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Return of the lysergamides. Part VII: Analytical and behavioural characterization of 1-valeroyl-d-lysergic acid diethylamide (1V-LSD)

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Return of the lysergamides. Part VII: Analytical and behavioural characterization of 1-valeroyl-d-lysergic acid diethylamide (1V-LSD)

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³ State Bureau of Criminal Investigation Schleswig-Holstein, Section Narcotics/Toxicology, Mühlenweg 166, D-24116 Kiel, Germany

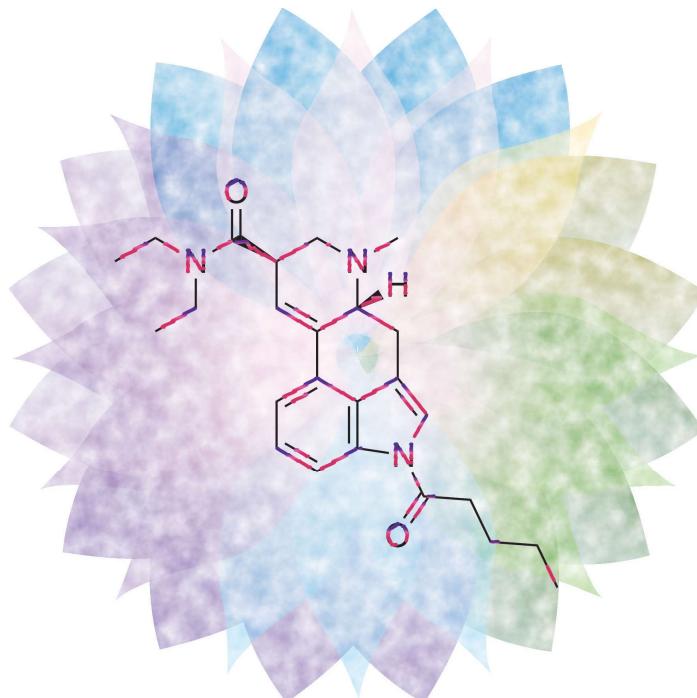
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⁵ Synex Synthetics BV, Karveelweg 20, 6222NH, Maastricht, The Netherlands

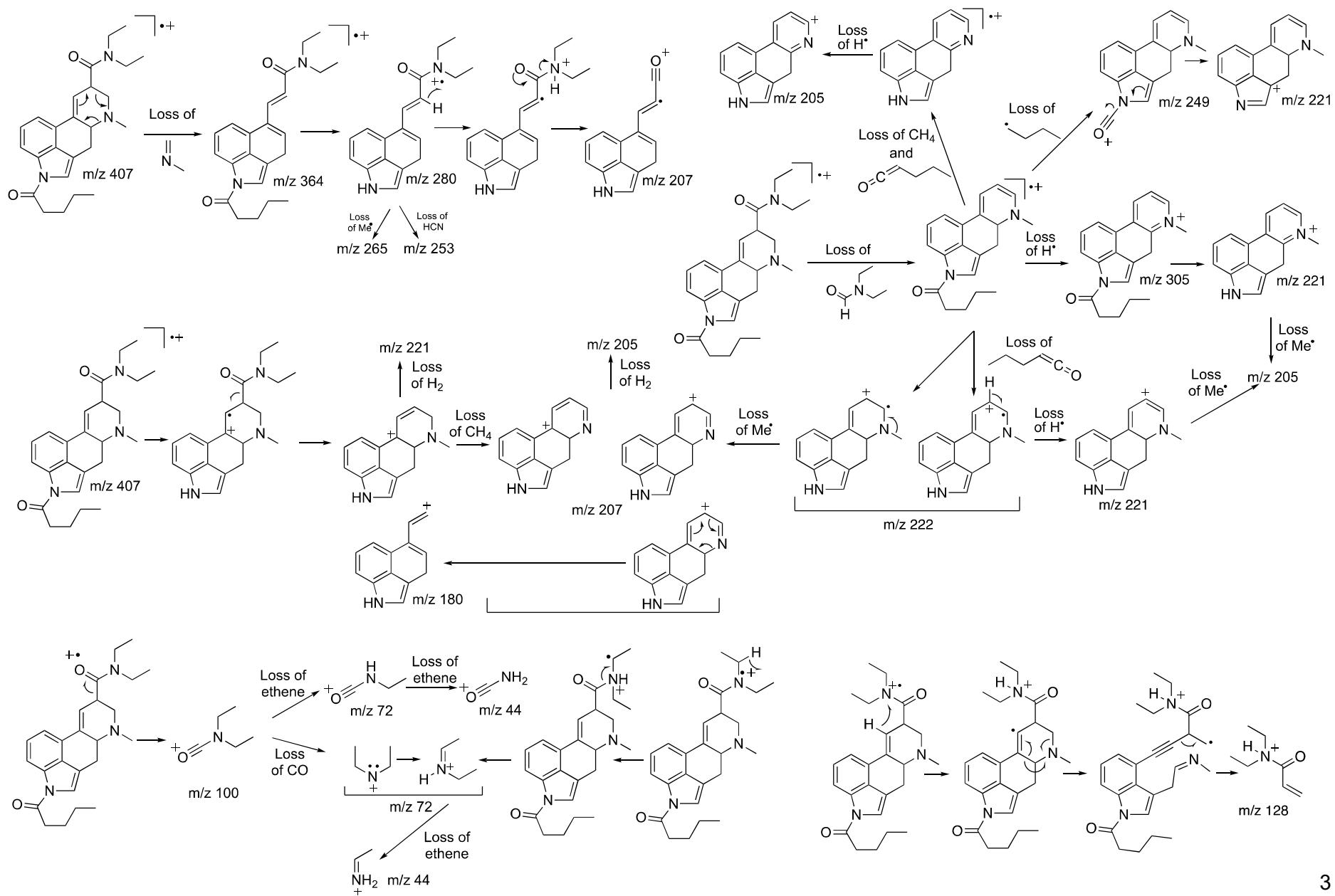
⁶ Department of Life Sciences, School of Science, Sligo Institute of Technology, Ash Lane, Sligo, F91YW50, Ireland

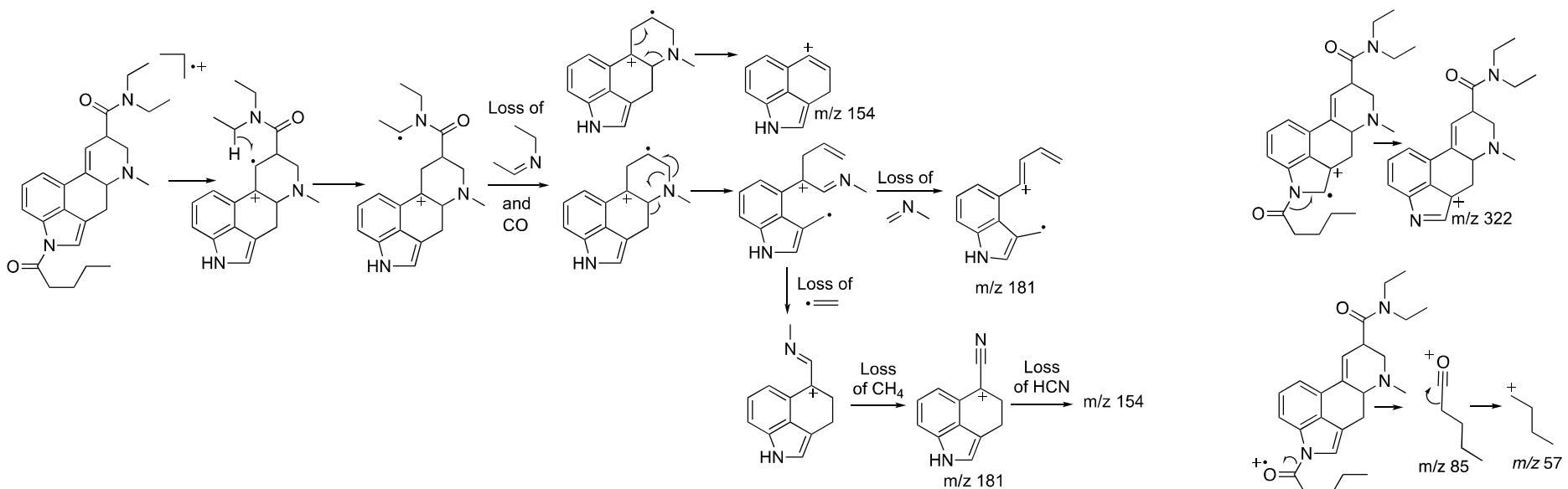
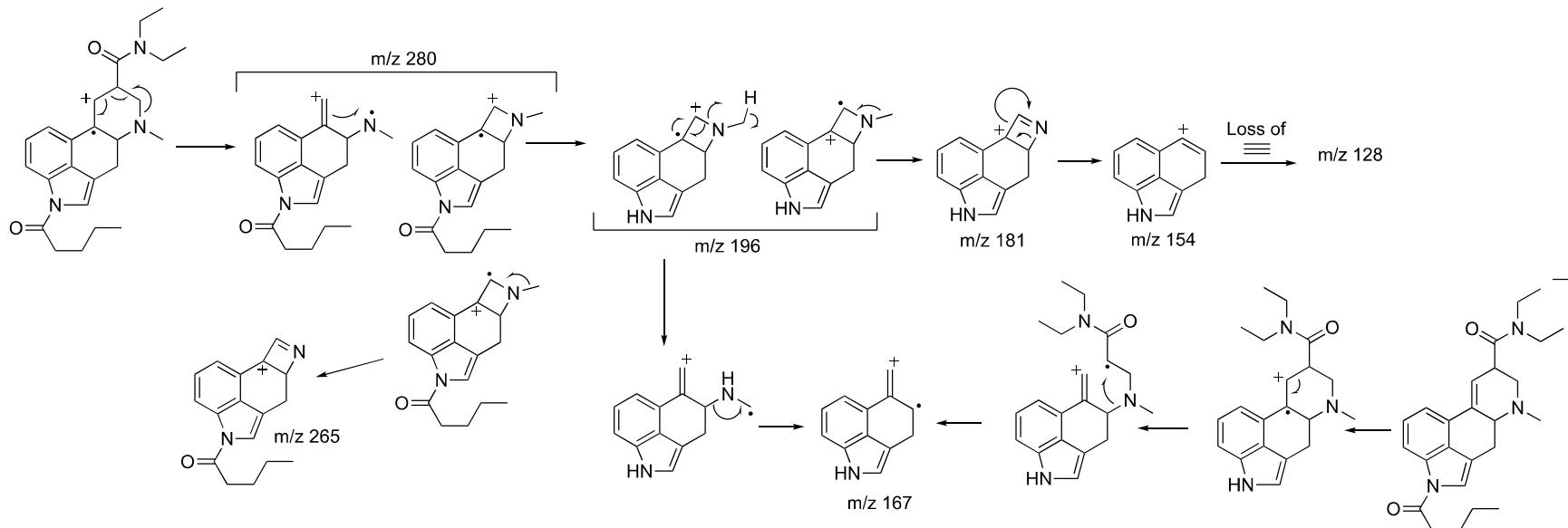
⁷ Research Service, VA San Diego Healthcare System, San Diego, CA 92161, USA

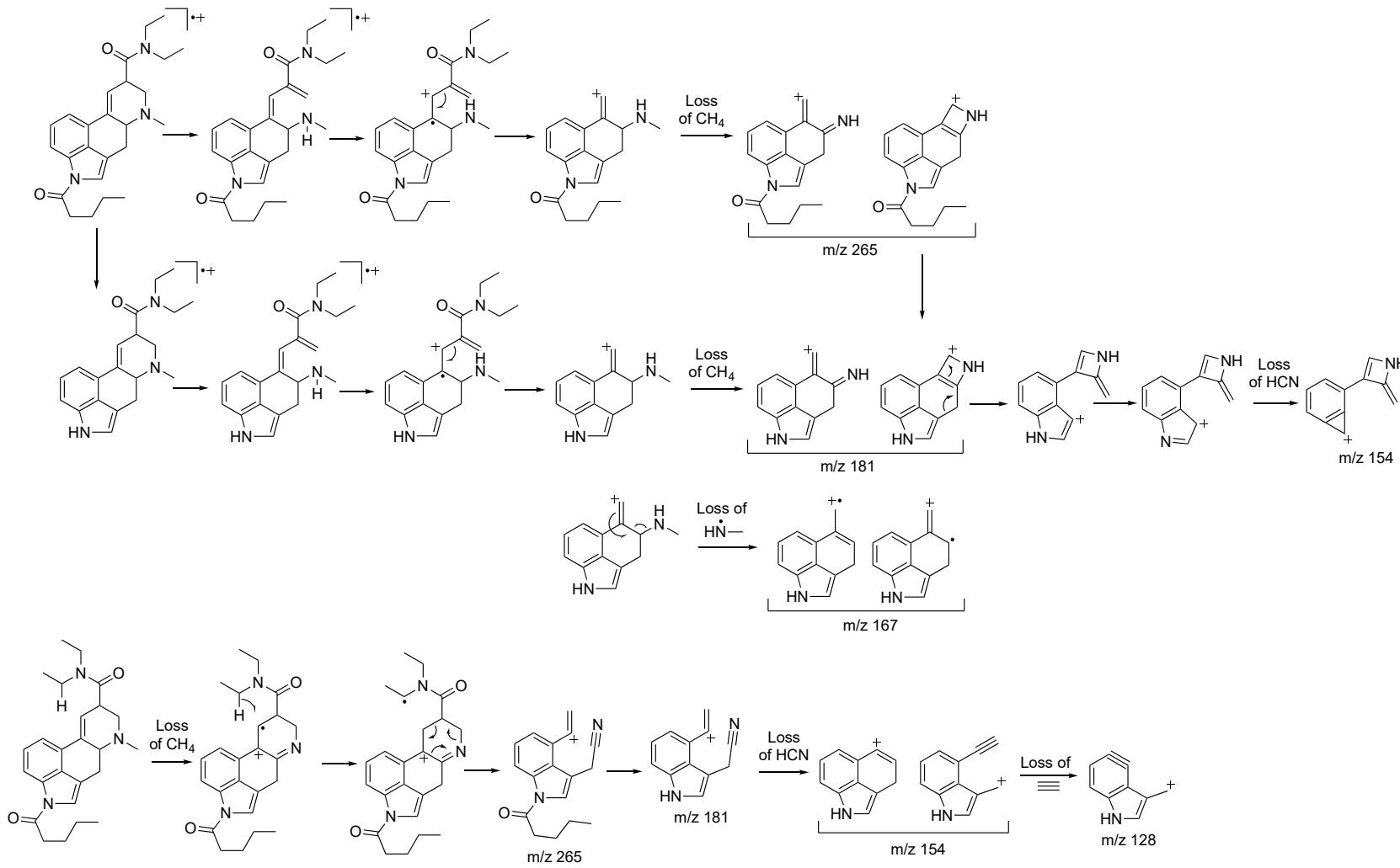
* Correspondence to: Simon D. Brandt, School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK. E-Mail: s.brandt@ljmu.ac.uk

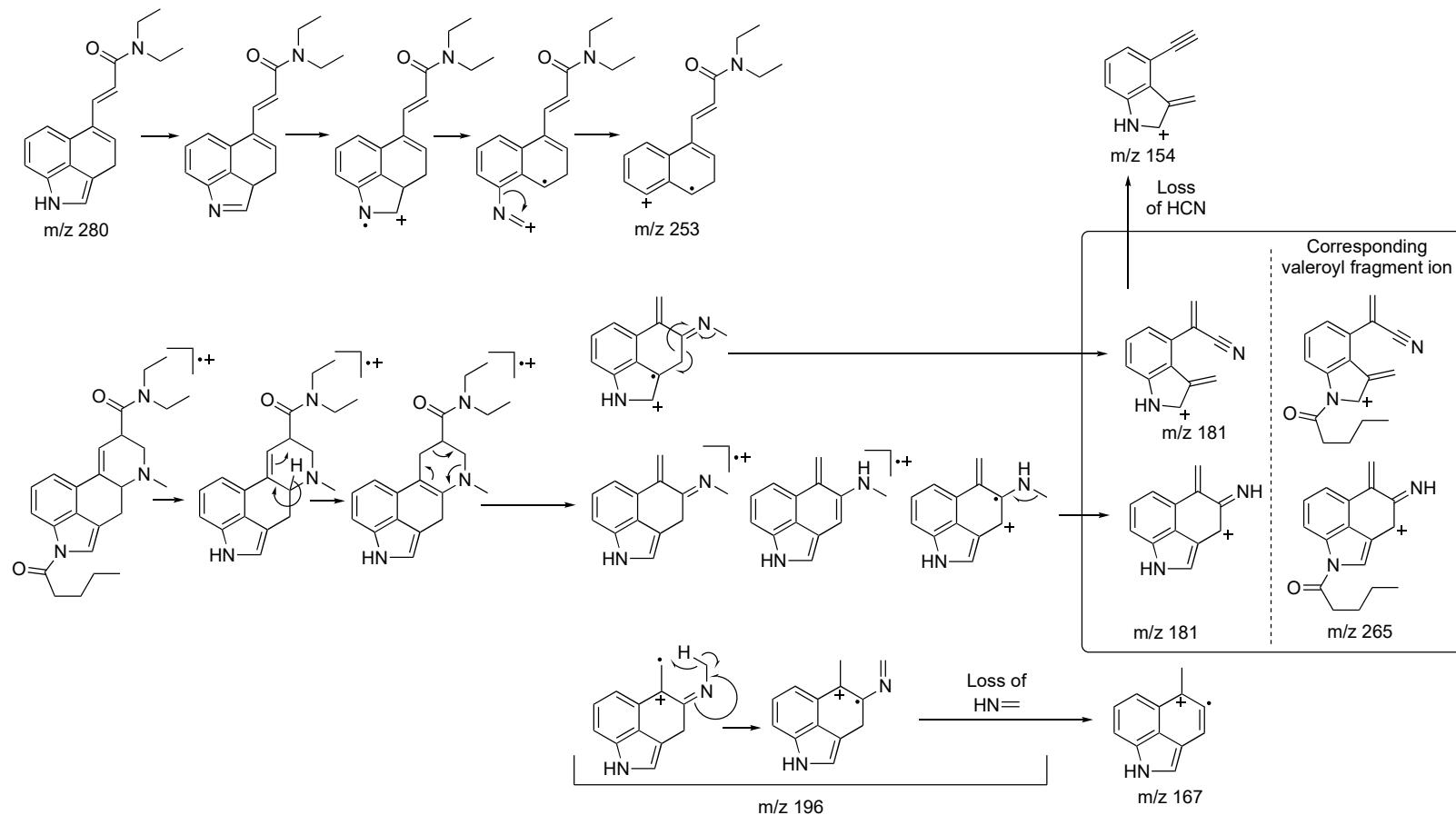


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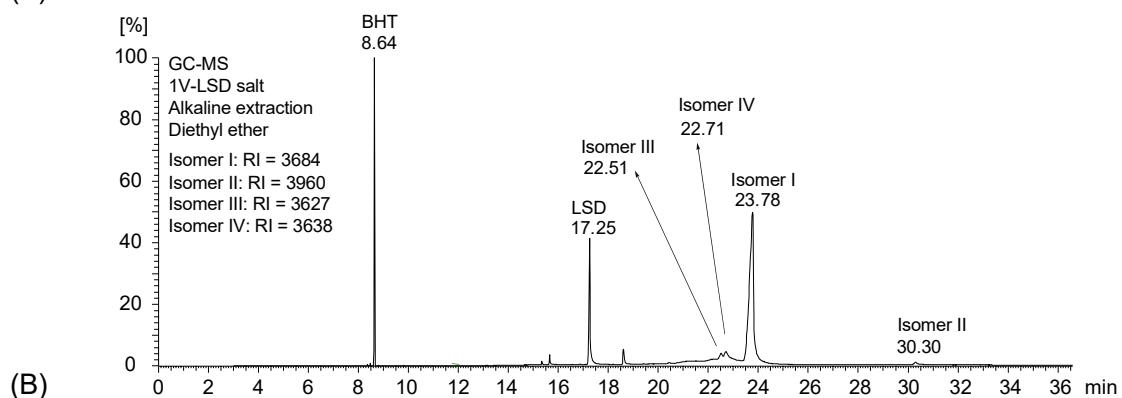




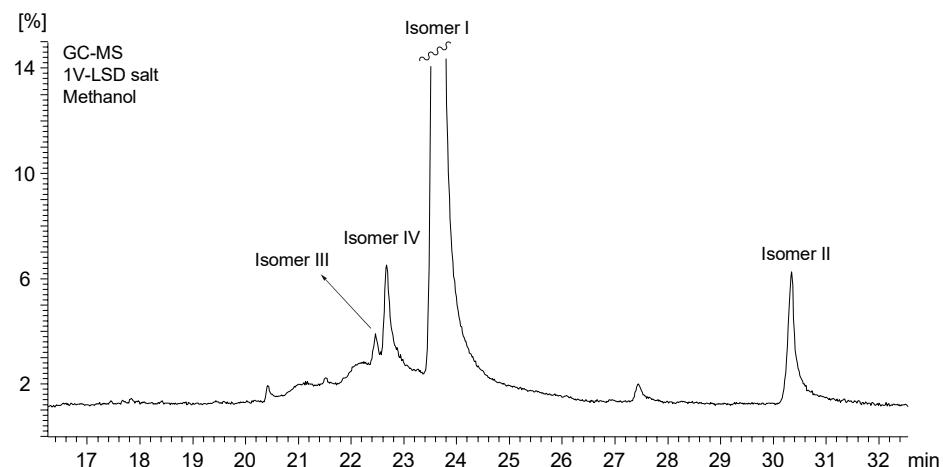


(A) GC-MS analysis of 1V-LSD hemitartrate (2:1) followed by alkaline extraction and GC-induced formation of three minor 1V-LSD isomers. The alkaline extraction procedure led to formation of LSD. BHT: butylated hydroxytoluene additive in diethyl ether. (B) GC-MS analysis of 1V-LSD salt dissolved in methanol. (C) LC-ESI-ion trap-MS analysis of 1V-LSD salt in methanol (extracted ion chromatogram) without detection of other isomers.

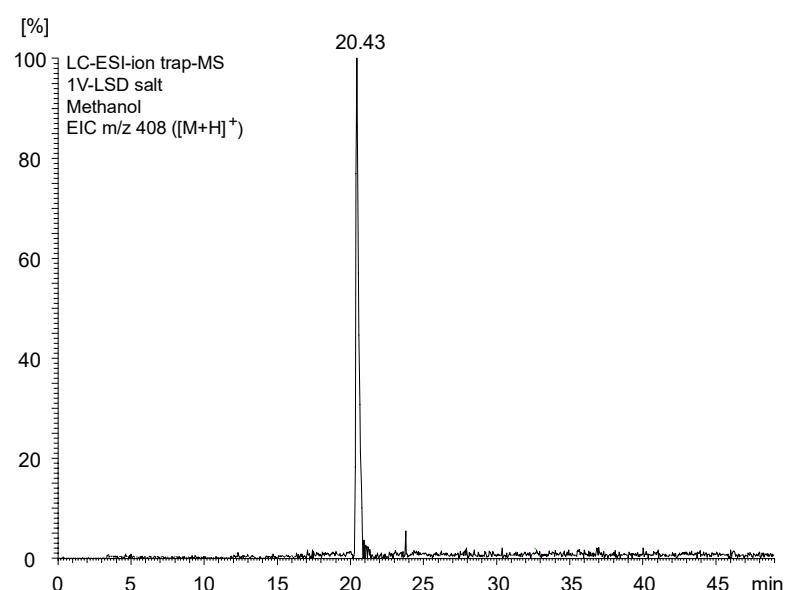
(A)



(B)

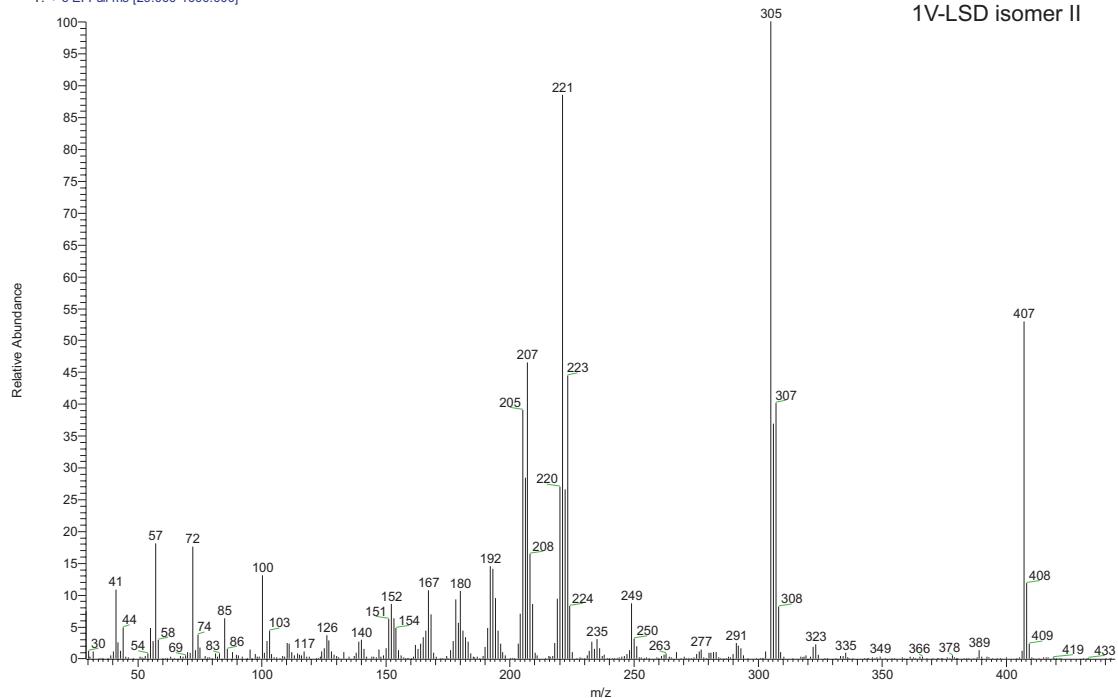


(C)

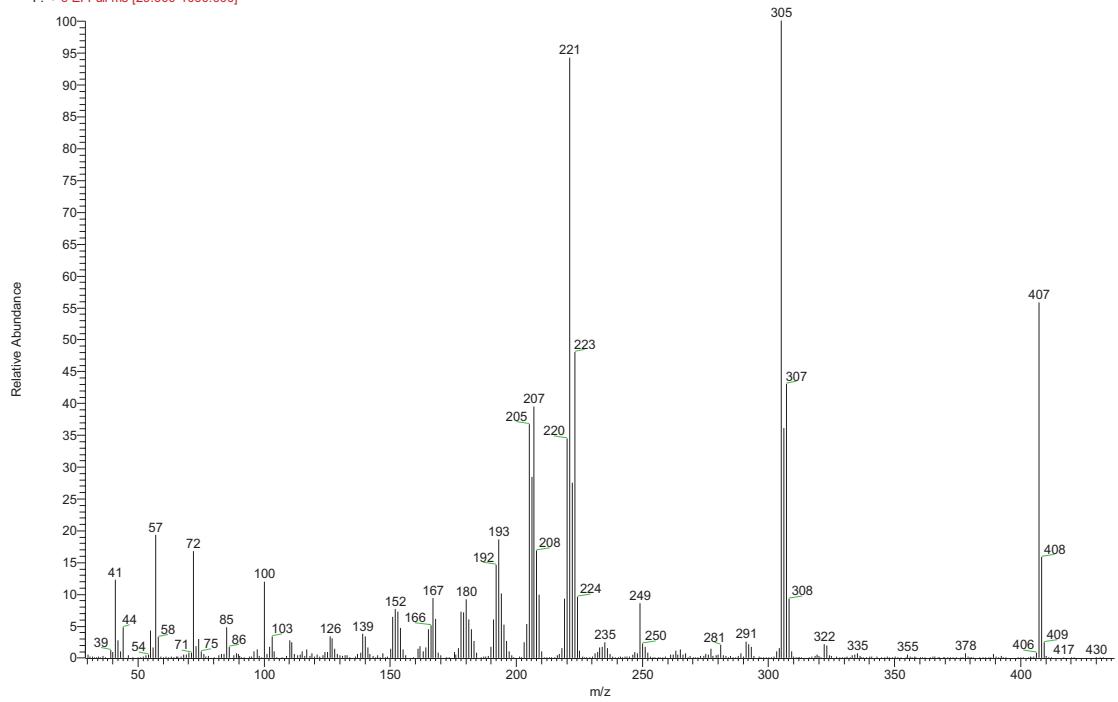


Supporting Information – Drug Testing and Analysis

21_ADB-088 #1637 RT: 30.30 AV: 1 SB: 2 30.12 , 30.77 NL: 1.87E6
 T: + c El Full ms [29.000-1000.000]

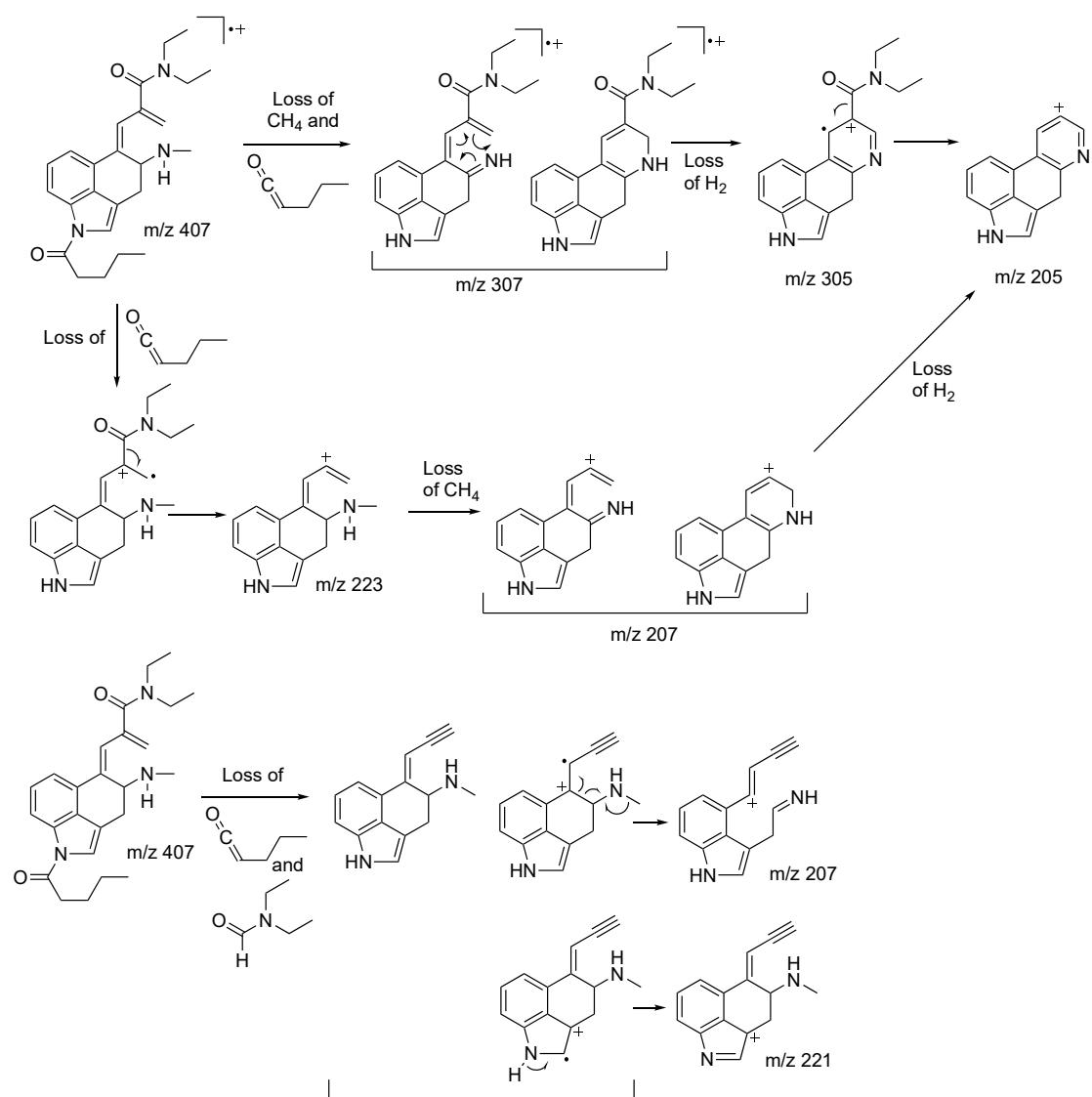


21_ADB-088_SB_MeOH #1639 RT: 30.33 AV: 1 SB: 2 30.03 , 30.63 NL: 2.97E6
 F: + c El Full ms [29.000-1000.000]



See also page 7. Top: GC-MS TIC trace following alkaline extraction into diethyl ether. Bottom: GC-MS TIC trace following analysis of 1V-LSD hemitartrate salt dissolved in methanol.

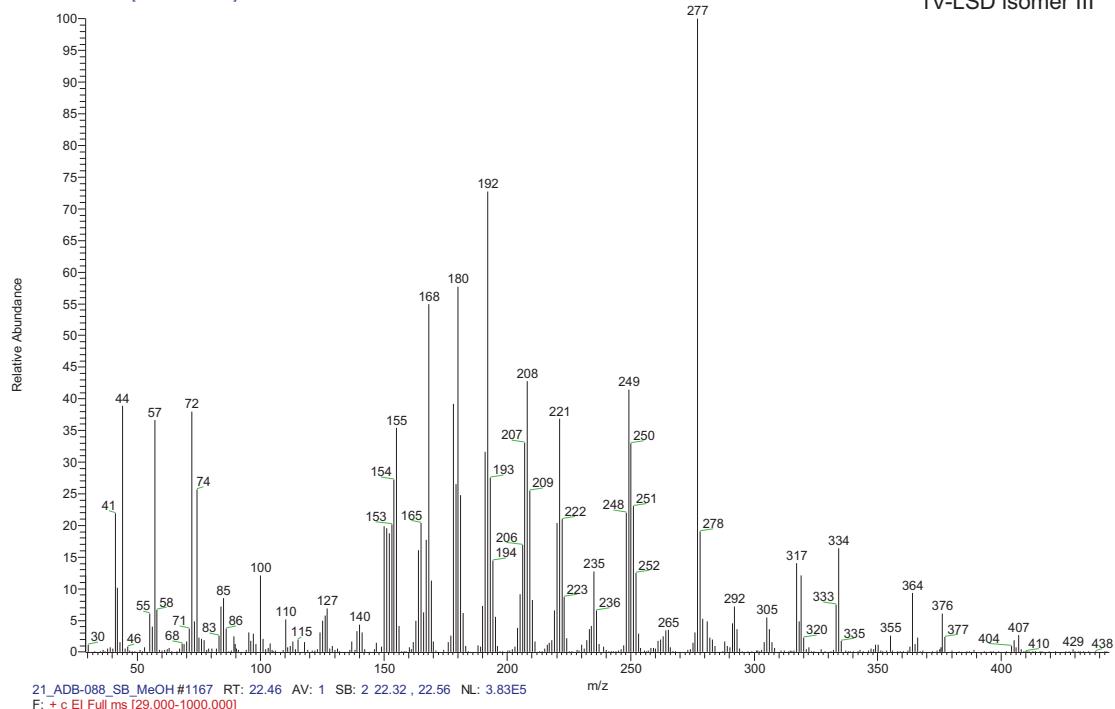
Tentative identification of isomer II



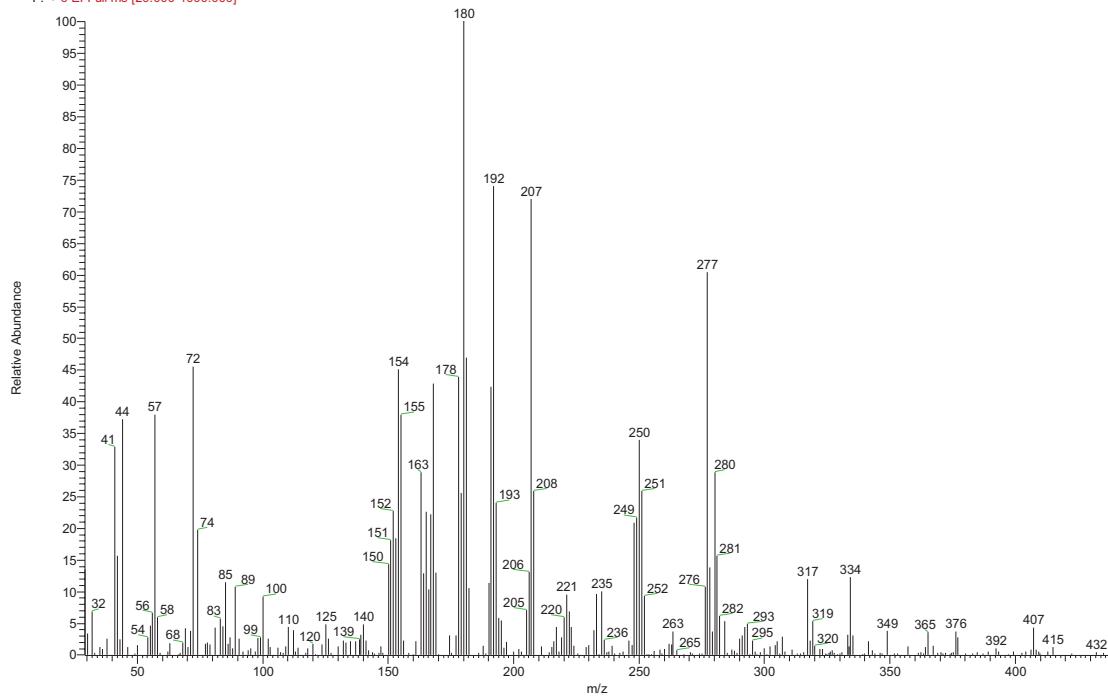
Supporting Information – Drug Testing and Analysis

21_ADB-088 #1170 RT: 22.51 AV: 1 SB: 2 22.36 , 22.61 NL: 1.97E6
 T: + c El Full ms [29.000-1000.000]

1V-LSD isomer III

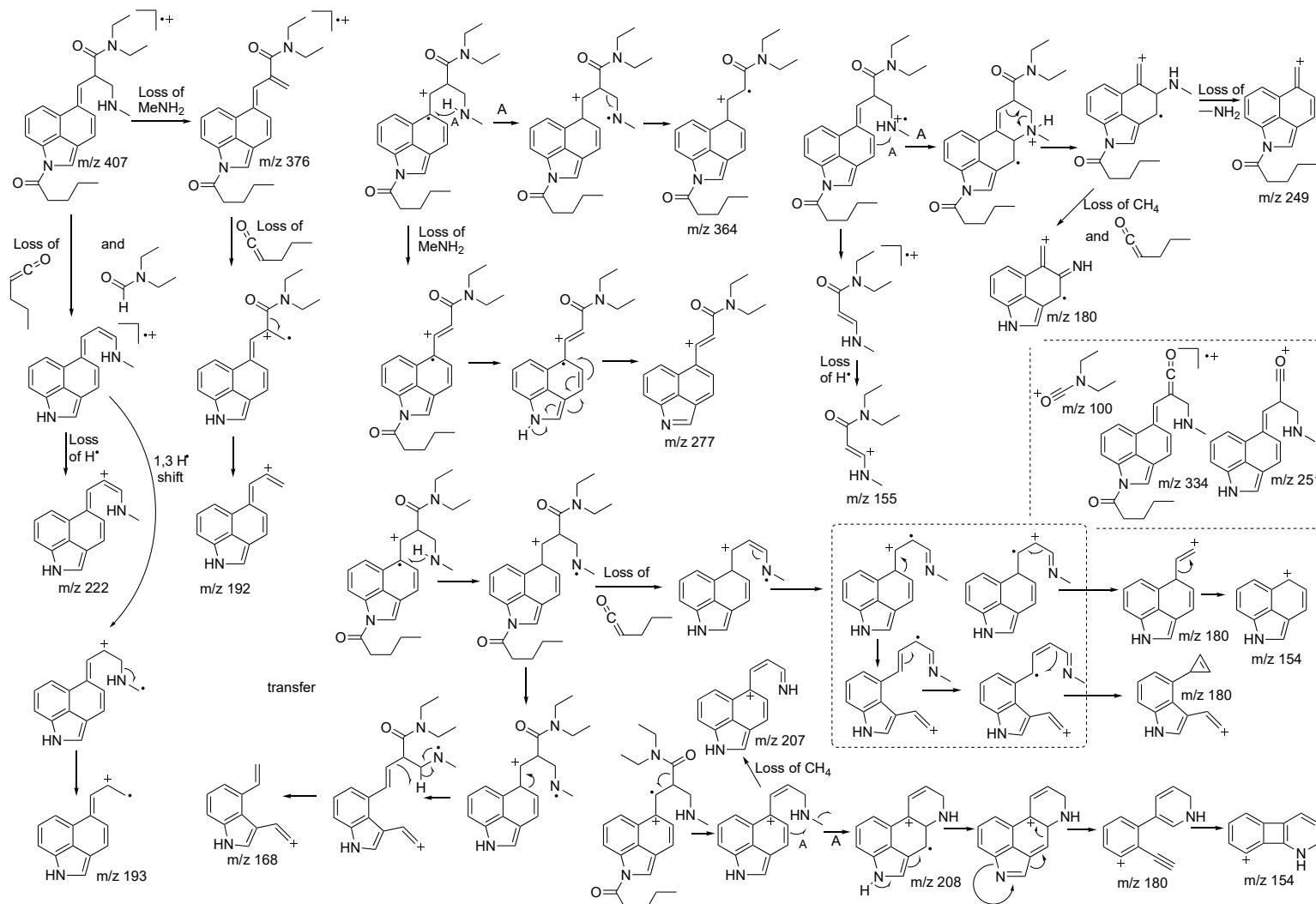


21_ADB-088_SB_MeOH #1167 RT: 22.46 AV: 1 SB: 2 22.32 , 22.56 NL: 3.83E5
 F: + c El Full ms [29.000-1000.000]



See also page 7. Top: GC-MS TIC trace following alkaline extraction into diethyl ether. Bottom: GC-MS TIC trace following analysis of 1V-LSD hemitartrate salt dissolved in methanol.

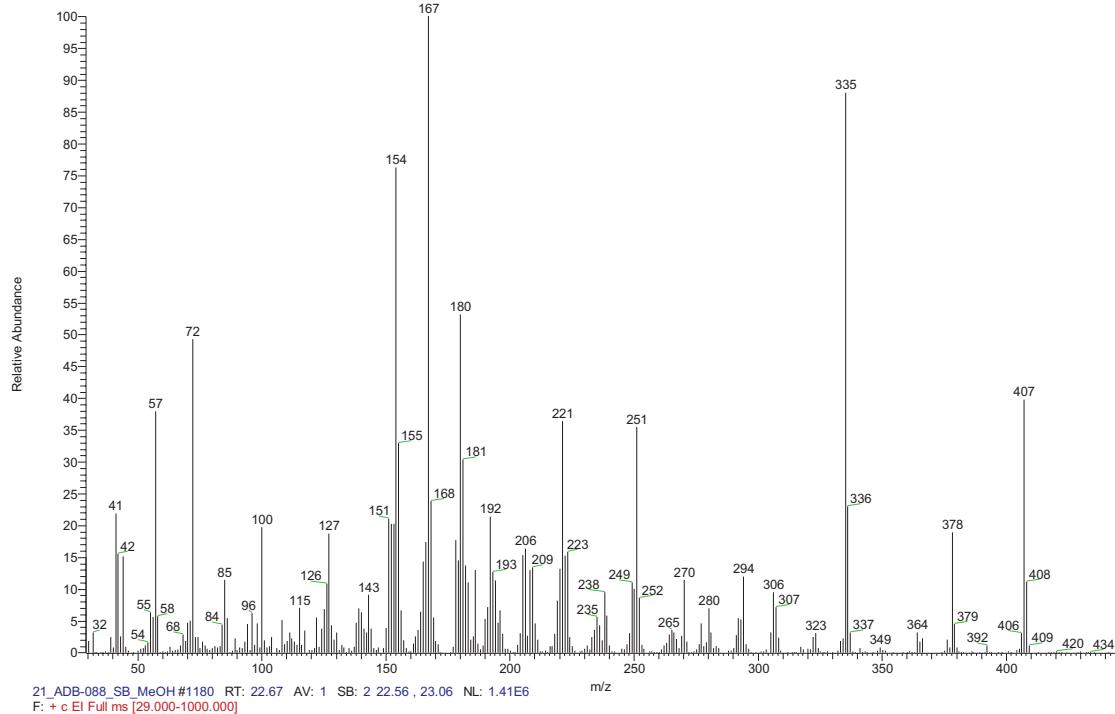
Tentative identification of isomer III



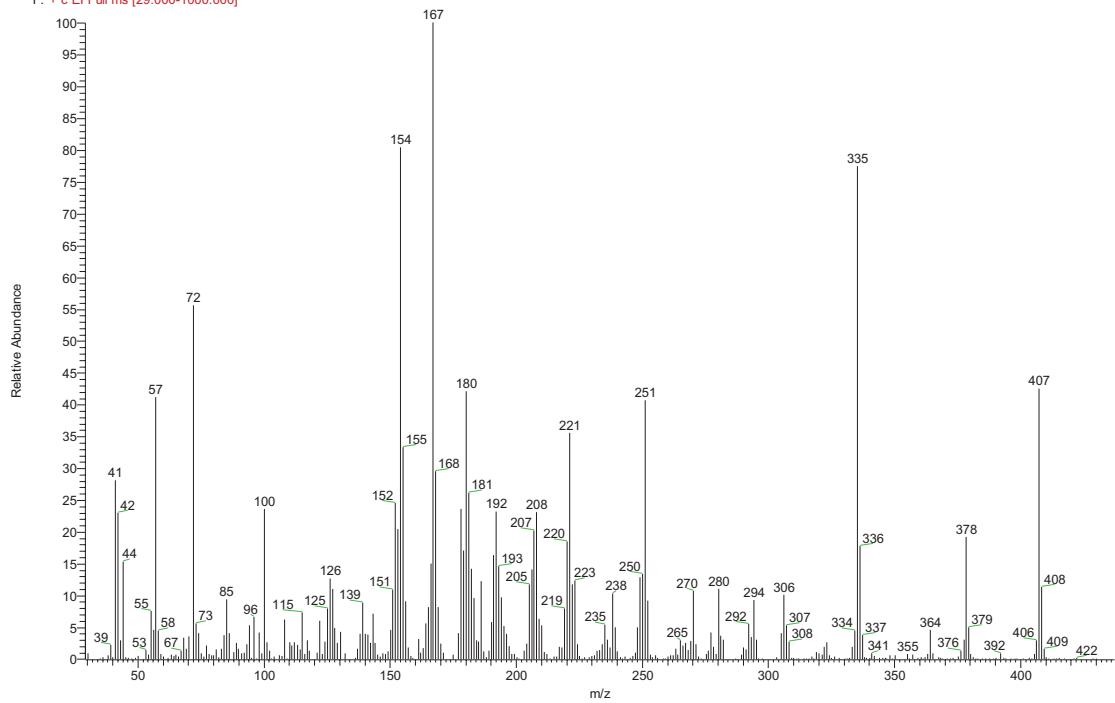
Supporting Information – Drug Testing and Analysis

21_ADB-088 #1183 RT: 22.72 AV: 1 SB: 2 22.61 , 23.03 NL: 2.54E6
 T: + c El Full ms [29.000-1000.000]

1V-LSD isomer IV

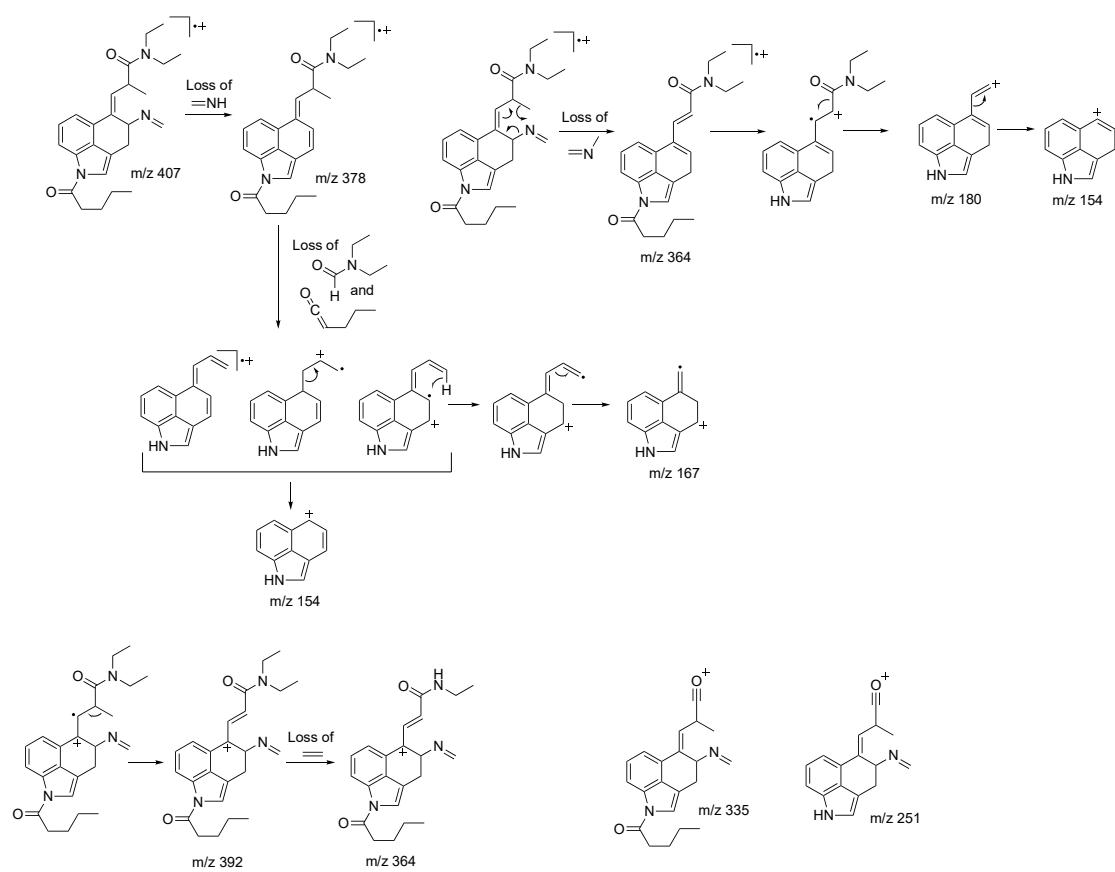


21_ADB-088_SB_MeOH #1180 RT: 22.67 AV: 1 SB: 2 22.56 , 23.06 NL: 1.41E6
 F: + c El Full ms [29.000-1000.000]



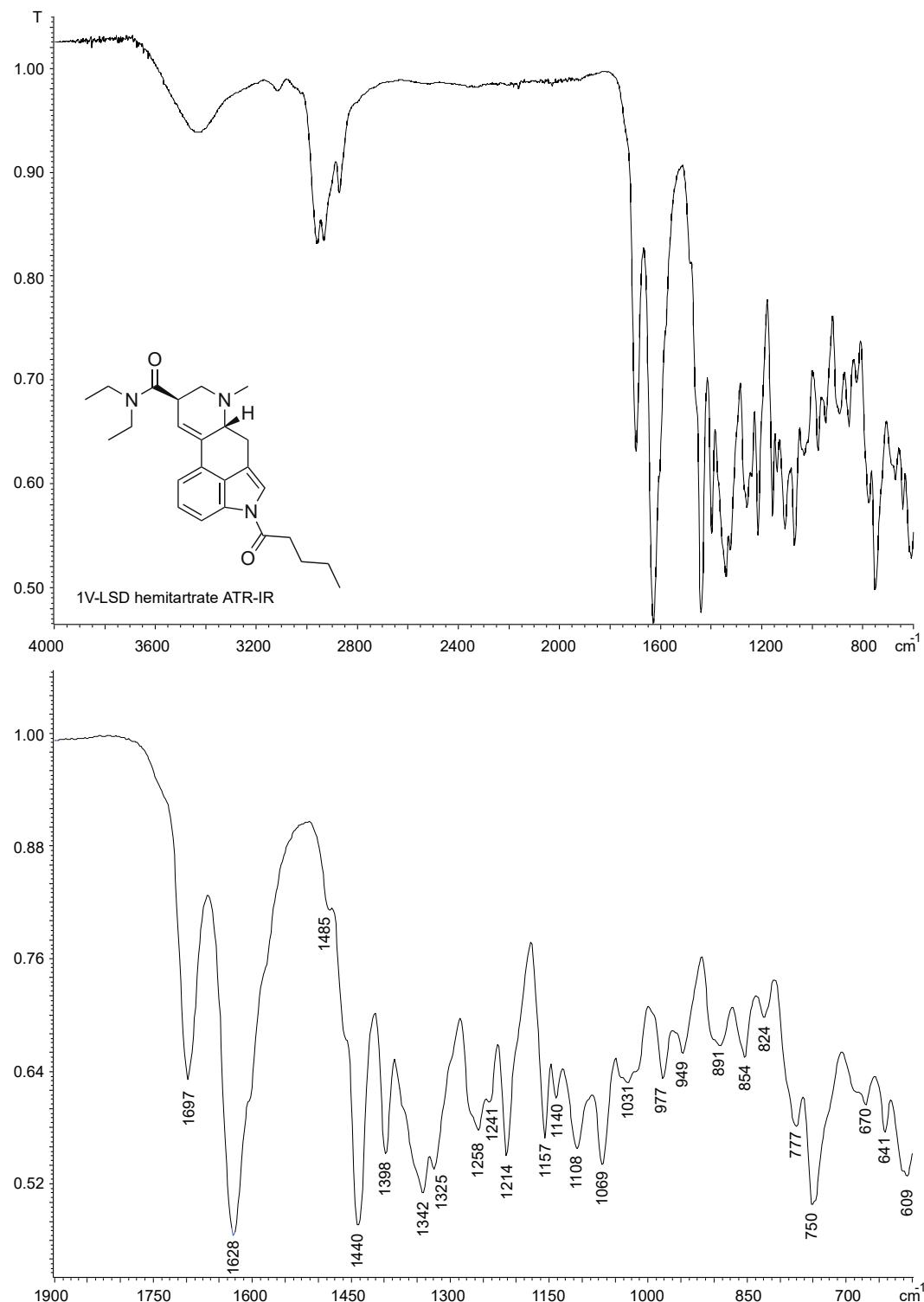
See also page 7. Top: GC-MS TIC trace following alkaline extraction into diethyl ether. Bottom: GC-MS TIC trace following analysis of 1V-LSD hemitartrate salt dissolved in methanol.

Tentative identification of isomer IV

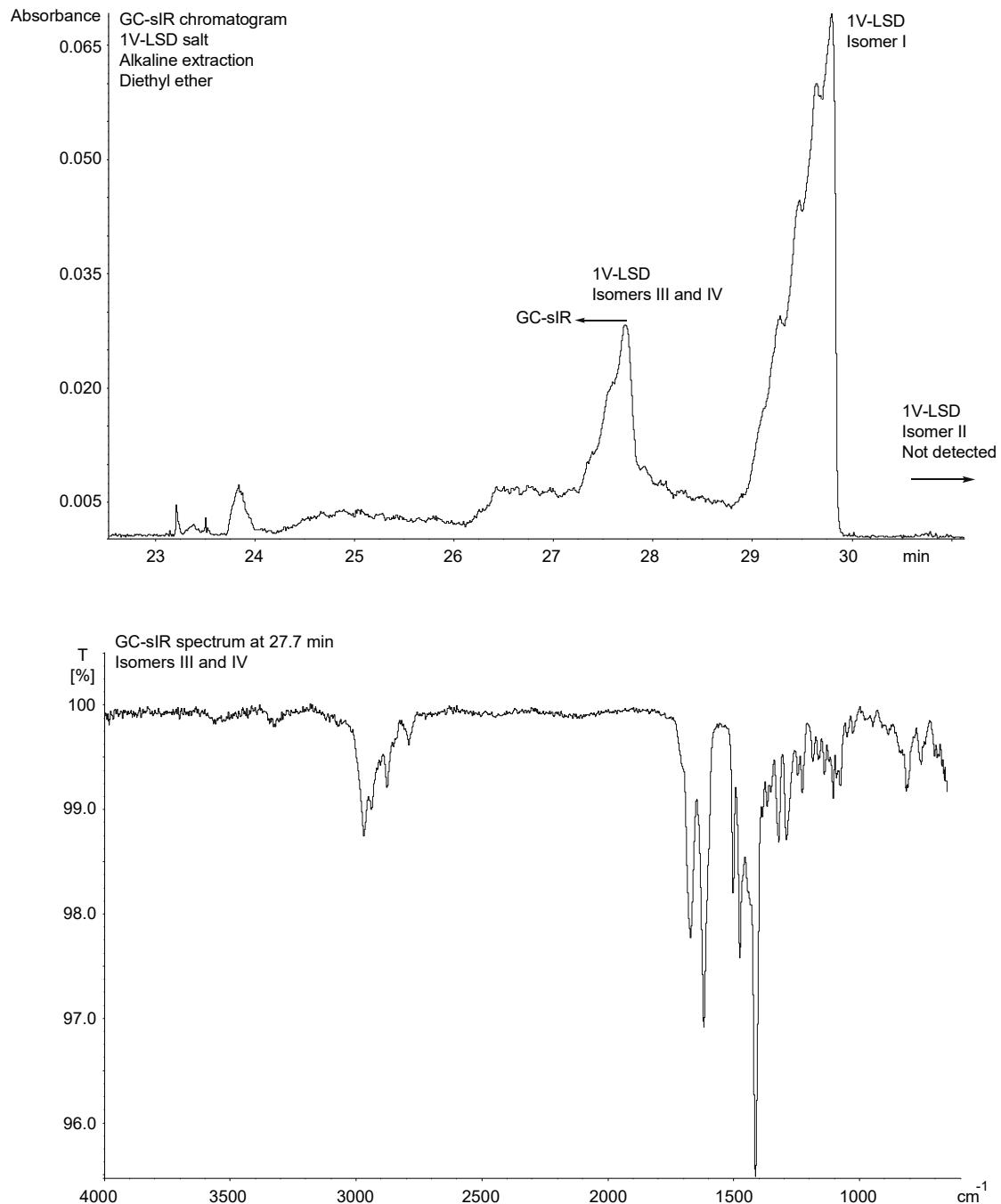


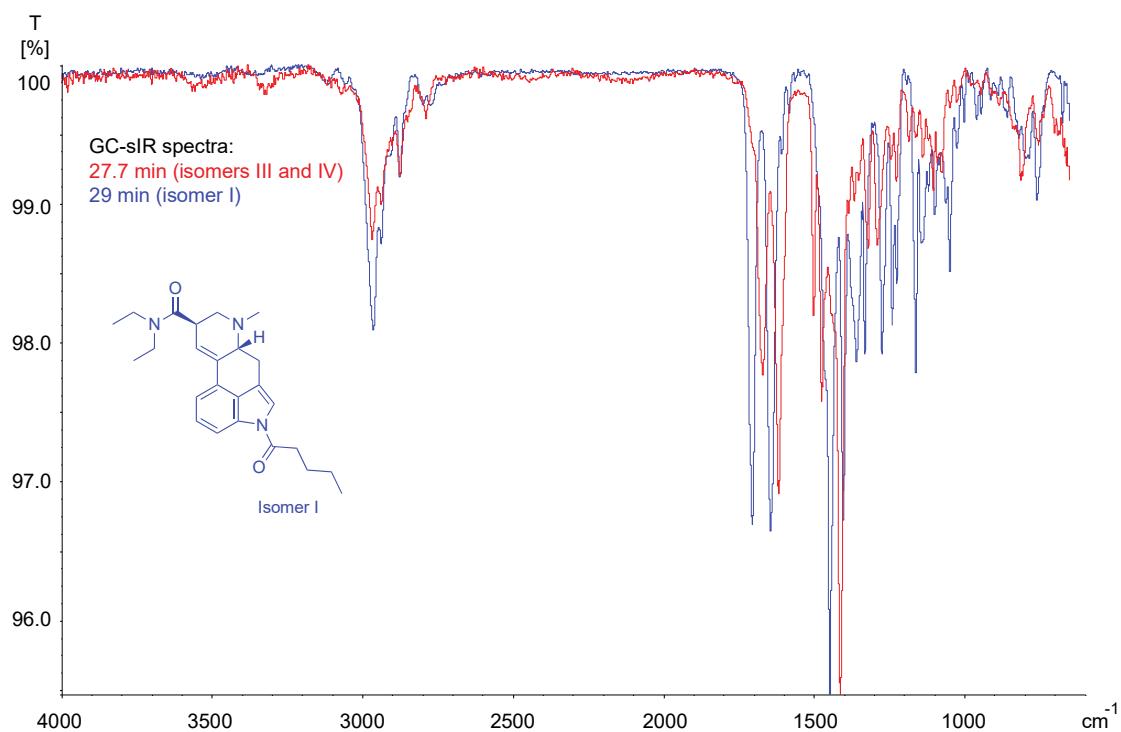
Attenuated total reflection-infrared spectroscopy (ATR-IR)

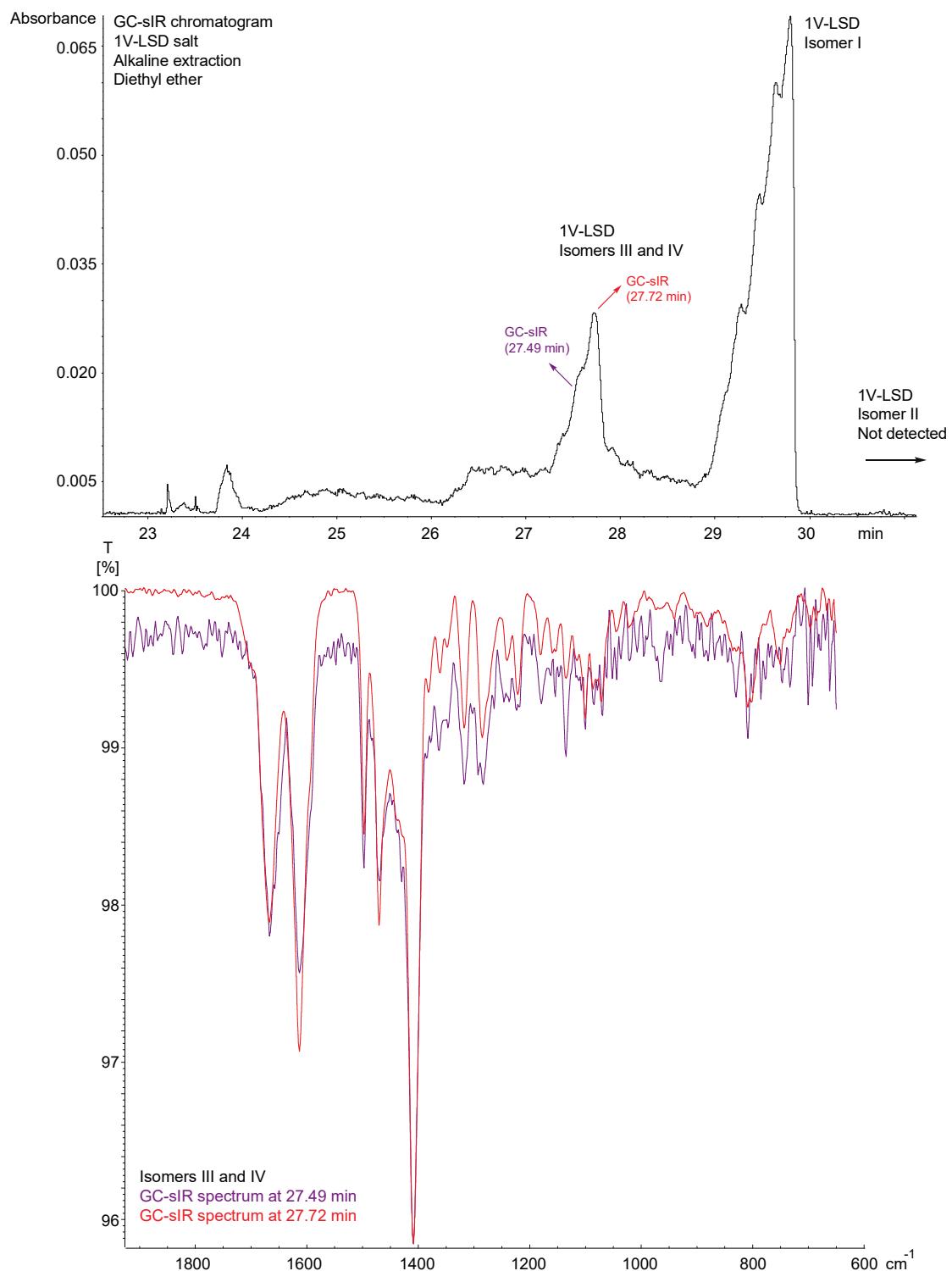
A Nicolet 380 FT-IR spectrometer with Smart Golden Gate Diamond ATR and the software OMNIC, Ver. 7.4.127 (Thermo Electron Corporation, Dreieich, DE) were used for data acquisition and data analysis, respectively. Wavelength resolution was 4 cm^{-1} , scan range $650\text{--}4000\text{ cm}^{-1}$, and 32 scans/spectrum were acquired. IR spectra were recorded from the solid.



Supporting Information – Drug Testing and Analysis

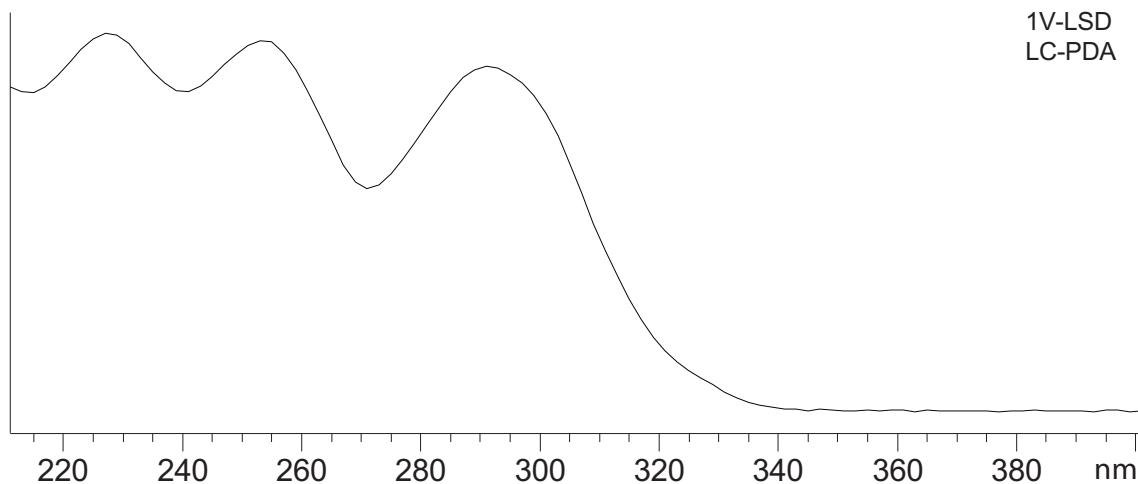
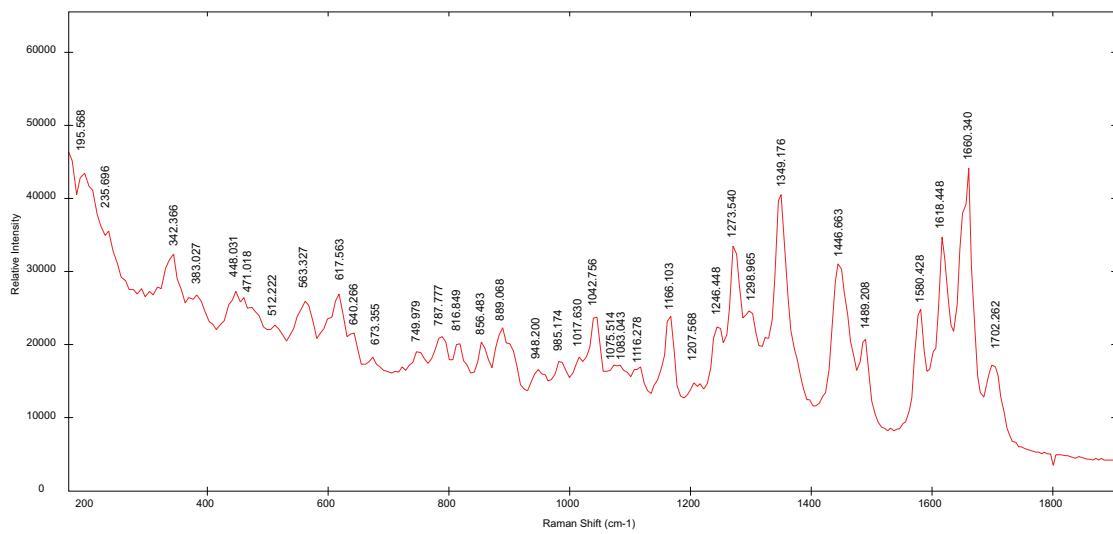




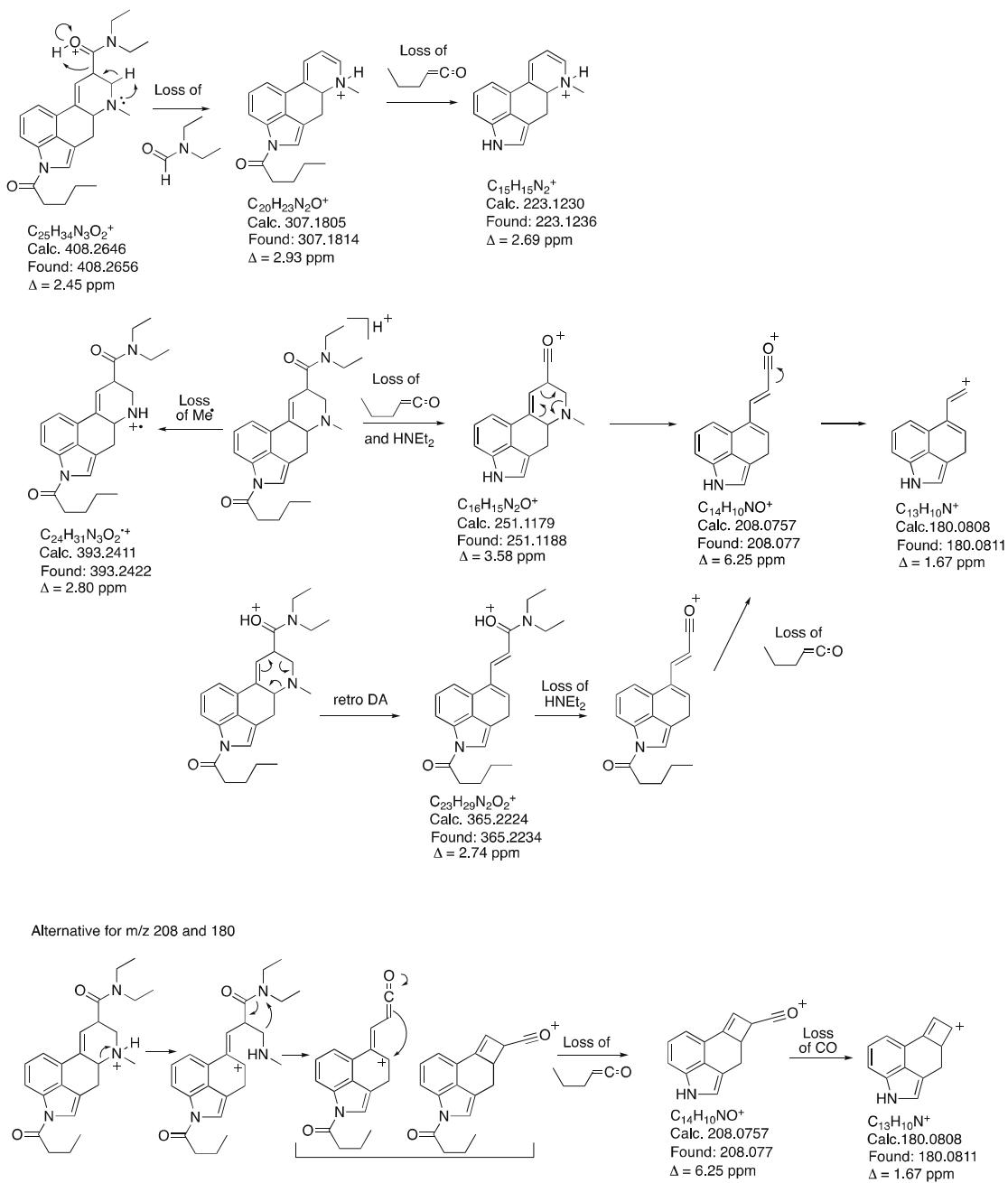


Raman spectroscopy

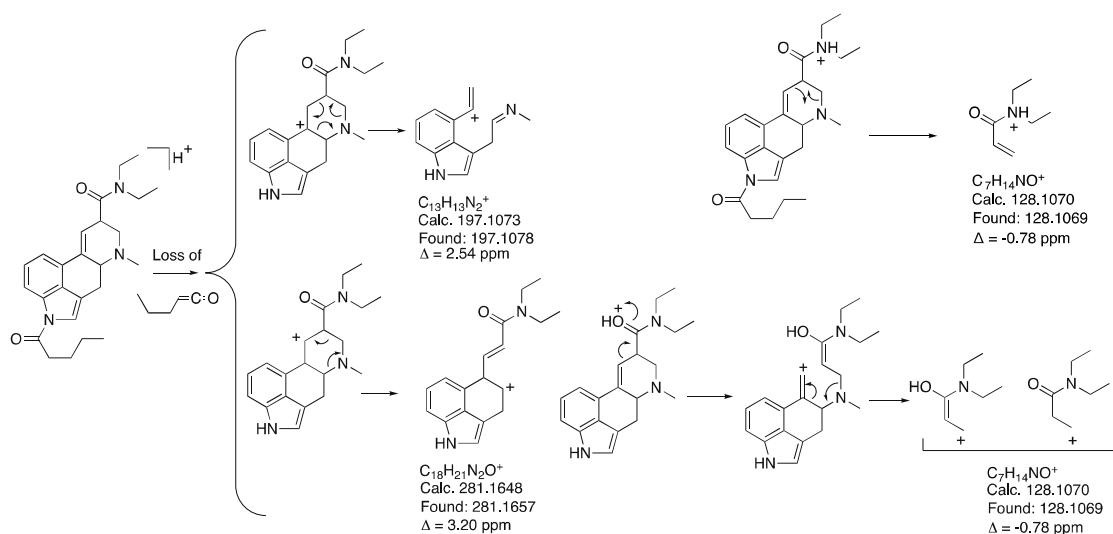
Raman spectroscopic data was acquired using laser irradiation at 1064 nm using an i-Raman® EX system and a BWS485-1064S-05 spectrometer using a scan range of 170 – 2502 cm⁻¹; the resolution was ~9.5 cm⁻¹ at 1296 nm. A BAC151B Raman Video Microsampling System was applied with an objective lens magnification of 20x (camera: active pixels 1280 x 1024). The applied software was BWSpec® 4.10_4 (B&W TEK). Integration time was chosen to reach a relative intensity above 45,000 arbitrary units for the most intensive peak.

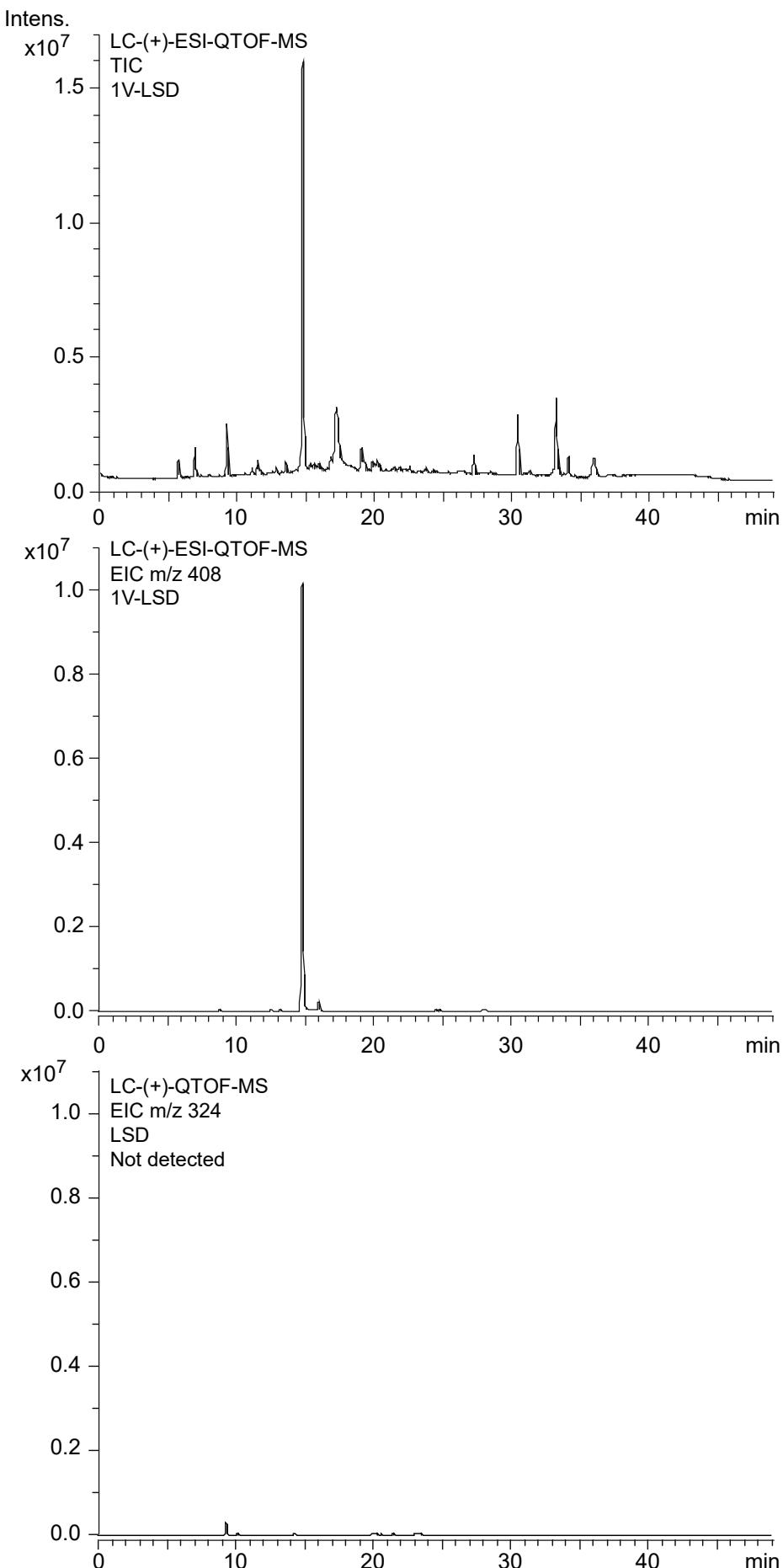


Proposed formation of product ions following collision-induced dissociation of 1V-LSD under QTOF-MS/MS conditions

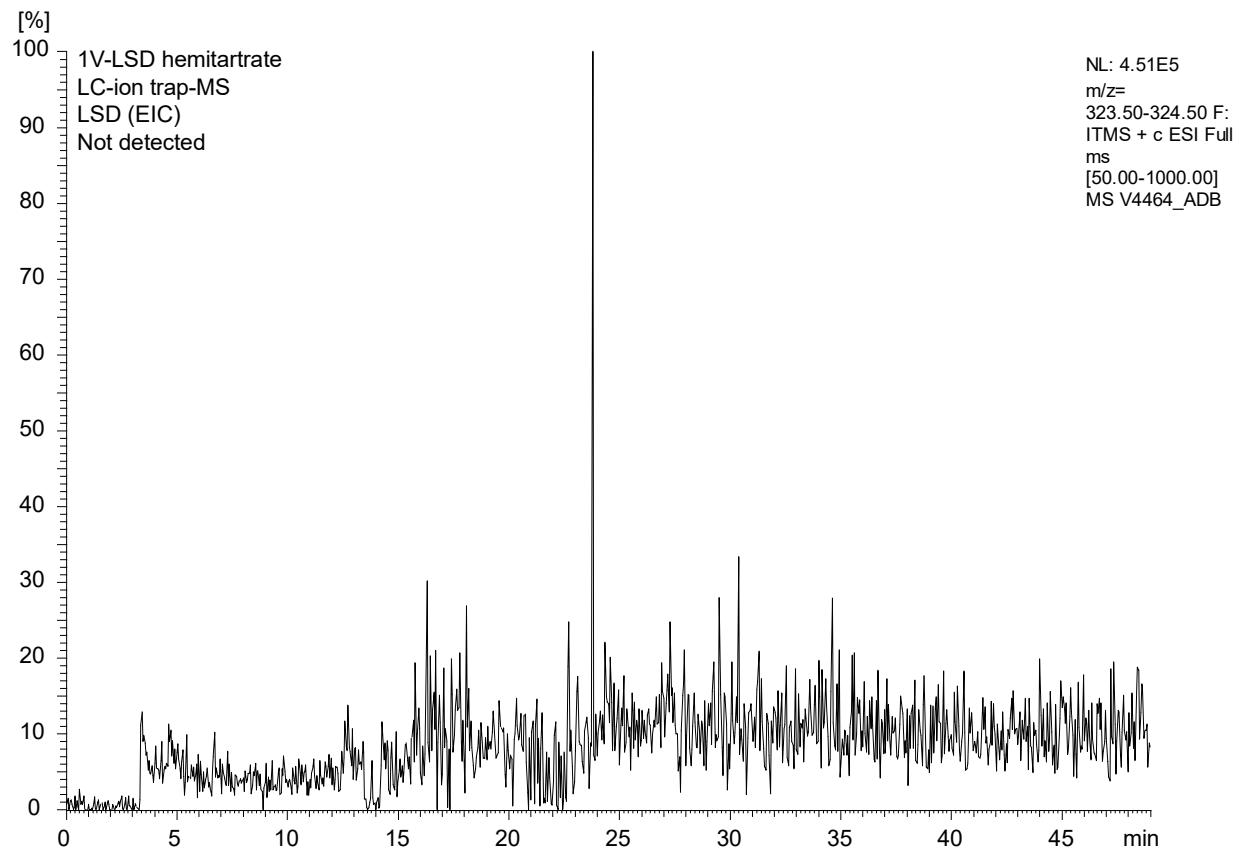


Proposed formation of product ions following collision-induced dissociation of 1V-LSD under QTOF-MS/MS conditions (continued).

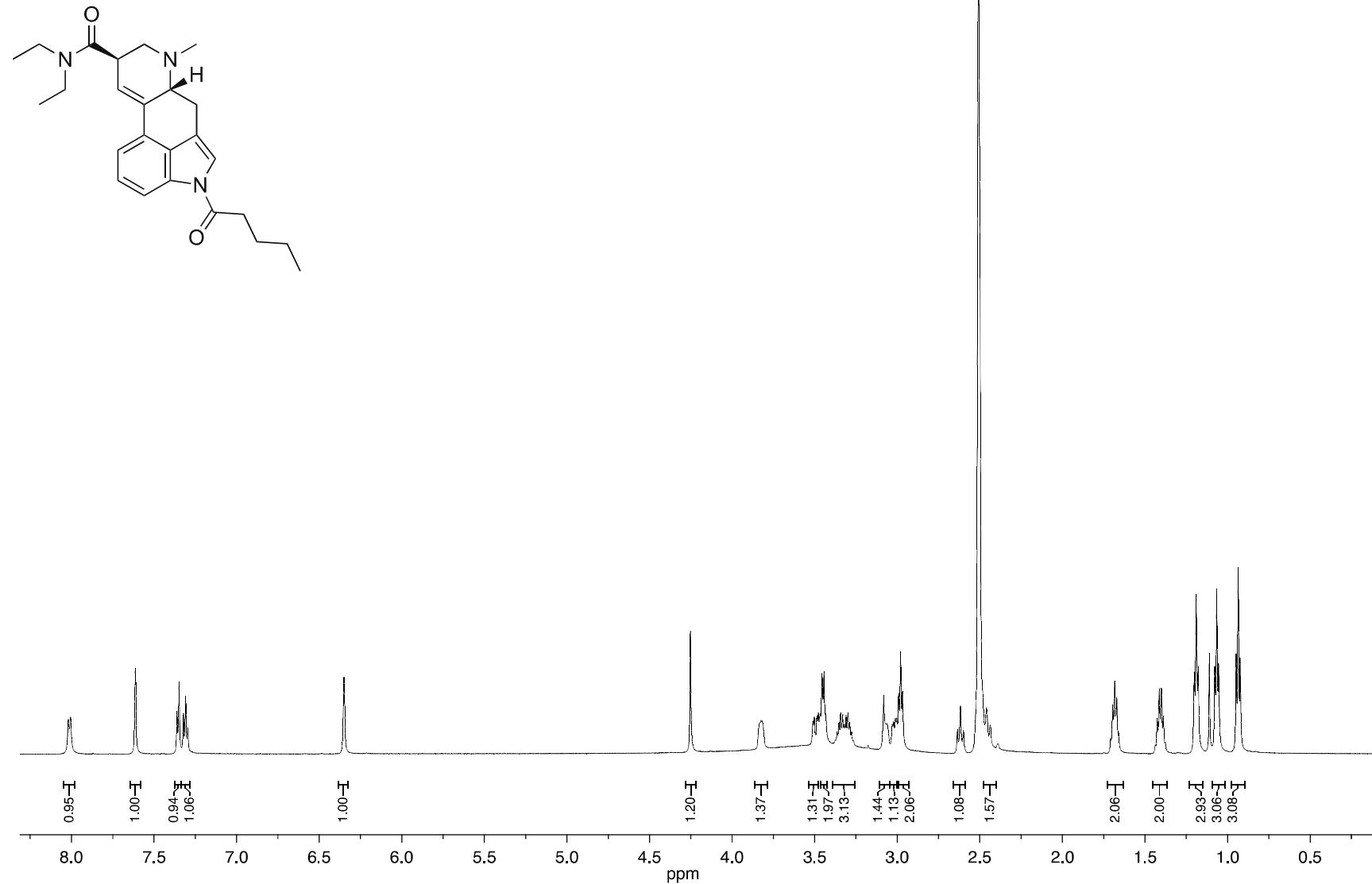




Supporting Information – Drug Testing and Analysis

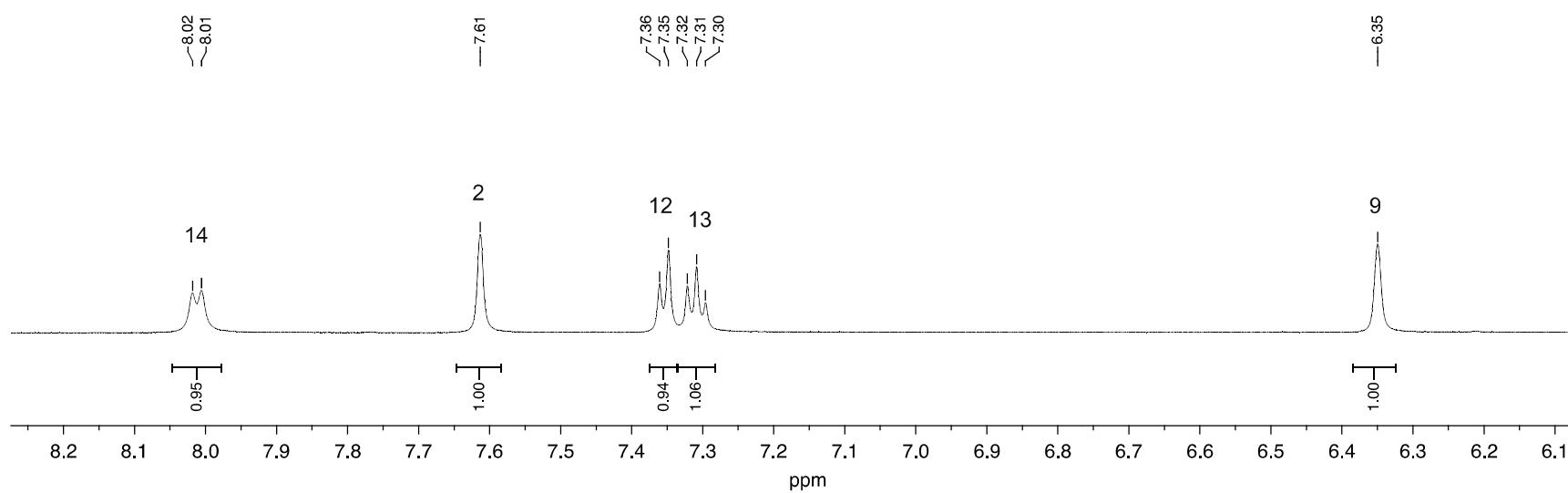
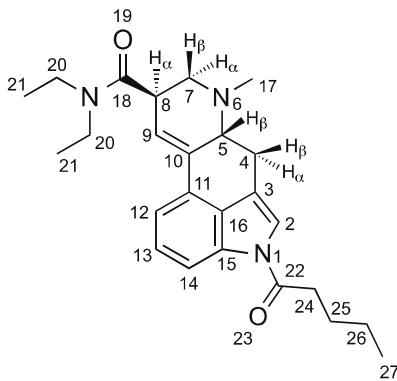


1V-LSD hemitartrate (2:1)
 ^1H -NMR (600 MHz)
 d_6 -DMSO



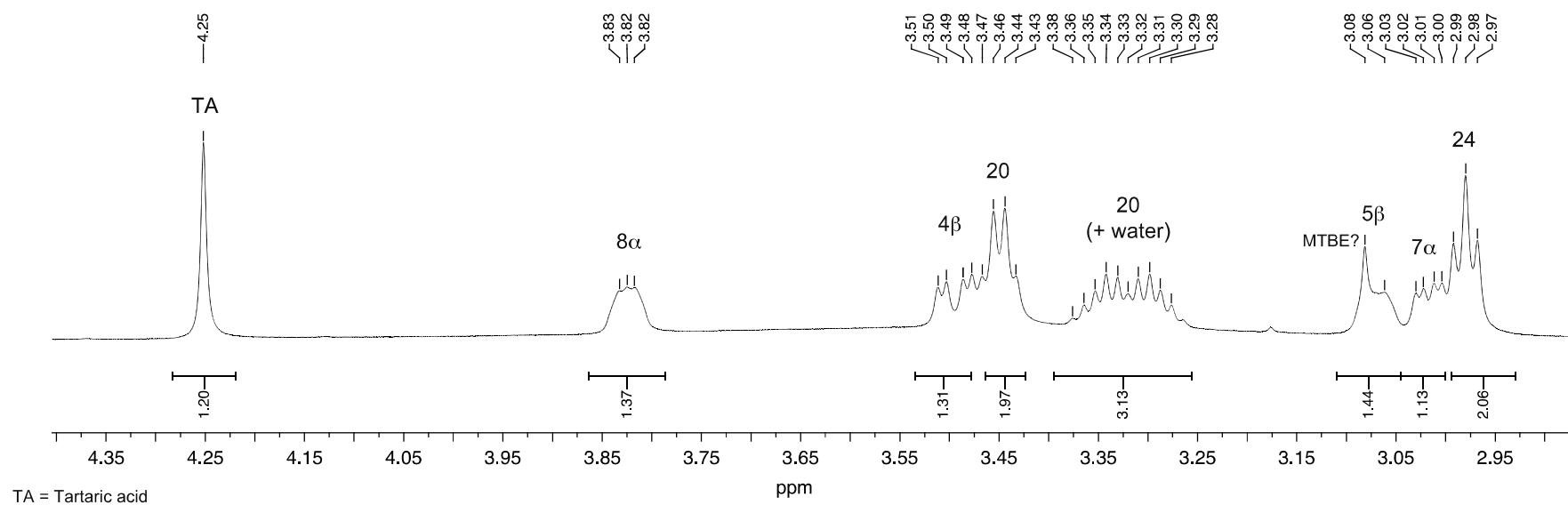
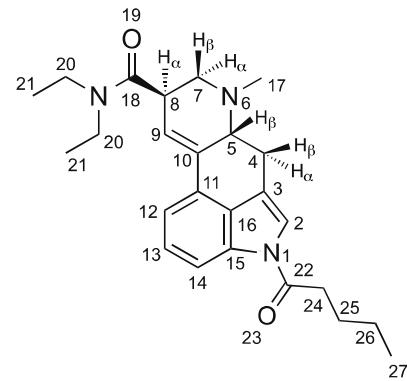
Supporting Information – Drug Testing and Analysis

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



Supporting Information – Drug Testing and Analysis

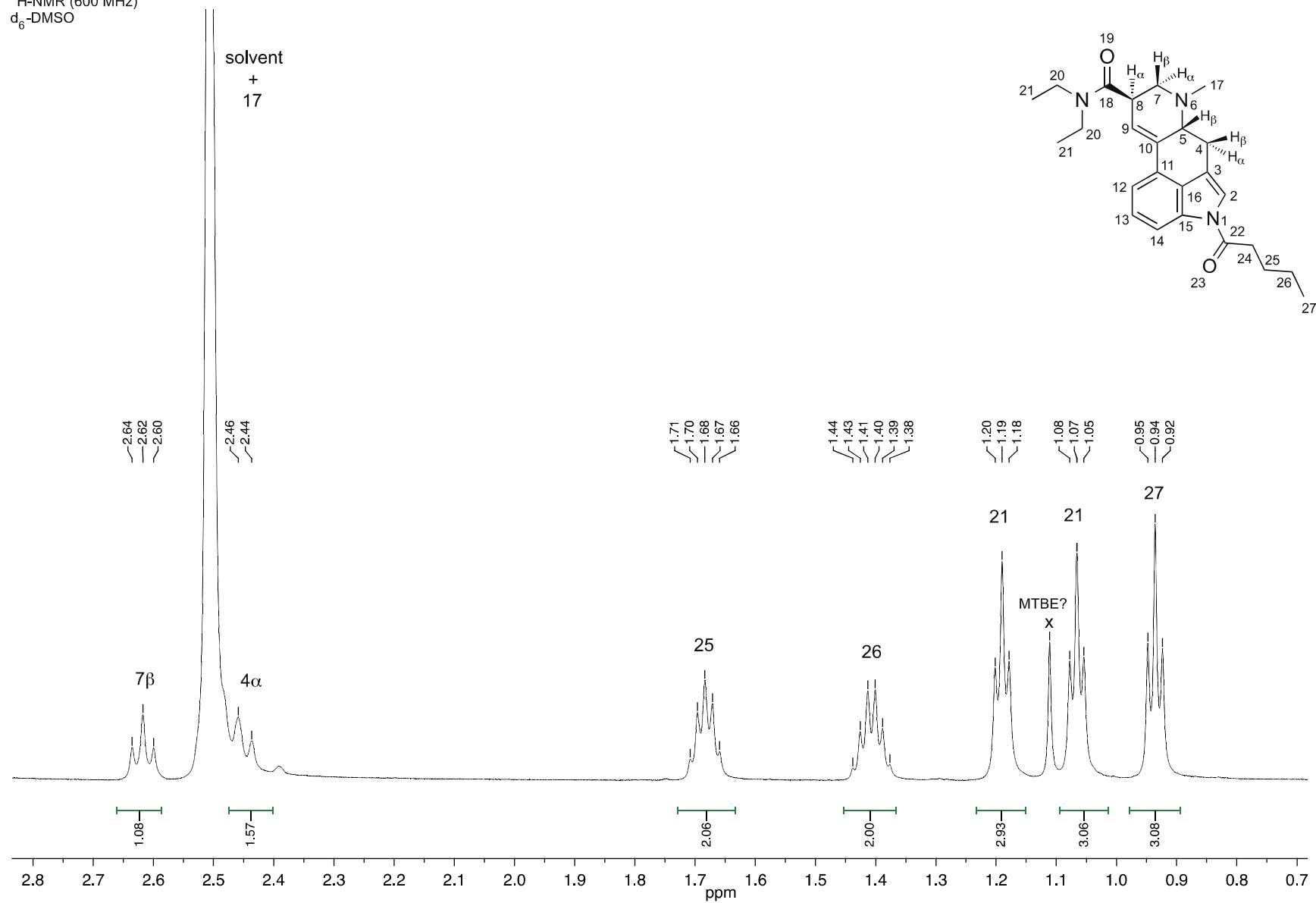
1V-LSD hemitartrate (2:1)
¹H-NMR (600 MHz)
d₆-DMSO



TA = Tartaric acid

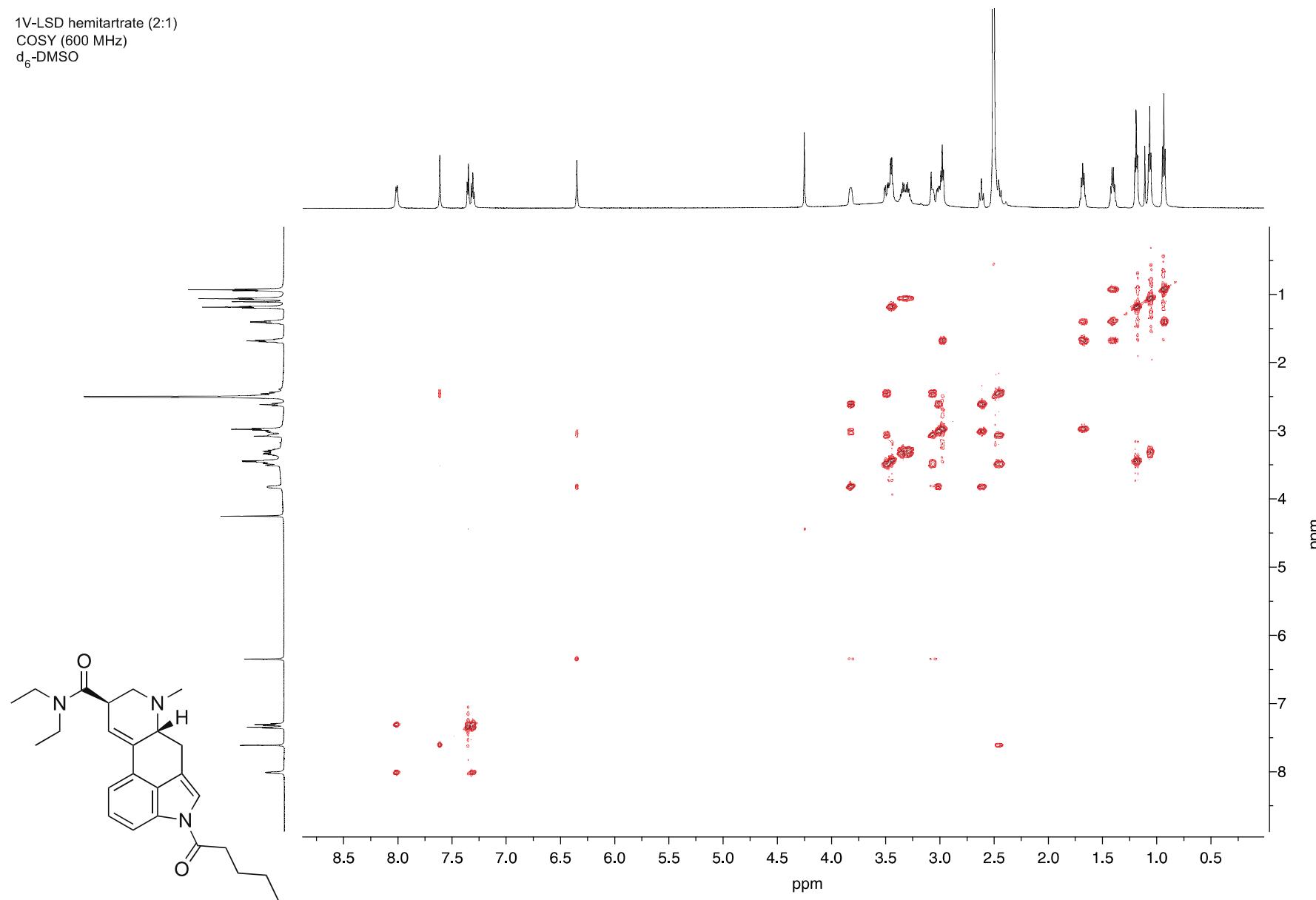
Supporting Information – Drug Testing and Analysis

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



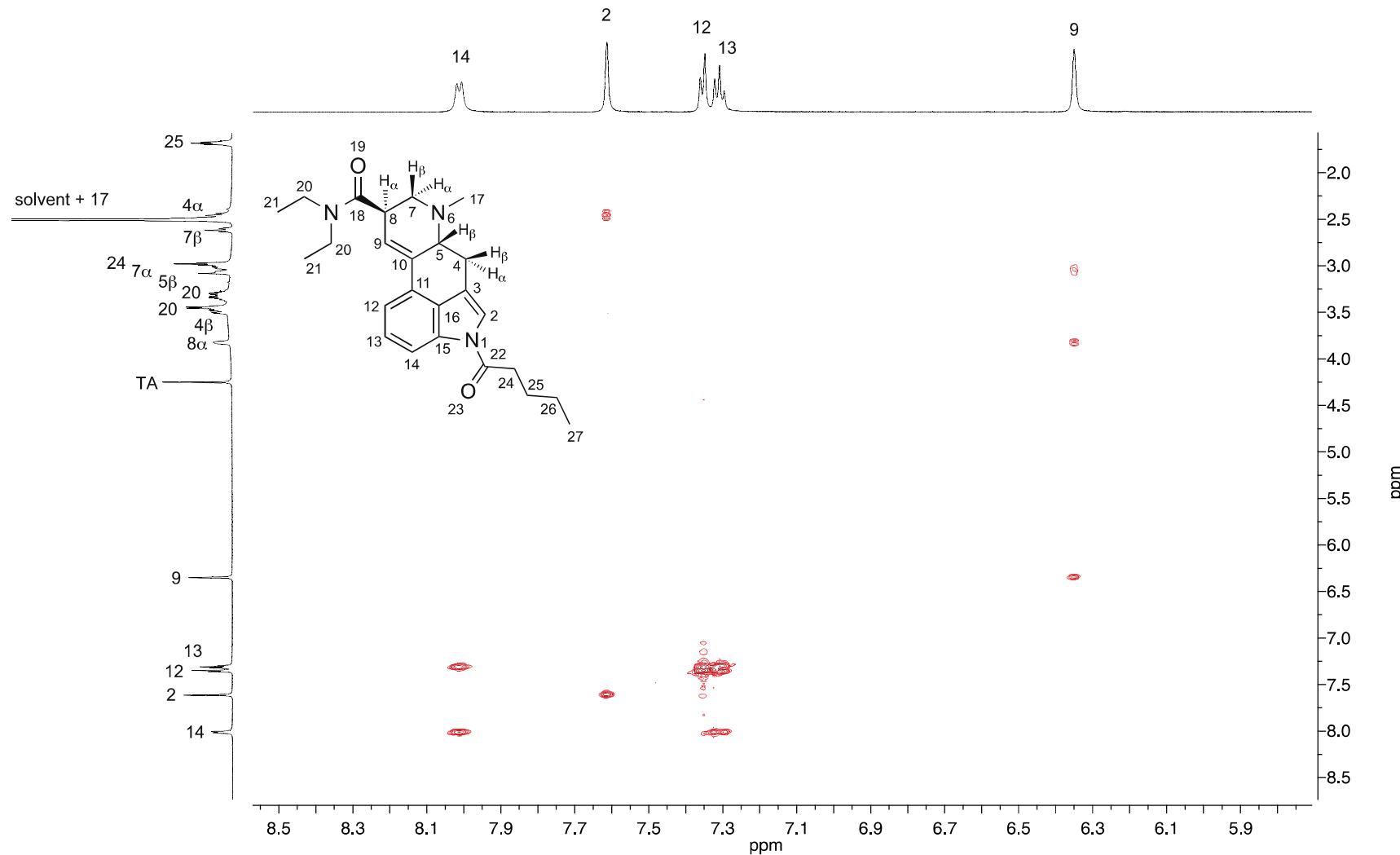
Supporting Information – Drug Testing and Analysis

1V-LSD hemitartrate (2:1)
COSY (600 MHz)
 d_6 -DMSO



Supporting Information – Drug Testing and Analysis

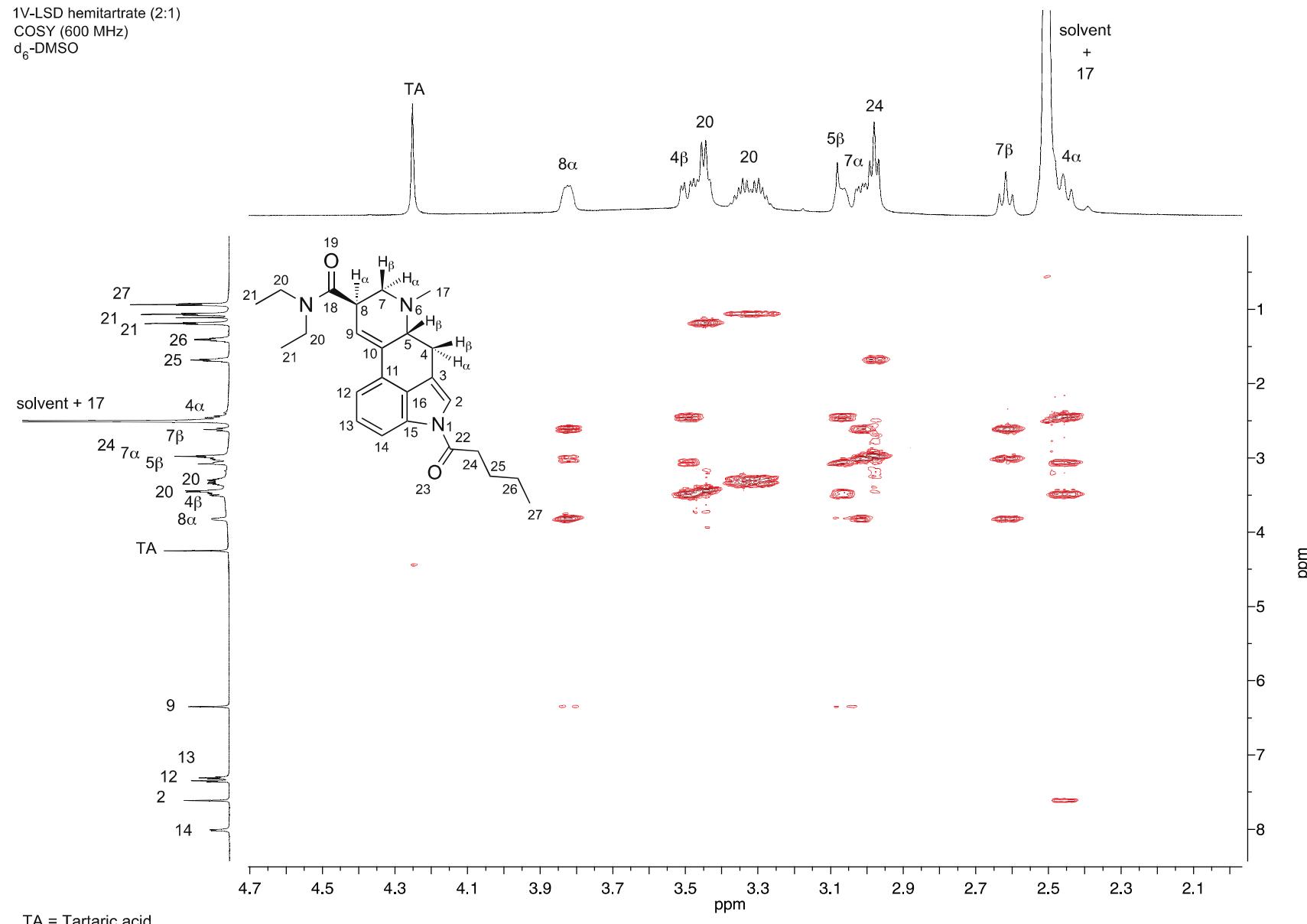
1V-LSD hemitartrate (2:1)
COSY (600 MHz)
 d_6 -DMSO



TA = Tartaric acid

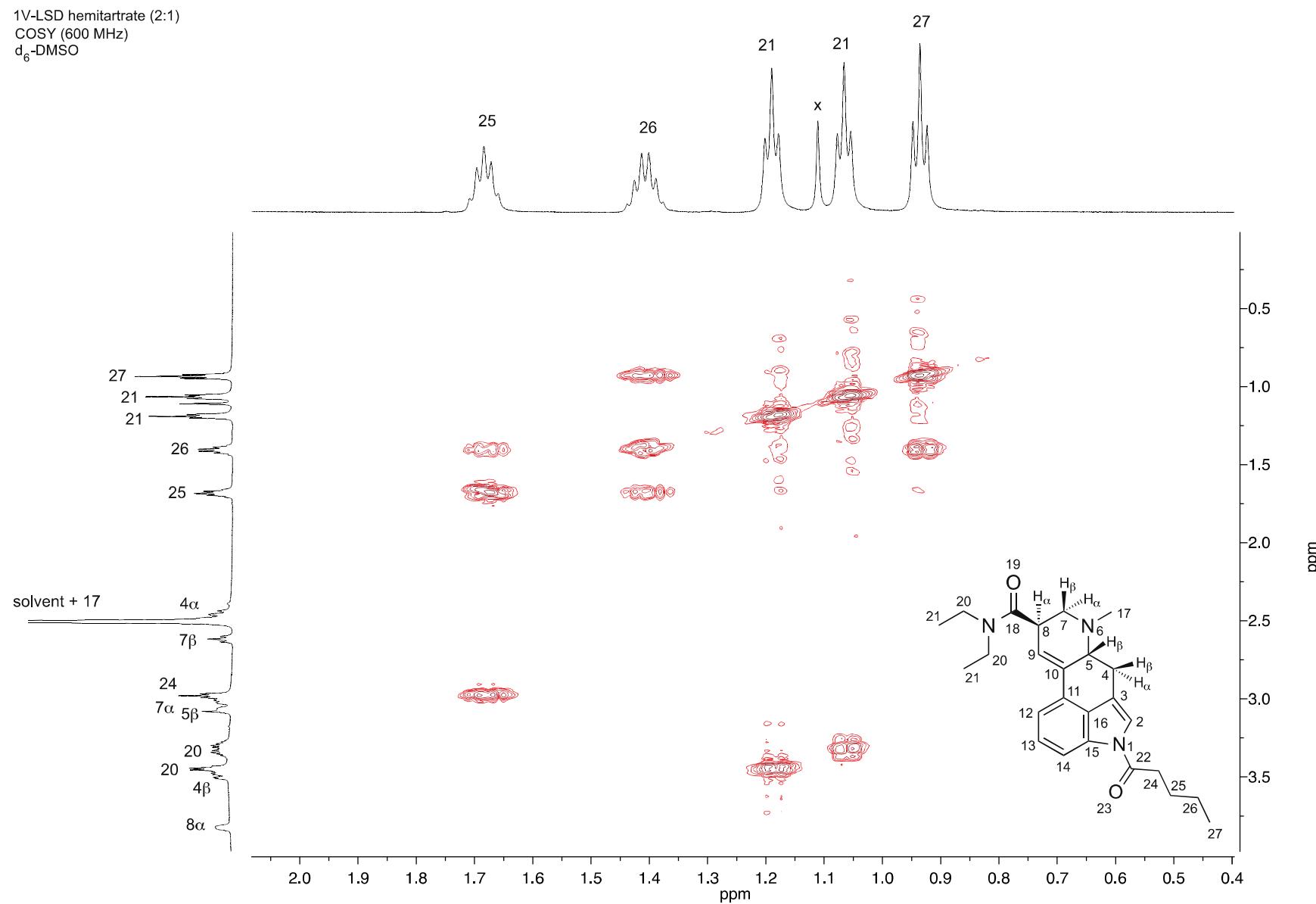
Supporting Information – Drug Testing and Analysis

1V-LSD hemitartrate (2:1)
COSY (600 MHz)
 d_6 -DMSO



TA = Tartaric acid

Supporting Information – Drug Testing and Analysis

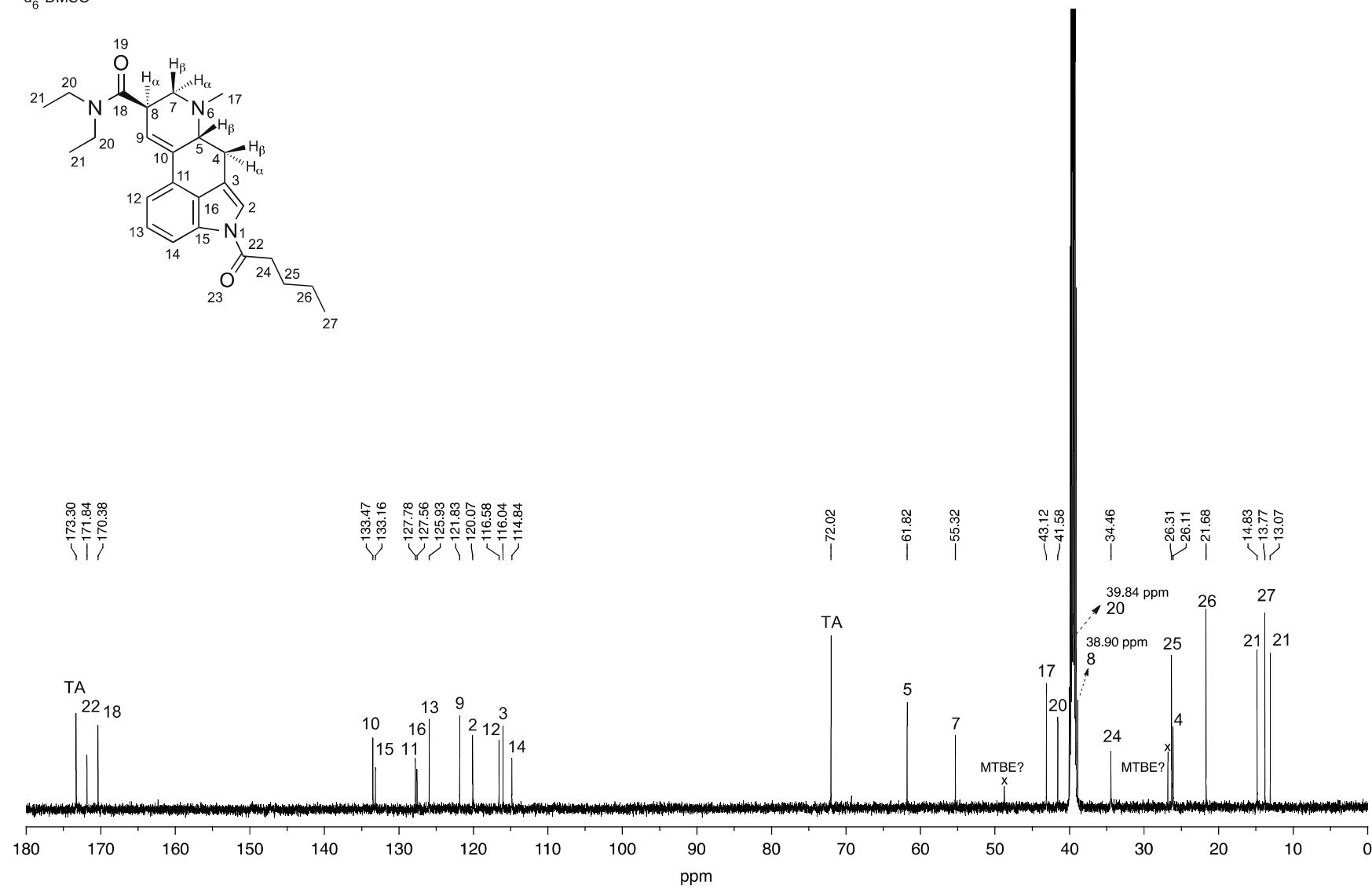
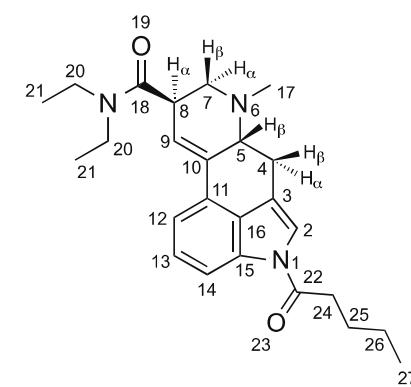


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1V-LSD hemitartrate (2:1)

^{13}C -NMR (150 MHz)

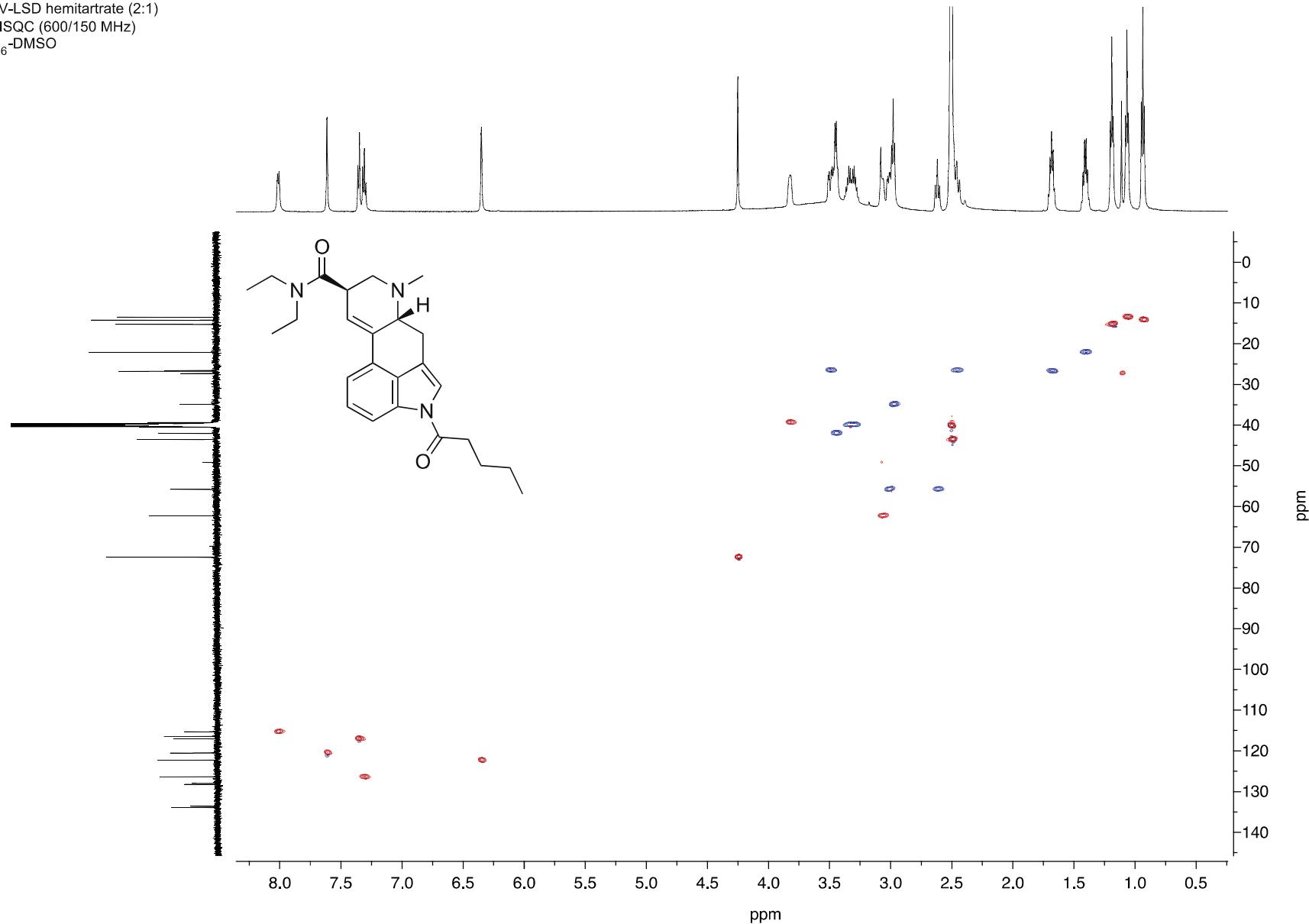
d_6 -DMSO



TA = Tartaric acid

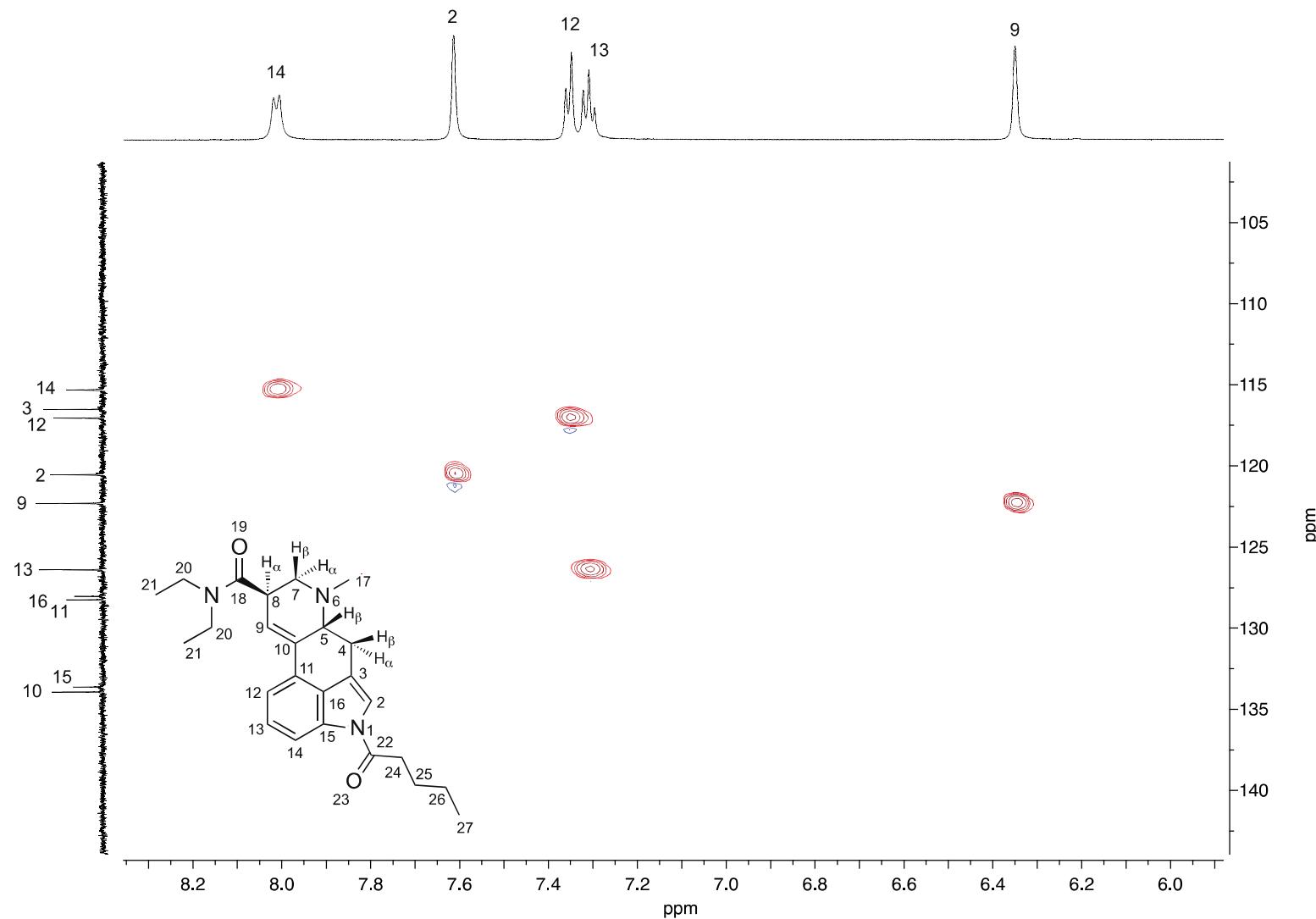
Supporting Information – Drug Testing and Analysis

1V-LSD hemitartrate (2:1)
HSQC (600/150 MHz)
 d_6 -DMSO

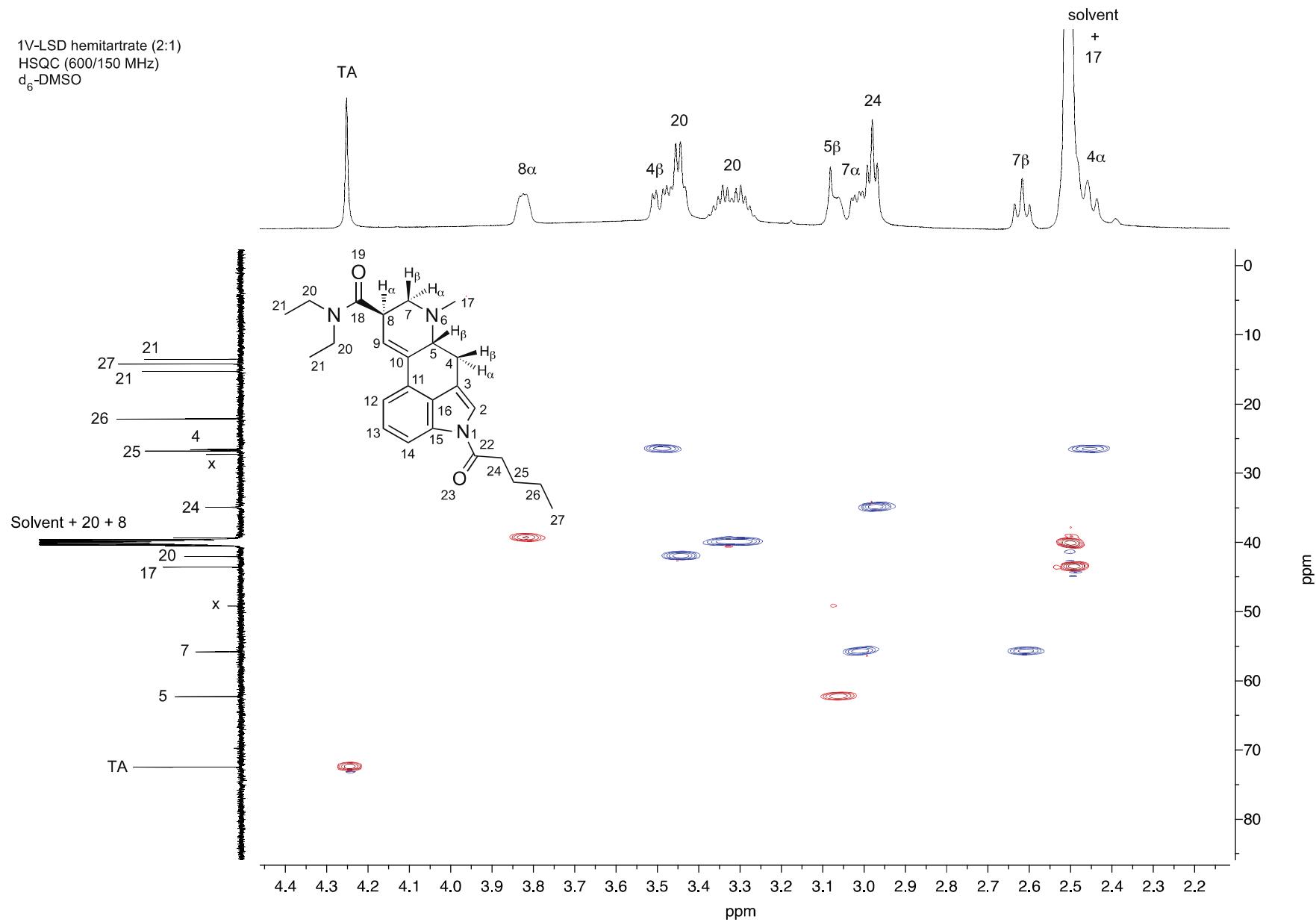


Supporting Information – Drug Testing and Analysis

1V-LSD hemitartrate (2:1)
HSQC (600/150 MHz)
 d_6 -DMSO

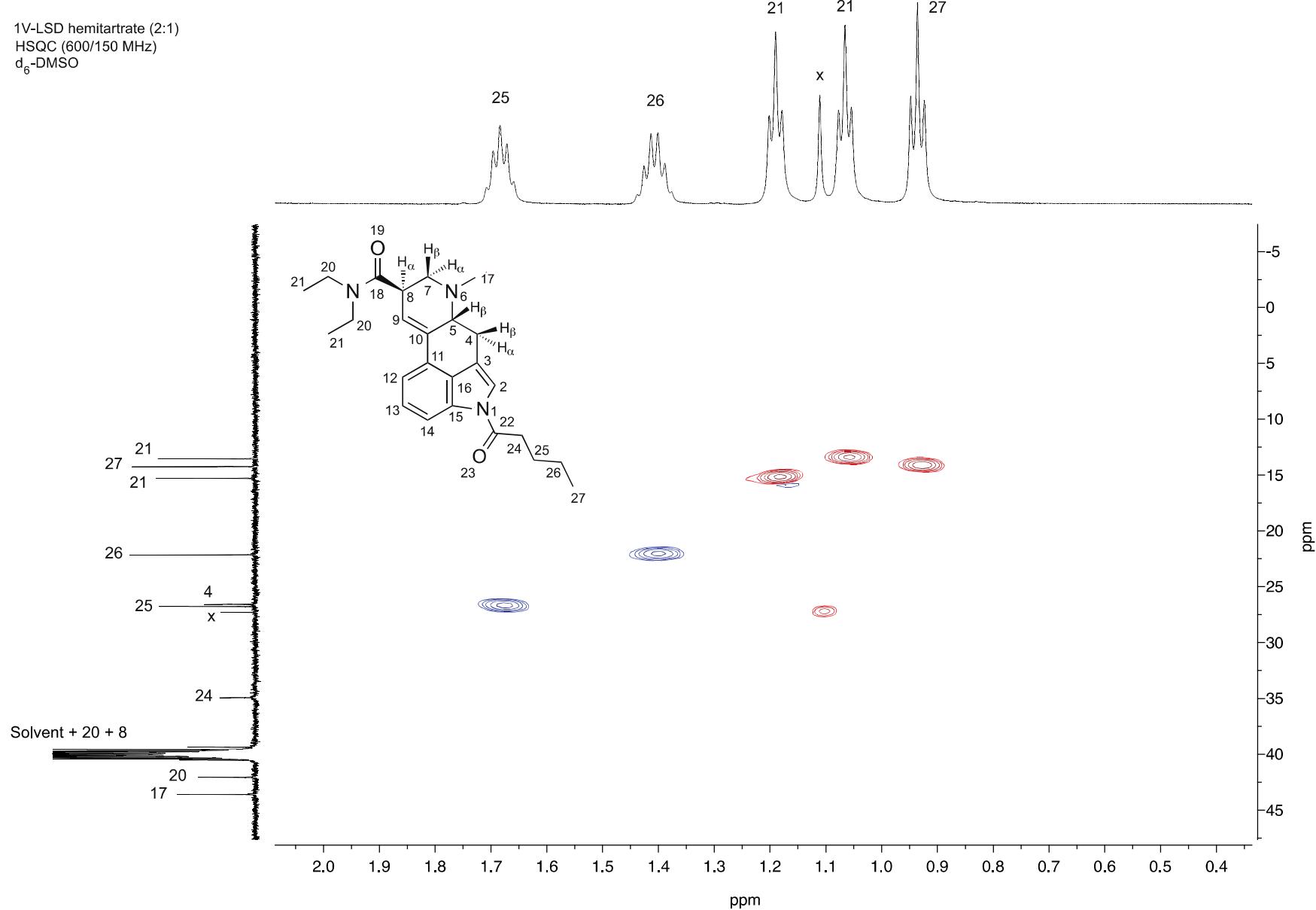


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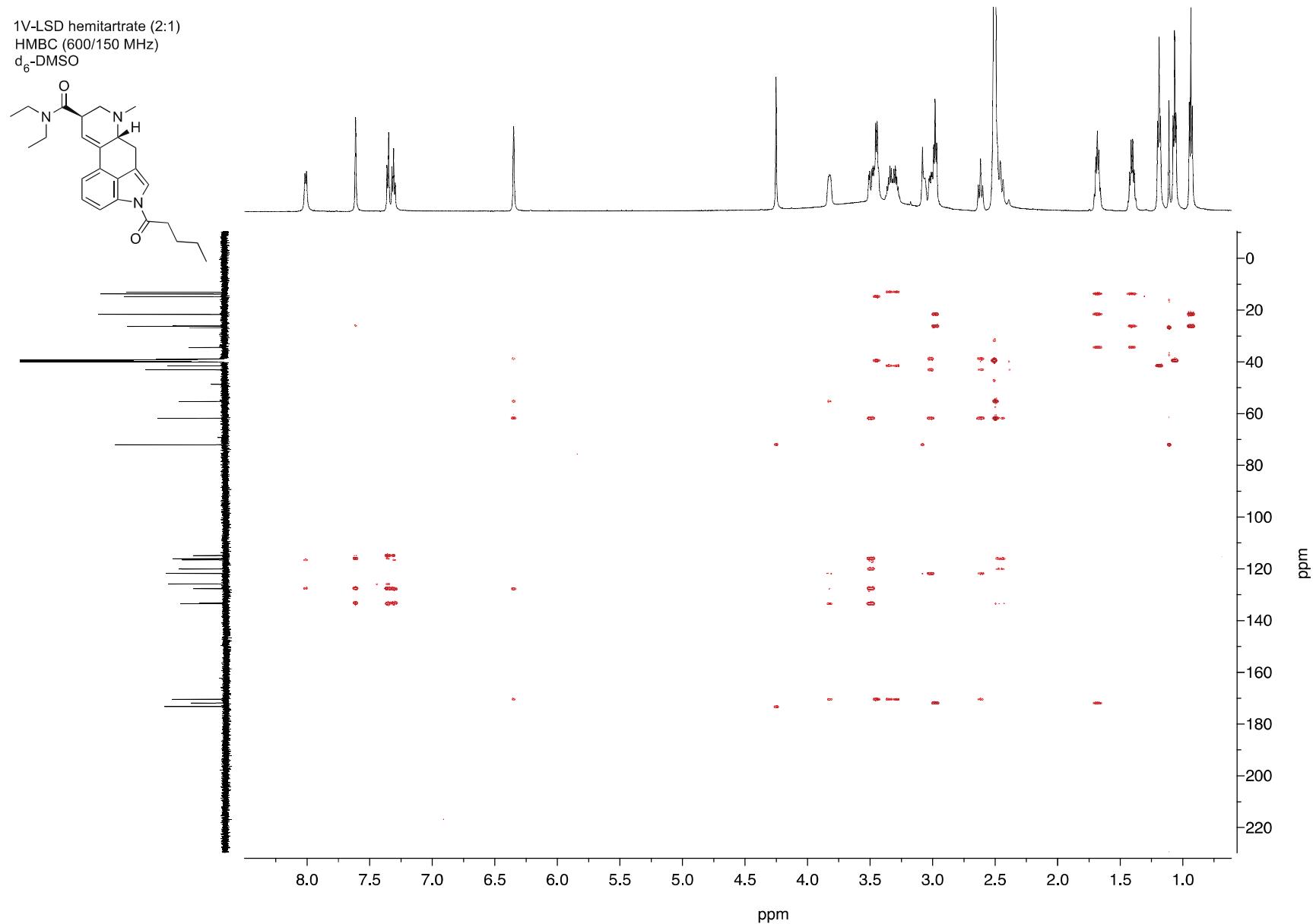


TA = Tartaric acid

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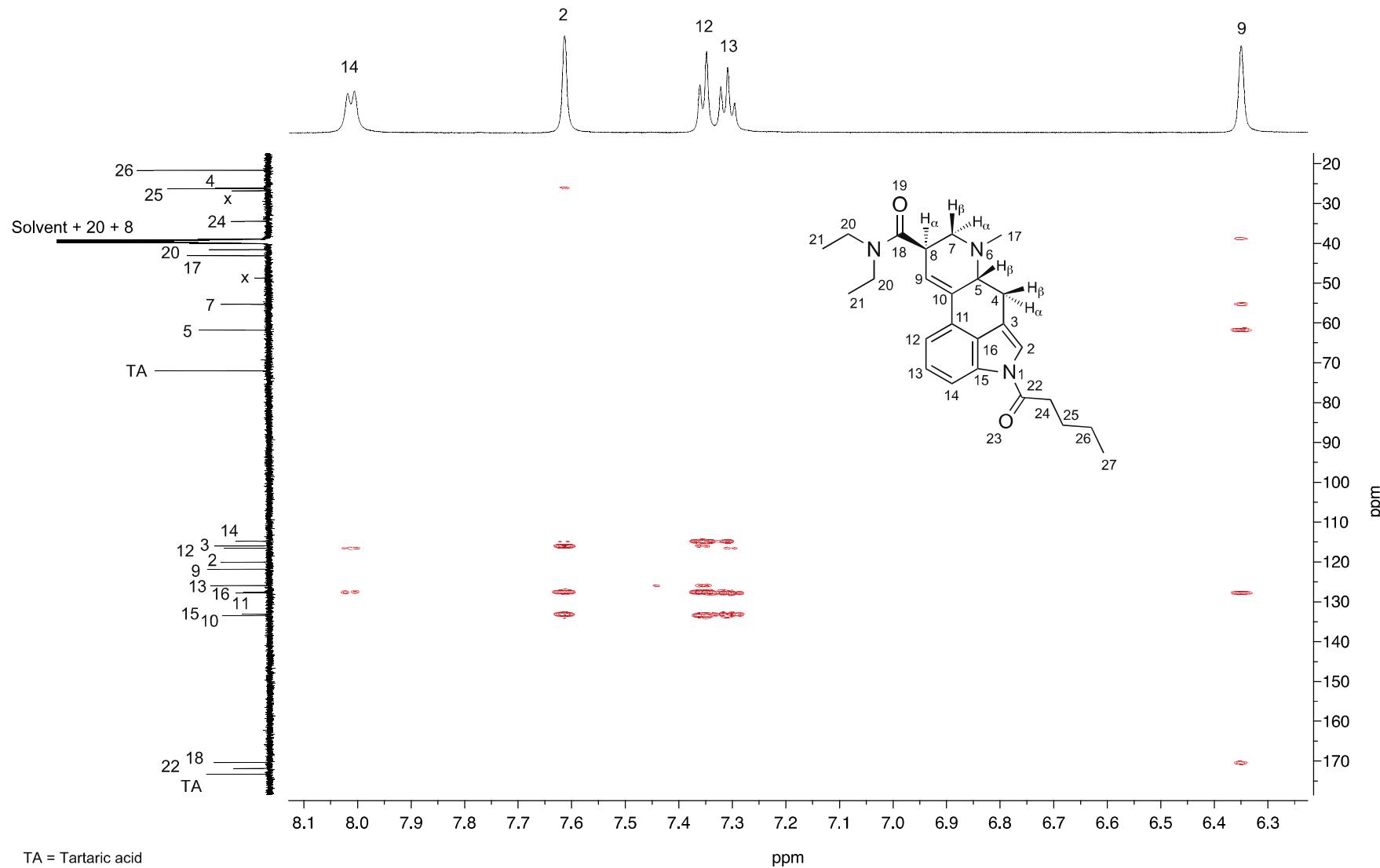


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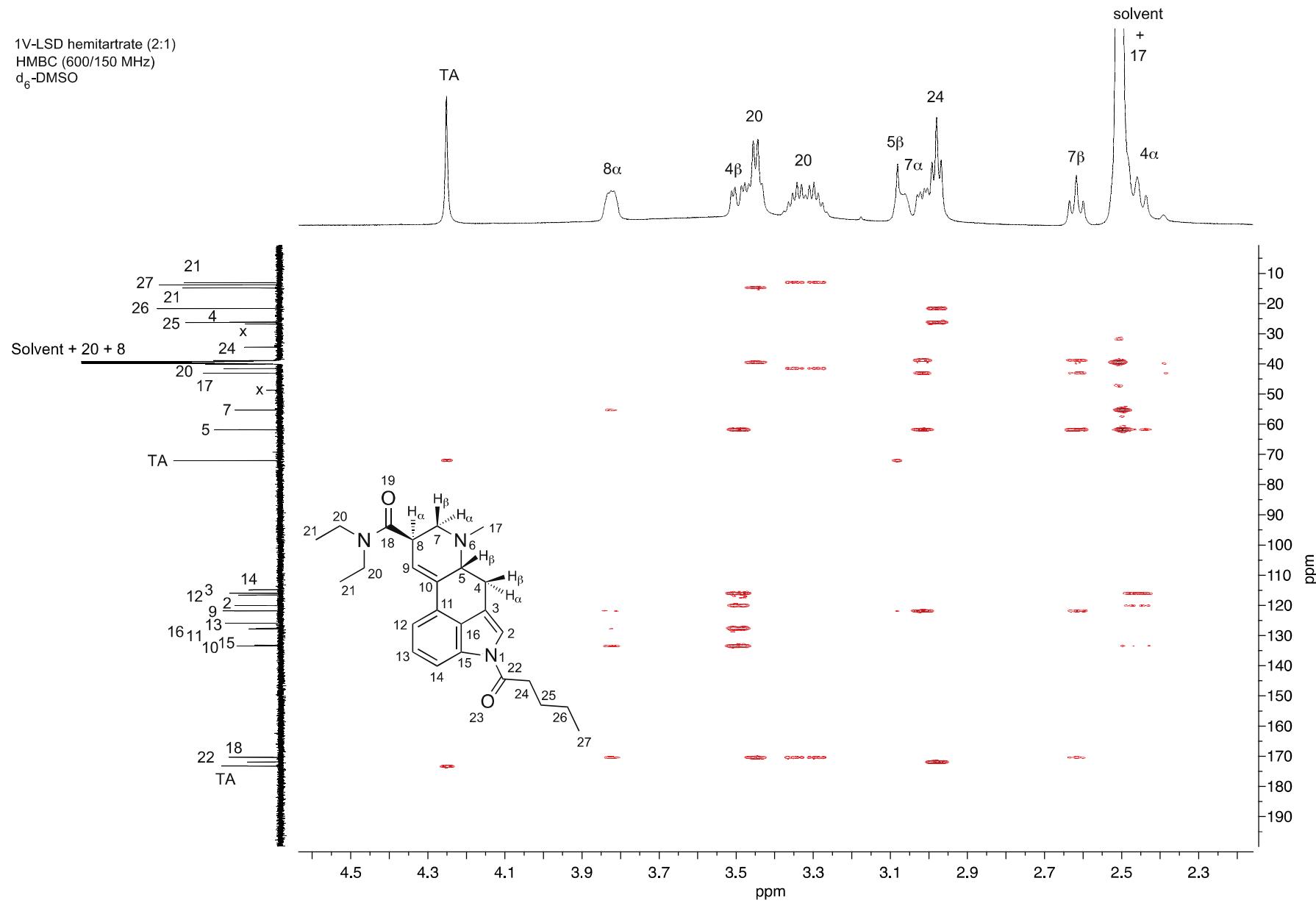


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1V-LSD hemitartrate (2:1)
HMBC (600/150 MHz)
 d_6 -DMSO

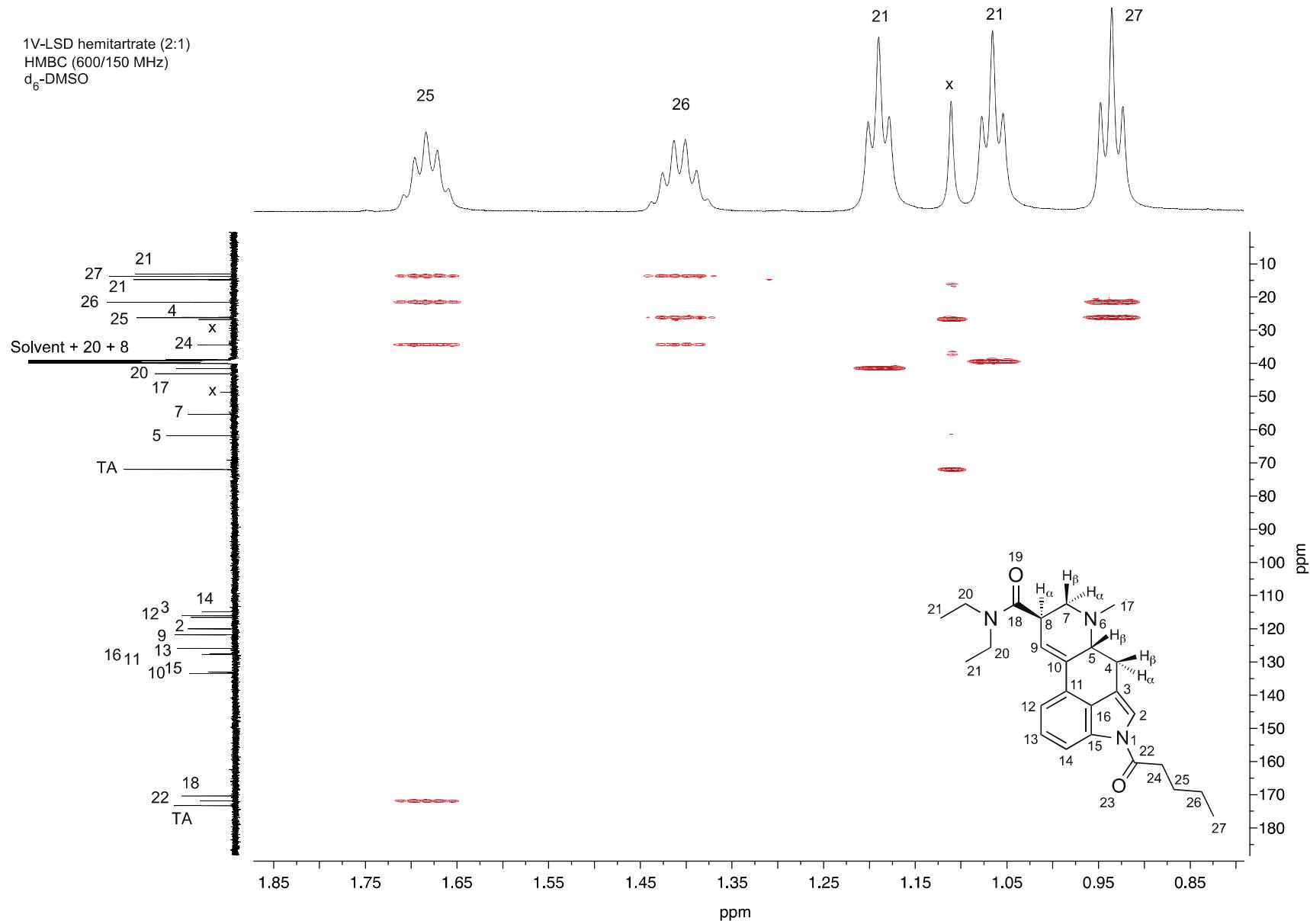


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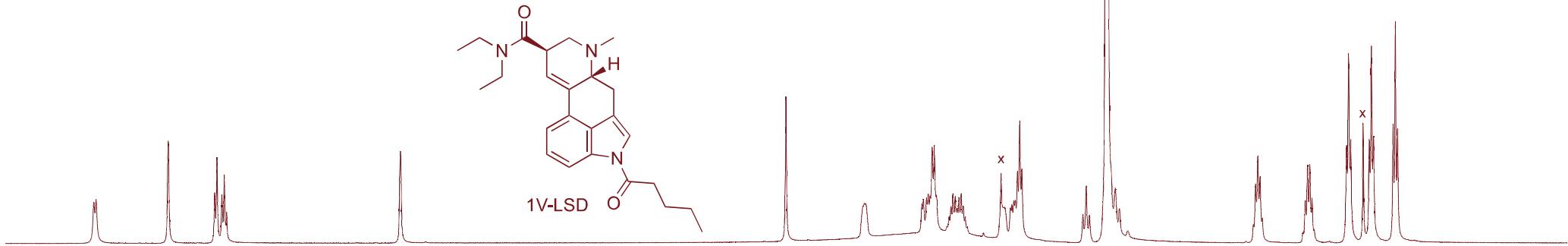
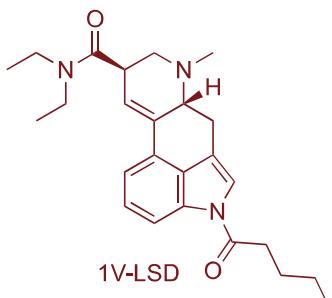
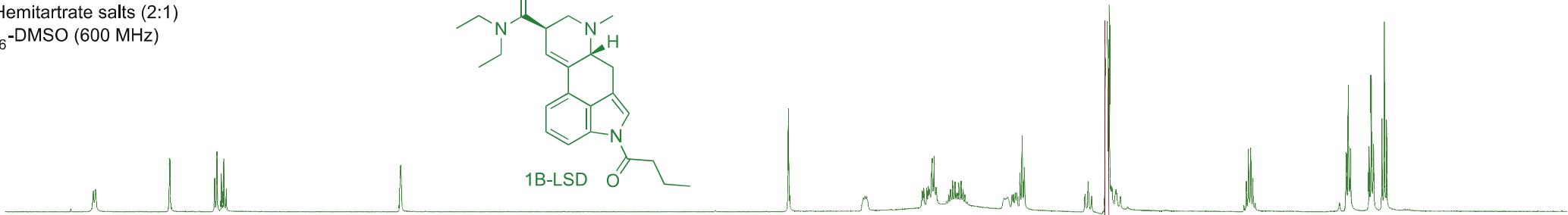
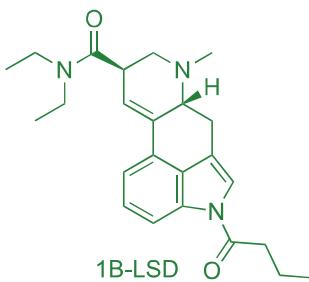
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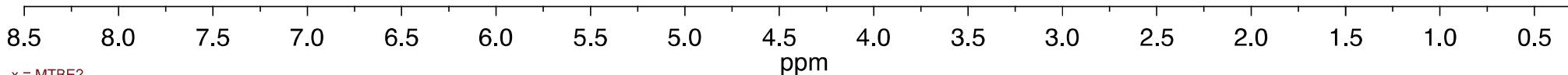


TA = Tartaric acid

Proton NMR
Hemitartrate salts (2:1)
 d_6 -DMSO (600 MHz)



x = MTBE?



Lysergamides - hemitartrate salts (2:1)
Carbon NMR (150 MHz)
DMSO-d₆

