# Notes on Troubleshooting LC/MS Contamination

## Table of Contents

- General Discussion ............................................................................................................. 2
- Identifying the source of contamination ............................................................................. 2
- HPLC troubleshooting. ....................................................................................................... 3
- Contamination in the MS .................................................................................................... 3
  - Phthalate Contamination  (391, 413 798, 803) .............................................................. 3
  - +44 Series ........................................................................................................................... 5
  - Background Ramps Up With Gradient ............................................................................ 5
  - +59 Series ......................................................................................................................... 6
  - +77 Series ......................................................................................................................... 6
  - +74 Series ......................................................................................................................... 6
  - 615.7 and 1229.8  CHAPS ............................................................................................... 6
  - Nanospray Peaks (371, 445 and others) ......................................................................... 8
  - +136 Series ....................................................................................................................... 8
  - Pentafluoropropionic Acid ............................................................................................... 9
  - Water ................................................................................................................................ 9
  - 798 and 803 Contamination ............................................................................................ 9
  - 453.3, 679.5 and 905.7 Contamination .......................................................................... 10
  - Peak Clusters at +21, -17, -35, -52 ............................................................................... 10
  - Iron-Acetate clusters 538, 555 and 534 ....................................................................... 10
  - Common Contaminants in Protein/Peptide Analysis ..................................................... 10
  - Removing Ultramark Contamination ............................................................................ 14
  - Trouble Shooting Excessive Noise In the LCQ, DUO and DECA .............................. 16
  - HPLC Equipment as sources of contamination ......................................................... 18
  - Ghost Peaks .................................................................................................................... 18
  - Purge Procedure for Surveyor MS Pump Pulse Dampener ........................................... 19
General Discussion

Chemical contamination is one of the more common problems with LC/MS. I recommend using the highest purity chemicals available.

- Try Burdick and Jackson solvents and water. These seem to be uniformly better than other suppliers. Switch to this brand if you have contamination problems.
- I recommend this high purity acetic acid: Aldrich p/n 38,012-1 double distilled in Teflon bottle $175 for 500 mL

In my experience a chemical contamination problem (with specific mass peaks) is almost never deep inside the MS. I always prioritize like this:
1. HPLC pre or trap column
2. HPLC column
3. HPLC hardware
4. Chemicals
5. ESI or APCI probe
6. Spray shield area
7. Heated capillary – reaming only removes blockages
8. MS Tube lens/skimmer
9. MS Octopoles/Multipoles
10. MS Trap
11. MS Dynode

Mostly if 8 – 11 are dirty you will see random peaks not specific masses.

Identifying the source of contamination

To Start

1. Is the contamination always there or does it elute as specific peaks during the HPLC run? If the later the problem is most likely with the HPLC.
2. Turn off flow. If the noise disappears take the column out of the flow path and test again. If the noise is still there try these items.
3. If you suspect that there is chemical contamination from the HPLC mobile phase use a clean syringe and tubing to infuse the mobile phase components individually into the MS (e.g. water, then water + acid, then methanol or acetonitrile). Most commonly the acid is the source of contamination.
HPLC troubleshooting.

Many times contaminant peaks occur during a gradient. Try this procedure to locate the source of the contamination:

- Inject a solvent blank.
- Make a zero volume injection
- Run a gradient with the injection valve or autosampler out of the liquid flow.

Contamination in the MS

If the noise disappears when HPLC flow is turned off and you have eliminated contamination in the mobile phase:

- Trim or replace the sample tube and set it to the correct distance within the needle.
- Clean the ESI spray nozzle (cone and needle).
- Replace the Teflon needle seal behind the needle
- If still noisy, the cause could be neutral chemical noise (non-ionized materials). Change source conditions (e.g. increase heated capillary temperature 10 or 20 C, increase Sheath gas flow 10 or 20 units, increase Auxiliary gas flow 10 units.

Other possibilities

- Solvent reservoir pickup filters are common sources of contamination. Usually the aqueous phase one will be the first to go.
- Offline solvent filters are common sources of contamination. I have seen serious contamination with the Nylon 0.22 um filters used to filter buffer solutions.
- You might want to think about the containers you are using for your acetonitrile. We had a similar problem that we traced to using glass scintillation vials with polypropylene insert caps to make up our solvent. After switching to a different type of glass vial the problem went away.
- Surveyor MS pump. The pump has a pulse dampener which is not directly in the liquid flow. It is Tee'd off the flow. A dampener containing a badly contaminated working fluid can leach contamination into the LC solvent. A thorough cleaning procedure is listed at the end of this document.

Phthalate Contamination  (391, 413 798, 803)

391 protonated dioctyl phthalate (M+H)+.
413 sodium adduct of dioctyl phthalate (M+Na)+.
803 sodiated dimer of dioctyl phthalate (2M+Na)+.

This is usually from contaminated solvents. It can concentrate on the column and elute during a gradient. Follow the normal solvent checking procedures.
Glassware run through a “dishwasher” often picks up phthalate contamination. Remove this with a rinse of 30% nitric acid followed by a rinse with 2M NH4OH.

The APCI probe can retain this. Baking the APCI will eliminate this problem. Try 550 deg C for 15 min.
**+44 Series**

Possible polymer contamination. If you have shot detergent containing samples on the system that could explain the background. Also PEGs and other ethoxylated polymers give +44 ion series. The PEG’s could also be from the water, or extracted polymer from plastic ware/silicon coatings.

Example:

Noise is overwhelming the entire spectrum. Every 44 there is a peak from 400 to 800. They see the problem when they have an LC input. The impurity increased as the gradient went on. Mobile phase is acetonitrile:water. Pattern of 75 from 1500 up to 1900. They changed the pump and the rest of the system components. ESI probe. Cleaned the spray shield, needle, nozzle, changed the ESI capillary. They have changed bottles of both acetonitrile and water. She will fax some example data. Also she will try spectra with cap on heated capillary, cap off heated capillary no flow and LC flow on.

First pump was HP 1100 (mixing from 2 different bottles. Second pump was Beckman (isocratic only so they pre-mixed)

Infusing methanol get peaks at 812, 817, 1211.7, 1610.8 level is E5. Noise is below 1%. What are these?
Pumped in 50:50 acetonitrile:water with the HPLC. Got the same patterns that they had sent to me. Not the same ones as with the infused methanol except for 812 which is still there. Level is still E5 also. The noise is about 15%.

She will try acetonitrile:water (as used with the Beckman pump) through the syringe pump.

9-9-97: New fax from her. She saw the contamination with the acetonitrile:water mixture. I asked her to try the water and the acetonitrile separately. The water is from their nanopure system. The acetonitrile is from Fisher. She wants to clean the API but this is unlikely to be the problem. Probably the source is a filter in the water system.

If the problem might be from solvent clusters try source CID at 10 to 20% to confirm clustering.

If every infused solvent shows contamination it may be due to some residual olyethylene glycols (PEG’s) in the MS. They could be in the API stack, the spray shield area or in the ESI probe, or all of the above. Flush the spray shield, capillary and needle with methanol, acetonitrile or isopropyl alcohol. This means around the spray shield area, directly on the end of the heated capillary, around the base of the heated capillary at the PEEK sleeve and all around the needle of the ESI probe.

**Background Ramps Up With Gradient**

This can be contaminated organic phase or the high organic content can be eluting retained junk from the column. Try Burdick and Jackson organic phase and a new column.
+59 Series

And what about a +59 ion series? I'm using acetic acid in a 5% concentration. 59 Da is the mass of the acetate ion. But how acetate interact with the peptide? Is it possible to have acetate polymerization? There is another explanation for the +59 ion series?

Polymers of +59 might be iron in some form, presumably leaching from the steel in acid. We saw a lot of that before we eliminated as much metal as possible. We also saw +59 adducts onto larger, acidic peptides.

+77 Series

I had been experiencing contamination on the Deca (ca 77 u clusters, mostly across the mass range). This appears to have resolved itself upon replacing the heated capillary and seal (kelrez ?, the soft black one). The seal was visibly "chewed-up". I don't know how this might related to the problem, but at least it appears to be gone.

Contamination peaks related to a bad heated capillary o-ring is a new one on me. I could postulate that the worn o-ring was allowing leakage into the tube lens skimmer area and the leak was also leaching something out of the o-ring. Normally the o-ring is a total block and any polymers in it would not enter the MS.

+74 Series

Peaks at 536, 610 and 684 have been seen with LCQDecaXP and LCQAdvantage and TSQ Quantum. The contamination came from connecting the opaque silicon tube to the API source housing. The proper tubing is Tygon.

The instrument parts kits contain both types of tubing. The TSQ Quantum contains a 12-foot length of clear Tygon tube and a 2.5-foot length of opaque silicon tube. The intended function of the silicon tube is to connect from the waste container (P/N 00301-57020) to the fume exhaust system. If the silicon tube is instead connected to the API source housing, background ions at m/z 536, 610, and 684 might be observed.

Solution: Remove all silicon tubing from the solvent waste system.

615.7 and 1229.8 CHAPS

615.7 is MH+ of CHAPS and 1229.8 is (2M+H)+ in the sample. Removal can be tough. Acetone precipitation removes the excess CHAPS but there may still be an appreciable amount remaining (determined by the above ions in the mass spectrum).

CHAPS won't kill your SCX chromatography, but it will chromatograph nicely on reversed phase and you'll get an intense ion at 615 m/z (MH+).

LC/MS system contamination by CHAPS.
If the contamination came from your sample (likely with CHAPS contamination). Then all the bits that contacted the liquid sample should be thoroughly cleaned or replaced. This means from the injector to the skimmer in the MS.

Details
On the HPLC side I recommend a complete replacement of the parts that were touched by the CHAPS. This starts with the sample pickup needle, continues through the injection loop to the high voltage tee and column and spray tip (for nanospray). You need to replace all at once. For example, I expect that some CHAPS upstream from a new column may have already contaminated it.

On the MS side if this is ESI disassemble the probe and clean. All fittings to the probe should be replaced. Internally the ion transfer tube, skimmer and tube lens are stainless and can be soaked in 30% nitric acid for cleaning. Usually I do not worry about contamination past the skimmer. Very little material gets deeper into the MS. You should also clean any areas that the spray might contact.

Be thorough!
Nanospray Peaks (371, 445 and others)

Peaks at 371 and 445 amu are commonly observed with the nanospray probe. This may result from the silica.

A long troubleshoot for a source of contamination in Nanospray ended when the user replaced:

97044-60290 HV Emmitter nanospray ay $141 (the nanospray micro-Tee)

The spectrum was: 442.0, 459.7, 477.5, 495.4 (see picture)

The emitter/Tee assembly had obviously become contaminated. This piece could be hard to clean as the electrode side of the Tee cannot be taken apart and assembled again correctly.

+136 Series

Background (contamination) has a repeating sequence of 136 amu.

TFA sodium salt is 136 and there was a nice spectrum of peaks 136 units apart when the sample contained it.

Try removing TFA from the sample and/or mobile phase. TFA may be difficult to remove from the column so take off the column and flow mobile phase directly into the MS. This could be from the chromatograph or internal to the LCQ. Identify the source by infusing methanol with a clean syringe and a new piece of tubing.

If the source of the contamination is the LCQ then try the following:
• Using the syringe pump spray acetonitrile at a relatively high flow rate and cycle the heated capillary from 150 to 270 C two times. Allow a few minutes before changing the temperature.
• Disassemble and clean the ESI head.
• Disassemble and clean the API stack.
• Remove and clean the octopoles.
• Disassemble and clean the Trap.

If the source of the contamination is the HPLC I will leave you to deal with it.

• Test the mobile phase for contamination by infusing at 500 uL/min with 500 uL syringe.
• To minimize contamination use fresh chemicals, particularly acids.

**Pentafluoropropionic Acid**

May stick on PEEK tubing and fittings. Usually I associate contamination with areas that are poorly swept by the flowing liquid (e.g. unions).

If you have concerns about the pentafluoropropionic acid then use fused silica and steel unions. The ferrules should be kel-F. PEEK tube nuts should be ok. They do not touch the liquids.

What is the purity of the pentafluoropropionic acid? I always worry about purity of mobile phase additives.

**Water**

If you see possible contamination use high grade bottled water (Burdick & Jackson HPLC grade.) Avoid ANY nanoPure or MilliQ water. The equipment may not be maintained correctly.

**798 and 803 Contamination**

They are two main ions were seen, one at 798.1 and 1 at 803.4. They both appeared at high organic during the LC/MS run. Because it was also seen without the column and trap, it must either be coming from the HPLC or MS.

This may be phthalate contamination. 798 could be the gas phase dimer of di-octylphthalate (MW 390) plus ammonium [2M+NH4]⁺. The m/z 803 would then be [2M+Na]⁺. Sometimes this can be really hard to track down. If it's coming from your HPLC it can concentrate on your column until you ramp the gradient to knock it off. We recommend checking the solvents by infusion to determine if it's coming from your HPLC or if it's in one of your reagents (e.g., TFA).

In this case the source was finally identified as the dishwasher used to clean the HPLC reservoir bottles. These could be cleaned by rinsing well with 30% Nitric Acid followed, after a water rinse, by 2M NH4OH. This has removed many odd artifacts from our LC/MS/MS runs.
**453.3, 679.5 and 905.7 Contamination**

Nylon HPLC solvent filters can produce nylon (6,6monomer) peaks at masses of 226 Da a dimer 452, trimer 678 and tetramer 905 Da. The contaminant is very hard to get rid of since it binds very well to C18.

**Peak Clusters at +21, -17, -35, -52**

+21 is sodium, -17, -35, -52 are various losses of ammonia and water (if you look at the -17 ion you will probably see a small amount of the -18, as well).

**Iron-Acetate clusters 538, 555 and 534**

Mobile phases with acetic acid can react with iron in the LC/MS system to produce Iron-acetate clusters (as established by Ijames, Dutky, and Fales: J Am Soc Mass Spec 6 1226 1995.) The general formula is FeO(CH3COO)6(L)x where L is a ligand from the mobile phase (water or organic component, acetonitrile solvent or water. 538 could be 6 acetates with no additional ligand. The 555 presumably has additional water.

533.88 could be a chromium cluster similar to the iron 537.88. Stainless steel in the system contains both iron and chromium. Potential sources in the ESI probe could be the ESI spray needle or the with the metal needle kit. This is not common try replacing the needle.

**Common Contaminants in Protein/Peptide Analysis**

From Sequest Browser (Bill Lane)
The lines have the following format:

MH+ z graphlabel SEQUENCE description after the number sign

note that the first column is the calculated MH+ not the m/z of the z need not be sorted
graphlabel, (no longer optional), will be displayed on the chromatogram, marking the peak to which it corresponds SEQUENCE will be displayed as a hyperlink appended to the description put just “X” (without the quotes) for the SEQUENCE if not applicable

entries were originally made with the experimental m/z OBSERVED not calculated! They should vary between exact & average mass since they were determined by TSQ and LCQ over a long period of time. Since tolerance was expected to be +/- 1.0, no attempt has been made to correct these values.

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1657.8 2 Keratin SGGGSSGAGINYYOR # Keratin 67K Tyll CytoKer 1
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2240.1 3 Keratin ADLEFQIESLTEELAYLKK # Keratin Tyll CytoSkel 10 (Mx->F)

Type II Cyto Keratin 7
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1060.6 2 Keratin TLLIDNTR # Keratin Tyll CytoKer 7
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1315.7 2 Keratin LDNLLEATLQQAK # Keratin Tyll Cytoskel 7
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1851.9 2 Keratin TLNDMRQEYEELIQAQ # Keratin Tyll Cytoskel 7
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1350.7 2 Keratin IGLGGRGGSGGSYGR # Keratin Tyll CytoKer 10
2872.4 3 Keratin NVSTGDVNMNAPGVDLTQLLNMR # Keratin Tyll Cytoskel 10
2082.9 2 Keratin AETECNTETEYQQLLIDK # Keratin Tyll CytoSkel 10
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1003.5 2 Keratin SEITELRR # Keratin Tyll CytoSkel 10
1241.6 2 Keratin SLLEGEGSSGGSGR # Keratin Tyll CytoSkel 10
1381.6 2 Keratin ALEESNYELEKG # Keratin Tyll CytoSkel 10
995.5 1 Keratin IKEWYEK # Keratin Tyll CytoSkel 10
2904.3 2 Keratin NVSTGDVNMNAPGVDLTQLLNMR # Keratin Tyll CytoSkel 10
2240.1 3 Keratin ADLEFQIESLTEELAYLKK # Keratin Tyll CytoSkel 10 (Mx->F)

Type II Cyto Keratin 8
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1344.7 2 Keratin ASLEAAIADEOR # Keratin Tyll CytoKer 8
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**Porcine Trypsin autolytic fragments observed**

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**trypsin-like** artifact sequences below are "best-fit" sequences and not intended to be rigorously determined sequence interpretations. They are merely entered to flag these ions as possible contaminants.

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UNCHARACTERIZED POLYMER (44AMU SUBUNIT)
#801.7 2 * X # 801.7 series +44 ladder 1
#845.65 2 * X # 801.7 series +44 ladder 2
#889.72 2 * X # 801.7 series +44 ladder 3
#933.87 2 * X # 801.7 series +44 ladder 4
#977.87 2 * X # 801.7 series +44 ladder 5
#1021.87 2 * X # 801.7 series +44 ladder 6
#1065.61 2 * X # 801.7 series +44 ladder 7
#1110.00 2 * X # 801.7 series +44 ladder 8
#1154.13 2 * X # 801.7 series +44 ladder 9
#1198.1 2 * X # 801.7 series +44 ladder 10
#1242.1 2 * X # 801.7 series +44 ladder 11
#1286.1 2 * X # 801.7 series +44 ladder 12
#1330.1 2 * X # 801.7 series +44 ladder 13
#1374.1 2 * X # 801.7 series +44 ladder 14
#1418.1 2 * X # 801.7 series +44 ladder 15
#1462.4 2 * X # 801.7 series +44 ladder 16
#1506.4 2 * X # 801.7 series +44 ladder 17
#1550.5 2 * X # 801.7 series +44 ladder 18
#1594.5 2 * X # 801.7 series +44 ladder 19
#1638.5 2 * X # 801.7 series +44 ladder 20

523 1 * X # unknown

UNCHARACTERIZED POLYMER (44AMU SUBUNIT)
300.3 1 * X # 300.3 series +44 ladder
344.1 1 * X # 300.3 series +44 ladder
388.13 1 * X # 300.3 series +44 ladder
432.20 1 * X # 300.3 series +44 ladder
476.20 1 * X # 300.3 series +44 ladder
520.30 1 * X # 300.3 series +44 ladder
564.30 1 * X # 300.3 series +44 ladder
608.30 1 * X # 300.3 series +44 ladder
652.30 1 * X # 300.3 series +44 ladder
696.30 1 * X # 300.3 series +44 ladder
740.30 1 * X # 300.3 series +44 ladder
784.30 1 * X # 300.3 series +44 ladder
828.30 1 * X # 300.3 series +44 ladder

392.10 1 Z X # Zwit-316 monomer
783.40 1 Z X # Zwit-316 dimer

PERSISTENT TUNING SOLUTION IONS
522.5 1 U X # Ultramark
1022.5 1 U X # Ultramark
1122.5 1 U X # Ultramark
1222.5 1 U X # Ultramark
1322.5 1 U X # Ultramark
1422.5 1 U X # Ultramark
1522.5 1 U X # Ultramark
1622.5 1 U X # Ultramark

ANGIOTENSIN STANDARDS (ANGIOMIX)
884.07 2 RVVYHPI RVVYHPI # xx Angio I 2-7
918.09 2 RVVYHPI RVVYHPIF # bo Angio I 2-7
932.12 2 RVYIHPF RVYIHPF # hu Angio I 2-7
1263.50 3 DRVYHPPHL DRVYHPPHL # bo Angio I
1297.50 3 DRVYIHPFHL DRVYIHPFHL # hu Angio I
1297.50 2 DRVYIHPFHL DRVYIHPFHL # hu Angio I

UNKNOWN ARTIFACTS -- NOT SEEN IN A WHILE
883.0 2 unk_x85 X # 285 385 485 585 unk
869.0 2 unk_x85 X # 285 385 485 585 unk
983.5 2 unk_x85 X # 285 385 485 585 unk

BSA TRYPTIC PEPTIDES
1479.8 2 BSA LGEYGFQNALIVR # BSA
1567.7 2 BSA DAFLGSFLYEYSR # BSA
1305.7 2 BSA HLVDEPQNLIK # BSA
LC-MS Contamination

1443.6 2 BSA YICDNQDTISSK # BSA
1002.6 2 BSA LVVSTQTALA # BSA (cterm)
1640.3 2 BSA KVPQVSTPTLVEVSR # BSA
1440.8 3 BSA RHPEYAVSVLLR # BSA
1400.7 2 BSA TVMENFVAFVDK # BSA
1072.6 2 BSA HCIAEVEK # BSA
1567.7 2 BSA DAFLGSFLYEYSR # BSA
1491.8 2 BSA FYAPELLYYANK # BSA
732.5 1 BSA GLVLIAF # BSA

CASEIN TRYPTIC PEPTIDES
1384.7 2 Casein FFVAPFPEVFGK # bo Casein alpha-S1
1267.7 2 Casein YLGYLEQLLR # bo Casein alpha-S1
742.5 1 Casein GPFPILV # bo Casein alpha-S1
1251.7 2 Casein YIPIQYVLSR # bo Casein alpha-S1

PROTEASE I PRECURSOR (API) (LYSYL ENDOPEPTIDASE)
1830.8 2 API_ACHLY SDQYGRVFTSWTGGGAAA # Lys-C
1367.7 2 API_ACHLY VFTSWTGGGAAAASR # Lys-C
2055.0 3 API_ACHLY RVLGQLHGGPSSCATGNT NR # Lys-C
1366.7 2 API_ACHLY RISNSTPSTSFV # Lys-C
1064.6 2 API_ACHLY DIIRAVGAYS # Lys-C

Removing Ultramark Contamination

Empty the syringe that contained the tuning solution.
Flush the syringe 3 times with HPLC grade acetone
Set the sheath gas to 80
Manually inject acetone three times
Open the source and squirt acetone (clean squeeze bottle) onto the end of the fused silica and on the heated capillary and the spray shield. Thoroughly flush this area then wipe dry with Kim wipes.
Repeat this procedure with acetonitrile then inject methanol and water and look for the Ultramark.

Another Technique
Before the sensitivity test is run you must clean out the residual Ultramark with methanol or isopropanol. Flush spray shield, capillary and needle with alcohol, then:
1 uL syringe with methanol
2 Cool to 150C
3 Heat capillary to 270C
4 Repeat 2 times.
Must get rid of the Ultramark because it is an electron acceptor.

The APCI is a particular problem with Ultramark contamination because the heat causes the Ultramark to boil out of the contamination sites. These areas must be cleaned thoroughly. The areas of concern are the outer surface of the heated capillary, the PEEK bushing between the heated capillary and the spray shield, the o-ring and the inner diameter of the spray shield. Disassemble the API stack and clean these thoroughly with acetone and then methanol. You might replace the bushing and o-ring.
Trouble Shooting Excessive Noise In the LCQ, DUO and DECA

The purpose of this document is to help isolate the cause of noise and to provide possible causes and fixes. The type of noise covered in this document is defined as random noise spikes seen in the spectrum. The sources of noise covered are: Chemical, ESI source, Main RF, Dynode, Multiplier and associated electronics and cabling.

The manufacturing specification for random noise spikes is:
Height of noise spikes should be less than the low E4 range.

1) If you suspect that there is chemical contamination from the HPLC mobile phase use a clean syringe and tubing to infuse the mobile phase components individually into the MS (e.g. water, then water + acid, then methanol or acetonitrile). Most commonly the acid is the source of contamination.

2) If the noise disappears when HPLC flow is turned off and you have eliminated contamination:
   a) Trim the sample tube and set it to the correct distance within the needle.
   b) If still noisy, the cause could be chemical noise. Change source conditions (e.g. increase heated capillary temperature 10 or 20 C, increase Sheath gas flow 10 or 20 units, increase Auxiliary gas flow 10 units.
   c) If still noisy, there is a possibility of a bad 8kv-power supply, HV cable or if this is an LCQ (API 1) and spray current is high, there may be moisture between HV connector and ESI probe metal can.

3) If there is still excessive noise with the heated capillary capped, the ESI source is not the problem.
   a) Go to Diagnostics - Toggles and Detectors and turn off the Main RF.
      1) If noise is gone, then clean the Trap (End Caps, Ring Electrode and Insulators).
      2) Inspect the Insulators for scratches, deposits, chips or cracks. Inspect the RF feed through for deposits. Replace parts if necessary.
      3) If still noisy, go to next step.
   b) Go to Tune - Multiplier and turn off the Dynode.
      4) If noise is gone, then clean the Dynode and Shields. Scratches on the Dynode are also a source of noise. Inspect the Dynode feed through for deposits. Replace parts if necessary.
      5) If still noisy, possibly the Dynode power supply is bad or go to the next step.
   c) Go to Diagnostics - Toggles and Detectors and turn off the Multiplier
      6) i) If the noise is gone, rinse the multiplier with methanol and blow dry.
      7) ii) If still noisy, possibly the multiplier power supply is bad or go to the next step.
   d) Possible bad Top cover PCB, Blue cable, Acquisition pcb, or loose or dirty or multiplier anode.

When finished turn on all devices turned off during trouble shooting.
Cleaning Processes (preferably done by a qualified service engineer)

Cleaning the Dynode
1) Remove the dynode power supply, and then remove the dynode feed through from the manifold.
2) Loosen the set screw attaching the dynode to the shaft.
3) Sonicate the dynode in acetone for 1 minute then 1 minute in methanol.
4) Blow-dry with clean dry gas. Do not touch the dynode, even with gloves (especially when wet with solvents.) Handle with a kimwipe or a lint free paper.
5) Reassemble dynode onto the shaft and feed through, blow off parts, then insert into the manifold.
6) While the dynode is out, clean the shields by sonicating them in methanol. Reinstall them, then reinstall the dynode.

Cleaning the multiplier
1) remove the analyzer assembly, then the multiplier.
2) Rinse in methanol and blow dry. Blow off the anode (mounted in top cover assembly).
3) While the multiplier is removed, check to see if the anode is loose. It should be hand tight. Do not use any tools to tighten or loosen it. Clean if necessary.
4) Reinstall multiplier and blow off the analyzer assembly before installation.

Cleaning the trap end caps, ring electrode and quartz insulators
1) sonicate the parts separately, first in acetone for one minute, then in methanol for one minute. If the insulators are cleaned, a 24 hour pump down will be required, or RF noise will occur.
2) If parts are contaminated or a heavy accumulation of sample is suspected, analyzer parts (stainless steel or plastic) can be scrubbed with soap and water, rinsed well with water, then rinsed well with methanol. Do not clean the multiplier with soap and water. Methanol rinse only. All parts can be sonicated except octopoles/quadrupoles. When sonicating parts, keep the parts separate to minimize surface damage.
HPLC Equipment as sources of contamination

Autosampler
- Could be bad rotor seal.
- Needle seal.

Pump:
- Pulse dampener
- Reservoir filters

Ghost Peaks

<table>
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<tr>
<th>Problem: Ghost peaks</th>
<th>Possible Cause</th>
<th>Solution</th>
</tr>
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<tbody>
<tr>
<td>Peaks from previous injection; column contamination</td>
<td>Use strong solvent flush after each run; improve sample cleanup</td>
<td></td>
</tr>
<tr>
<td>Unknown interferences in samples</td>
<td>Improve sample cleanup</td>
<td></td>
</tr>
<tr>
<td>Upset equilibrium (ion pair)</td>
<td>Use mobile phase as injection solvent</td>
<td></td>
</tr>
<tr>
<td>Oxidation of TFA (peptide mapping)</td>
<td>Prepare fresh daily; use antioxidant</td>
<td></td>
</tr>
<tr>
<td>Contaminated water (reversed-phase)</td>
<td>Check water by varying equilibration time; use HPLC-grade water</td>
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Purge Procedure for Surveyor MS Pump Pulse Dampener

The following procedure should be used to periodically purge the Surveyor MS Pump as part of a comprehensive preventive maintenance program:

1. Prepare a series of beakers containing 50ml each of the following: water, 5% nitric acid, iso-propanol and n-hexane. The water, iso-propanol and n-hexane should all be HPLC-grade.
2. Disconnect the 1/16th inch 316 SS solvent line from the pulse dampener outlet and plug the pulse dampener outlet fitting.
3. Locate the 1/16th inch PEEK line connecting the MS Pump’s Liquid Displacement Assembly to the pulse dampener’s inlet fitting.
4. Disconnect this line at the LDA outlet and place the free end of the tubing into the beaker containing HPLC-grade water.
5. Attach a luer-tipped, priming syringe to the pulse dampener’s purge valve.
6. Open the purge valve and draw 10ml of water through the pulse dampener.
7. Close the purge valve and empty the priming syringe into a suitable waste container.
8. Replace the beaker containing water with the beaker containing 5% nitric acid solution.
9. Reconnect the priming syringe to the purge valve, open the purge valve and draw 10ml of the nitric acid solution through the pulse dampener.
10. Close the purge valve and empty the priming syringe into a suitable waste container.
11. Replace the beaker containing nitric acid with the beaker containing water.
12. Reconnect the priming syringe, open the purge valve and draw 10ml of water through the pulse dampener.
13. Close the purge valve and empty the priming syringe into a suitable waste container.
14. Replace the beaker containing water with the beaker containing iso-propanol.
15. Reconnect the priming syringe, open the purge valve and draw 10ml of iso-propanol through the pulse dampener.
16. Close the purge valve and empty the priming syringe into a suitable waste container.
17. Replace the beaker containing iso-propanol with the beaker containing n-hexane.
18. Reconnect the priming syringe, open the purge valve and draw 10ml of n-hexane through the pulse dampener.
19. Close the purge valve and empty the priming syringe into a suitable waste container.
20. Replace the beaker containing n-hexane with the beaker containing iso-propanol.
21. Reconnect the priming syringe, open the purge valve and draw 10ml of iso-propanol through the pulse dampener.
22. Close the purge valve and empty the priming syringe into a suitable waste container.
23. Reconnect the 1/16th inch PEEK line going to the pulse dampener inlet to the LDA outlet.
24. Reconnect the 1/16th inch 316 SS solvent line from Surveyor Autosampler to the pulse dampener outlet.
25. Empty all beakers into a suitable waste container.