESI-linear ion trap-MS/MS



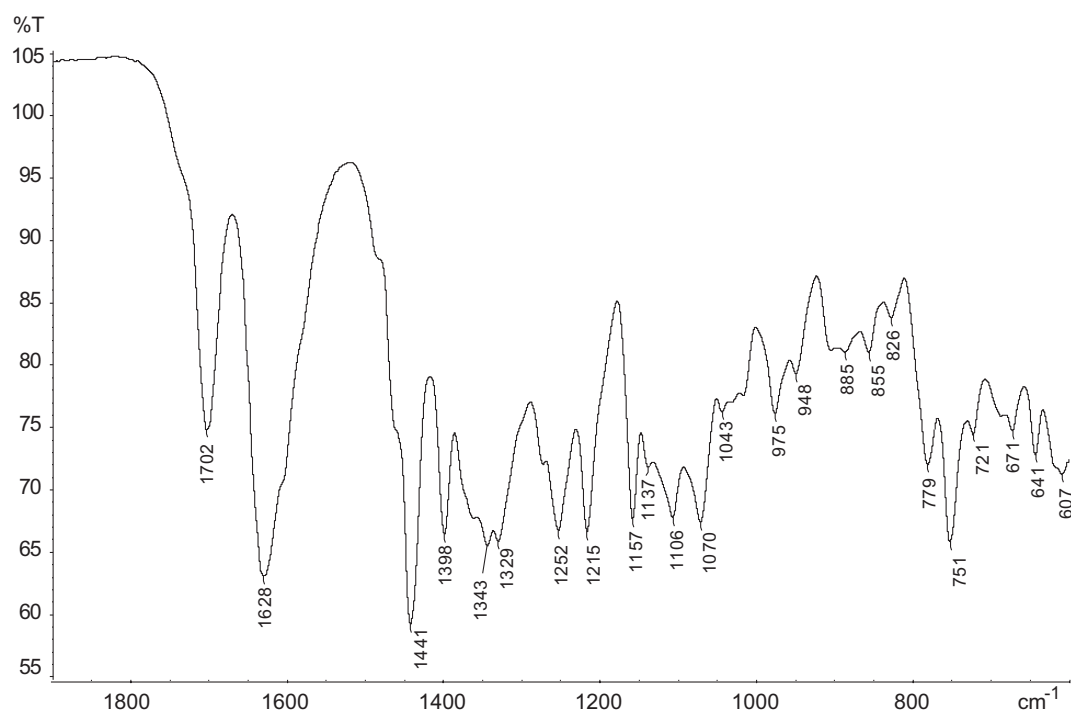
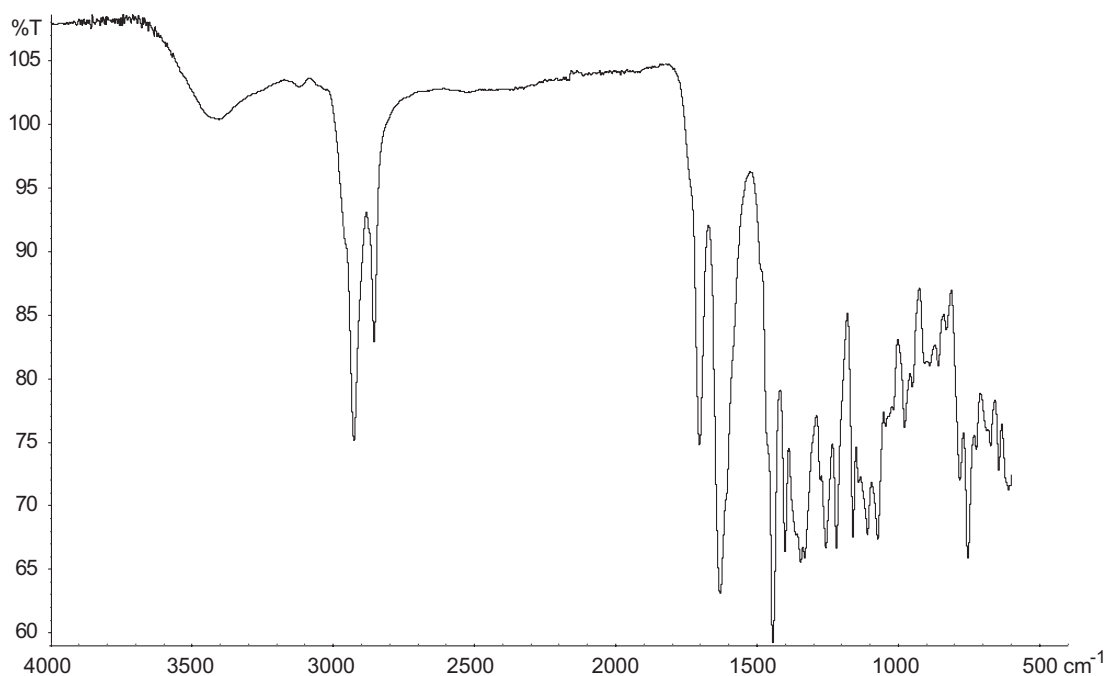
Supporting Information – Drug Testing and Analysis

LC-ESI-linear ion trap-MS



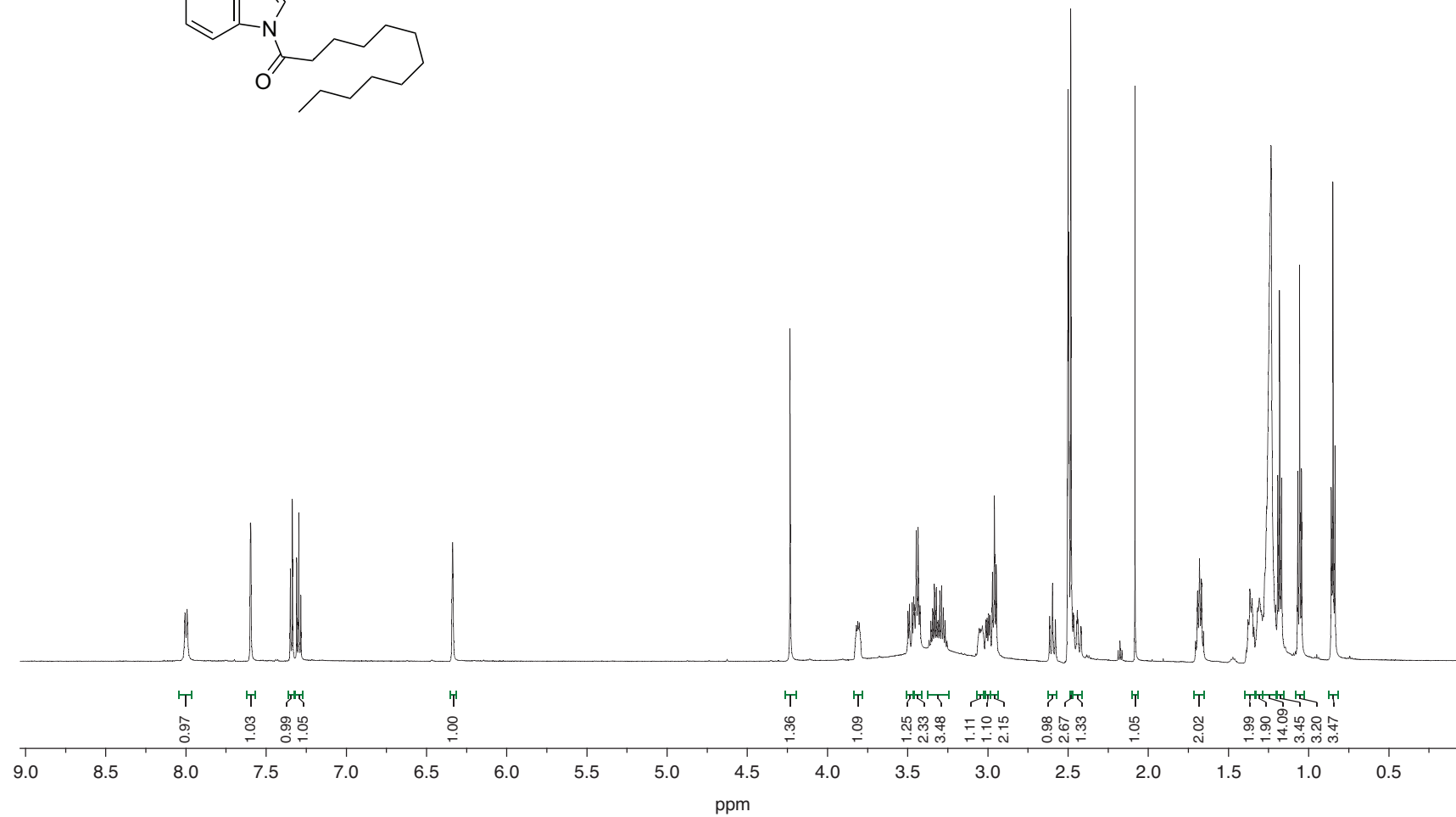
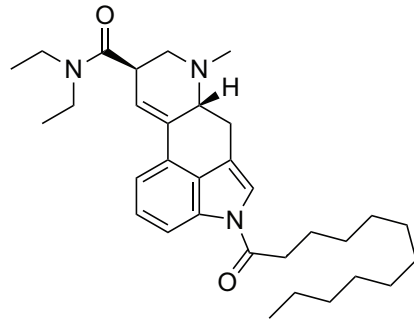
Attenuated total reflection-infrared spectroscopy (ATR-IR)

The IR spectrum of the powdered 1DD-LSD tartrate (3:2) was recorded on a Perkin Elmer Spectrum 100 FT-IR with Universal ATR sampling accessory (Perkin Elmer, Waltham, MA, USA). The wavelength resolution was set to 2 cm^{-1} . IR spectra were collected in a range of $650\text{--}4000\text{ cm}^{-1}$ with 16 scans per spectrum. The IR data were processed using Spectrum Perkin Elmer Version 6.3.4 Software (Perkin Elmer, Waltham, MA, USA).



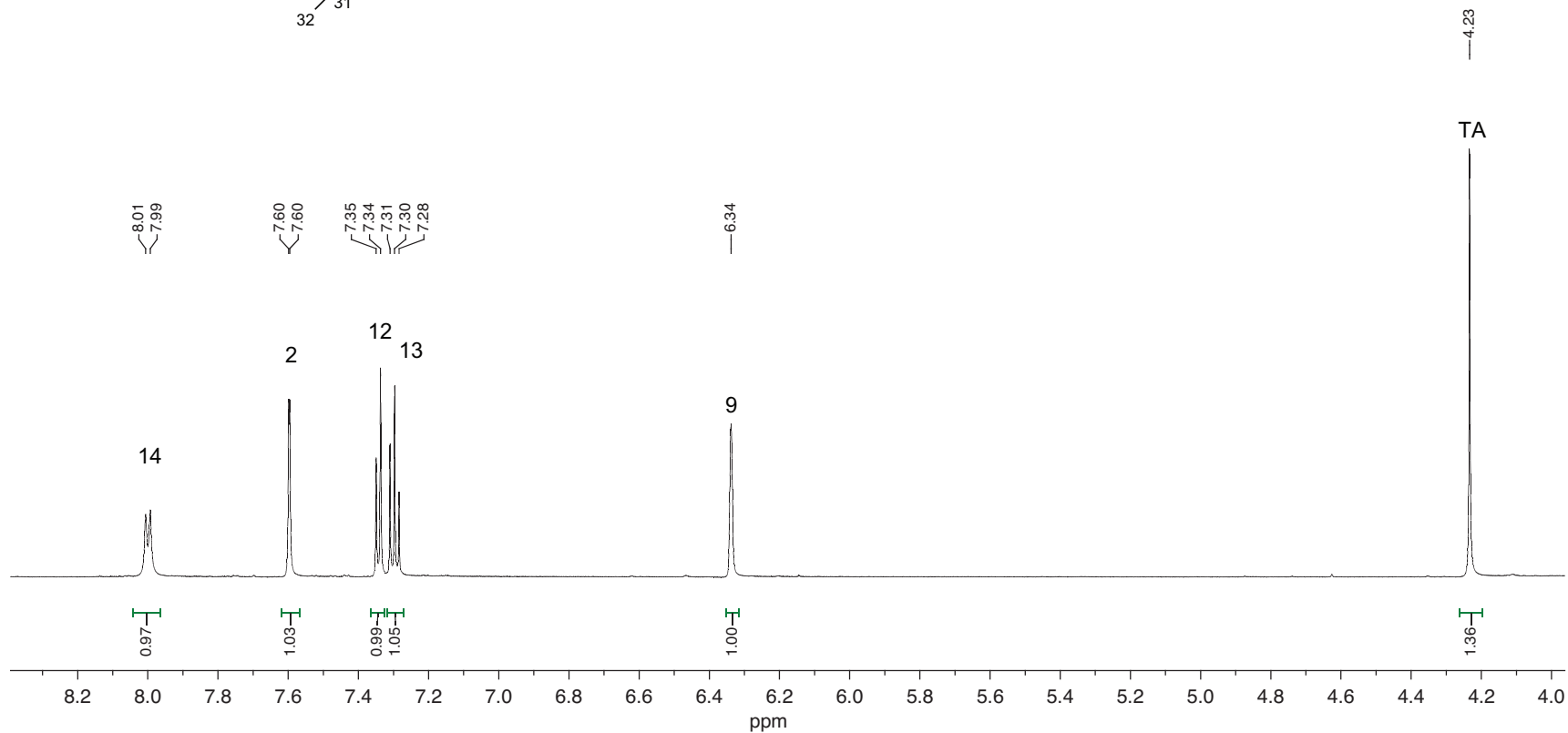
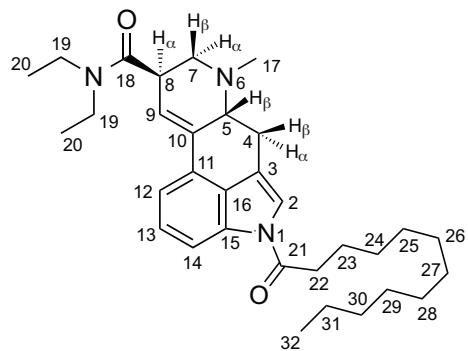
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
1H NMR (600 MHz)
DMSO-d₆



Supporting Information – Drug Testing and Analysis

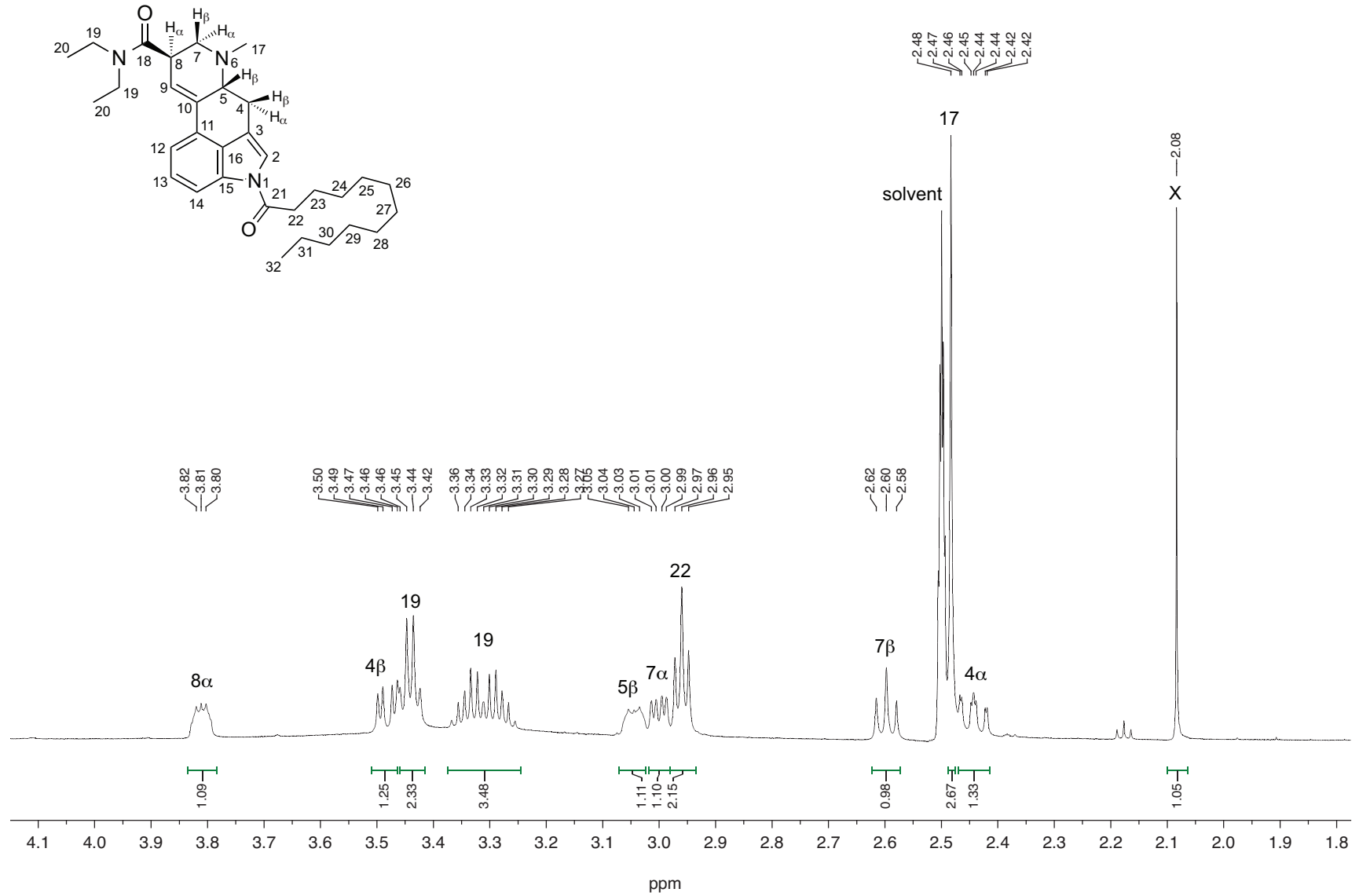
1DD-LSD tartrate (3:2)
 1H NMR (600 MHz)
 DMSO-*d*₆



TA: tartaric acid

Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
 1H NMR (600 MHz)
 DMSO-*d*₆



Supporting Information – Drug Testing and Analysis

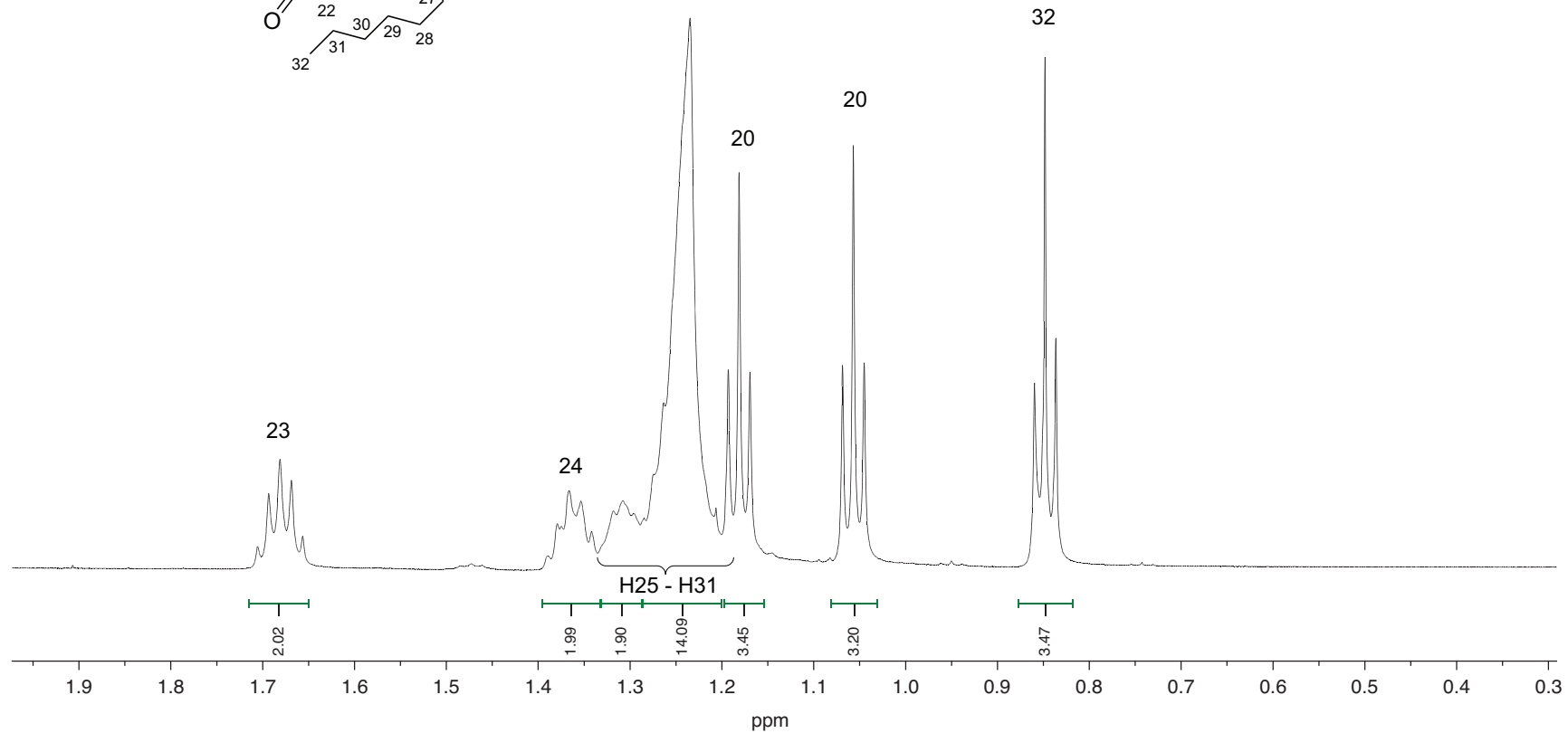
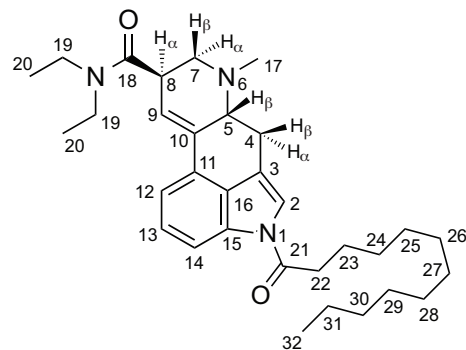
1DD-LSD tartrate (3:2)
 1H NMR (600 MHz)
 DMSO-*d*₆

1.71
 1.69
 1.68
 1.67
 1.66

1.39
 1.38
 1.37
 1.37
 1.35
 1.34
 1.32
 1.31
 1.30
 1.28
 1.26
 1.23
 1.19
 1.18
 1.17

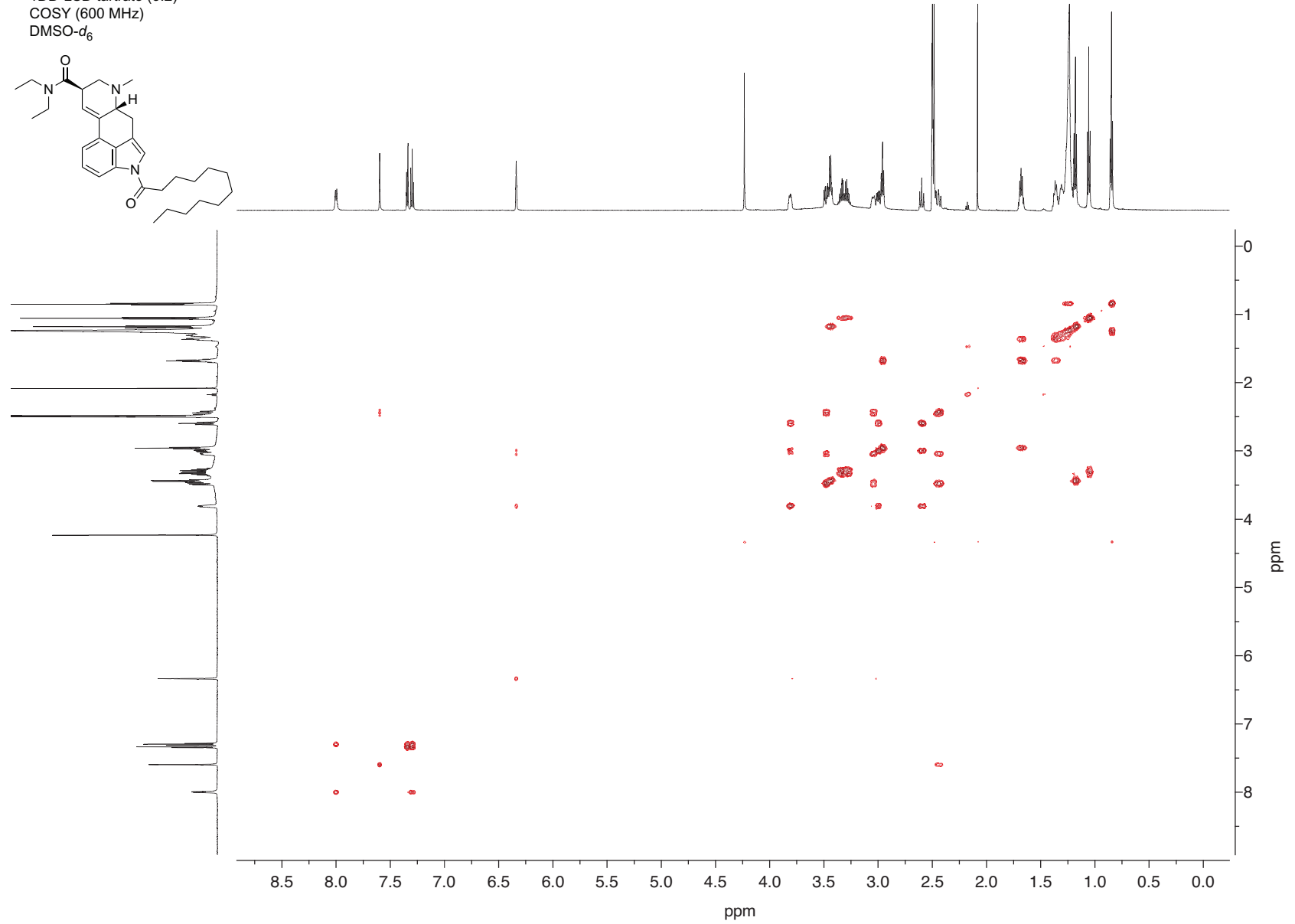
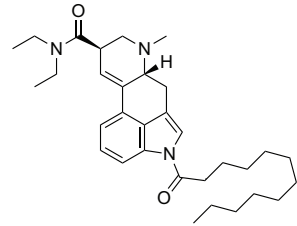
1.07
 1.06
 1.05

0.86
 0.85
 0.84



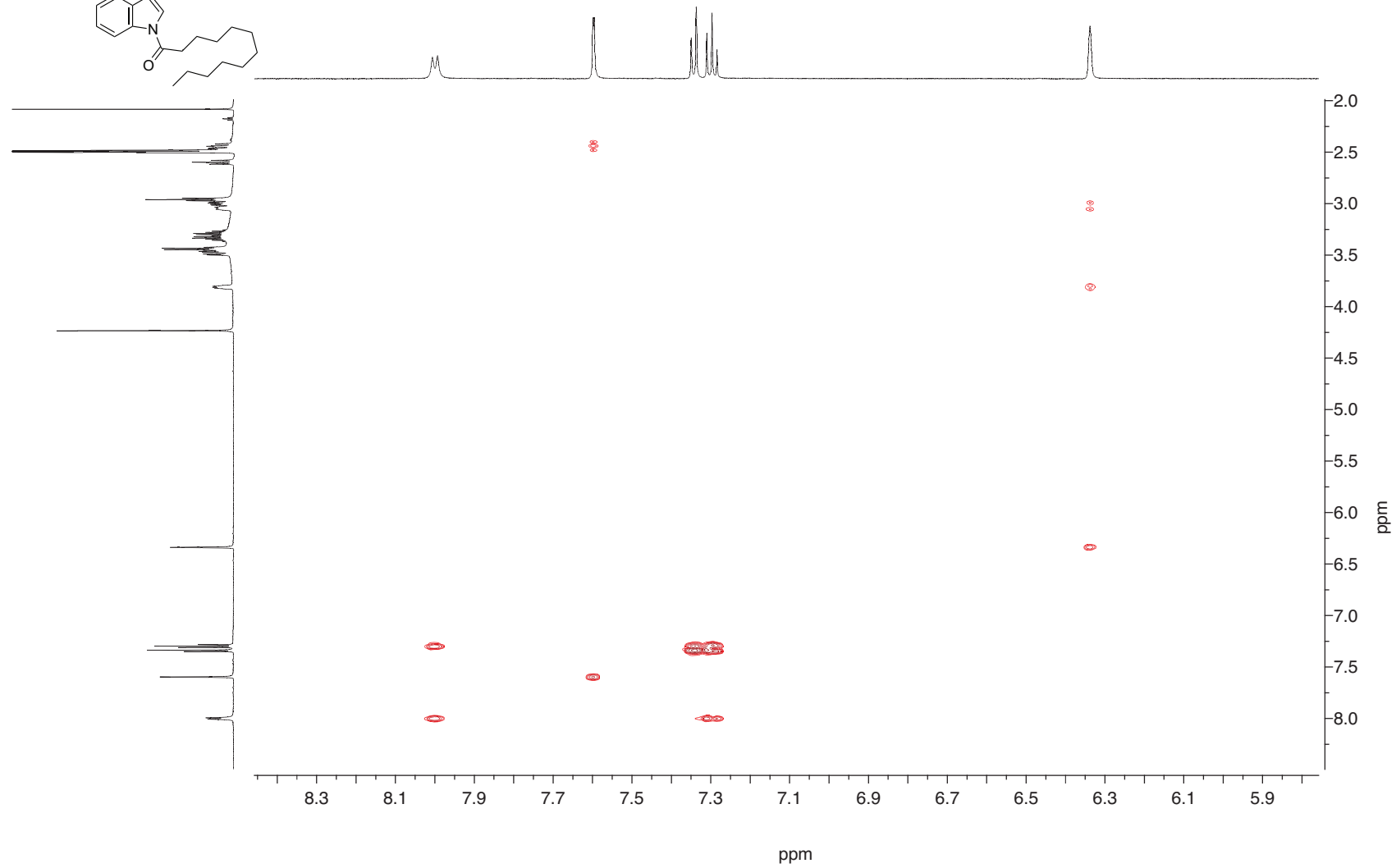
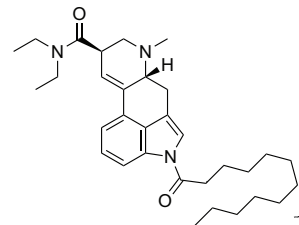
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
COSY (600 MHz)
DMSO- d_6



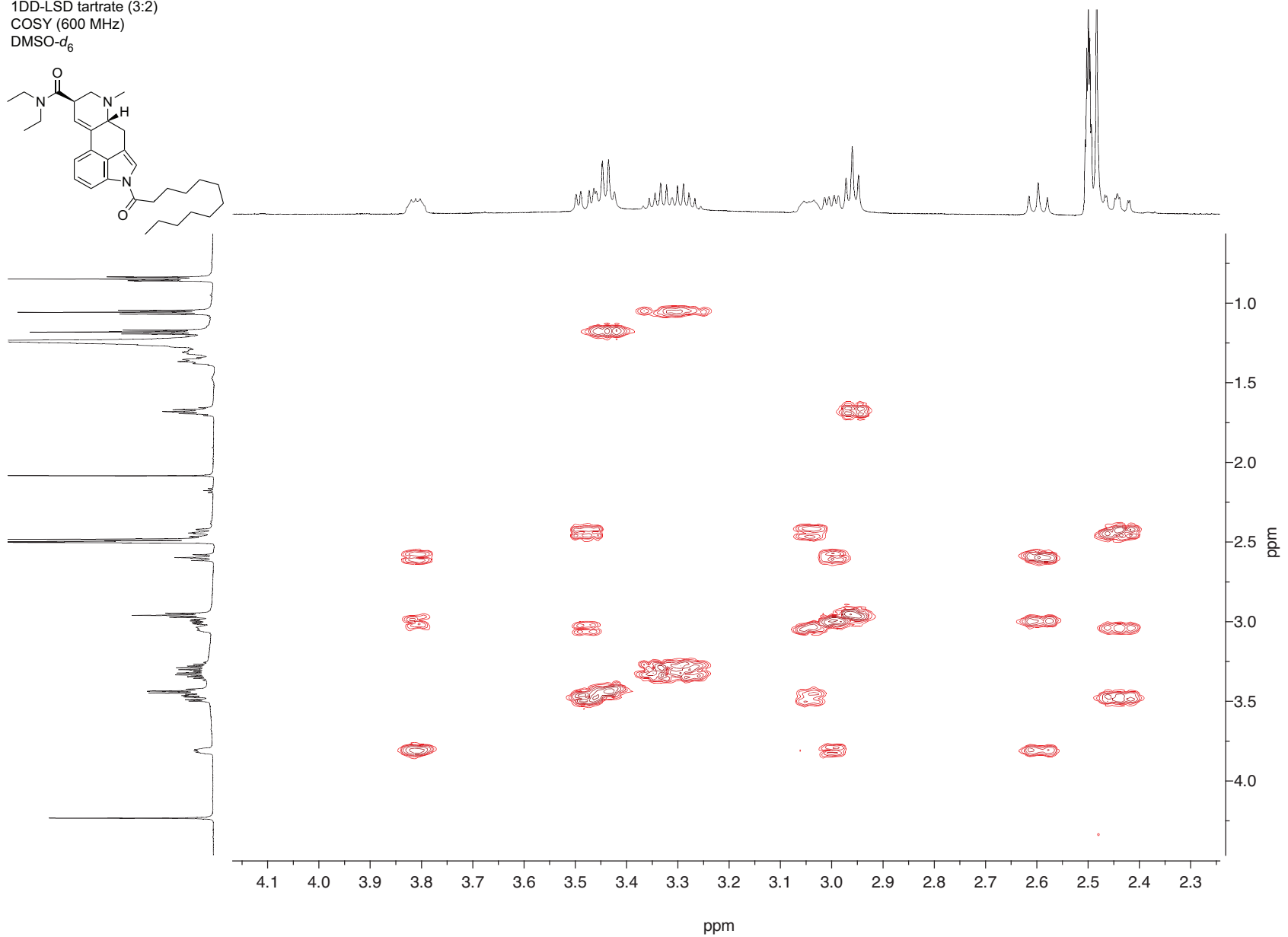
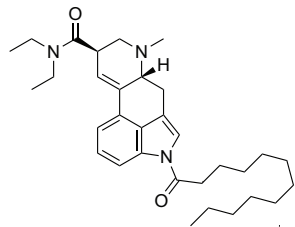
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
COSY (600 MHz)
DMSO-*d*₆



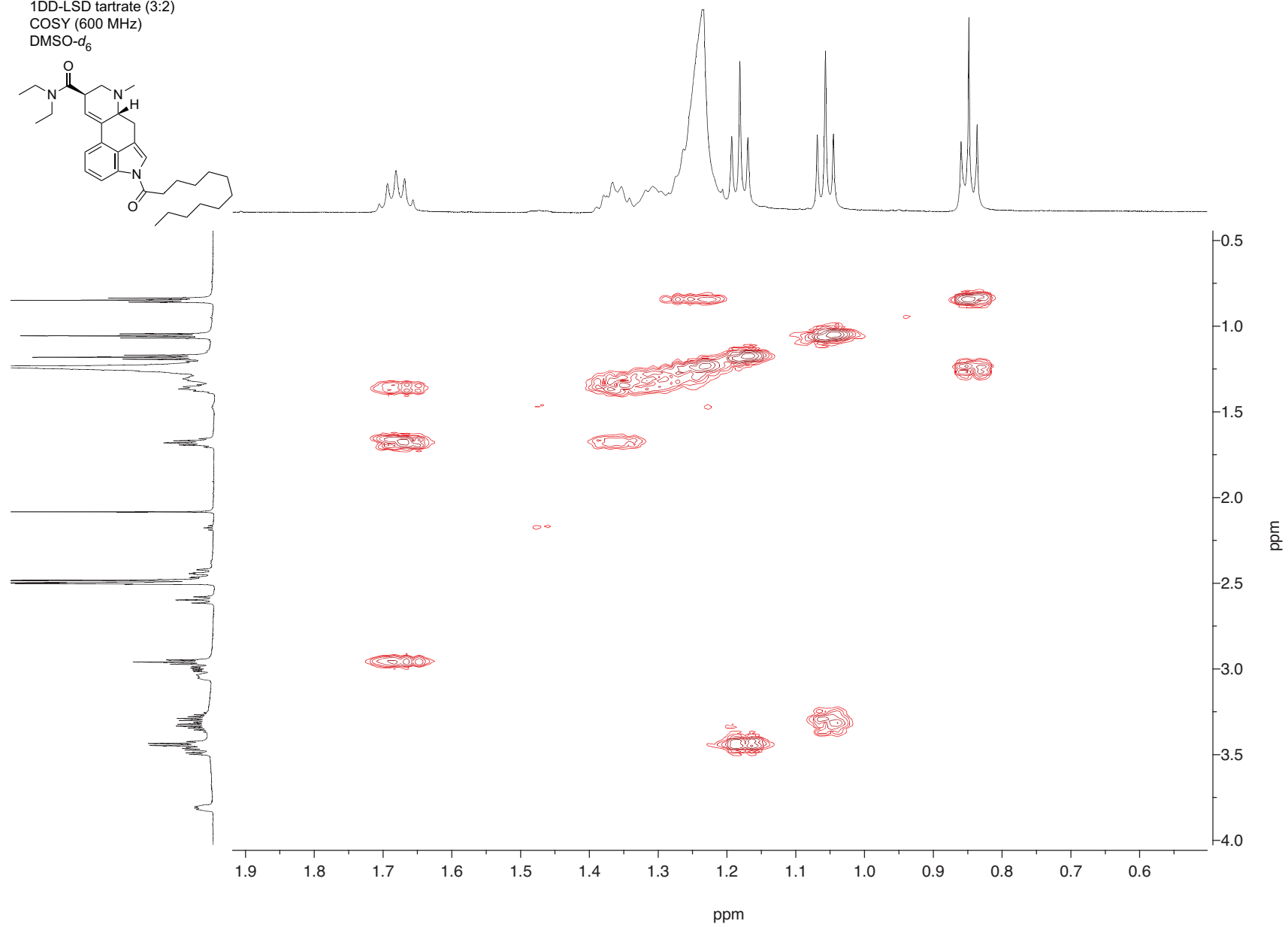
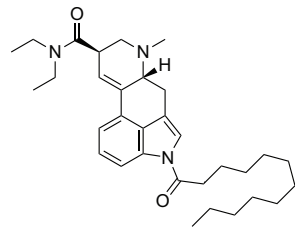
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
COSY (600 MHz)
DMSO- d_6



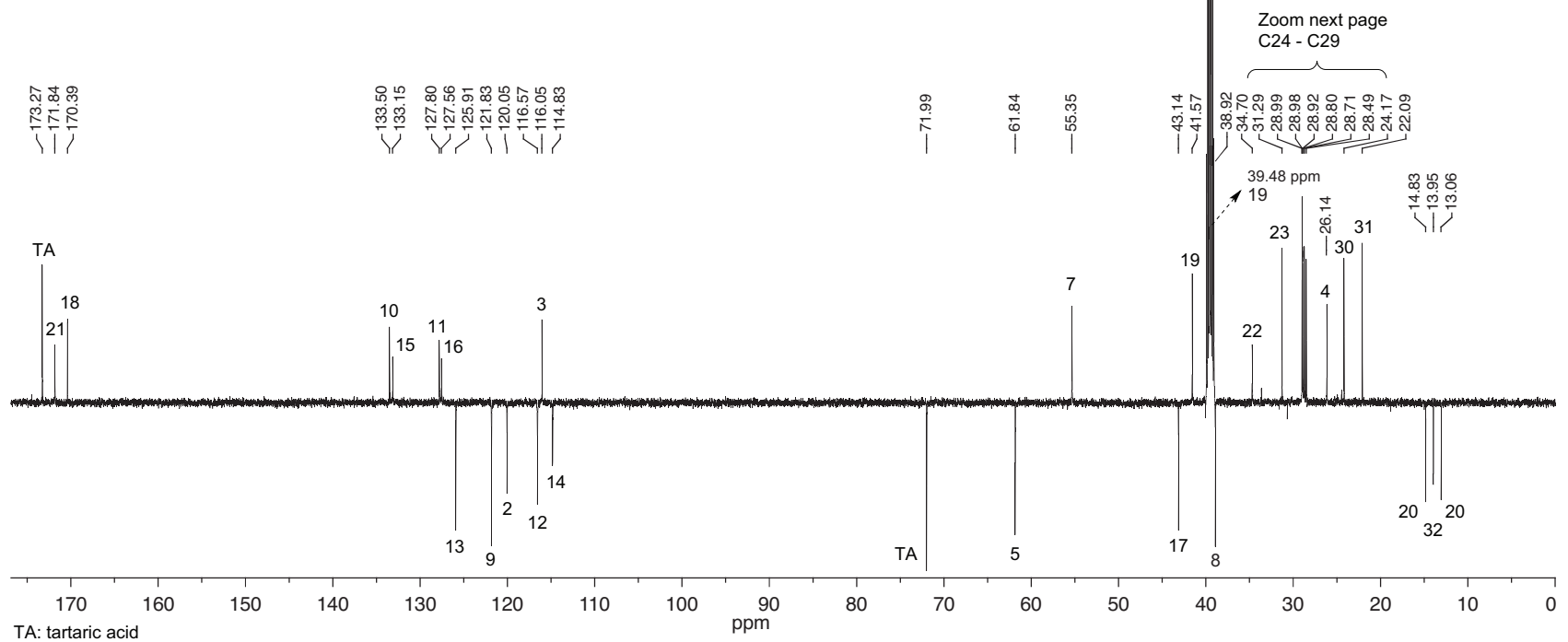
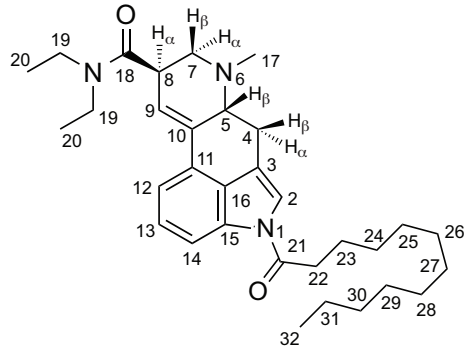
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
COSY (600 MHz)
DMSO-d₆



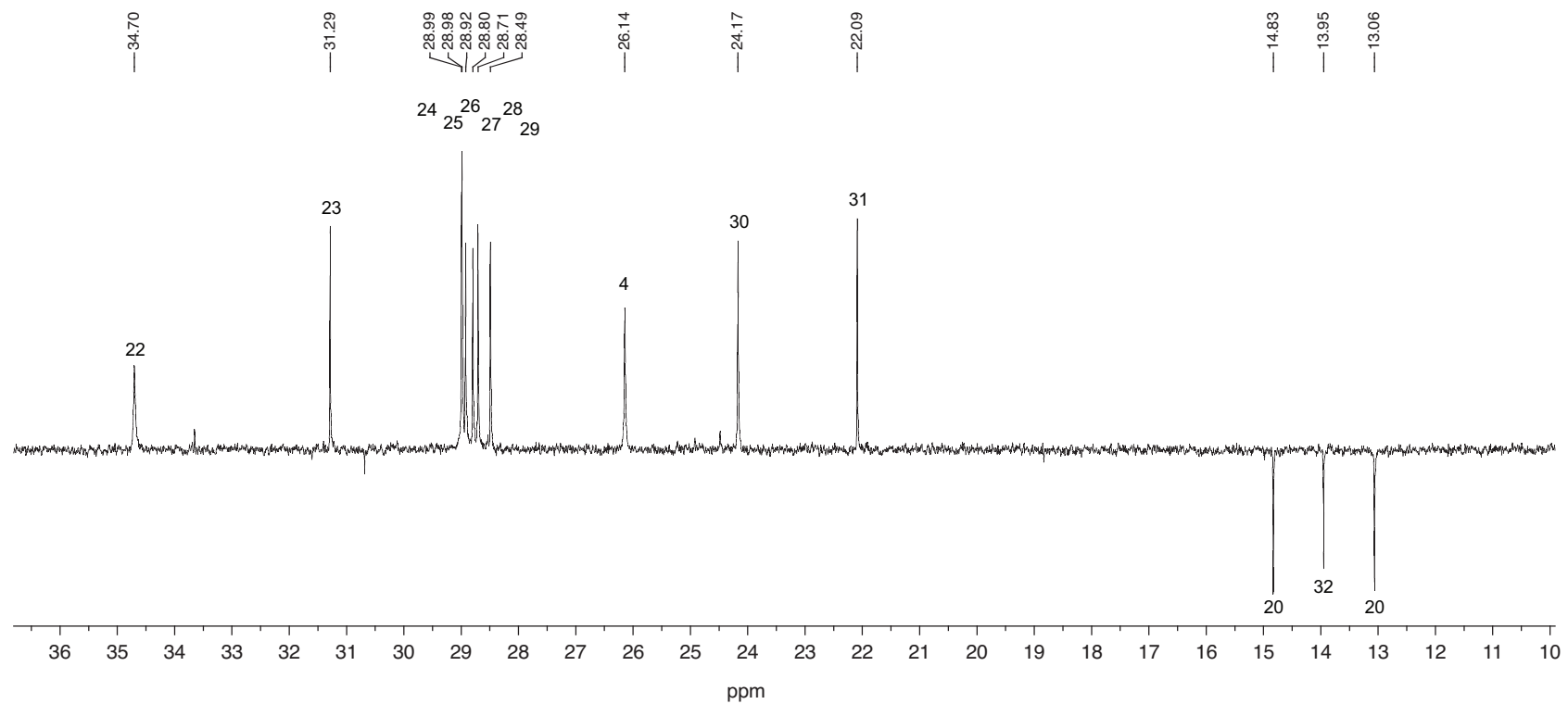
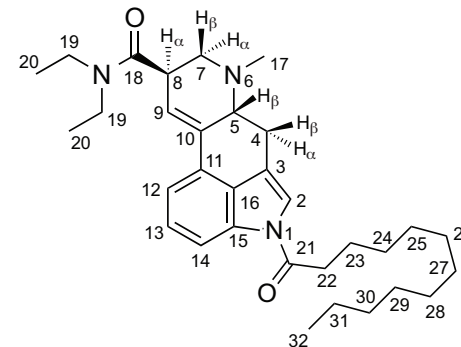
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
 DEPTQ (150 MHz)
 DMSO-*d*₆



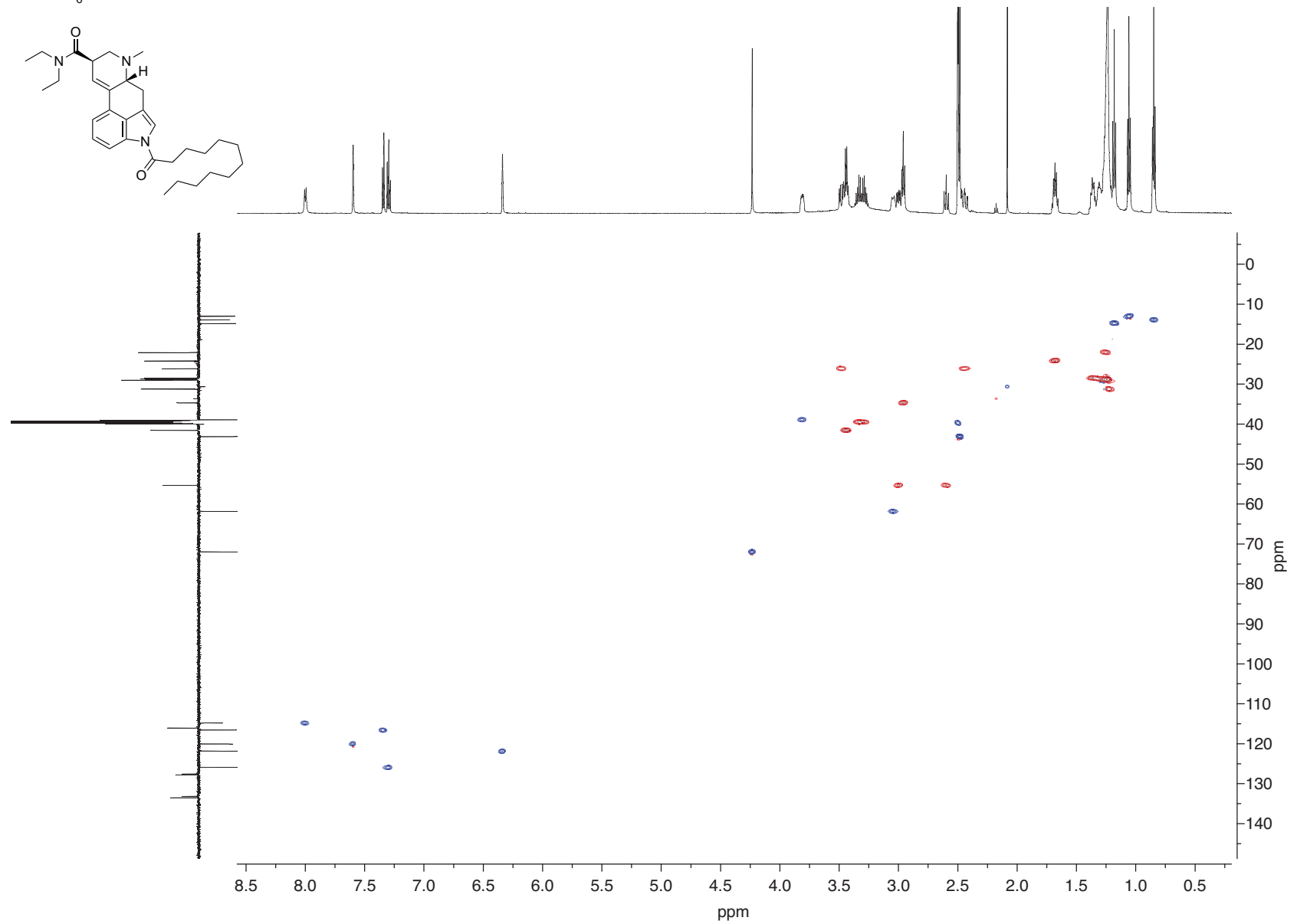
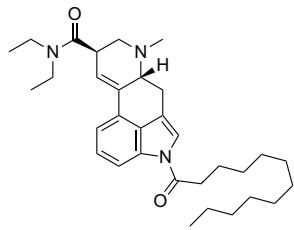
Supporting Information – Drug Testing and Analysis

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 DEPTQ (150 MHz)
 DMSO-*d*₆



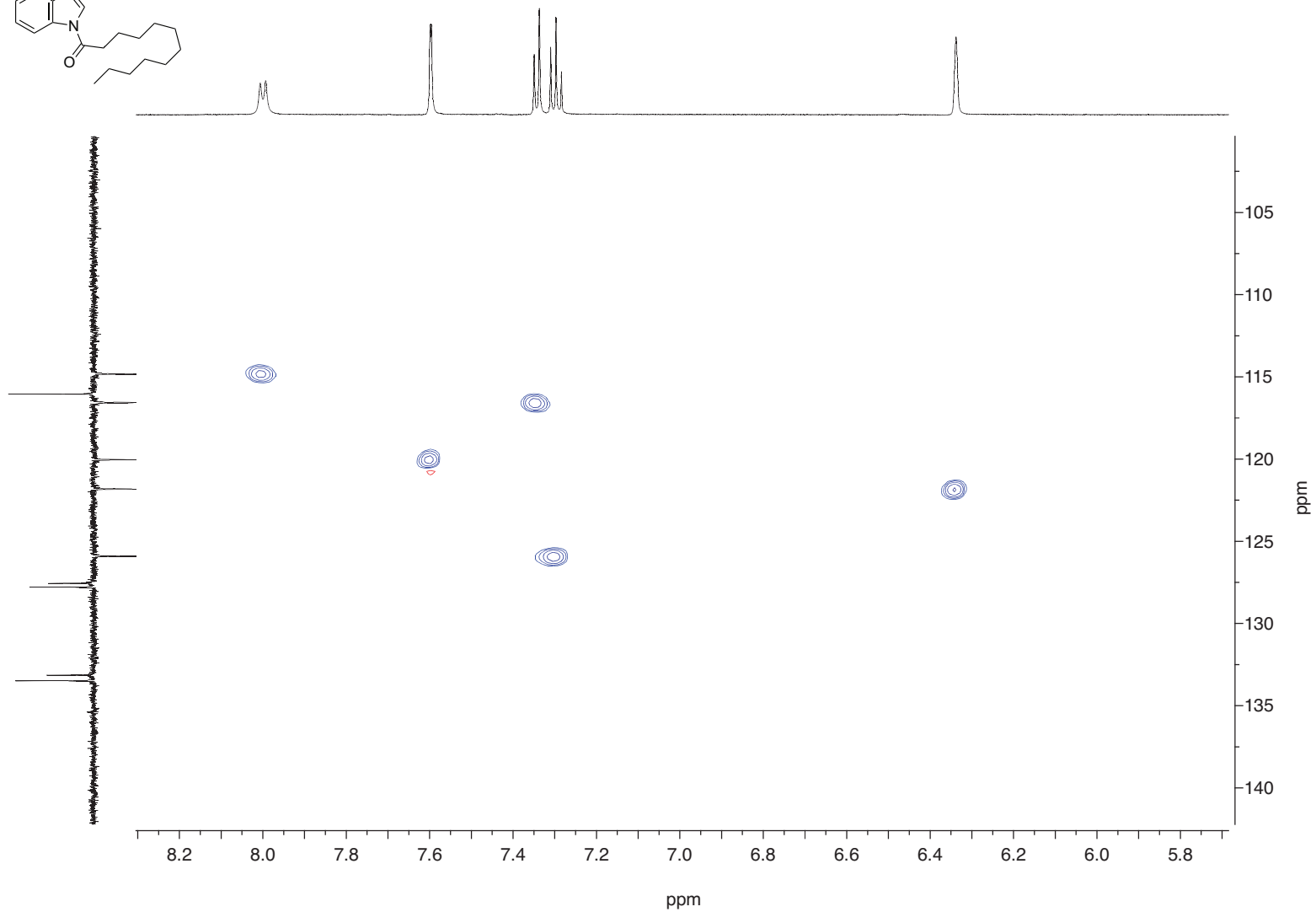
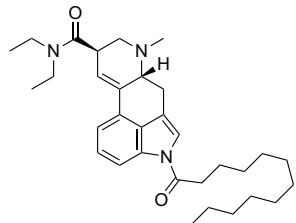
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
HSQC (600/150 MHz)
DMSO- d_6



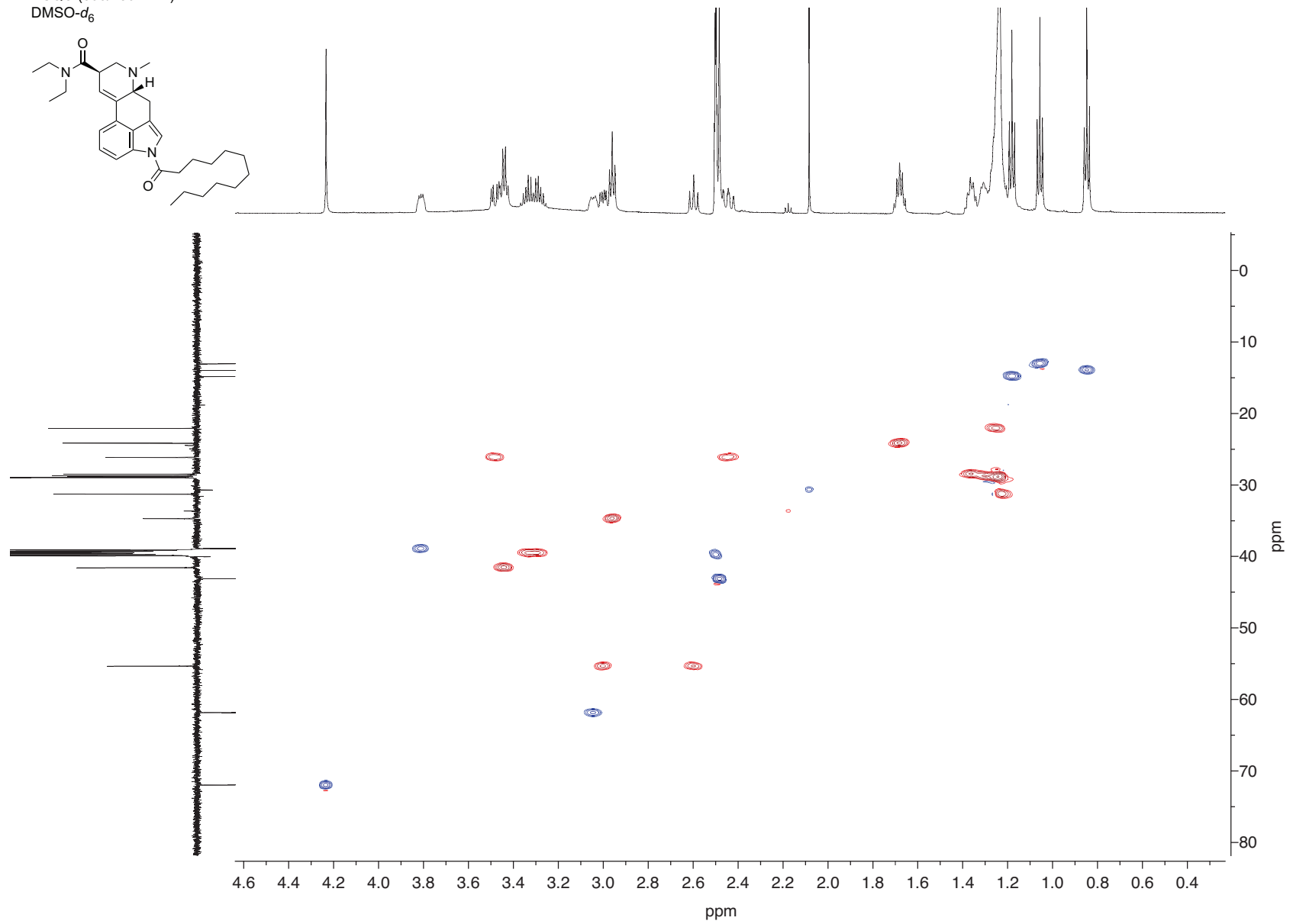
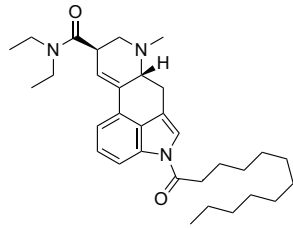
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
HSQC (600/150 MHz)
DMSO-*d*₆



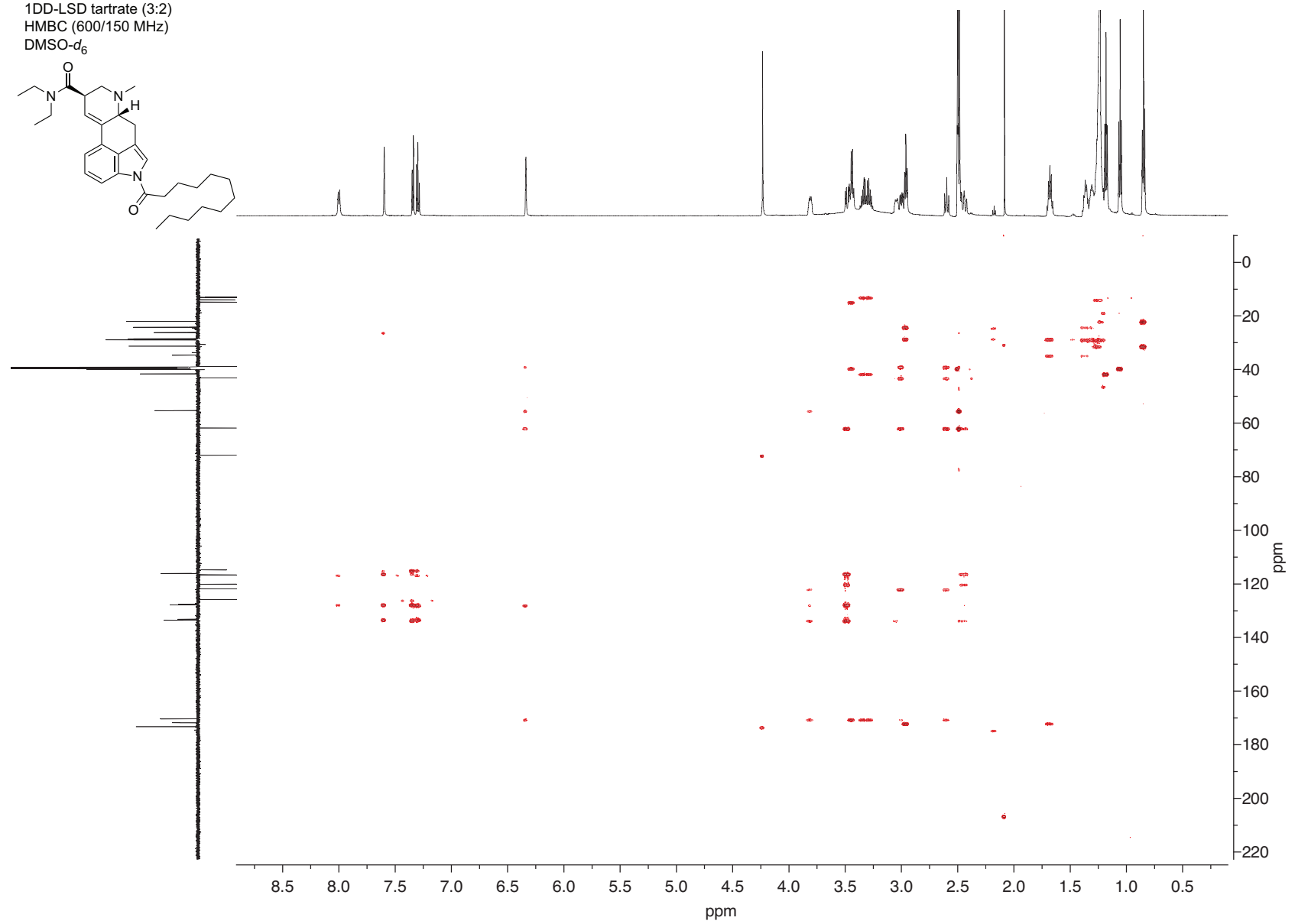
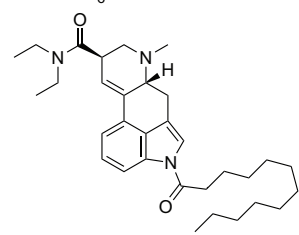
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1DD-LSD tartrate (3:2)
HSQC (600/150 MHz)
DMSO- d_6



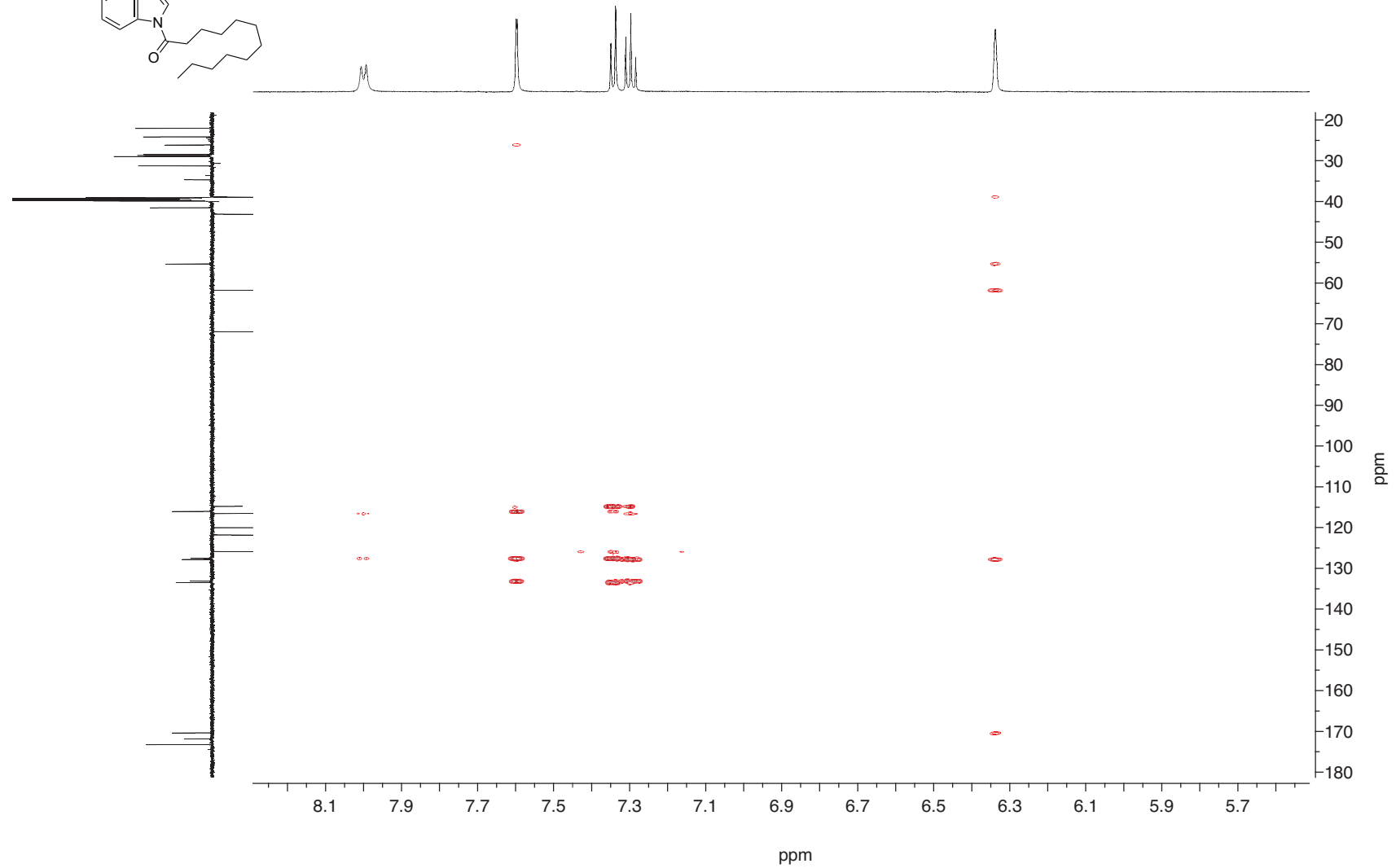
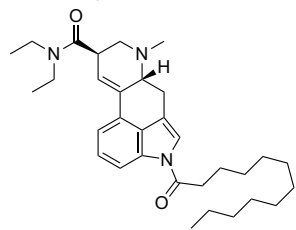
Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
HMBC (600/150 MHz)
DMSO-*d*₆



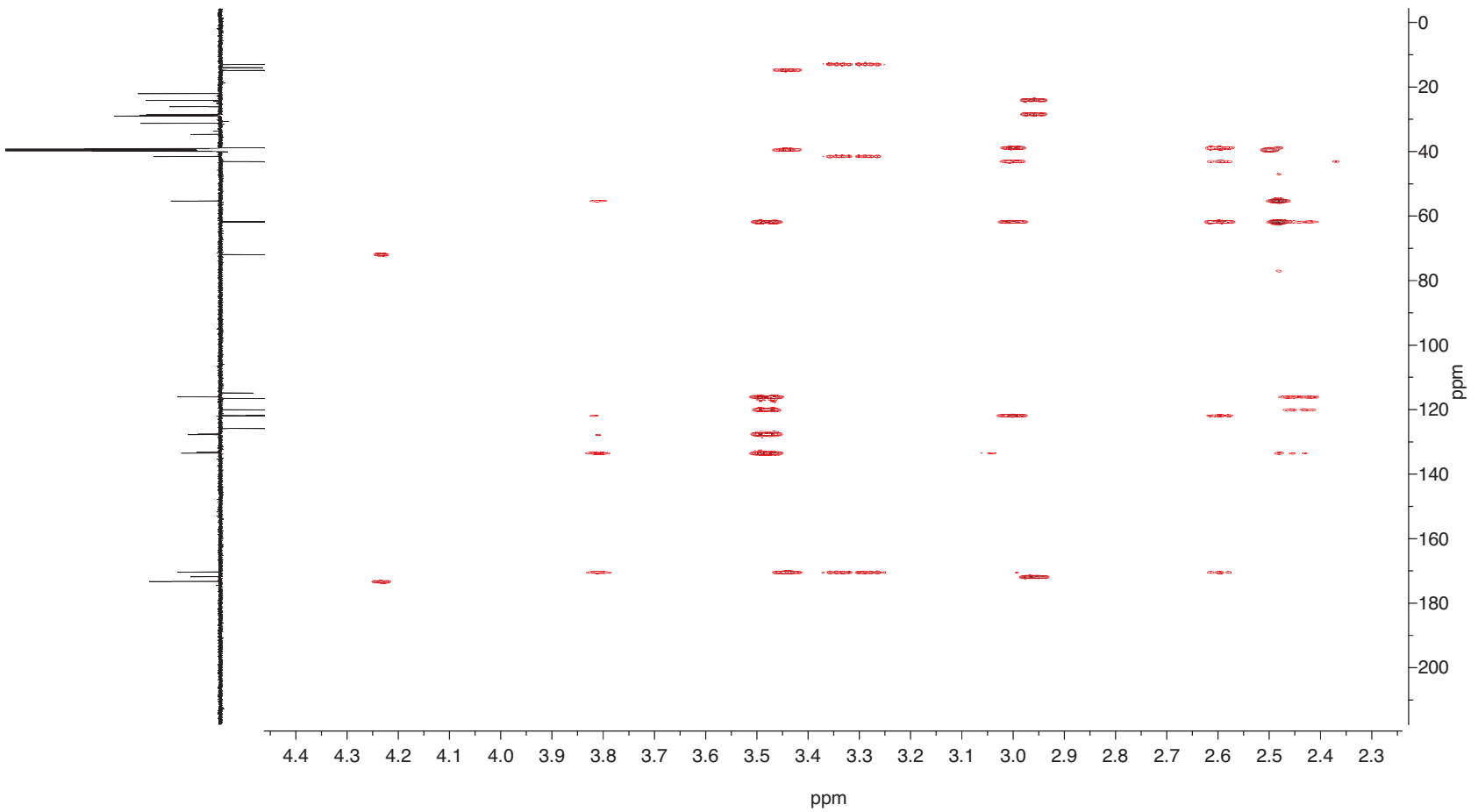
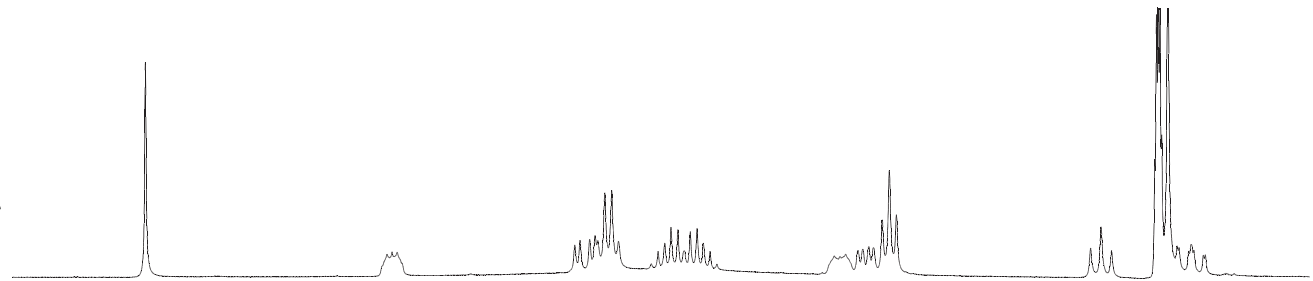
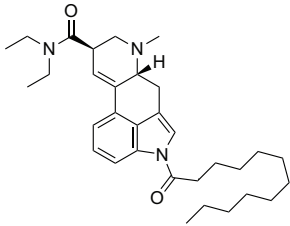
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1DD-LSD tartrate (3:2)
HMBC (600/150 MHz)
DMSO-*d*₆



Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)
HMBC (600/150 MHz)
DMSO-*d*₆



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1DD-LSD tartrate (3:2)
HMBC (600/150 MHz)
DMSO- d_6

