## SOP-072 LC-MS Test Mix Evaluation and Troubleshooting

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# SOP-072 LC-MS Test Mix Evaluation and Troubleshooting

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Toricology Eutoratory				
Approving Authority Name	Approving Authority Signature	Approval Date		
Ruth E. Winecker, Ph.D.	Hut Ellinden	04/15/2015		
Ruth E. Winecker, Ph.D.	Stuttellinden	06/10/2016		
Ruth E. Winecker, Ph.D.	Puttellinden	09/12/2017		
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#### 1. Principle

- 1.1. Prior to beginning an analytical run on an LC-MSLC-MS or LC-MS-ion trap instrument a test-mix should be run prior to analysis, when able, to ensure acceptable performance.
  - 1.1.1. The test mix could be either a neat containing the analytes to be analyzed or an injection of an extracted standard or QC.

#### 2. Evaluation and Troubleshooting Guide

- 2.1. Chromatography
  - 2.1.1. Make sure peaks are present, if not:
    - 2.1.1.1. Check that the right vial was injected
    - 2.1.1.2. Make sure the correct instrument and processing method is chosen, in accordance with the assay SOP.
    - 2.1.1.3. Check the volume of the LC solvents.
    - 2.1.1.4. Check for leaks or clogs in the LC-MS system.
    - 2.1.1.5. Check that the mass spectrometer is working properly by observing signal/scans in TSQ-Tune program
    - 2.1.1.6. Consult with the LC-MS chemist to troubleshoot the problem
  - 2.1.2. Unless specific to the analyte, make sure that the peaks do not front, tail or have shoulders. If chromatography is poor:
    - 2.1.2.1. Inject another neat to make sure it is not extraction related.
    - 2.1.2.2. Make sure the correct instrument method is chosen, in accordance with the assay SOP.
    - 2.1.2.3. Check to see when the column was last replaced, and replace if necessary.
    - 2.1.2.4. Consult with the LC-MS chemist
- 2.2. Mass spectrometry
  - 2.2.1. For triple quadrupole mass spectrometers:

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- 2.2.1.1. Check that calculated ion ratio agrees with the target, if not:
  - 2.2.1.1.1. Check instrument worksheet for the last time the transfer ion tube, housing, and sweep cone was cleaned.
    - 2.2.1.1.1.1. Clean if necessary or consult LC-MS chemist
  - 2.2.1.1.2. Check instrument worksheet for the last time the instrument had a preventative maintenance, calibration, or cleaning of Q0/Q00.
    - 2.2.1.1.2.1. If needed, consult LC-MS chemist
- 2.2.2. For LC-MS-ion trap and Orbi Trap instruments:
  - 2.2.2.1. Check that the full scan spectra contains the expected ions in approximately the expected ratios.
    - 2.2.2.1.1. Check instrument worksheet for the last time the transfer ion tube, housing, and sweep cone was cleaned.
      - 2.2.2.1.1.1. Clean if necessary or consult LC-MS chemist
    - 2.2.2.1.2. Check instrument worksheet for the last time the instrument had a preventative maintenance, calibration, or cleaning of Q0/Q00.
      - 2.2.2.1.2.1. If needed, consult LC-MS chemist
- 2.3. Instrument and Processing Methods
  - 2.3.1. If there appears to be a problem with the instrument or processing methods, inform the LC-MS chemist.

#### 3. Maintenance

- 3.1. If routine maintenance is suggested see SOP-071 LC-MS Routine Maintenance and references therein.
  - 3.1.1. For procedures and cleaning beyond the scope of SOP-071 please consult the LC-MS chemist