

Separating the wheat from the chaff: observations on the analysis of lysergamides LSD, MIPLA, and LAMPA

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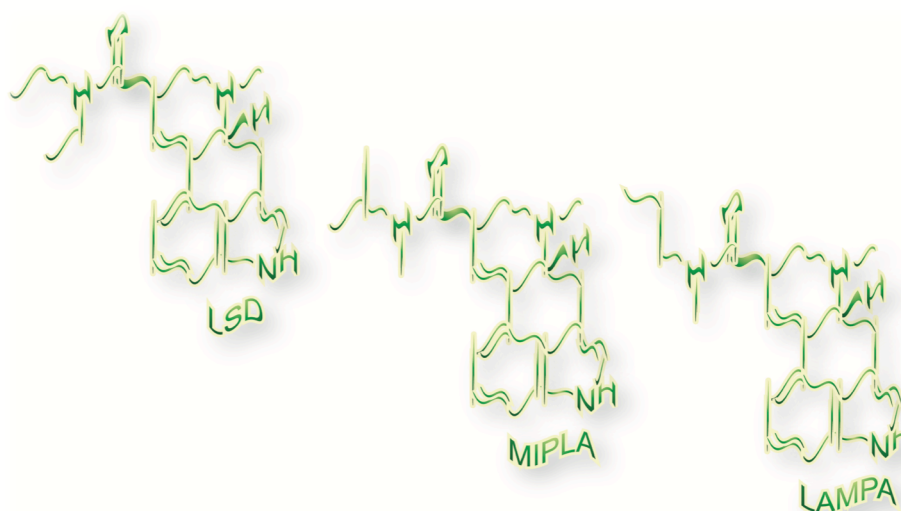
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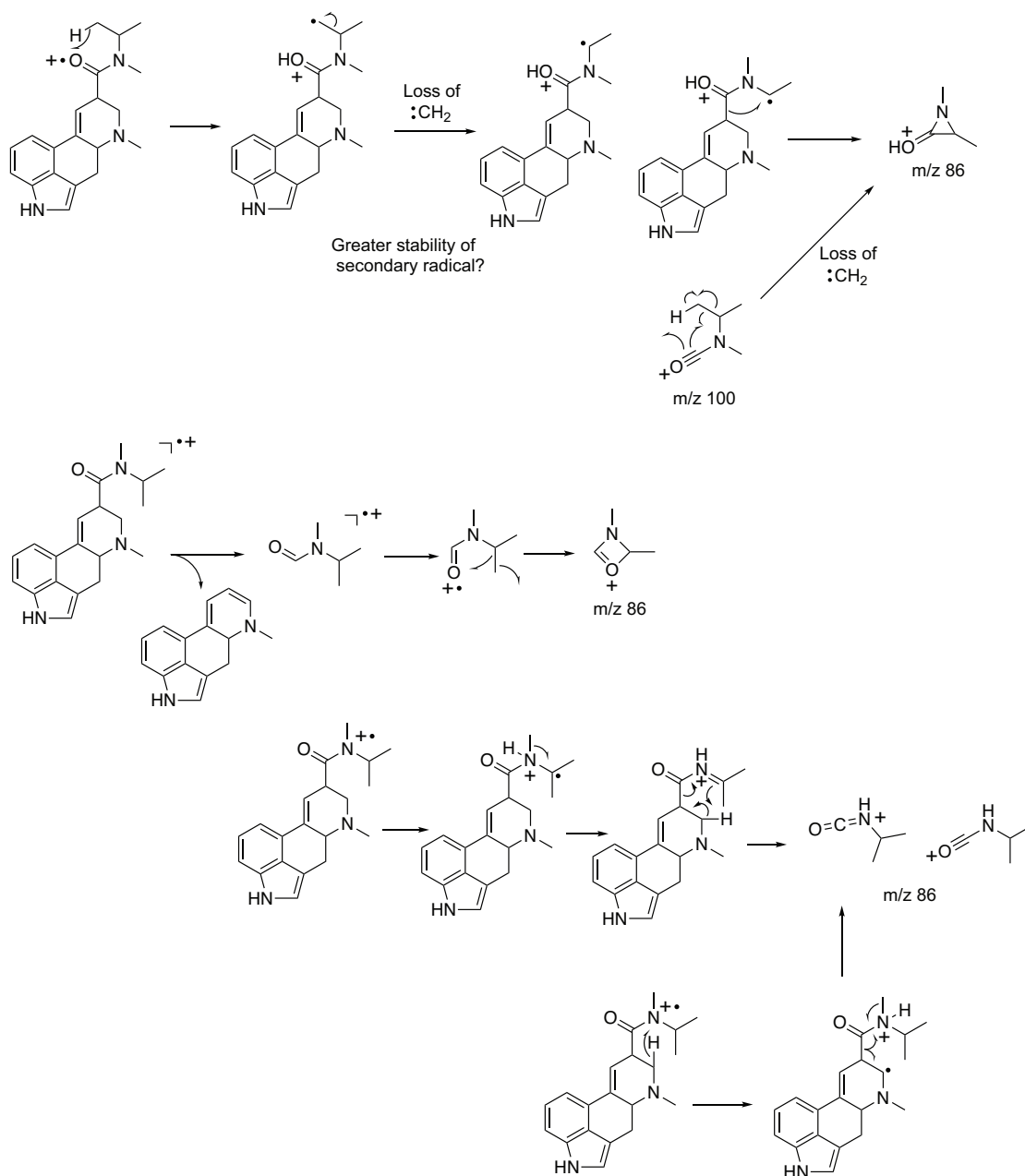
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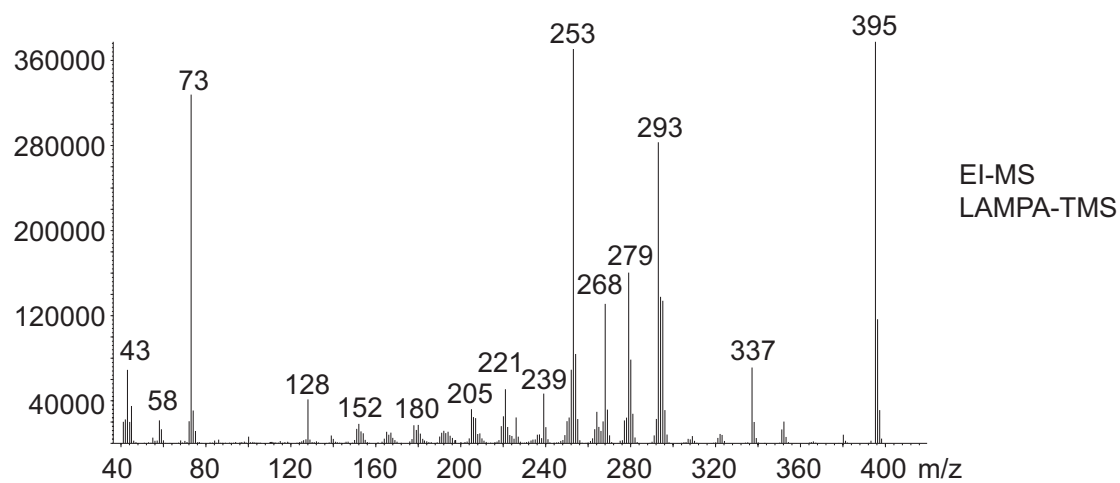
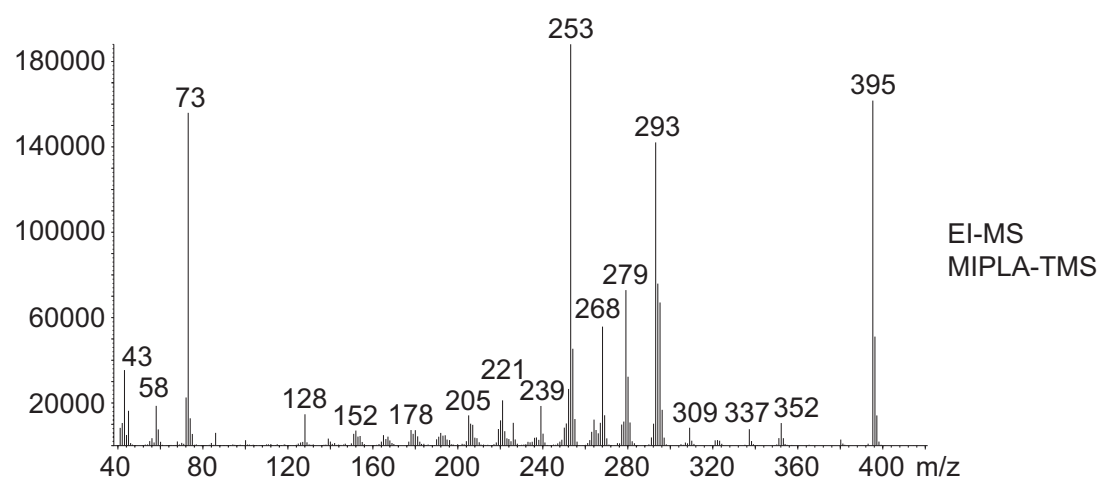
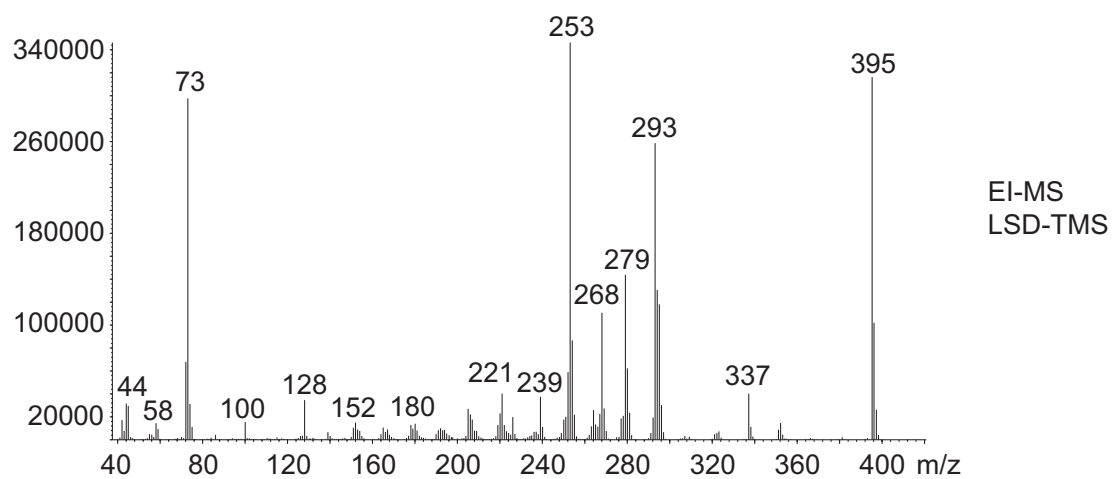


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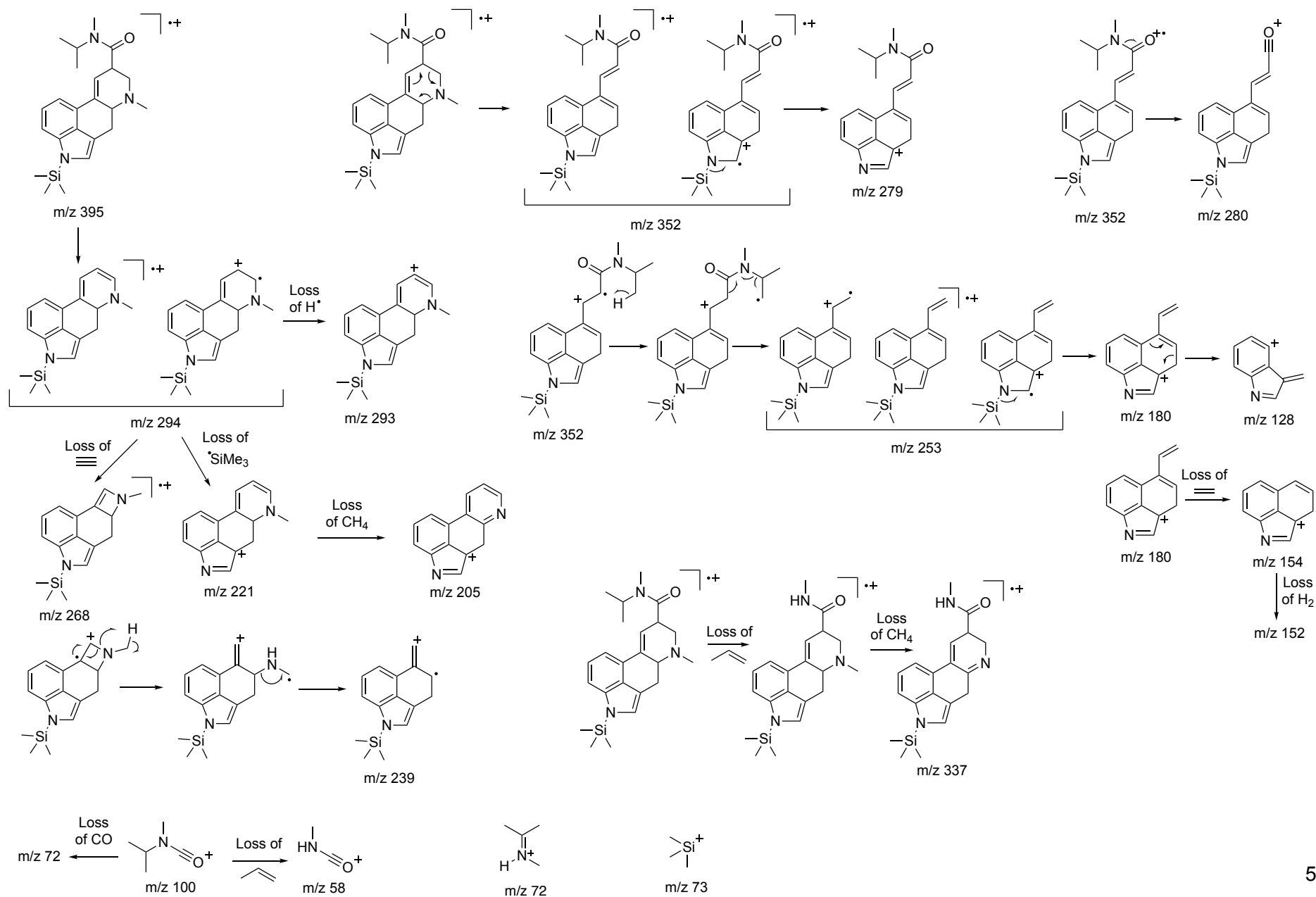
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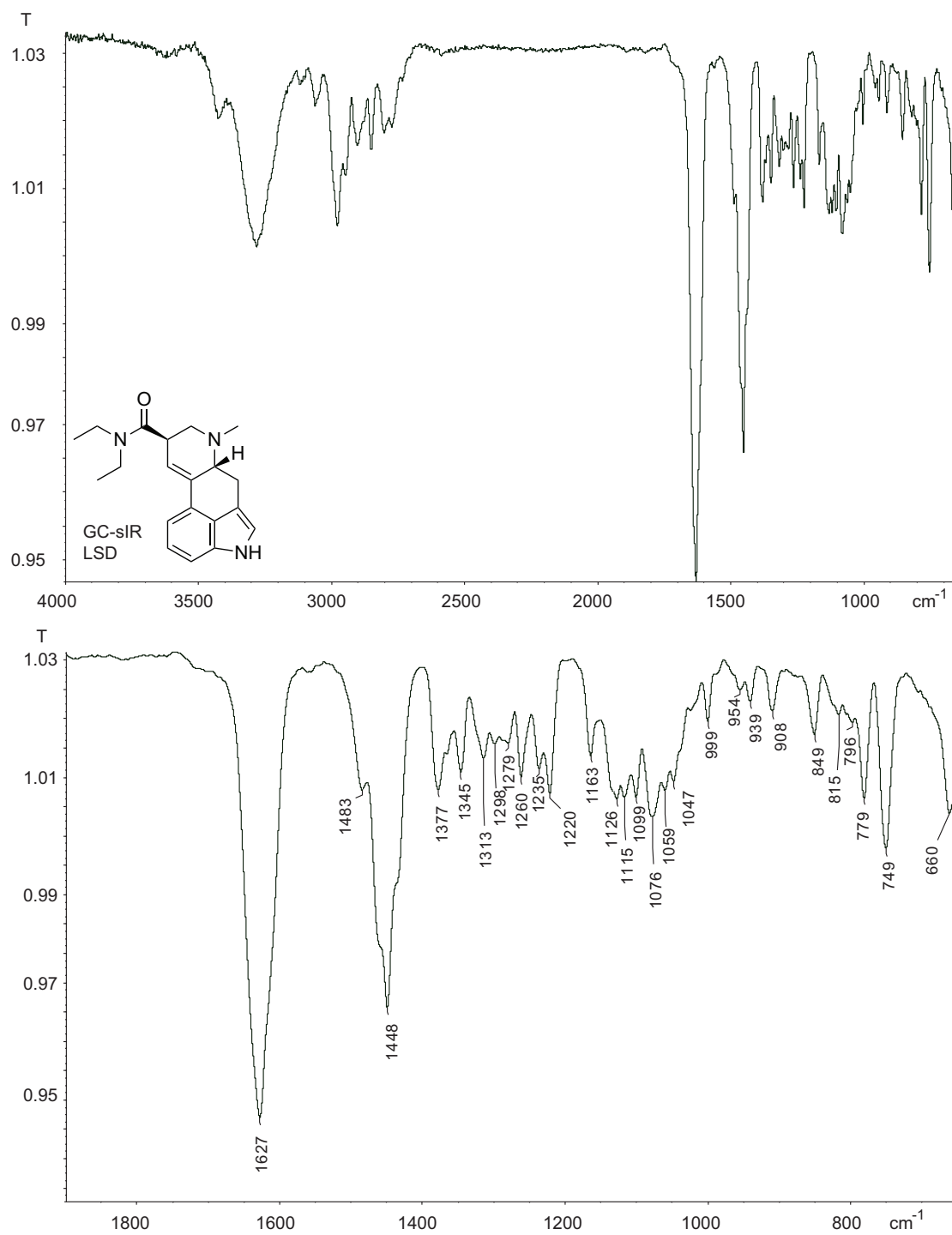
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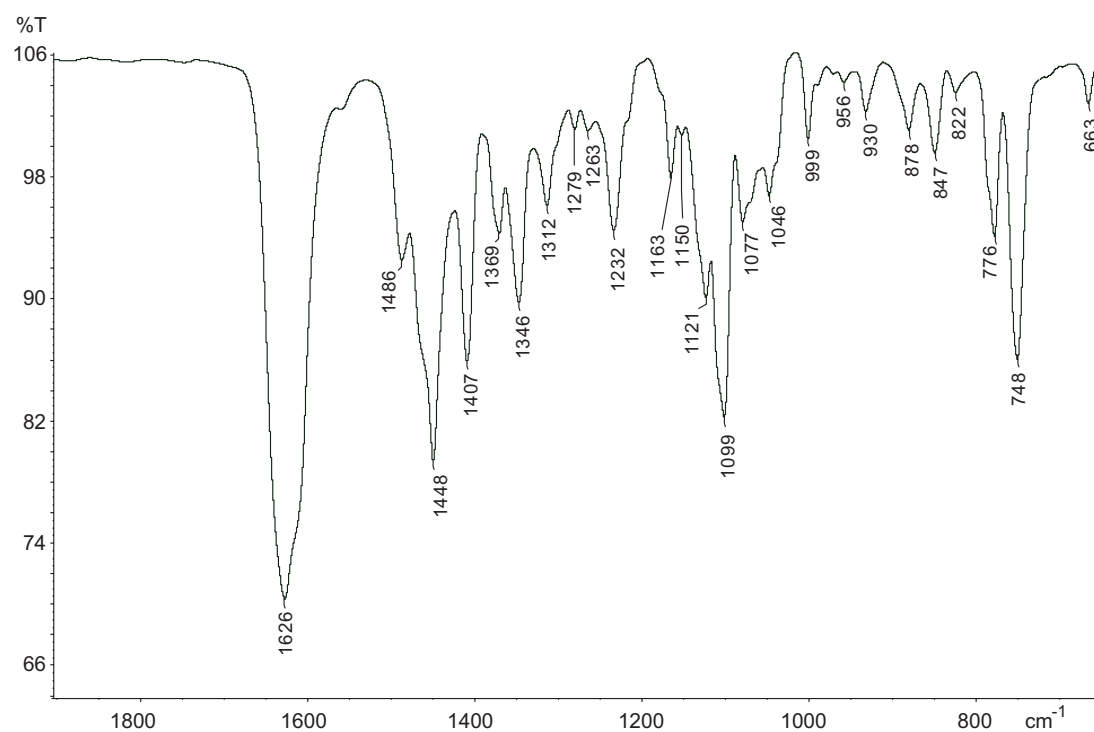
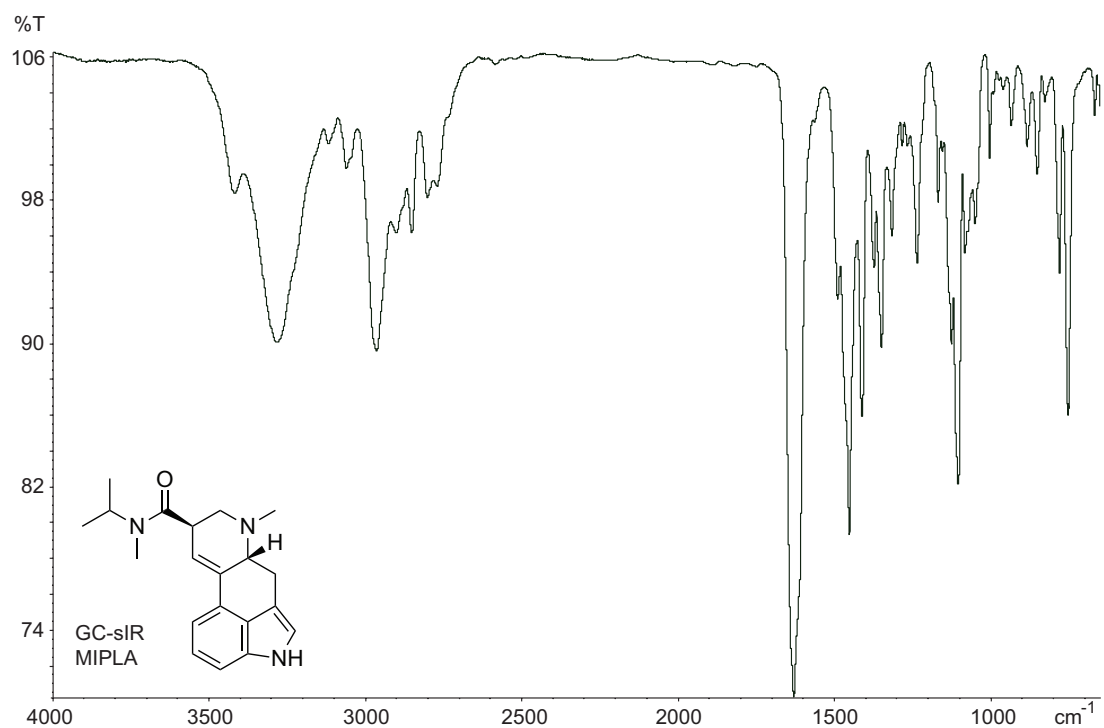
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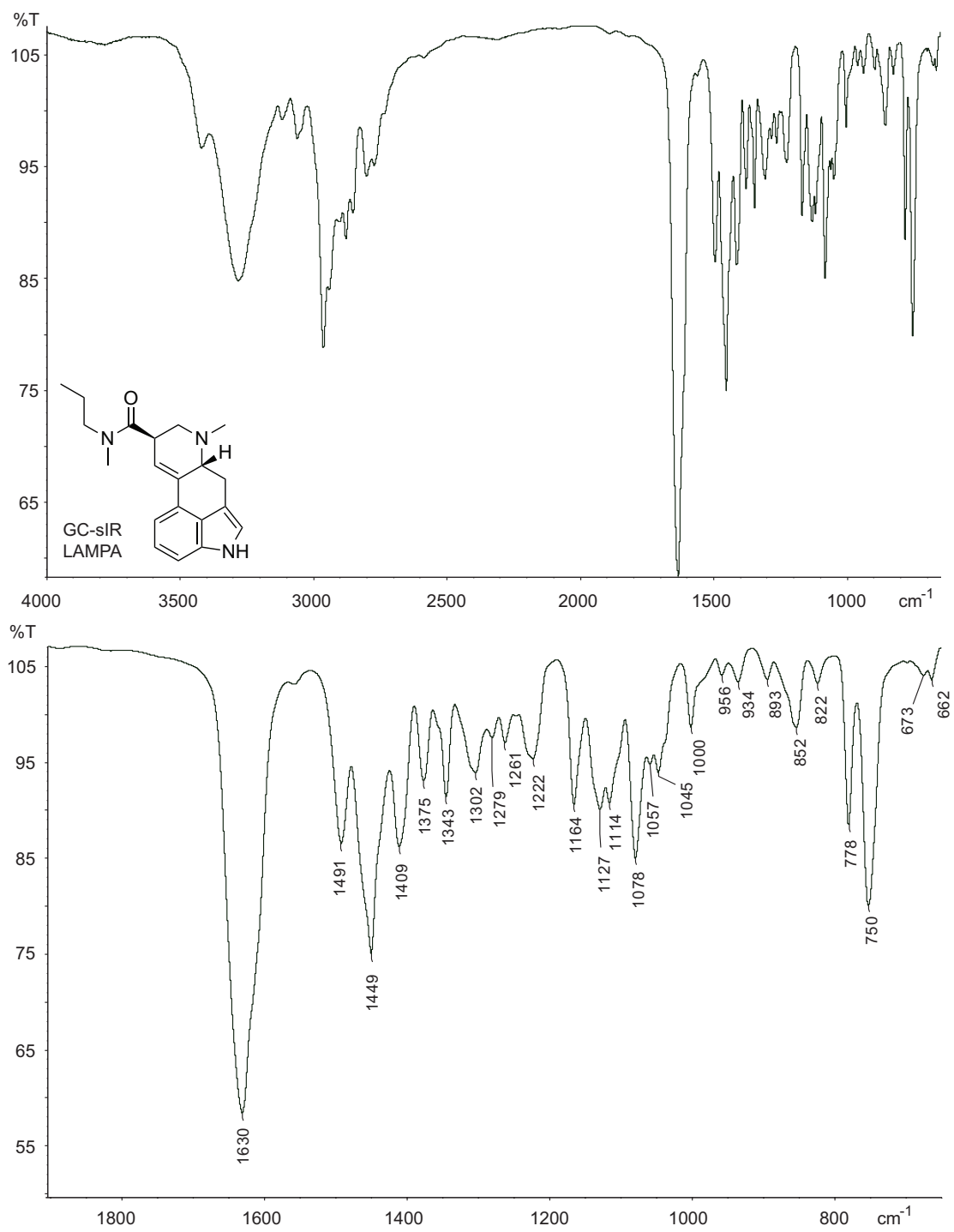
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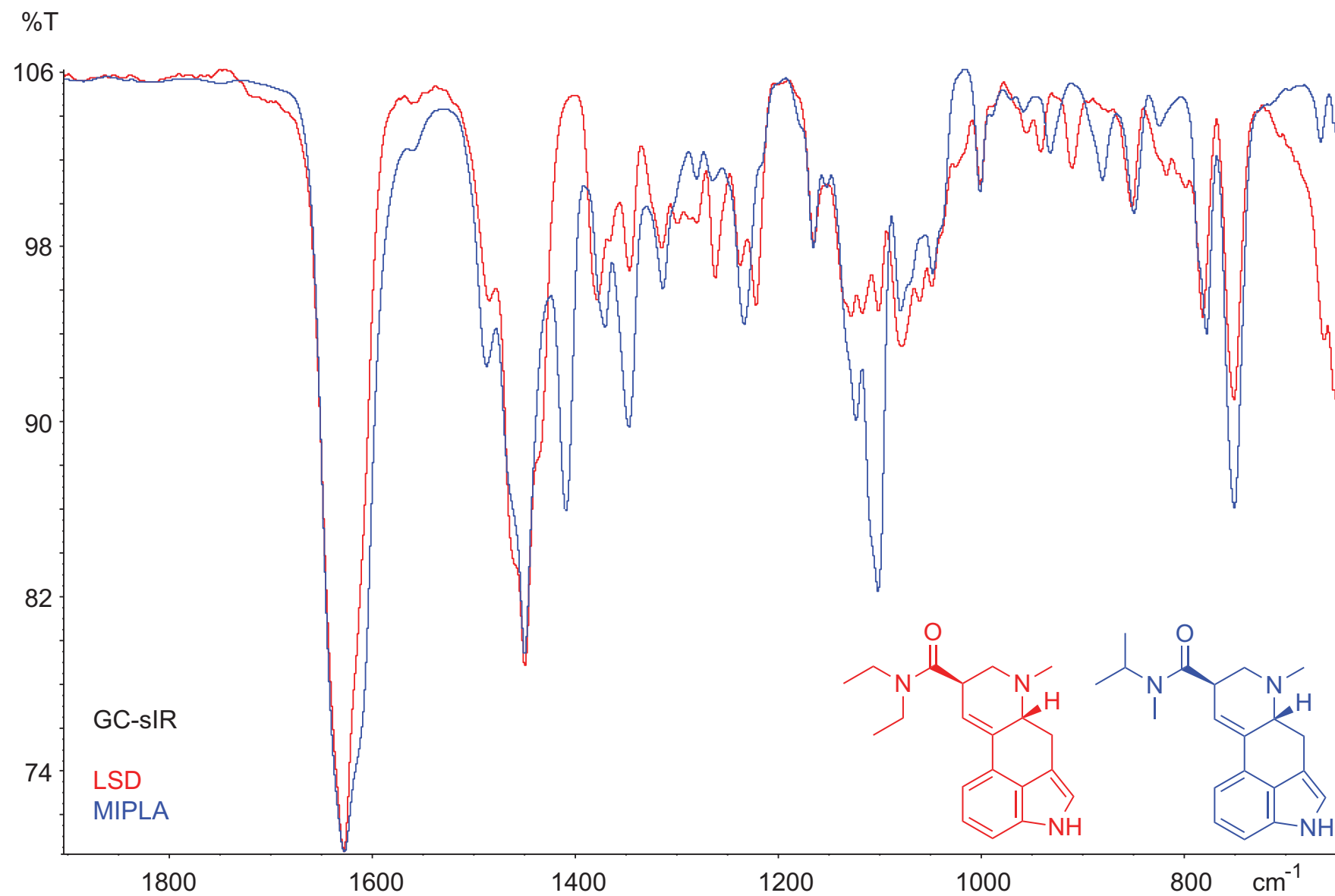


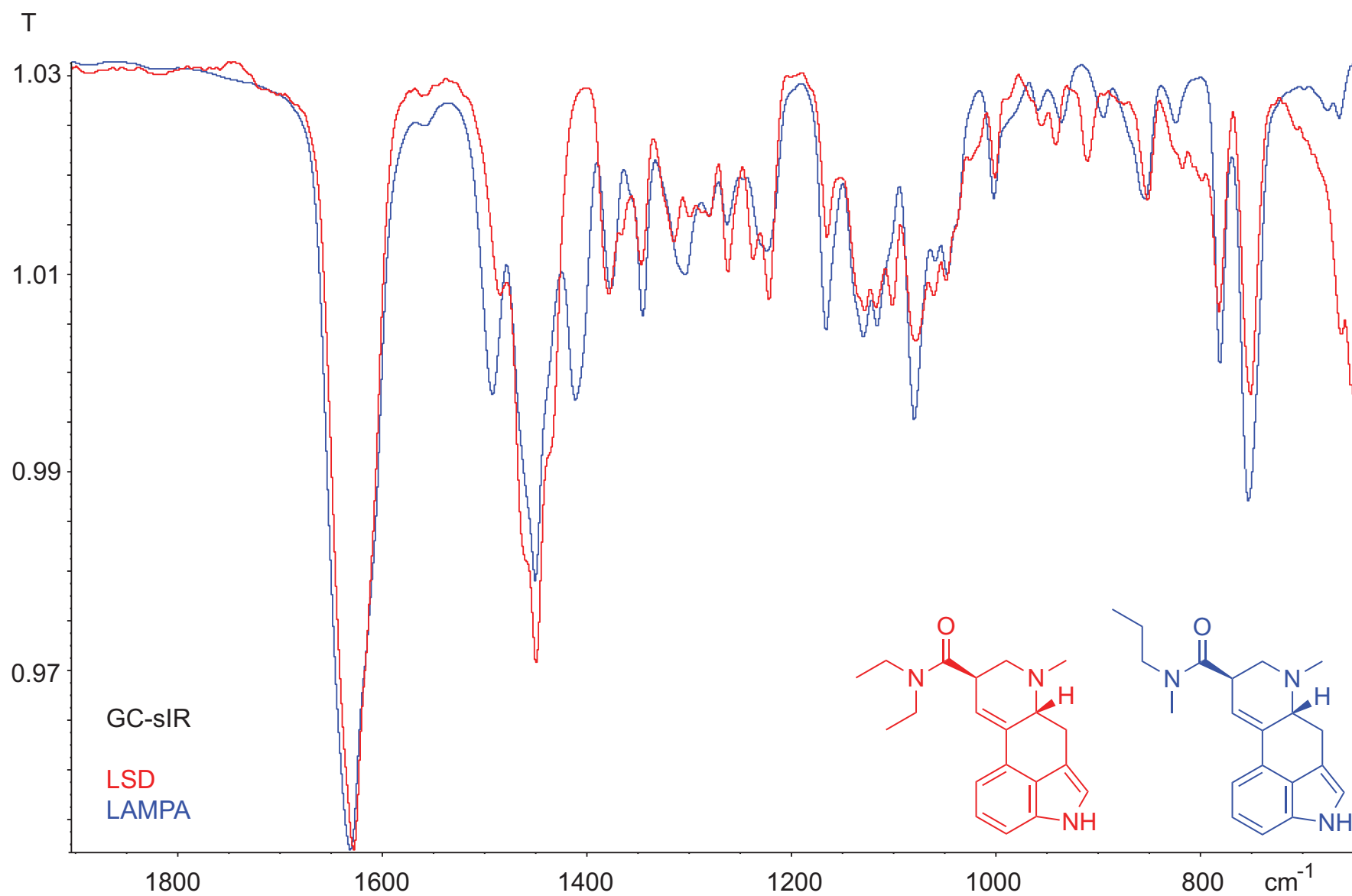
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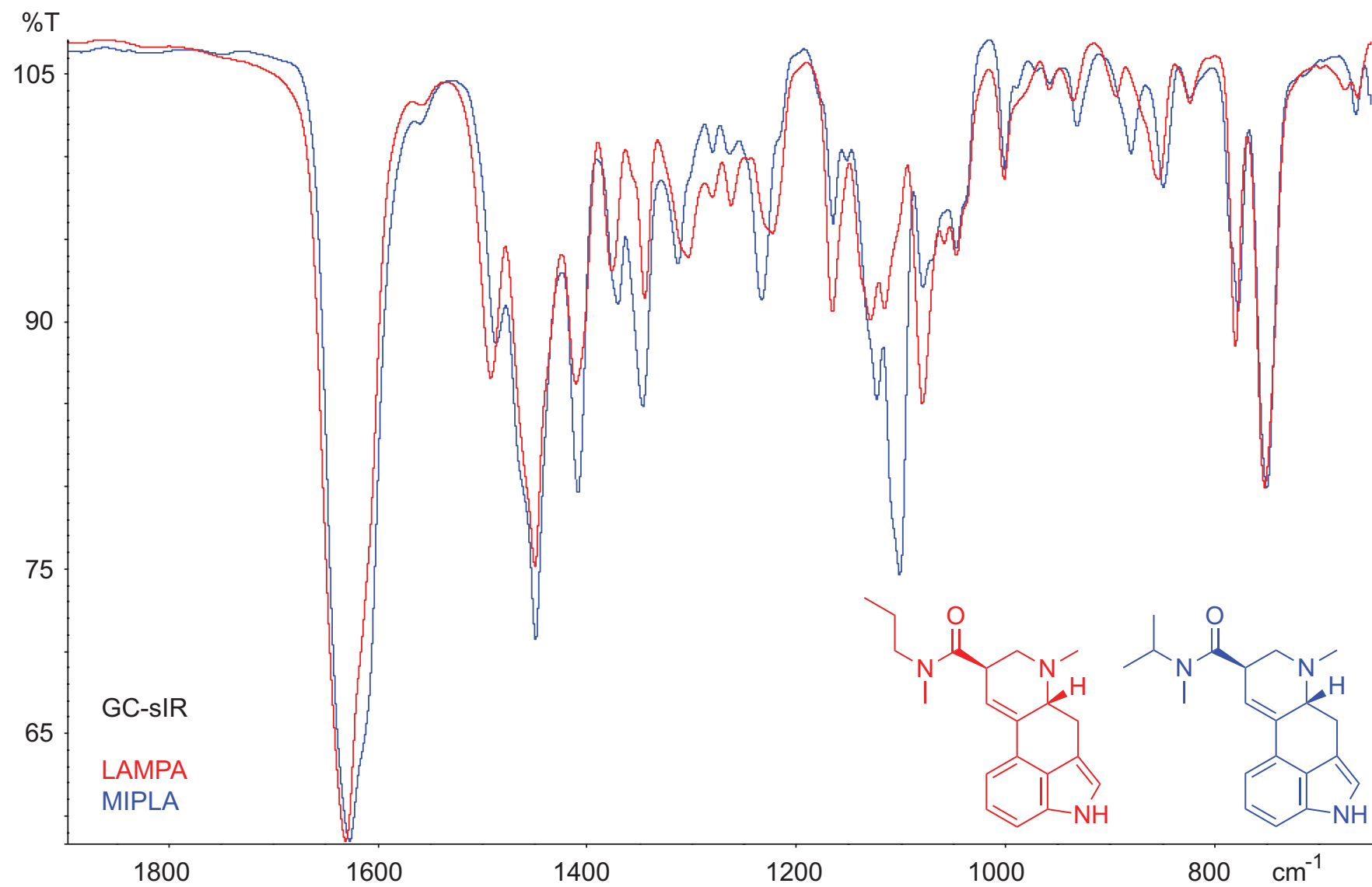


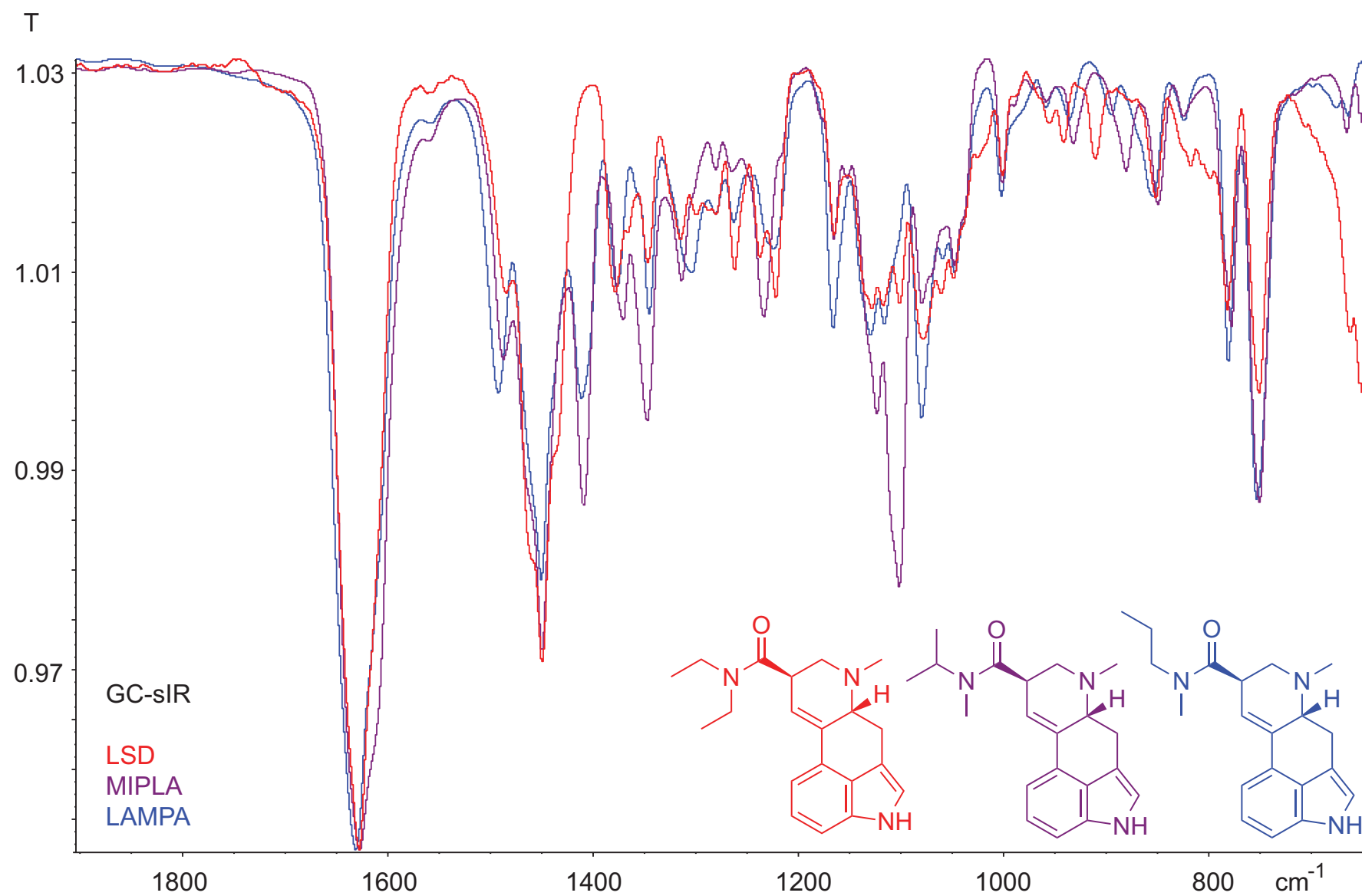
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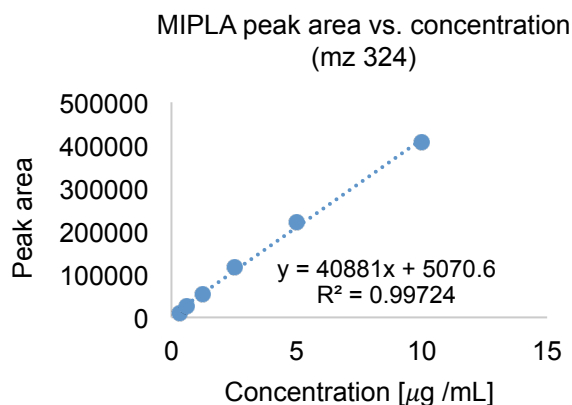
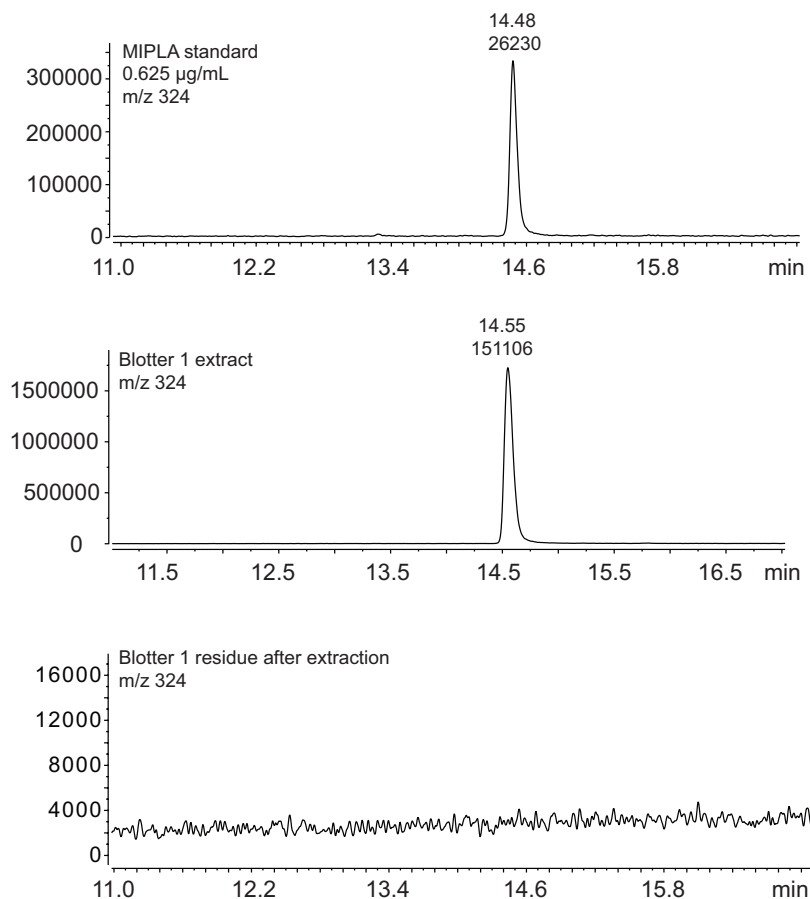




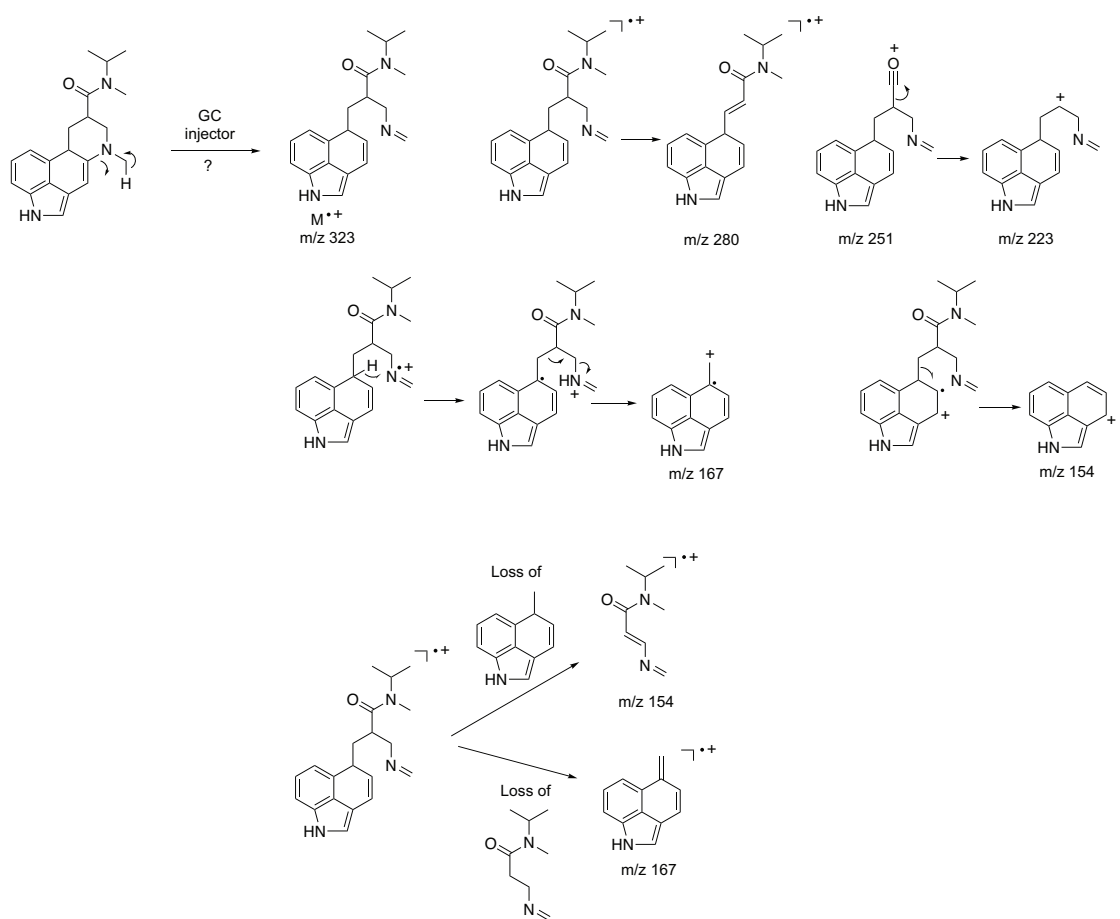
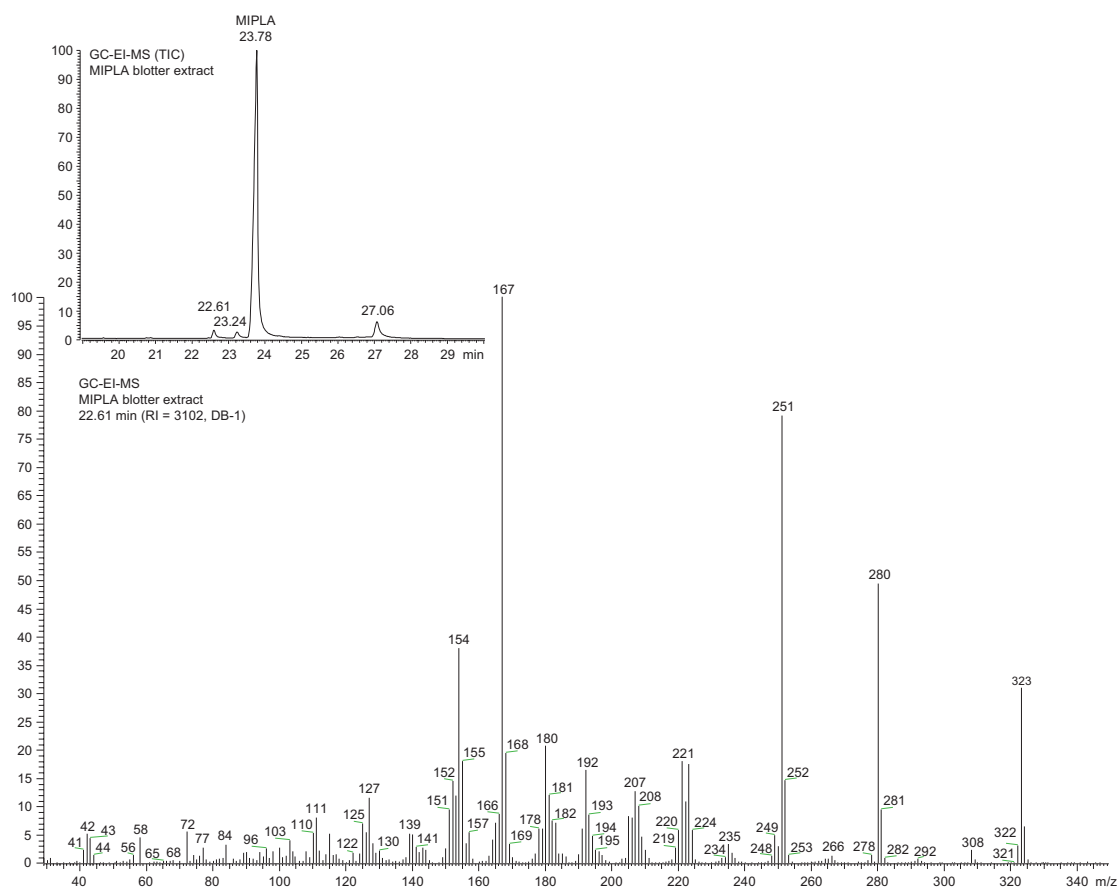


LC-Q-MS elution profile used for quantification of MIPLA blotter extracts

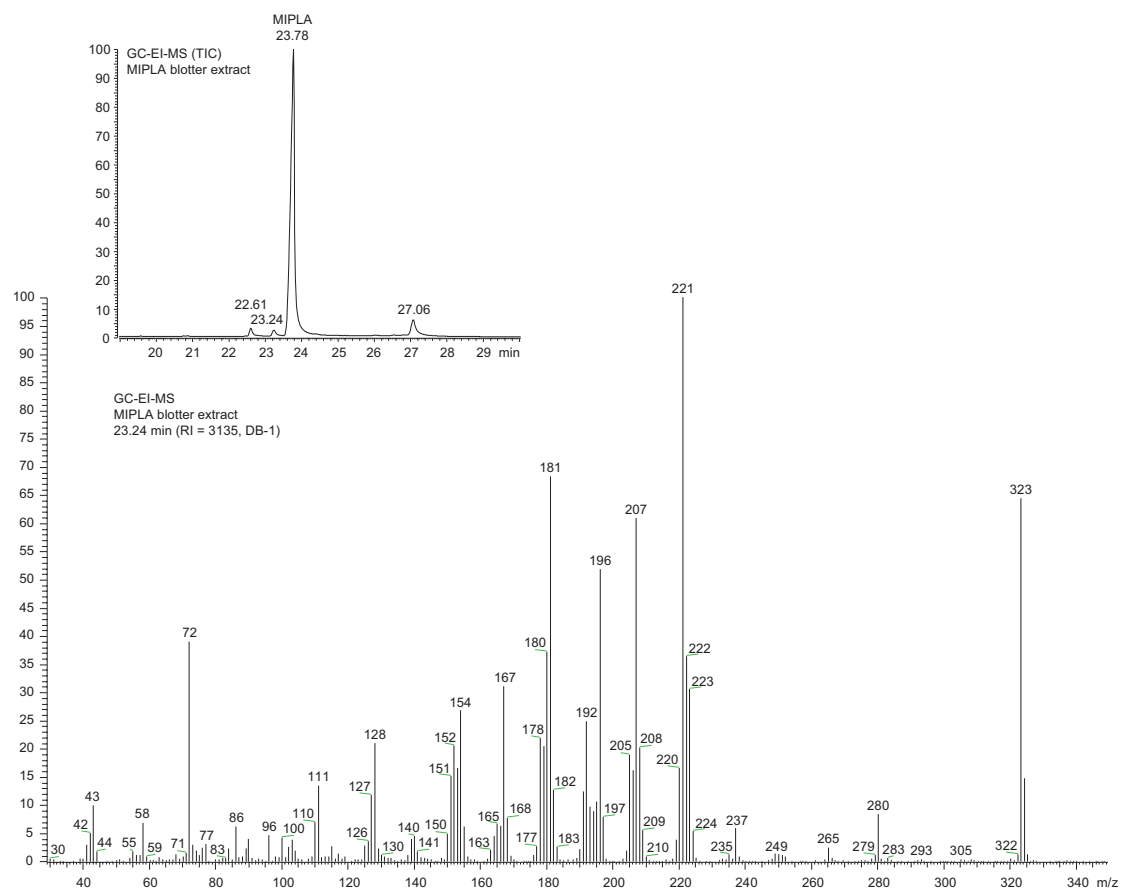
LC-ESI-MS was performed on Agilent 1100 LC system using a Kinetex[®] F5 column (2.6 μm , 100 \AA ; 100 x 2.1 mm) (Phenomenex, Macclesfield, Cheshire, UK): eluent A – acetonitrile containing 0.1% formic acid, eluent B – water containing 0.1% formic acid; 5% A (0–1 min) followed by a linear gradient up to 80% A at 25 min; followed by a linear gradient down to 5% A at 27 min and 5% A for 8 min; flow rate was 200 $\mu\text{L}/\text{min}$, 1 μL injected for SIM (protonated molecule) and 10 μL injected for TIC.



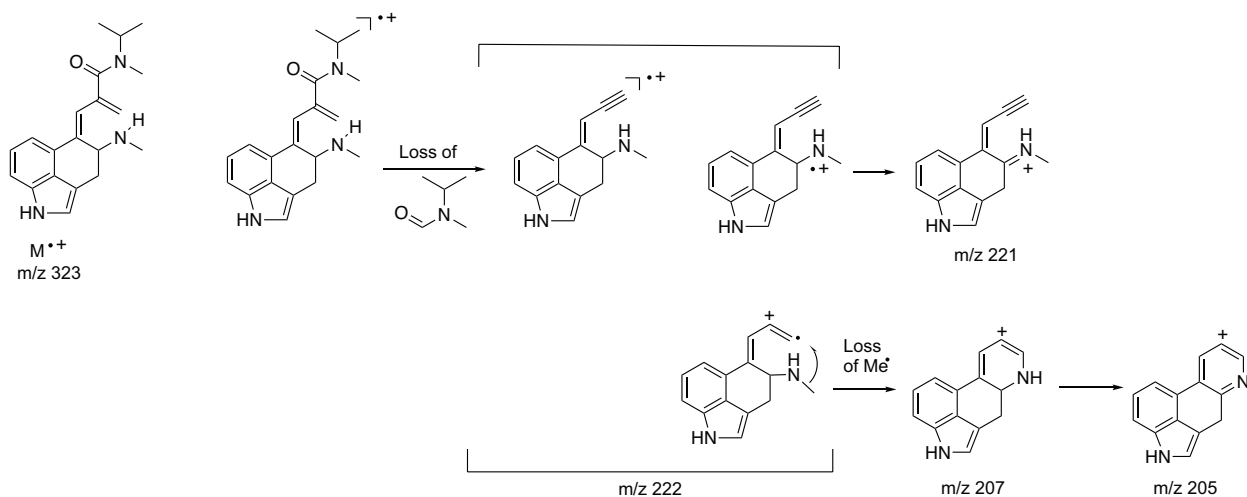
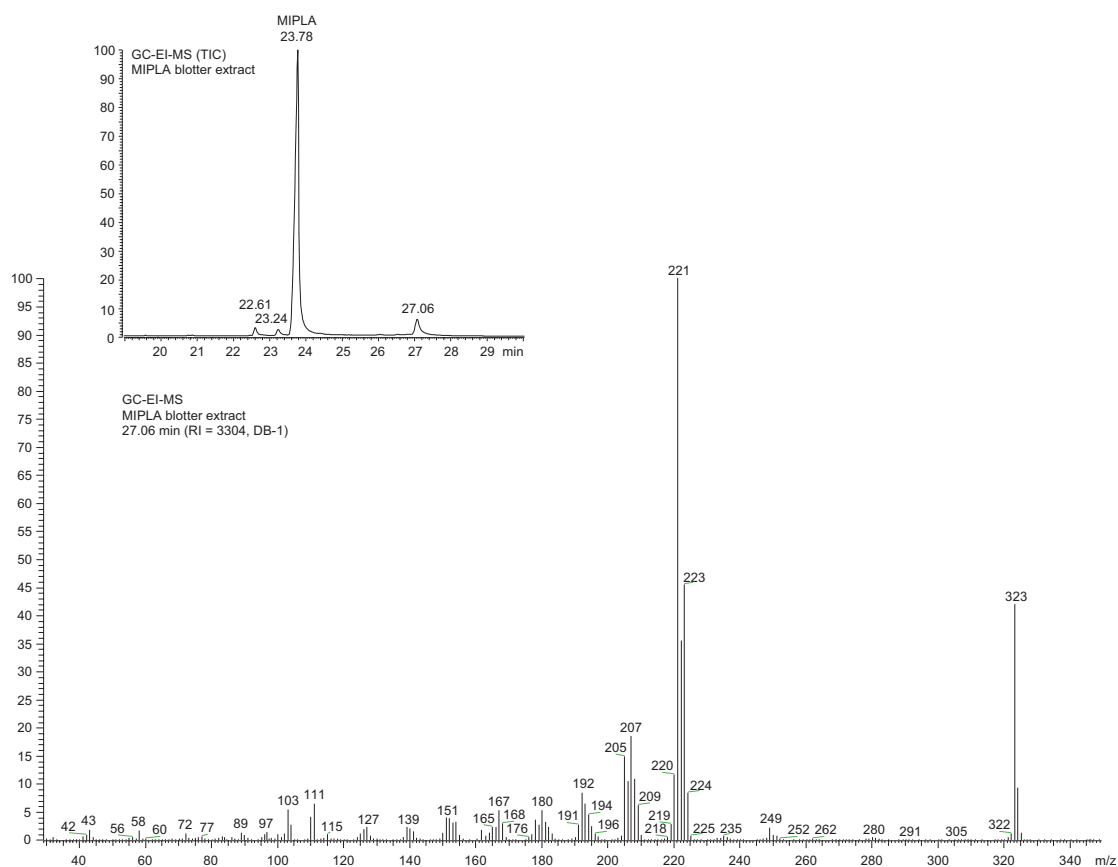
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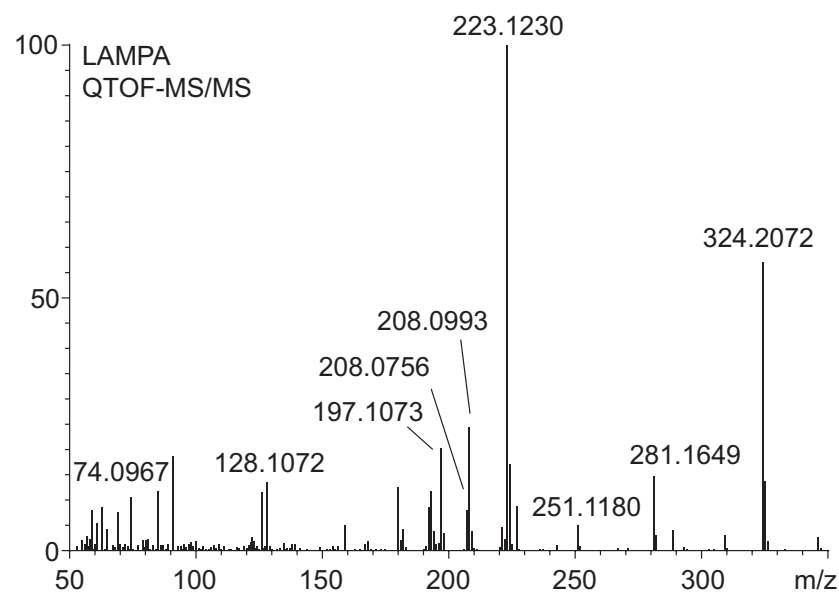
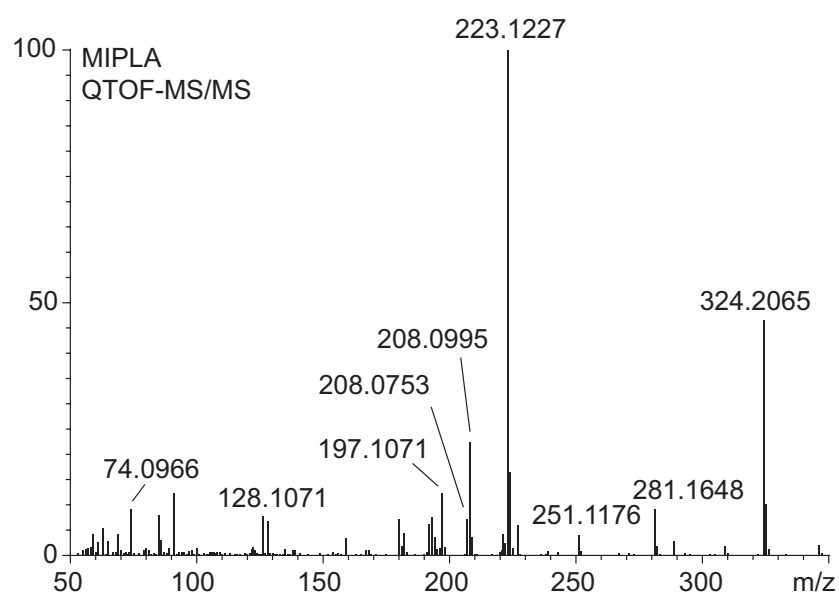
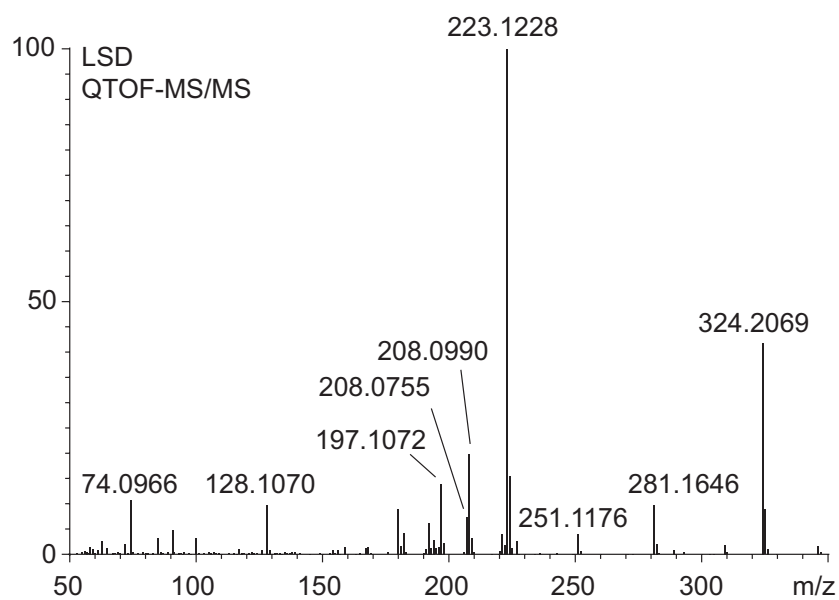
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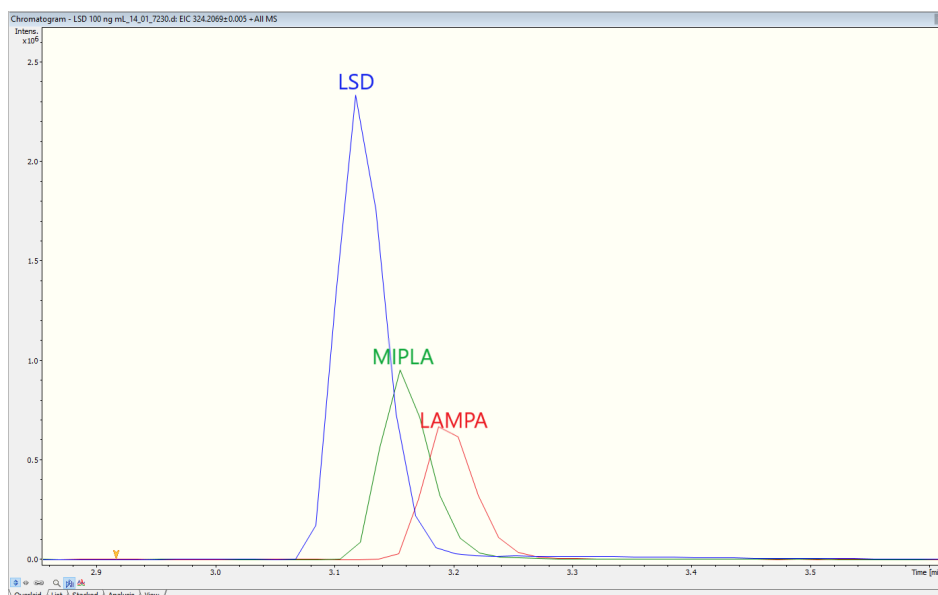


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LC-QTOF-MS conditions

Liquid chromatography-electrospray ionization-quadrupole time of flight-mass spectrometry (LC-QTOF-MS) analysis was performed on an impact IITM qToF instrument coupled with an Elute high-performance liquid chromatography (HPLC) system (both from Bruker Daltonik, Bremen, Germany). Chromatographic separation was performed on a Kinetex[®] Biphenyl column (100 × 2.1 mm, 2.6 µm particle size, Phenomenex, Aschaffenburg, Germany) equipped with a corresponding guard column (SecurityGuardTM ULTRA Cartridges UHPLC Biphenyl for 2.1 mm ID columns, Phenomenex). Mobile phase A consisted of 1% acetonitrile, 0.1% formic acid and 2 mM ammonium formate in deionized water. Mobile phase B consisted of 0.1% formic acid and 2 mM ammonium formate in acetonitrile. The applied gradient with a flow of 0.3 mL/min started at 5% mobile phase B, increased to 30% within 1.0 min, and to 35% within another 9.0 min. Mobile phase B was increased to 95% within 0.5 min and was held for 2 min before the initial conditions were restored within 0.2 min and held for 1.3 min. The total runtime was 14 min. The autosampler temperature was 10°C and the column oven temperature was 40°C. The injection volume was 10 µL. The MS was operated in positive ionization mode acquiring spectra in the range of m/z 50–500 at an acquisition rate of 4.0 Hz. The dry gas temperature was set to 200°C with a dry gas flow of 8.0 L/min. The nebulizer gas pressure was 200 kPa. Full scan (+MS) and broadband collision-induced dissociation (bbCID) data were acquired in one run. The collision energy applied for bbCID was 30 ± 6 eV. Nitrogen was used as collision gas. The voltages for the capillary and end plate offset were 2500 V and 500 V, respectively. External and internal mass calibration was performed using sodium formate/acetate clusters and high-precision calibration (HPC) mode. HyStarTM version 3.2 and DataAnalysis version 4.2 (both from Bruker Daltonik) were used for data acquisition and processing, respectively. The bbCID fragmentation experiments for MIPLA, LAMPA, and LSD were performed with solutions at 100 ng/mL, applying the LC-QTOF-MS parameters stated above.



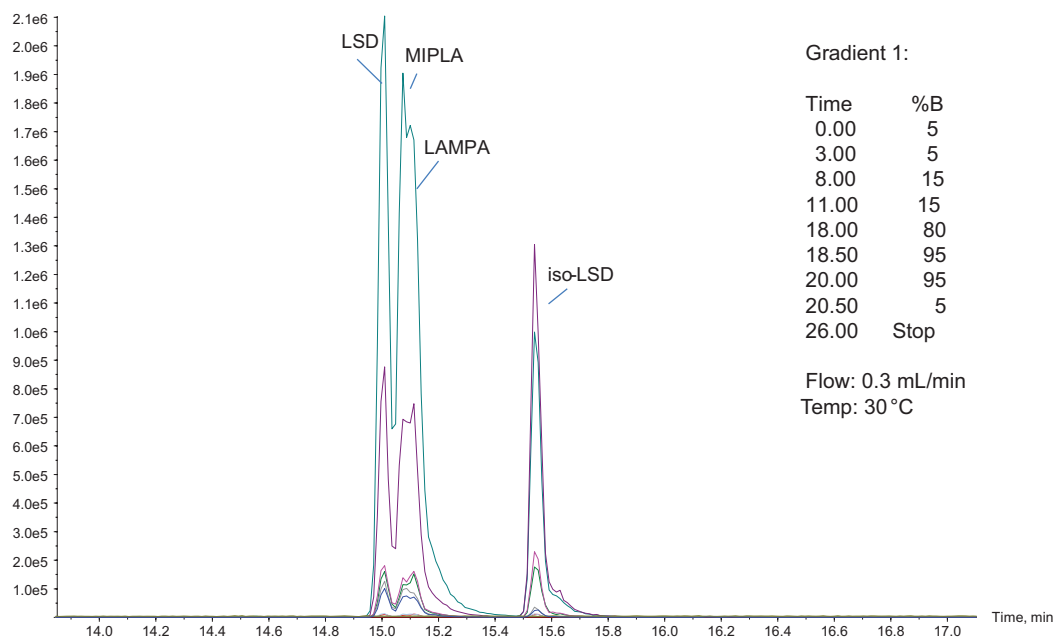
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Kinetex Biphenyl (100x 2.1 mm, 2.6 μ m particle size, Phenomenex, Aschaffenburg, Germany)

Mobile phase A: deionized water with 1% ACN, 0.1% formic acid, and 2 mM ammonium formate

Mobile phase B: 0.1% formic acid and 2 mM ammonium formate in ACN

Intensity, cps MIPLA, LAMPA, LSD, iso-LSD 50 ng/mL

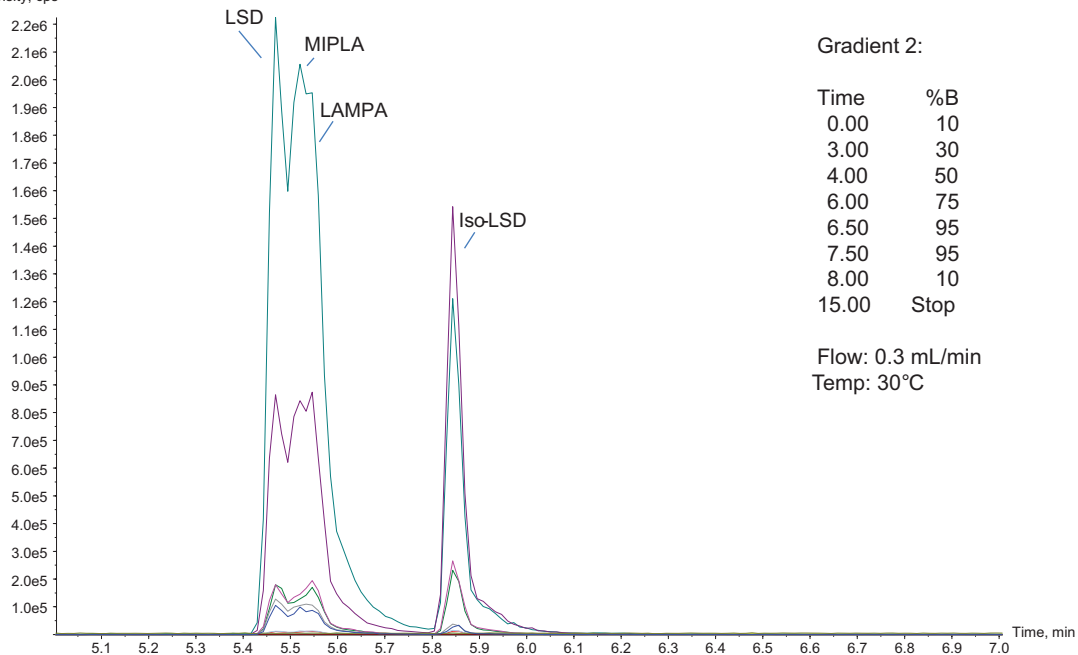


Kinetex Biphenyl (100x 2.1 mm, 2.6 μ m particle size, Phenomenex, Aschaffenburg, Germany)

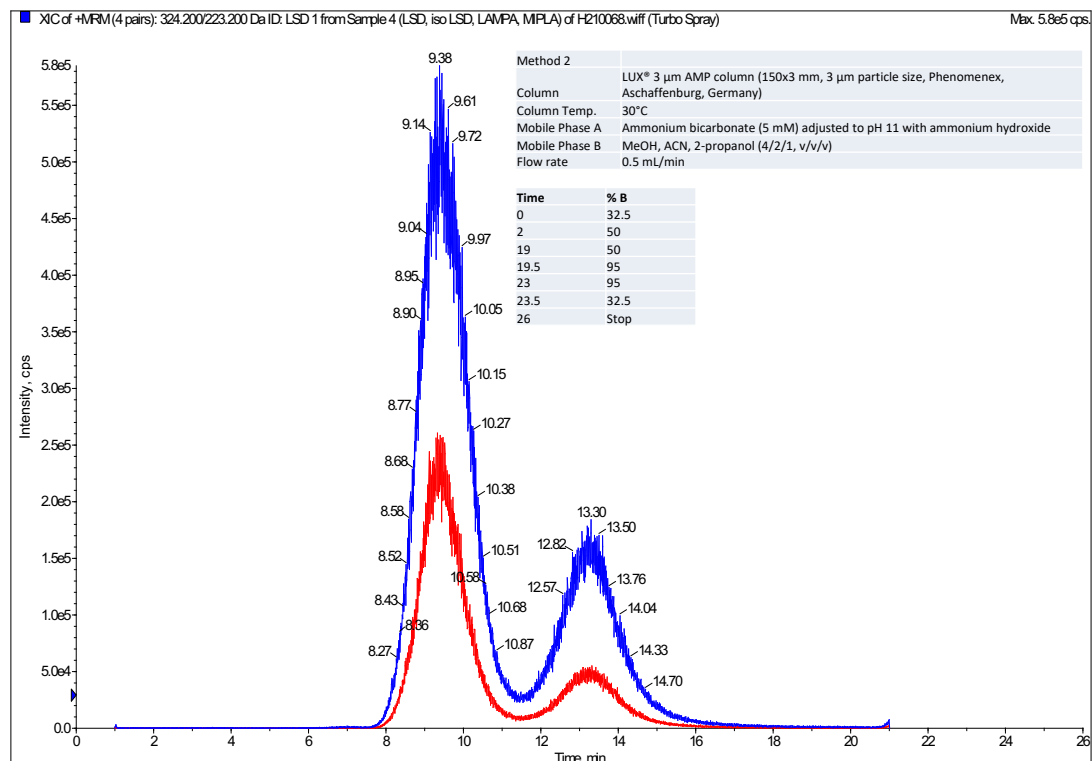
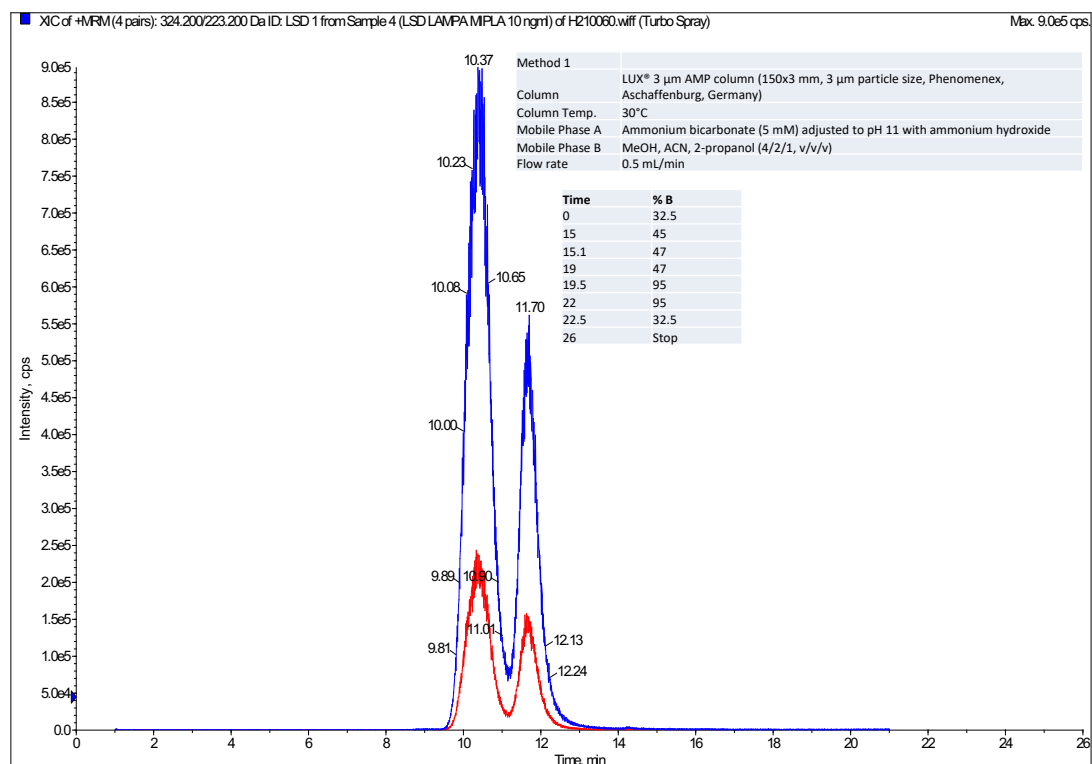
Mobile phase A: deionized water with 1% ACN, 0.1% formic acid, and 2 mM ammonium formate

Mobile phase B: 0.1% formic acid and 2 mM ammonium formate in ACN

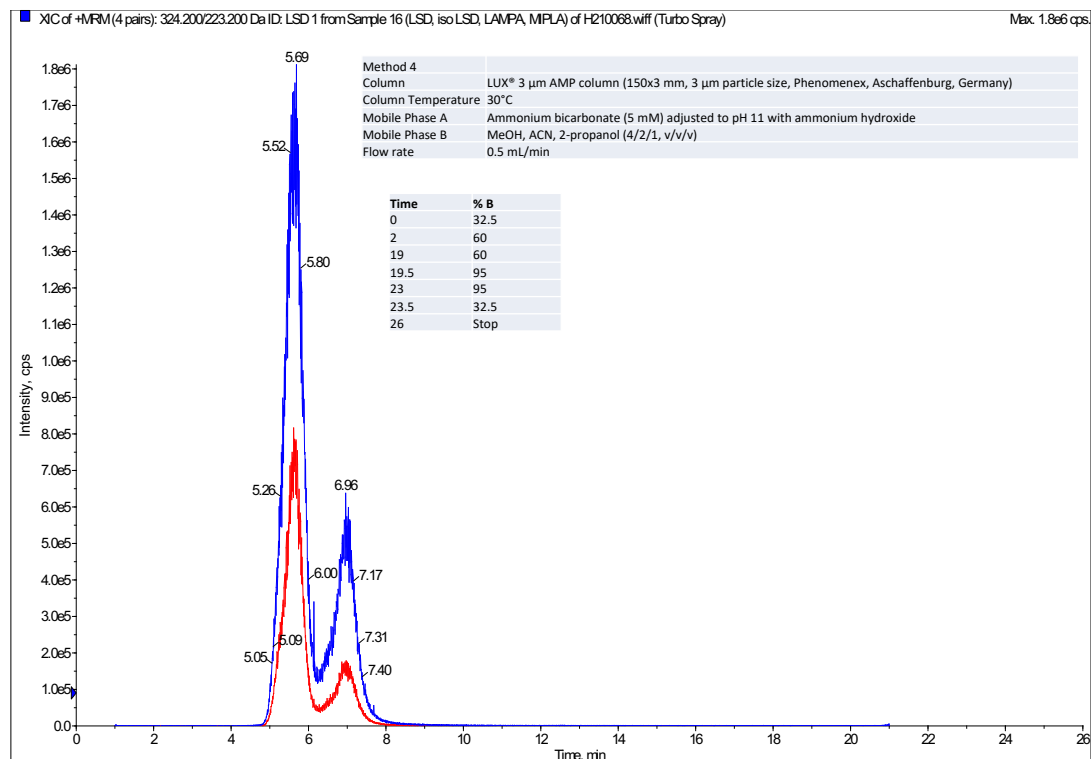
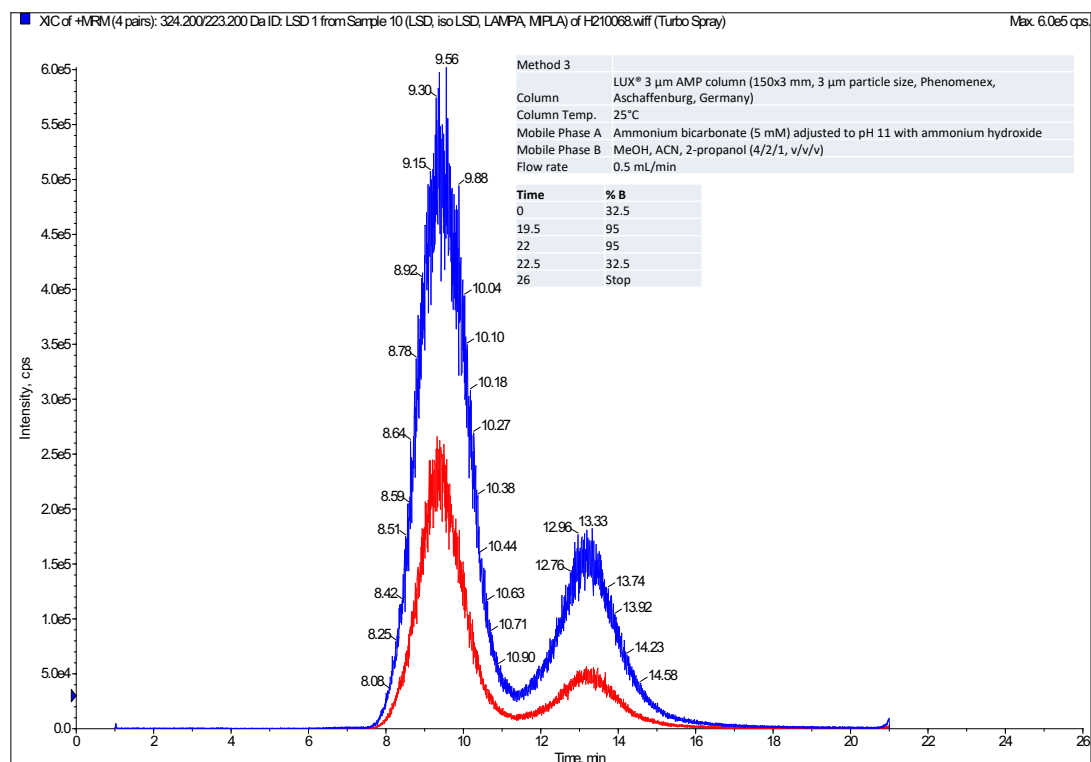
Intensity, cps



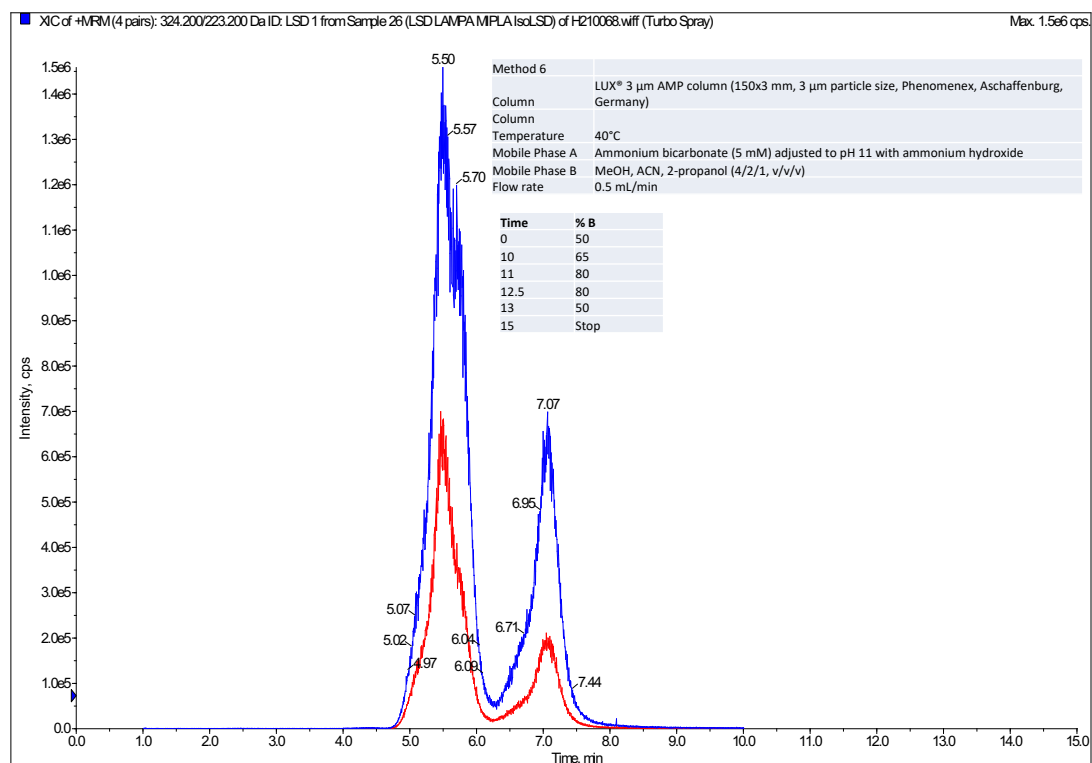
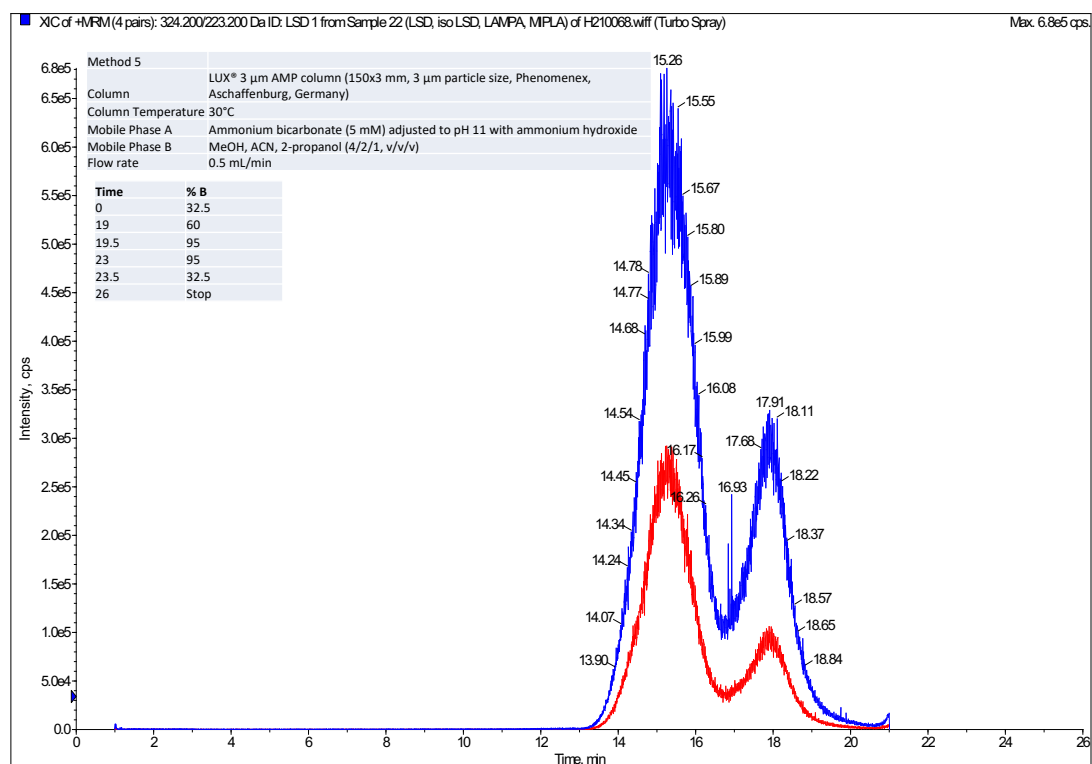
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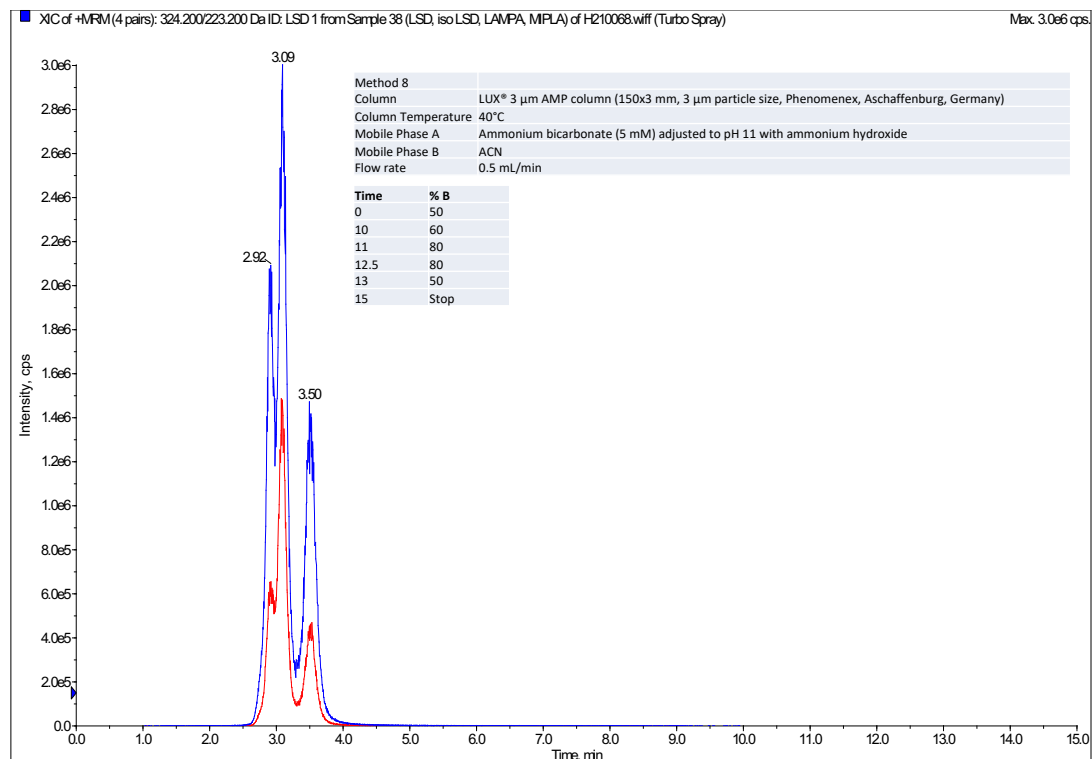
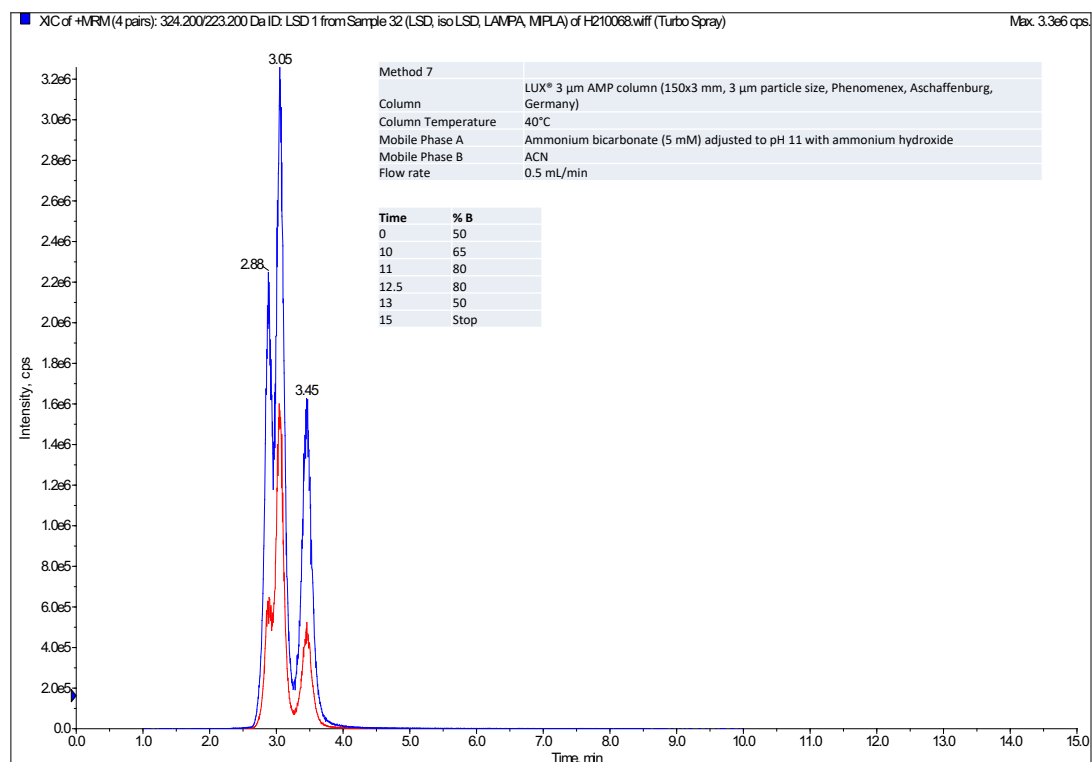
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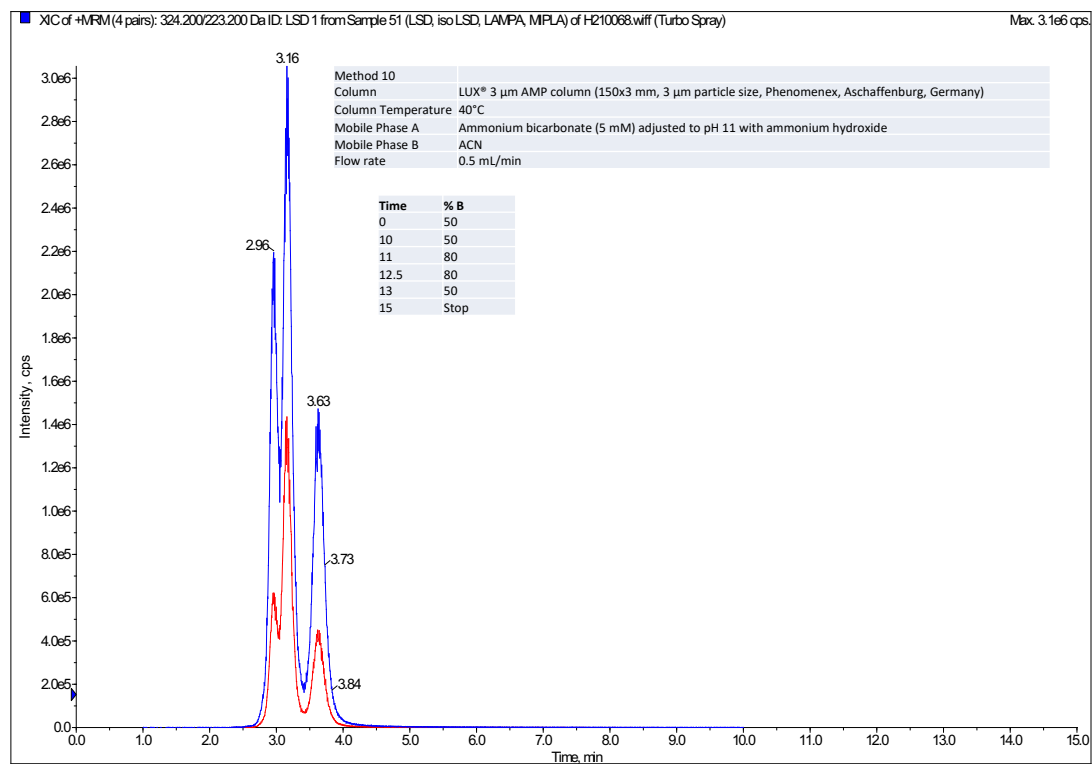
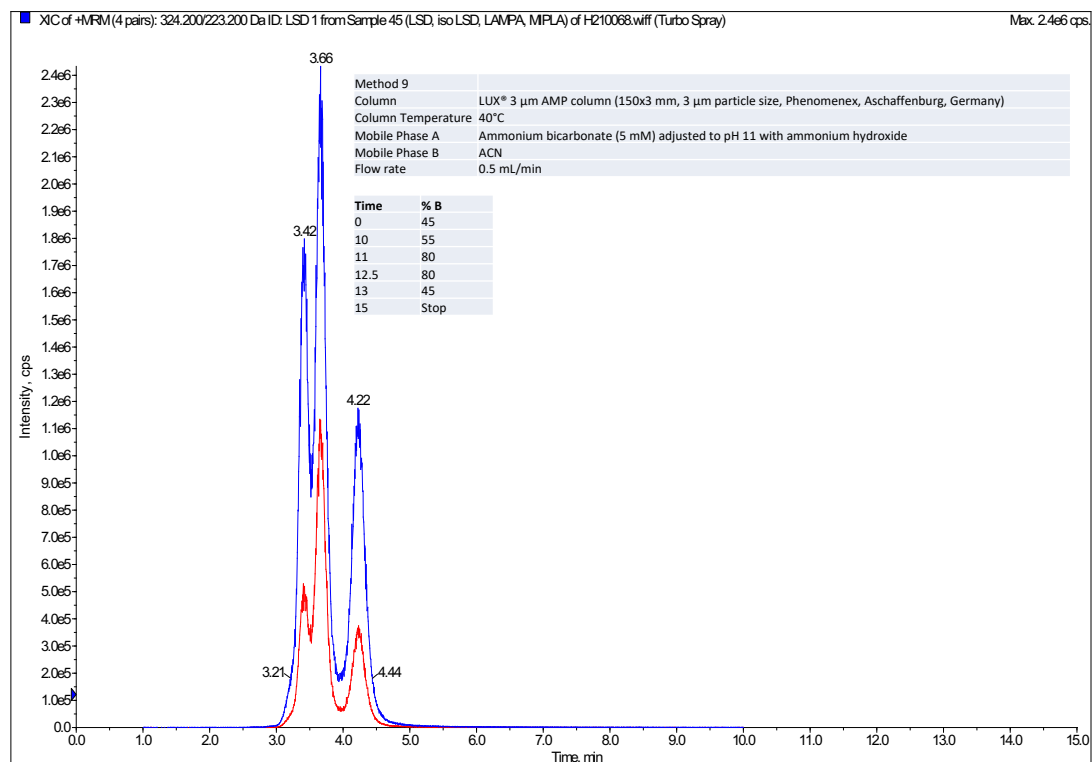
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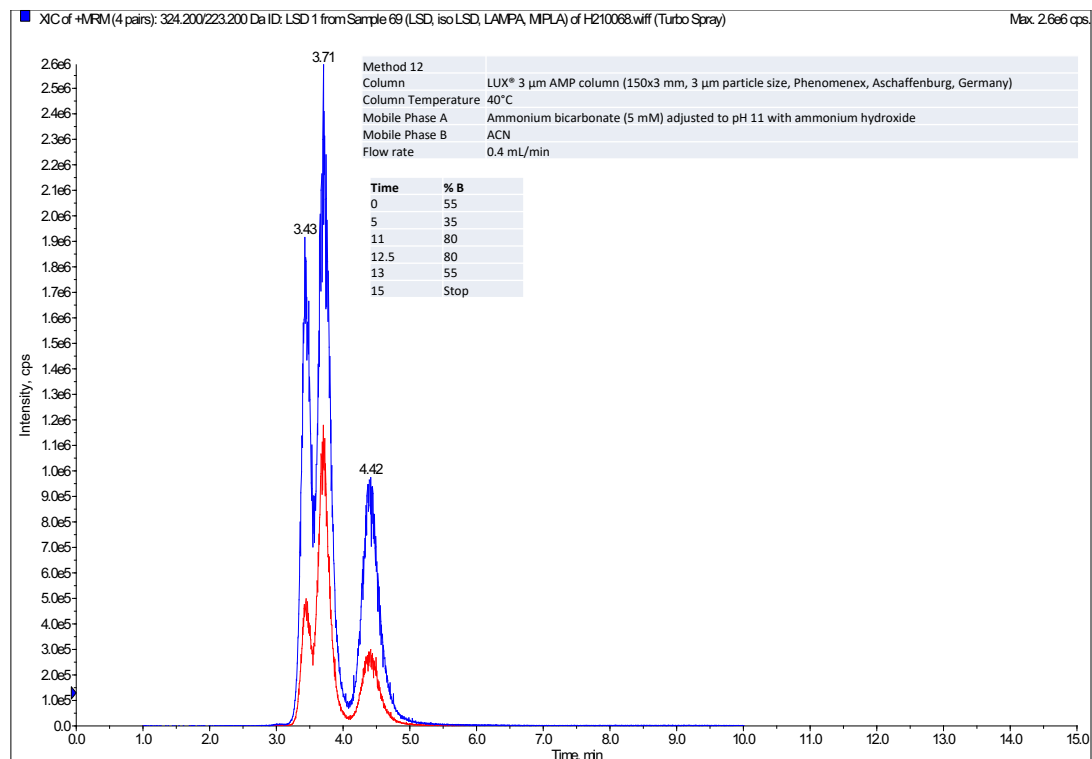
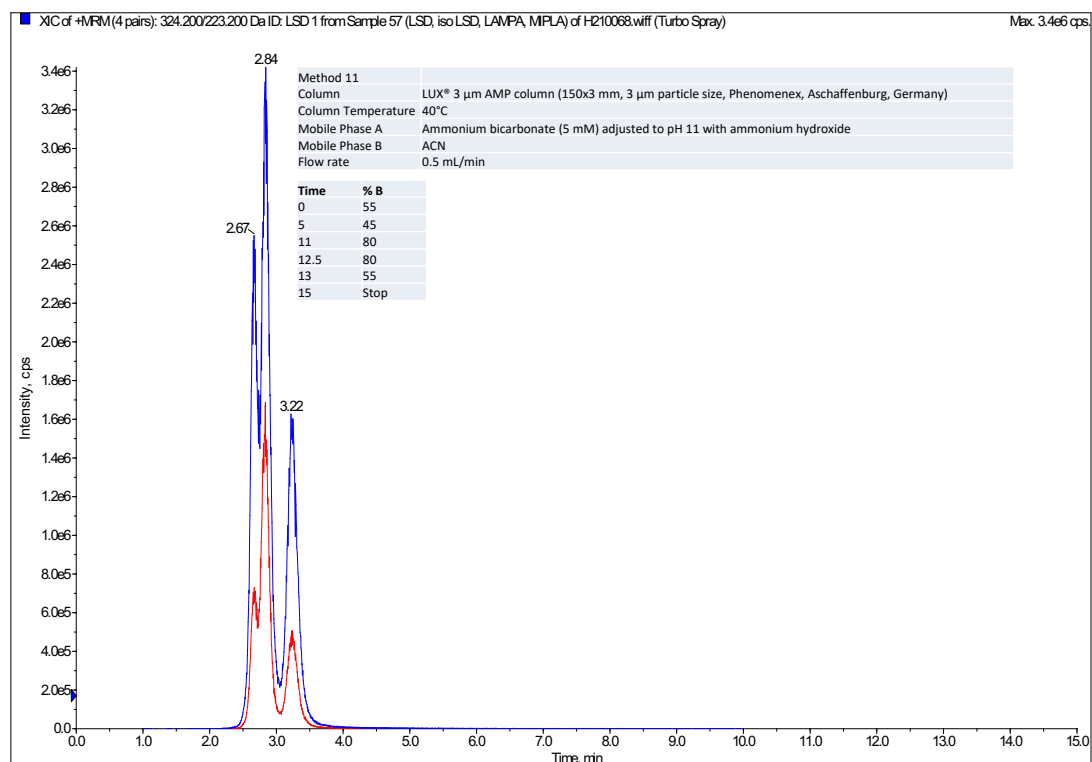
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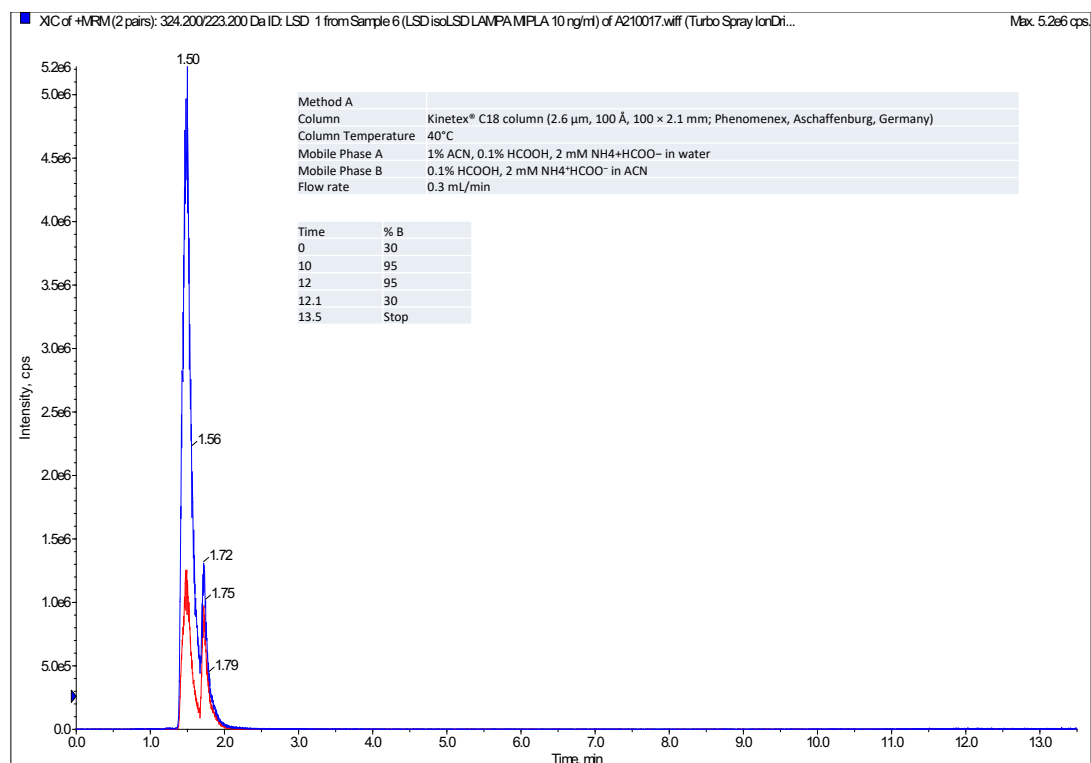
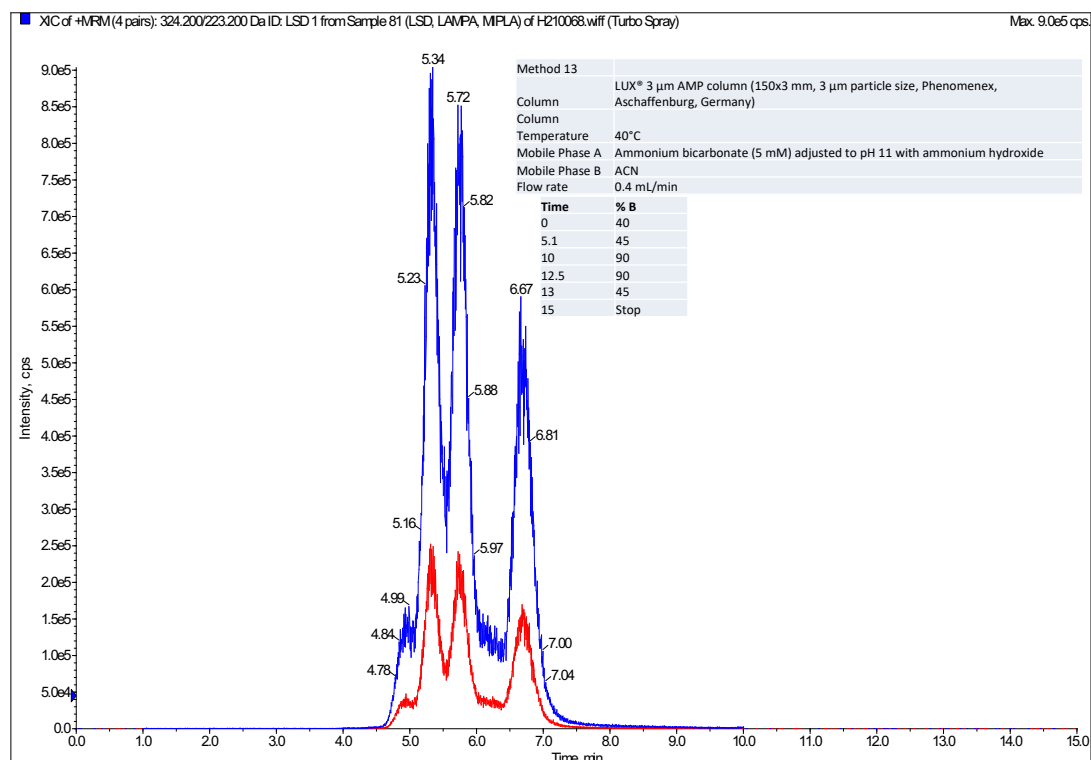
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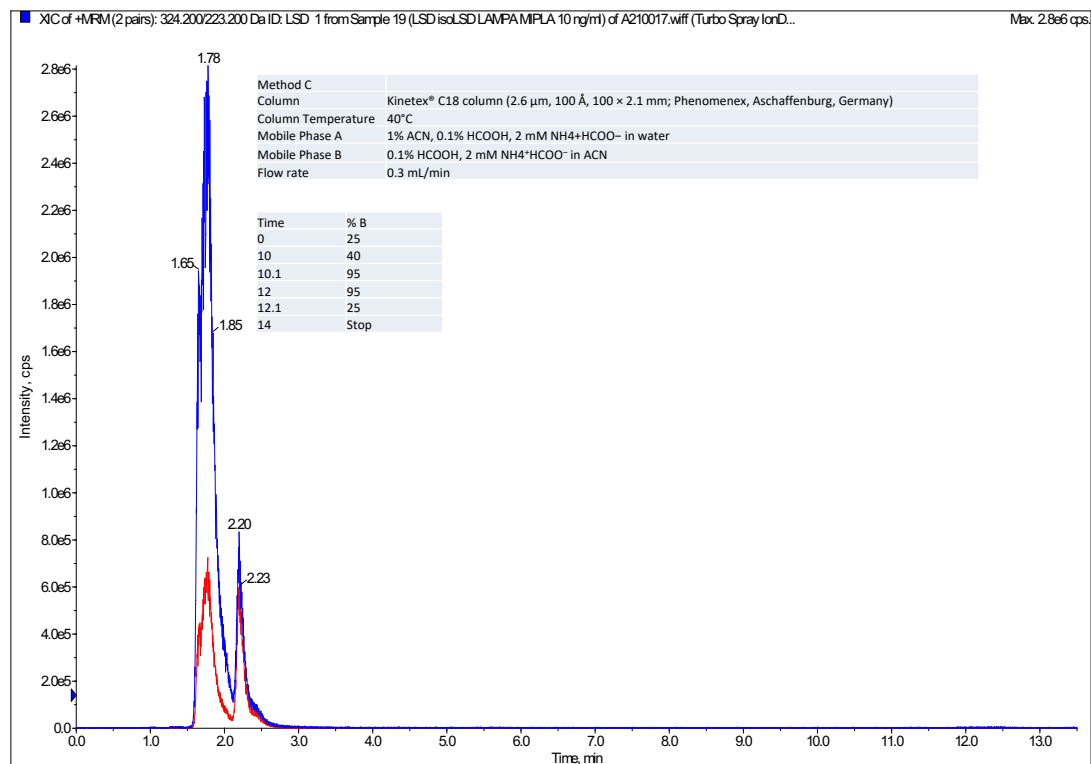
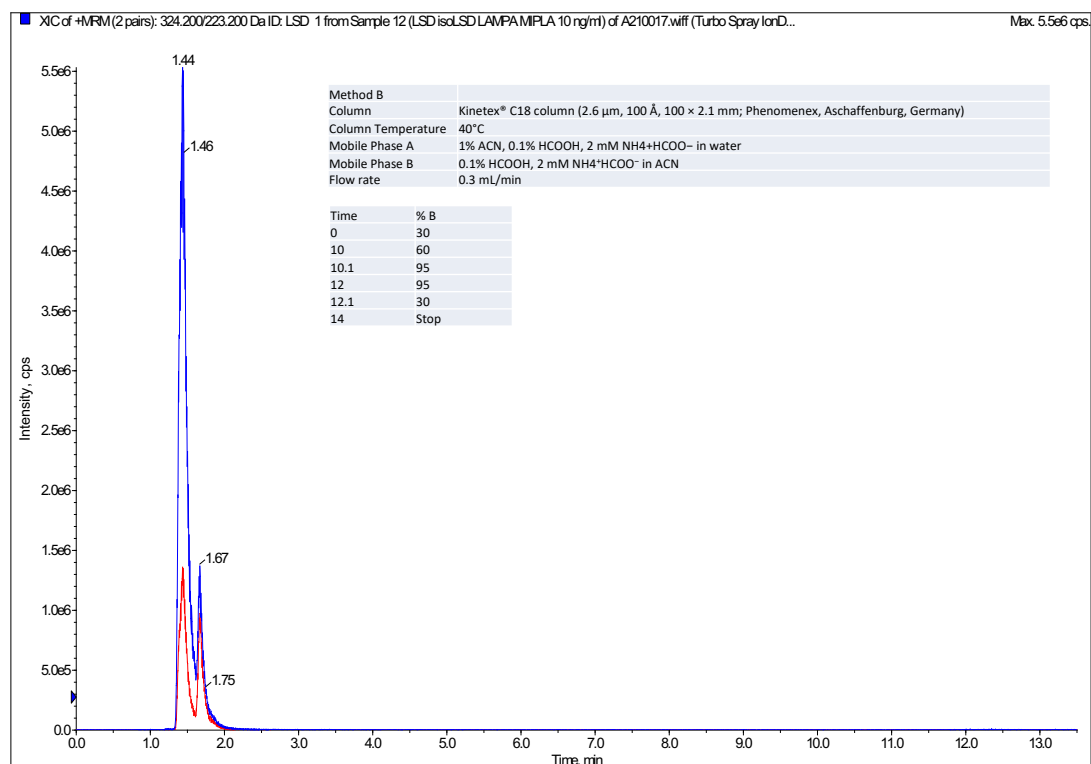
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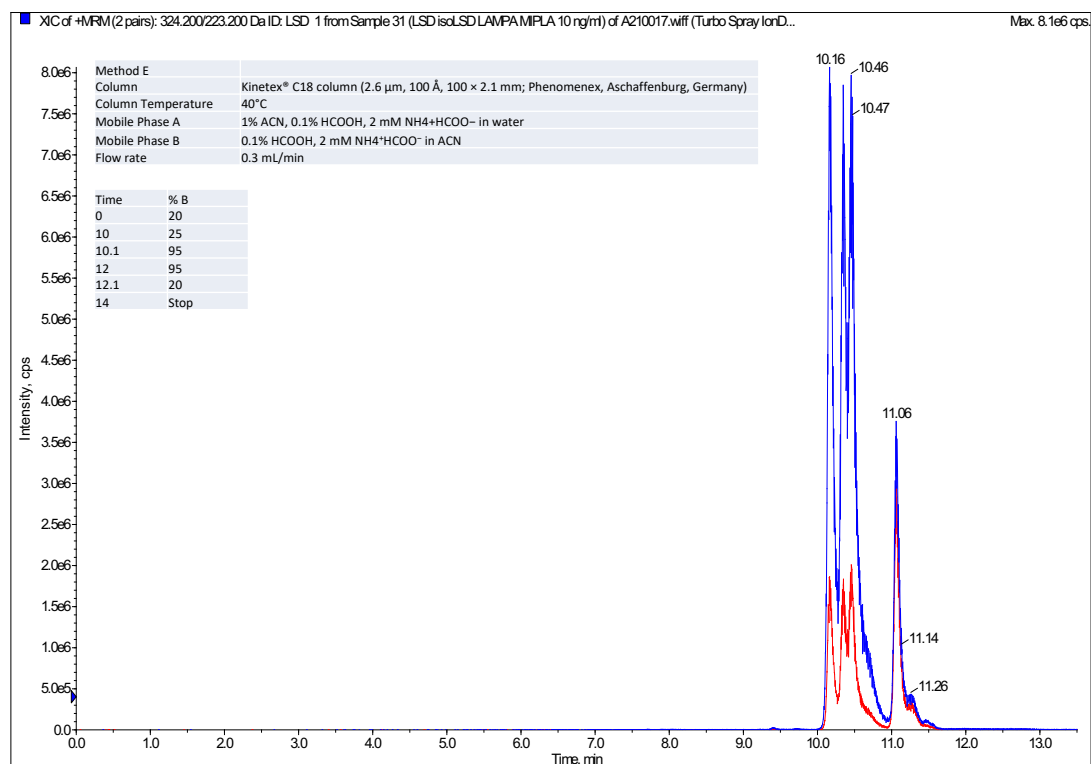
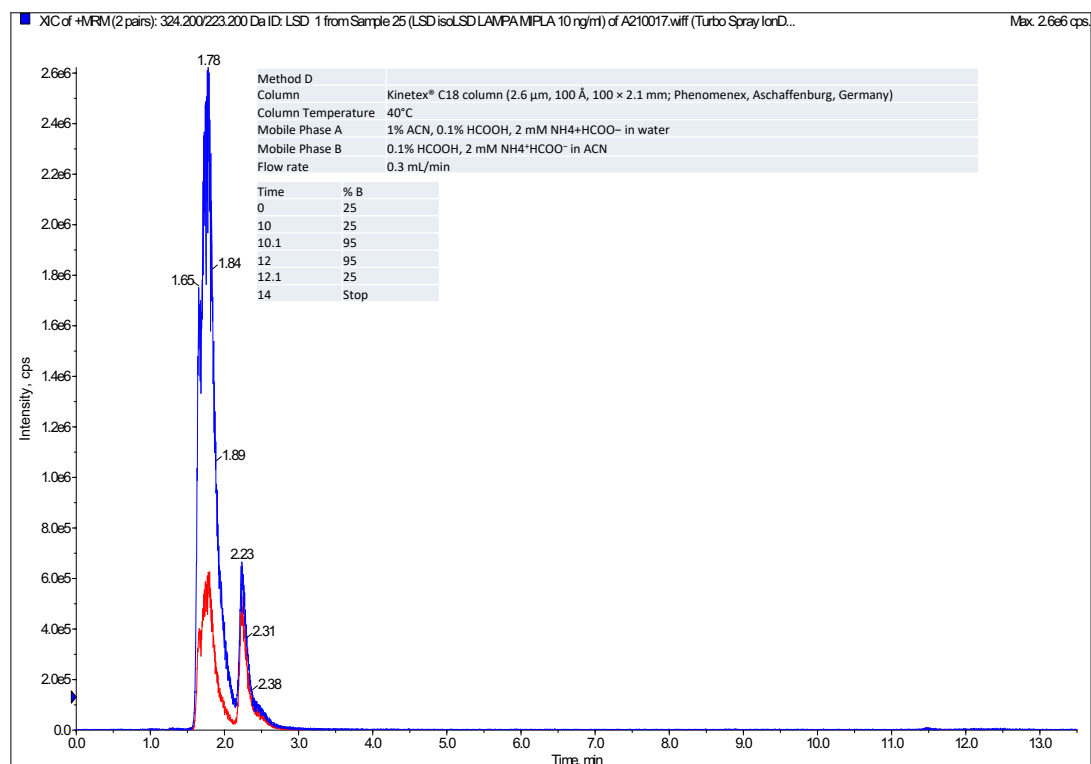
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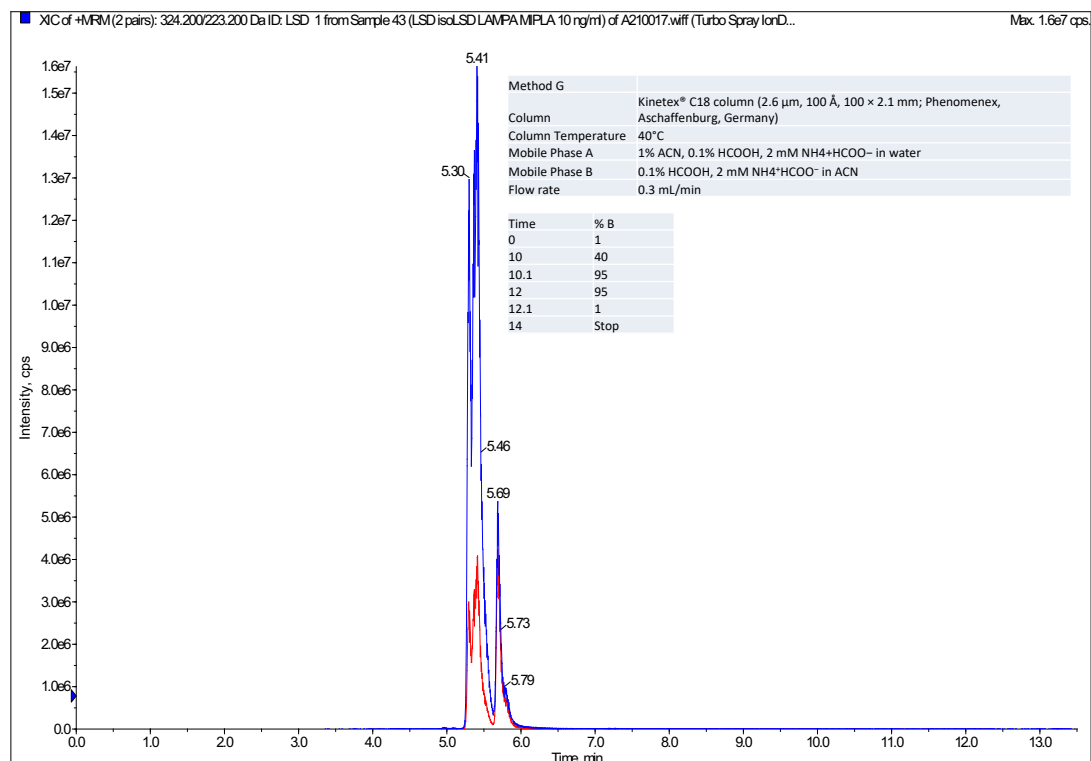
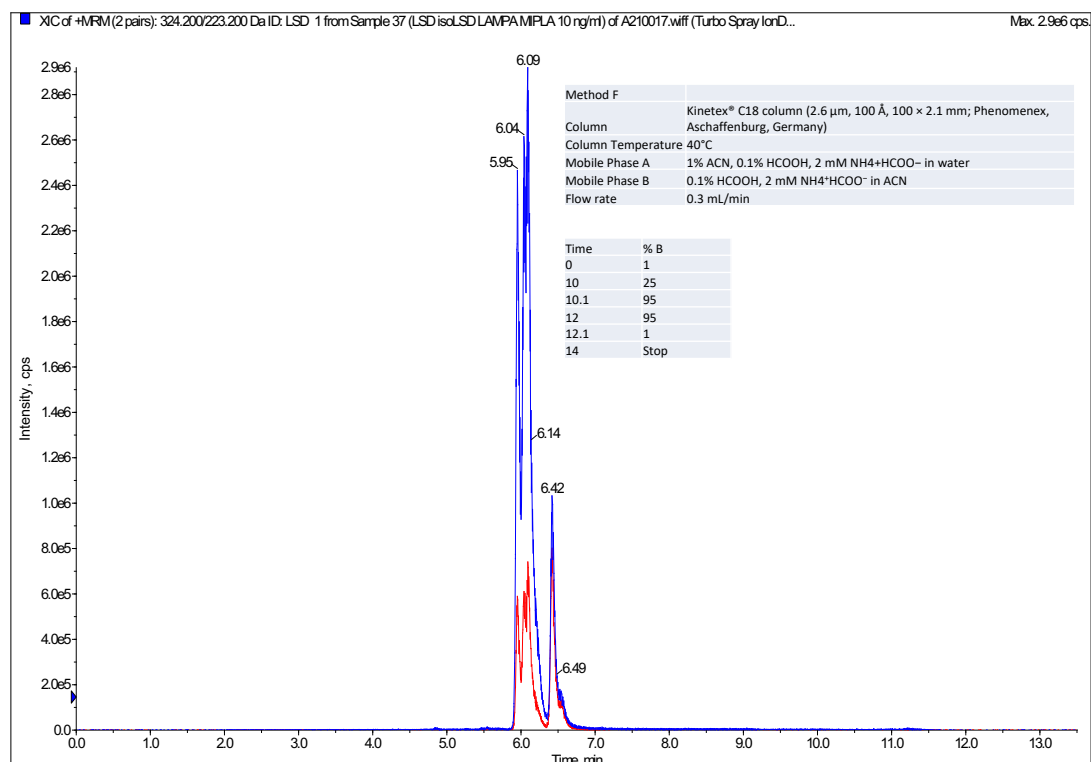
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