MS calibration and tune

Friday, August 03, 2012 1:26 PM

Calibration

Purpose: Establish X-axis (m/z). Calibration is independent of source type

LTQ/FT-Hybrid Positive Ion Mode Calibration Solution

The LTQ/FT-hybrid positive ion mode calibration solution consists of caffeine, MRFA, Ultramark 1621, and n-butylamine in an acetonitrile:methanol:water solution containing 1% acetic acid.

To prepare the positive ion mode calibration solution

1. Pipet 20 µL of the caffeine stock solution into a light-protected, clean, dry 10 mL volumetric flask.
2. Pipet 100 µL of the MRFA stock solution into the flask.
3. Pipet 100 µL of the Ultramark 1621 stock solution into the flask.
4. Pipet 100 µL of the stock solution of n-butylamine into the flask.

Caution Use only glass pipets or stainless steel syringes when measuring glacial acetic acid. Using plastic pipet tips causes contamination of acid stock solutions that can introduce contaminants in the calibration solution.

5. Pipet 100 µL of glacial acetic acid into the flask.
6. Pipet 5 mL of acetonitrile into the flask.
7. Bring the volume of the solution up to the 10 mL-mark on the flask with 50:50 methanol:water.
8. Mix the calibration solution thoroughly.
9. Transfer the solution to a light-protected, clean, dry vial.
10. Label the vial Positive Ion Mode Calibration Solution and store it in a freezer until it is needed.

LTQ/FT-Hybrid Negative Ion Mode Calibration Solution

The LTQ/FT-hybrid negative ion mode calibration solution consists of sodium dodecyl sulfate, sodium taurocholate, and Ultramark 1621 in an acetonitrile:methanol:water solution containing 1% acetic acid.

To prepare the negative ion mode calibration solution

1. Pipet 100 µL of the sodium dodecyl sulfate stock solution into a light-protected, clean, dry 10 mL volumetric flask.
2. Pipet 100 µL of the sodium taurocholate stock solution into the flask.
3. Pipet 100 µL of the Ultramark 1621 stock solution into the flask.

Caution Use only glass pipets or stainless steel syringes when measuring glacial acetic acid. Using plastic pipet tips causes contamination of acid stock solutions that can introduce contaminants in the calibration solution.

4. Pipet 100 µL of glacial acetic acid into the flask.
5. Pipet 5 mL of acetonitrile into the flask.
6. Bring the volume of the solution up to the 10 mL-mark on the flask with 50:50 methanol:water.
7. Mix the solution thoroughly.
8. Transfer the solution to a light-protected, clean, dry vial.
9. Label the vial Negative Ion Mode Calibration Solution and store it in a freezer until it is needed.

To prepare Caffeine Stock Solution

1 mg/mL stock solution of caffeine in 100% methanol is provided with your LTQ Orbitrap Series MS detector. You can also order this solution through Sigma. The Sigma product number for this solution is C6035.

To prepare the MRFA stock solution

1. Obtain the vial of L-methionyl-arginyl-phenylalanyl-alanine acetate × H2O (MRFA) in your accessory kit. In this form, the MRFA sample has an average molecular weight of 523.7 u. Carefully weigh 3.0 mg of the MRFA sample.
2. Dissolve the MRFA sample in a total volume of 1.0 mL of 50:50 methanol:water. Mix the solution (5.0 nmol/µL) thoroughly.
3. Transfer 50 µL of the 5 nmol/µL solution into a clean polypropylene tube.
4. Add 1.45 mL of 50:50 methanol:water to the tube. Mix this solution (166.7 pmol/µL) thoroughly.
5. Label the tube MRFA stock solution and store it in a freezer until it is needed.

To prepare the Ultramark 1621 stock solution

1. Obtain the vial of Ultramark 1621 in your accessory kit.
2. Using a syringe, measure out 10 µL of Ultramark 1621, and dissolve it in 10 mL of acetonitrile.
3. Mix the solution thoroughly.
4. Label the vial Ultramark 1621 stock solution and store it in a freezer until it is needed.

To prepare the n-butylamine stock solution

1. Using a syringe, transfer 5 µL of n-butylamine to a 25 mL (minimum) volumetric glass flask.
2. Add 9995 µL of 50:50 methanol/water to the flask.
3. Mix the solution thoroughly.
4. Transfer the solution to a vial.
5. Label the vial N-butylamine stock solution (5/1000 dilution).

Sodium Dodecyl Sulfate Stock Solution

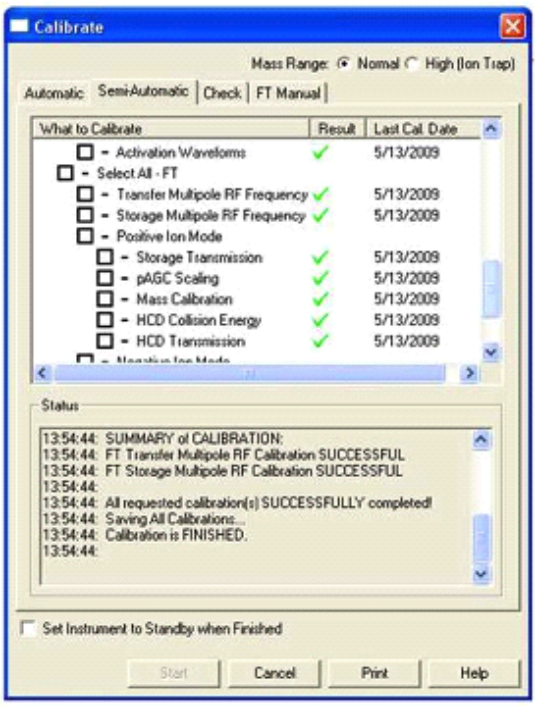
To prepare the sodium dodecyl sulfate stock solution

1. Obtain the vial of sodium dodecyl sulfate. In this form, the sample has an average molecular weight of 288.4 u.
2. Prepare the stock solution of sodium dodecyl sulfate by dissolving 2.88 mg in 10 mL of 50:50 methanol:water.
3. Mix the solution (1.0 nmol/µL) thoroughly.
4. Label the vial Sodium Dodecyl Sulfate stock solution (1 nmol/µL).

To prepare the sodium taurocholate stock solution

1. Obtain the vial of sodium taurocholate. In this form, the sample has an average molecular weight of 537.7 u.
2. Prepare the stock solution of sodium taurocholate by dissolving 5.38 mg in 10 mL of 50:50 methanol:water.
3. Mix the solution (1.0 nmol/µL) thoroughly.
4. Label the vial Sodium Taurocholate stock solution (1 nmol/µL).

Calibration procedure (Semi-Automatic calibration)

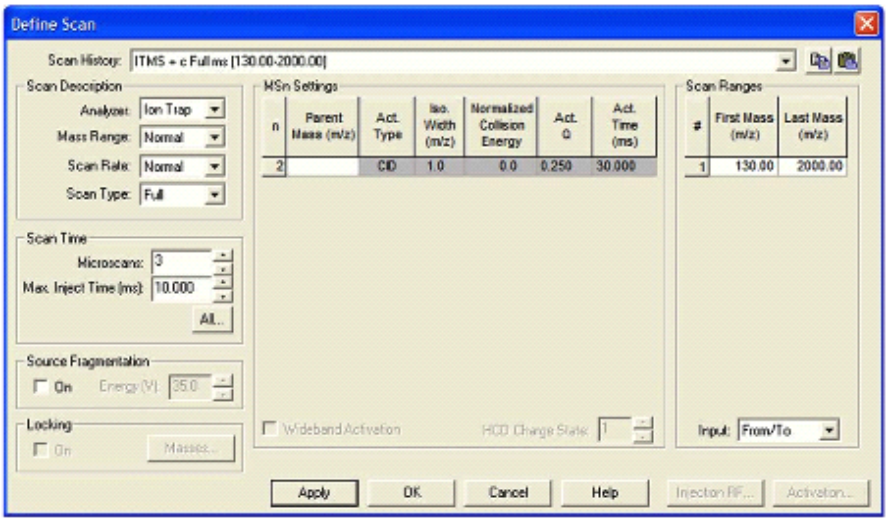


Usually, you do not need to perform a complete ion trap calibration or an FT ion transmission calibration unless the hardware is modified in some way. However, it is necessary to repeat the electron multiplier calibration monthly and the FT mass calibration weekly. To perform an FT calibration, the ion trap has to be successfully calibrated before. The calibration masses and all experimental parameters like target values, scan ranges, resolution settings, etc. are set automatically.

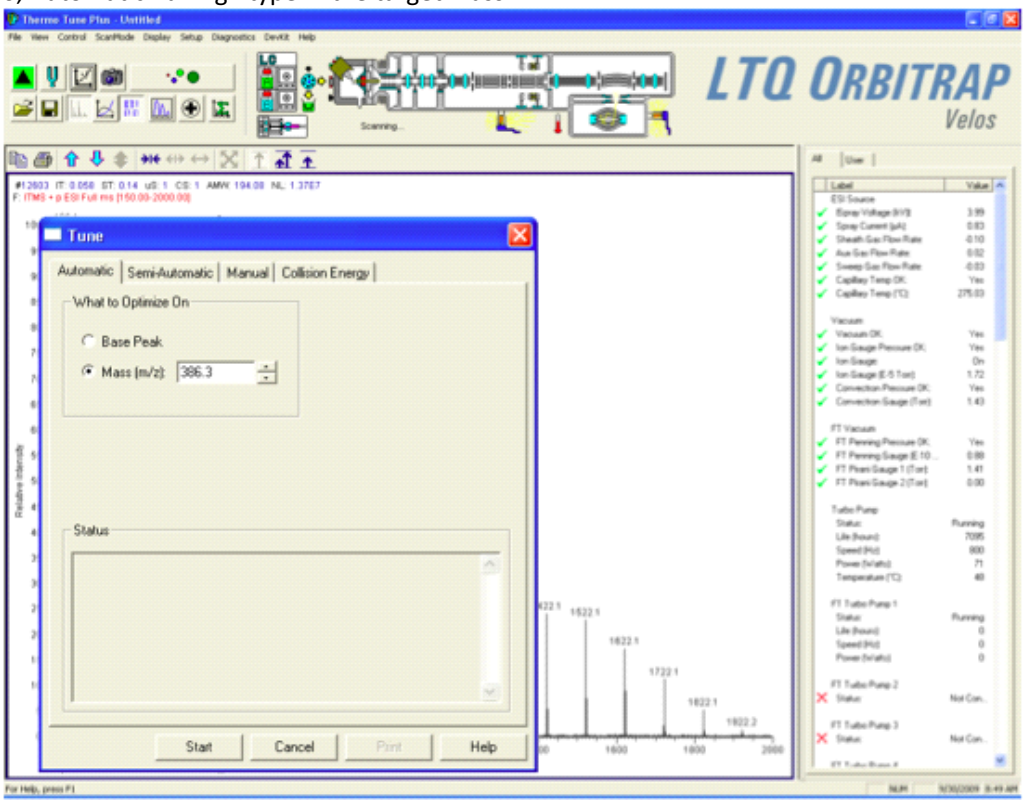
Tuning the Ion Trap

Purpose: Tuning optimizes voltage settings for highest sensitivity. Tuning is highly compound and technique dependent.

1, Use nanospray system.
2, Connect the blank silica analytical column to the syringe pump. Use Angiotensin I as tuning solution.
3, Turn on Syringe Pump to Infuse Sample. Flow rate is 600nl/min.
4, Turn on MS scan. Make sure the injection time is stable.
5, Open the Define Scan Dialog Box and Modify Parameters.



6, Automatic Tuning - type in the target mass



7, Save the tune method after a successful tuning.