"No Ions" Troubleshooting Strategy ELEMENT ICP-MS

The following is a guideline for identifying the cause of partial or total loss of ion signal. In the process of correctly diagnosing the problem there are important waypoints to be considered:

- a.) Ion species-dependent failure (analyte species versus gas species)
- b.) Resolution-dependent failure
- c.) Detector Mode-dependent failure (Analog versus Counting Mode)
- d.) Partial versus total loss of ion signal

Instrument Basics

Detector basics

Unlike in Ion Counting Mode (that will show NO counts when no ions are arriving at the detector) the Analog Mode will ALWAYS show a continuum of the equivalent of 3,000 to 50,000 ions/s, even when no ions are arriving at the detector. This is a characteristic of the integrator electronics used to record the analog signal. The magnitude of the continuum is a function of integration time. At long integration times (1s) the continuum in Analog Detector Mode is some 3,000-5,000 cps, at shorter integration times (10ms) the continuum is ca. 30,000 cps.

Ultra-trace users often forget that there is an Analog Detector Mode and they fail to test for ion transmission in THIS mode leading to wrong conclusions during trouble-shooting.

Slit basics

The LR slits are argon pressure-sensitive. If there is a failure or deviation of the "Argon middle" pressure then the LR slit will be physically blocked, causing a total loss of ion transmission in Low Resolution. It is advisable to record the "last good argon middle" pressure and use it as a trouble-shooting tool.

Shift of plasma energy basics

There is a difference in plasma energy depending upon the Guard Electrode (GE) status. The energy notch filter setting (Ua/Ub ratio) differs by approx. 0.2 divisions on the Ua/Ub scale. An unrecognized change of the GE status (e.g. loss of contact or GE relay failure) will cause a significant loss of ion transmission since the energy notch filter is now set for the wrong plasma energy causing both transmission and peak shape deterioration.

Extraction lens overload basics

Reducing the sample gas flow significantly (< 0.9 L/min.) or even turning it off causes the sample plume in the plasma to collapse giving way to an abundance of argon ions now entering the interface. As a consequence the voltage on the Extraction lens will either become unstable or even collapse, causing partial or total loss of ion transmission. Such failure of the Extraction lens voltage is indicated by red numerals in the appropriate box in TUNE.

Magnet failure basics

A failure of the magnet will cause total loss of ion transmission. Magnet failures are identifiable by appropriate red indication in the Magnet section of TUNE and DIAGNOSTICS.

Trouble shooting strategy

1. Wrong sample (partial or total loss of analyte signal)

2. Wrong tune file (partial or total loss of ion transmission)

Verify that currently used tune file matches "last good" tune file settings. (For quick tuning recovery it is advisable to always save a "good" tune file on a recovery floppy.

3. Nebulizer failure (partial or total loss of ion transmission)

Remove nebulizer from spray chamber, use DIAGNOSTICS to turn on nebulizer gas flows and visually confirm nebulization (e.g. nebulize against a flat surface or shine flashlight into the aerosol spray).

4. Injector failure (partial or total loss of analyte signal)

Check for blocked or melted nebulizer tip. In this case gas species are preset in the spectrum but no analyte ions.

5. Guard Electode status failure (partial or total loss of ion transmission)

Assuming GE status failure try to recover ion transmission by re-tuning Ua/Ub ratio.

With plasma off verify visually the physical GE contact between plug-in torch spring and opposing contact inside of the torch box.

Again, with plasma off attach an Ohm meter between GE contact spring (on plug-in torch) and instrument ground. Use DIAGNOSTICS/Statusbit INLET to switch "Valve 12" on and off. If GE relay works correctly then this should ground and un-ground the GE contact.

6. Cones blocked (partial or total loss of ion transmission)

Remove cones and inspect orifices with magnifying glass. Look for deterioration of the orifices.

7. Magnet failure (total loss of ion transmission)

Verify that neither magnet error LEDs show red (in TUNE and DIAGNOSTICS/StatusbitsIonOptics).

8. Accelerating voltage failed (total loss of ion transmission)

Verify voltage reading (in INSTRUMENT/Watchparameters) and compare reading with "last good" voltage.

9. Extraction lens failure (partial or total loss of ion signal)

While scanning (in TUNE) verify that numerals for Extraction lens voltage show black.

10. Ion optical lens failures (partial or total loss of ion transmission)

While scanning inspect DIAGNOSTICS/Statebits/ Ionoptics & ESA for any red LED indicators. Contact ThermoFinnigan service for interpretation of observation.

11. Gate valve failure (total loss of ion transmission)

Verify in DIAGNOSTICS/Statebit/PowerDistribution that LED for "Skimmer valve Open" shows green.

12. Mass calibration failure (apparent total loss of ion transmission)

Perform a wide-range magnet scan (in BOTH Mode) using INSTRUMENT/Manual in either resolution to identify possible significant shift in peak positions. If mass calibration seems corrupted then reload "last good" mass calibration from recovery floppy for fast recovery.

13. LR Slit failure (partial or total loss of ion transmission ONLY in LR resolution)

Verify current Argon Middle pressure matches "last good value" for "Argon Middle" pressure. Verify that transmission in MR and HR appears to be OK. If failure of Argon Middle pressure is diagnosed then an expert user can re-determine the correct pressure by doing a LR-slit-pressure-scan. Otherwise assistance of ThermoFinnigan service is required.

14. HR slit failure (partial loss of ion transmission ONLY in HR resolution)

If (under optimal tuning conditions) the transmission in HR falls below 1% relative to LR then the HR entrance slit has deteriorated and needs to be replaced.

15. Skip range blockage (apparent total loss of ion transmission)

Verify in EXECUTIVE/Skipmasses that observed ion is not blocked by skip mass range. Attempting to acquire an ion signal that is blocked by the skip mass range will result in no signal to be present in THAT mass range.

16. Counting detector safety tripped (total loss of Counting Detector Signal)

Verify presence of gas species ArO in Analog Detector Mode in MR and HR (MR= approx. 10 Mio cps and HR= approx. 1 Mio. cps). If analog mode ion signals are OK then either Counting Detector safety has tripped due to a large signal or due to failure of the Counting electronics.

17. Inappropriate SEM voltage (partial loss of ion transmission)

Verify that SEM voltage in the currently used tune file matches the "last good" setting of SEM voltage. If some counting signal can be observed then perform "Ion Counting Plateau Scan" to determine correct SEM voltage setting for the SEM voltage. If new plateau voltage exceeds 2600 V then replace detector.

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