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Return of the lysergamides. Part I: Analytical and behavioural characterization of 1-propionyl-d-lysergic acid diethylamide (1P-LSD).

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Return of the lysergamides. Part I: Analytical and behavioral characterization of 1-propionyl-*d*-lysergic acid diethylamide (1P-LSD)

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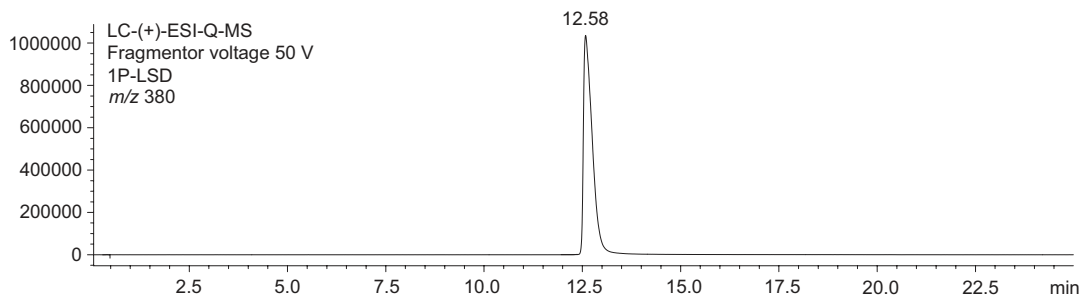
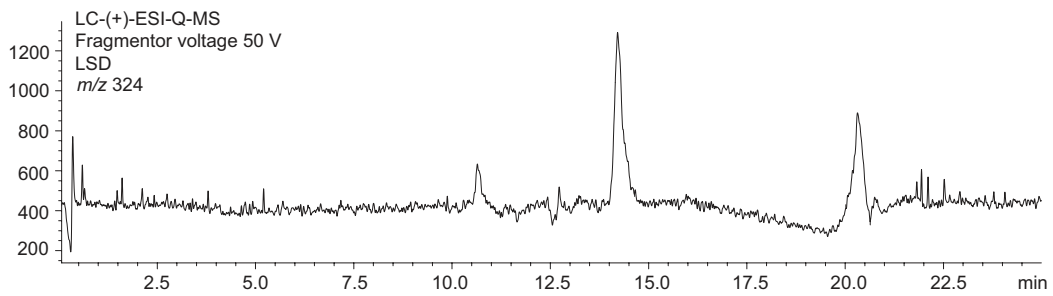
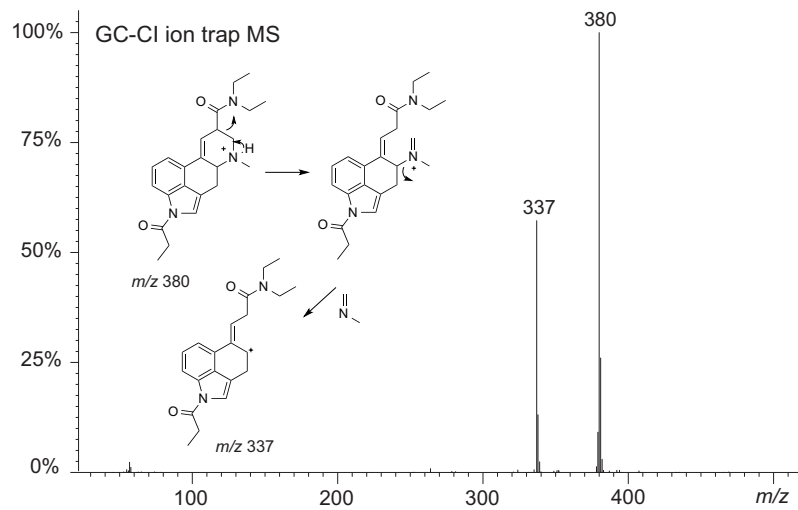
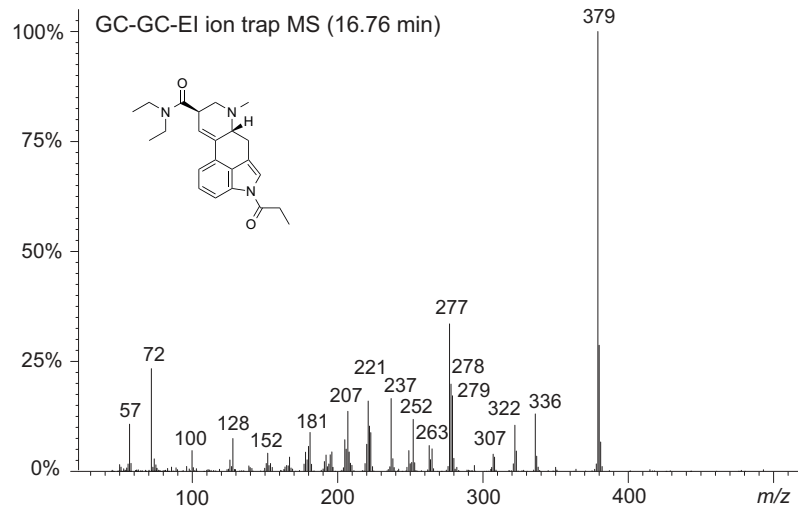
^f Department of Chemistry and Biochemistry, University of the Sciences, Philadelphia, PA 19104, USA

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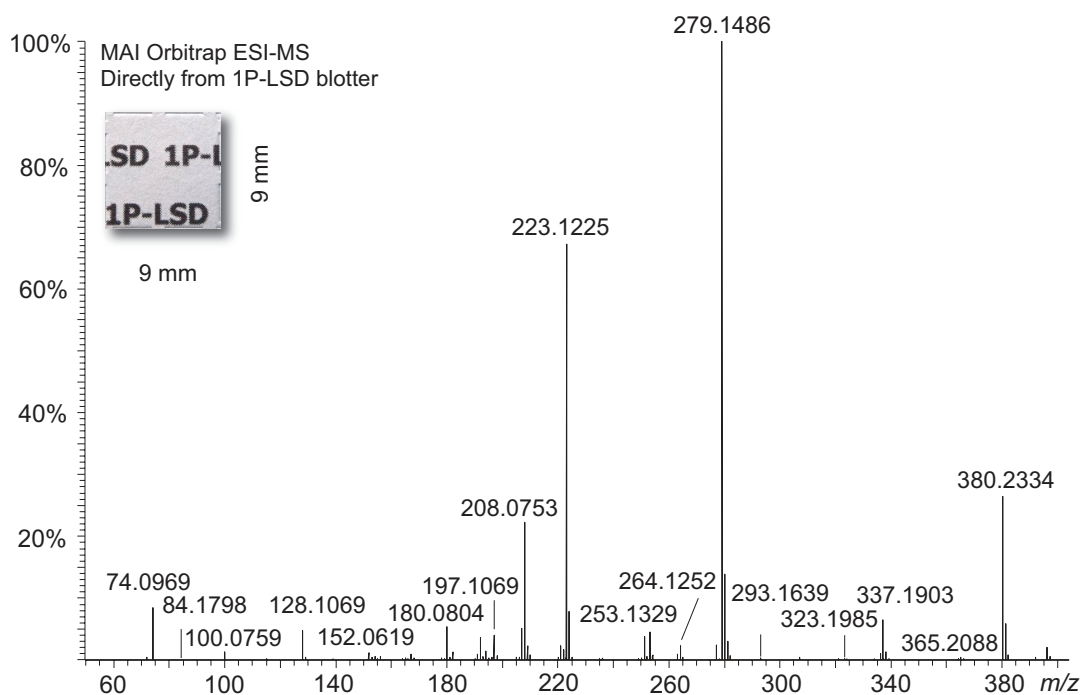
^h Department of Psychiatry, University of California San Diego, La Jolla, CA 92093-0804, USA

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* 1P-LSD = hemitartrate salt (powder); LSD = tartrate salt (powder).	



GC-MS (EI/CI) and LC-single quadrupole MS trace of 1P-LSD



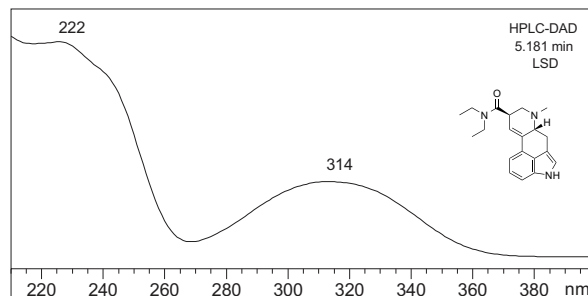
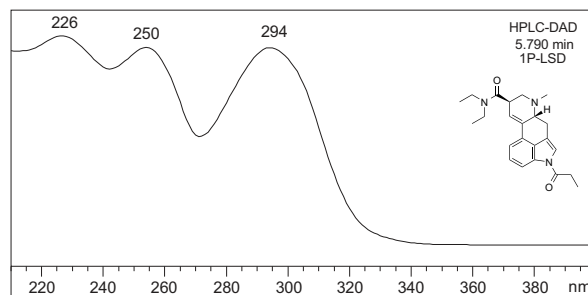
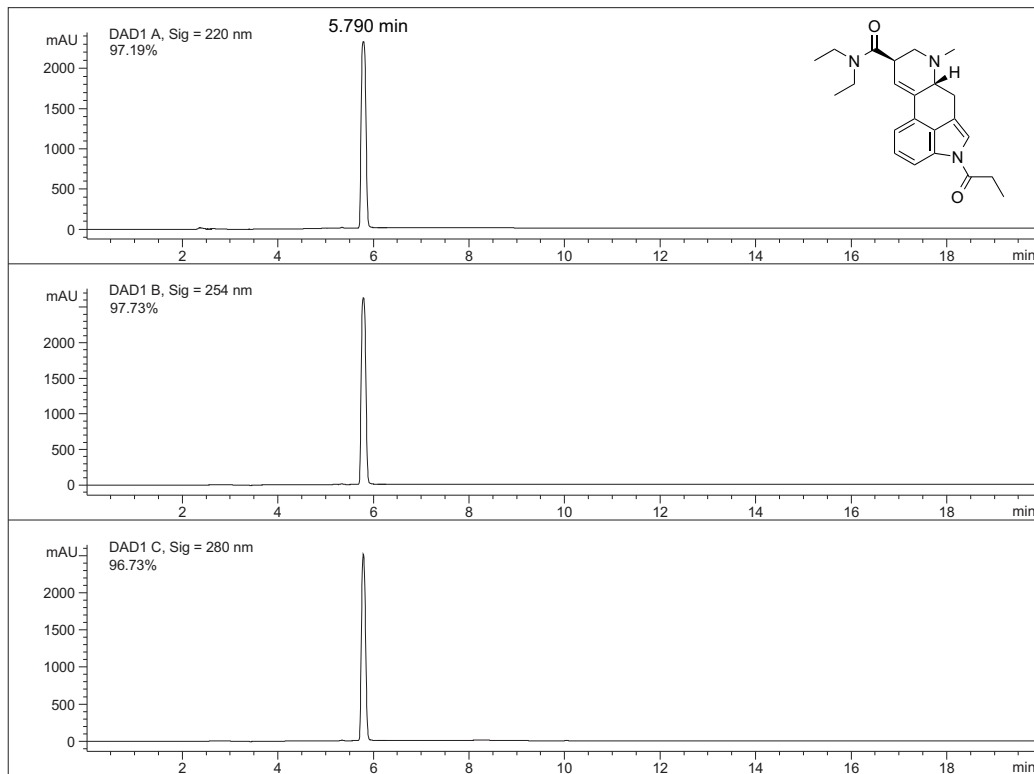
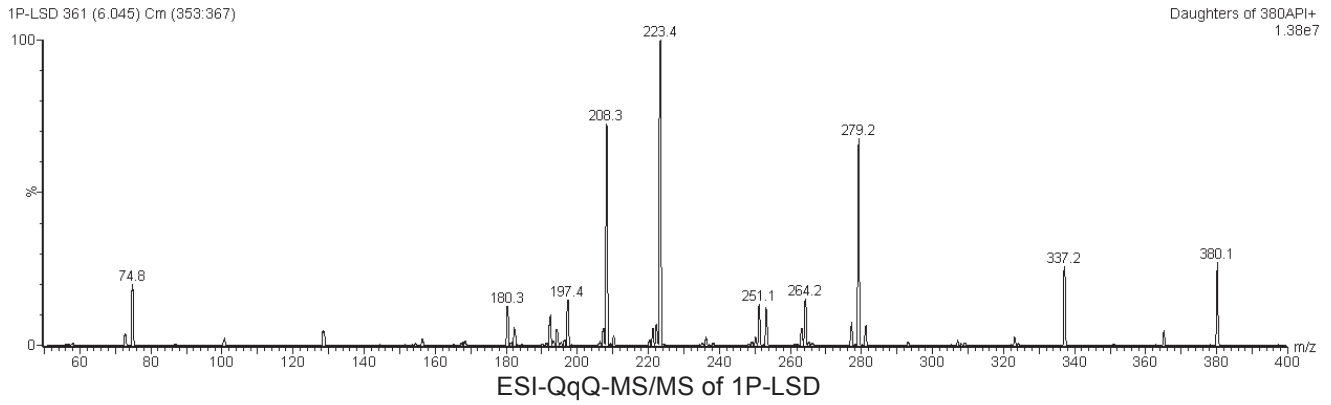
Matrix assisted ionization mass spectrum (MAI-MS) of 1P-LSD obtained from direct analysis of a 1P-LSD blotter.

Matrix assisted ionization mass spectrometry (MAI-MS)

A Thermo Scientific Exactive™ mass spectrometer (Thermo Fisher Scientific, Bremen, Germany) was modified by removing the Ion Max source to expose the inlet capillary for sample introduction using glass slides. The trap fill time was set at 1000 ms to correspond with the 1 s required to achieve a resolution of 100,000 (50% FWHH, m/z 200). The sheath, auxiliary, and sweep gas flow rates, as well as the electrospray ionization spray voltage, were set to zero. The inlet capillary temperature was set at 70 °C. The capillary, tube lens and skimmer voltages were optimized at 30, 60, and 18 V, respectively, and acquisition time was set to continuous mode. The higher-energy collisional dissociation (HCD) parameter was set at 35 eV to induce dissociation.

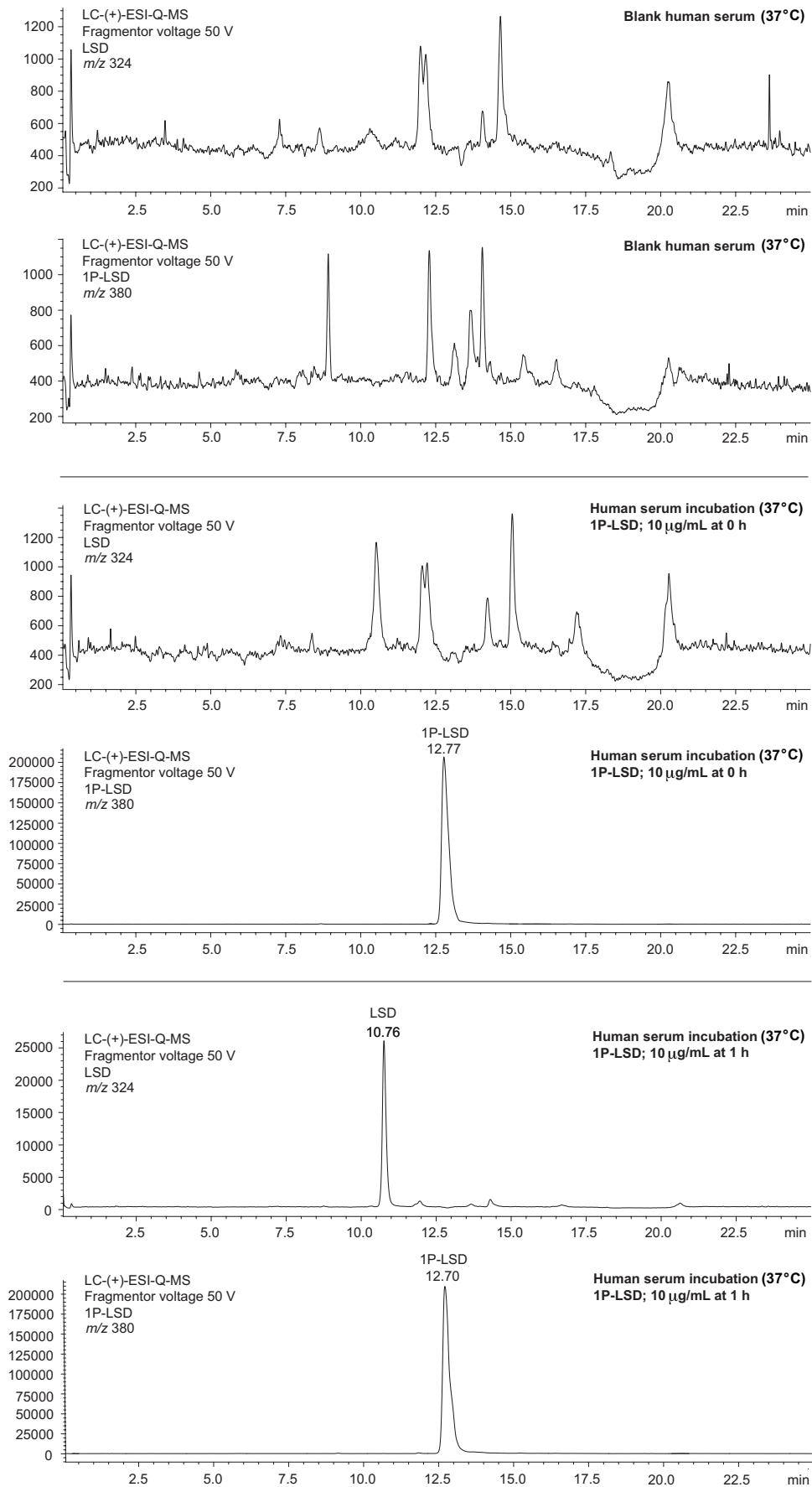
Blotter preparation for MAI-MS analysis

3-Nitrobenzotrile was used as the matrix and prepared at a 0.6 mg/mL concentration with acetonitrile/water (1:1). For direct MAI-MS analysis, 1 μ L of matrix solution was added onto the surface of the 1P-LSD blotter and allowed to air-dry.



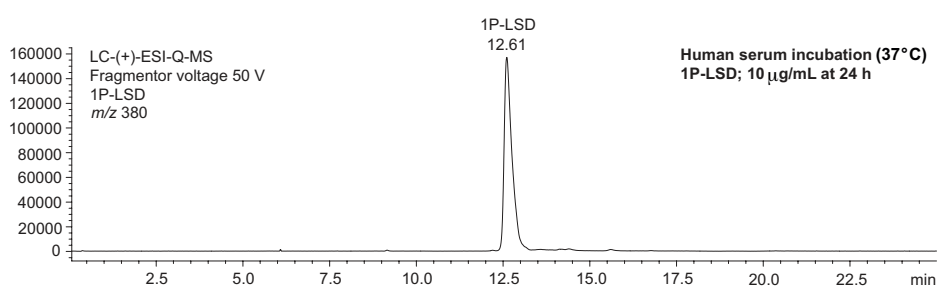
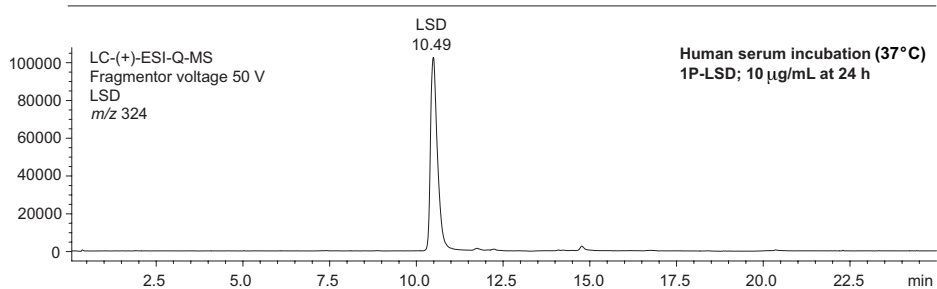
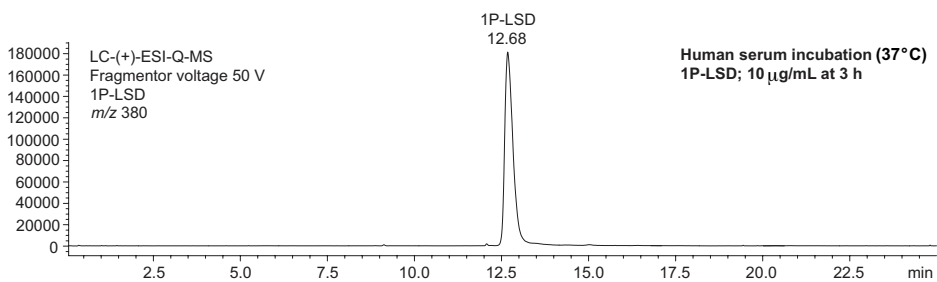
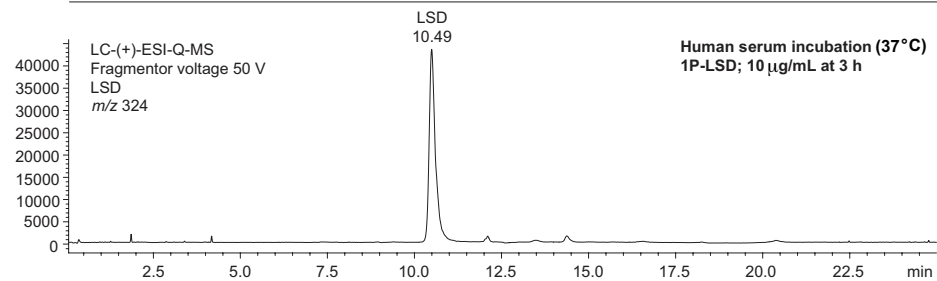
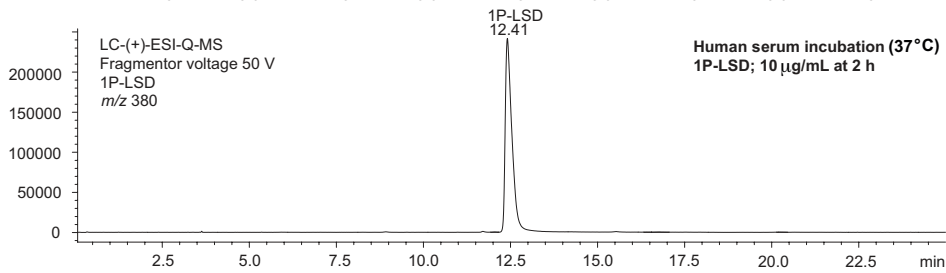
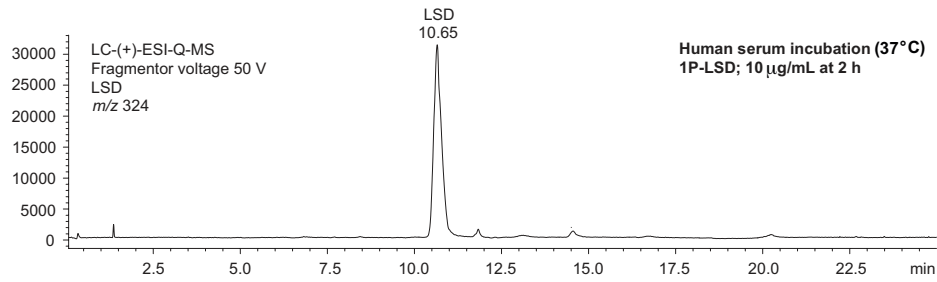
HPLC-UV (1P-LSD) traces and DAD spectra (1P-LSD vs. LSD)

Drug Testing and Analysis – Brandt *et al.* – Supplementary Information



Incubation of 1P-LSD in human serum at 37 °C and analysis of 1P-LSD vs. LSD using LC single quadrupole MS (blank serum, 0 h and 1h)

Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

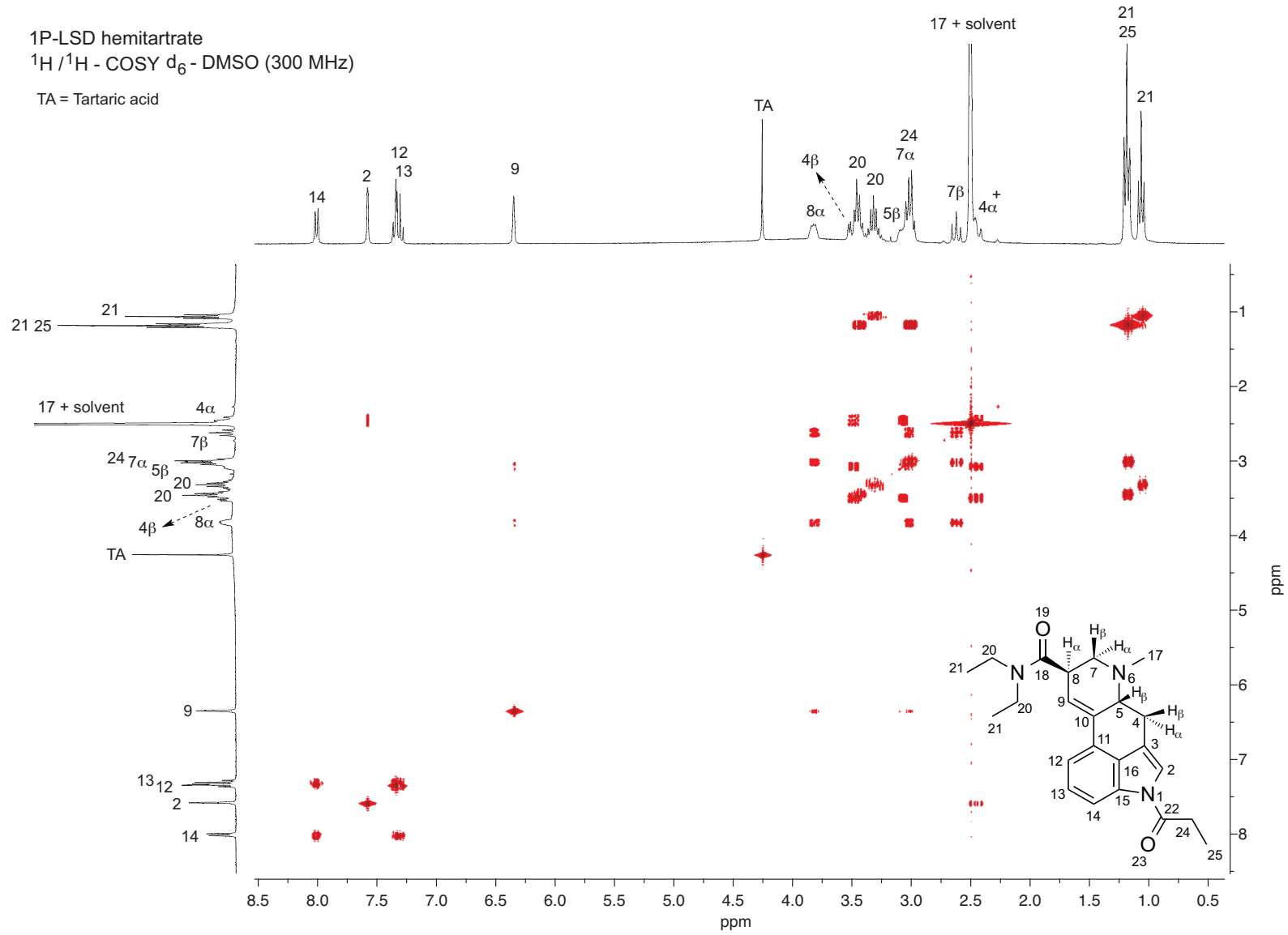


Incubation of 1P-LSD in human serum at 37 °C and analysis of 1P-LSD vs. LSD using LC single quadrupole MS (2 h, 3 h and 24h)

Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

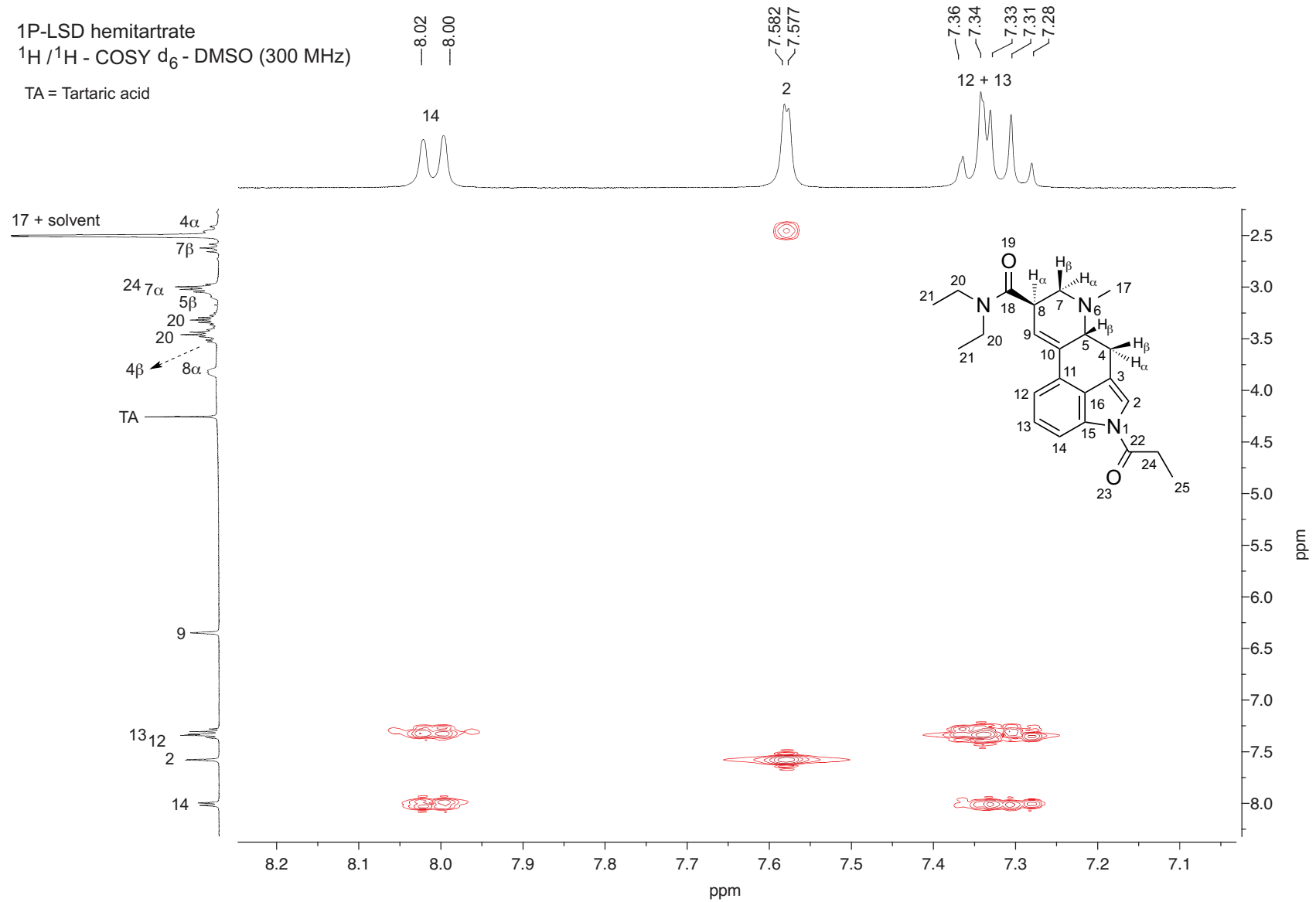
1P-LSD hemitartrate
 $^1\text{H}/^1\text{H}$ - COSY d_6 - DMSO (300 MHz)

TA = Tartaric acid

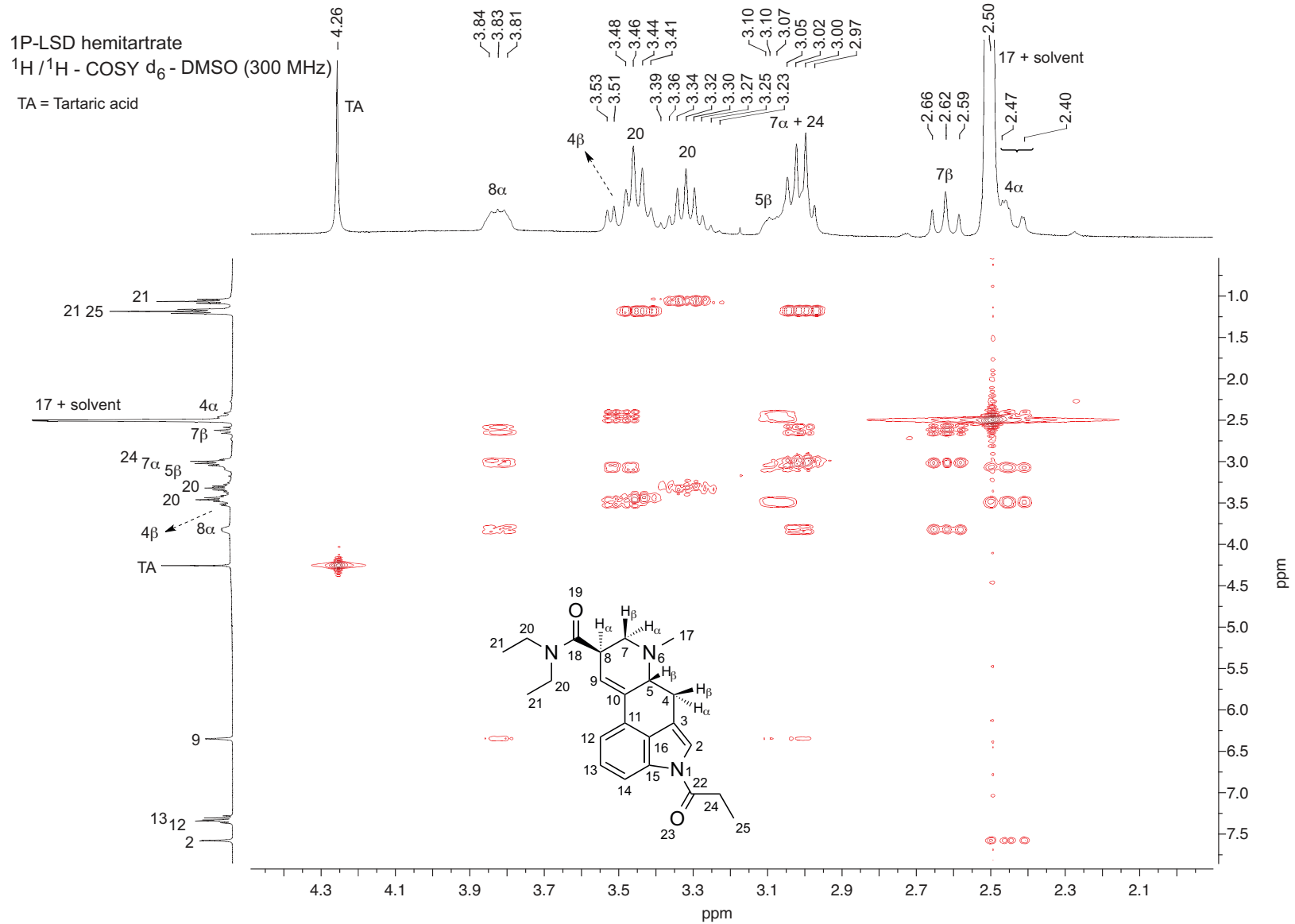


Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

1P-LSD hemitartrate
 $^1\text{H}/^1\text{H}$ - COSY d_6 -DMSO (300 MHz)
 TA = Tartaric acid



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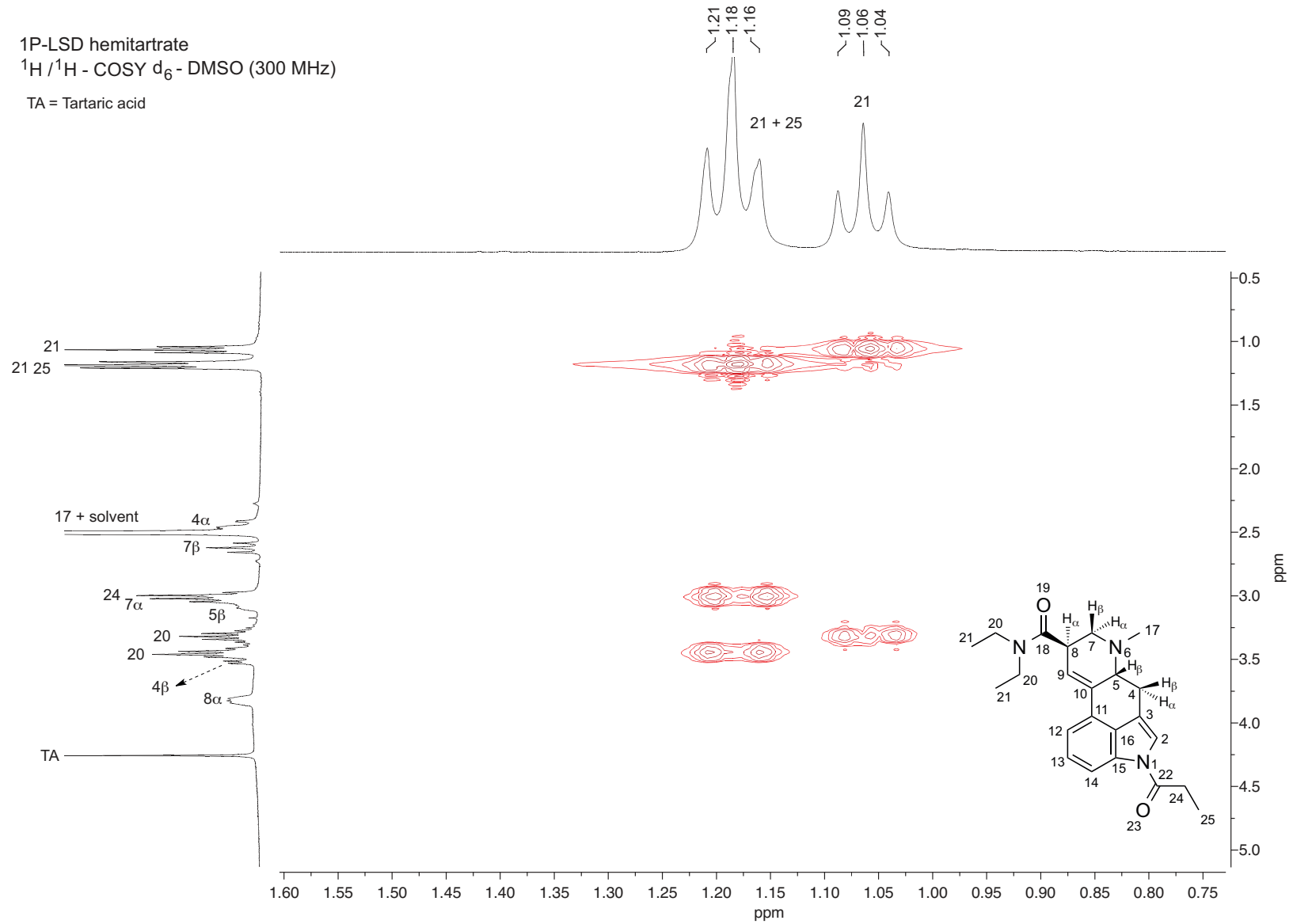


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1P-LSD hemitartrate

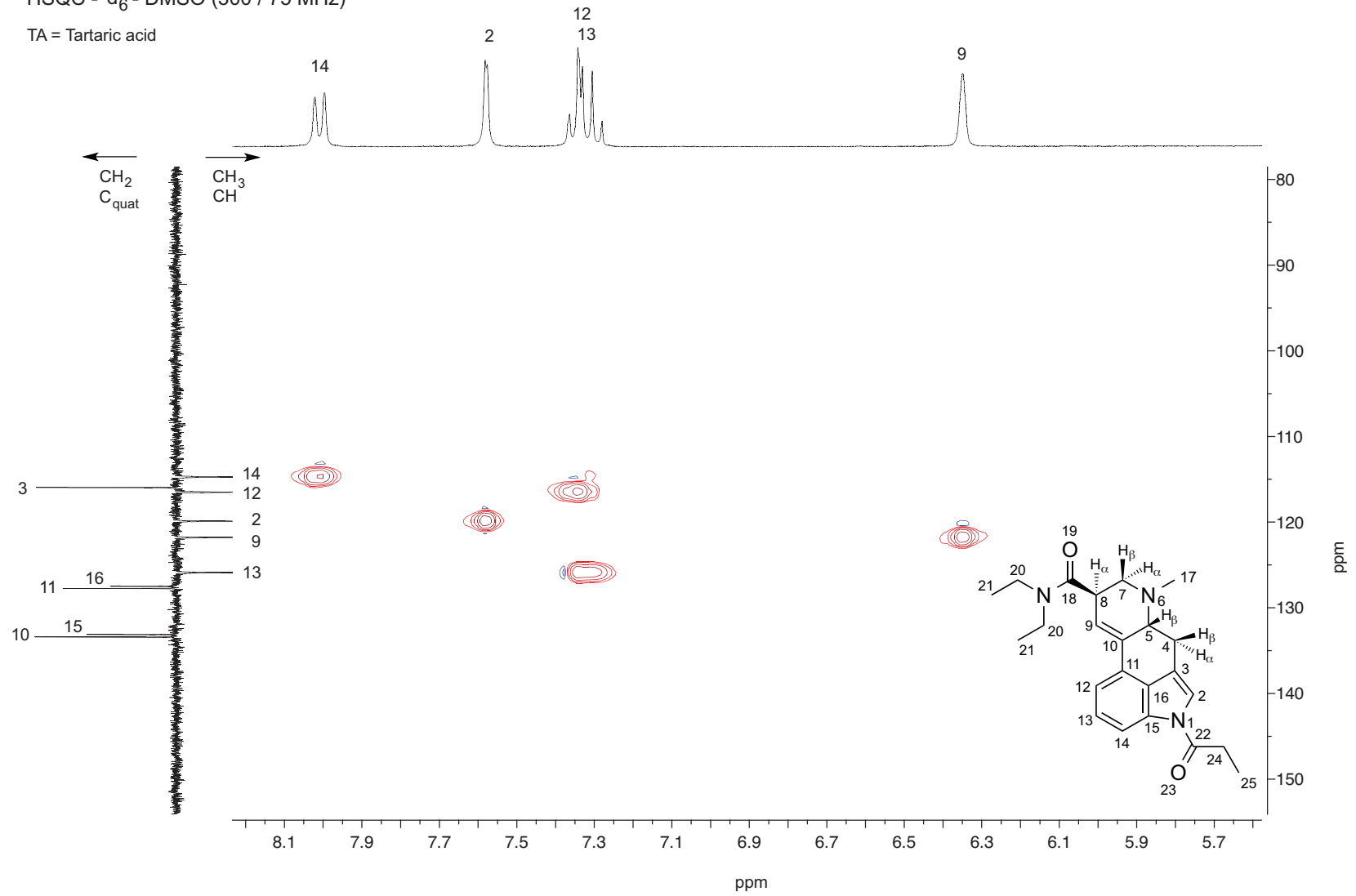
$^1\text{H}/^1\text{H}$ - COSY d_6 - DMSO (300 MHz)

TA = Tartaric acid



1P-LSD hemitartrate
 HSQC - d₆-DMSO (300 / 75 MHz)

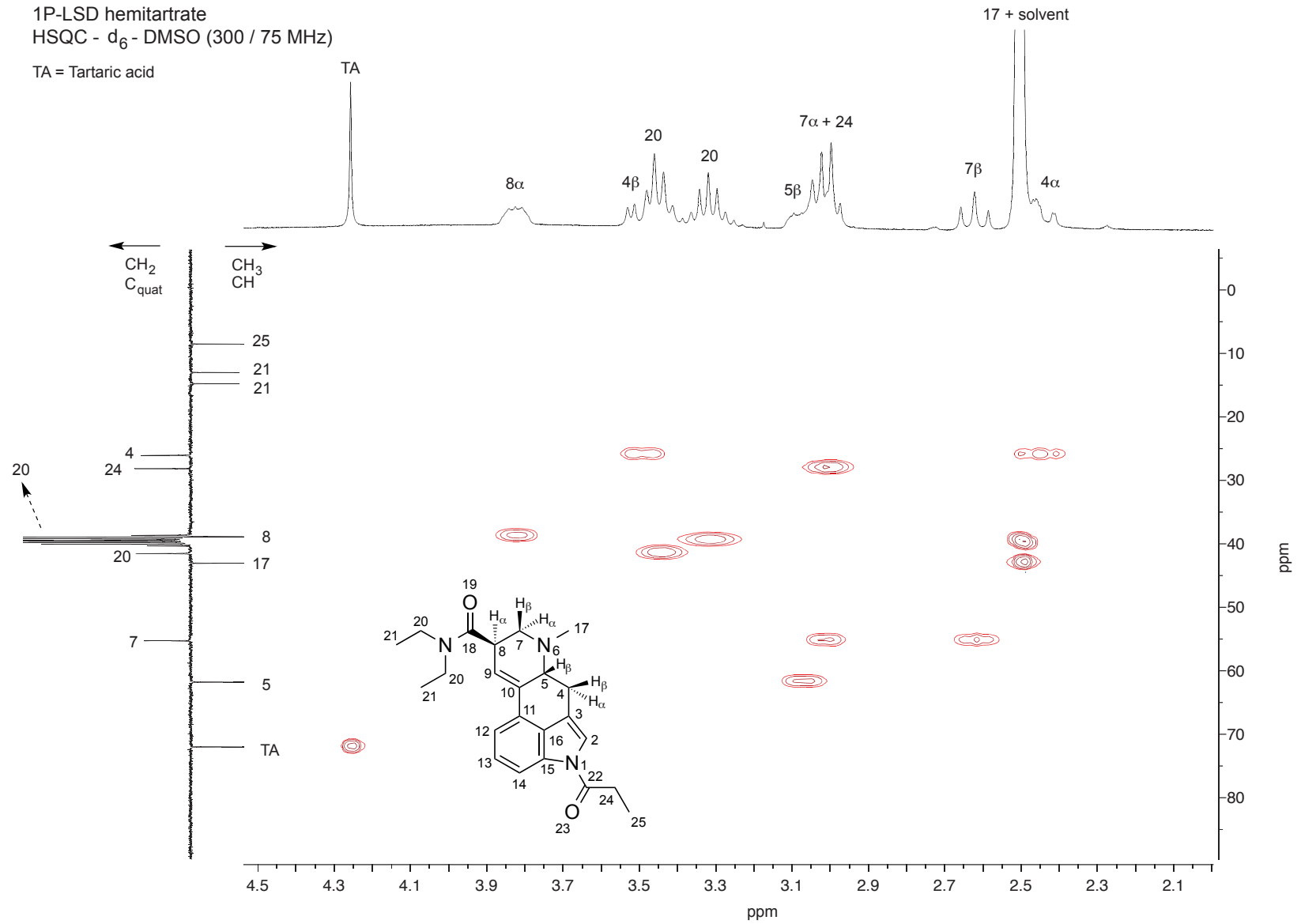
TA = Tartaric acid



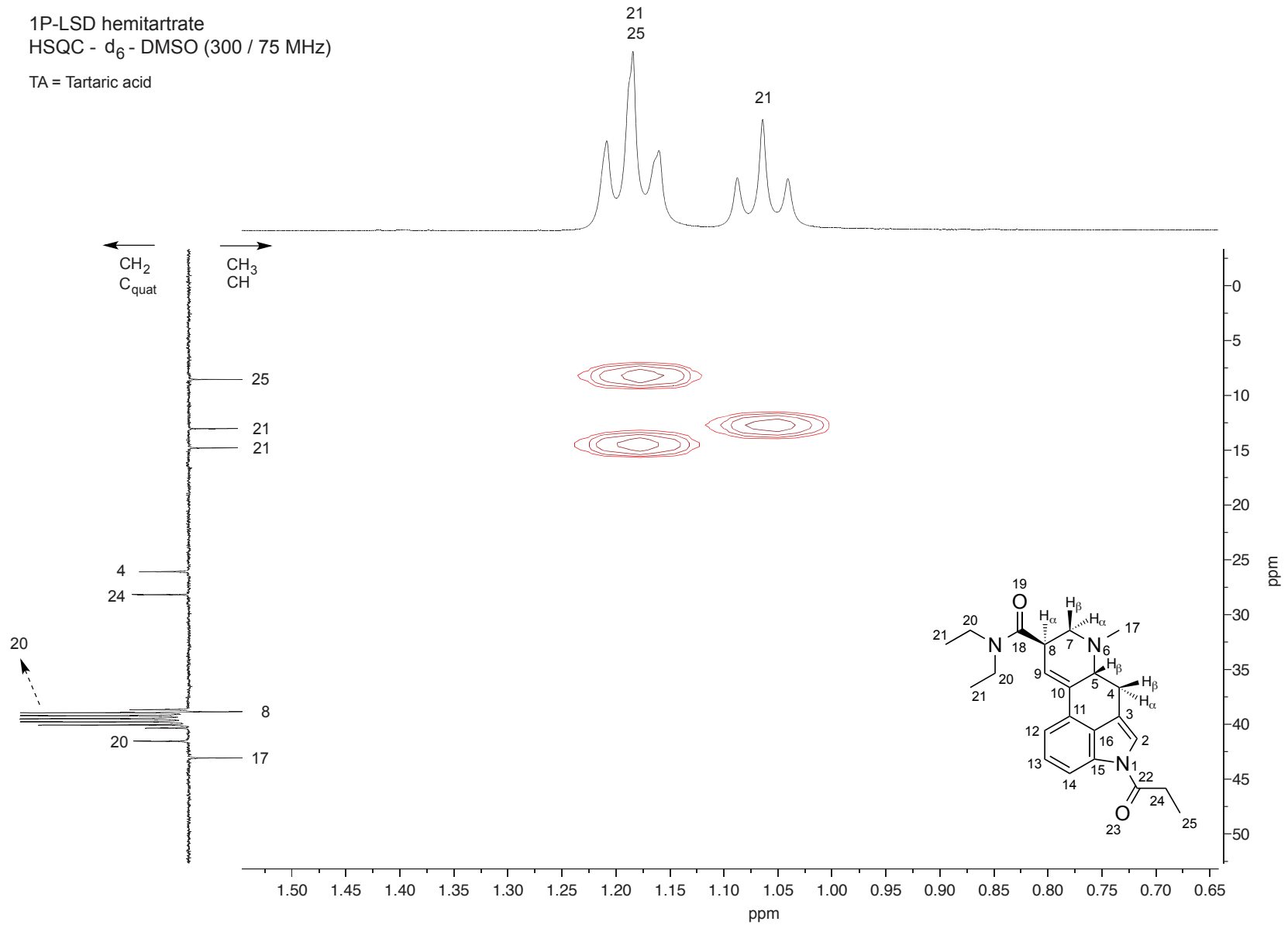
Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

1P-LSD hemitartrate
 HSQC - d₆ - DMSO (300 / 75 MHz)

TA = Tartaric acid

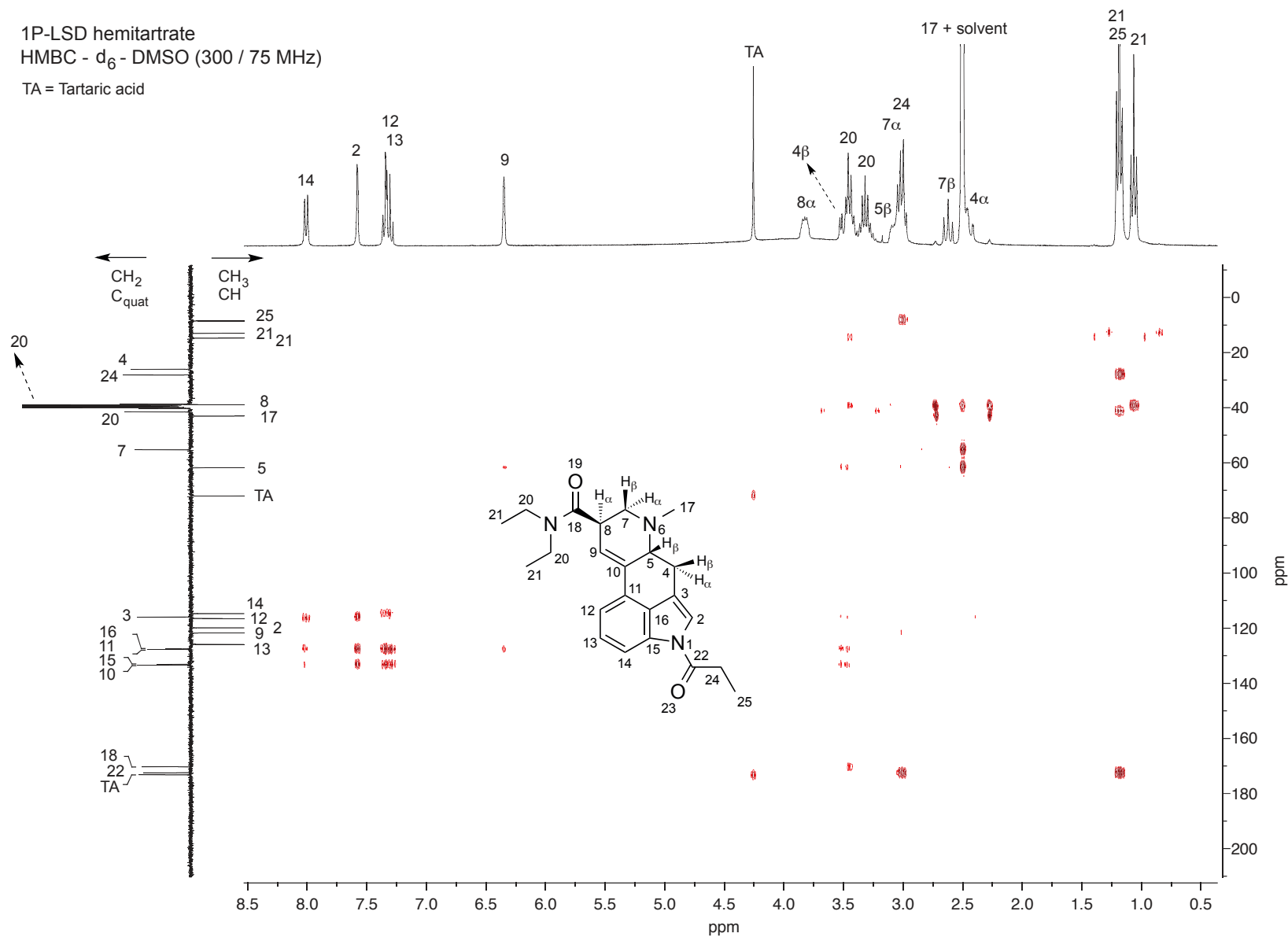


1P-LSD hemitartrate
 HSQC - d₆-DMSO (300 / 75 MHz)
 TA = Tartaric acid



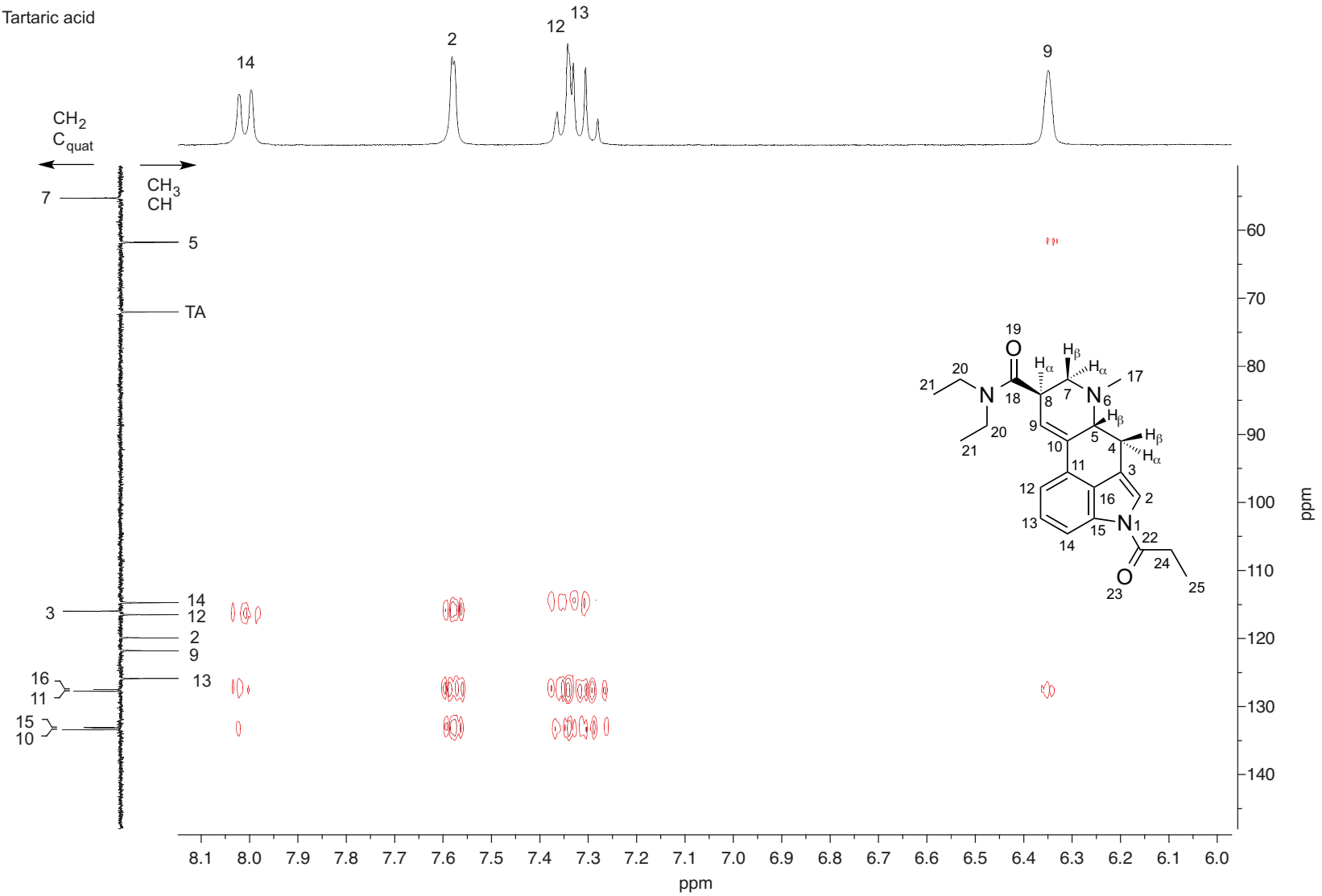
Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

1P-LSD hemitartrate
 HMBC - d₆ - DMSO (300 / 75 MHz)
 TA = Tartaric acid



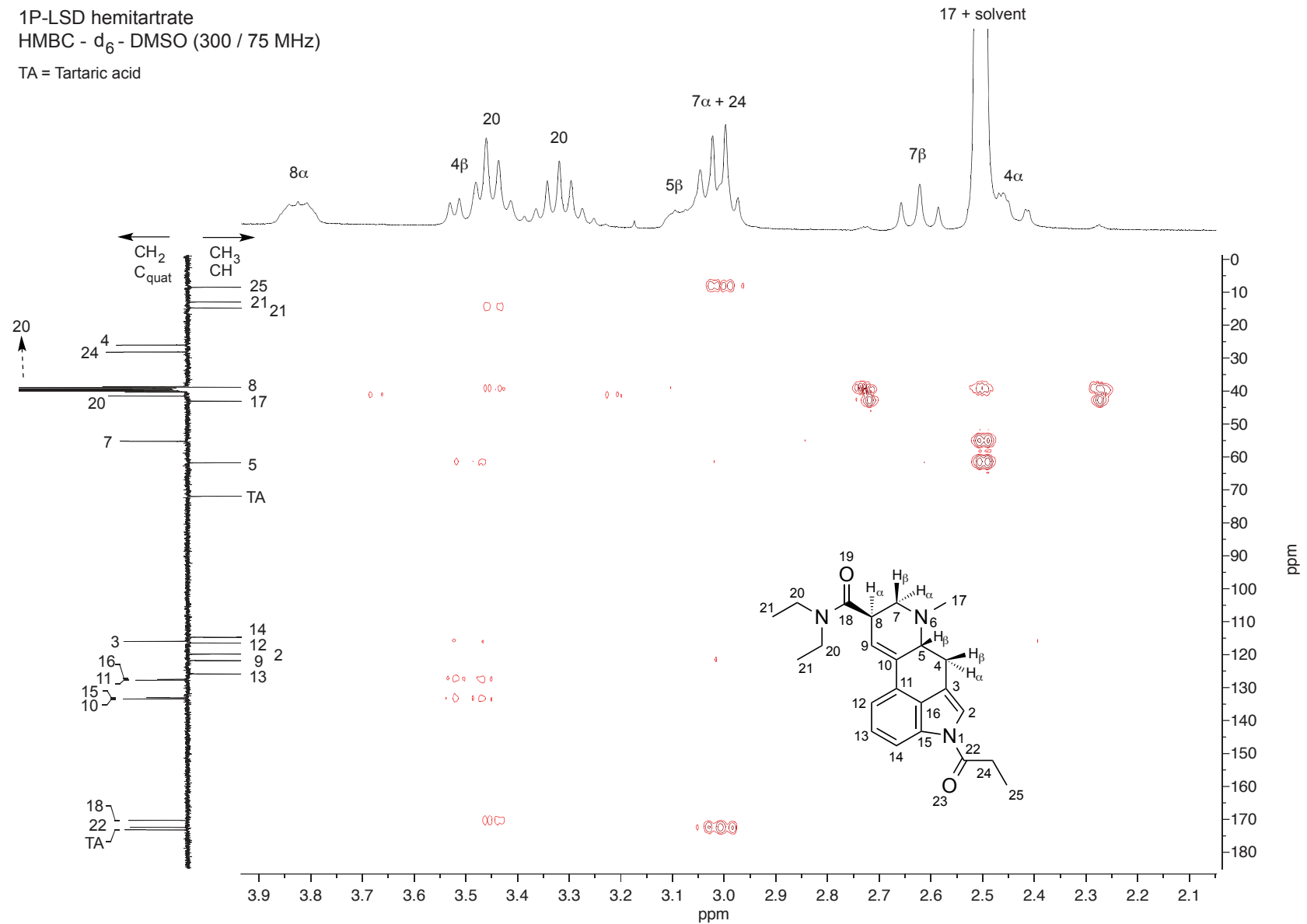
1P-LSD hemitartrate
 HMBC - d₆ - DMSO (300 / 75 MHz)

TA = Tartaric acid



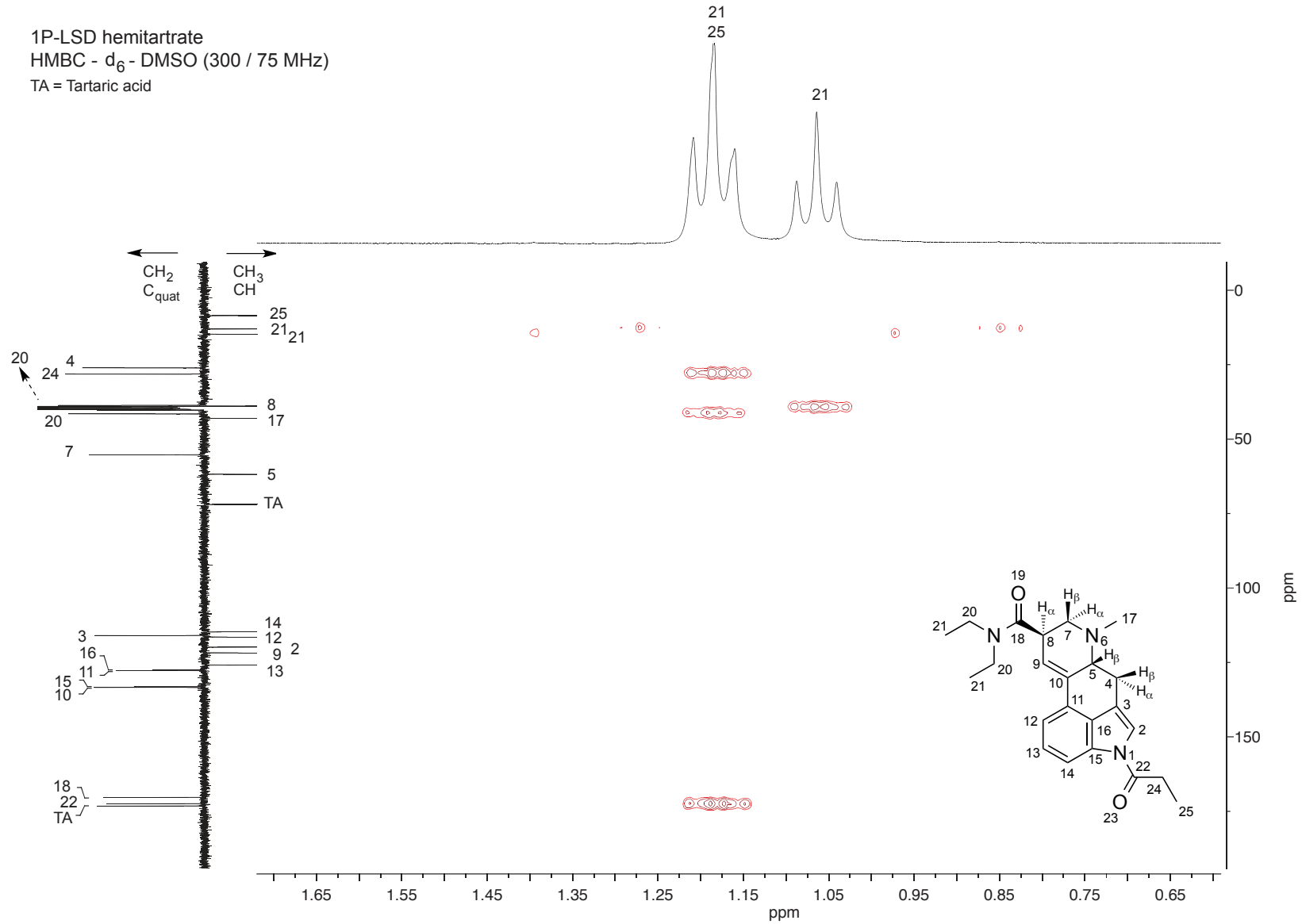
Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

1P-LSD hemitartrate
 HMBC - d₆ - DMSO (300 / 75 MHz)
 TA = Tartaric acid

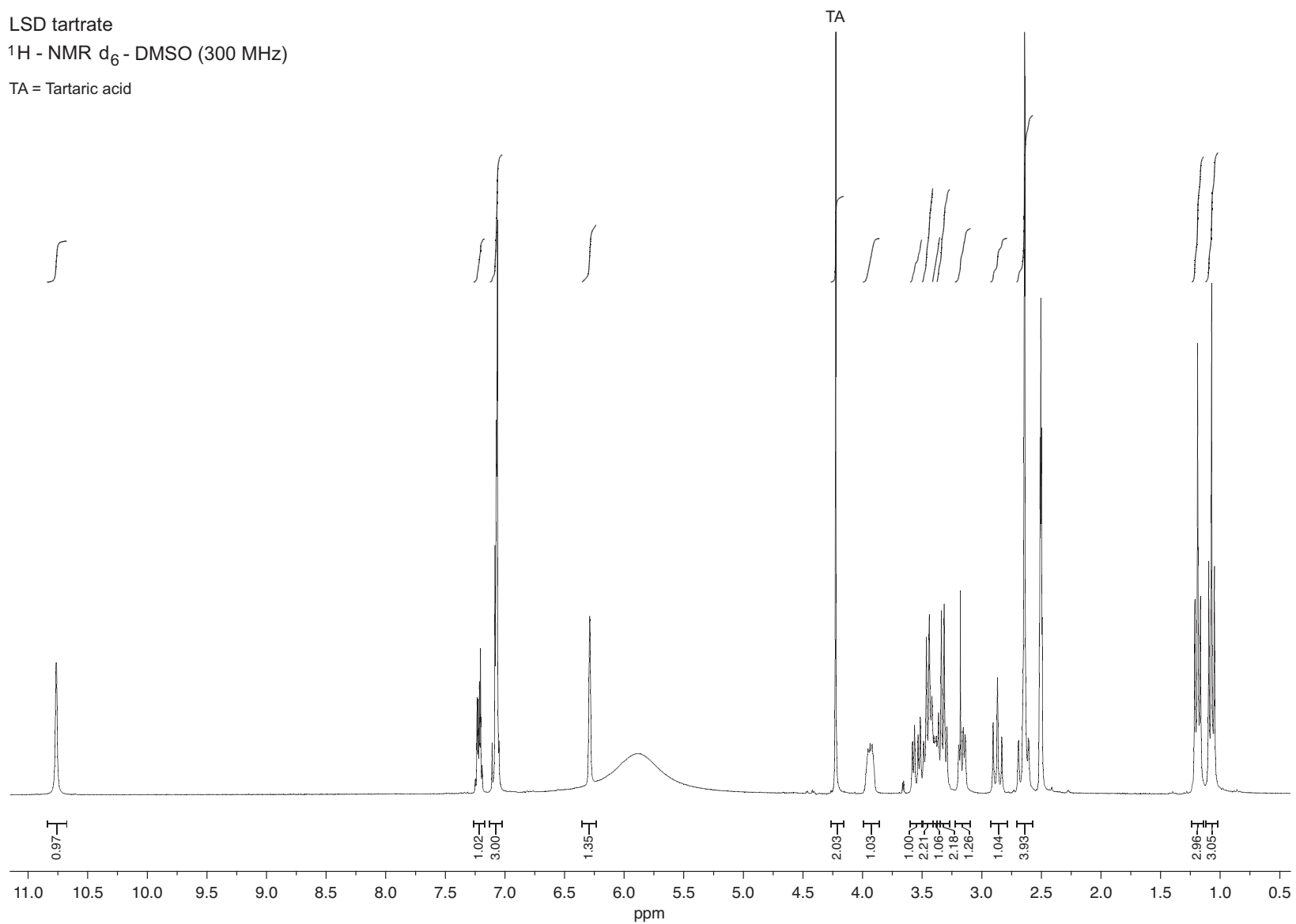


Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

1P-LSD hemitartrate
 HMBC - d₆ - DMSO (300 / 75 MHz)
 TA = Tartaric acid

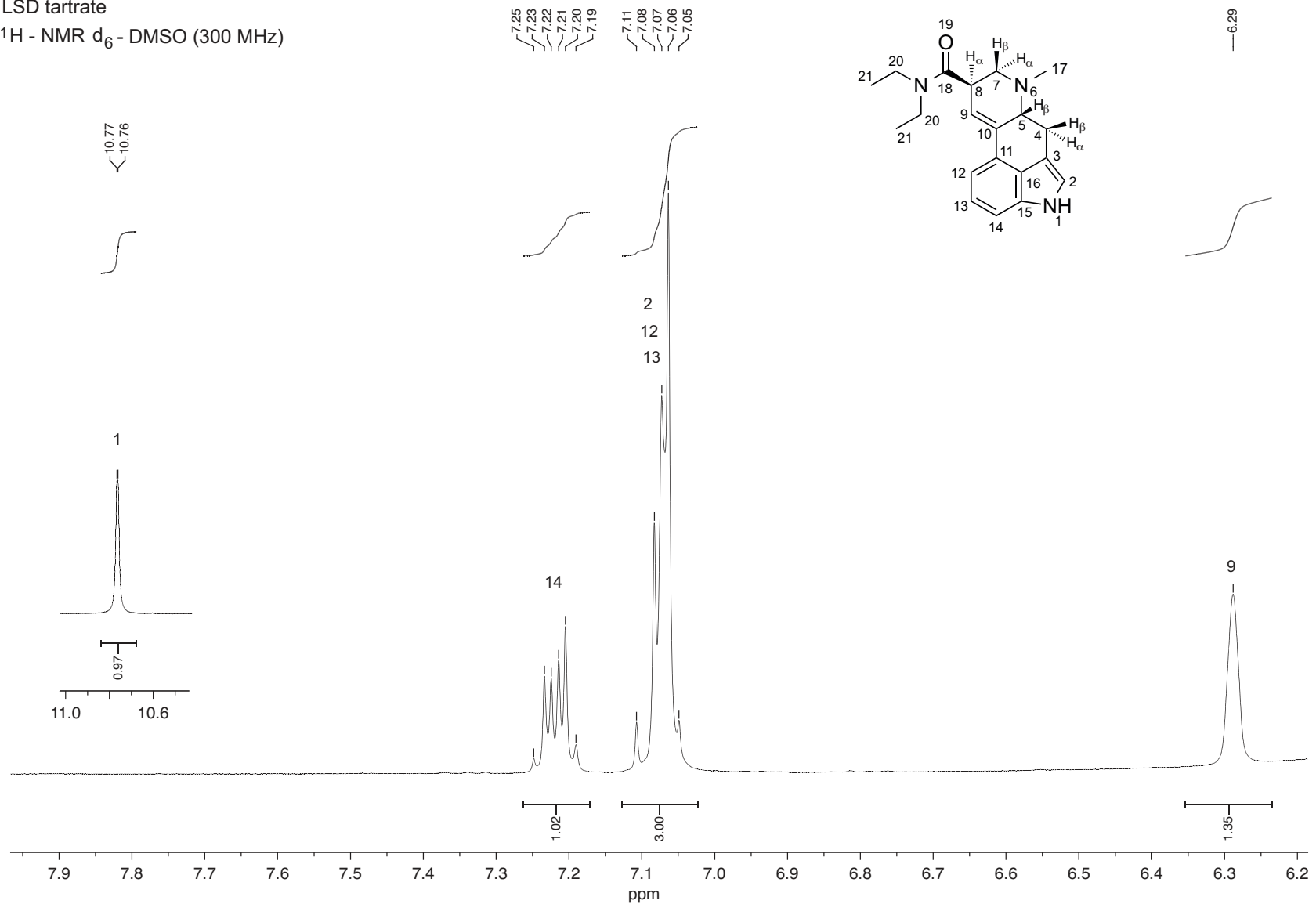


LSD tartrate
 ^1H - NMR d_6 - DMSO (300 MHz)
TA = Tartaric acid

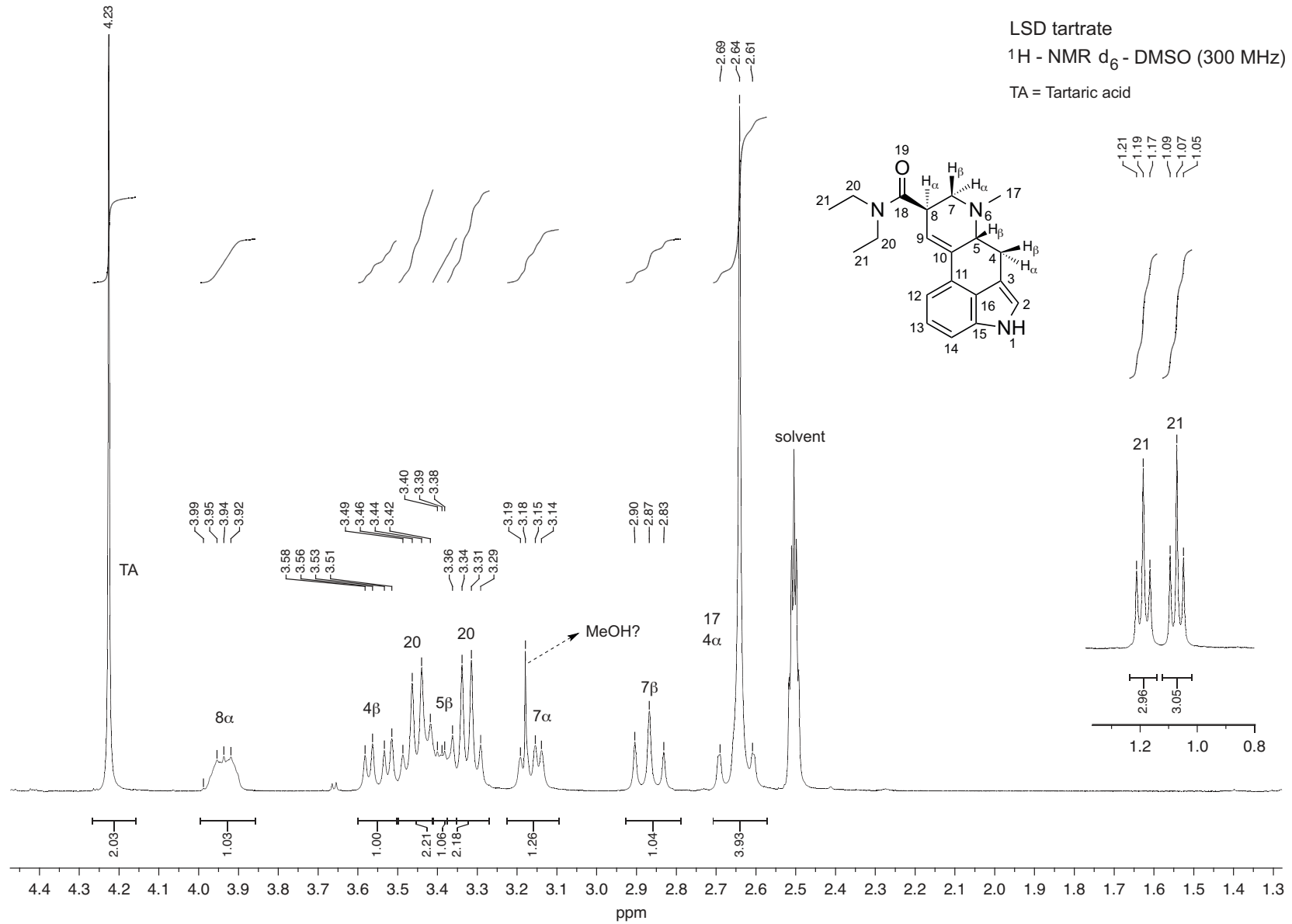


LSD tartrate

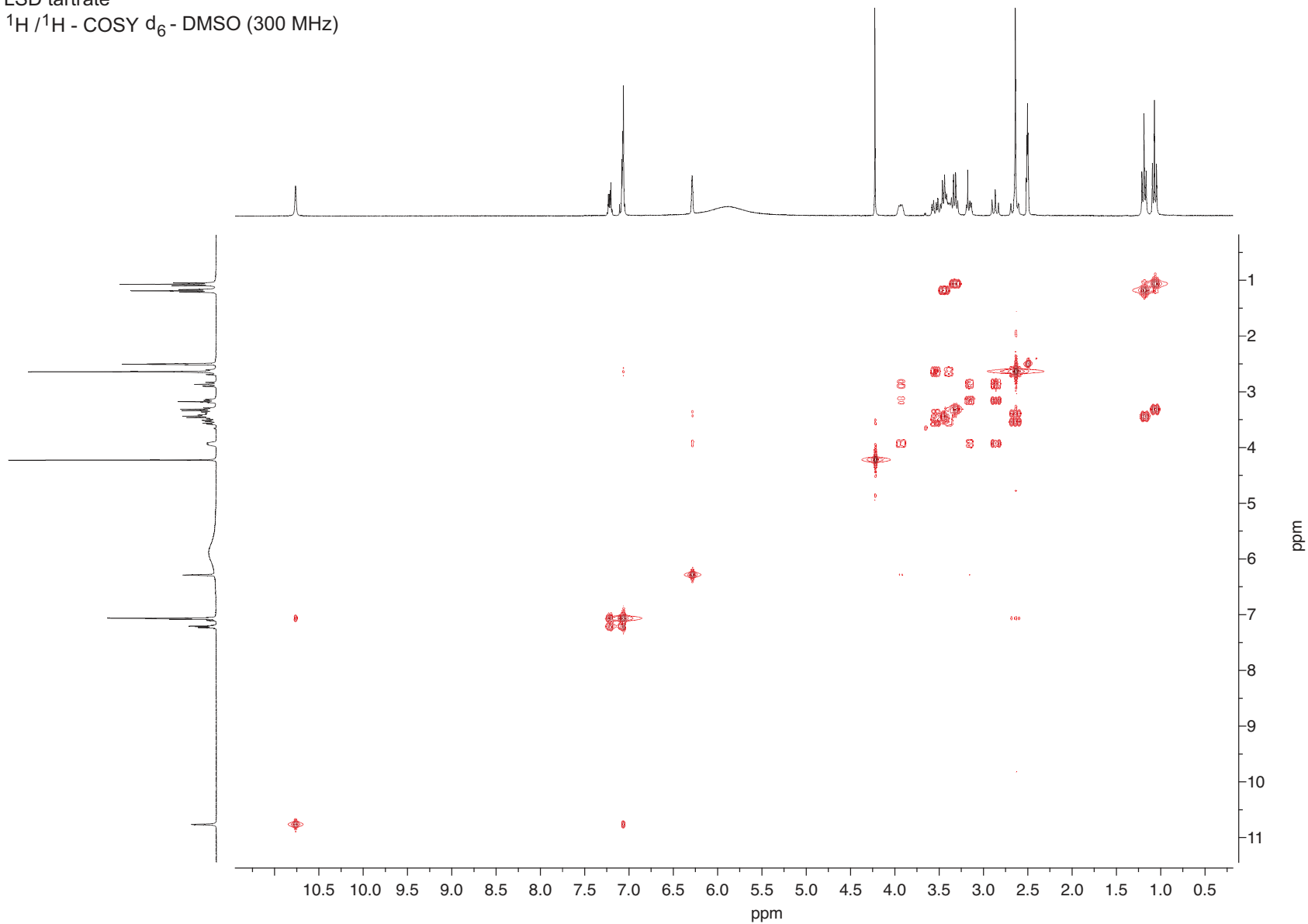
^1H - NMR d_6 - DMSO (300 MHz)



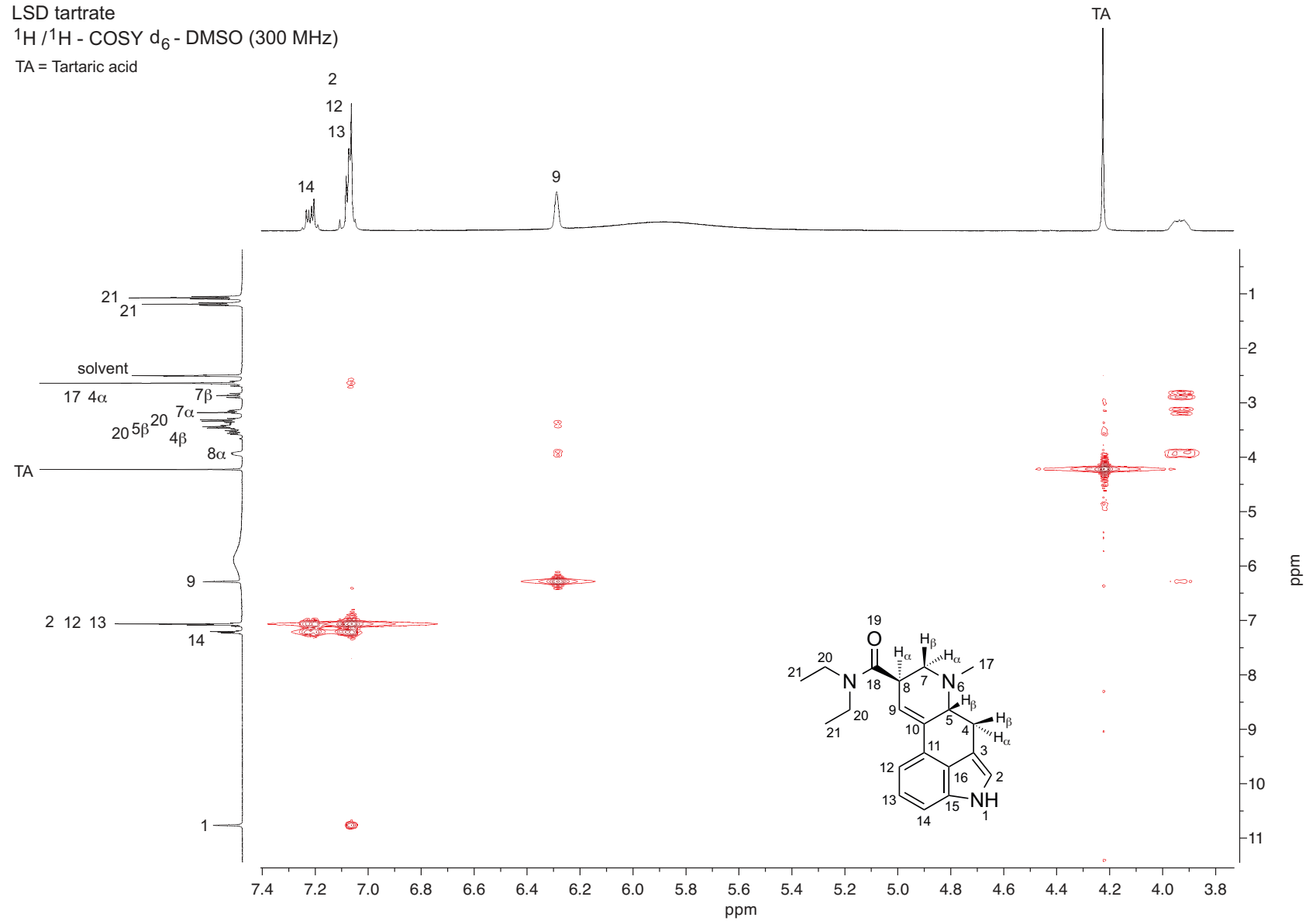
Drug Testing and Analysis – Brandt *et al.* – Supplementary Information



LSD tartrate
 $^1\text{H}/^1\text{H}$ - COSY d_6 -DMSO (300 MHz)



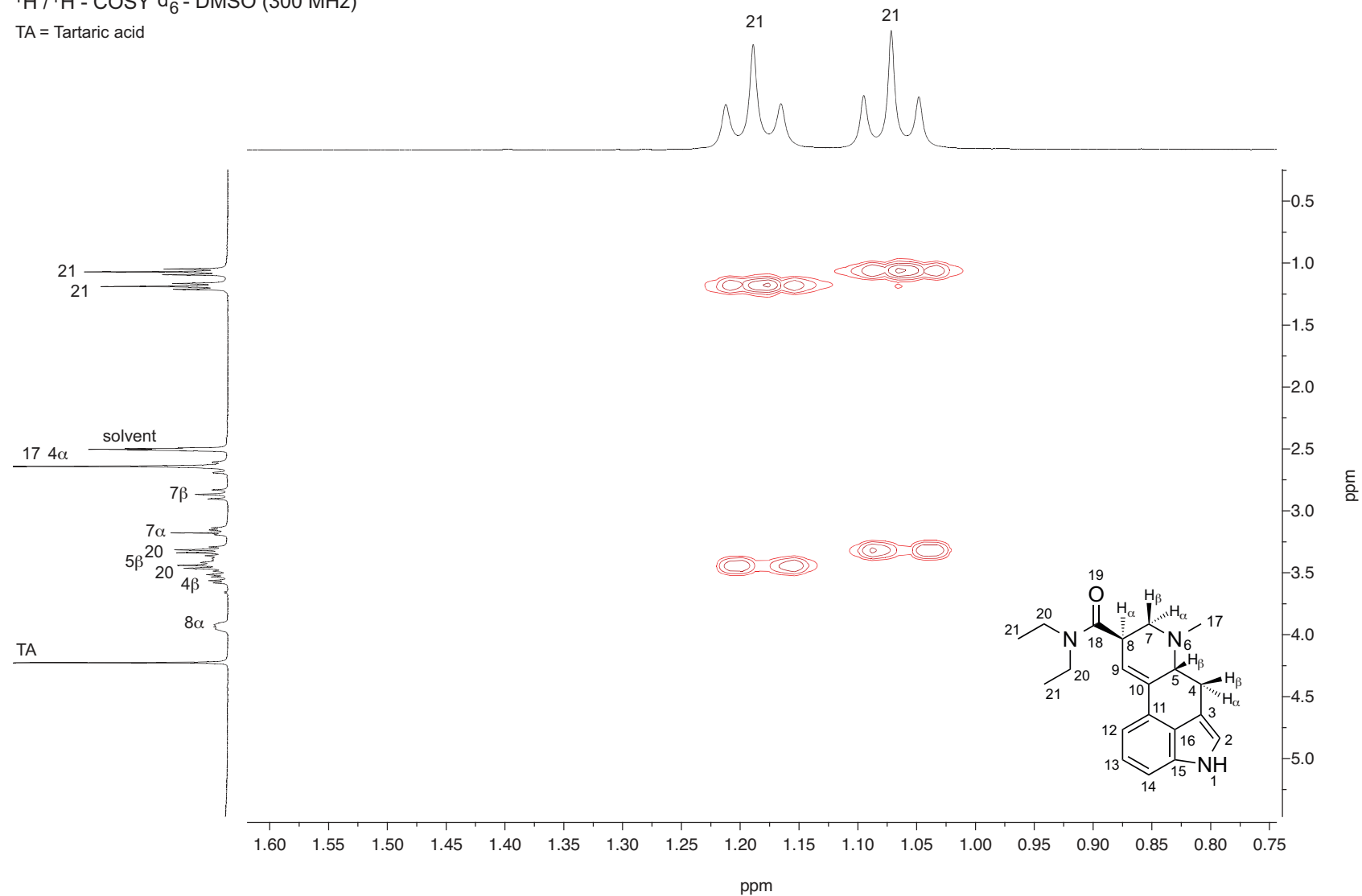
LSD tartrate
 $^1\text{H}/^1\text{H}$ - COSY d_6 - DMSO (300 MHz)
 TA = Tartaric acid



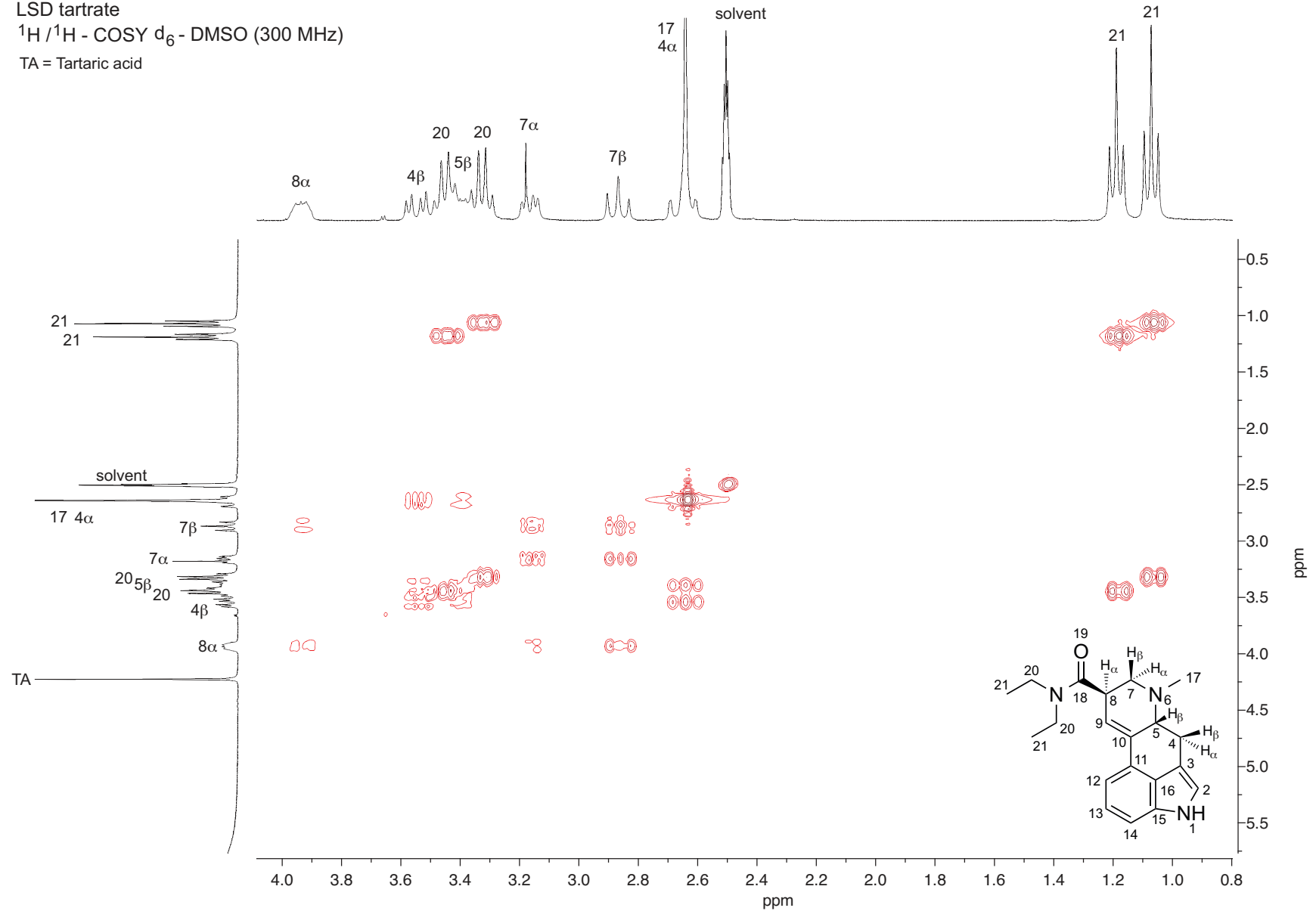
LSD tartrate

$^1\text{H} / ^1\text{H}$ - COSY d_6 -DMSO (300 MHz)

TA = Tartaric acid

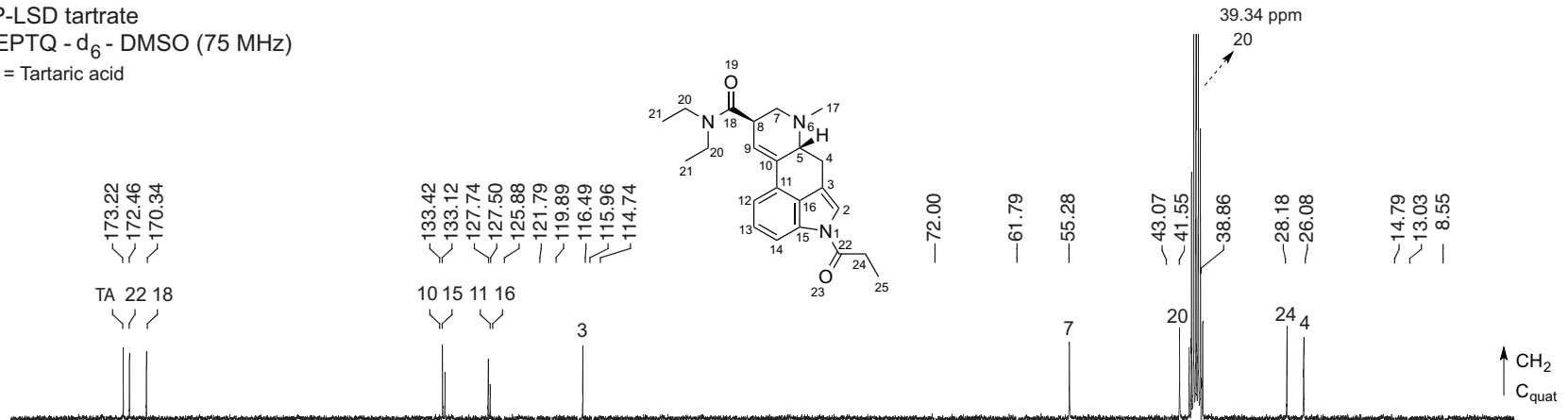


LSD tartrate
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 TA = Tartaric acid

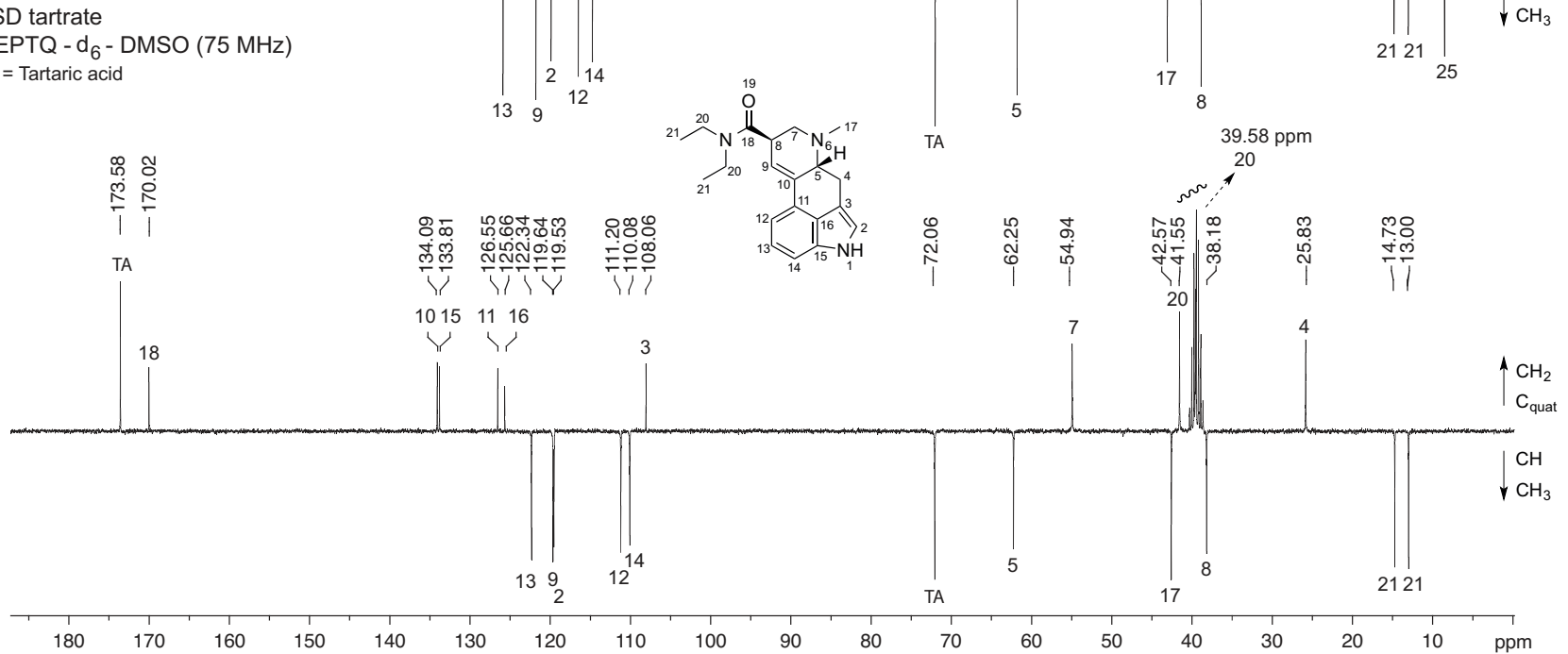


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1P-LSD tartrate
DEPTQ - d₆ - DMSO (75 MHz)
TA = Tartaric acid

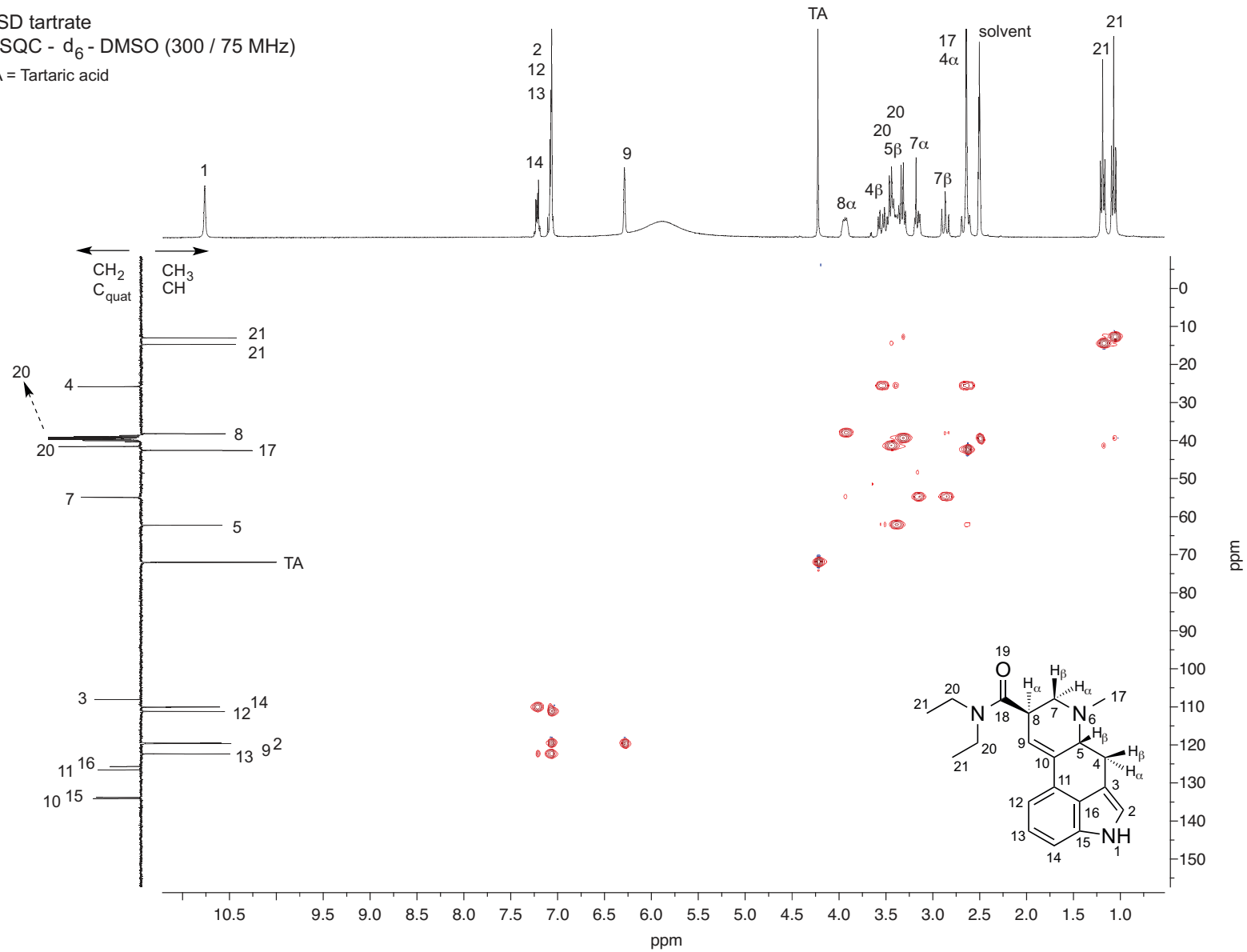


LSD tartrate
DEPTQ - d₆ - DMSO (75 MHz)
TA = Tartaric acid



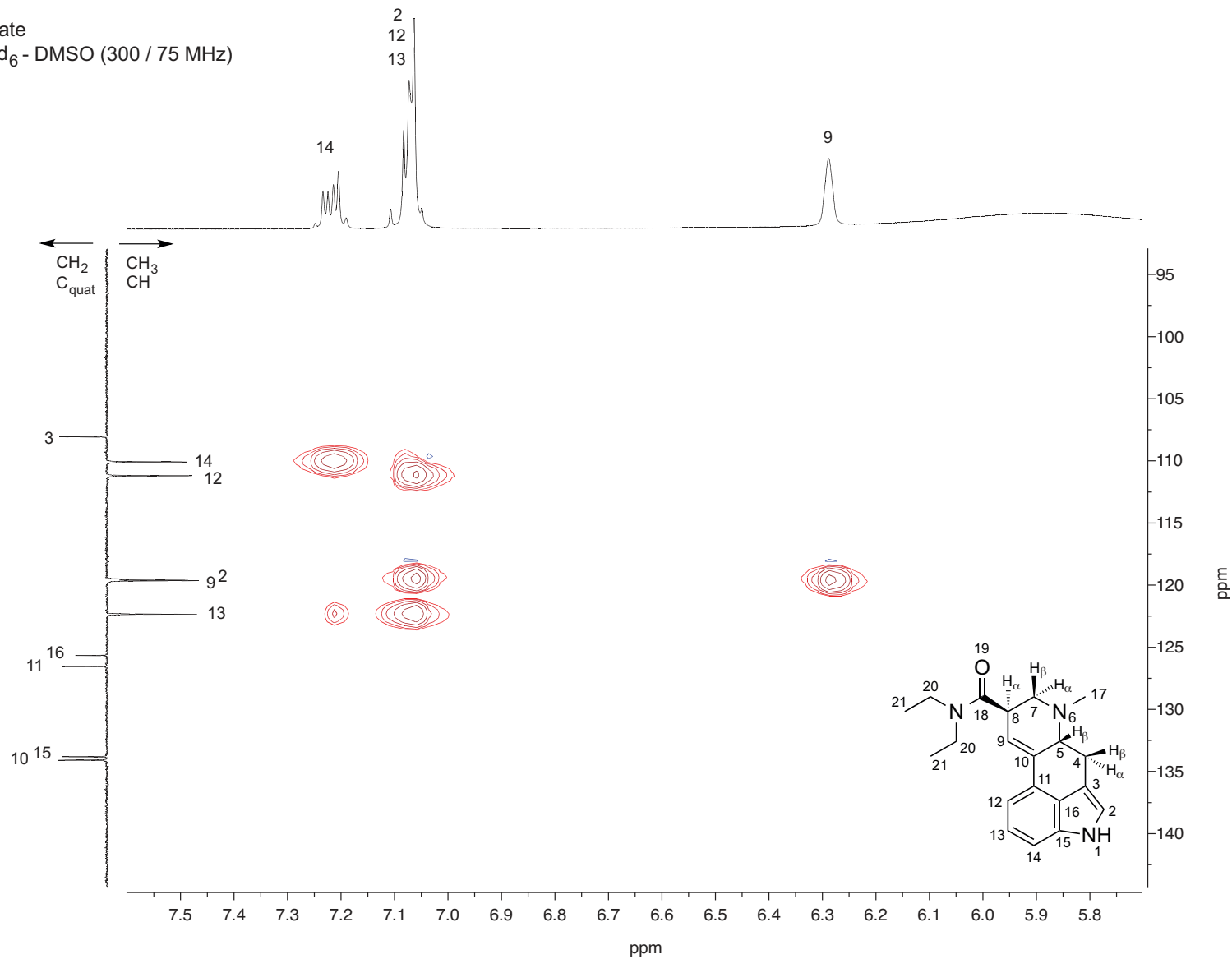
Drug Testing and Analysis – Brandt *et al.* – Supplementary Information

LSD tartrate
 HSQC - d₆-DMSO (300 / 75 MHz)
 TA = Tartaric acid

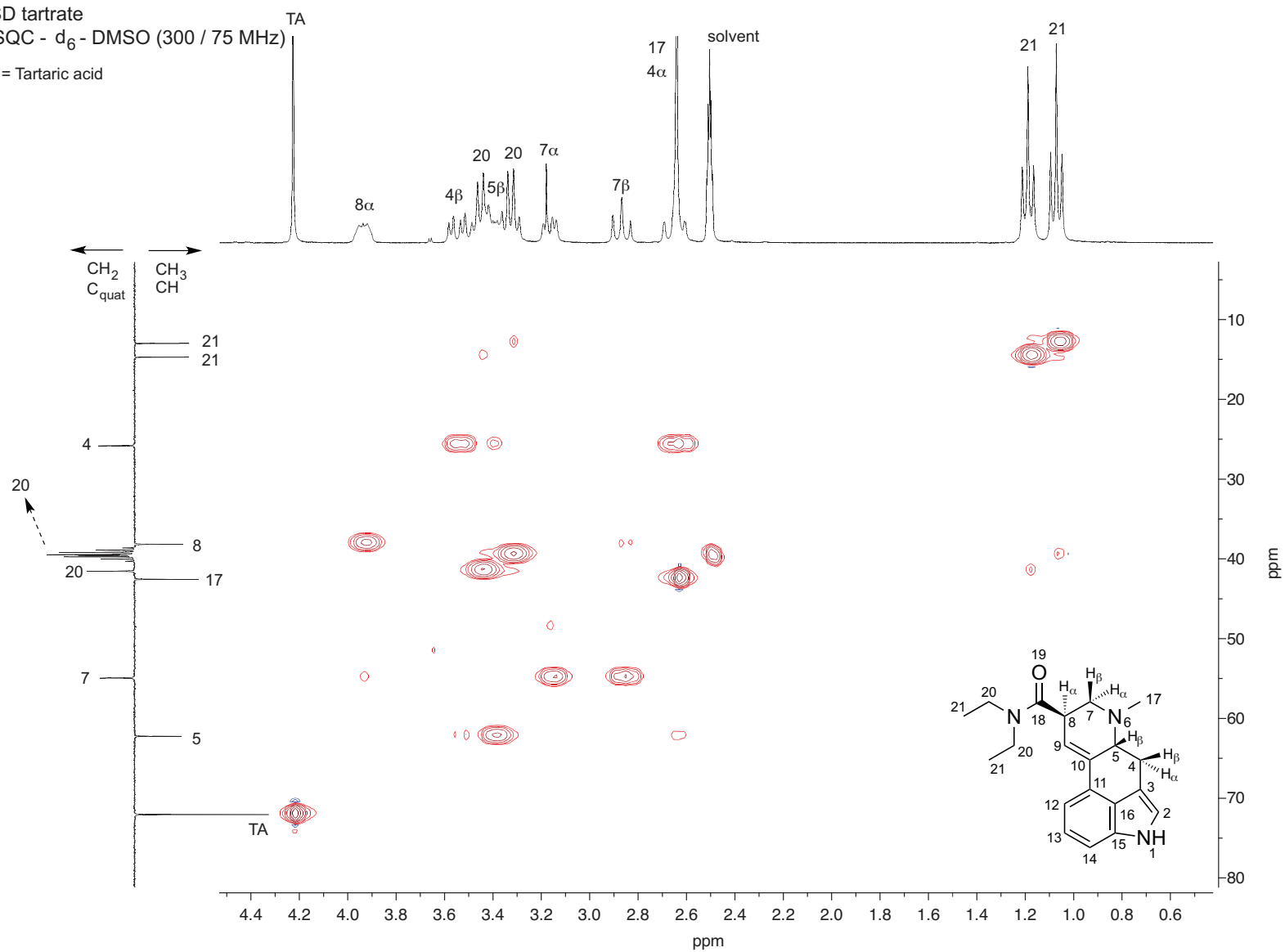


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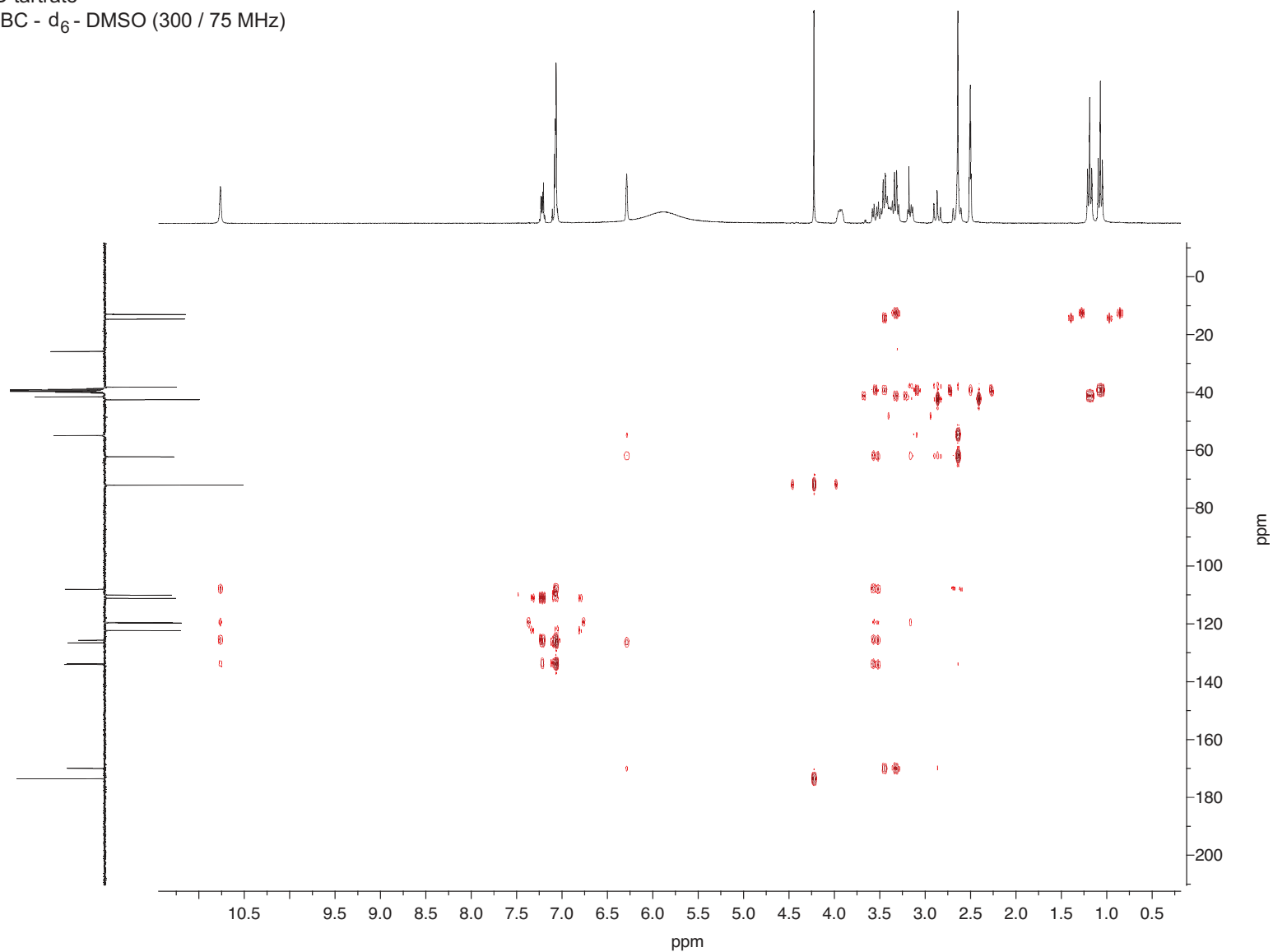
LSD tartrate
 HSQC - d₆-DMSO (300 / 75 MHz)



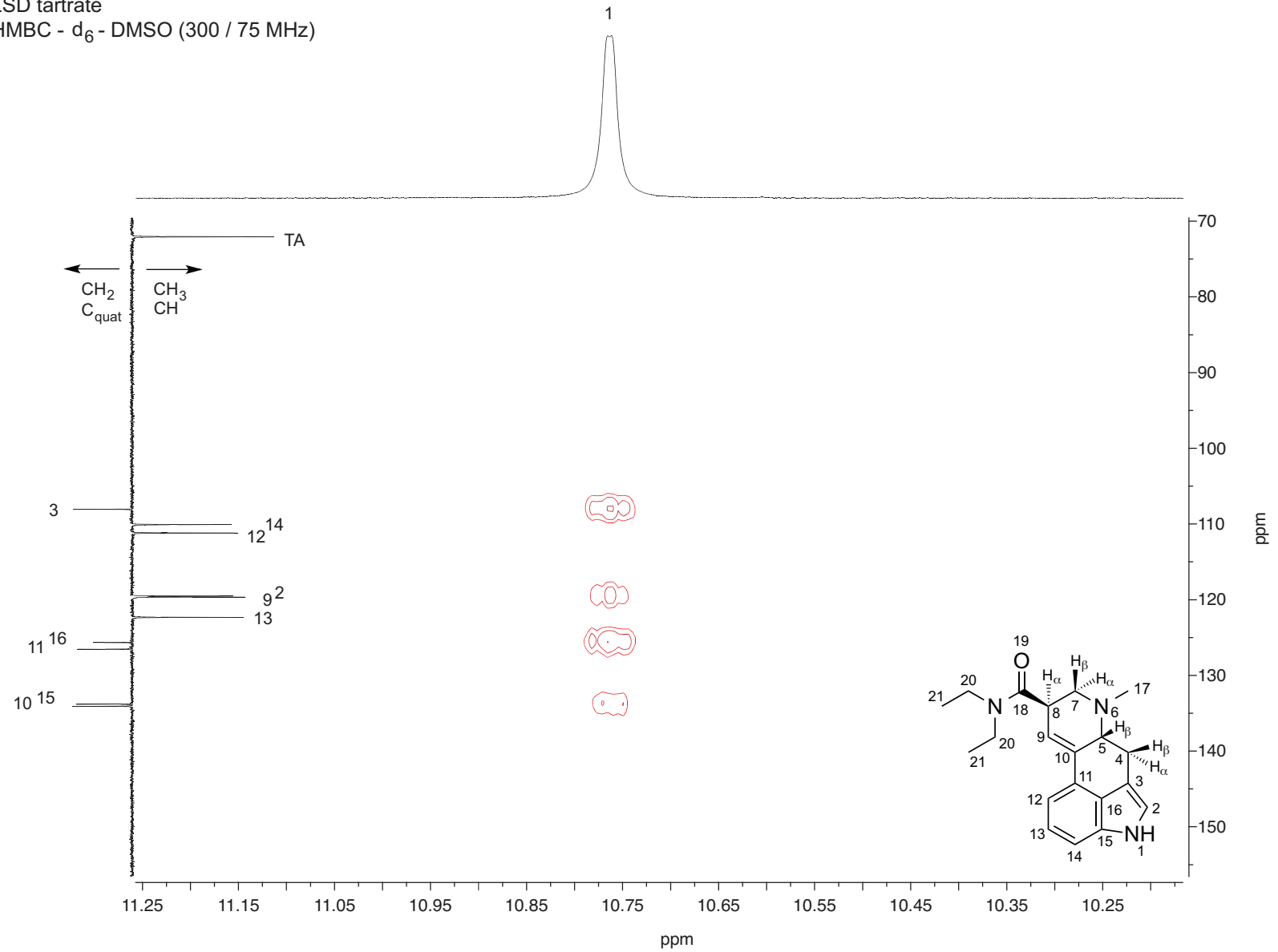
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 HSQC - d₆-DMSO (300 / 75 MHz)
 TA = Tartaric acid



LSD tartrate
HMBC - d₆ - DMSO (300 / 75 MHz)



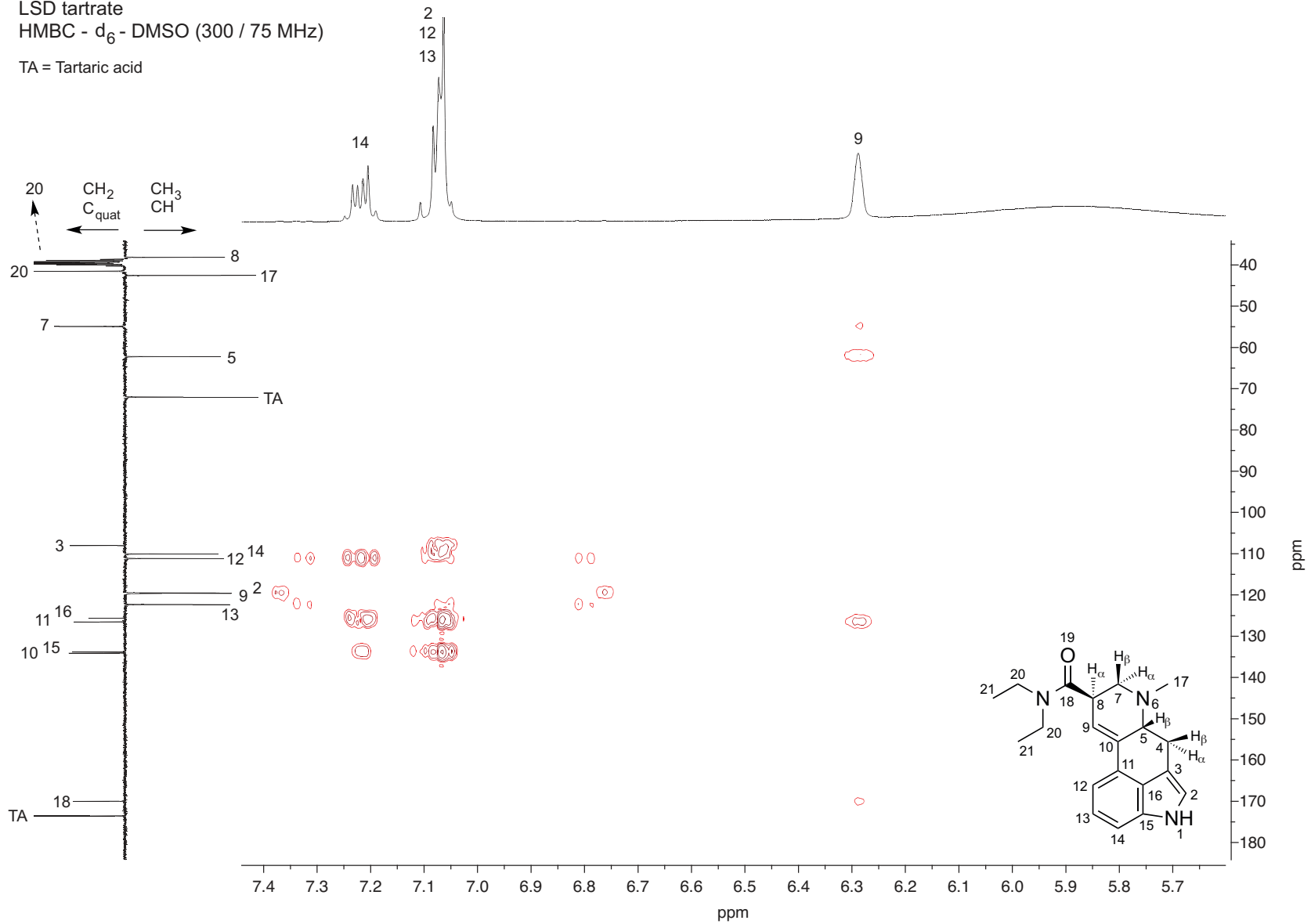
LSD tartrate
 HMBC - d₆ - DMSO (300 / 75 MHz)

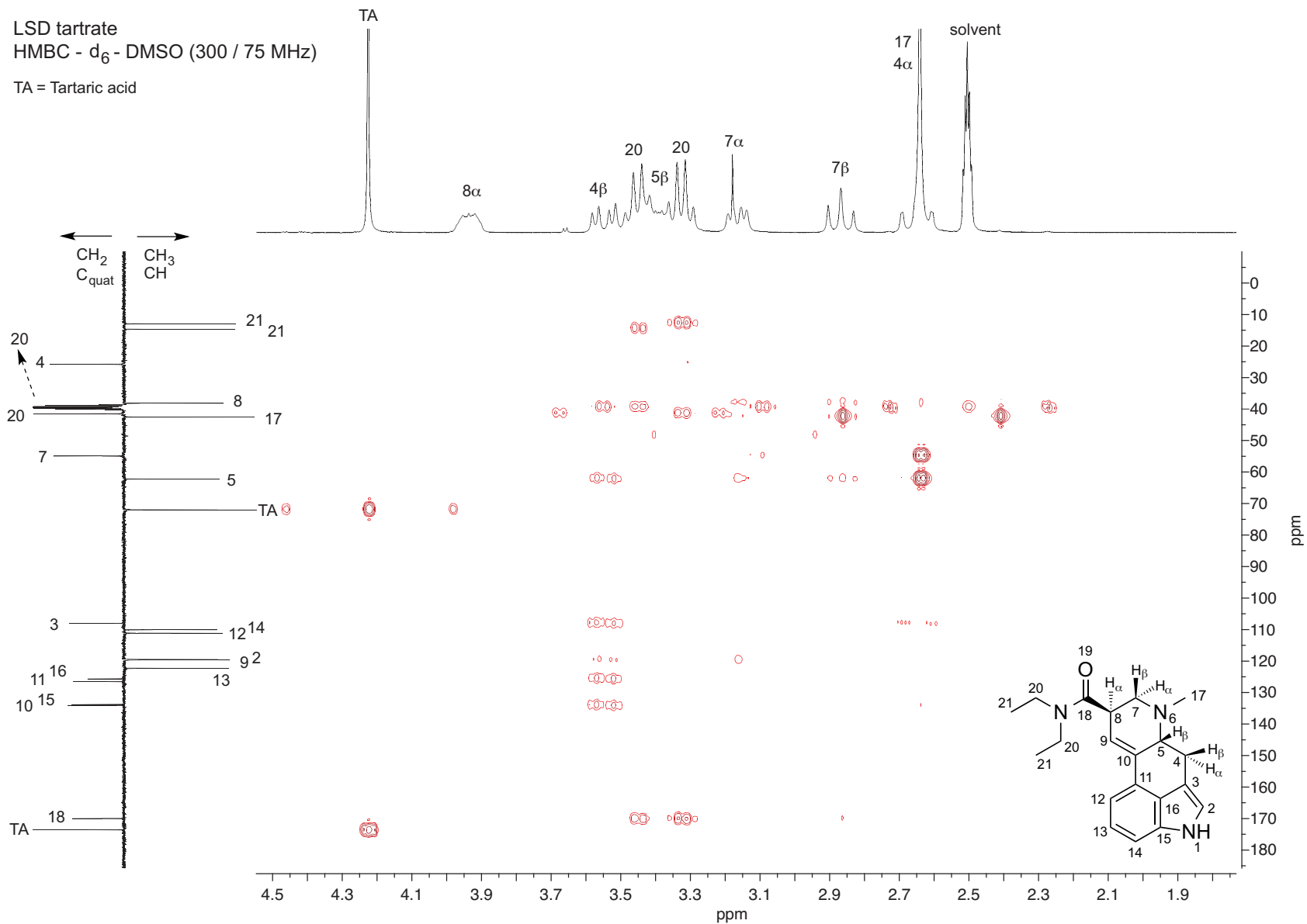


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 HMBC - d₆ - DMSO (300 / 75 MHz)

TA = Tartaric acid





LSD tartrate
 HMBC - d₆ - DMSO (300 / 75 MHz)

