Orbitrap Exploris Mass Spectrometer Performance Monitoring and Maintenance

Table of Contents

1 INTRODUCTION 2

2 SCOPE 2

3 EQUIPMENT 2

4 STANDARDS AND CONTROLS 2

4.1 Testmix (Explosives Chemistry) 2

4.2 Calibration Solution 3

5 PROCEDURE 3

5.1 Daily Checks 3

5.2 As Needed Maintenance/Checks 4

5.2.1 Mass Spectrometer 4

5.2.2 UPLC 4

5.3 Mass Calibration 4

6 INSTRUMENTAL CONDITIONS 5

6.1 Testmix 5

6.1.1 UPLC 5

6.1.2 Mass Spectrometer 5

6.2 Mass Calibration 6

6.2.1 Mass Spectrometer 6

7 ACCEPTANCE CRITERIA 6

7.1 Testmix 6

7.1.1 Explosives Chemistry 6

7.2 Mass Calibration 6

7.2.1 Positive Mode 6

7.2.2 Negative Mode 7

8 LIMITATIONS 7

9 SAFETY 7

10 REVISION HISTORY 7

Orbitrap Exploris Mass Spectrometer Performance Monitoring and Maintenance

1 Introduction

This document addresses the performance monitoring and maintenance of the Orbitrap Exploris LC/MS system consisting of a Thermo Scientific Exploris Mass Spectrometer (MS) and a Waters Ultra-High Performance Liquid Chromatograph (UPLC). This system can be used for high resolution accurate mass analyses and unit mass analyses. The instrument is configured with an API source that is capable of electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI), or atmospheric pressure photoionization (APPI). The instrument is primarily used in ESI mode. However, in can be used for APCI or APPI provided the method of ionization is clearly labeled in the resulting data and documentation. Definitions and guidelines are outlined in IOSS-701.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following subdiscipline: Explosives Chemistry.

3 Equipment

- Instrumentation
  - Thermo Scientific Orbitrap Exploris 120, API Source, Xcalibur software (or equivalent)
  - Waters Acquity UPLC (or equivalent)
- Materials
  - Nitrogen, 99.99% (high purity or equivalent)
  - Methanol (Optima grade or equivalent)
  - Deionized Water, 18.2 MΩ·cm (Milli-Q or equivalent)
  - Acetone (HPLC grade or equivalent)
  - Ammonium Nitrate (NH_4NO_3) (reagent grade or equivalent)

Redacted

  - 3.125 mM Ammonium Nitrate Mobile Phase (250 mg to 1 Liter water)
  - Waters Cortecs UPLC C18 1.6 µm, 2.1 mm X 50 mm (or equivalent)
  - 100, 250 or 500 µL LC syringe (Hamilton or equivalent)
  - Pierce FlexMix calibration solution (Thermo or equivalent)
  - General laboratory supplies

4 Standards and Controls

4.1 Testmix (Explosives Chemistry)

The testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. Record preparations in the appropriate discipline/subdiscipline reagent log.

- 100 µg/mL Stock Solution
  Pipette 1 mL of each Redacted standards in a separate 10 mL volumetric flask and dilute to the mark with acetone to achieve a final concentration of 100 µg/mL. Shelf life is two years when stored refrigerated in colored glass. This preparation may be appropriately scaled.

- 10 µg/mL Stock Solution
  Pipette 1 mL of each Redacted into a 10 mL volumetric flask and dilute to the mark with acetone to achieve a concentration of 10 µg/mL. Shelf life is two years when stored refrigerated in colored glass. This preparation may be appropriately scaled.

- Testmix Solution
  For daily use, dilute 20 µL of the 10 µg/mL stock solution to 1 mL with a 50:50 solution of methanol and water.

4.2 Calibration Solution

The FlexMix calibration solution is used for coarse tuning and calibrating the mass spectrometer over the entire mass range. This procedure only needs to be performed when warranted based on system performance or if the system has been significantly moved, down for a long period of time, or undergone a major repair. The calibration solution is purchased from Thermo Fisher Scientific or equivalent.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the instrument log.

A. Record the remaining disk space on the hard drive. Verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.
B. Record the line pressure of the building nitrogen supply or generator (API gas). The regulator should read between 70 and 100 psi. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the nitrogen is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 250 psi remaining.
C. Check the oil level of the vacuum pump.
D. Check the vacuum pressure under instrument status on the tune page. If a green circle with a white check mark in it is present, the system is ready.
E. Prime the UPLC system:
    1. Open the Acquity UPLC Console and select Acquity UPLC system in the menu on the left side of the screen.
    2. From the control drop down menu select ‘System Start Up.’
    3. Confirm for the sample manager (SM) that the strong wash, weak wash, and sample syringe are checked and 3 is entered in the cycles box.

4. Confirm for the binary solvent manager (BSM) that all the boxes are checked and that the duration time is set to 5 minutes.
5. Click the start button to start priming the system.

F. Analyze the testmix
   - Explosives:
     Conduct a performance verification of the appropriate testmix through the column. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the TIC, RICs, and spectra for components in the testmix.

G. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.2 As Needed Maintenance/Checks

The following steps are to be performed as needed. Enter the information in the appropriate instrument log to indicate completion. Refer to IOSS-701 for more information on instrument maintenance and documentation.

5.2.1 Mass Spectrometer

A. Check the faceplate for debris, clean as needed.
B. Bakeout the analyzer as needed.
C. Change the capillary.
D. Check level and color of rough pump oil. Top off or change oil as needed.
E. Perform a full evaluation/calibration. If any steps do not pass, repeat. Perform a full calibration if warranted.

5.2.2 UPLC

A. Replaced solvent frits as needed.
B. Replace drain valve as needed.
C. Replace degasser pump as needed.
D. Inspect pump plungers, diaphragms, and check valves. Replaced if needed.
E. Inspect oven fan.
F. Inspect degasser filters, replace if needed.
G. Check autosampler injection count and performance. Replace components (seals, rotors, stators, needle) if warranted.

5.3 Mass Calibration

The mass calibration procedure should be performed as needed based on system performance, when the instrument has been significantly moved, down for a long period of time, or undergone a major repair.

A. Load a 100, 250 or 500 µL syringe with the Pierce FlexMix Calibration Solution.
B. Connect the infusion syringe to the ESI probe assembly, and place in the syringe pump.

C. Set the syringe pump to the correct syringe type and set the pump rate to 5 µL/minute. The flow rate can be adjusted as needed.
D. Verify that the ESI heater is turned off.
E. Monitor spray stability to ensure a stable spray cutoff is achieved (2 < % RSD <15)
F. On the tune page Calibration tab:
    1. Select “Check” to perform a calibration check, or “Calibrate” to perform a calibration (the “Check, Calibrate if required” option may also be used).
    2. Select “Positive” Ionization for Positive Mode, or “Negative” Ionization for Negative Mode.
    3. Select “Orbitrap Mass” to perform only a mass calibration, or “Orbitrap Mass & System” to perform a full check/calibration.
G. Turn on the syringe pump and verify that the solution is flowing out the ESI needle.
H. Ensure the ESI probe is engaged and turn on the MS.
I. Click the "Start" button at the bottom of the Calibration tab to start the calibration.
J. When the calibration is complete, it will display whether the calibration was successful.
    - If the procedure fails, repeat the calibration.
    - When the procedure passes, save the report and evaluate the calibration solution spectrum using the 'Acceptance Criteria' section.
K. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

6 INSTRUMENTAL CONDITIONS

6.1 Testmix

6.1.1 UPLC

Mobile Phase:	(From discipline-specific procedure)
Flow Rate:	0.5 mL/min
Column:	Waters Cortecs UPLC C18
Injection Volume:	8 µL

6.1.2 Mass Spectrometer

Ionization:	ESI
Polarity:	Negative
Tune File:	exp_tune (or equivalent)
Sheath Gas Flow:	20 (arb)
Aux Gas Flow:	5 (arb)
Sweep Gas Flow:	0 (arb)
Scan Mode:	Full Scan
Scan Range:	200-400 m/z (minimum)
Resolution:	17,500

6.2 Mass Calibration

6.2.1 Mass Spectrometer

Ionization:	ESI
Polarity:	Positive or Negative (select as appropriate)
Scan Mode:	Full Scan
Scan Range:	100-2000 m/z

7 ACCEPTANCE CRITERIA

Redacted

7.2 Mass Calibration

Verify the results of the calibration. The calibration will indicate if the procedure was successful. For reference, the individual ions for the FlexMix calibration solution are listed below for each mode.

7.2.1 Positive Mode

○ Triethylamine	102.1277 m/z
○ Tetramethylpiperidine	142.1590 m/z
○ Caffeine	195.0877 m/z
○ Hexamethoxyphosphazine	322.0481 m/z
○ MRFA	524.2650 m/z
○ Hexakis(2,2-difluoroethoxy)phosphazene	622.0290 m/z
○ Hexakis(2,2,3,3-tetrafluoropropoxy)phosphazene	922.0098 m/z
○ Ultramark	1022.0033 m/z
	1121.9969 m/z
	1221.9905 m/z
	1321.9841 m/z
	1421.9777 m/z
	1521.9713 m/z
	1621.9649 m/z
	1721.9585 m/z
	1821.9521 m/z

7.2.2 Negative Mode

- Trifluroacetic acid 112.9845 m/z
- Pentafluropropionic acid 162.9813 m/z
- Perfluoroheptanoic acid 362.9685 m/z
- [2,4,6-Tris(heptafluoropropyl)-1,3,5-triazine + OH]$^-$ 601.9779 m/z
- [Hexakis(2,2,3,3-tetrafluoropropoxy)phosphazene + TFA]$^-$ 1033.9870 m/z
- Ultramark 1133.9816 m/z
  1233.9752 m/z
  1333.9688 m/z
  1433.9624 m/z
  1533.9560 m/z
  1633.9496 m/z
  1733.9432 m/z
  1833.9368 m/z

8 LIMITATIONS

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

9 SAFETY

Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

10 REVISION HISTORY

Revision	Issued	Changes
00	02/18/2025	New Document