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Return of the lysergamides. Part III: Analytical characterization of N6-ethyl-6-norlysergic acid diethylamide (ETH-LAD) and 1-propionyl ETH-LAD (1P-ETH-LAD)

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Return of the lysergamides. Part III: Analytical characterization of *N*⁶-ethyl-6-norlysergic acid diethylamide (ETH-LAD) and 1-propionyl ETH-LAD (1P-ETH-LAD)

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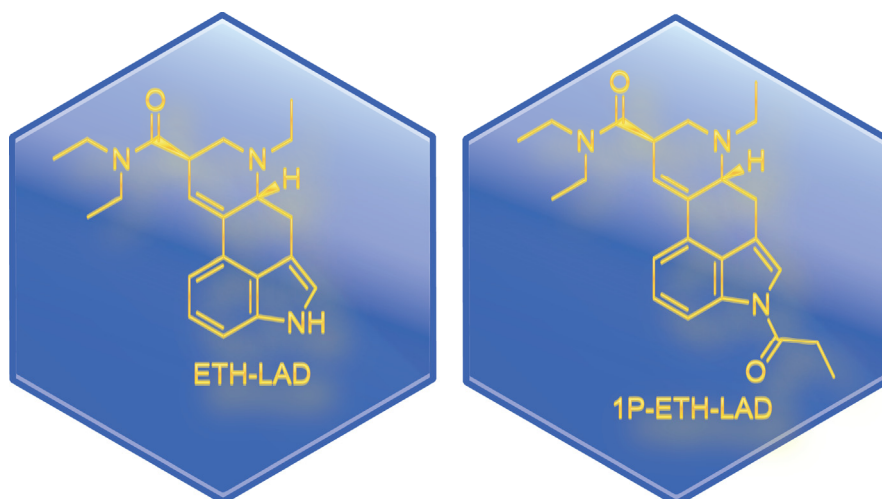
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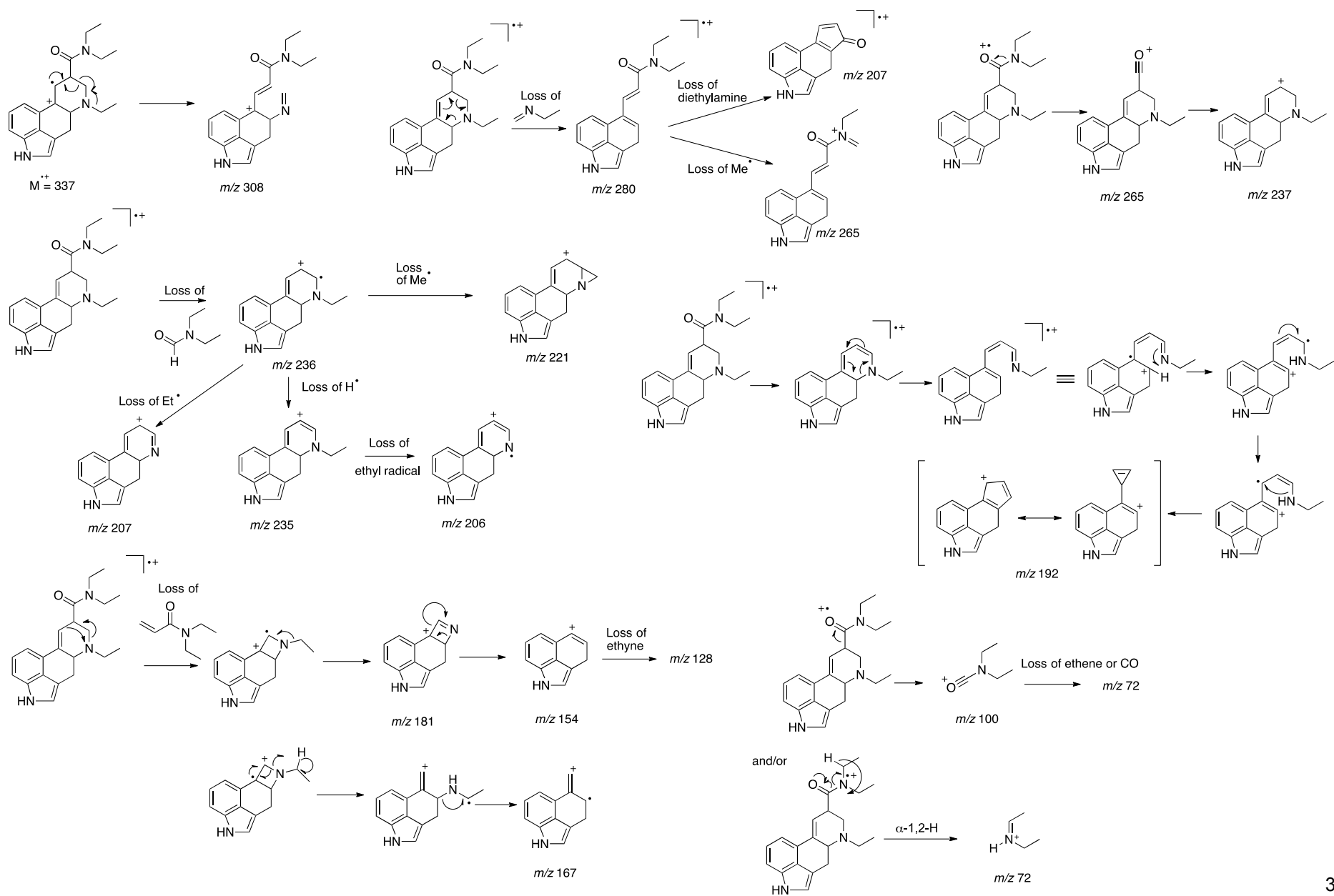
^h Department of Psychiatry, University of California San Diego, 9500 Gilman Drive, La Jolla, CA 92093-0804, USA

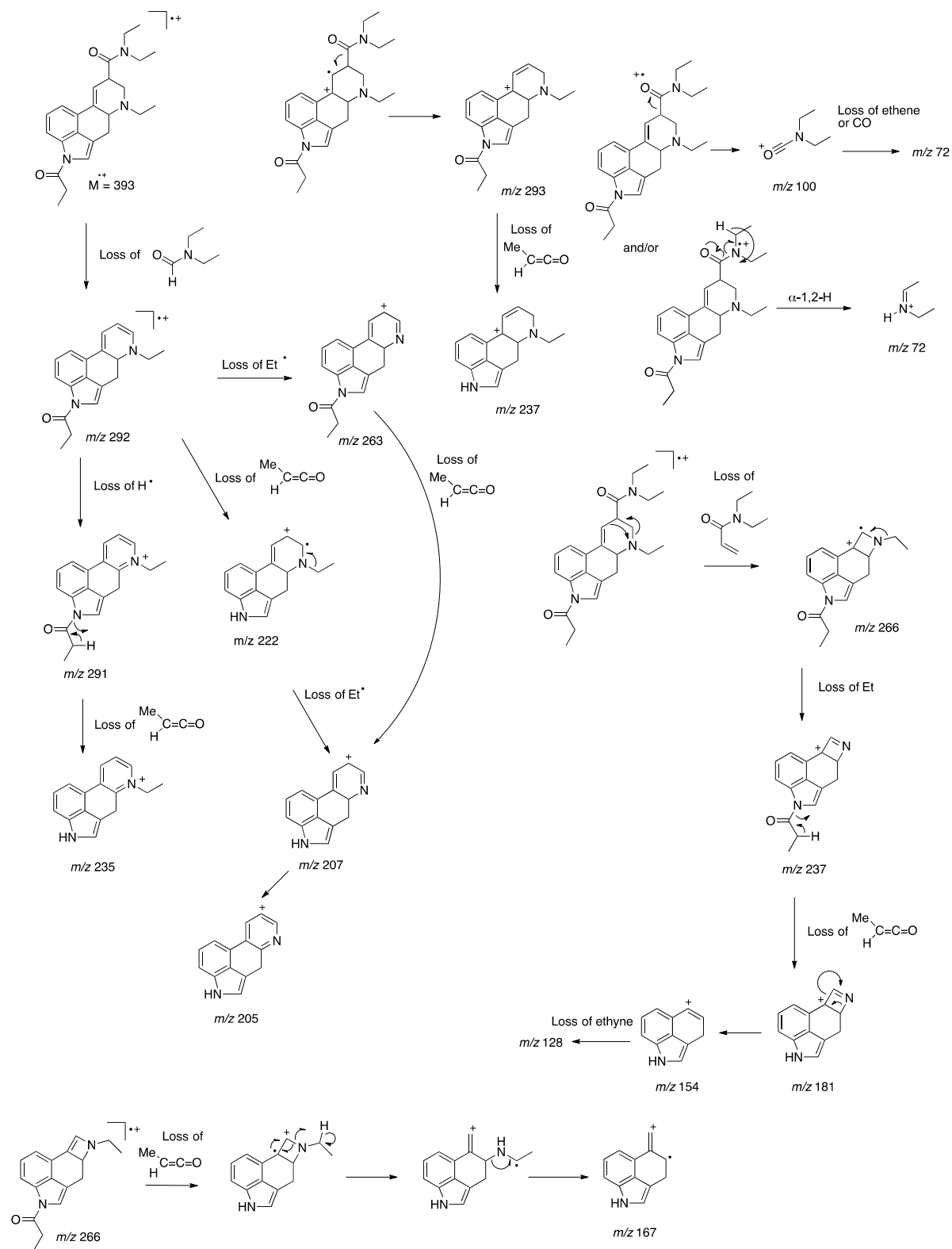
* Correspondence to: Simon D. Brandt, School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK. E-Mail: s.brandt@ljmu.ac.uk

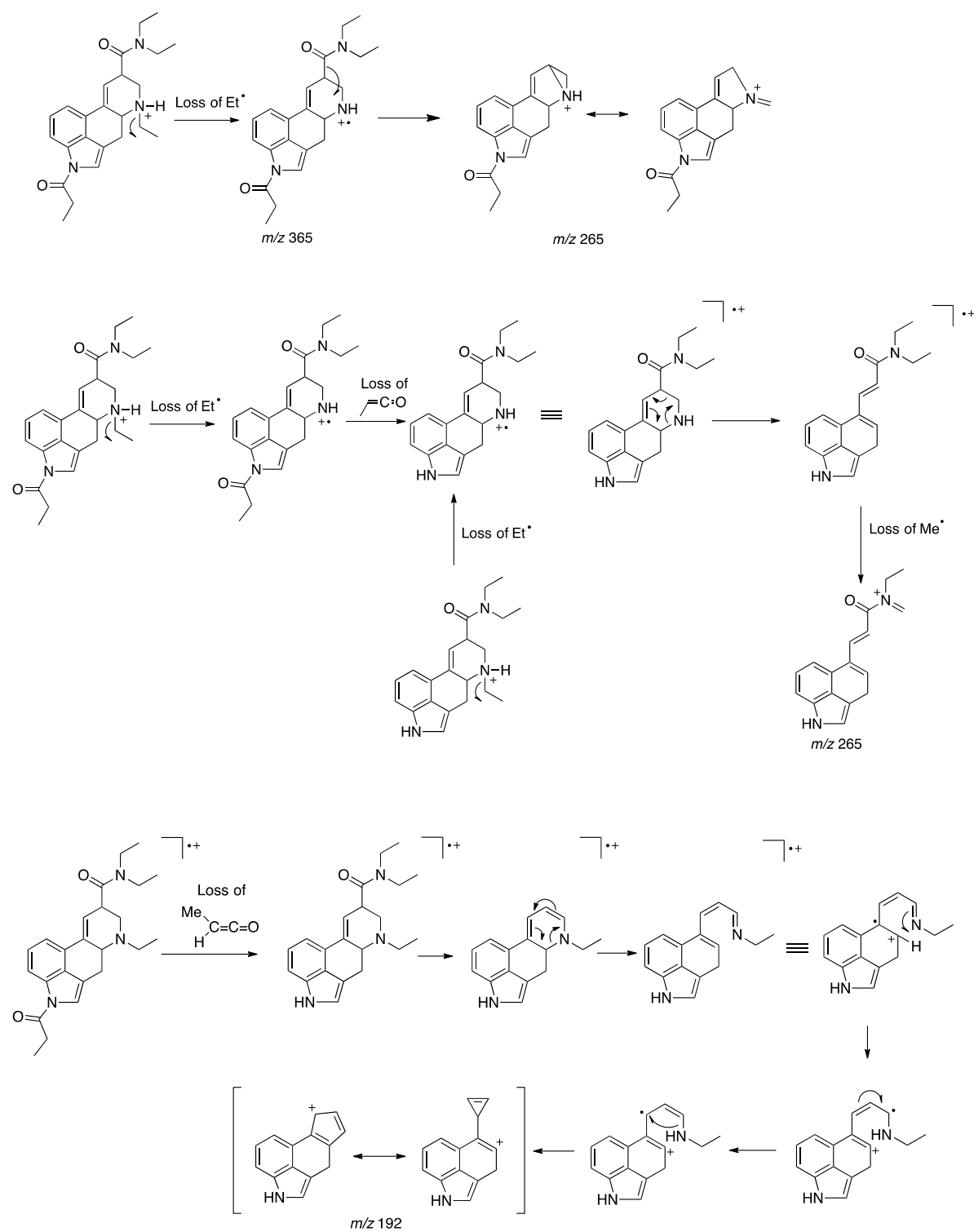


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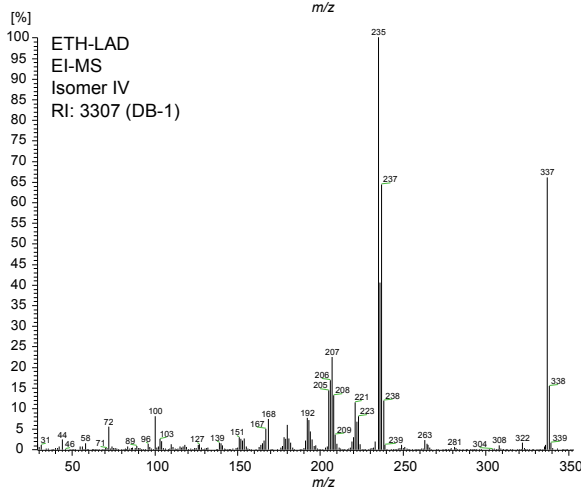
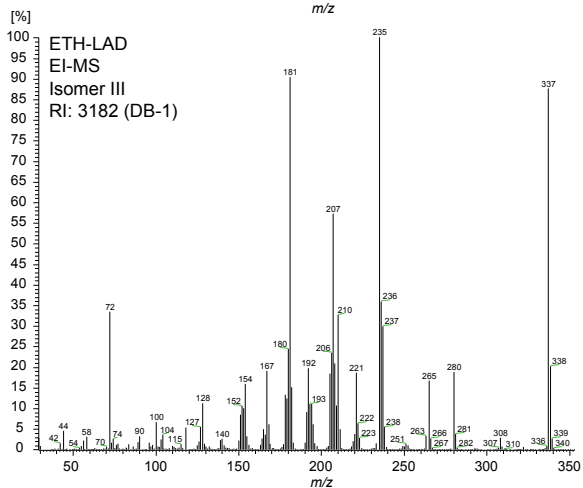
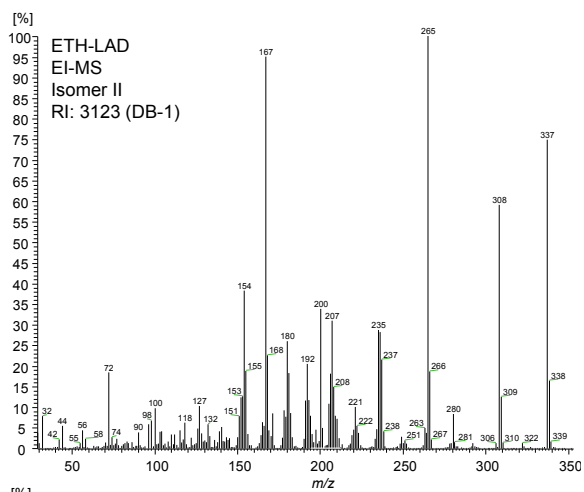
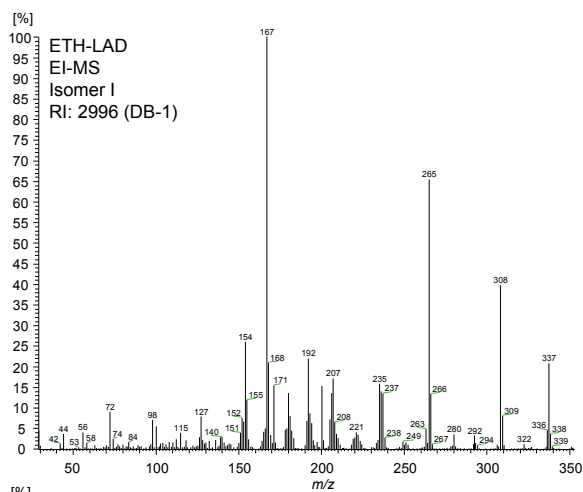
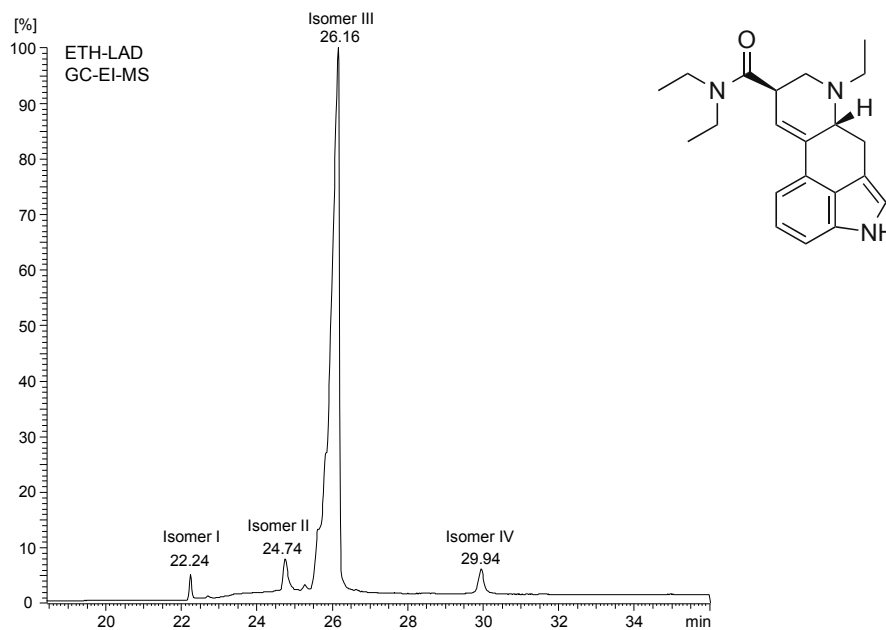
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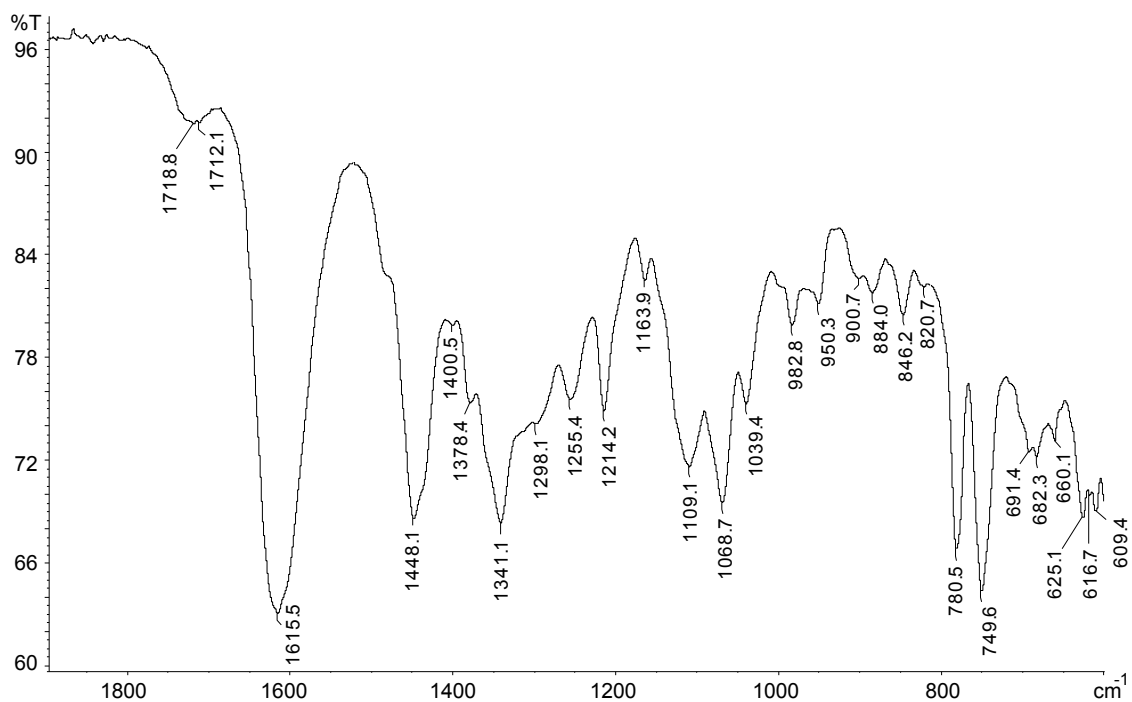
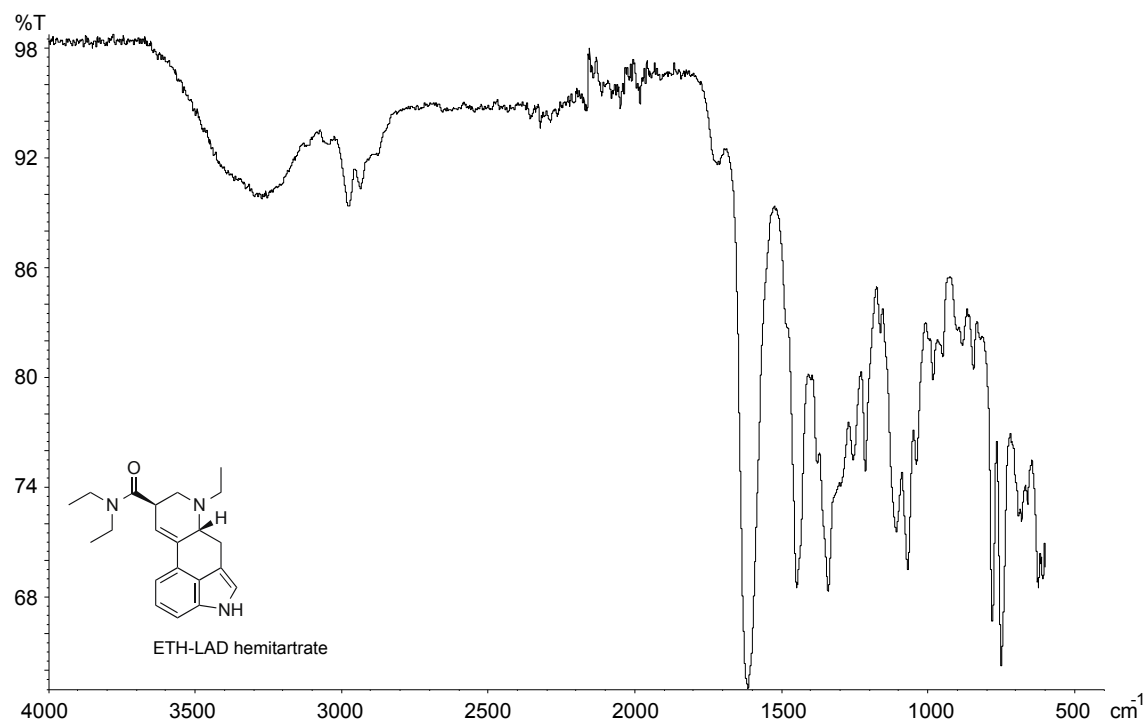


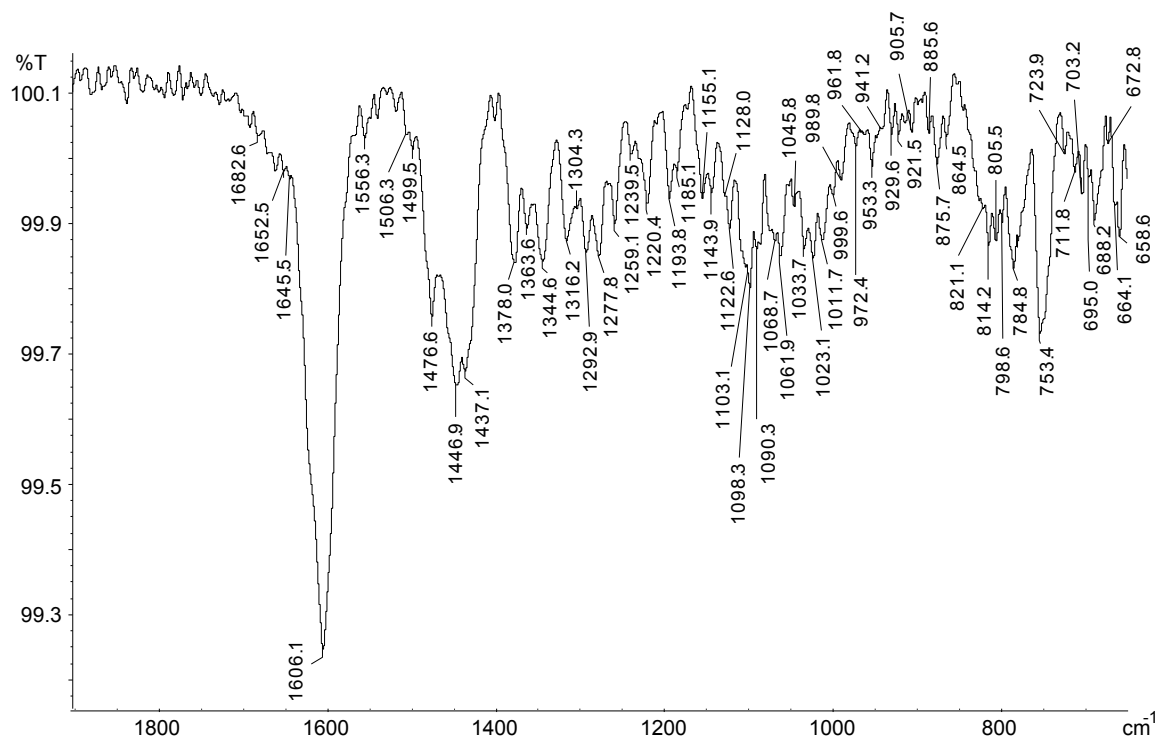
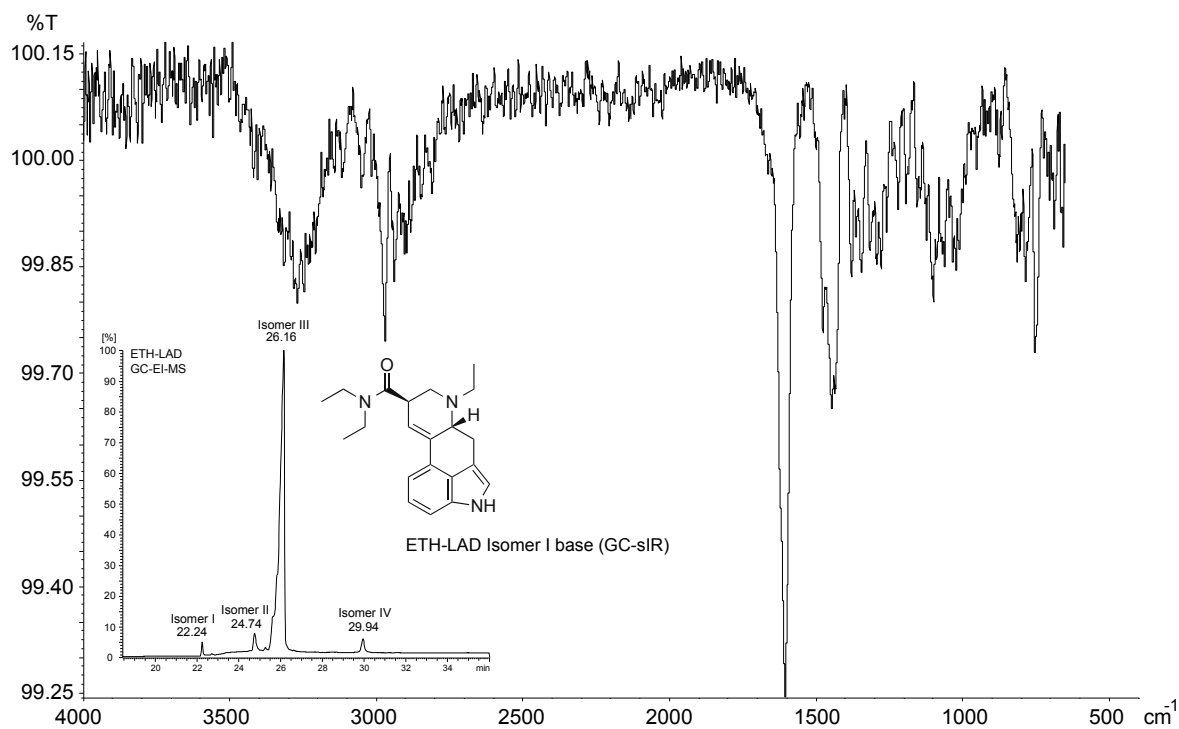


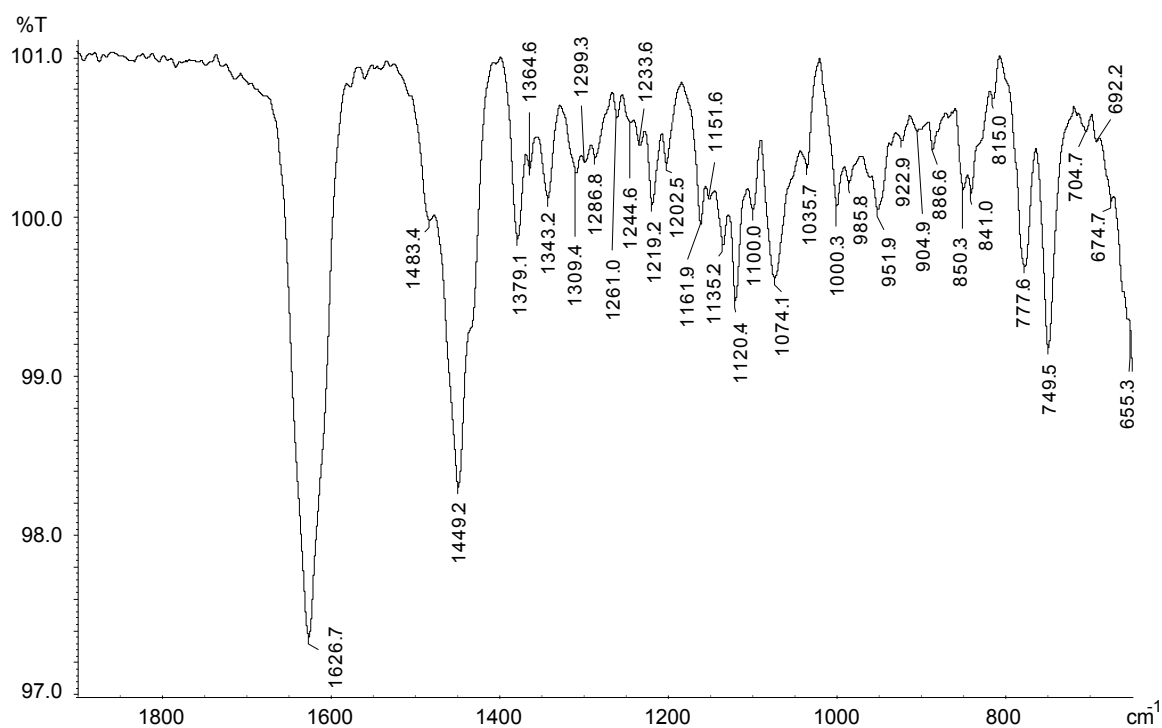
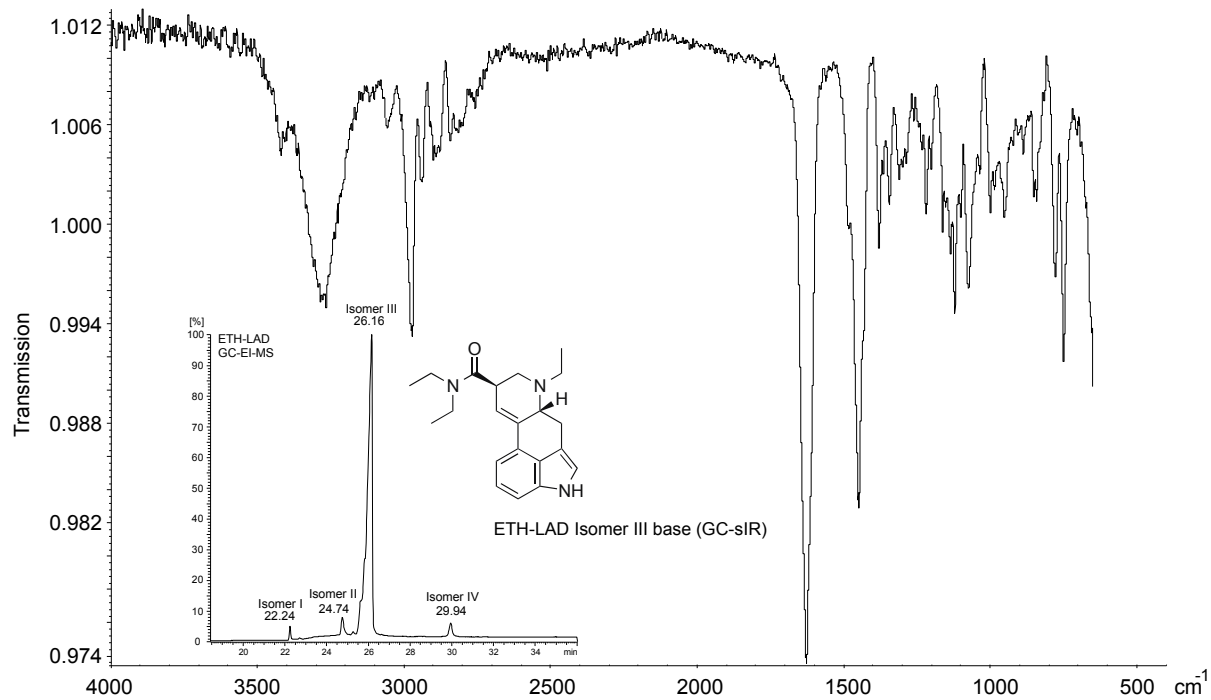
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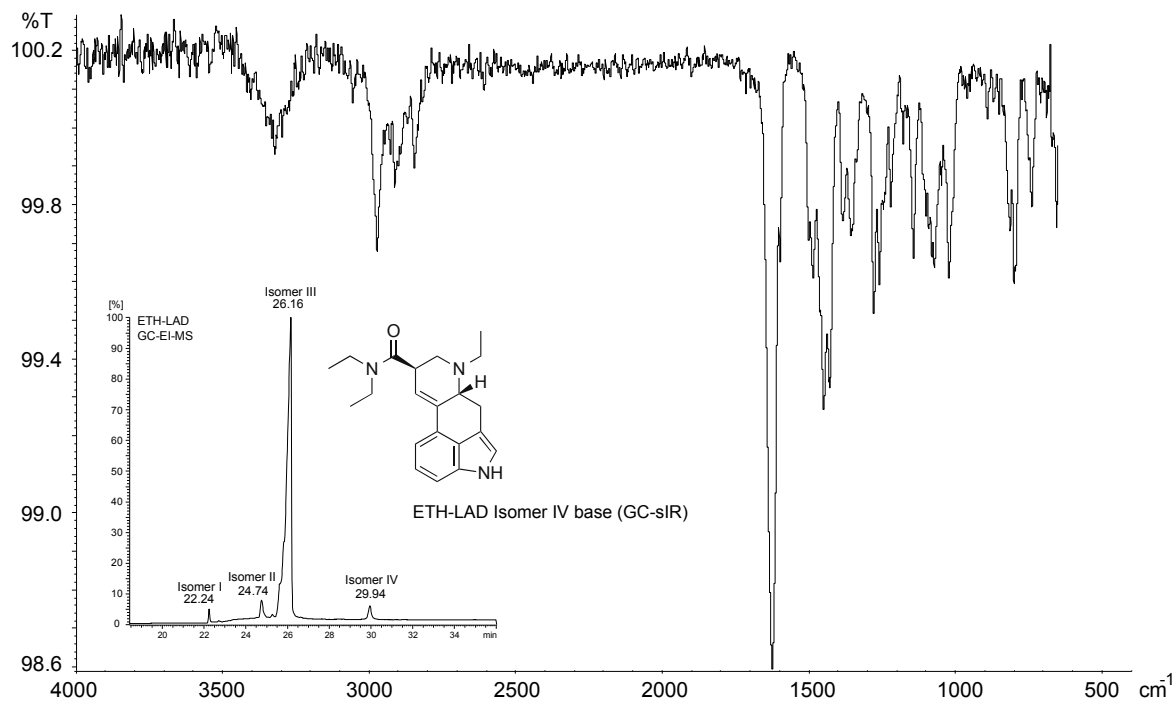


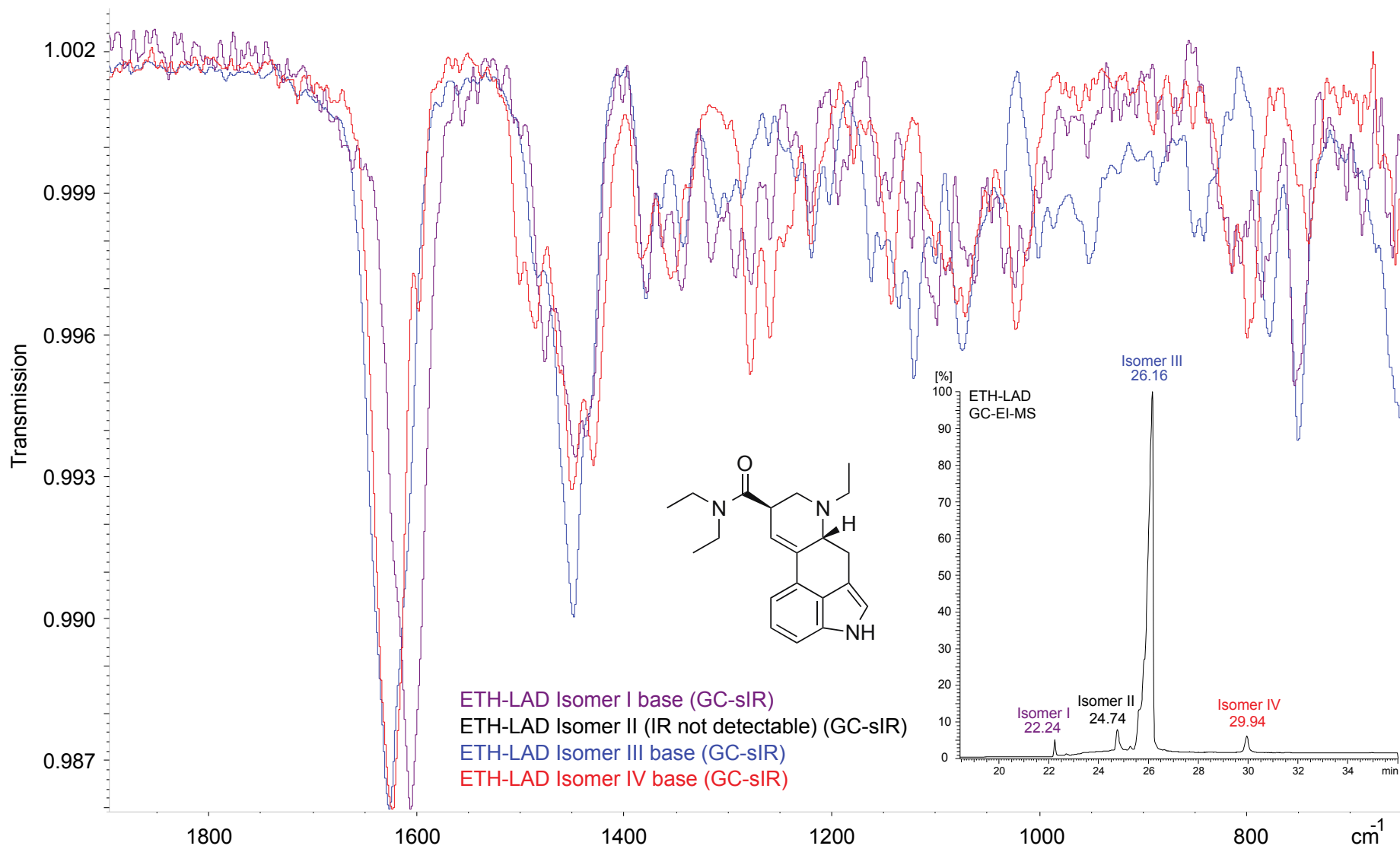
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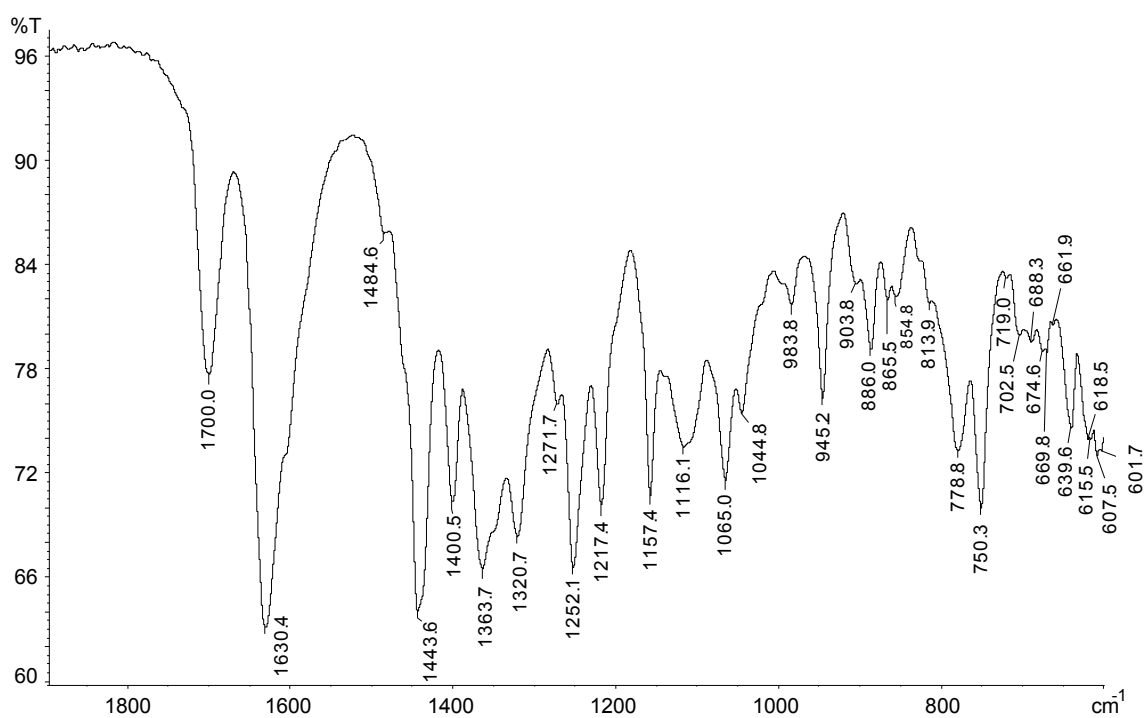
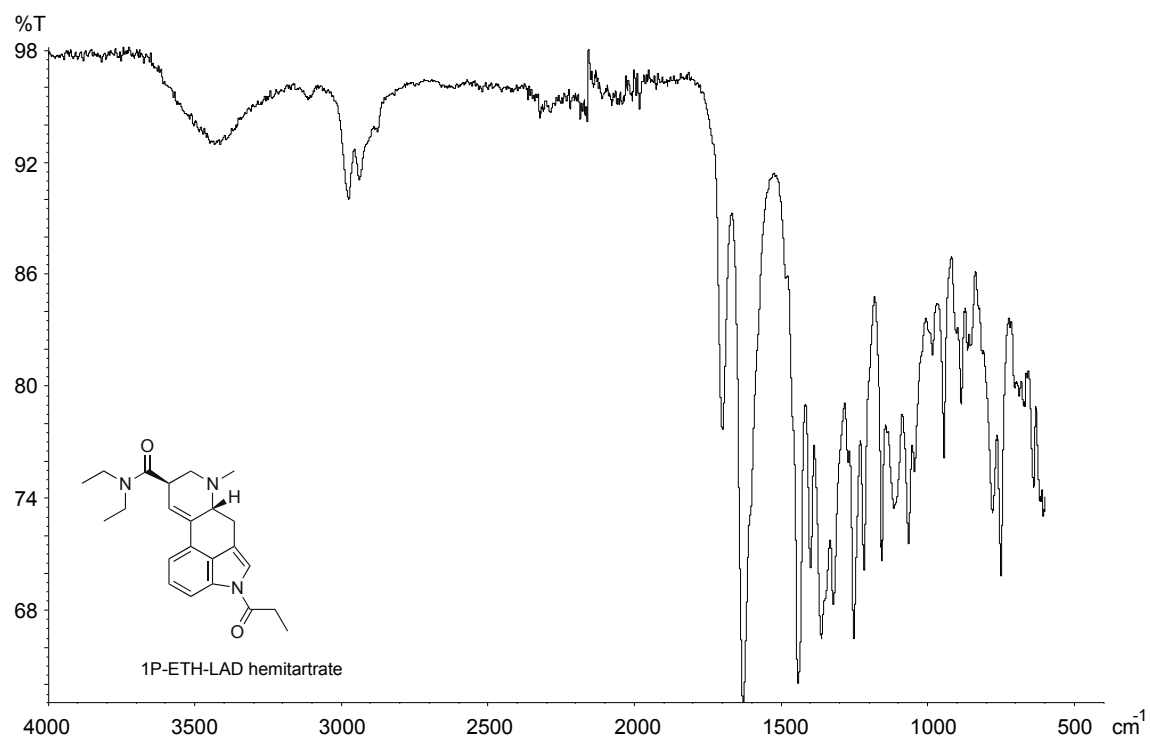


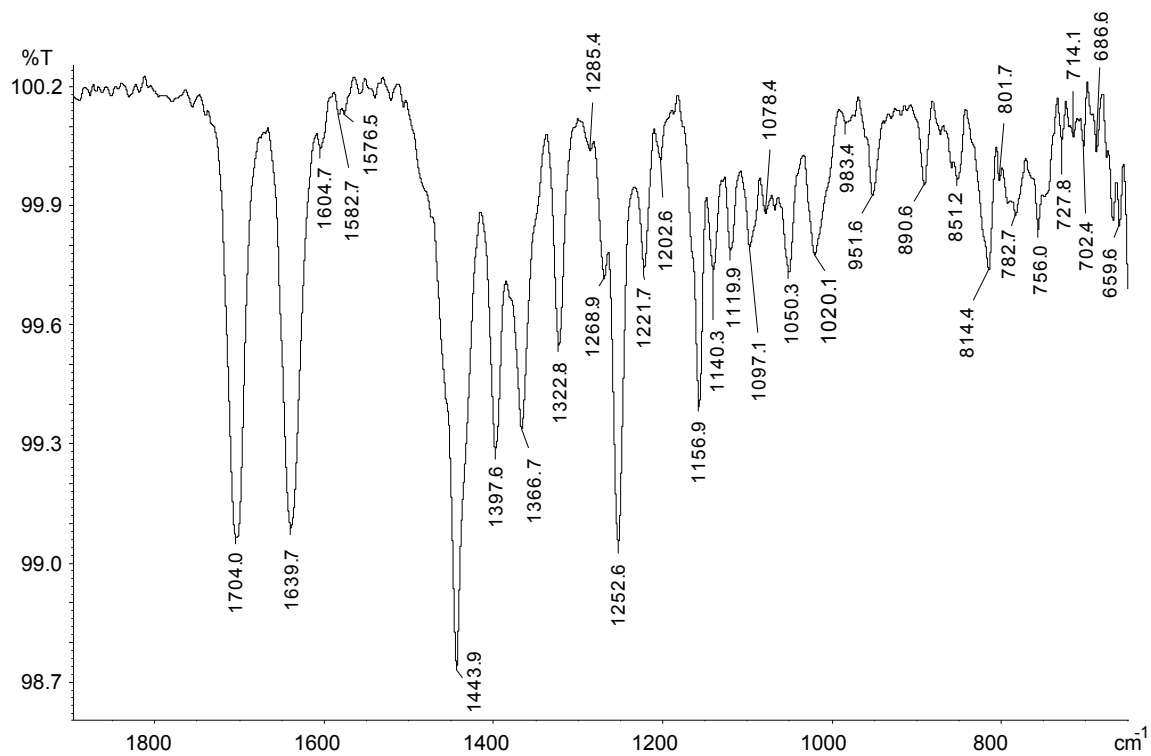
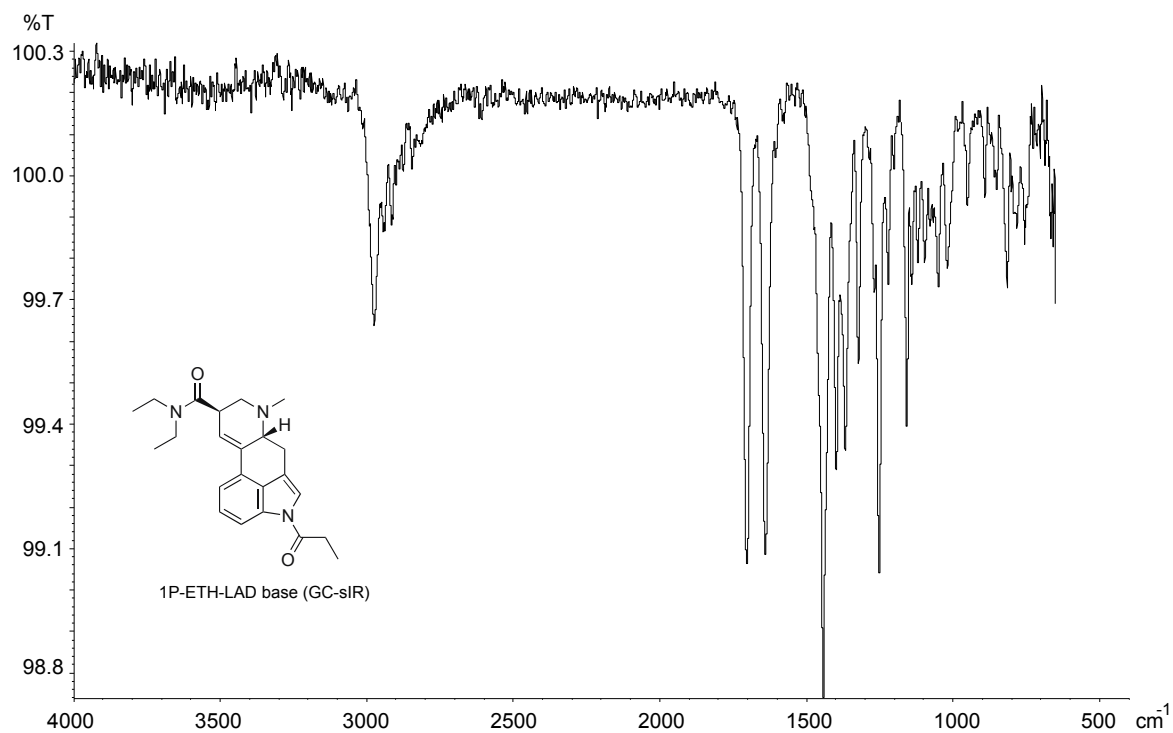






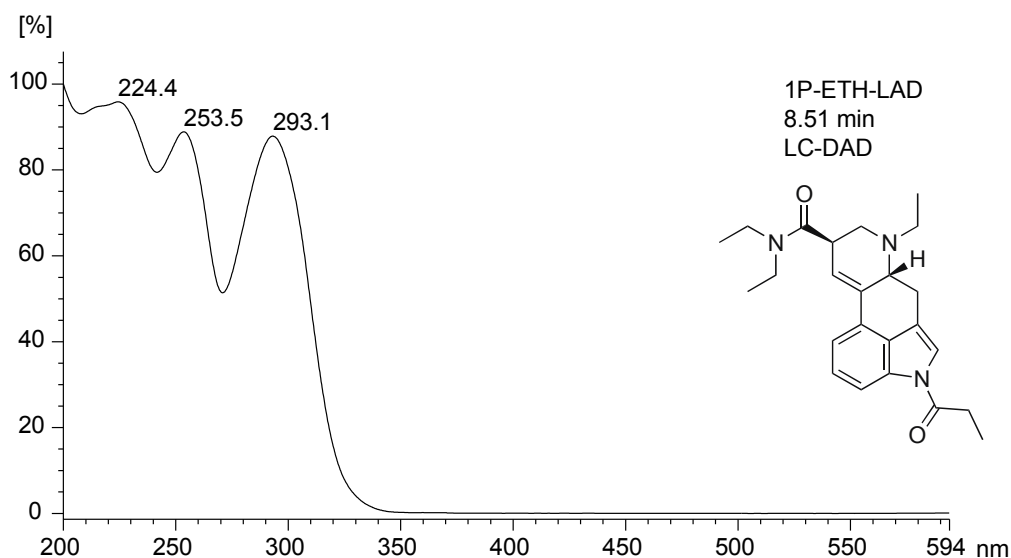
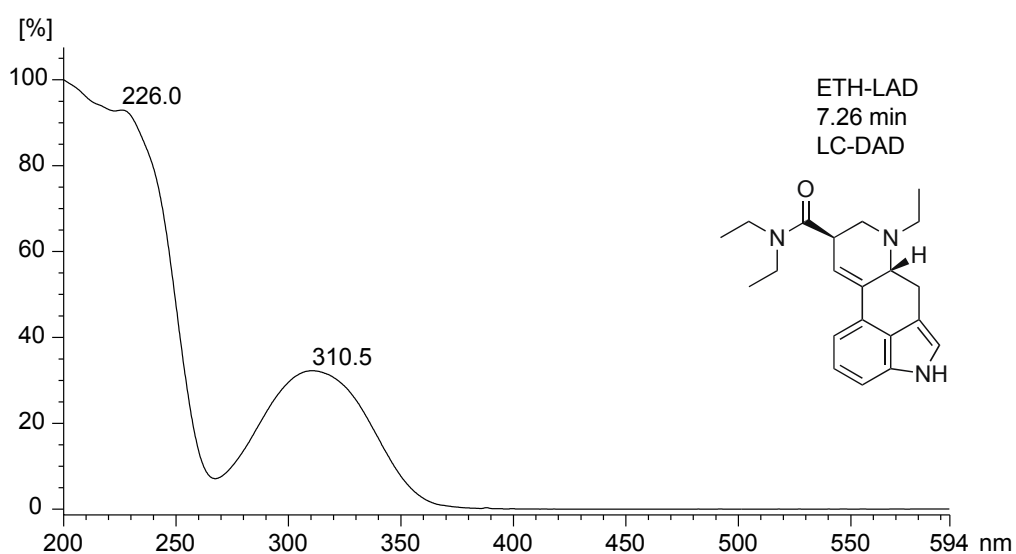






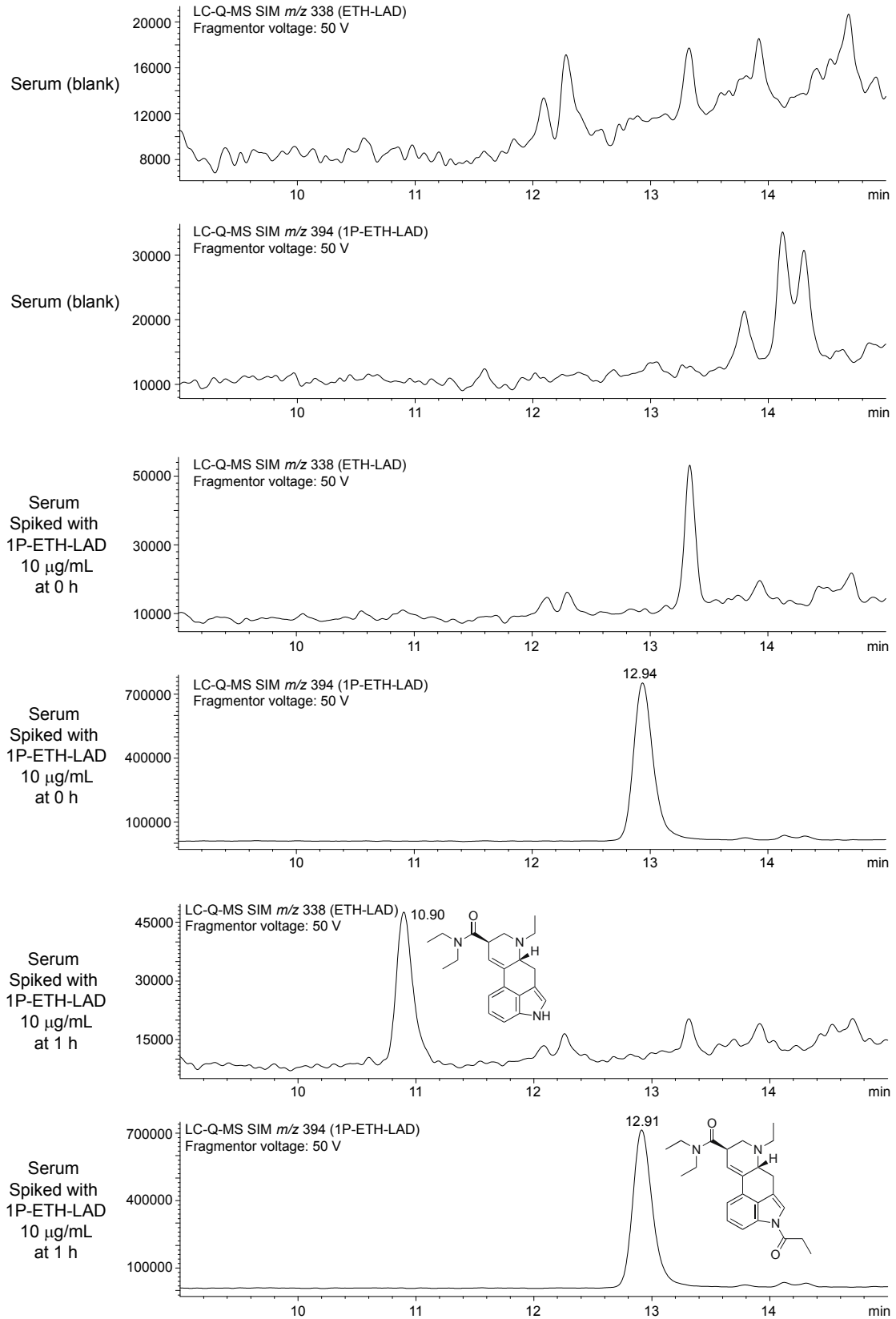
Liquid chromatography diode array detection

A Dionex 3000 Ultimate liquid chromatography system coupled to a UV diode array detector (Thermo Fisher, St. Albans, UK) was used for analysis using a Phenomenex Synergi Fusion column (150 mm × 2 mm, 4 μm) that was protected by a 4 mm × 3 mm Phenomenex Synergi Fusion guard column (Phenomenex, Cheshire, UK). The Mobile phases were 70% acetonitrile with 25 mM of triethylammonium phosphate buffer (TEAP) and aqueous TEAP (25 mM) buffer. The gradient elution commenced with 4% acetonitrile and ramped to 70% acetonitrile in 15 min and held for 3 min, resulting in a total acquisition time of 18 min at a flow rate of 0.6 mL/min. The diode array detection window was set at 200 nm–595 nm (collection rate 2 Hz).

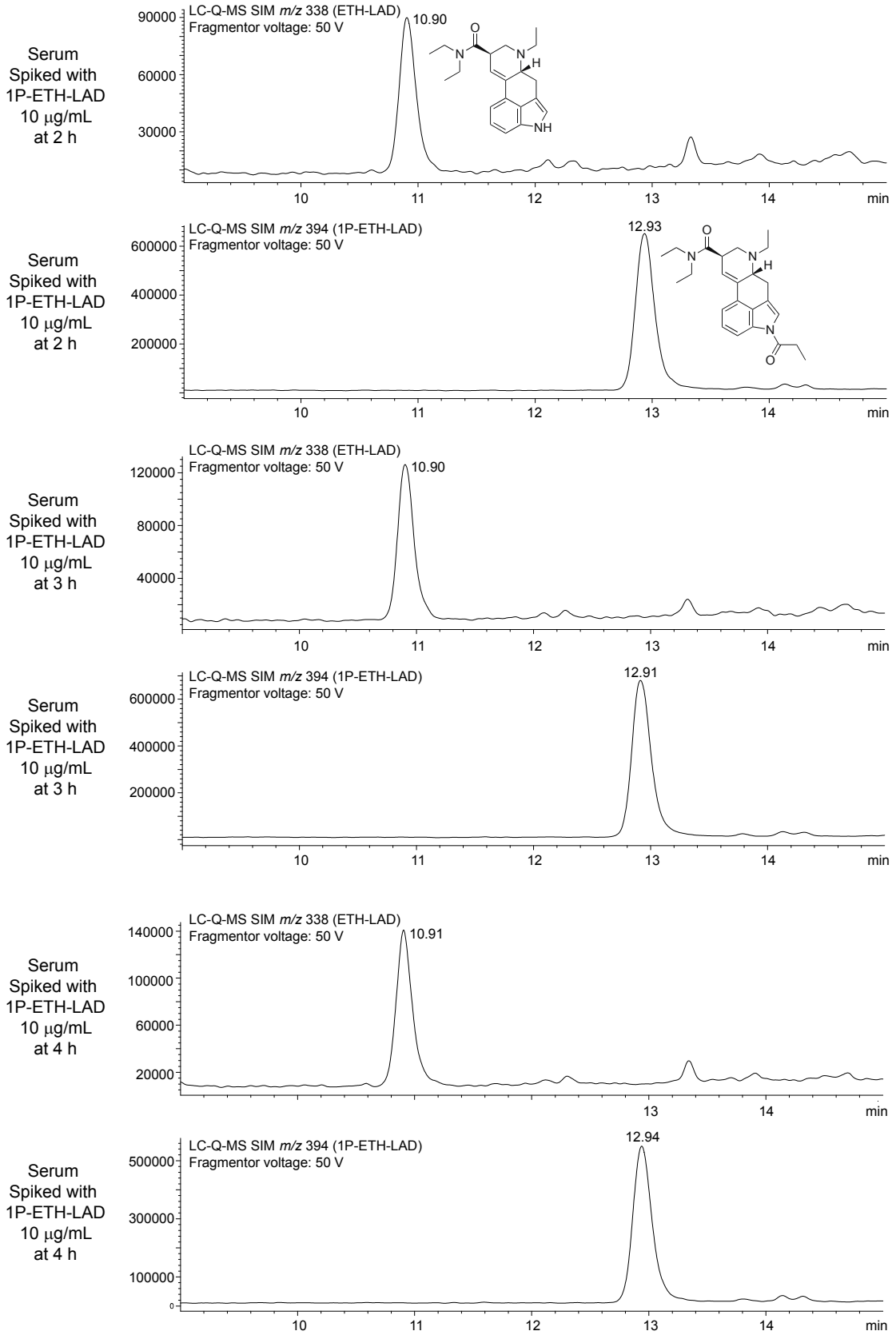


1P-ETH-LAD incubation (10 µg/mL) in human serum at 37°C.

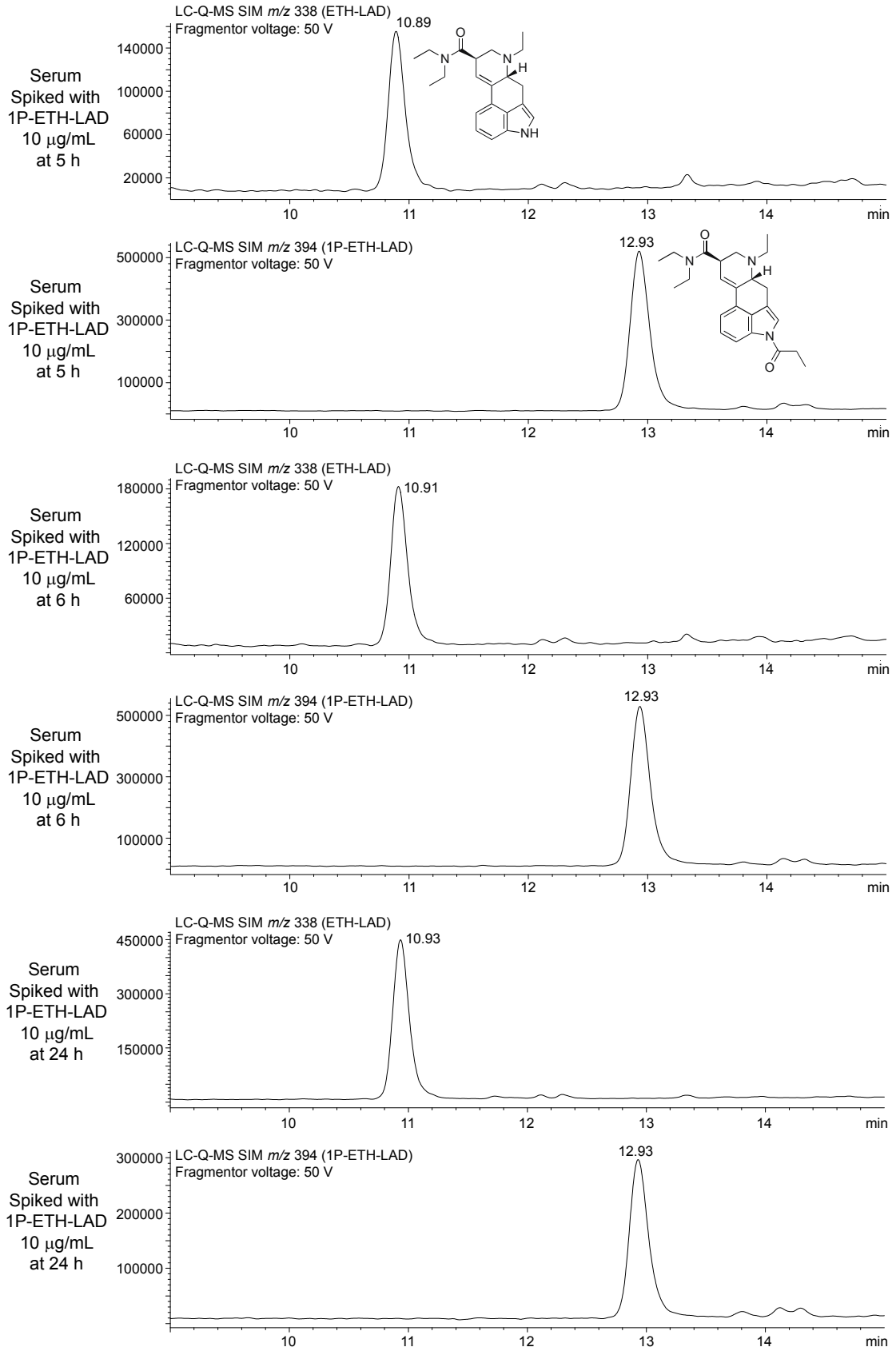
Fifty µL and 950 µL (acetonitrile/water, 1/1 + 0.1 % formic acid); centrifuged at 18,000 rpm for 3 min, then passed through a Nylon spin filter (0.2 µm). Samples were further diluted for LC-MS: 50 µL of the above and 950 µL (acetonitrile /water, 1/1 + 0.1 % formic acid).



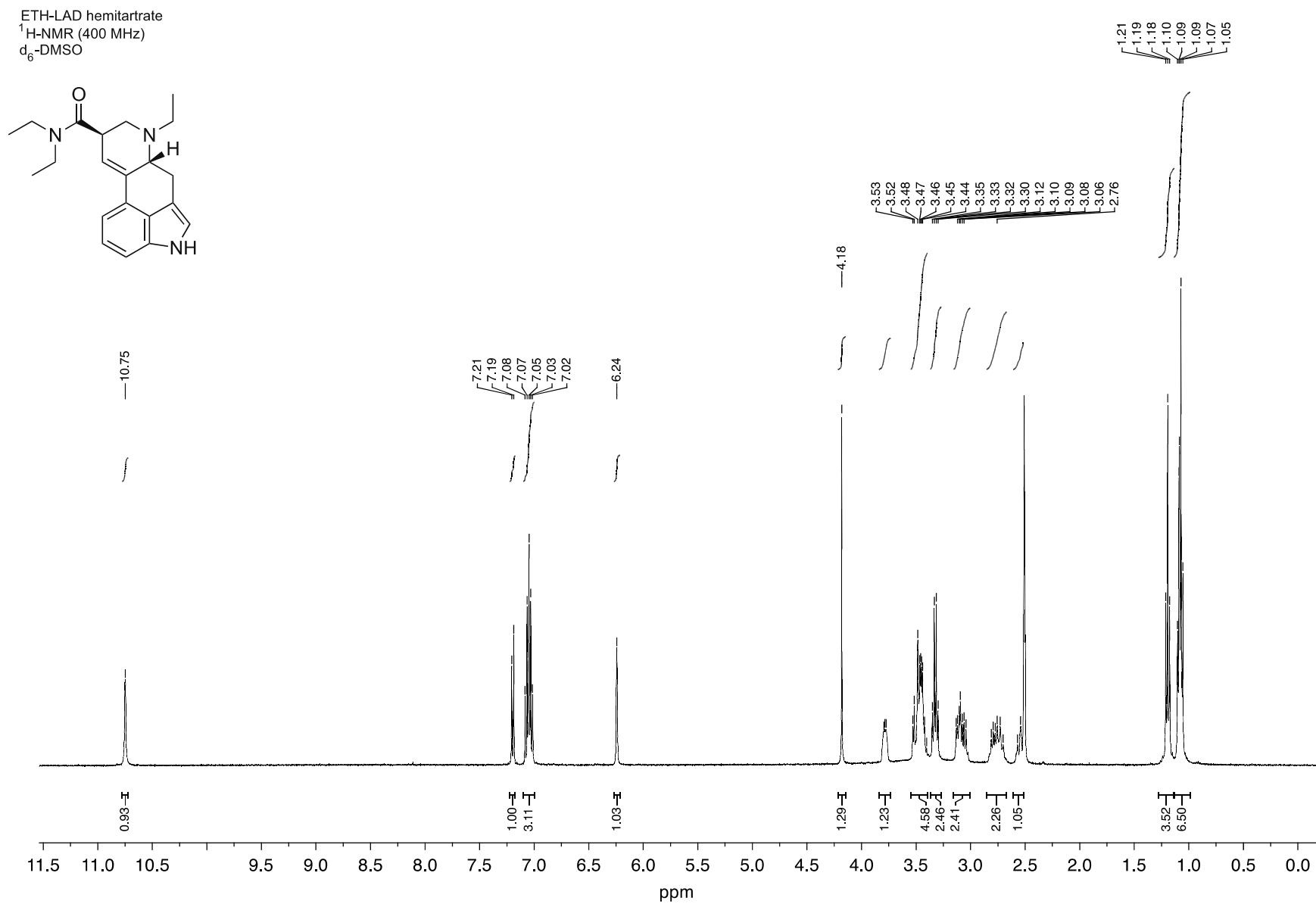
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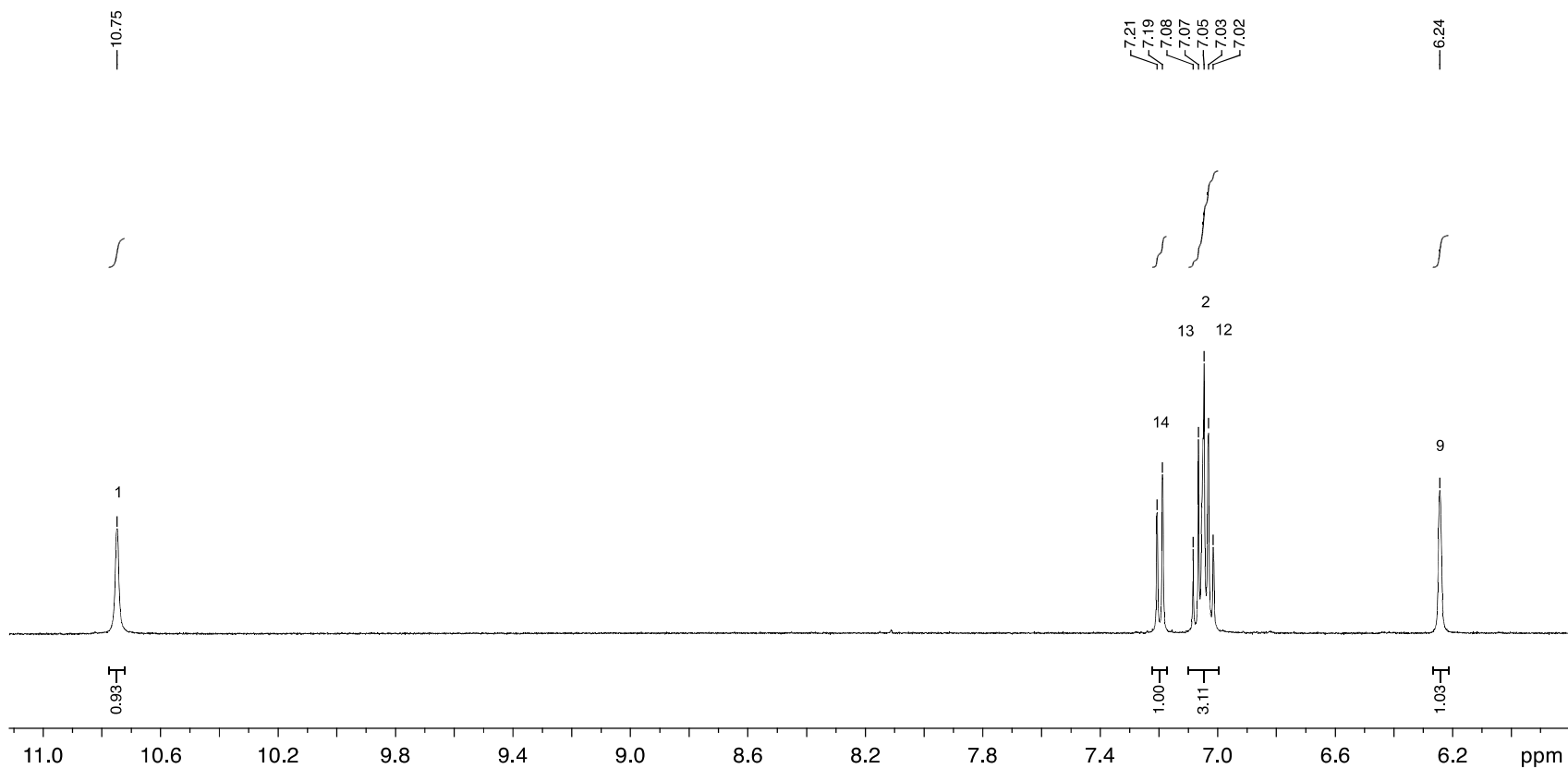
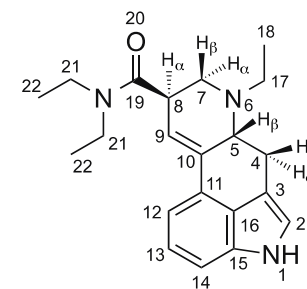


Drug Testing and Analysis – Brandt *et al.* – Supporting Information



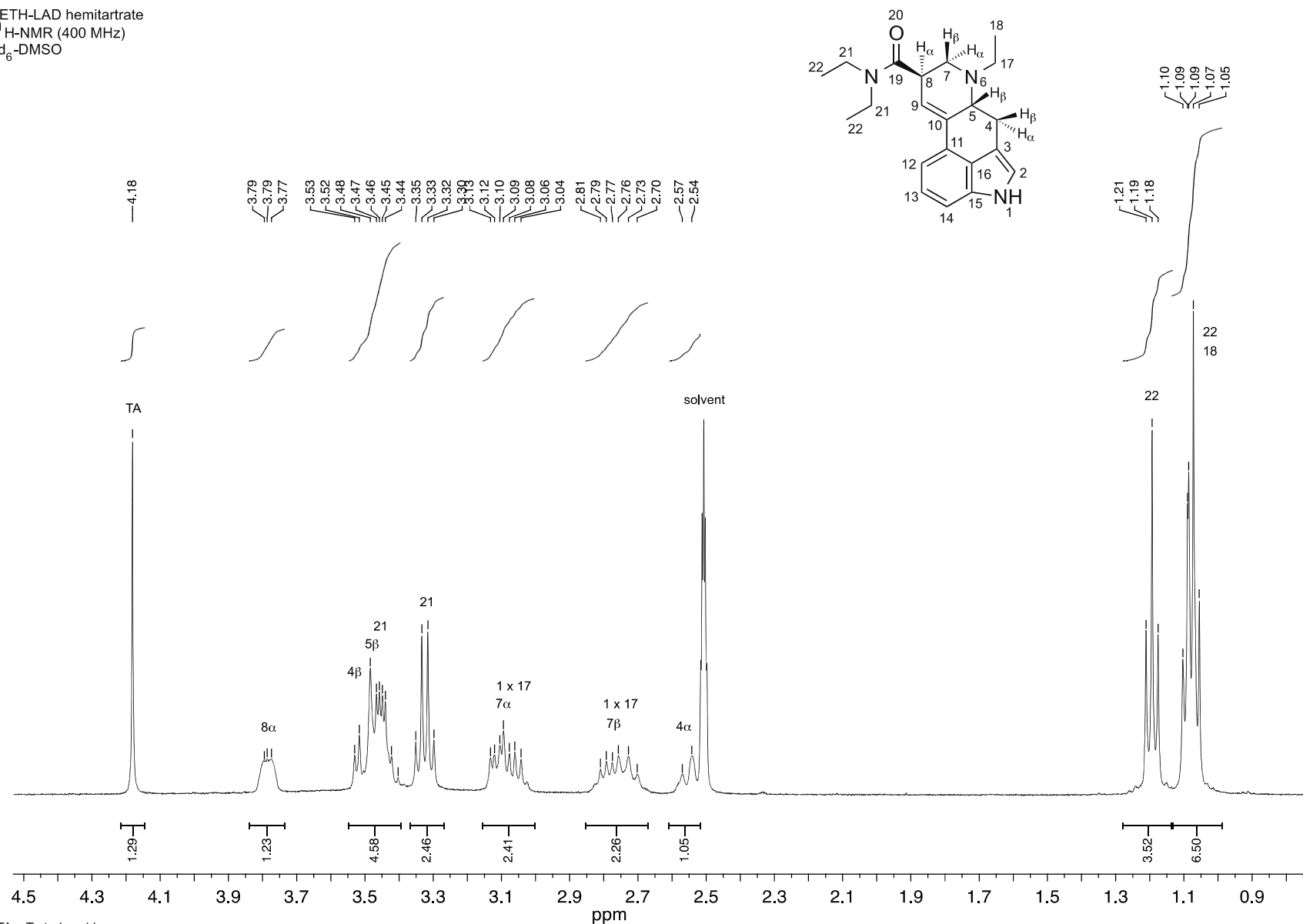
Drug Testing and Analysis – Brandt *et al.* – Supporting Information

ETH-LAD hemitartrate
¹H-NMR (400 MHz)
 d₆-DMSO



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

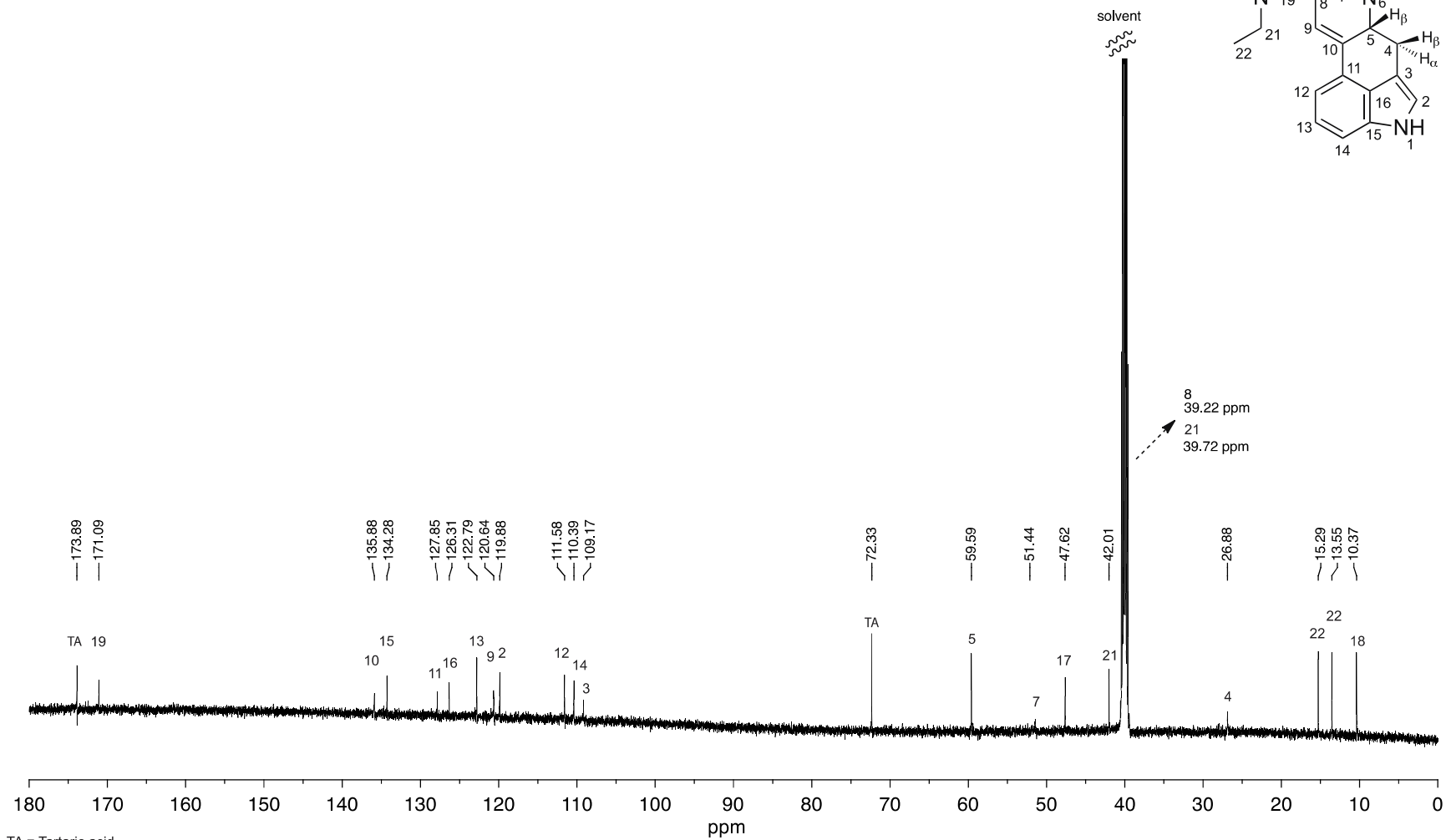
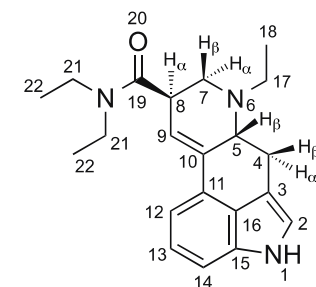
ETH-LAD hemitartrate
¹H-NMR (400 MHz)
d₆-DMSO



TA = Tartaric acid

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

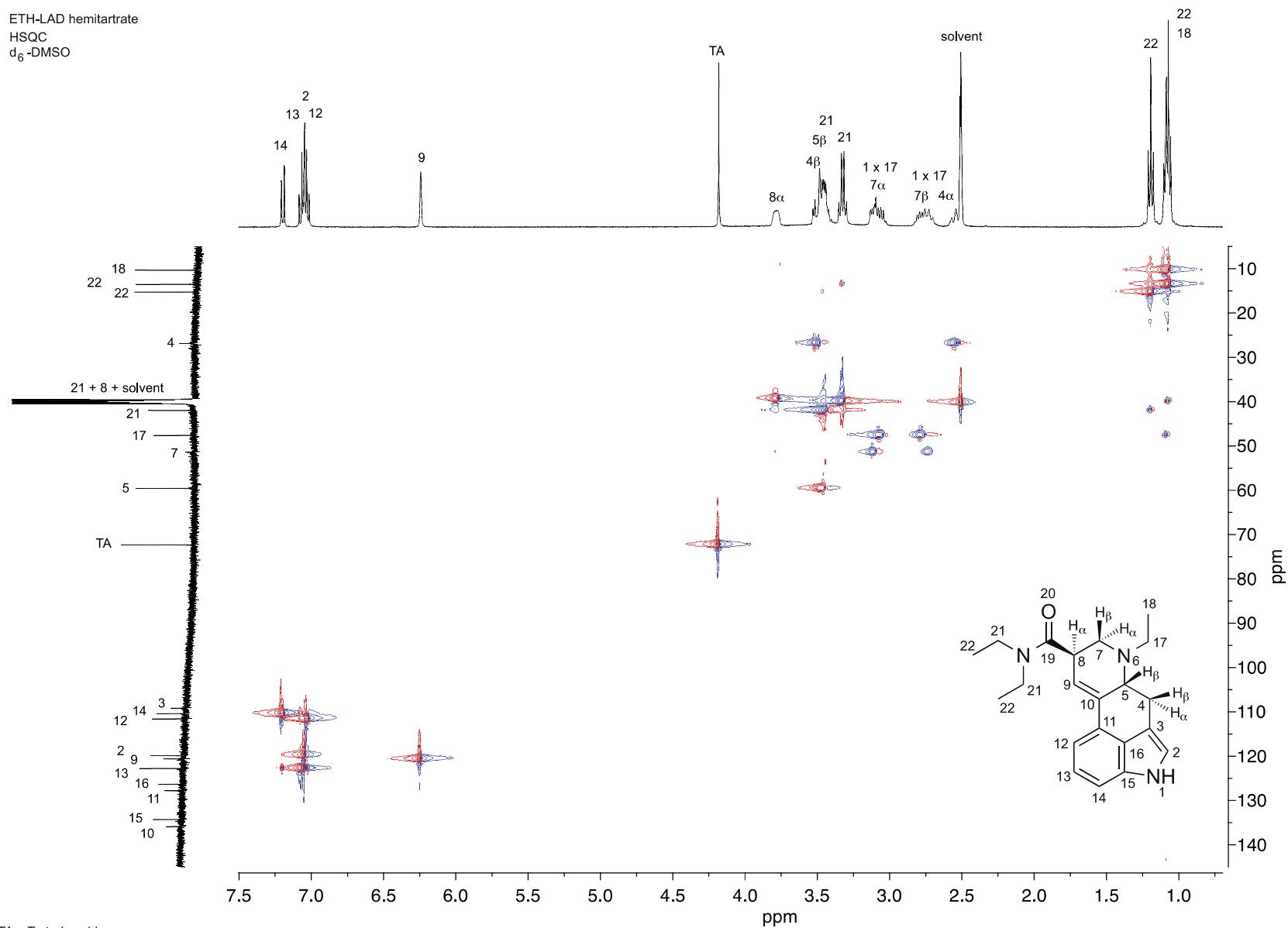
ETH-LAD hemitartrate
¹³C-NMR (150 MHz)
 d₆-DMSO



TA = Tartaric acid

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

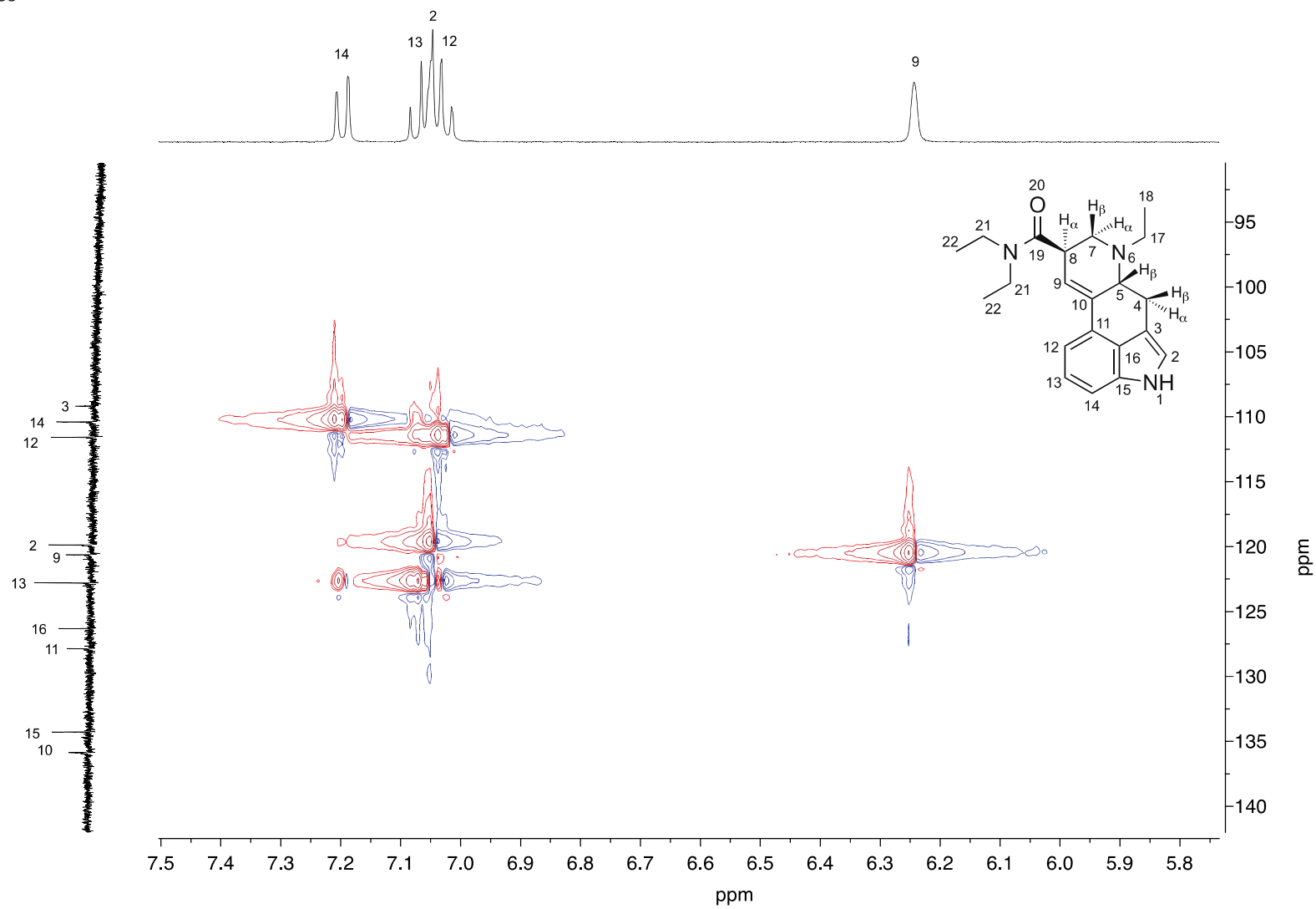
ETH-LAD hemitartrate
 HSQC
 d₆-DMSO



TA = Tartaric acid

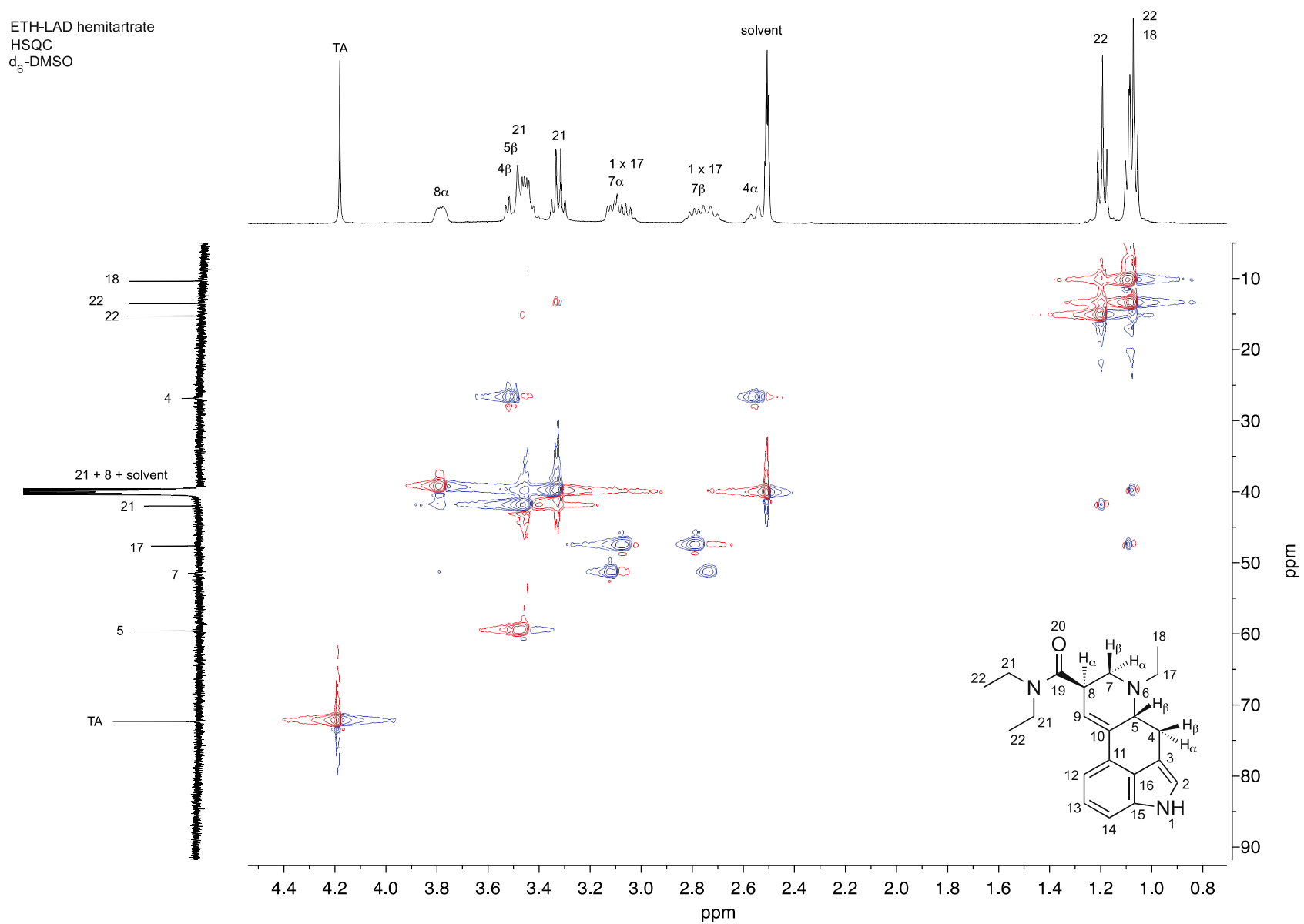
Drug Testing and Analysis – Brandt *et al.* – Supporting Information

ETH-LAD hemitartrate
 HSQC
 d₆-DMSO



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

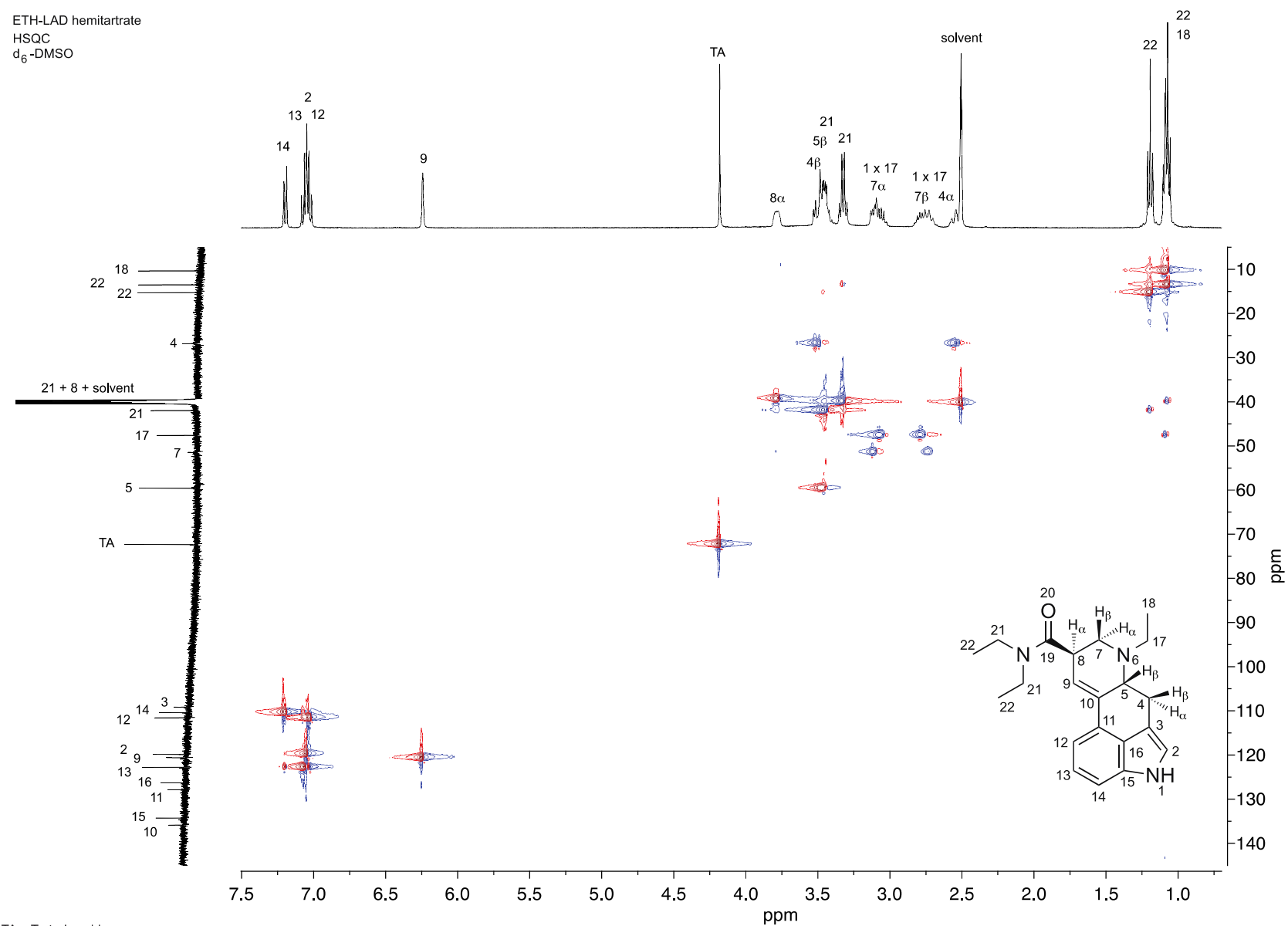
ETH-LAD hemitartrate
HSQC
d₆-DMSO



TA = Tartaric acid

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

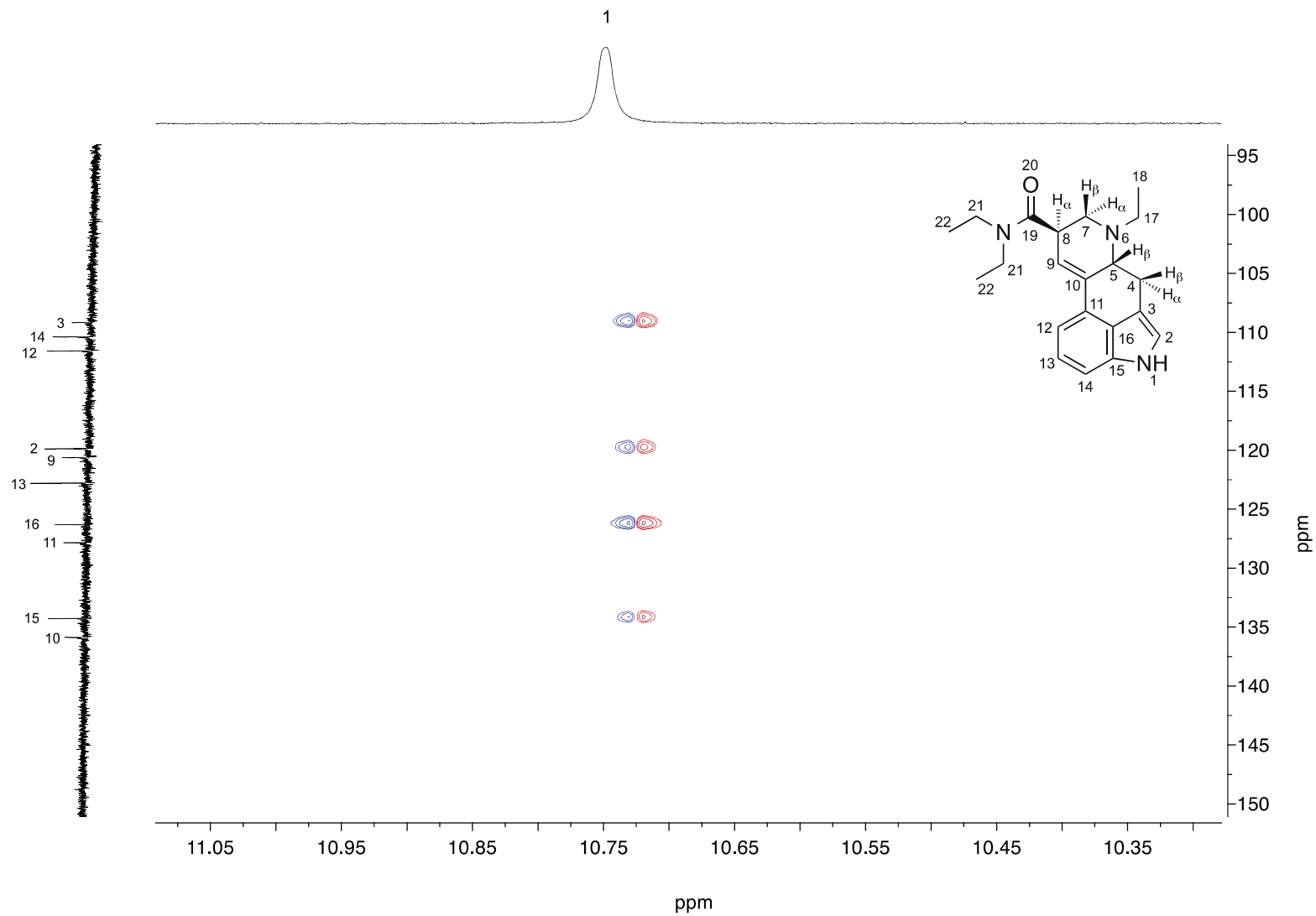
ETH-LAD hemitartrate
 HSQC
 d₆-DMSO



TA = Tartaric acid

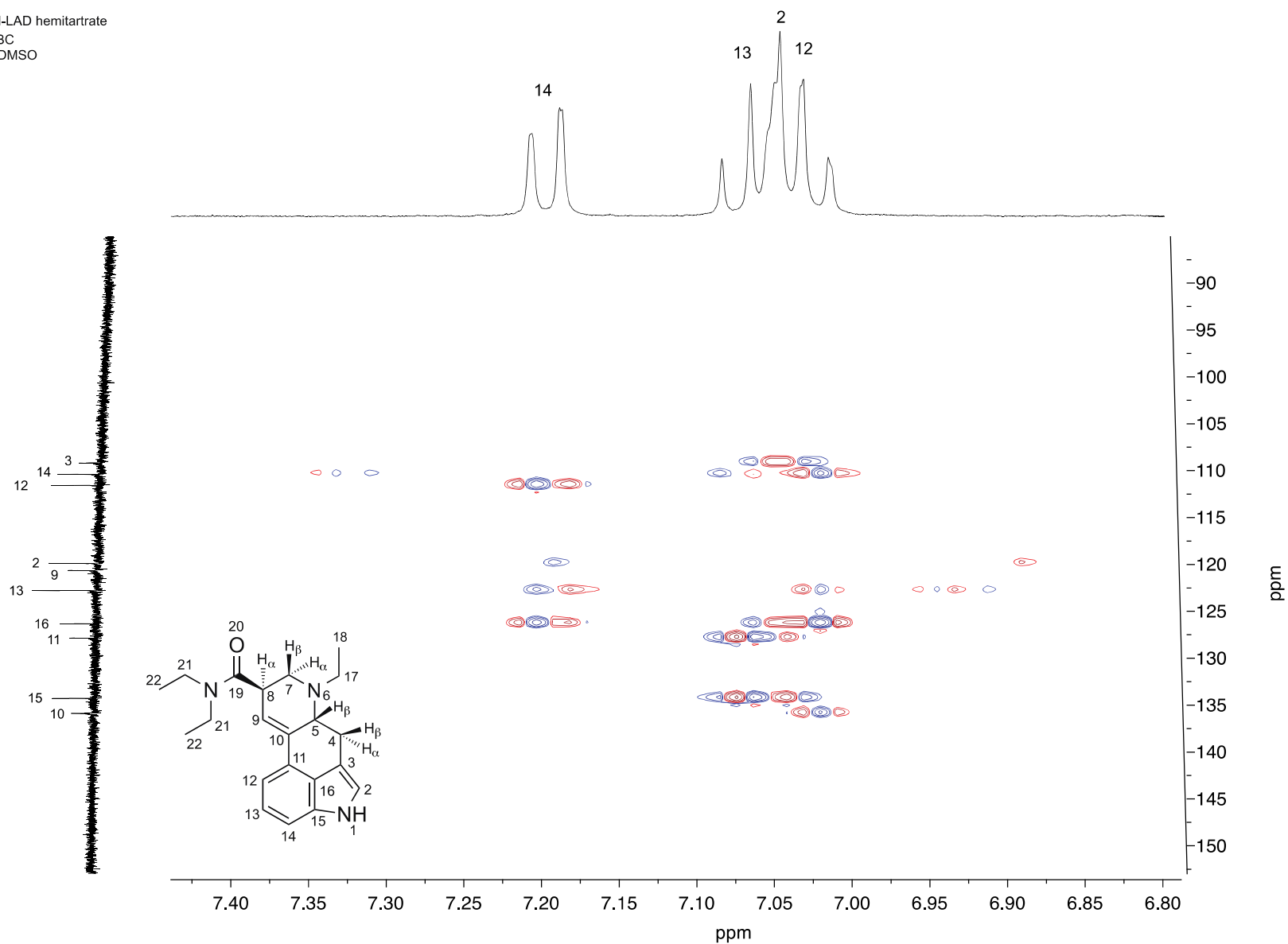
Drug Testing and Analysis – Brandt *et al.* – Supporting Information

ETH-LAD hemitartrate
HMBC
d₆-DMSO



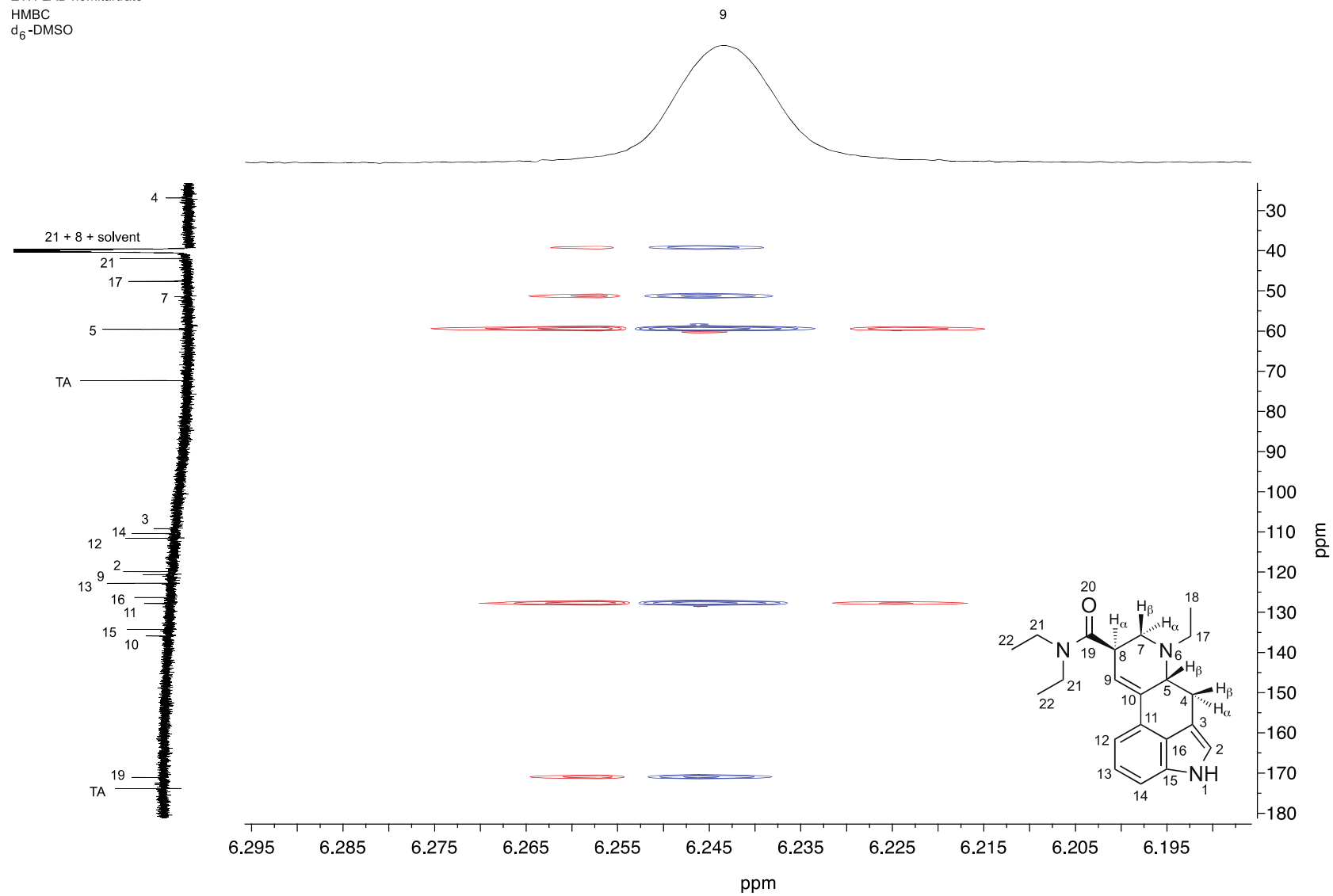
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ETH-LAD hemitartrate
 HMBC
 d₆-DMSO



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

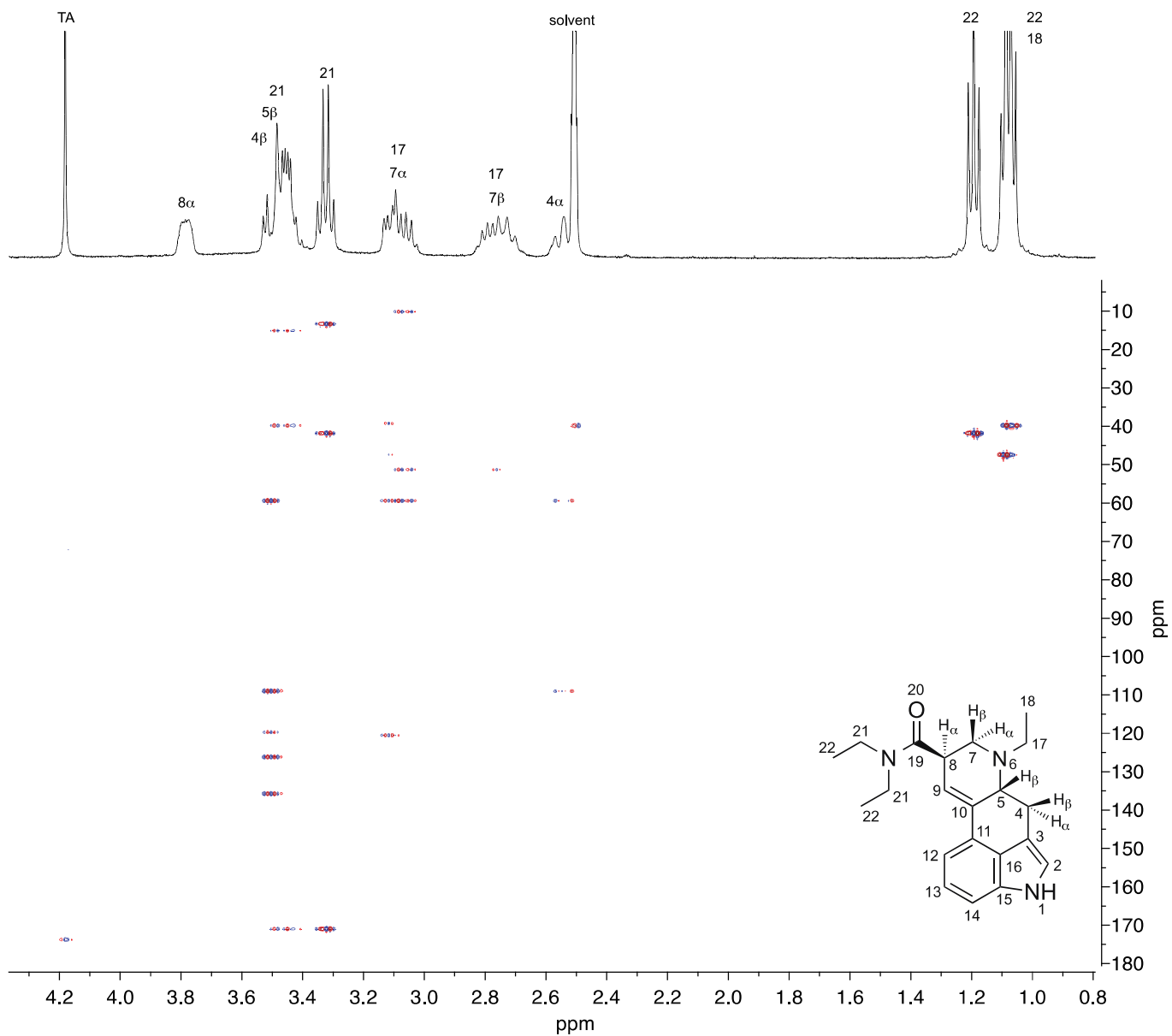
ETH-LAD hemitartrate
 HMBC
 d₆-DMSO



TA = Tartaric acid

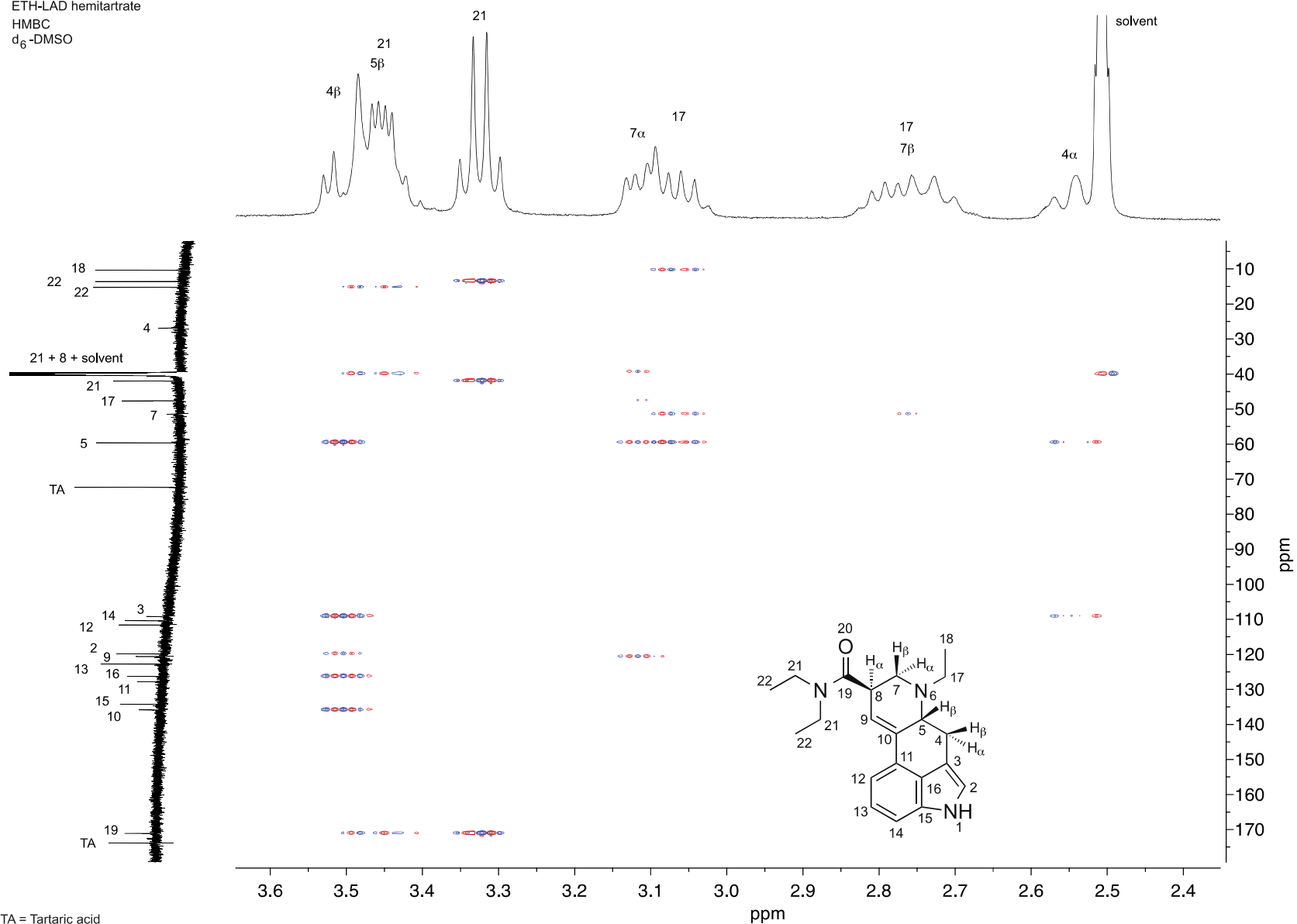
Drug Testing and Analysis – Brandt *et al.* – Supporting Information

ETH-LAD hemitartrate
 HMBC
 d₆-DMSO



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

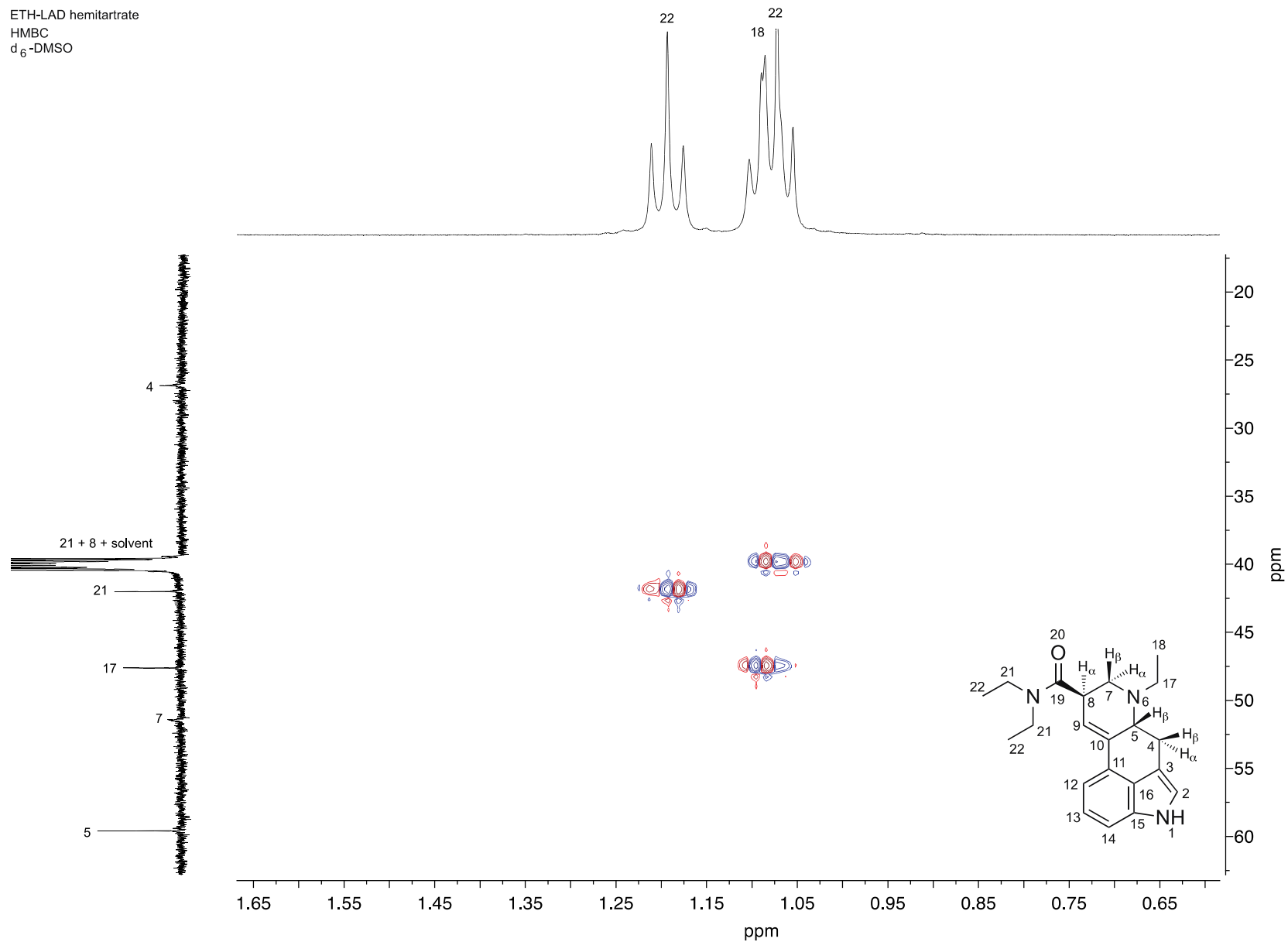
ETH-LAD hemitartrate
 HMBC
 d_6 -DMSO



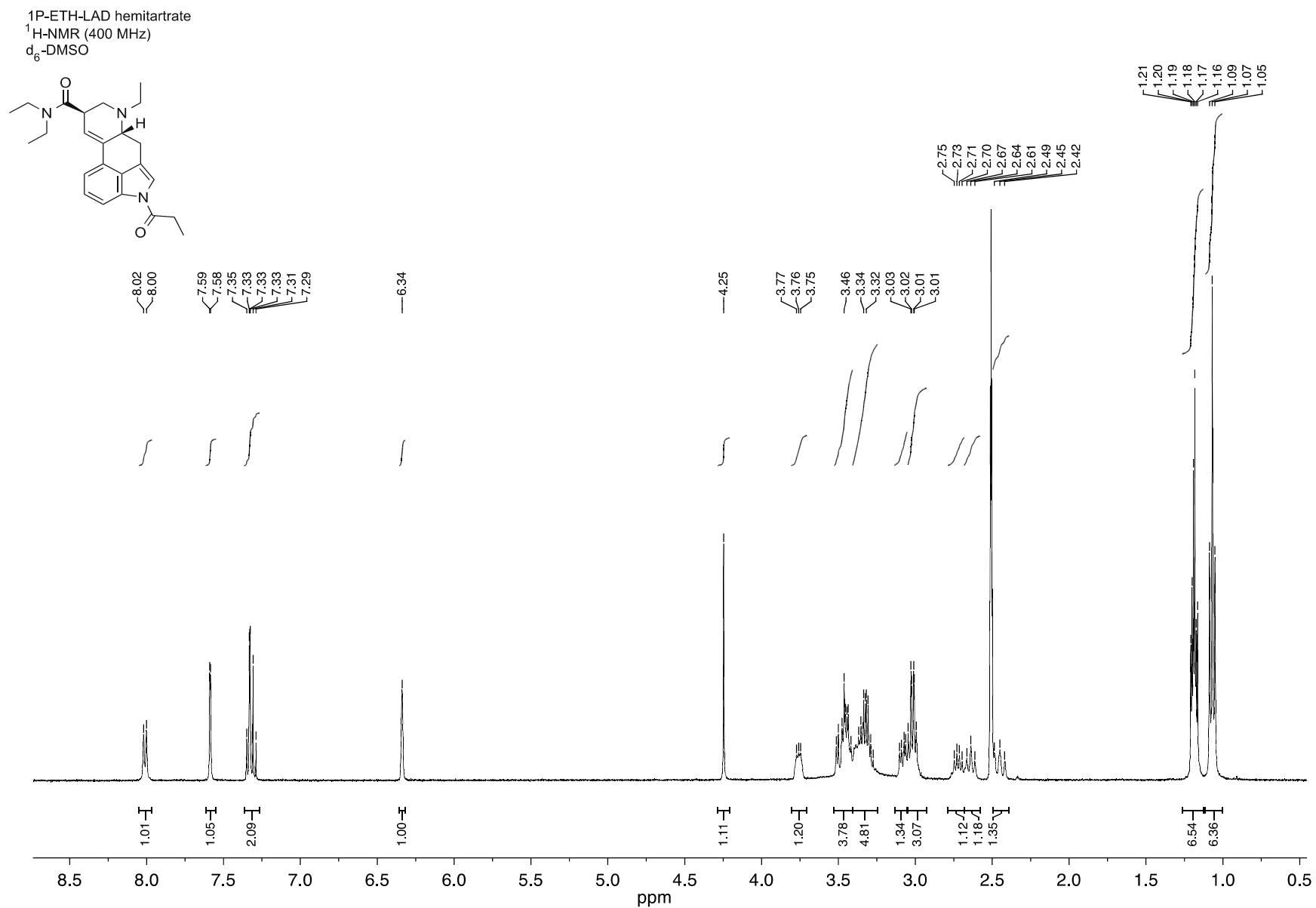
TA = Tartaric acid

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

ETH-LAD hemitartrate
 HMBC
 d₆-DMSO

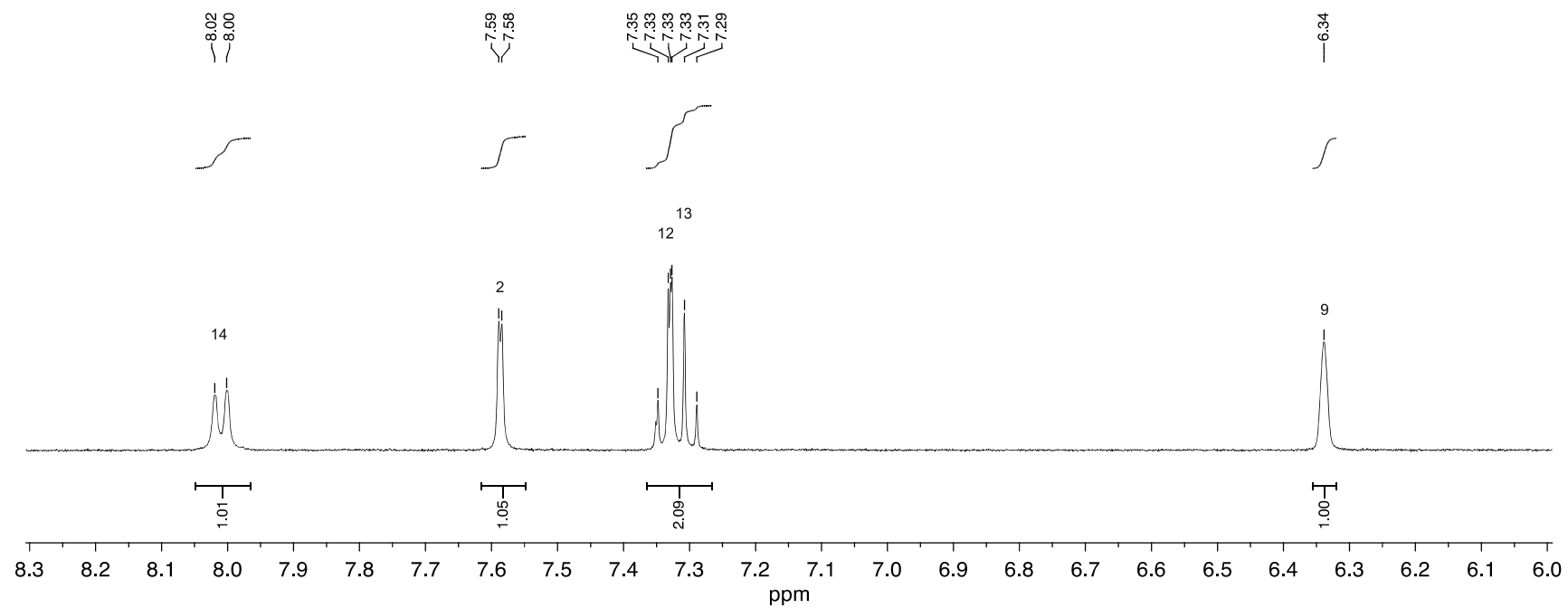
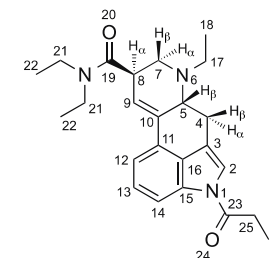


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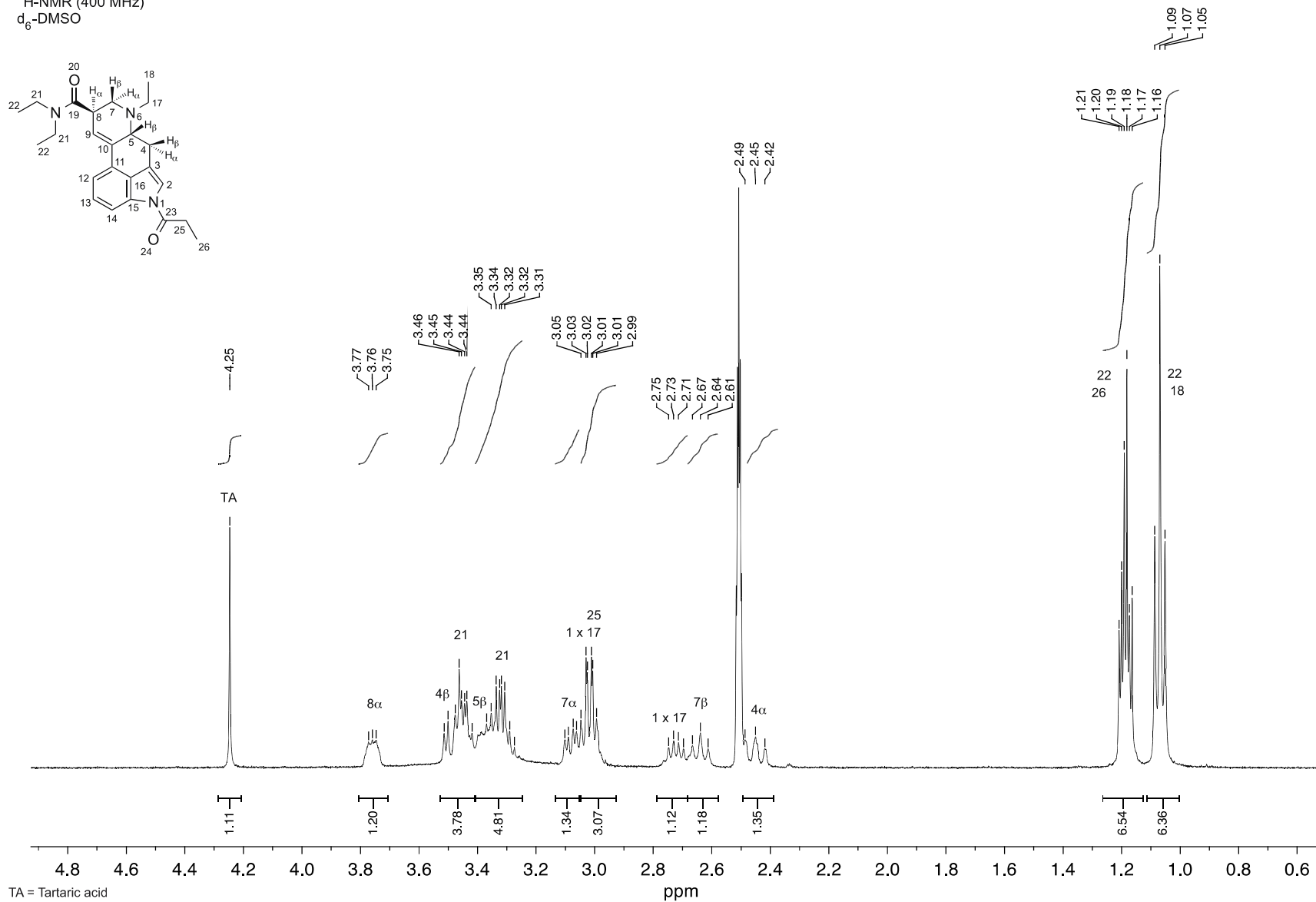
1P-ETH-LAD hemitartrate
¹H-NMR (400 MHz)
d₆-DMSO



1P-ETH-LAD hemitartrate

¹H-NMR (400 MHz)

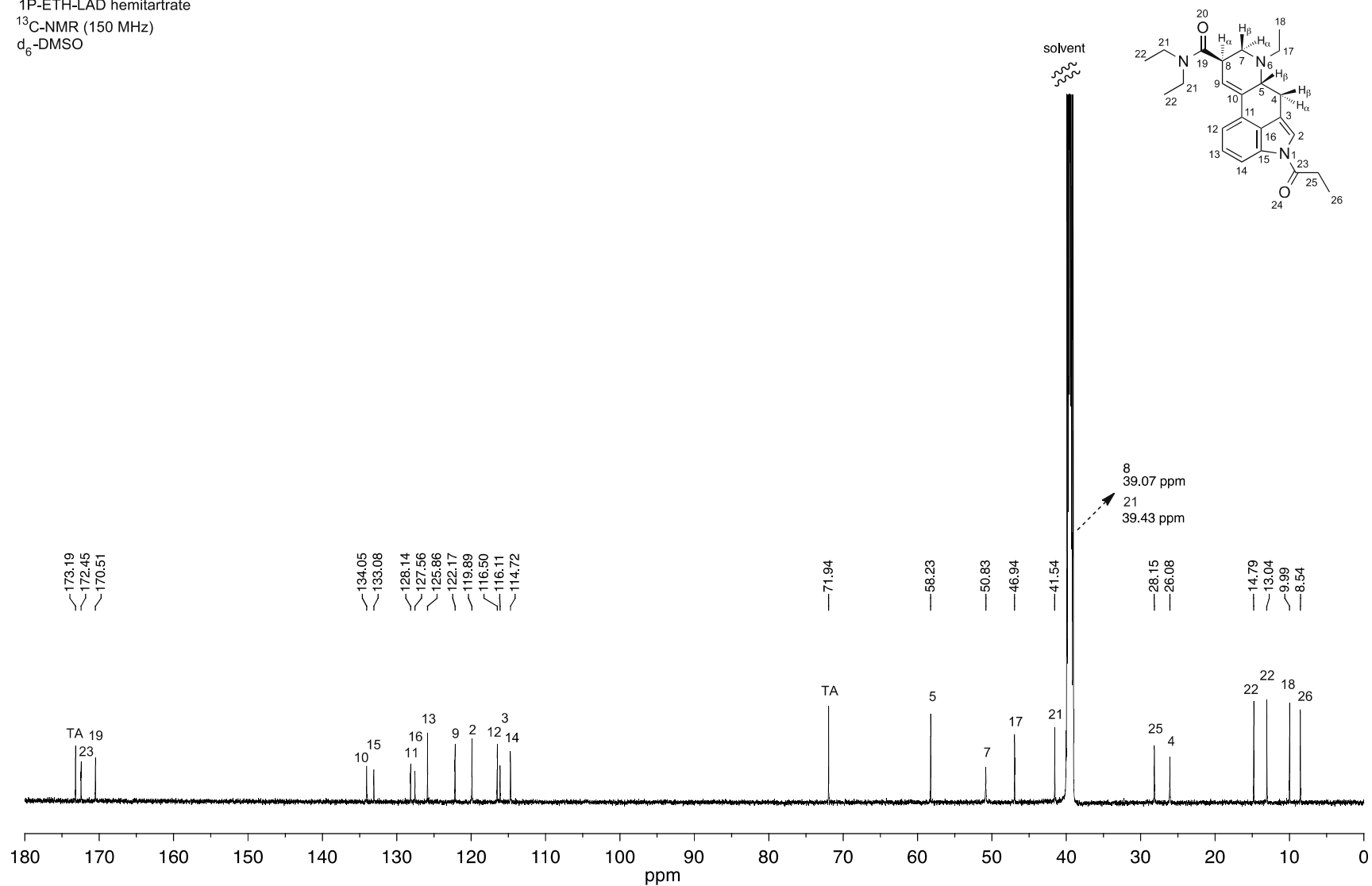
*d*₆-DMSO



TA = Tartaric acid

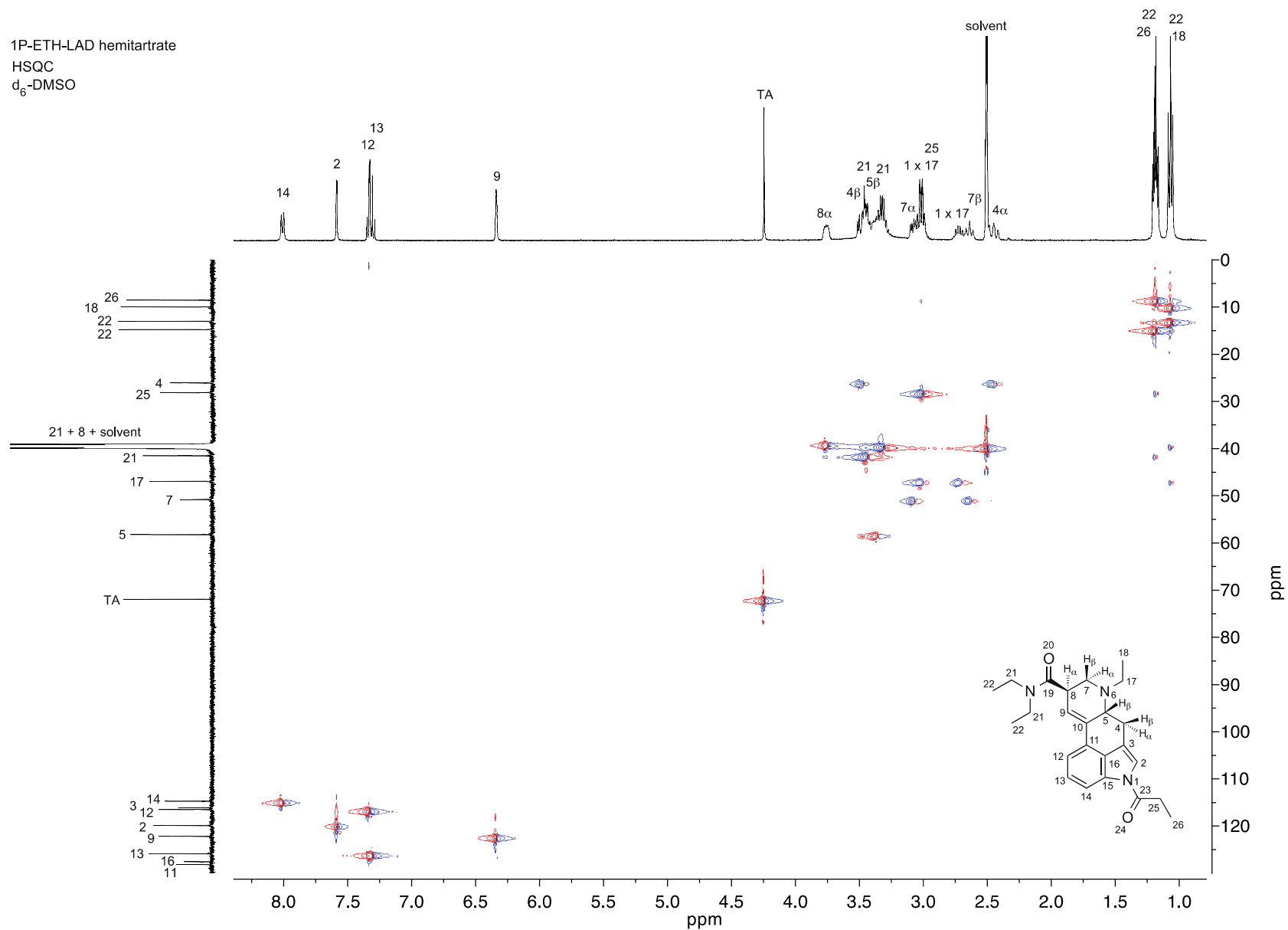
Drug Testing and Analysis – Brandt *et al.* – Supporting Information

1P-ETH-LAD hemitartrate
¹³C-NMR (150 MHz)
 d₆-DMSO



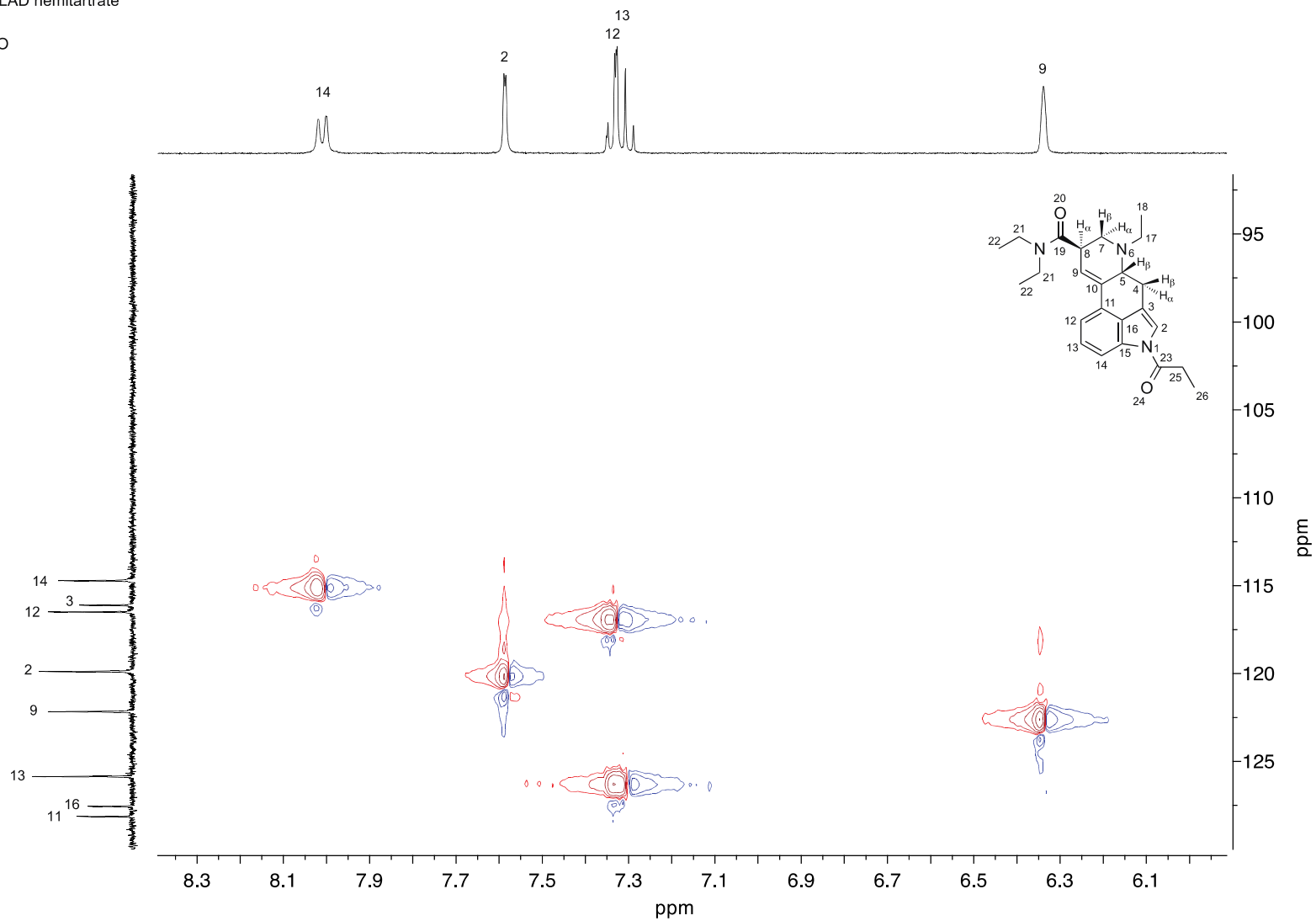
TA = Tartaric acid

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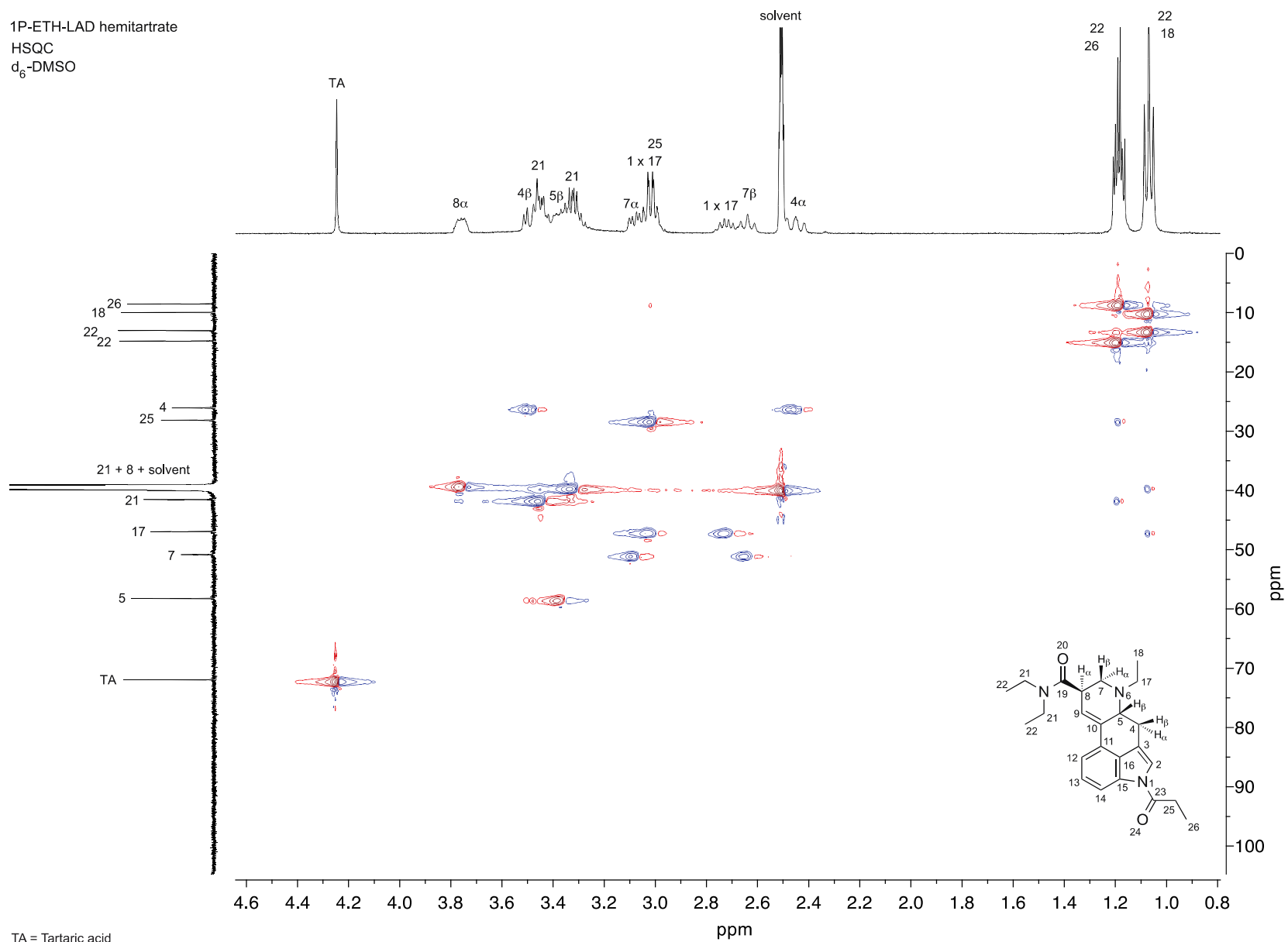
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1P-ETH-LAD hemitartrate
HSQC
d₆-DMSO



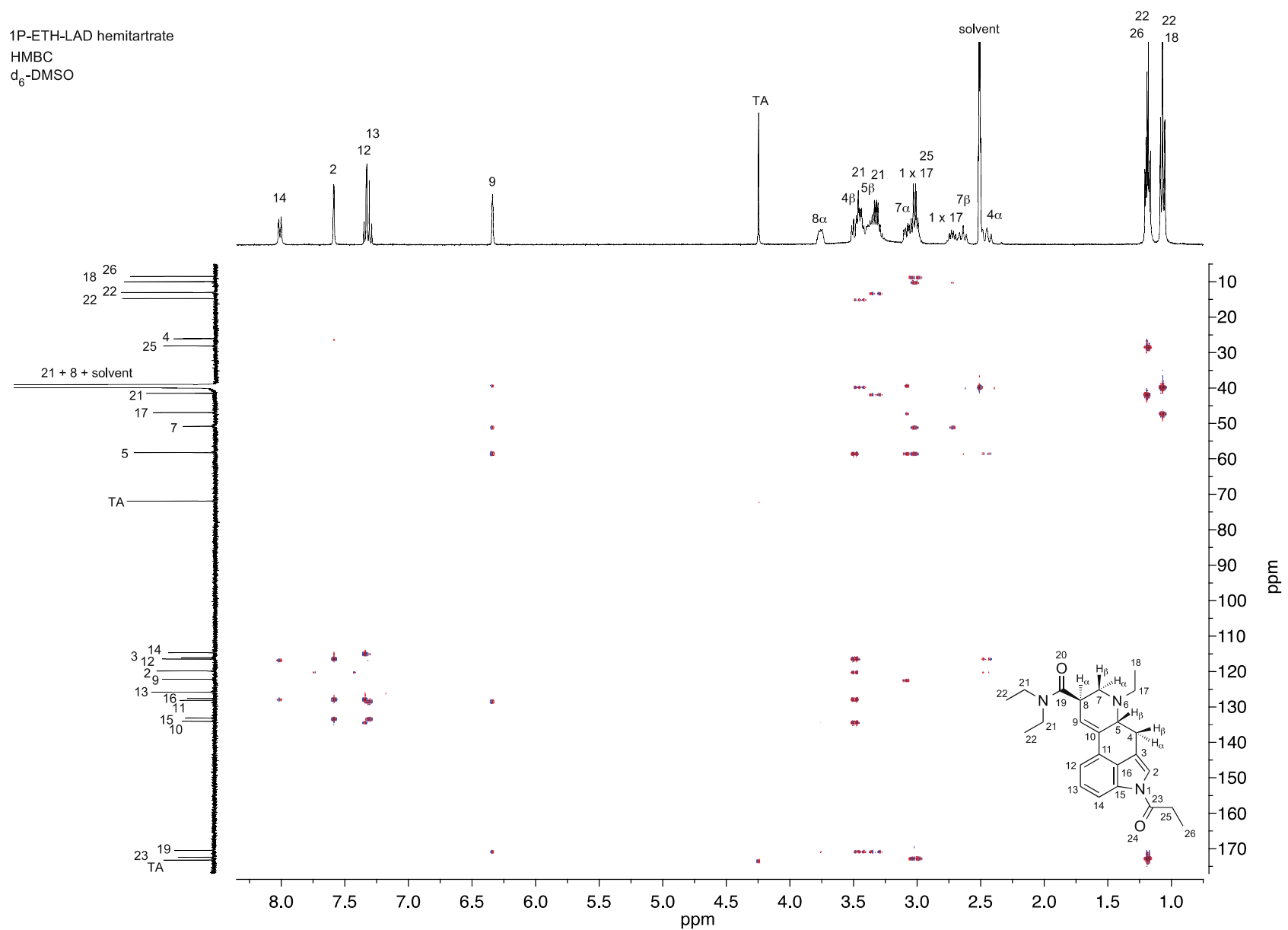
Drug Testing and Analysis – Brandt *et al.* – Supporting Information

1P-ETH-LAD hemitartrate
HSQC
d₆-DMSO



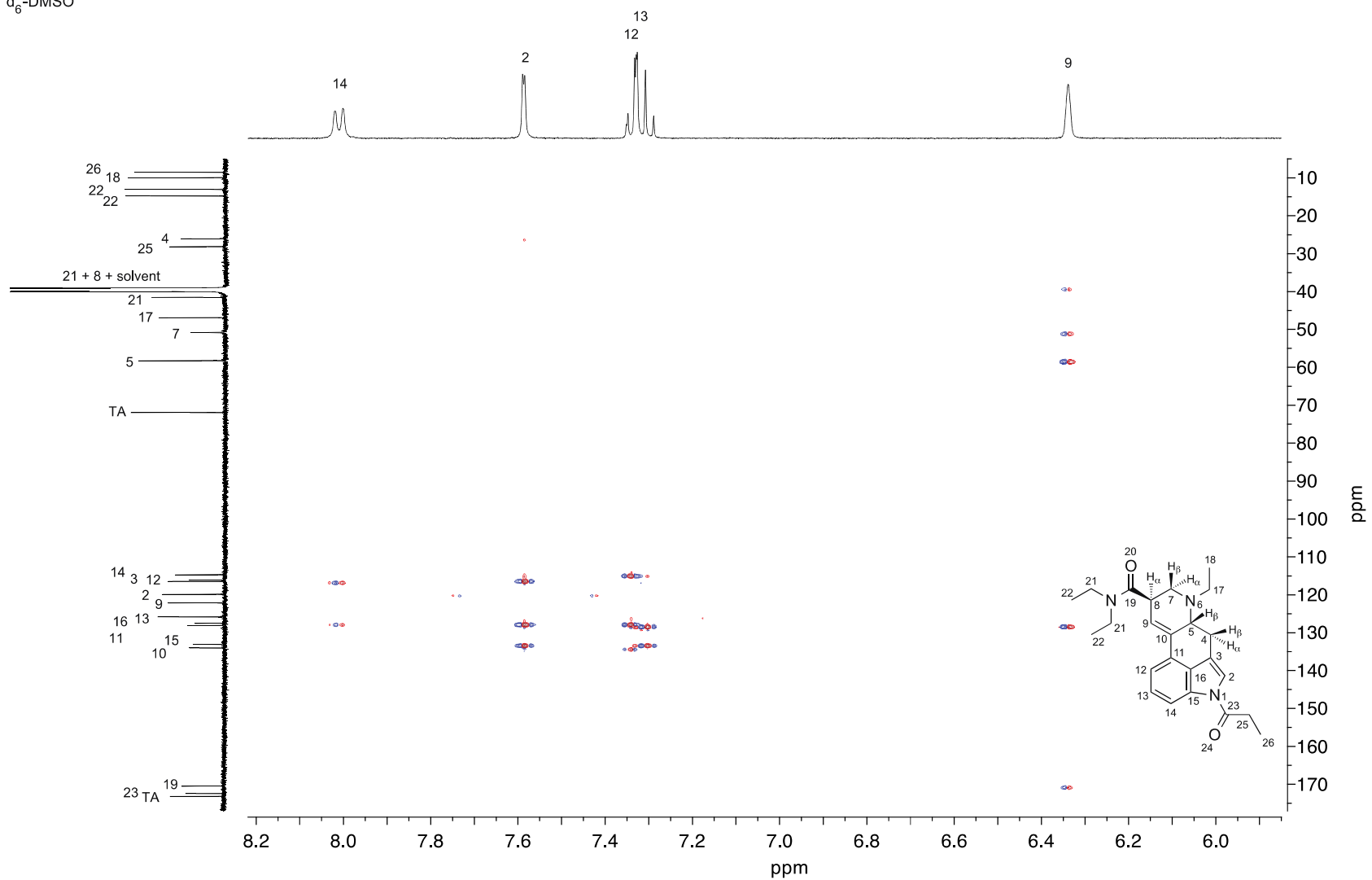
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1P-ETH-LAD hemitartrate
HMBC
d₆-DMSO



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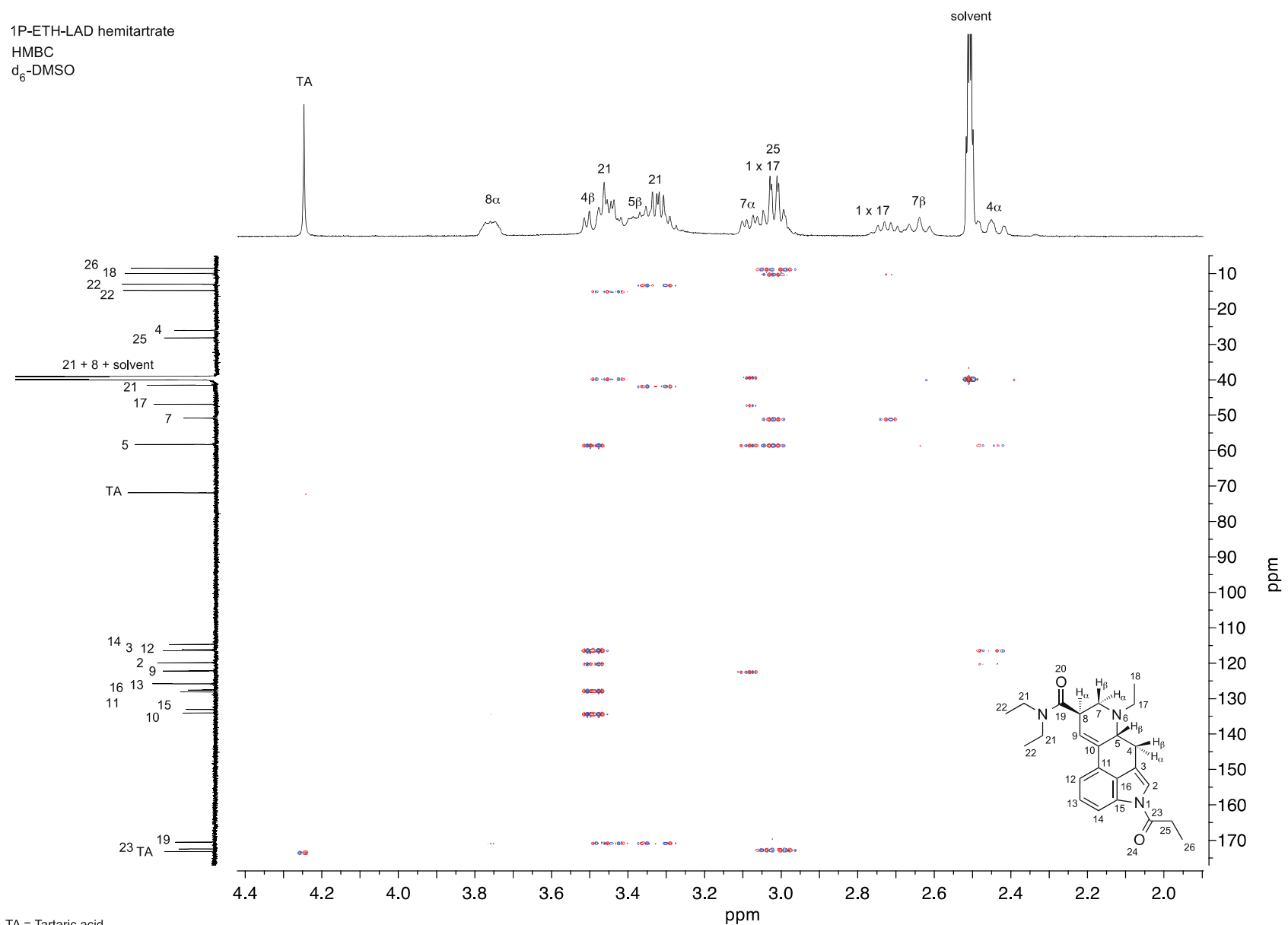
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 HMBC
 d₆-DMSO



TA = Tartaric acid

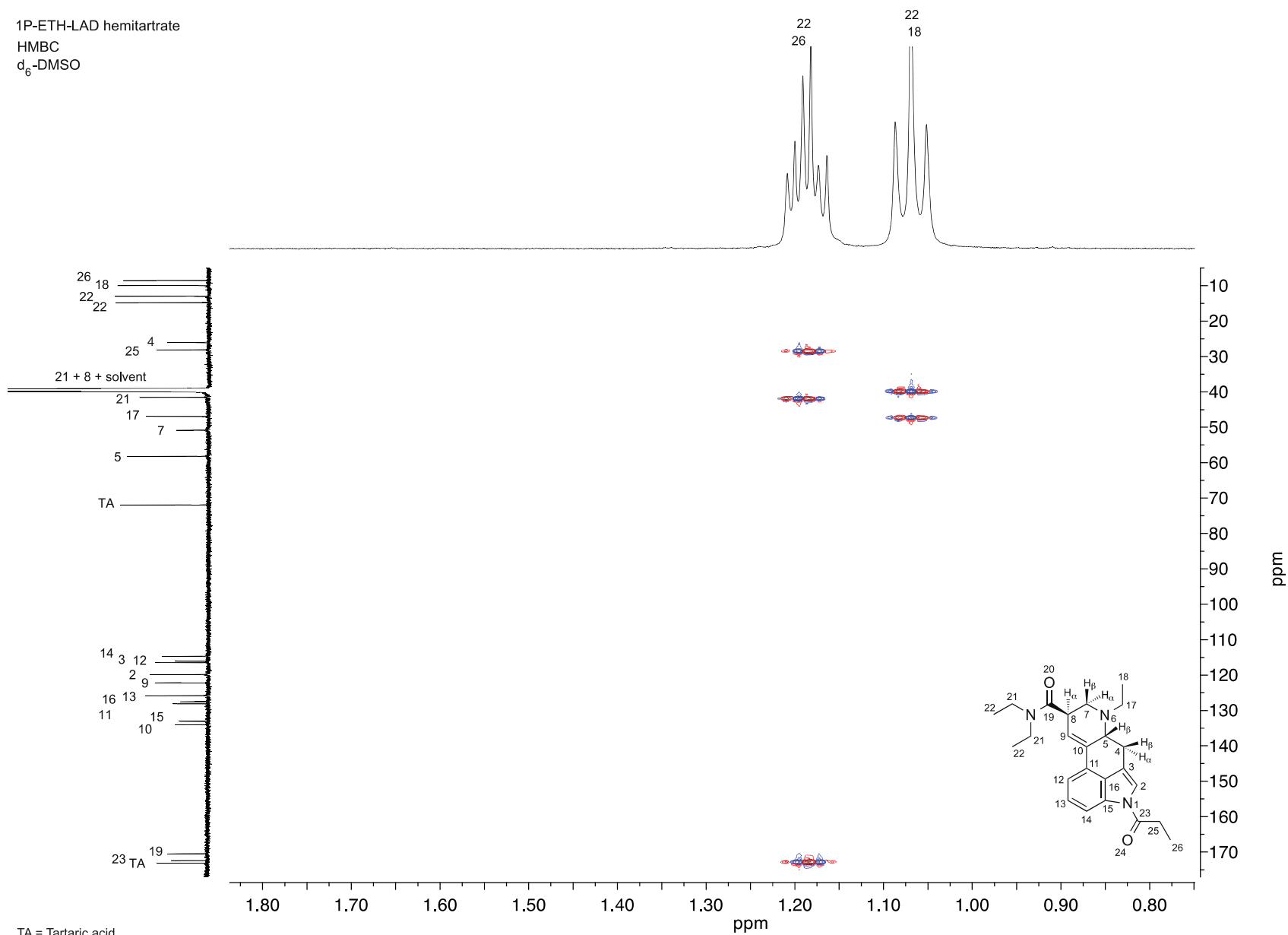
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1P-ETH-LAD hemitartrate
 HMBC
 d₆-DMSO



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1P-ETH-LAD hemitartrate
HMBC
d₆-DMSO



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