GERMAN FEDERAL INSTITUTE FOR RISK ASSESSMENT

BfR
Bundesinstitut für Risikobewertung

# IRMS – Part 3
# System Tests and Trouble Shooting

Dr. Melanie Gimpel

# Overview

- System tests - daily checks
- System tests -  after maintenance work and repair
- Source tuning
- Troubleshooting

# System Tests I – daily checks

Some system tests depend on the method, the measured isotope and the system, but most of the tests are required for all IRMS-applications:

- ✓ Check gas supplies
- ✓ Check vacuum
- ✓ Check carrier gas flow
- ✓ Check temperatures (Combustion, Reduction, GC-oven)
- ✓ Check background
- ✓ Check reference gas, perform a stability test

- ✓ For some tests it is recommended to document the test results regularly.

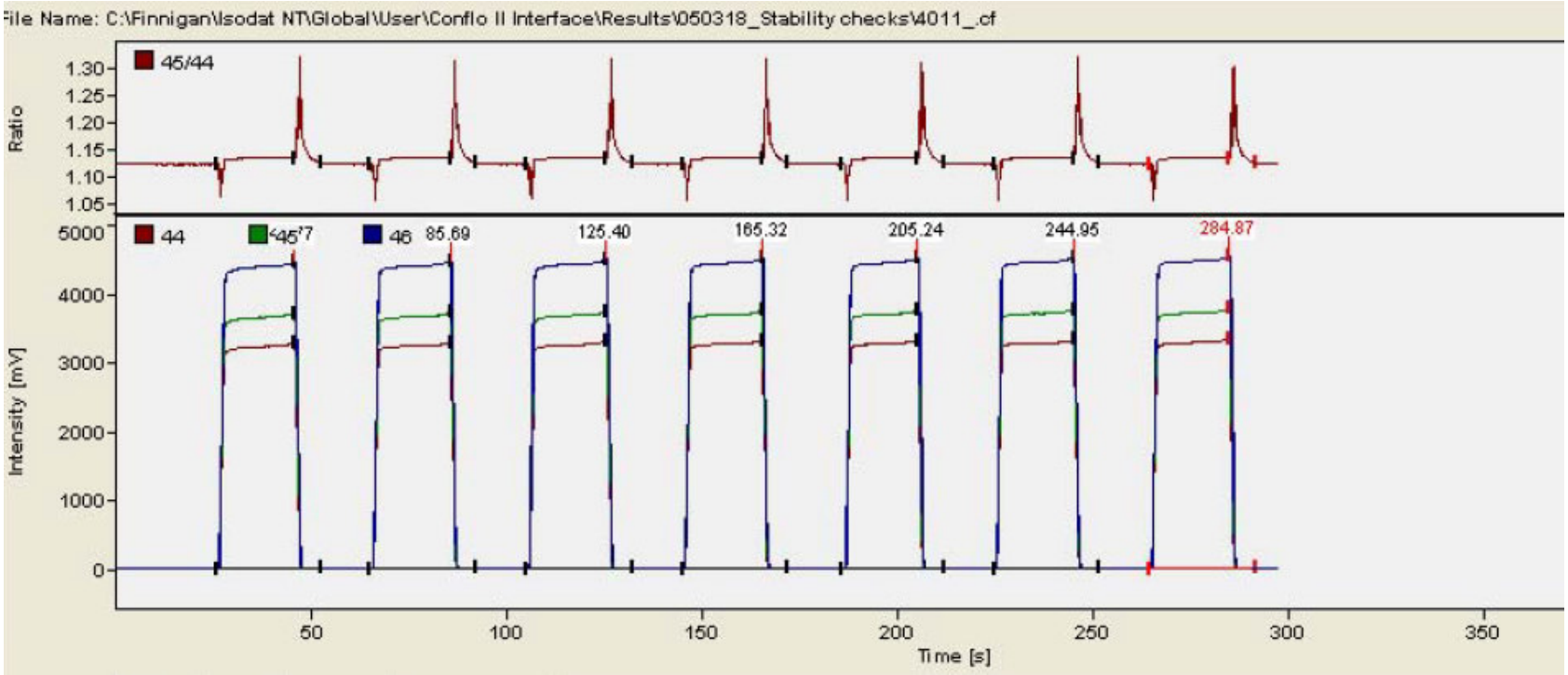
# Background measurement

- Background measurement is a good tool for identifying problems
- Background values vary from lab to lab
- Instrument manufactures often specify acceptable levels

| *m/z* | Mol species | Problem and possible cause |
|---|---|---|
| **2** | $He^{2+}$ | High background in D/H measurements, electron energy can be ajusted to produce acceptable values |
| **18** | $H_2O^+$ | Produces protonated species which may interfere with ions containing heavy isotopes |
| **28** | $N_2^+$ | Guide to ingress of atmospheric gases (also CO by thermolysis) → indicates a leak |
| **40** | Ar+ | Best guide to the ingress of atmospheric gases → indicates a leak |
| **44** | $CO_2^+$ | Contamination of C/N analysers or oxygen ingress into H/O analysers |

# System Tests – reference gas stability

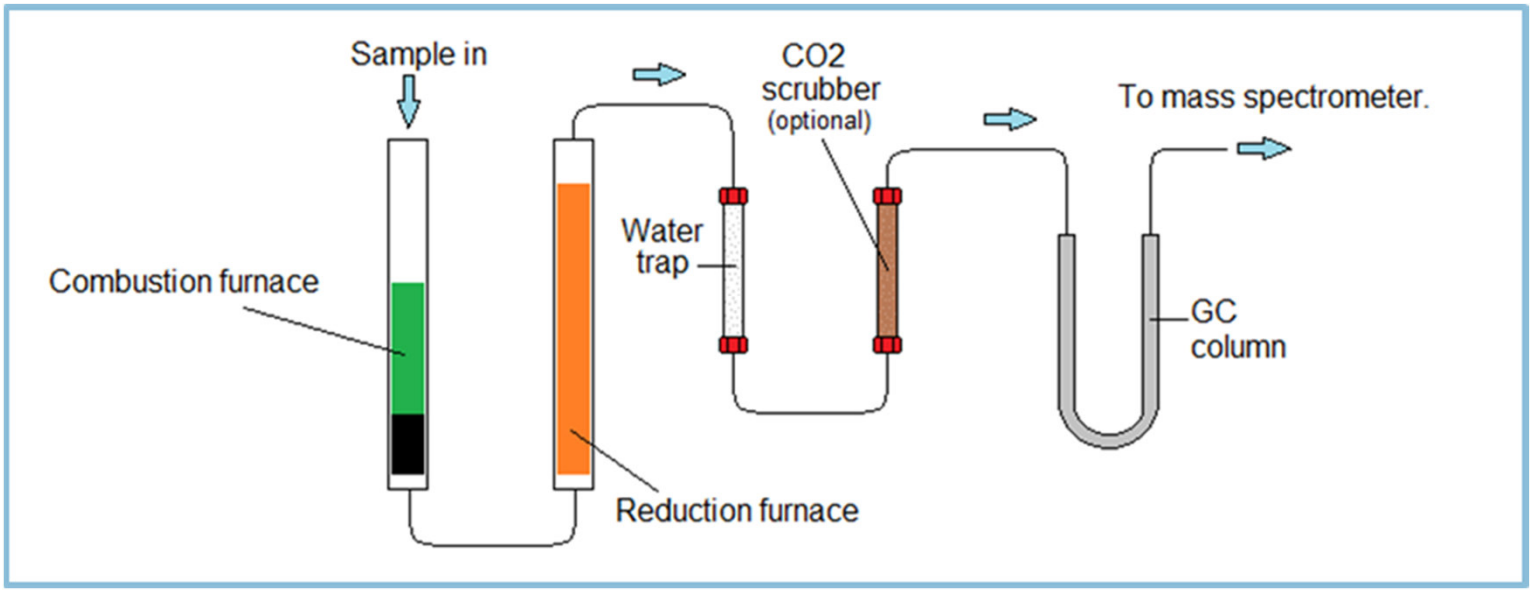
- ✓ Check reference gas
  - Monitor the stability of the measurement of the isotopic composition, on a daily basis
  - "**Zero enrichment**" or **"on-off" test**: introducing ten pulses of the working gas into the instrument, record standard deviation of the $\delta$-values (SD for $CO_2$, $N_2$ and CO should be less than 0.1)




J.F. Carter and V. J. Barwick (Eds), Good practice guide for isotope ratio mass spectrometry, FIRMS (2011)

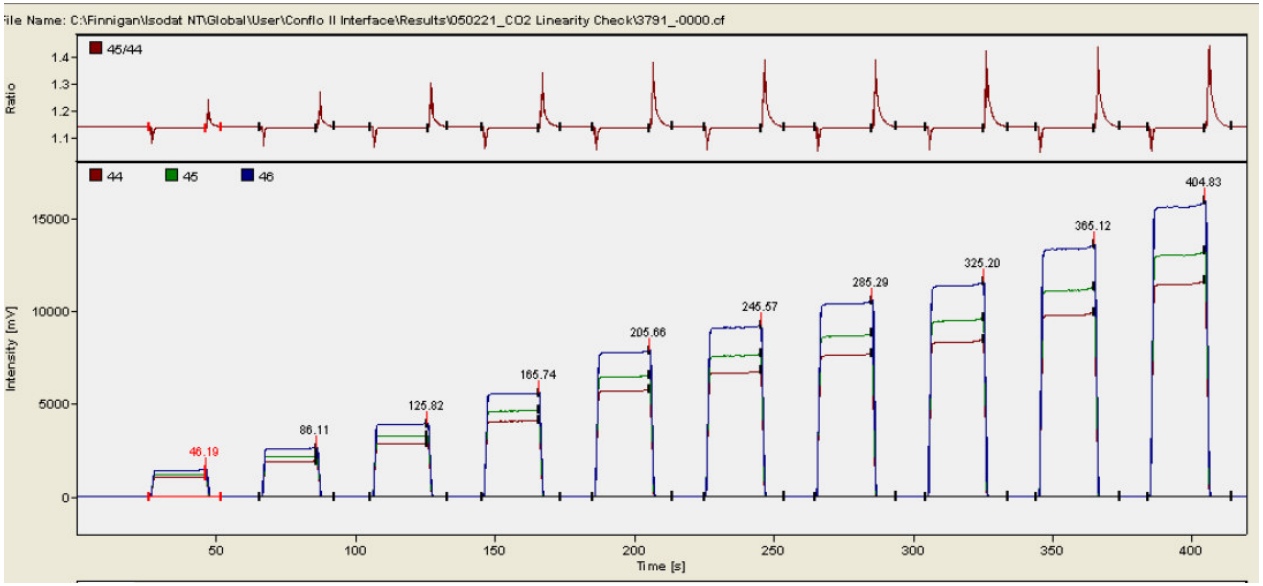
# System check II and maintenance

- ✓ **Source / Pumps**
  - Source Tuning
  - Replacing of the filament
  - Cleaning of the source
  - Oil control and oil change
- ✓ **Linearity test**
- ✓ **EA-Maintenance**
  - Replacing of the oxidation reactor reagents
  - Replacing of the reduction reactor reagents
  - Replacing of the trap reagents and Nafion
  - Baking the column
  - Cleaning or replacing of the ash collector



# System Tests - linearity

- ✓ Check linearity
  - Should be checked periodically, particularly after any modification of source parameters
  - Similar to the stability test, except that the intensity of the working gas is increased during the sequence
  - Intensity of working gas pulses must encompass the intensities of the samples to be determined (i.e. samples 5000-15000 mV, linearity measurement 4000-16000 mV)
  - Linearity of $CO_2$, $N_2$ and CO must be less than 0.1 ‰ per volt

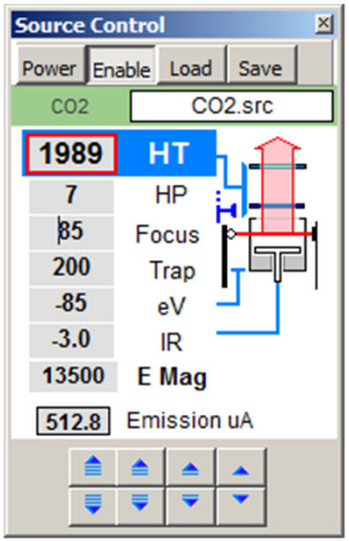



J.F. Carter and V. J. Barwick (Eds), Good practice guide for isotope ratio mass spectrometry, FIRMS (2011)

# Source Tuning

- A tuning is necessary:
  - after changing the source
  - after filament change
  - after cleaning the source
  - when signal decreases caused by a slightly dirty source
- An IRMS can be tuned either for **sensitivity** or for **linearity**
- Tuning is performed with working gas ($CO_2$)
- For **sensitivity** all ion source parameters to maximum signal intensity.
- For **linearity** some parameters to critical values. All other parameters to maximize the signal.

# Tuning – Source Control Sercon




**Callisto ion source control window. [20-22 User's Manual V5.1]**

The following source parameters can be adjusted:

**Acceleration Voltage** (HT) - Sets the voltage of the source block (Vs).

**Half Plates** (HP) - Sets a differential voltage across the two half plates.

**Focus** - Sets the voltage of the half plates with respect to ground.

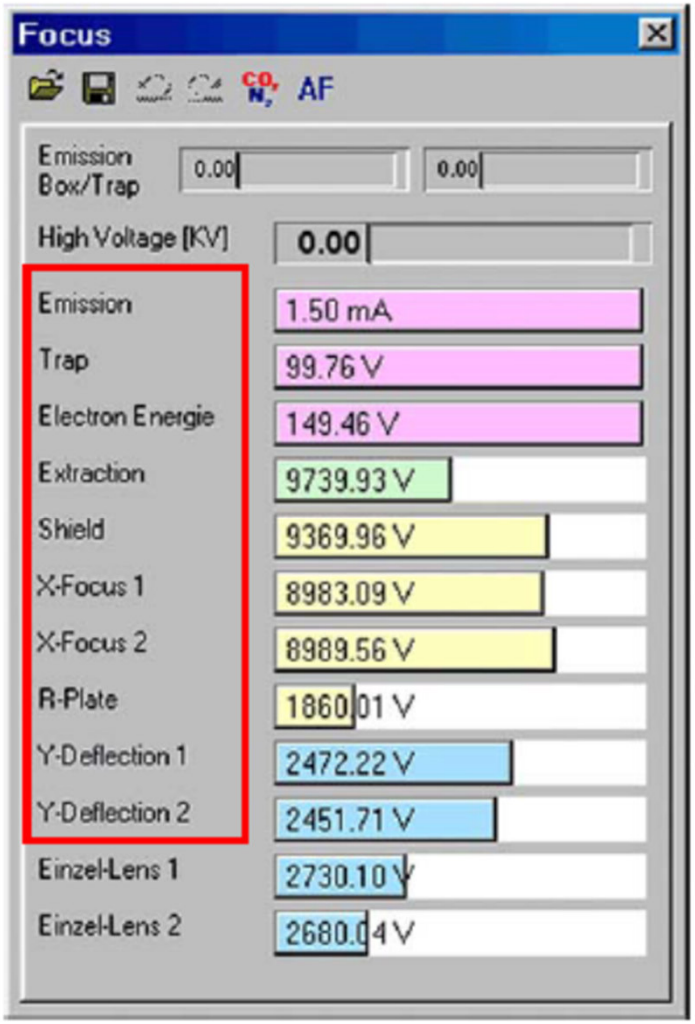
**Trap Current**- Controls the flux of electrons through the source chamber.

**Electron Energy** (eV) - Sets the voltage between the filament and the source block.

**Ion Repeller** (IR) - Sets the voltage difference between the ion repeller and source block.

The Emission box is a readback and cannot be adjusted.

# Source Tuning – Thermo




**Source: Thermo Finnigan User Manual MAT 253, Issue 04/2002**

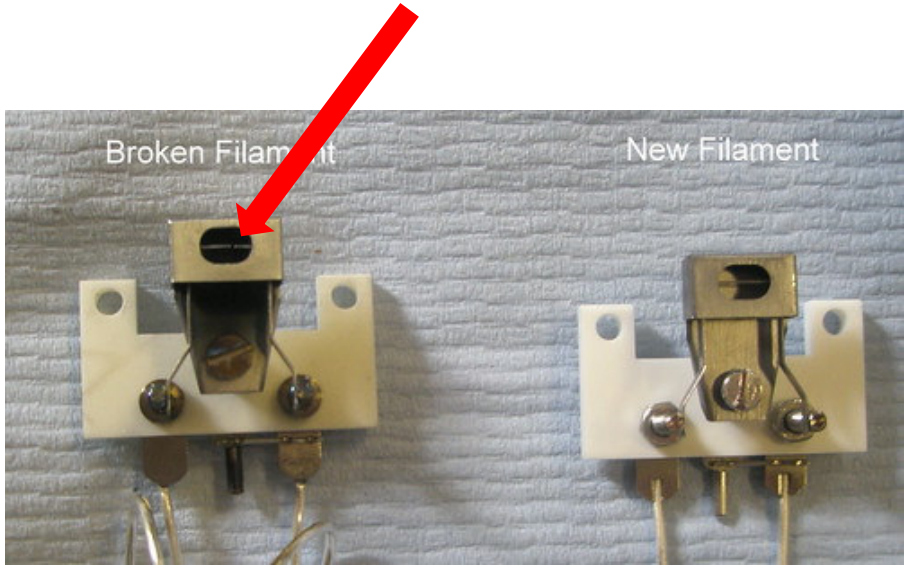
1. ***Basic Adjustment of Parameters***

   Before the first focusing run, the following parameters should be preset:

- Set ***Trap*** to 40 V.
- Set ***Electron Energy*** to the maximum value.
- Set ***Emission*** to 1 mA (i.e. to 50 % below the maximum value of 1.5 mA).
- Set ***Extraction*** to a middle position (e.g. to 2600 V).

# Trouble Shooting – ion source I

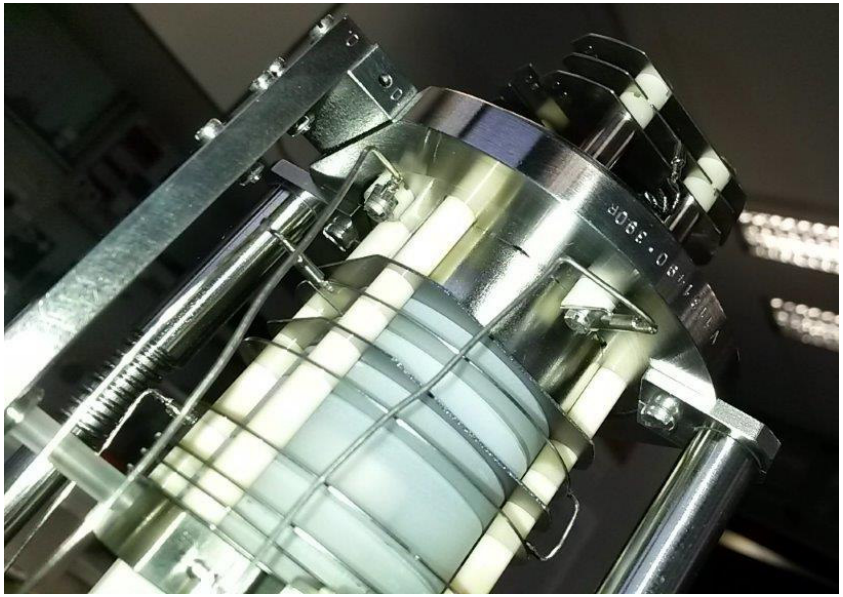
- No emission
- Box and Trap values are fluctuating
- Poor linerarity
- Poor sensitivity

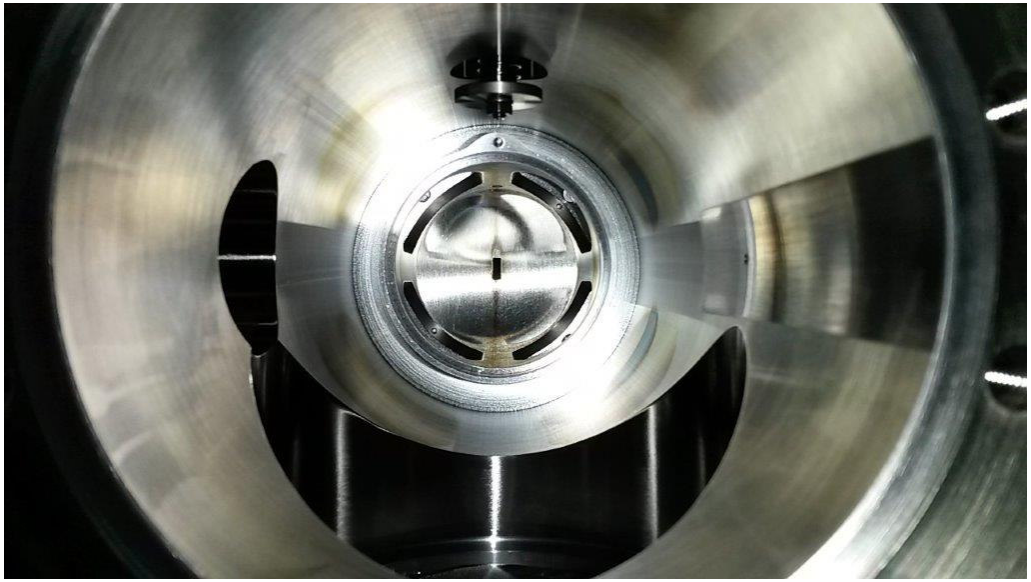



- Ion source filament failed
- Filament has weakened
- Misaligned filament or source
- Poor tuning parameters

- Check filament continuity, check connections
- Replace filament
- Check all connectors for shortings
- Check ion source tuning

# Trouble Shooting – Ion Source II





# Trouble Shooting – sample load

- No sample peak
- To large sample peak
- Unexpected δ values

- Problems with the autosampler

- Check if samples were loaded correctly
- Look for trapped capsules
- Crimp the capsules not to flat

# Trouble Shooting – peak shapes

- Peak tailing
- Peak broadening
- Poor peak separation

- Reactors or traps may have dead volumes
- Carrier gas to slow
- GC column aged or contaminated

- Check packing of reactors
- Check carrier gas flows
- Bake out or replace GC column

# Trouble Shooting - Heater

- Furnace heater does not operate

- Insufficient He flow
- Thermocouple failed
- Furnace heater failed

- Check helium flow
- Replace thermocouple
- Replace furnace heater

# Trouble Shooting - water

- Baseline drift after $CO_2$ Peak

- Water in the system

- Check m/z 18 Background
- Replace packing of water trap

# Trouble Shooting – Backgrounds

- High Backgrounds for $N_2$, $O_2$, Ar, $H_2O$

- Leaks
- GC column contaminated
- Traps chemicals exhausted
- Incorrect gas purity
- Heaters in ion source or inlet valve failed

- Test autosampler for leaks, replace seals
- Bake out or replace GC column
- Ensure correct gas supply
- Replace trap chemicals

# Trouble Shooting - summary

- Check the background
- Search for leaks
- Source
  - Tuning: Sensitivity ↔ Linearity
  - Cleaning
  - Filament Exchange
- Replacement of water traps
- Baking out GC columns
- Replacement of Ox/Red-reactors

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# Thank you for your attention

Melanie Gimpel

German Federal Institute for Risk Assessment
Max-Dohrn-Str. 8-10 ● 10589 Berlin, GERMANY
Phone +49 30 - 184 12 - 0 ● Fax +49 30 - 184 12 - 47 41
bfr@bfr.bund.de ● www.bfr.bund.de/en