ICP-MS

Technique and applications

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  - (Determination of metals in air filters)

ICP-MS technique

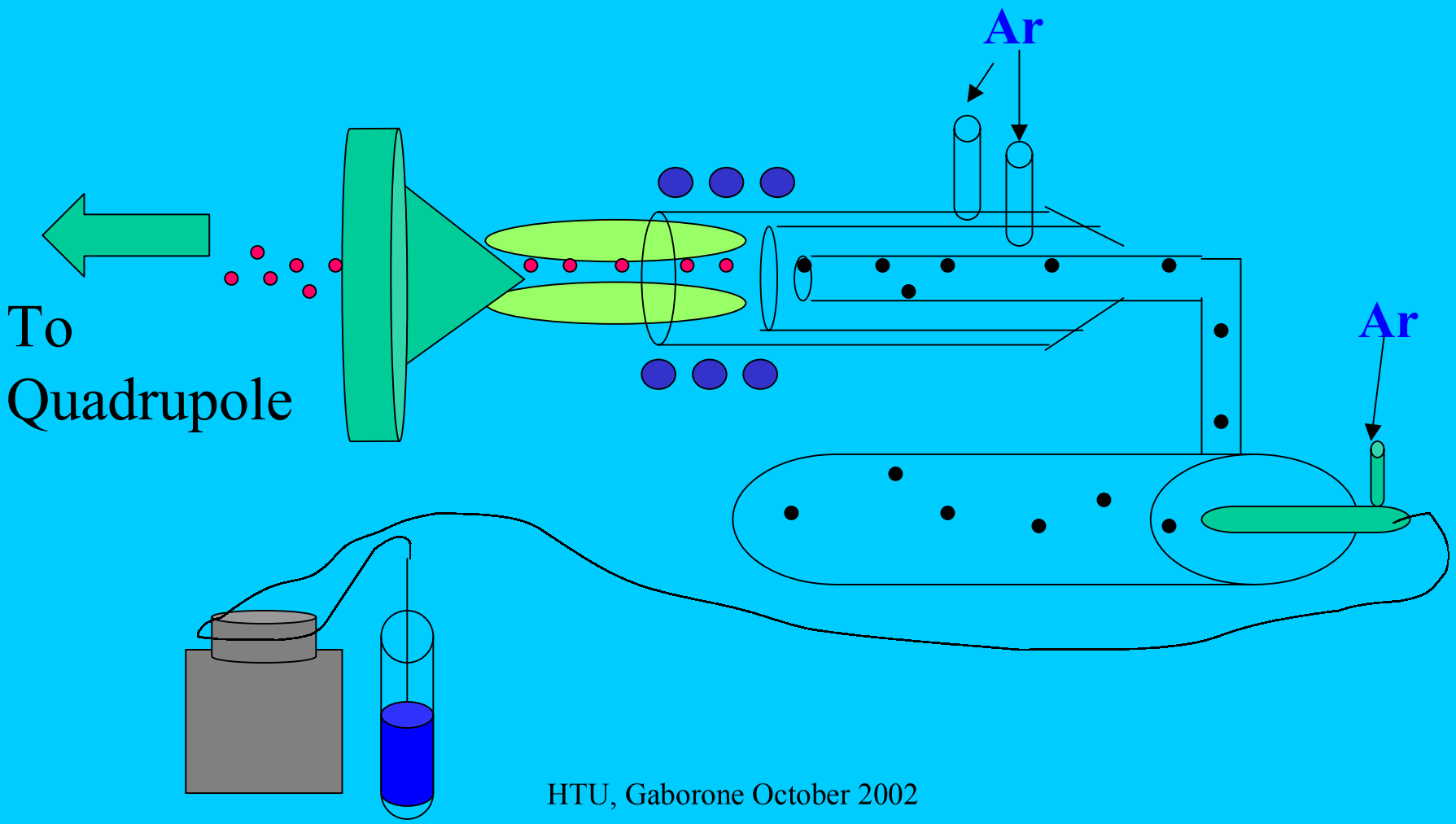
Advantages

- Low detection limits
- Long dynamic range
- Multielement technique

Limitations

- Interferences
- Low sample introduction efficiency
- Expensive

Interface



Use good laboratory practise to avoid contamination of samples

- Keep laboratory as clean as possible
- Acid wash all equipment in 2% HNO_3 v/v
- Use gloves
- Avoid contact between sample and metal items
- Check water quality regularly
- Cover sample tubes when not analysing
- Participate in laboratory intercomparisons

Sequence of analysis

Bottle no.	Sample ID	Sample definition
1-3	Blank	Calib.blank
4.	Standard1	Calib. standard
5.	Standard2	Calib. standard
6.	Standard3	Calib. standard
7.	Blank	Unknown
8.	QA Standard1	Unknown
9.	QA Standard2	Unknown
10.	Blank	Unknown
11-38	Samples	Unknown
39	Blank	Unknown
40	QA sample	Unknown

Analysing procedure

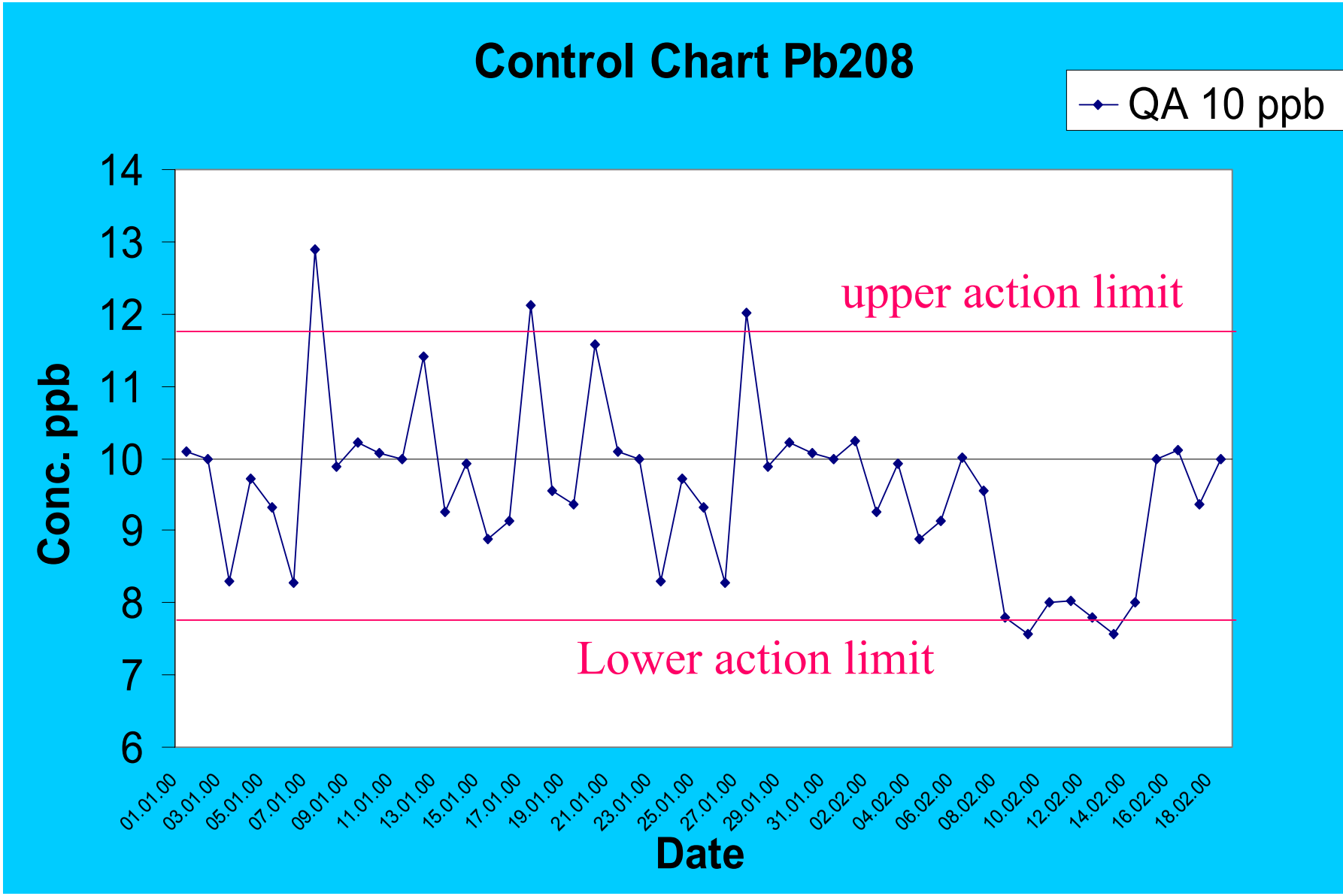
Inspect blank signal
Inspect signal from internal standard (i.s.)
Inspect conc. in QA-sample

Result	Cause	Action
High blank	Contamination	Make new blank
Low i.s. signal	Low intensity	Retune Recalibrate
Wrong QA-conc.	Calibration error	Retune Recalibrate
Acceptable results	⇒	Run samples

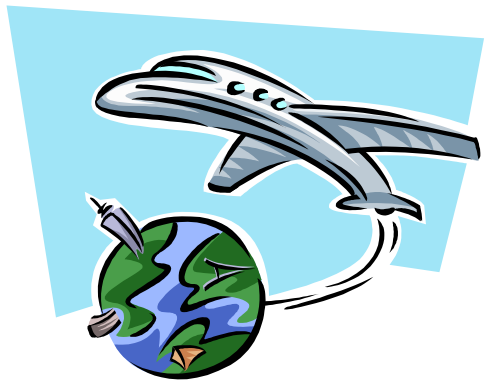
Result handling procedure

- Inspect and edit calibration curve
- Inspect QA-sample concentration
- Enter QA-values in control chart
- Identify samples for dilution and re-run
- Inspect sample values (raw-data)
- Identify possible interferences

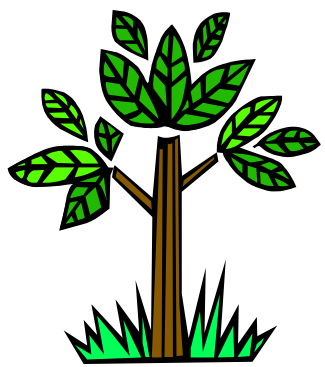
Result	Action
Not acceptable	Re-run
Acceptable	Calculate and report


Control Chart Pb208
QA 10 ppb
upper action limit
Lower action limit
Conc. ppb
Date
14
13
12
11
10
9
8
7
6
01.01.00
03.01.00
05.01.00
07.01.00
09.01.00
11.01.00
13.01.00
15.01.00
17.01.00
19.01.00
21.01.00
23.01.00
25.01.00
27.01.00
29.01.00
31.01.00
02.02.00
04.02.00
06.02.00
08.02.00
10.02.00
12.02.00
14.02.00
16.02.00
18.02.00

Matrixes analysed at NILU



- Air filters
- Water (freshwater; precipitation)
- Fish oil
- Plant material
- Road dust
- Absorption solutions
- Paper
- Bark
- Mine water
- Teeth
- Soil
- Ash
- Sediments
- Waste water



Metal analysis on air-filter

Sample types

Sample type	Purpose
Blank filters	Contribution from clean filter and calculation of LOD
Field blank	Contribution from handling
Blank MW .	Contamination from digestion
Calib.blank	Calibration
Standards	Calibration
Unknown samples	To be analysed

Procedure for mass determination of air filters (for metal analysis)

1. Air filters are conditioned for 24 hrs at 50% rel. humidity.

2. Gloves and plastic- or teflon layered tweezers are used for handling of filters.

3. After weighing, filters are placed in acid washed filterholders or petrislides.

Microwave digestion for air filters (47 mm)

1. Add 1.0 ml concentrated HNO_3 (sp) to MW-container and 2 ml concentrated H_2O_2 (sp)

2. Heat in MW oven for 25 min at 220 ^oC

3. Cool to ambient temp. and add 7 ml 1% v/v HNO_3 containing 14.3 ppb internal standard

4. Make sure that filter is covered with fluid before digestion in MW-oven

Analysing air-filters using ICP-MS

- Run samples using 1% v/v HNO_3 standards
- Report results as µg/m3

- 1-3 Calib.blank
- 4-6 Standards
- 7 Blank
- 8-9 QA-standards
- 9 Blank
- 10-11 Filter blank
- 12-13 MW-blank
- 14 Field blank
- 15-39 Filter samples

Thank you for your attention