

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

Pierce V. Kavanagh,<sup>1</sup> Folker Westphal,<sup>2</sup> Benedikt Pulver,<sup>2,3</sup> Simon P. Elliott,<sup>4,5</sup> Alexander Stratford,<sup>6</sup> Adam L. Halberstadt,<sup>7,8</sup> Simon D. Brandt,<sup>9\*</sup>

<sup>1</sup> *Department of Pharmacology and Therapeutics, School of Medicine, Trinity Centre for Health Sciences, St. James Hospital, Dublin 8, Ireland*

<sup>2</sup> *State Bureau of Criminal Investigation Schleswig-Holstein, Section Narcotics/Toxicology, Kiel, Germany*

<sup>3</sup> *Institute of Forensic Medicine, Forensic Toxicology, Medical Center, Faculty of Medicine, University of Freiburg, Freiburg, Germany.*

<sup>4</sup> *Elliott Forensic Consulting, Birmingham, UK*

<sup>5</sup> *Department of Analytical, Environmental and Forensic Sciences, King's College London, London SE1 9NH, UK*

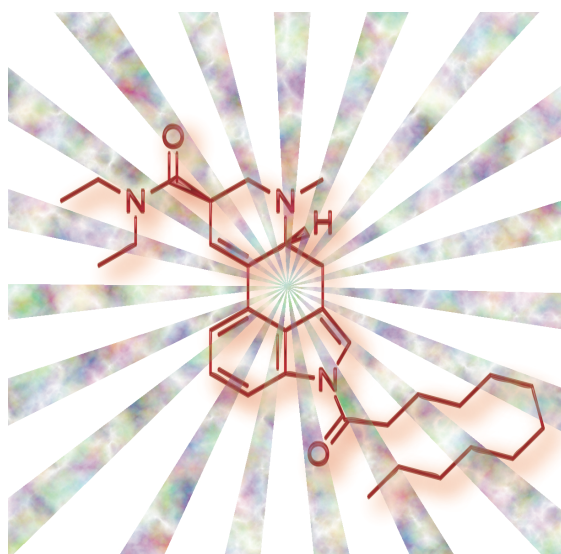
<sup>6</sup> *Synex Synthetics BV, Maastricht, The Netherlands*

<sup>7</sup> *Department of Psychiatry, University of California San Diego, La Jolla, USA*

<sup>8</sup> *Research Service, VA San Diego Healthcare System, San Diego, USA*

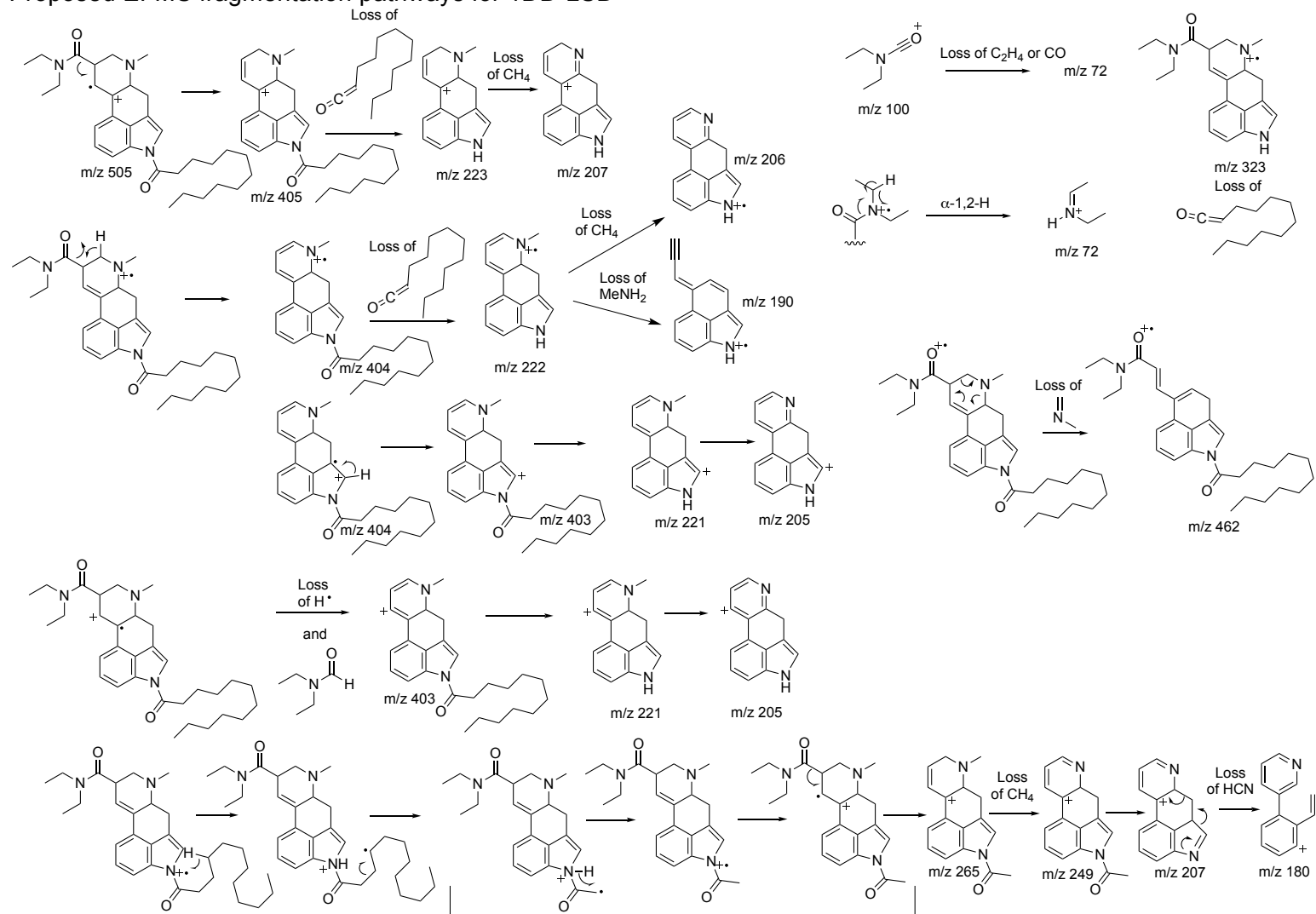
<sup>9</sup> *School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK*

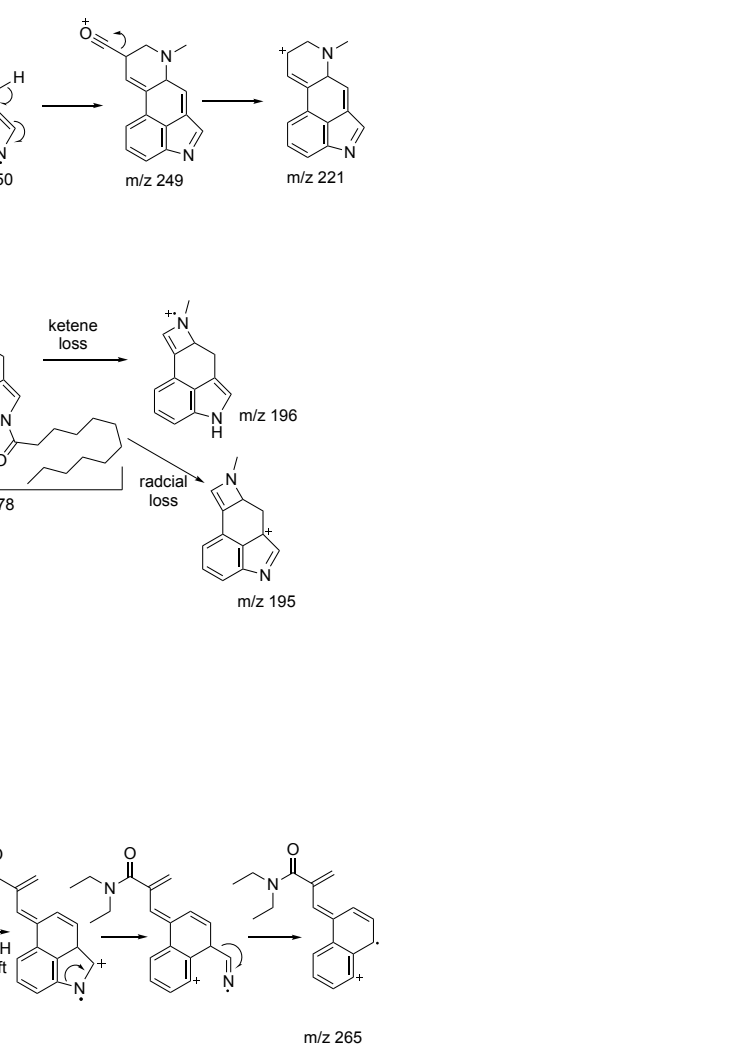
\* Correspondence to: Simon D. Brandt, School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK. E-Mail: [s.brandt@ljmu.ac.uk](mailto:s.brandt@ljmu.ac.uk)



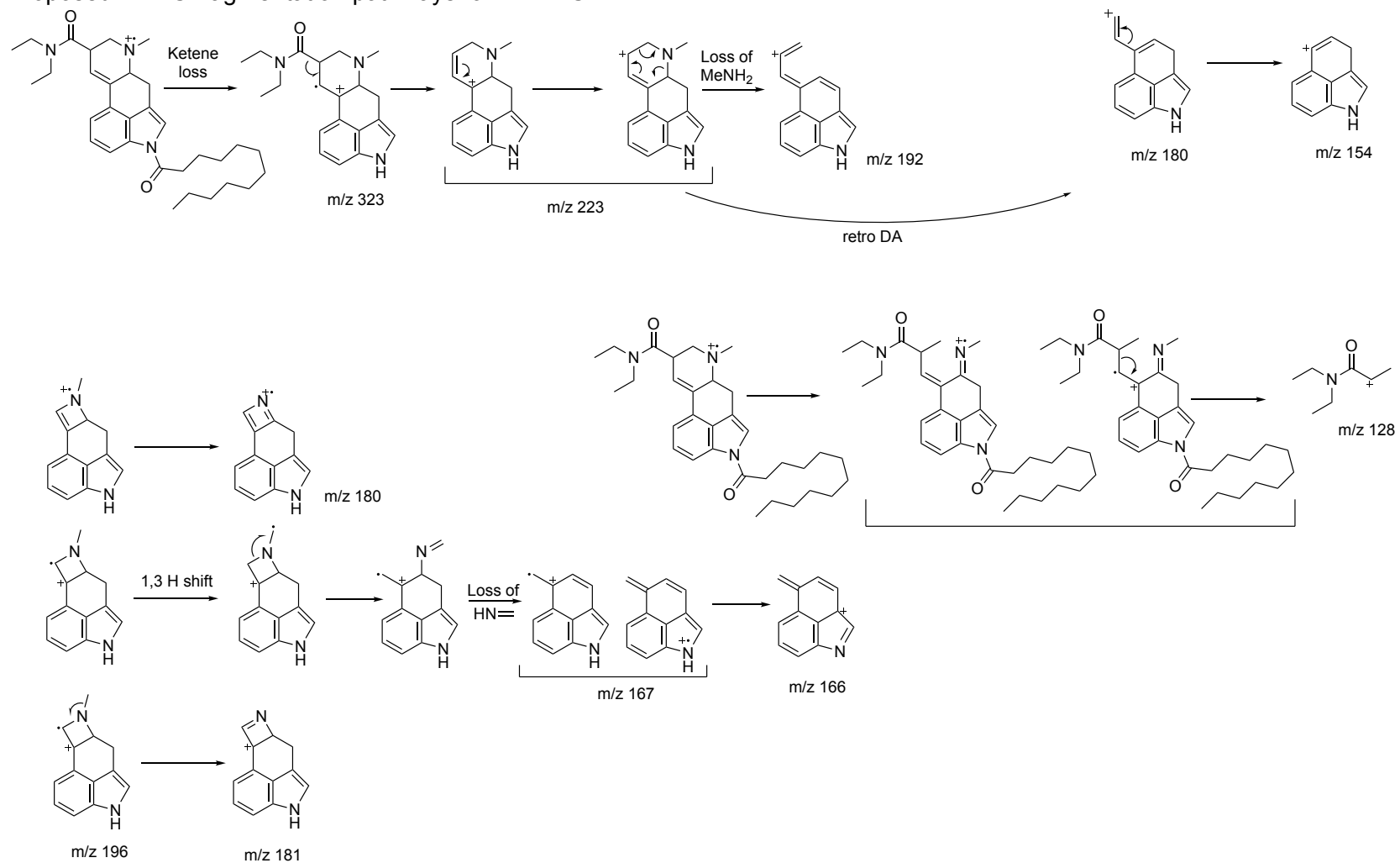
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## Proposed EI-MS fragmentation pathways for 1DD-LSD

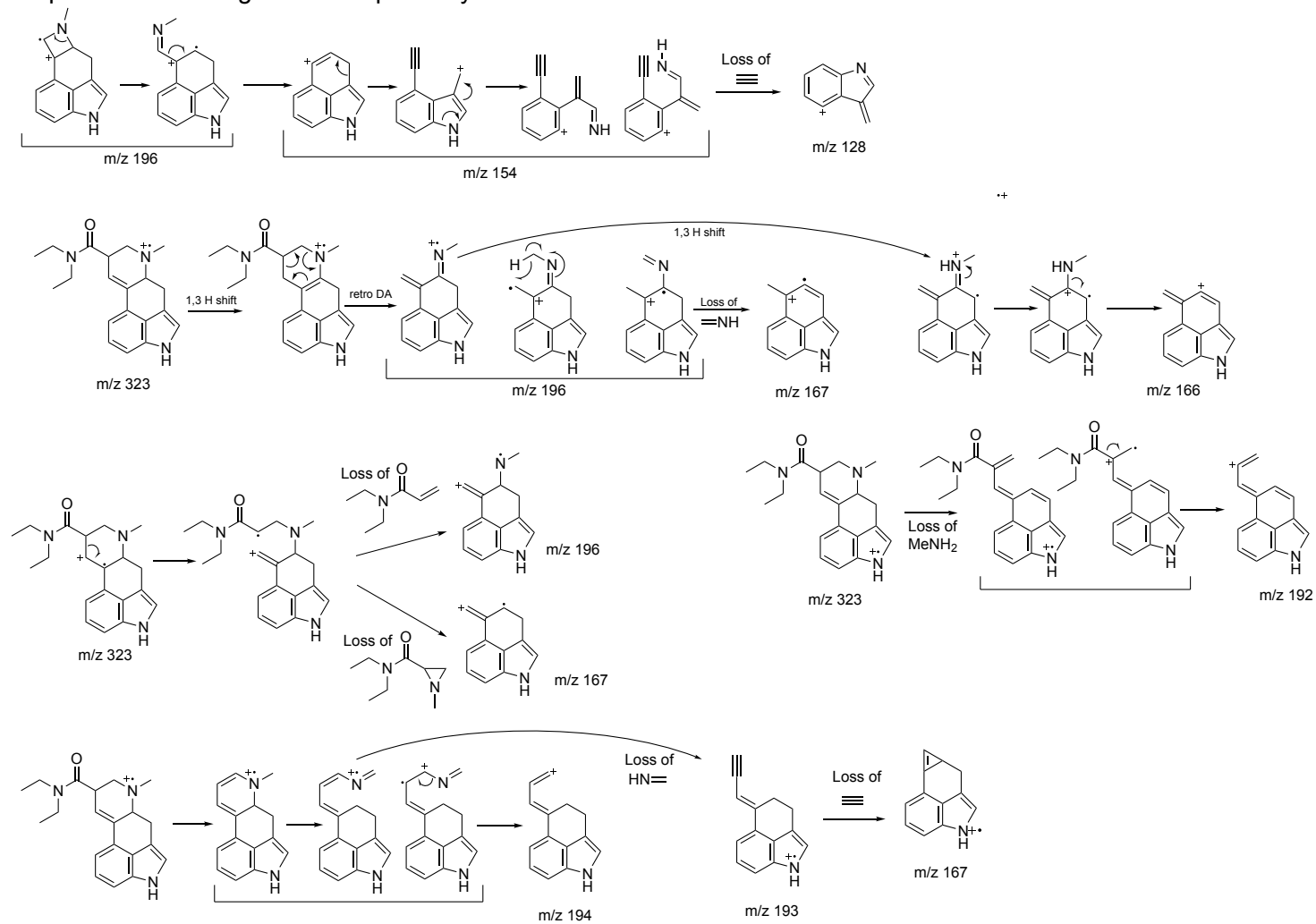




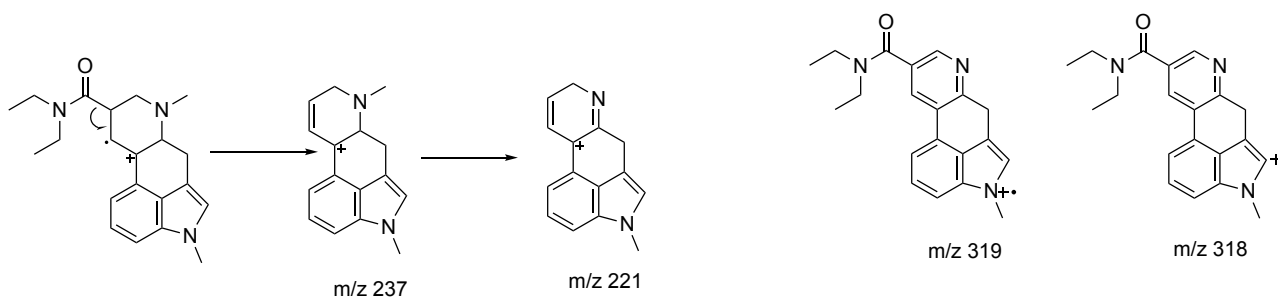
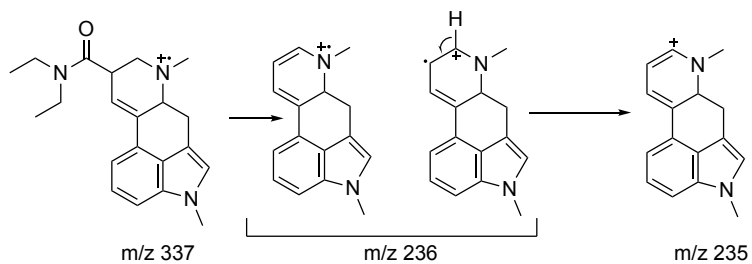
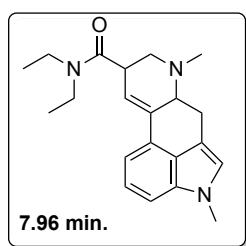
## Proposed EI-MS fragmentation pathways for 1DD-LSD



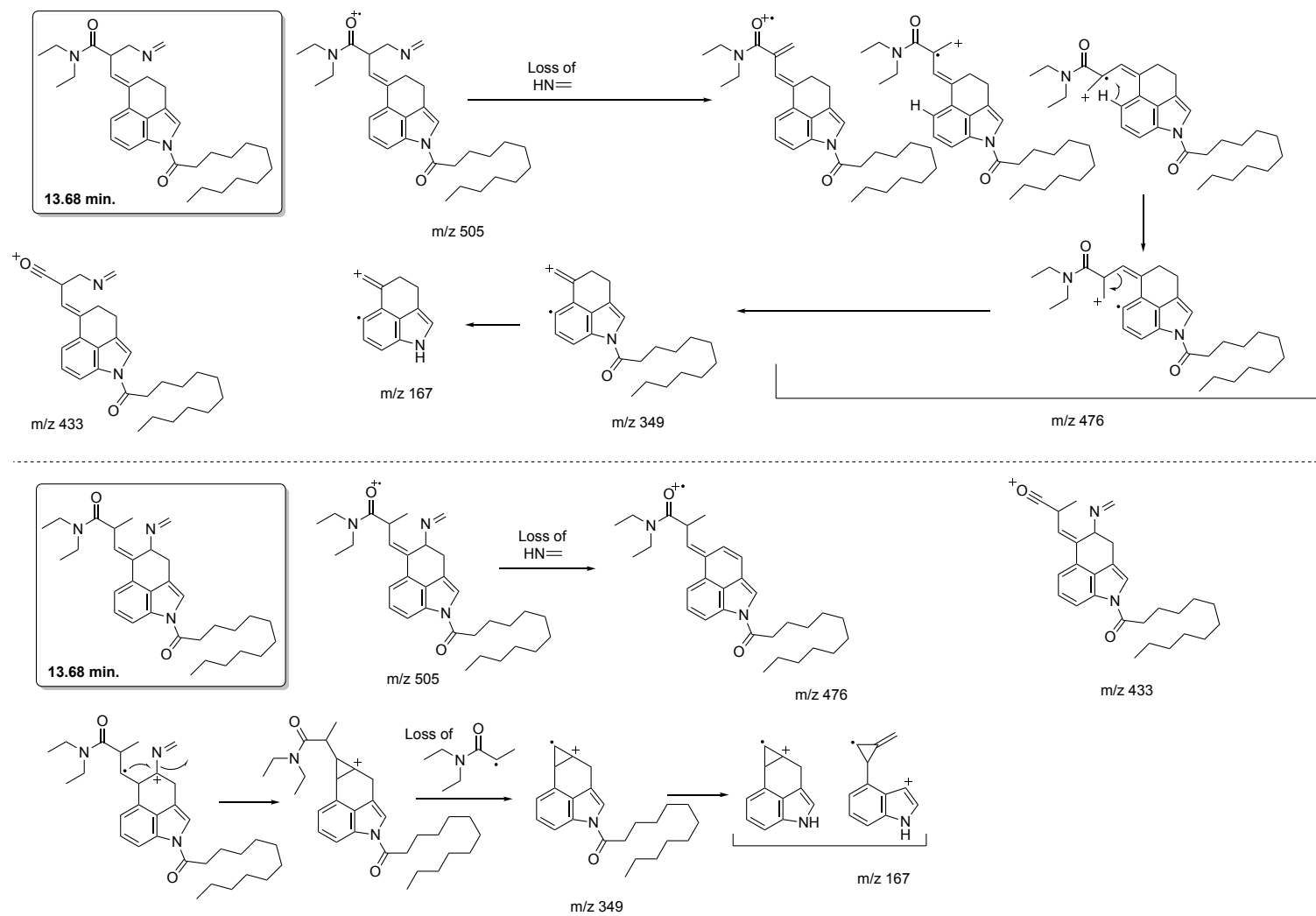
## Proposed EI-MS fragmentation pathways for 1DD-LSD



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

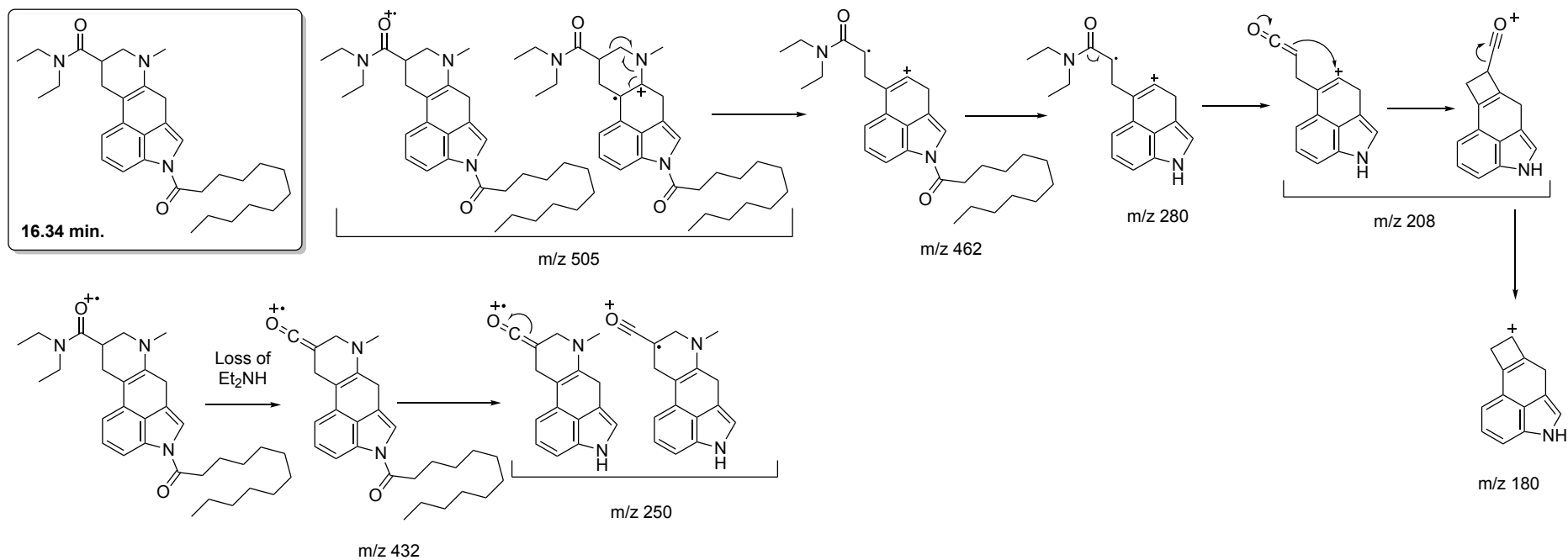


## Tentative identification of GC-induced artifacts (GC-MS method 1)

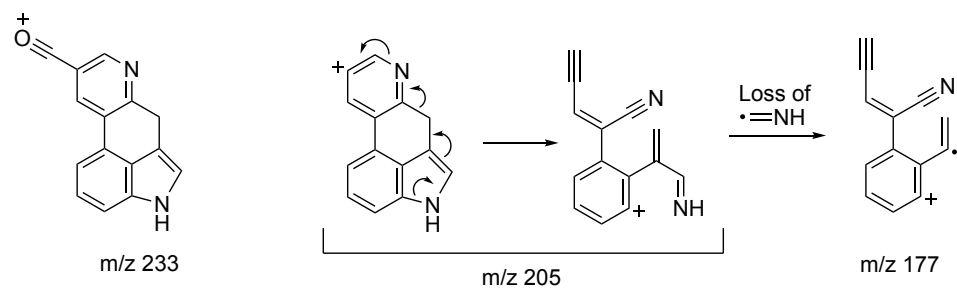
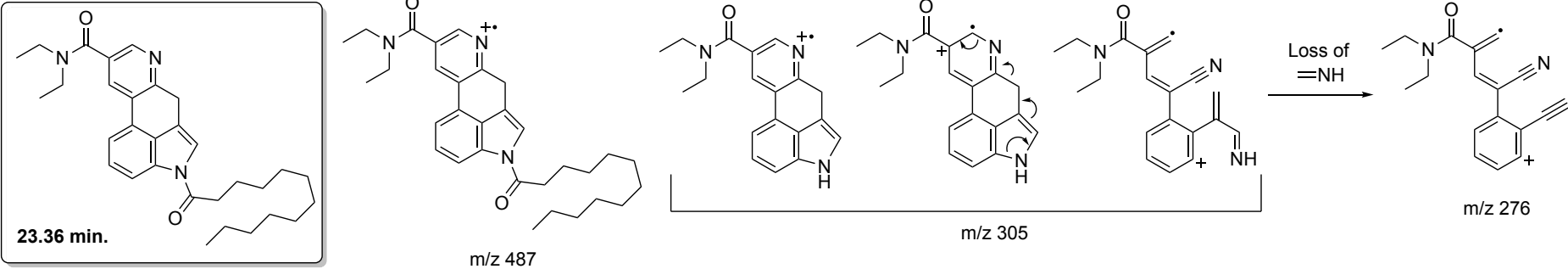




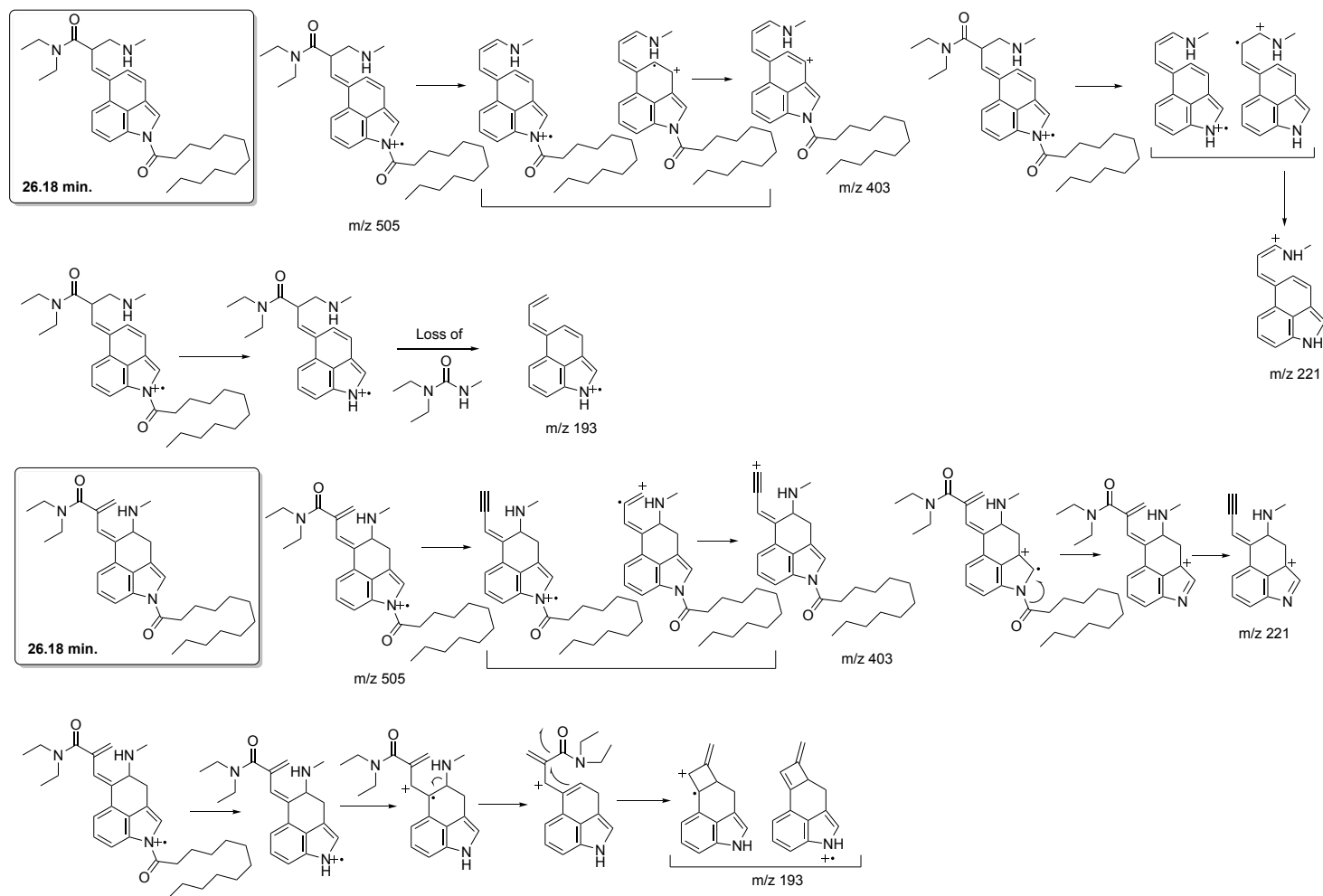
## Tentative identification of GC-induced artifacts (GC-MS method 1)



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



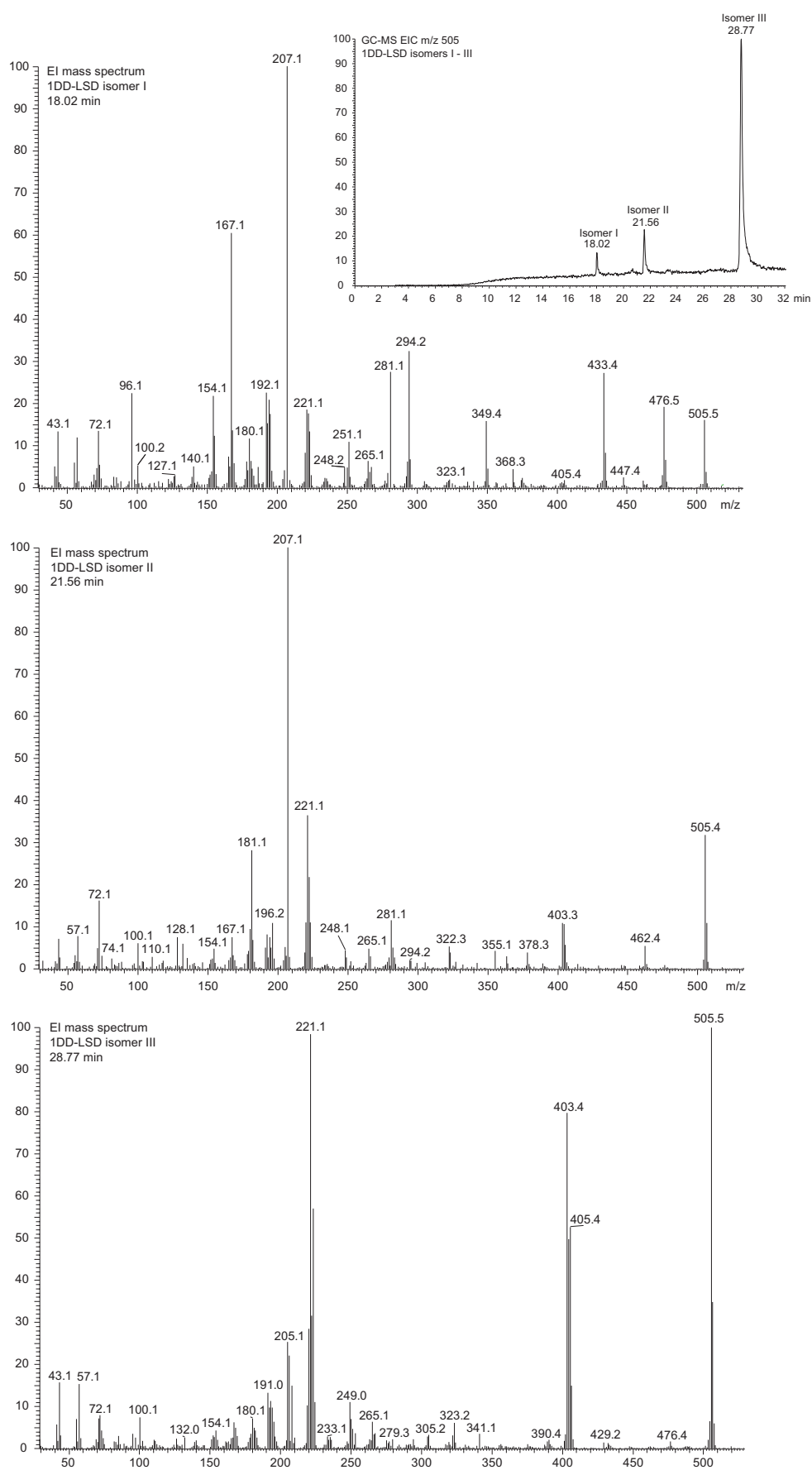
## Tentative identification of GC-induced artifacts (GC-MS method 1)



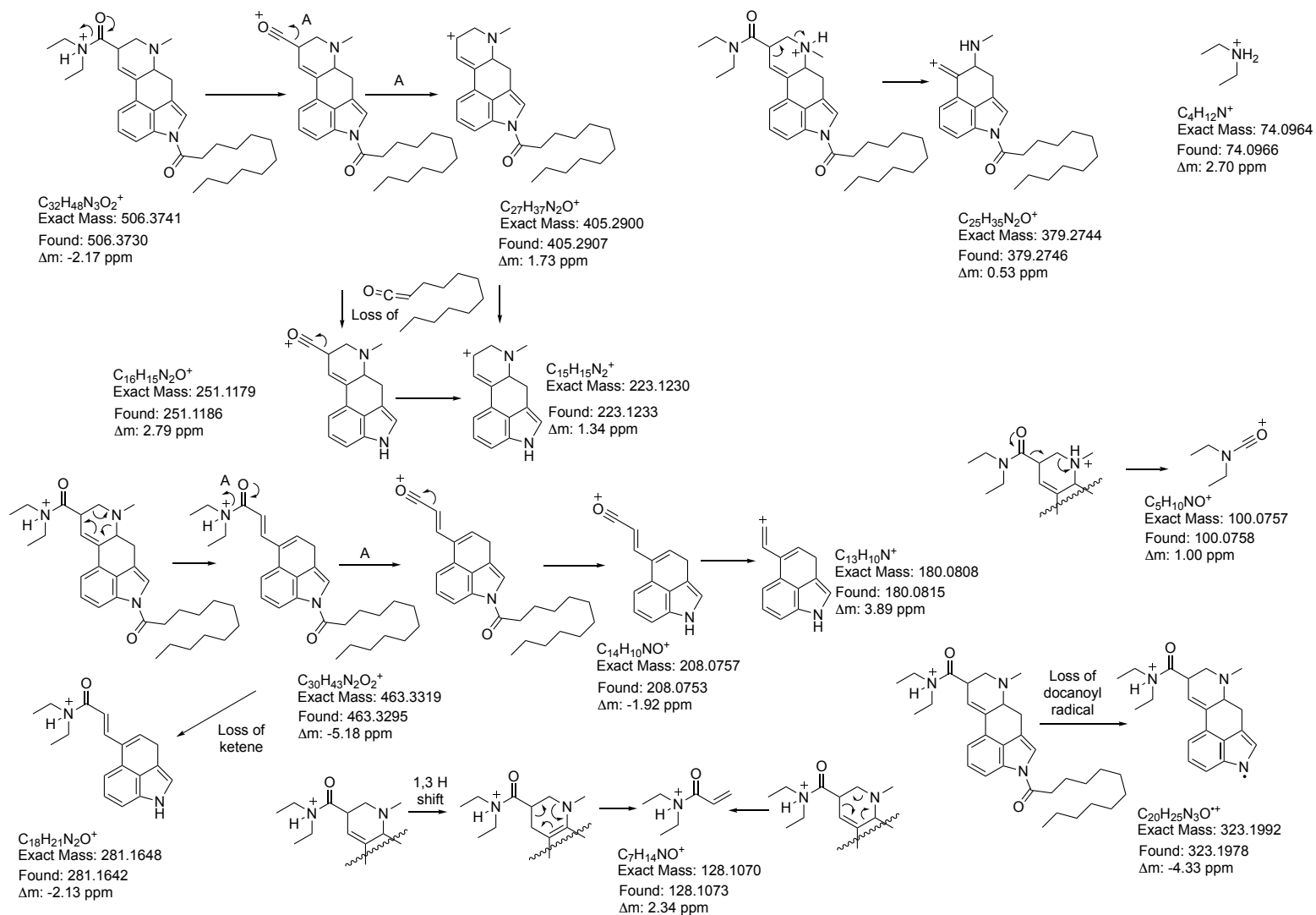
## **Gas chromatography-mass spectrometry (GC-MS) – method 2**

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  $\mu$ A. 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  $\times$  0.25 mm, film thickness 0.25  $\mu$ m). 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).

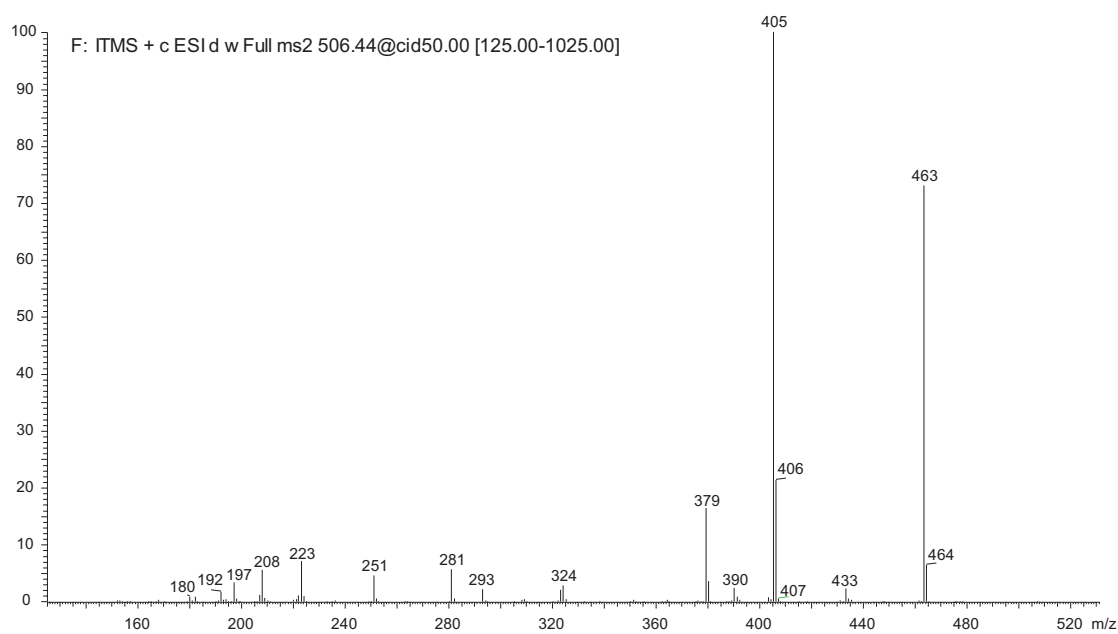
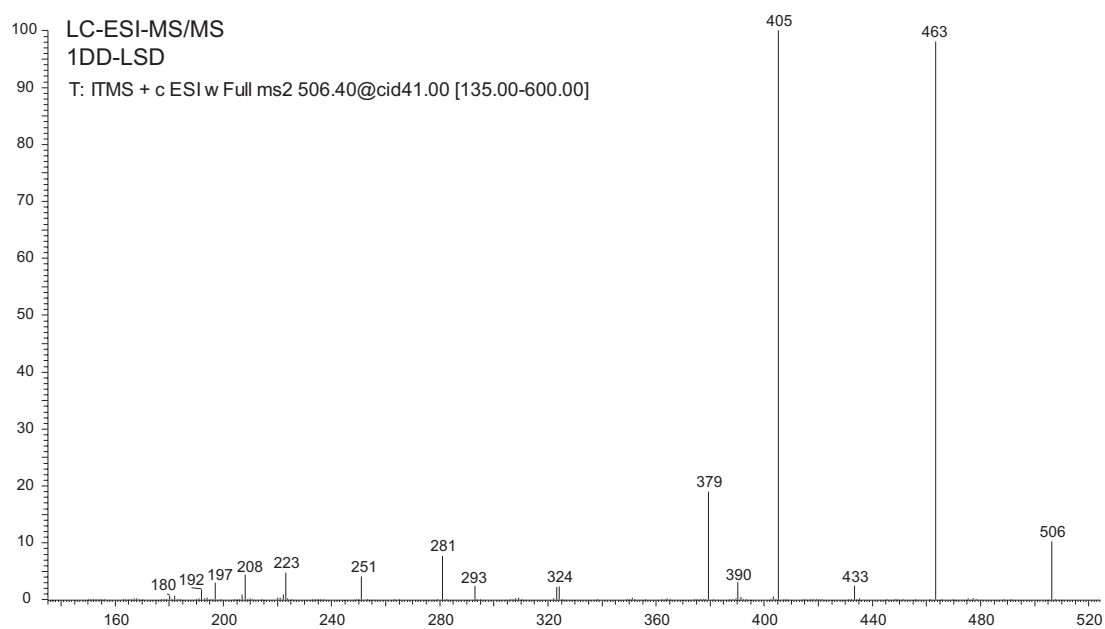
## GC-MS data (method 2)



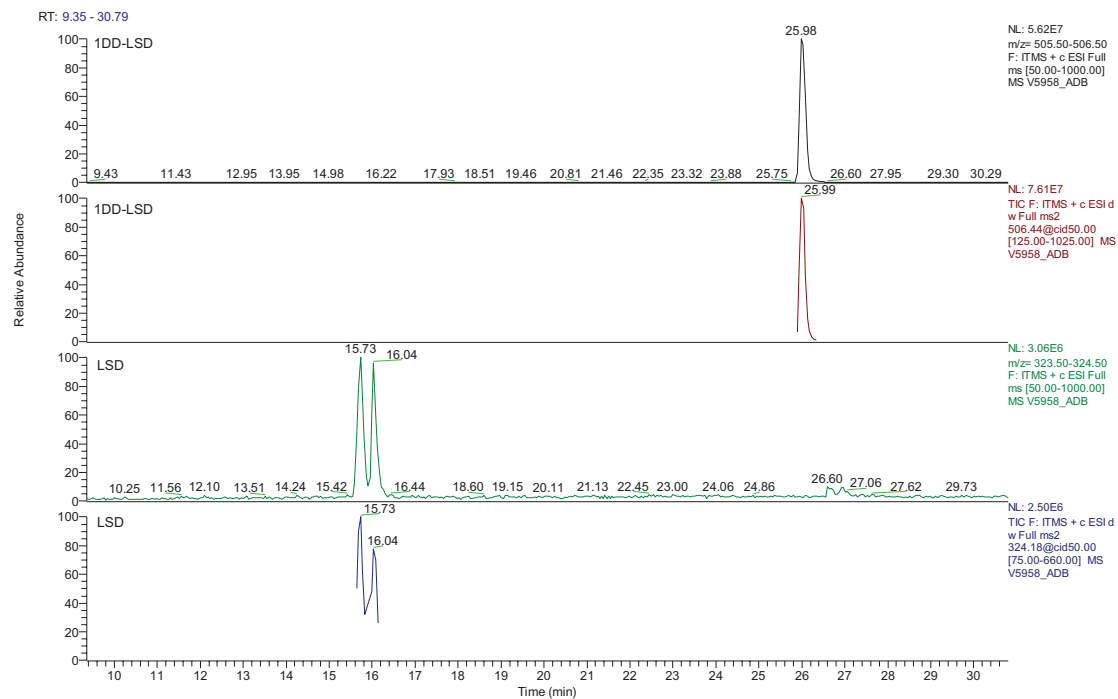
## Proposed ESI-QTOF-MS/MS fragmentation pathways for 1DD-LSD



# LC-ESI-linear ion trap-MS/MS



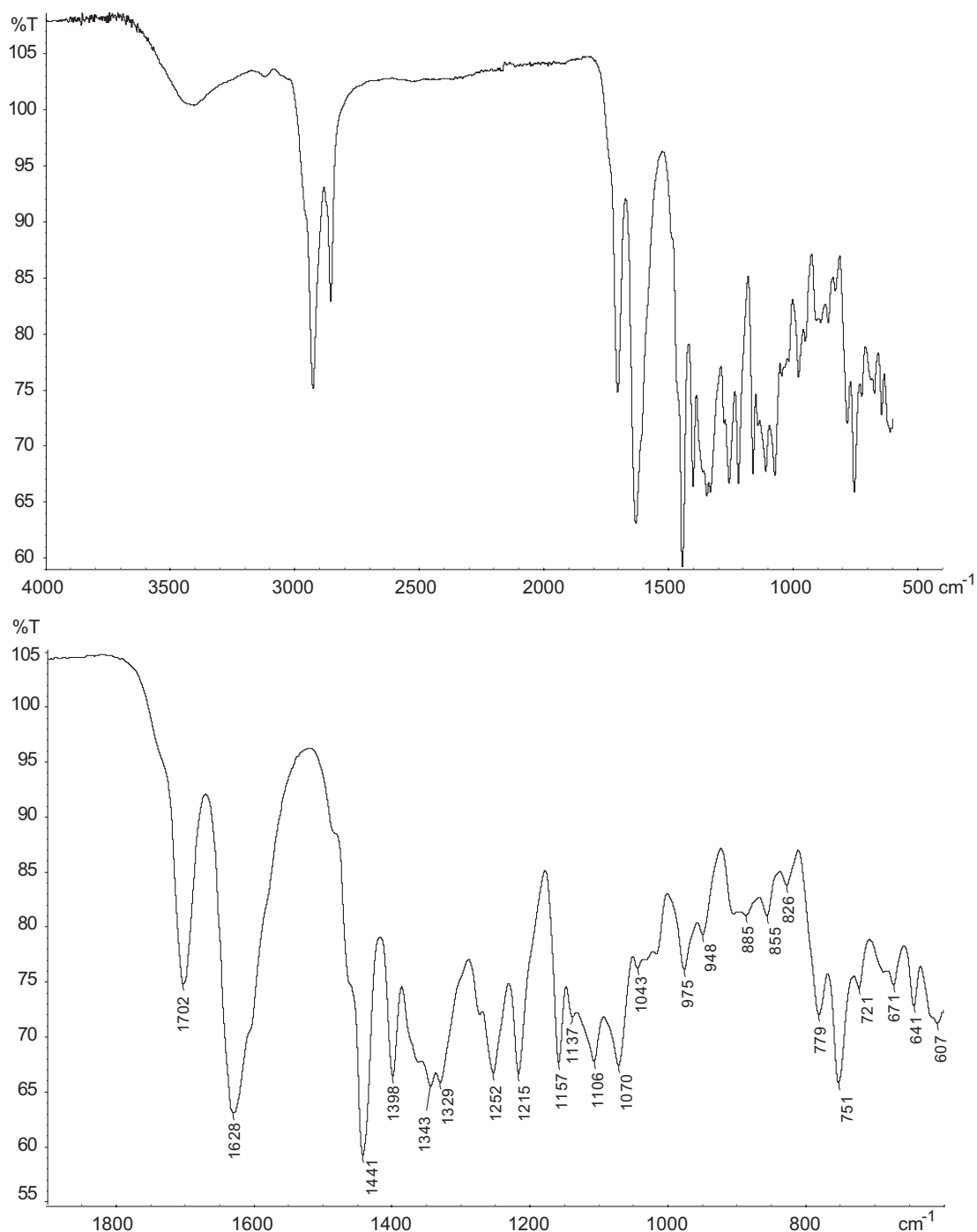
# LC-ESI-linear ion trap-MS





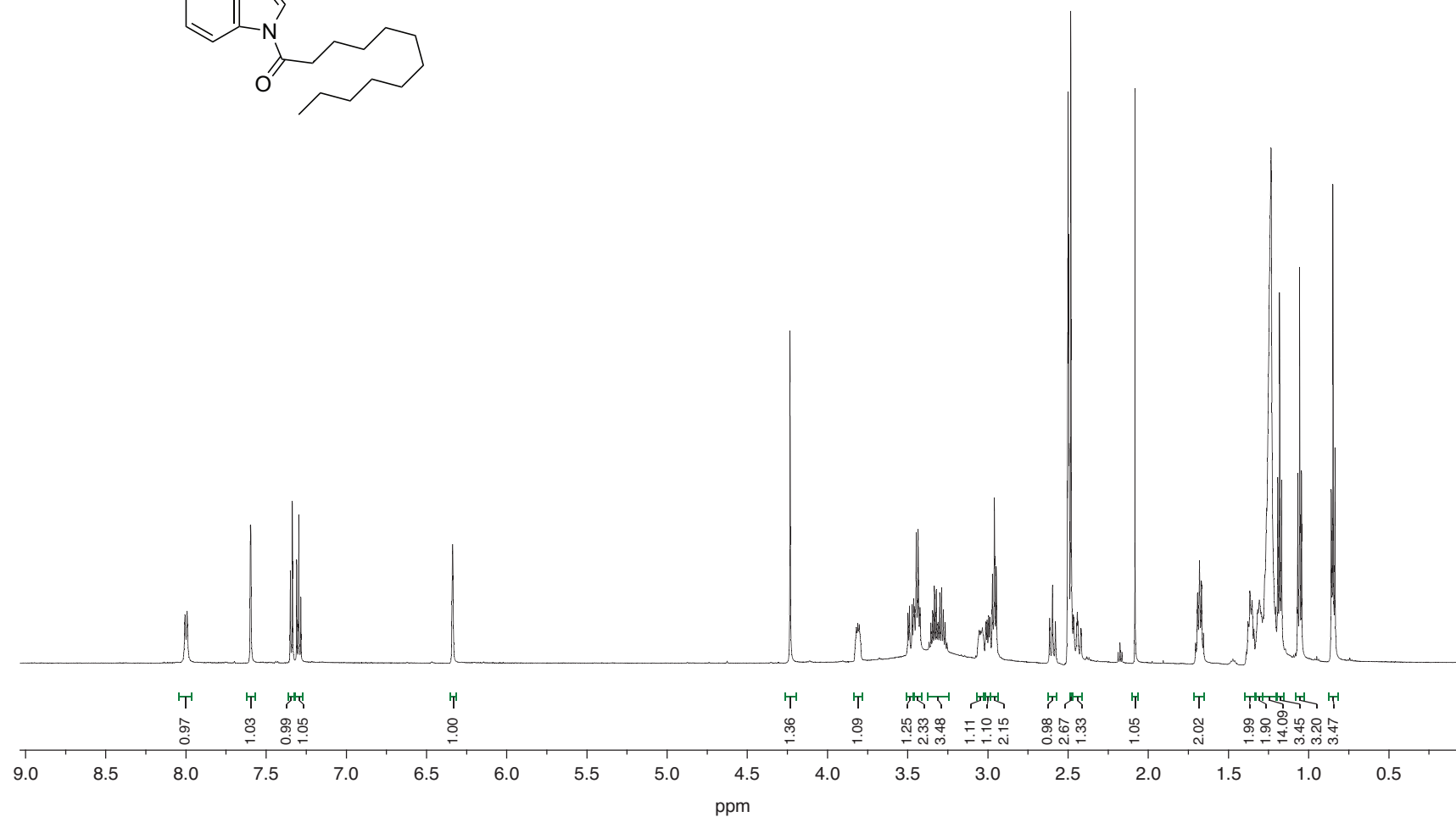
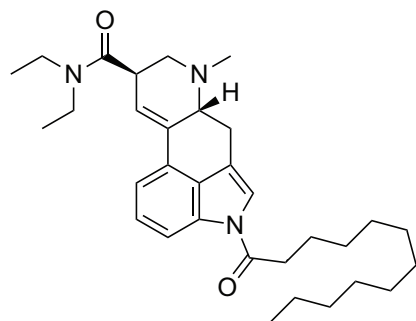
## Attenuated total reflection-infrared spectroscopy (ATR-IR)

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  $2\text{ cm}^{-1}$ . 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).



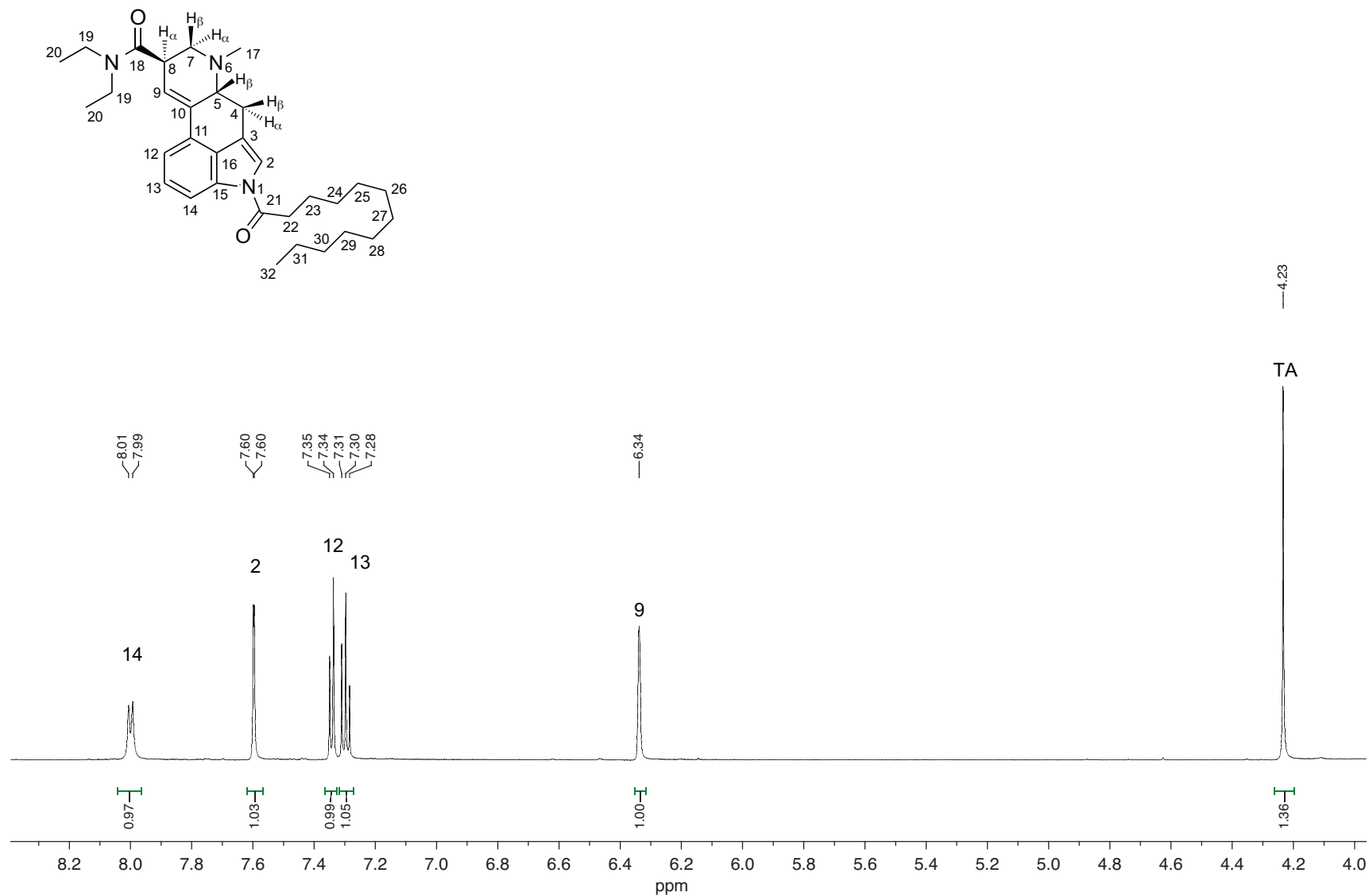
# Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)  
<sup>1</sup>H NMR (600 MHz)  
 DMSO-*d*<sub>6</sub>



# Supporting Information – Drug Testing and Analysis

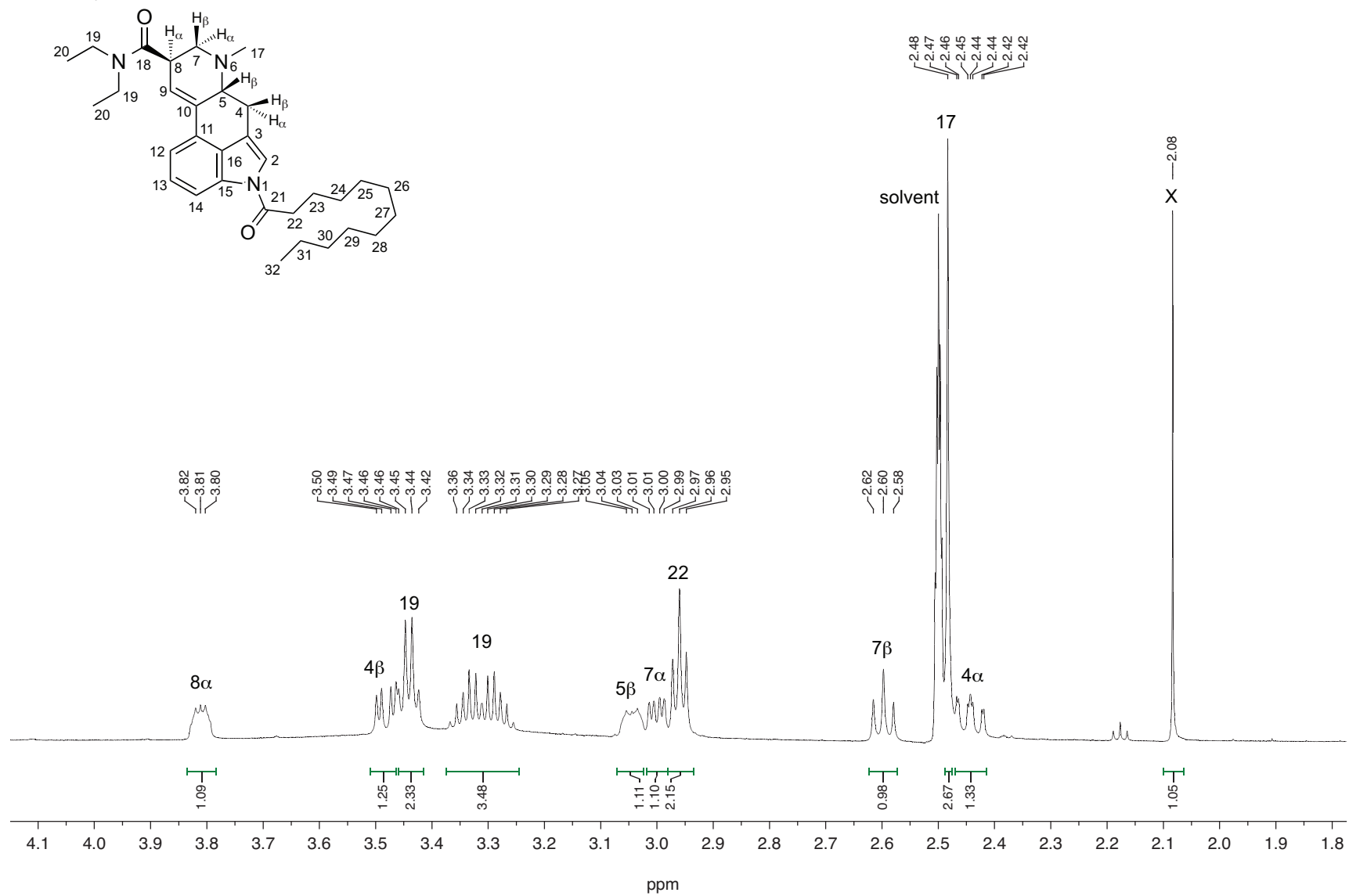
1DD-LSD tartrate (3:2)  
<sup>1</sup>H NMR (600 MHz)  
 DMSO-*d*<sub>6</sub>



TA: tartaric acid

# Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)  
<sup>1</sup>H NMR (600 MHz)  
DMSO-*d*<sub>6</sub>



# Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)

<sup>1</sup>H NMR (600 MHz)

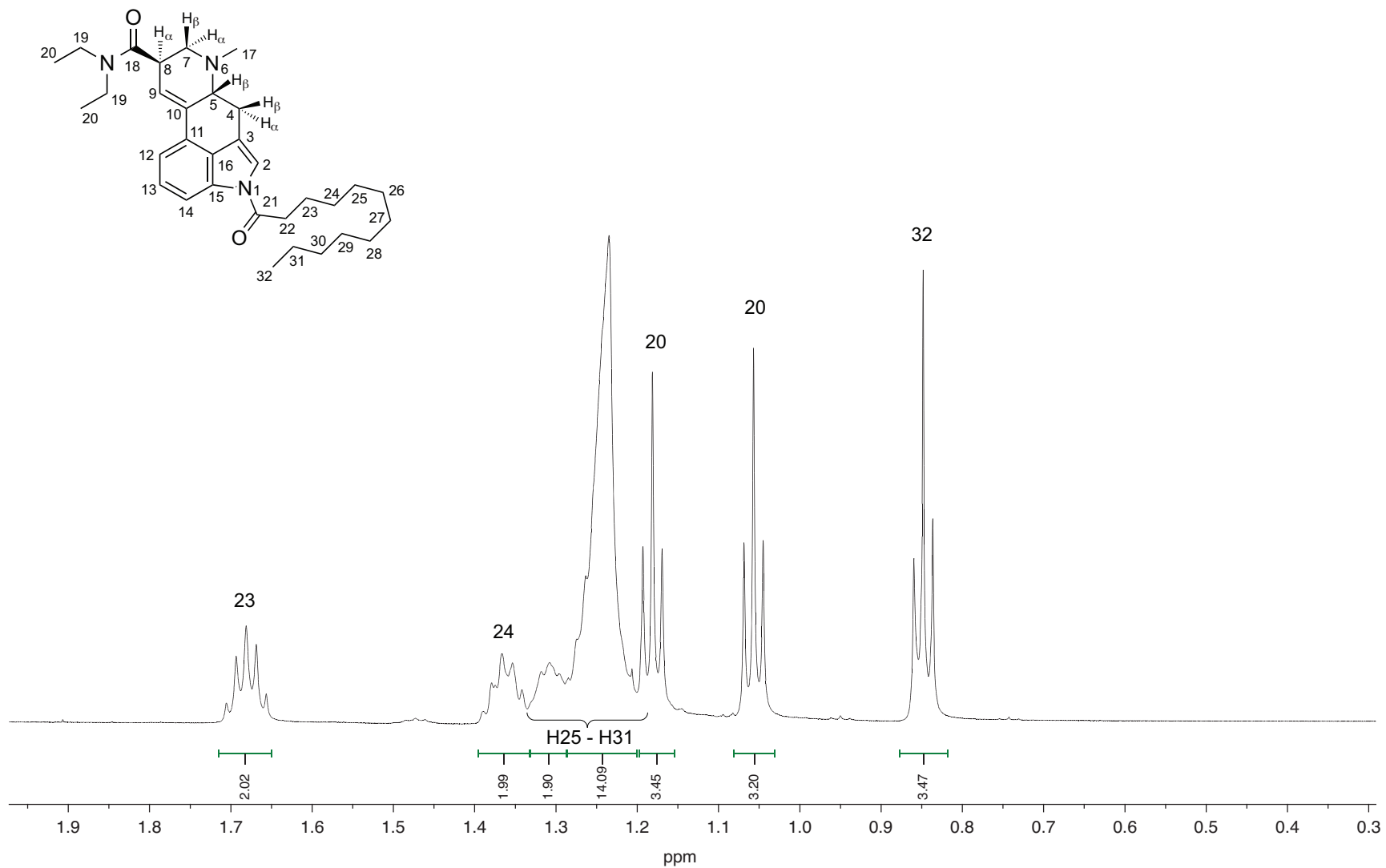
DMSO-*d*<sub>6</sub>

1.71  
1.69  
1.68  
1.67  
1.66

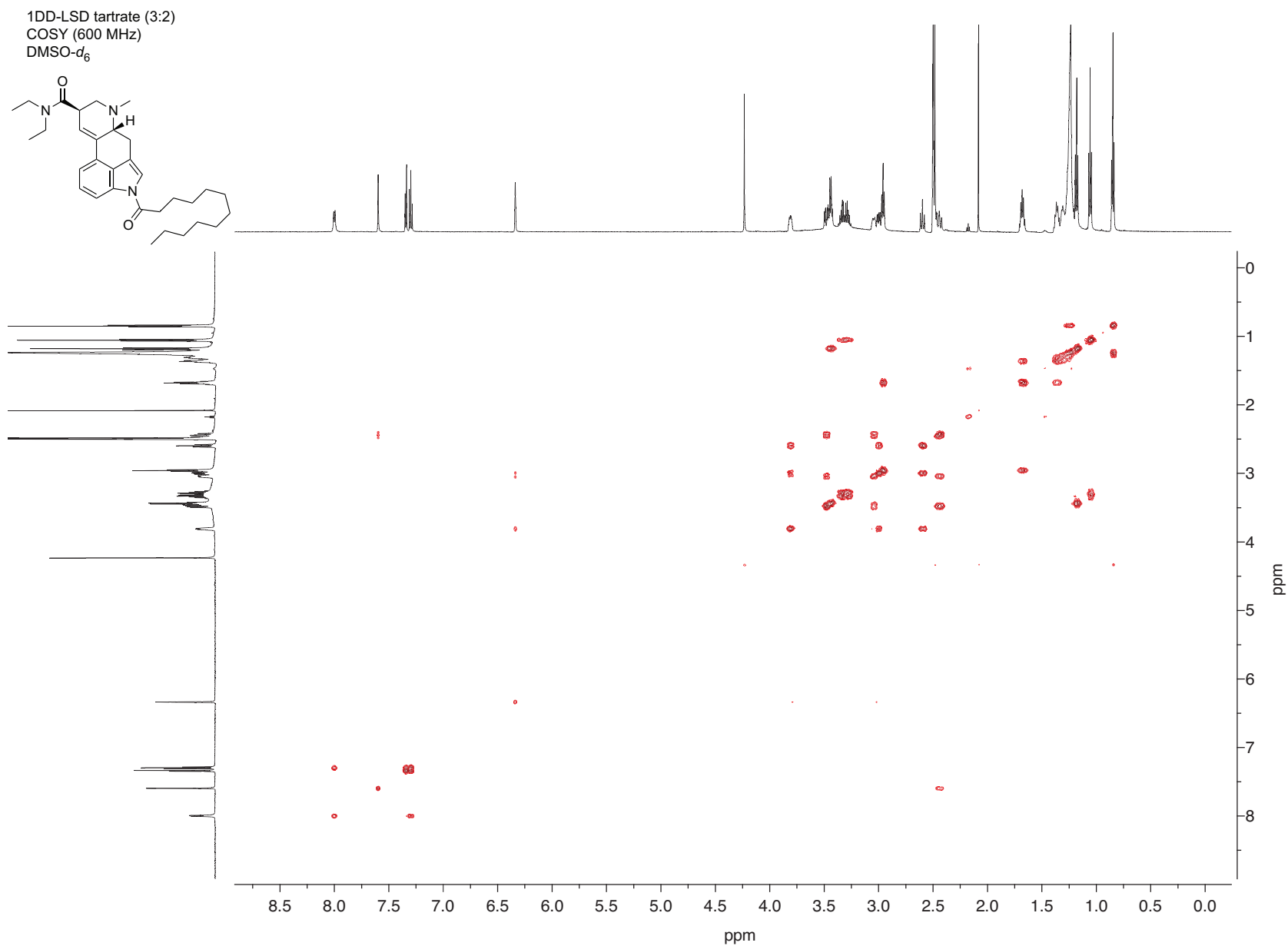
1.39  
1.38  
1.37  
1.37  
1.35  
1.34  
1.32  
1.31  
1.30  
1.28  
1.26  
1.23  
1.19  
1.18  
1.17

1.07  
1.06  
1.05

0.86  
0.85  
0.84

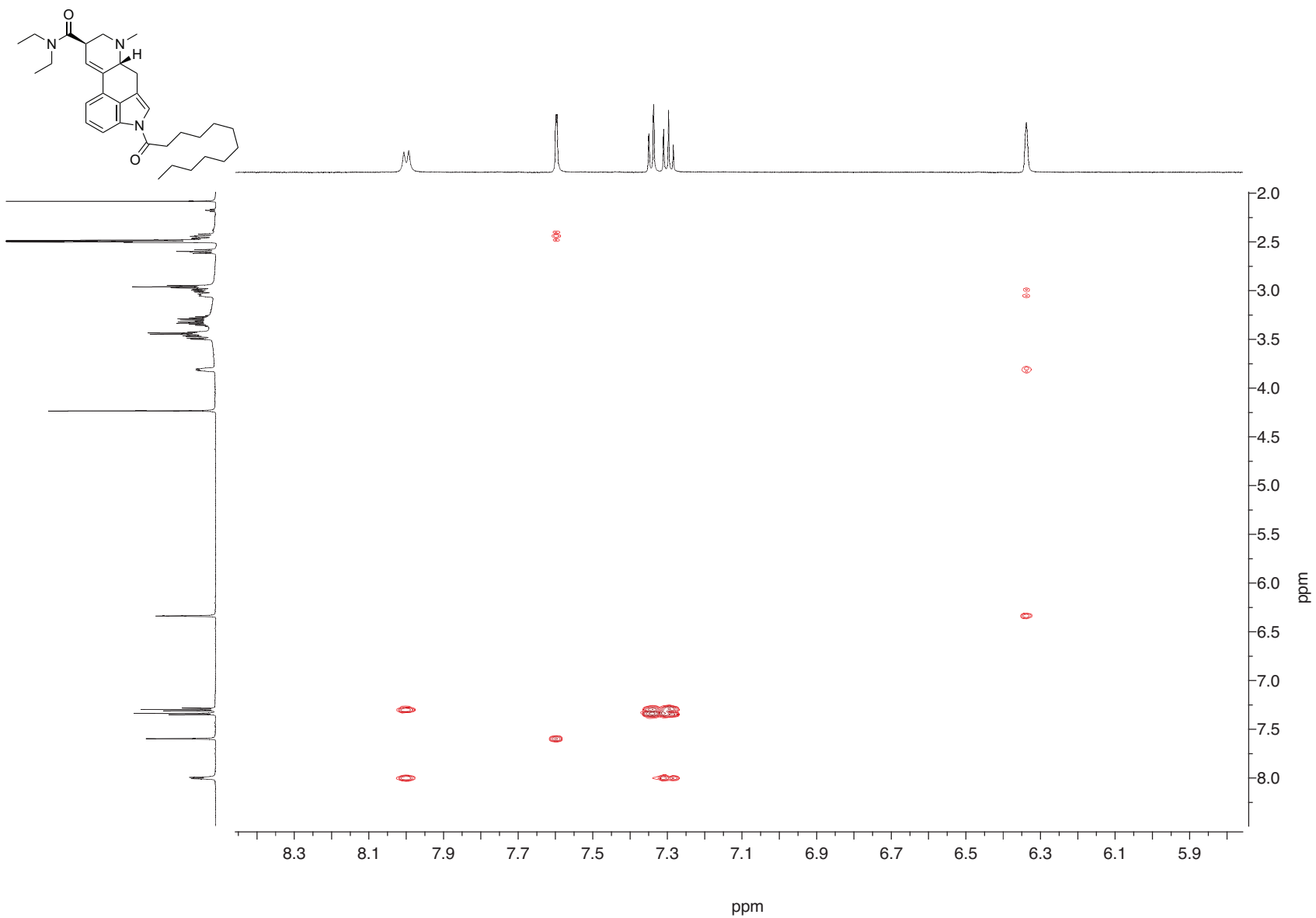


# Supporting Information – Drug Testing and Analysis



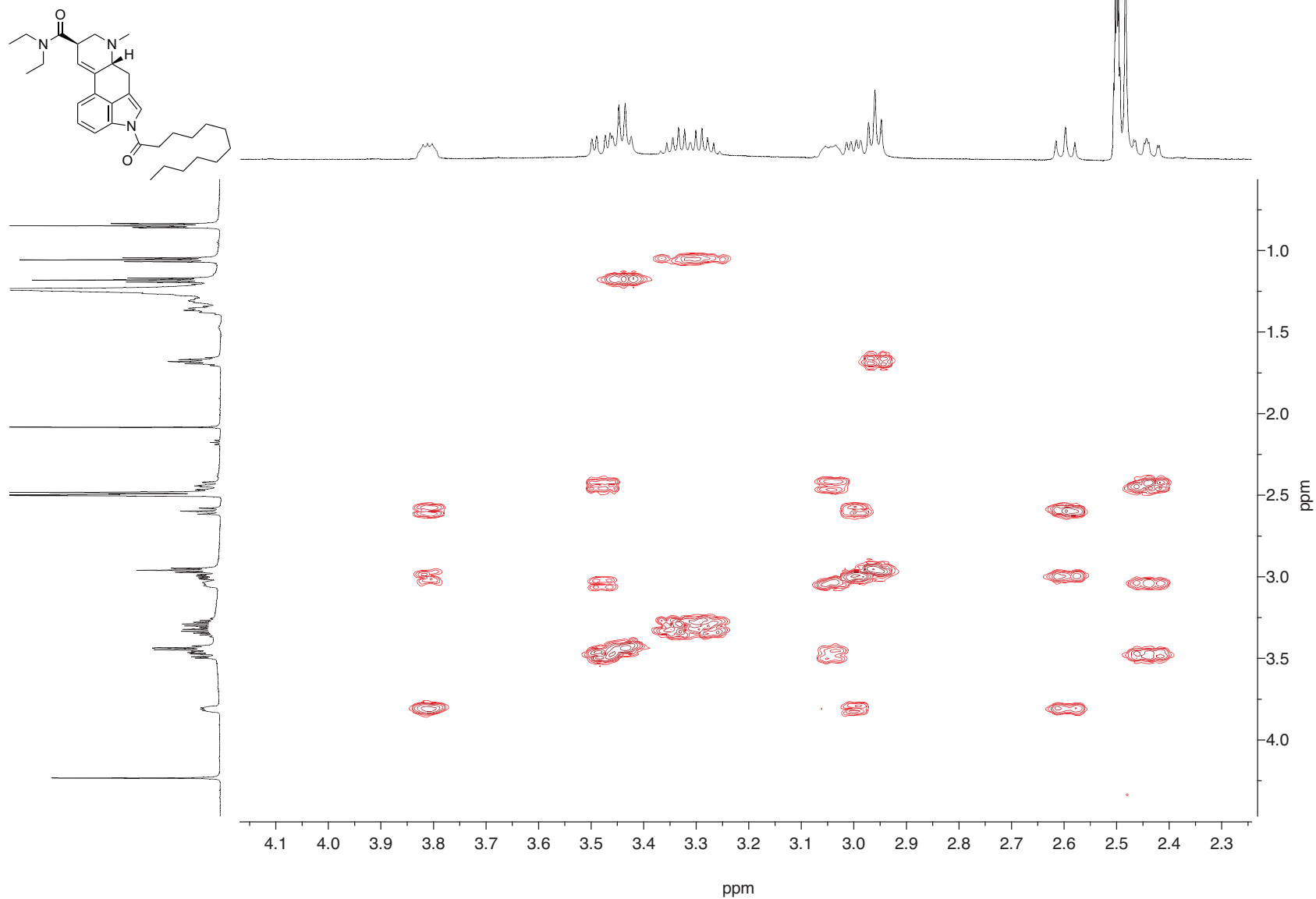
# Supporting Information – Drug Testing and Analysis

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



# Supporting Information – Drug Testing and Analysis

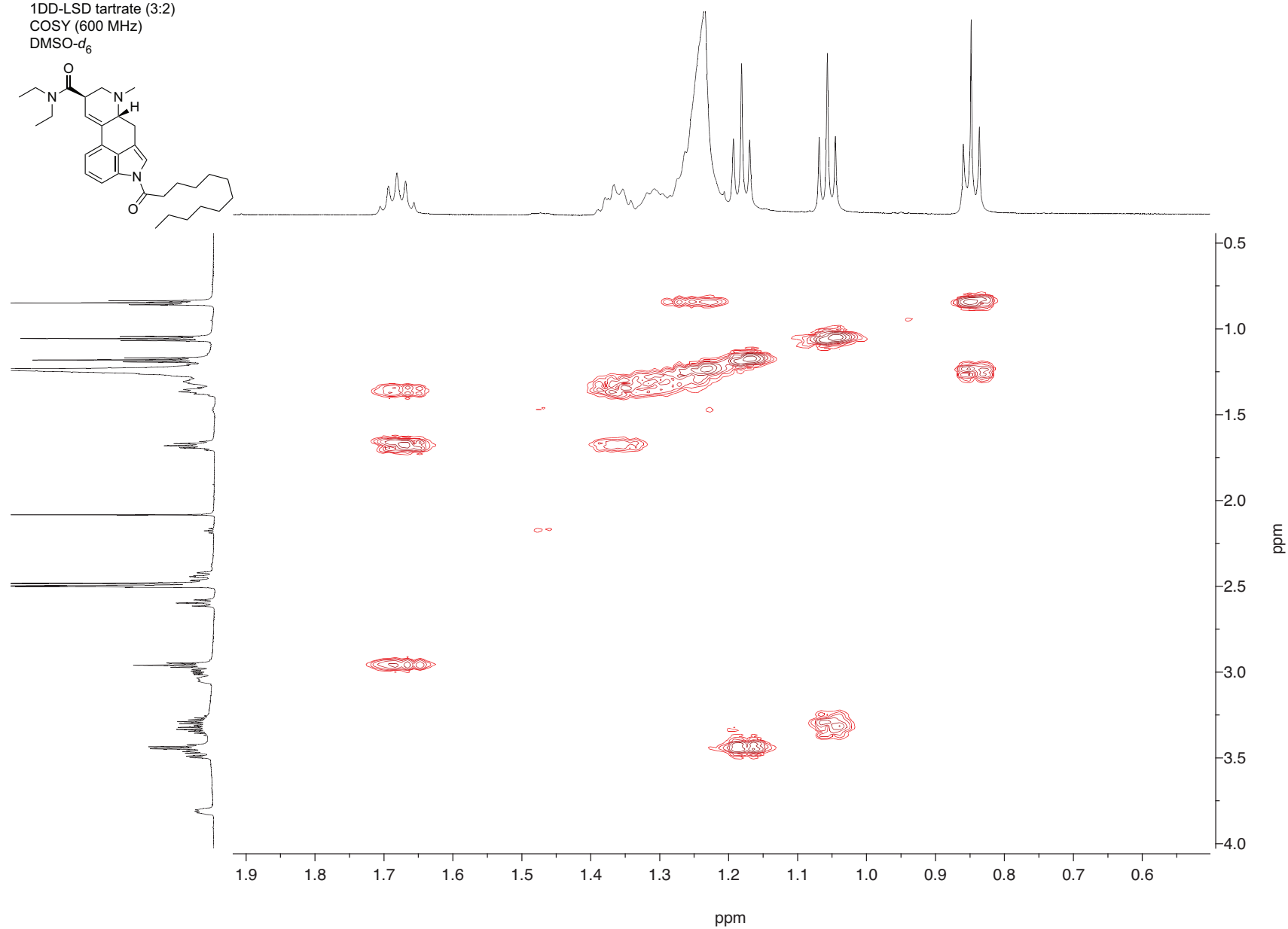
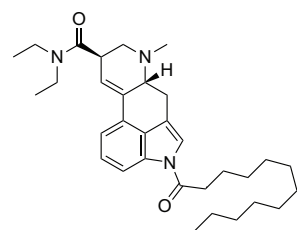
1DD-LSD tartrate (3:2)  
COSY (600 MHz)  
DMSO- $d_6$





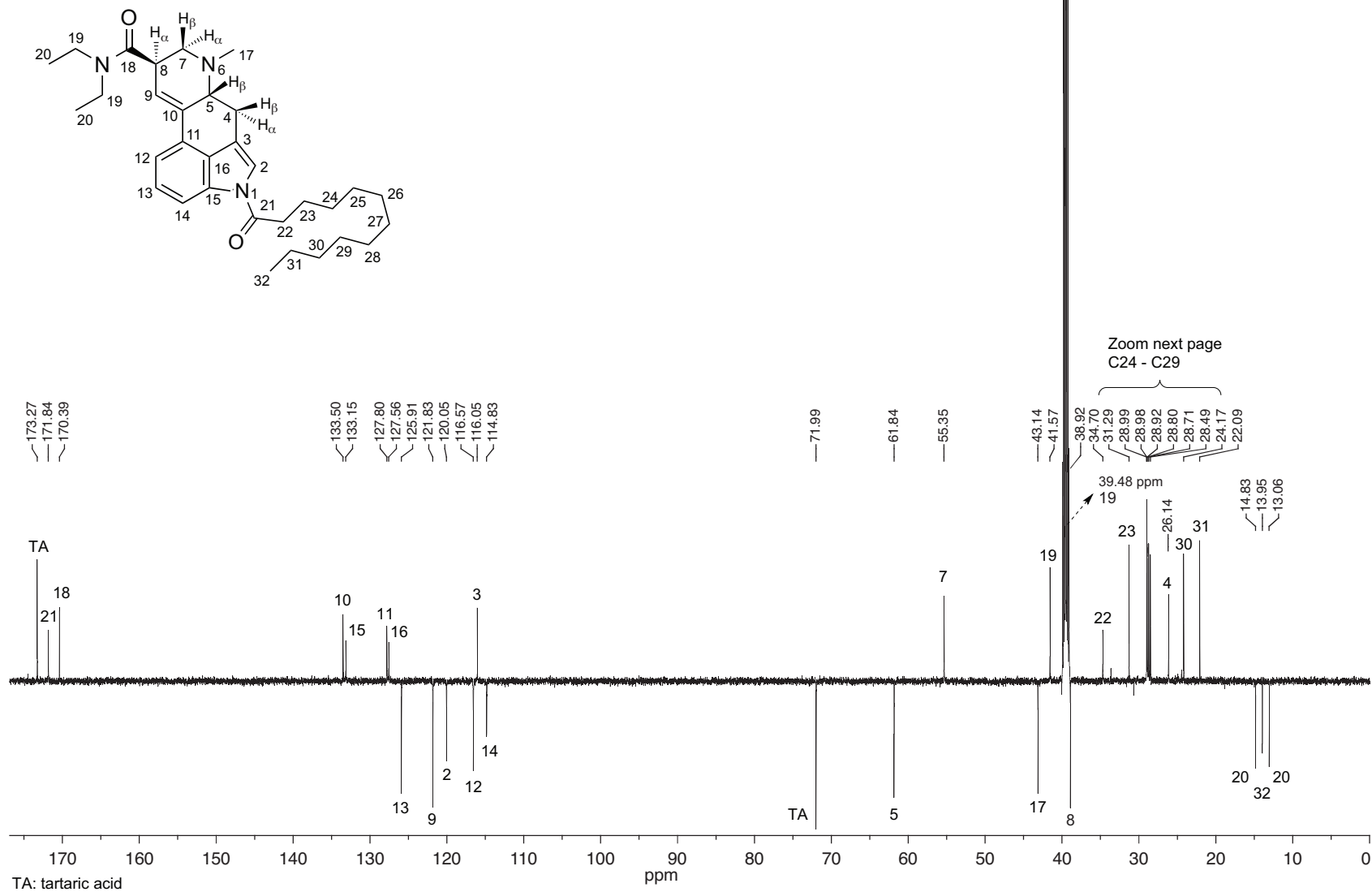
# Supporting Information – Drug Testing and Analysis

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



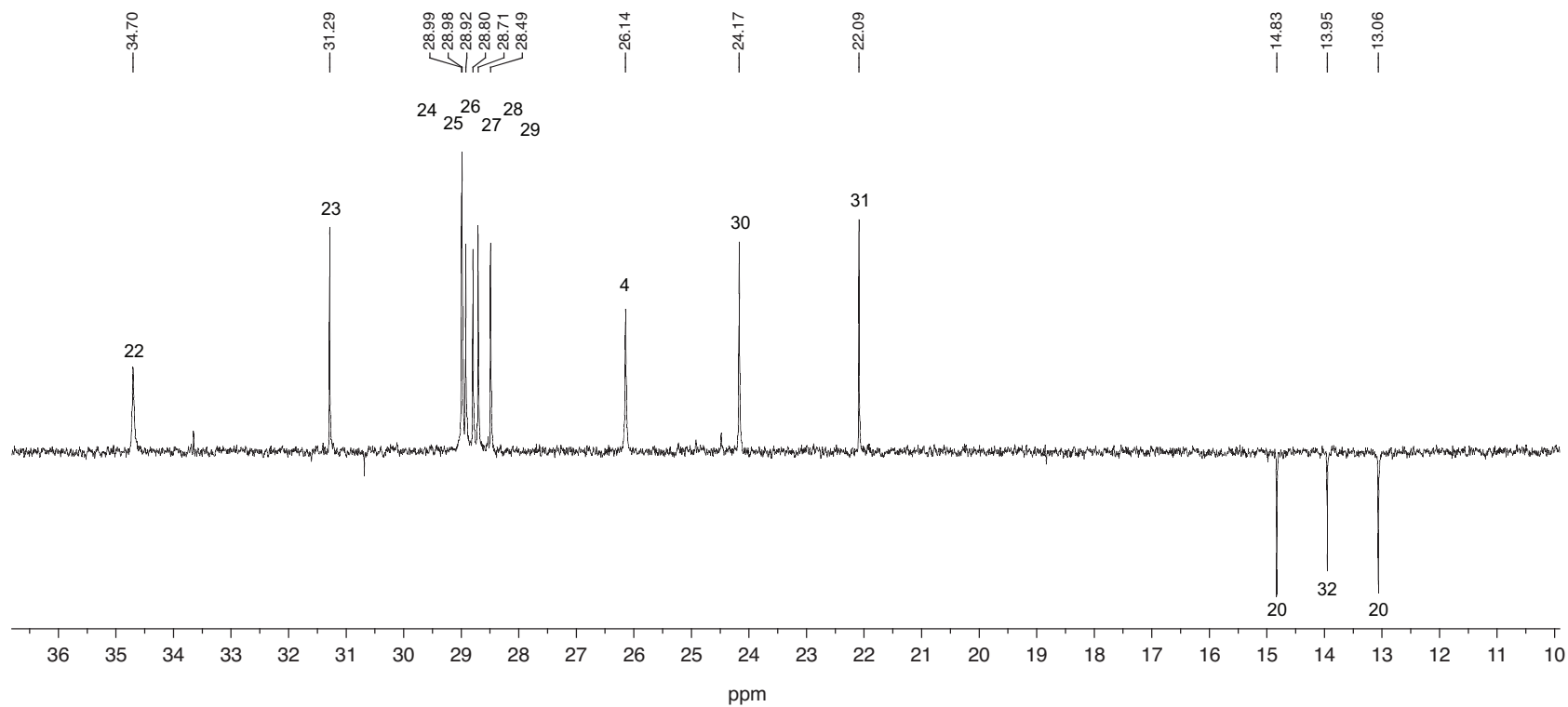
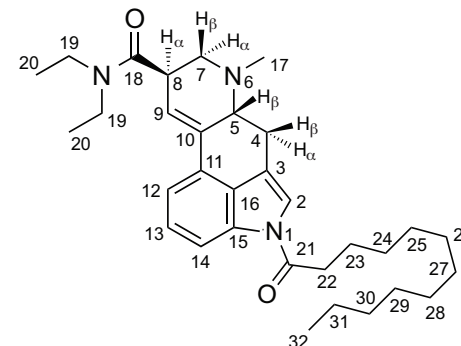
# Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)  
DEPTQ (150 MHz)  
DMSO- $d_6$



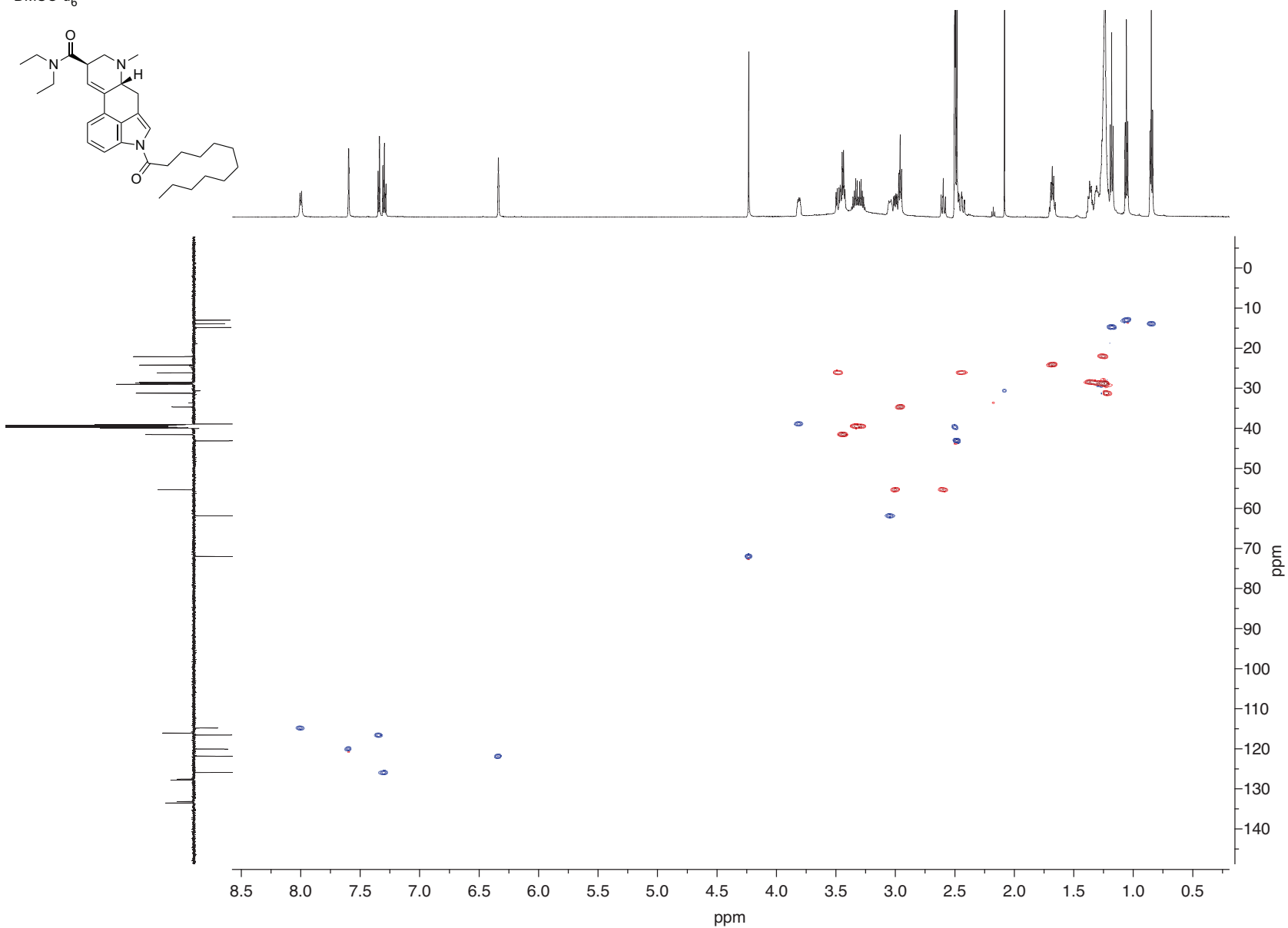
# Supporting Information – Drug Testing and Analysis

1DD-LSD tartrate (3:2)  
DEPTQ (150 MHz)  
DMSO- $d_6$



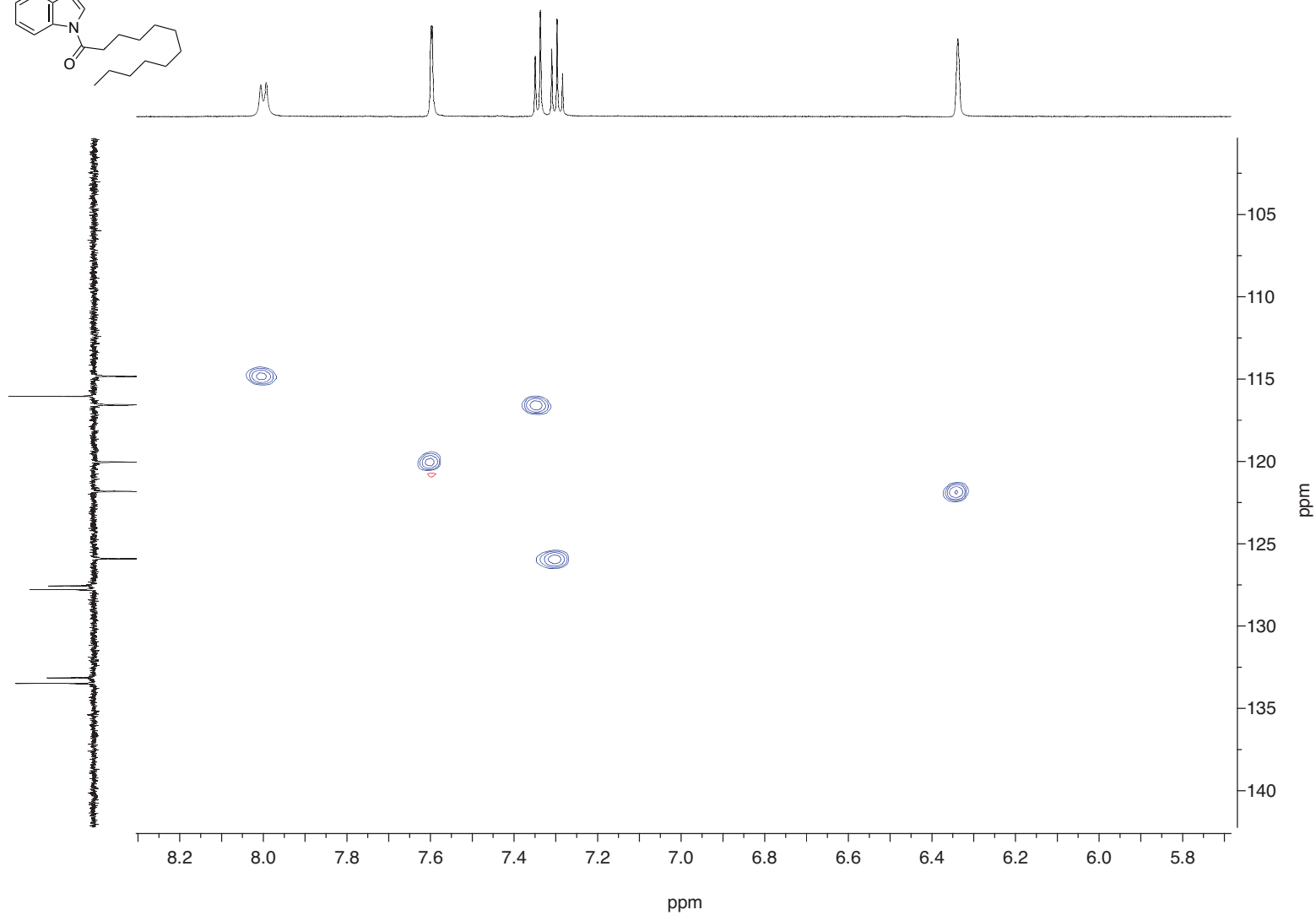
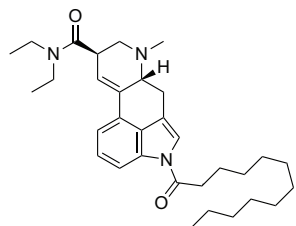
# Supporting Information – Drug Testing and Analysis

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



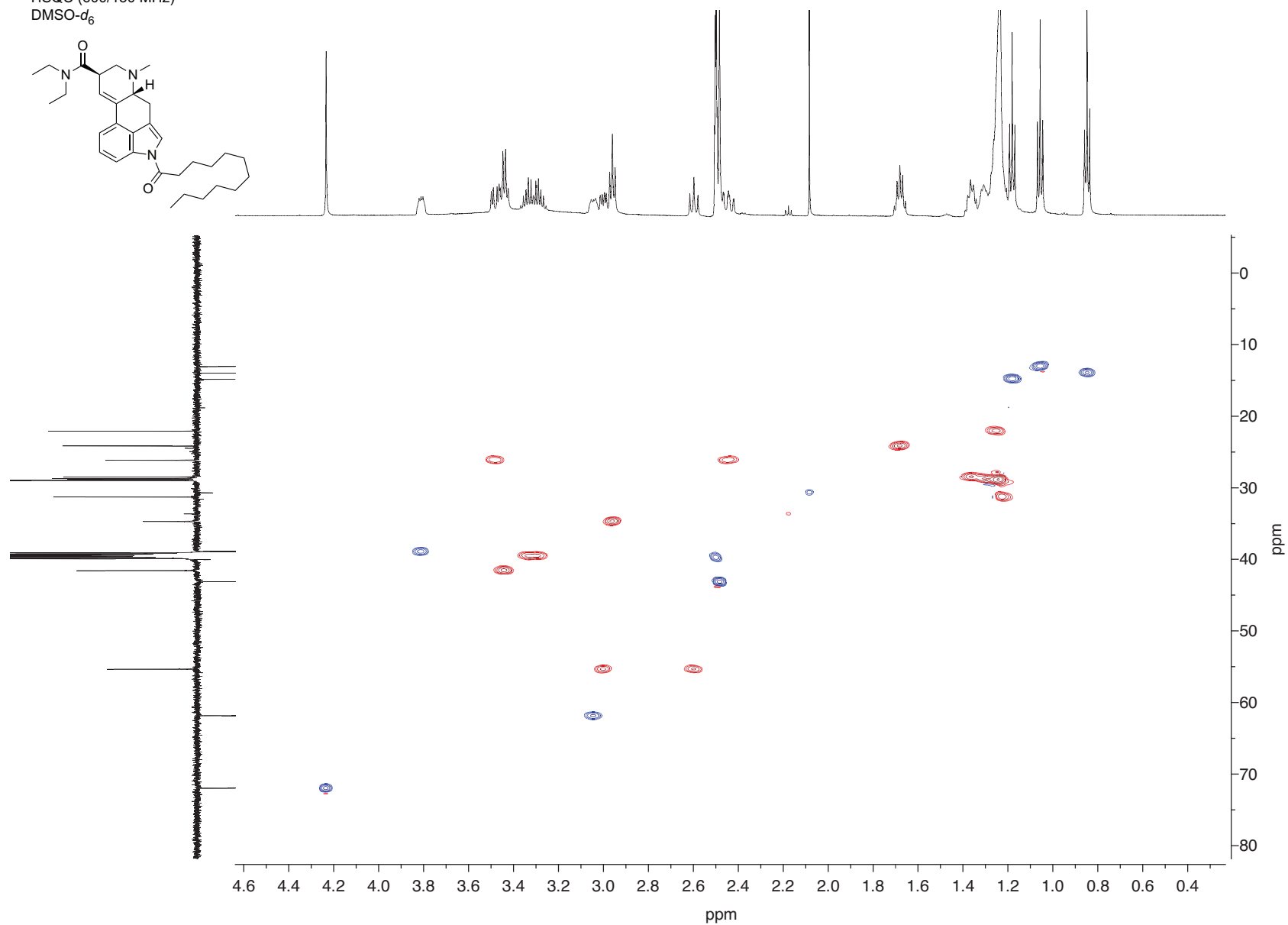
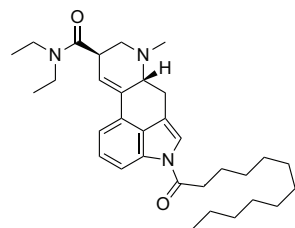
## Supporting Information – Drug Testing and Analysis

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



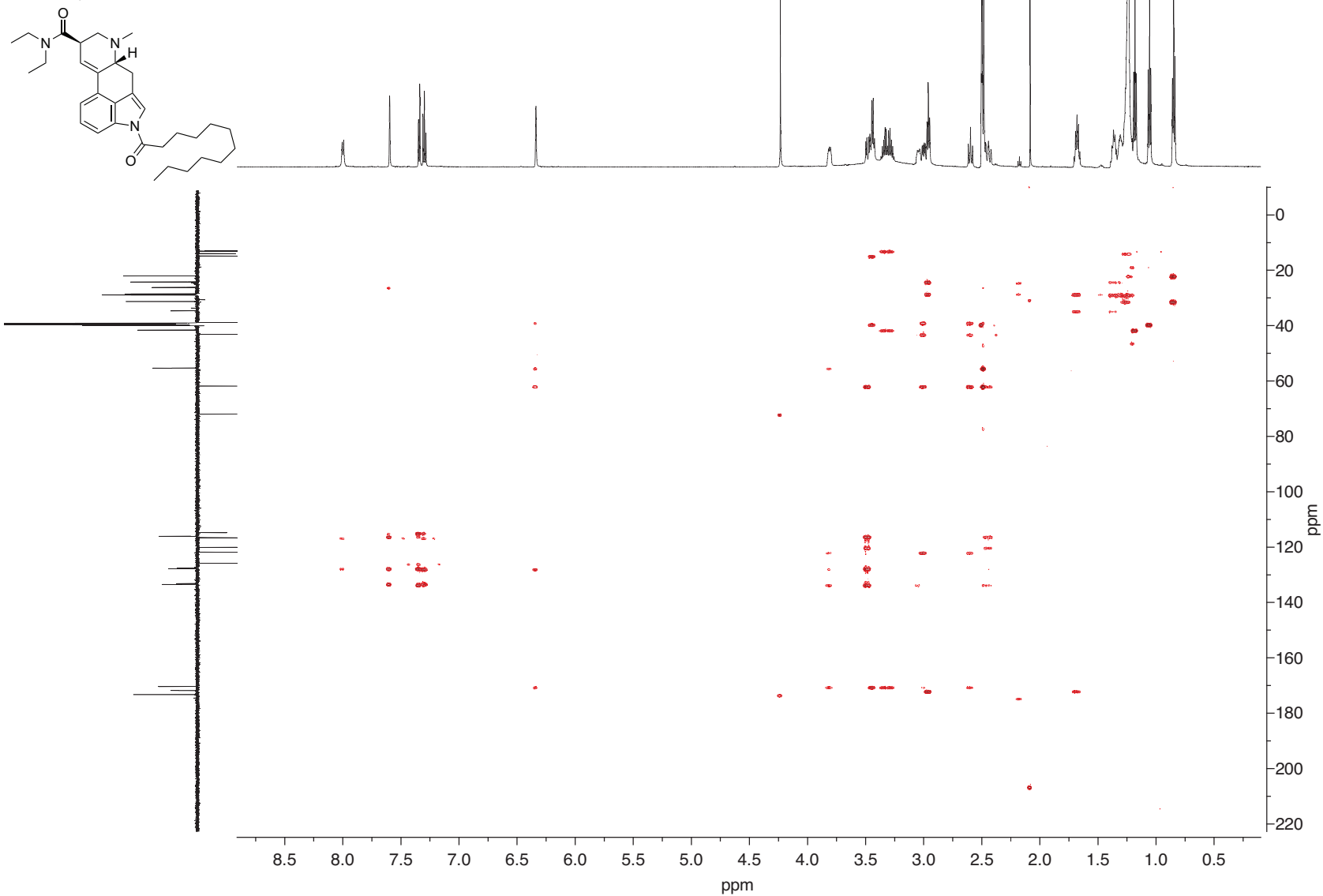
# Supporting Information – Drug Testing and Analysis

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



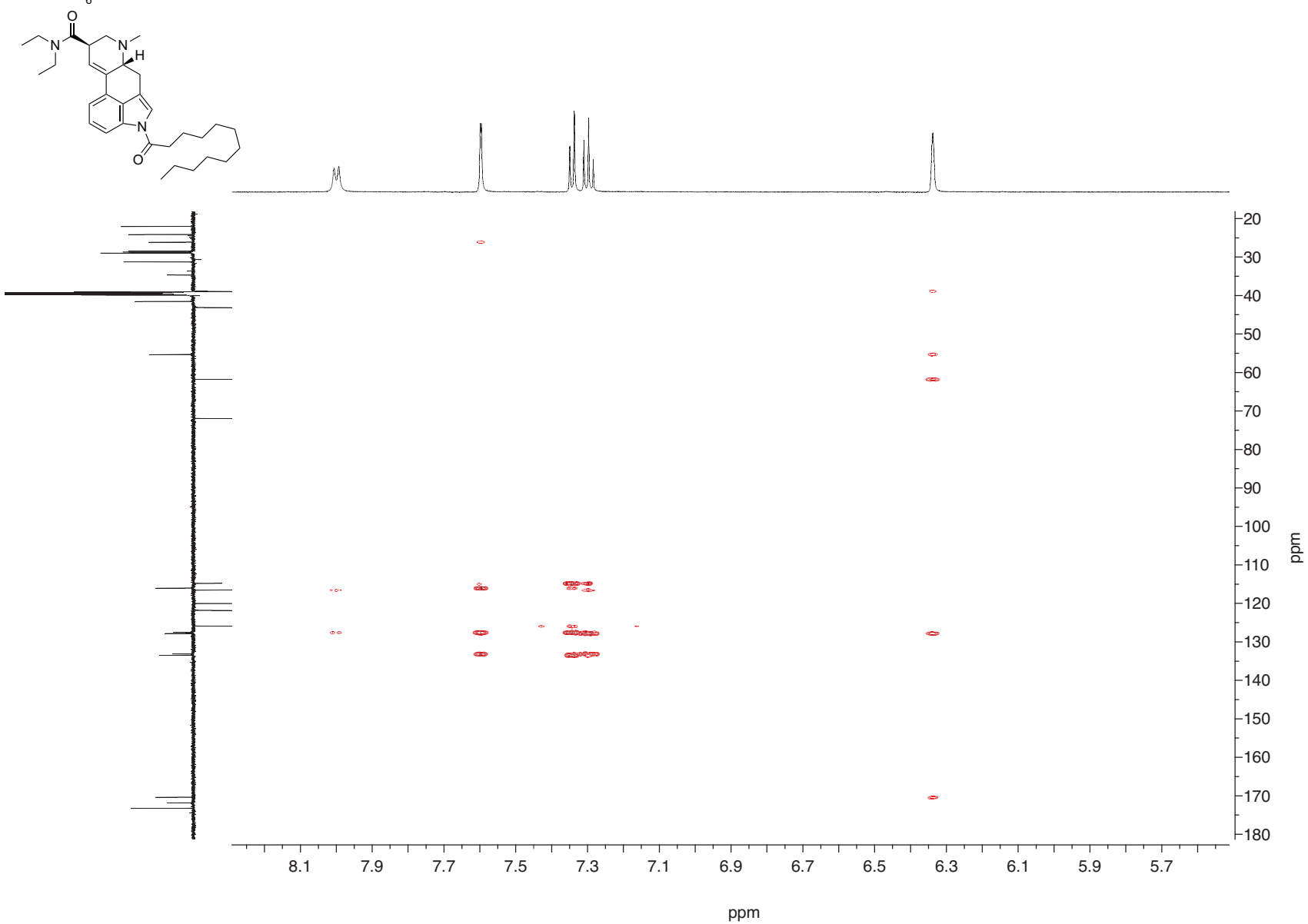
# Supporting Information – Drug Testing and Analysis

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



# Supporting Information – Drug Testing and Analysis

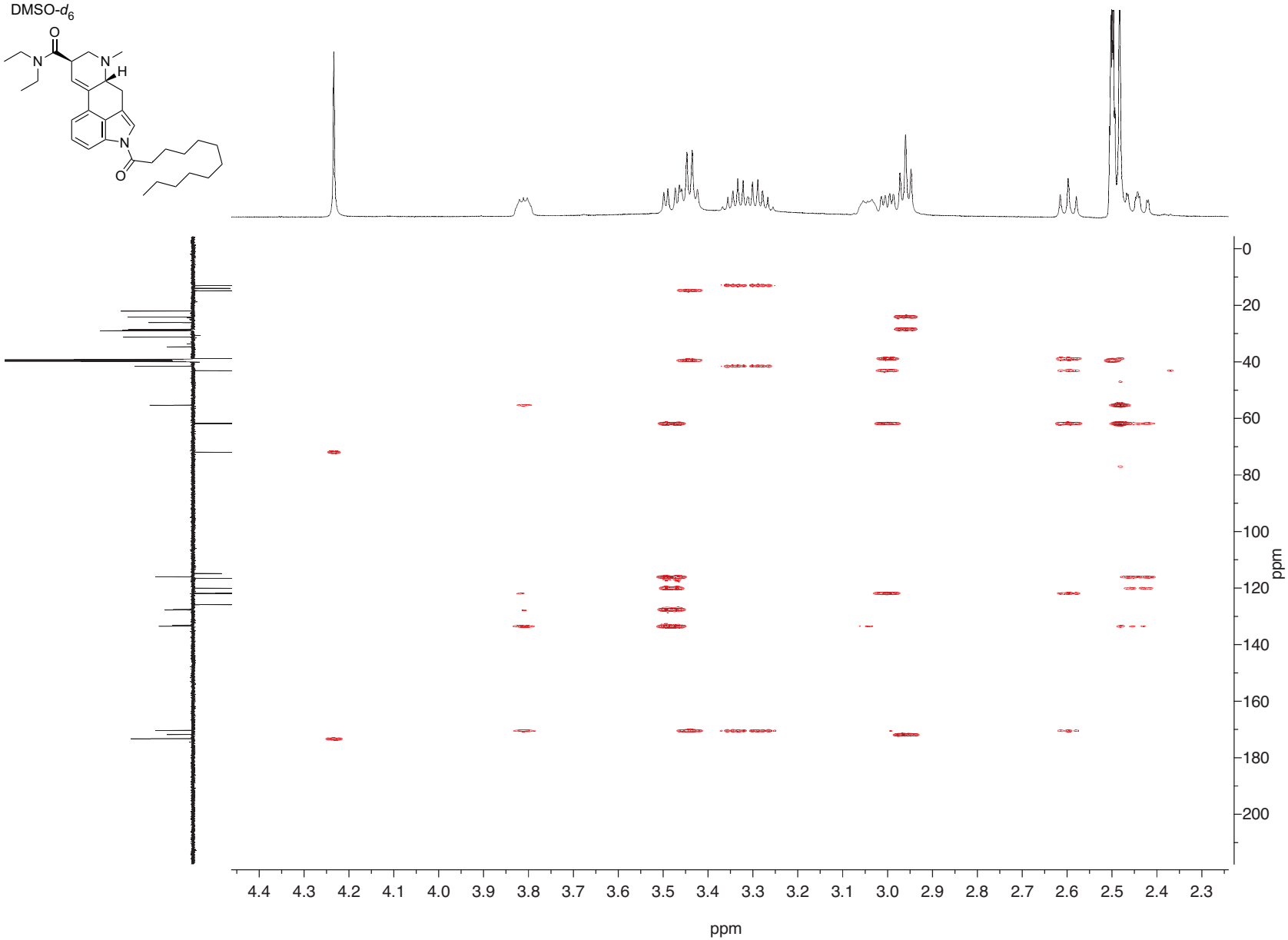
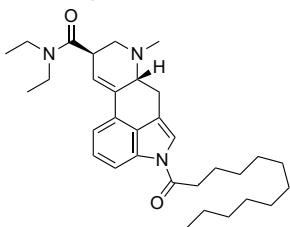
1DD-LSD tartrate (3:2)  
HMBC (600/150 MHz)  
DMSO- $d_6$





# 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- $d_6$

