

Tune and Calibration Mixture for LCQ DecaXP^{Plus} and Advantage:

Preparation Process and Specifications

(10mL batch size for field installation)

Lot No. _____ (Date calibration mixture was prepared)

Lot Size: 10mL

1. Materials and Equipment: (record manufacturers and lot numbers)

Chemical or Solvent	Quantity Required	Manufacturer	Lot Number
Methanol	10mL		
Water	10mL		
Acetonitrile	10mL		
Acetic Acid, Glacial	1.0mL		
Caffeine, 1mg/mL in methanol (<i>Caffeine Stock Solution</i>)	1 ampoules (1.0mL)	Sigma	
MRFA	1 bottle	-	
Ultramark 1621	1 bottle	Lancaster	

Equipment or Glassware	Number Required
25mL glass scintillation vial with cap	3
10mL glass pipettes	3
Pipette bulb	1
200-1000µL Pipetteman or equivalent, with 1000µL tips	1
20-200µL Pipetteman or equivalent, with 200µL tips	1
10mL glass volumetric flask	1
25mL glass volumetric flask	1

2. 30X Diluted MRFA Solution in 50:50 methanol:water:

Initials

- a. Combine 10.0mL of methanol with 10.0mL water:

_____ mL methanol

_____ mL water

Mix thoroughly.

- b. Weigh out 6.0mg of MRFA powder:

_____ mg MRFA

- c. Pipette 2.0mL of the 50:50 methanol:water into a labeled, clean, dry vial.

_____ mL 50:50 methanol:water.

Add and dissolve completely the 6.0mg of MRFA from step 2b in the methanol:water.

Label as *MRFA Stock Solution (3.0mg/mL)*.

- d. Pipette 2.9mL of 50:50 methanol:water into another clean, dry vial:

- _____ mL 50:50 methanol:water
- e. Pipette 100 μ L MRFA stock solution from step 2c into the 2.9mL of 50:50 methanol:water from step 2d. _____
- f. Mix thoroughly. Label as ***30X Diluted MRFA Stock Solution (0.1 μ g/ μ L)***. _____

3. Ultramark 1621 Stock Solution:

- a. Put about 23mL of acetonitrile into a 25mL volumetric flask:
_____ mL acetonitrile _____
- b. Pipette 25 μ L of Ultramark 1621 into the 23mL of acetonitrile from step 3a. Use a pipette tip that has been cut off (internal diameter (ID) very approximately 1/8" or 1mm. An exact ID is not essential. The idea is to make pipetting the viscous ultramark easier).
_____ μ L Ultramark _____
- c. Bring the total volume to the 25mL mark with acetonitrile. _____
- d. Mix thoroughly by inverting the capped volumetric flask 20 times. _____
- e. Pour into a clean, dry vial. Label as ***Ultramark 1621 Stock Solution (1/1000 dilution)***. _____

4. ESI Calibration Solution: Caffeine, 3X Diluted MRFA, 50X Diluted Ultramark 1621

- a. Obtain a clean, dry 10mL volumetric flask.
- b. Pipette 5.0mL of acetonitrile into the 10mL volumetric flask:
_____ mL acetonitrile _____
- c. Pipette 200 μ L of the ***Caffeine Stock Solution*** into the volumetric flask:
_____ Caffeine stock solution ampoules _____
- d. Pipette 100 μ L of ***30X Diluted MRFA Stock Solution***
- e. (***0.1 μ g/ μ L***) into the 10mL volumetric flask:
_____ μ L 30X Diluted MRFA stock solution _____
- f. Pipette 100 μ L of ***Ultramark 1621 Stock Solution (1/1000 dilution)*** into the 10mL volumetric flask:
_____ μ L Ultramark 1621 stock solution _____
- g. Pipette 100 μ L of glacial acetic acid into the 10mL volumetric flask:
_____ μ L glacial acetic acid _____
- h. Bring the total volume to the 10mL mark using 50:50 methanol:water. _____
- i. Cap the volumetric flask and invert 20 times to mix. _____
- j. Pour the solution into a clean, dry vial. Label the vial _____

Diluted Calibration Mix (Caffeine, 3X diluted MRFA, 50X diluted Ultramark). Write the **Lot Number** and **expiration date** on the label. _____

5. Store the Calibration Mix at 2 – 8 °C (Refrigerator temperature). Under these conditions, the expiration date is one month from the preparation date.

Changes to Calibration Procedure and Suggested New Target Values

1. Use the above *Calibration Mix for LCQ DecaXP/Advantage*.
2. Install the **sweep cap** prior to calibration.
3. Verify that the **instrument specific** target values given in the Tables below are used in the Calibration Tune File.

Positive Ion Mode DILUTED CAL MIX		
<i>DecaXP</i> Target Values	Scan Modes	<i>Advantage</i> Target Values
1 X 10e8	Full	5 X 10e7
5	uScans	5
2 X 10e7	SIM	2 X 10e7
4 X 10e7	MSN	4 X 10e7
4 X 10e7	Zoom	4 X 10e7

Negative Ion Mode DILUTED CAL MIX		
<i>DecaXP</i> Target Values	Scan Modes	<i>Advantage</i> Target Values
2 X 10e7	Full	2 X 10e7
5	uScans	5
2 X 10e7	SIM	2 X 10e7
2 X 10e7	MSN	2 X 10e7
2 X 10e7	Zoom	2 X 10e7