



No.46

LC-MS Liquid Chromatograph Mass Spectrometer

## Analysis of Drugs in Putrefied Human Pleural Fluid using Triple Quadrupole LC/MS/MS

This application illustrates a drug screening method in putrefied human pleural fluid using the Shimadzu UFMS triple quadrupole mass spectrometer, LCMS-8050.

In forensic and toxicology fields, it is important to develop a highly sensitive and exhaustive methodology for screening and identifying drug substances. A wide range of these compounds in various biological matrices, such as whole blood, urine and tissue, need to undergo a simple and uniform sample pretreatment protocol prior to Liquid Chromatography Triple Quadrupole Mass Spectrometry (LC-MS/MS) analysis. This data sheet presents a drug screening method using the LCMS-8050 with newly developed sample preparation protocol in putrefied human pleural fluid.

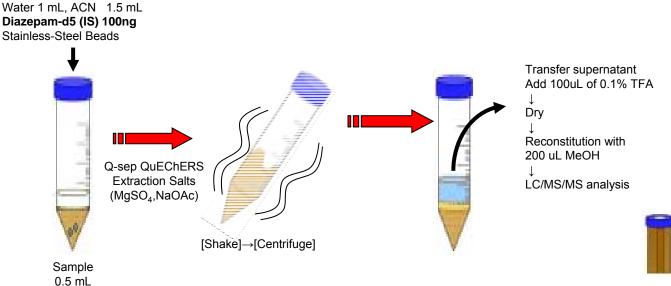
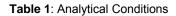


Figure 1: Scheme of the modified QuEChERS procedure



## Liquid Chromatography

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Column	:Shim-Pack FC ODS (150x2mm, 3µm)
<ul> <li>Temperature</li> </ul>	:40 °C
<ul> <li>Injected volume</li> </ul>	:5 μL
<ul> <li>Mobile phases</li> </ul>	:A: Water + 10 mM Ammonium Formate
	B: Methanol
<ul> <li>Flow rate</li> </ul>	:0.3 mL/min
<ul> <li>Gradient</li> </ul>	:5%B (0 min) – 95%B (15 min – 20min) – 5%B (20.1 min – 30 min)

## Mass Spectrometry

<ul> <li>Configuration</li> </ul>	:LCMS-8050
<ul> <li>Ionization mode</li> </ul>	:Heated ESI positive and negative
<ul> <li>Nebulizing gas flow</li> </ul>	:2 L/min
<ul> <li>Drying gas flow</li> </ul>	:10 L/min
<ul> <li>Heating gas flow</li> </ul>	:10 L/min
<ul> <li>DL temperature</li> </ul>	:250 °C
HB temperature	:400 °C
Analysis mode	:MRM



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Traditional sample preparation strategies for biological fluids, such as protein precipitation and solid phase extraction, require multiple time-consuming steps. In addition, commercially available sample preparation techniques lack the ability to extract all compounds of interest. A modified QuEChERS protocol for drug screening in biological fluids described here illustrates stable recoveries for drug substances regardless of sample or chemical properties.

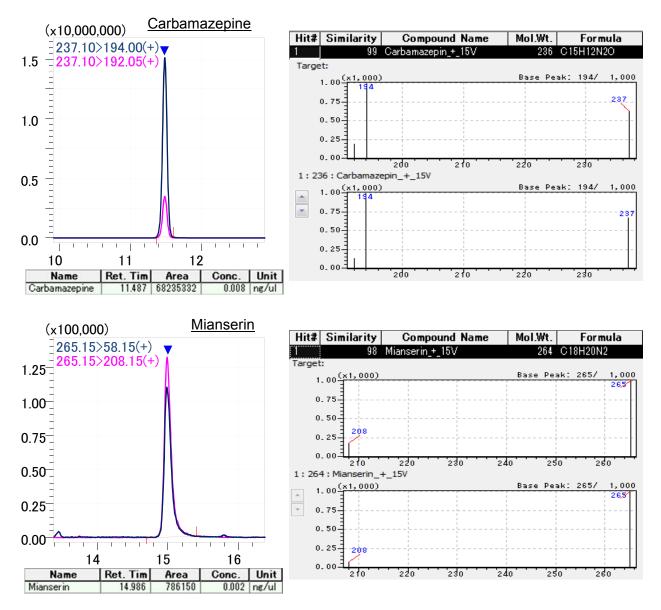


Figure 3: MRM Chromatograms, semi-quantitative value and Library Search results of Carbamazepine and Mianserin in putrefied human pleural fluid

Shimadzu's LCMS-8050 UFMS can perform Synchronized Survey Scan<sup>®</sup> (SSS), which automatically conducts a product ion scan triggered by preset MRM intensity thresholds. SSS provides both quantitative (MRM chromatograms) and qualitative data (Product ion spectrum) in a single run.

Furthermore, utilizing diazepam-d5 as an internal standard, semi-quantitative results can be determined using the method's built-in calibration curves (slope and intersection).

This software functionality is a very effective way for one to understand the quantitative values obtained during a simultaneous analysis.



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