Workplace Air - Sampling and determination of metals and metalloids in airborne particulate in precious metal labs

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Agosi

Part of Umicore

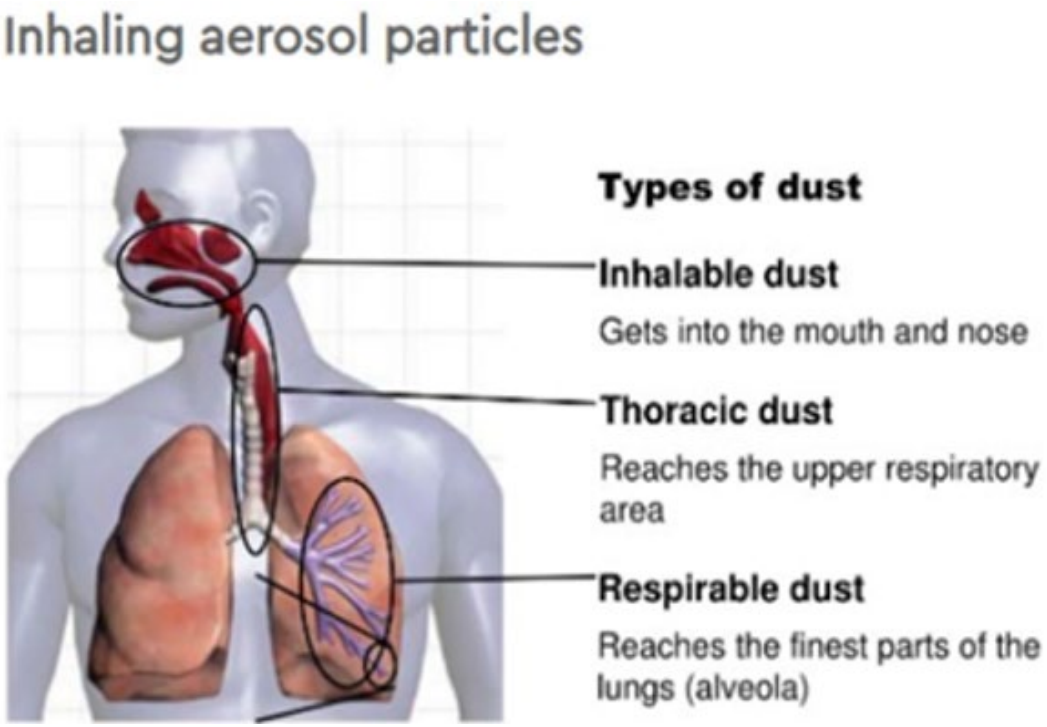
- The business model of Agosi and its subsidiaries is based on precious metal technology and precious metal expertise in the closed-loop business.
- Indispensable elements are precious metal refining, precious metal products and precious metal services.
- Our home market is Germany and Europe, supplemented by a multi-regional presence in markets relevant to us.
- Umicore Galvanotechnik GmbH complements this model as a solution provider for surface finishing in the international markets.

This presentation shows

- Description of air sampling
- description and critical elements
- The equipment used in Agosi Lab
- Sampling for lead in the fire assay lab
- Sampling for chlorinated platinum salts in the lab
- Analyze of the filters with ICP-OES
- Results
- Comparision of sampling heads

Description of air sampling

- In order to safeguard workers, national and EU regulations have established specific threshold values for pollutants and airborne dust, referred to as occupational exposure limits.
- There is a differentiation between respirable dust (fine dust) and inhalable dust.
- Also aerosols and acids can be analyzed by air sampling



Why air sampling ?

- Protecting employees and improving health
- Legal requirements and company standards
- Lower social costs associated with healthcare
- Optimization of employee potential

Measurement in each department should be repeated minimum 1 time / year or after process changes

Base for sampling and measurements ISO – regulations

The procedure of sampling, sample preparation and determination with ICP-OES or ICP-MS is described in the following ISO regulations

ISO 15202 - 1	Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 1: Sampling
ISO 15202 - 2	Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 2: Sample preparation
ISO 15202 - 3	Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 3: Analysis
ISO 30011	Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma mass spectrometry

Example of typical elements for Agosi

Element	regulatory limit value in Germany	dust type	internal limit
As	0,00083 mg/m³	fine dust	
Ni	0,006 mg/m³	fine dust	
In	0,0001 mg/m³	fine dust	
Cd	0,002 mg/m³	total dust	
Co	0,04 mg/m³	total dust	
Pb	0,15 mg/m³ but reduced to 0,03 in 2024	total dust	
Li	0,1 mg/m³	total dust	
Mn	0,02 mg/m³	total dust	
Pt salts	0,002 mg/m³		0,0001 mg/m³

Examples for health risks – especially in our lab

lead is a critical metal with a clear toxic profile

- Reprotoxic
- Neurological effects

Chlorinated platinum salts are considered as critical metals with a clear toxicological profile

- "Platinum Salt Sensitivity" (PSS): Respiratory sensitization Asthma
- Dermal reactions (contact dermatitis)

Principle

Metal and metalloid particles in the air are trapped by passing a measured volume of air through a collecting substrate (e.g. filter or foam). This substrate is mounted in a collector that targets a specific particle size fraction.

The sample is then treated according to the methods described in acids dissolve the metals and metalloids. The resulting solution is then analysed using inductively coupled plasma

$$Element\ concentration\ mg/m^3 = \frac{mass\ elment\ in\ mg}{air\ volume\ in\ m^3}$$

$$\text{Amount air in m}^3 = \frac{\text{pump flow } \frac{\text{L}}{\text{min}} \times \text{sampling time in min}}{1000}$$

What kind of sampling equipment do we use?

Sampling Pump conforme to ISO 15202-1 for personal or stationary sampling

0,0 - 5,0 L/min

Cyclon or IOM sampling heads

VC-25 for respirable or inhalable dust measurement

IOM-Sampler

- Both dust fractions are separated using a foam filter, while respireable dust is isolated with an MCE filter of 0.8 µm.
- Simultaneous measurement of respirable and inhalable dust.
- Flow rate at 2.0 L/min.

Foams for inhalable dust

MCE-Filter for respirable dust

Cyclon Sampler

- Separation of respirable-dust on an MCE filter 0,8 µm . Separation of the respirable and inhalable fractions by a cyclone
- Only measurement of respirable-dust
- Flow Rate 2,2 L/min

MCE-Filter for respirable dust

VC-25 - sampler

- High flow measurement 22,5 m³/h.
- Only measurement of respirable-dust or inhalable-dust. Not simultaneously

Configuration for respirable dust

Configuration for inhalable dust

Sampling protocol

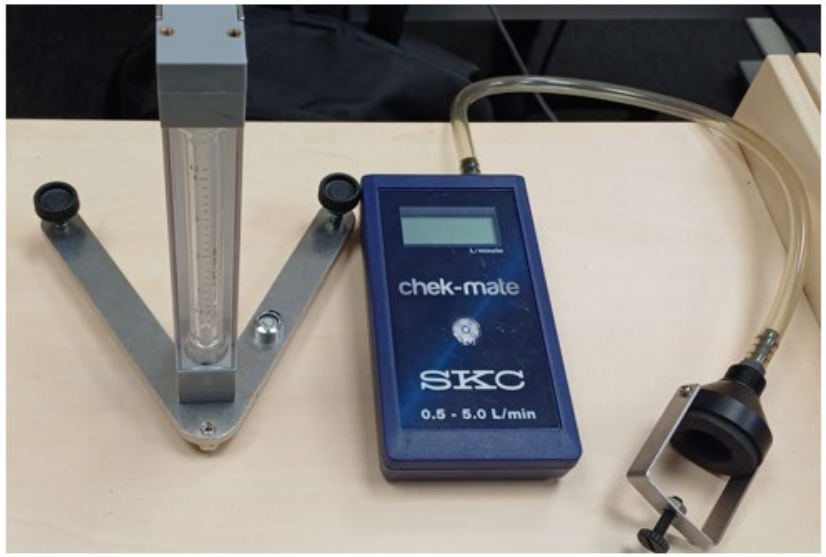
Sampling protocoll

- Department
- Start and end time for calculation of the air volume
- Pump flow rate
- Does the worker wear new or old clothes
- Used samplers (IOM, Cyclone,VC-25)

important points

- Date
- Work task of the worker
- Material processed on that day
- Available protective equipment
- Elements of interesst

Calibration of the pumps



Rotameter for classical calibration

Calibration is done with a mass flow meter SKC chek-mate

After every sampling the calibration must be checked and should be into 5% range of the calibration before sampling

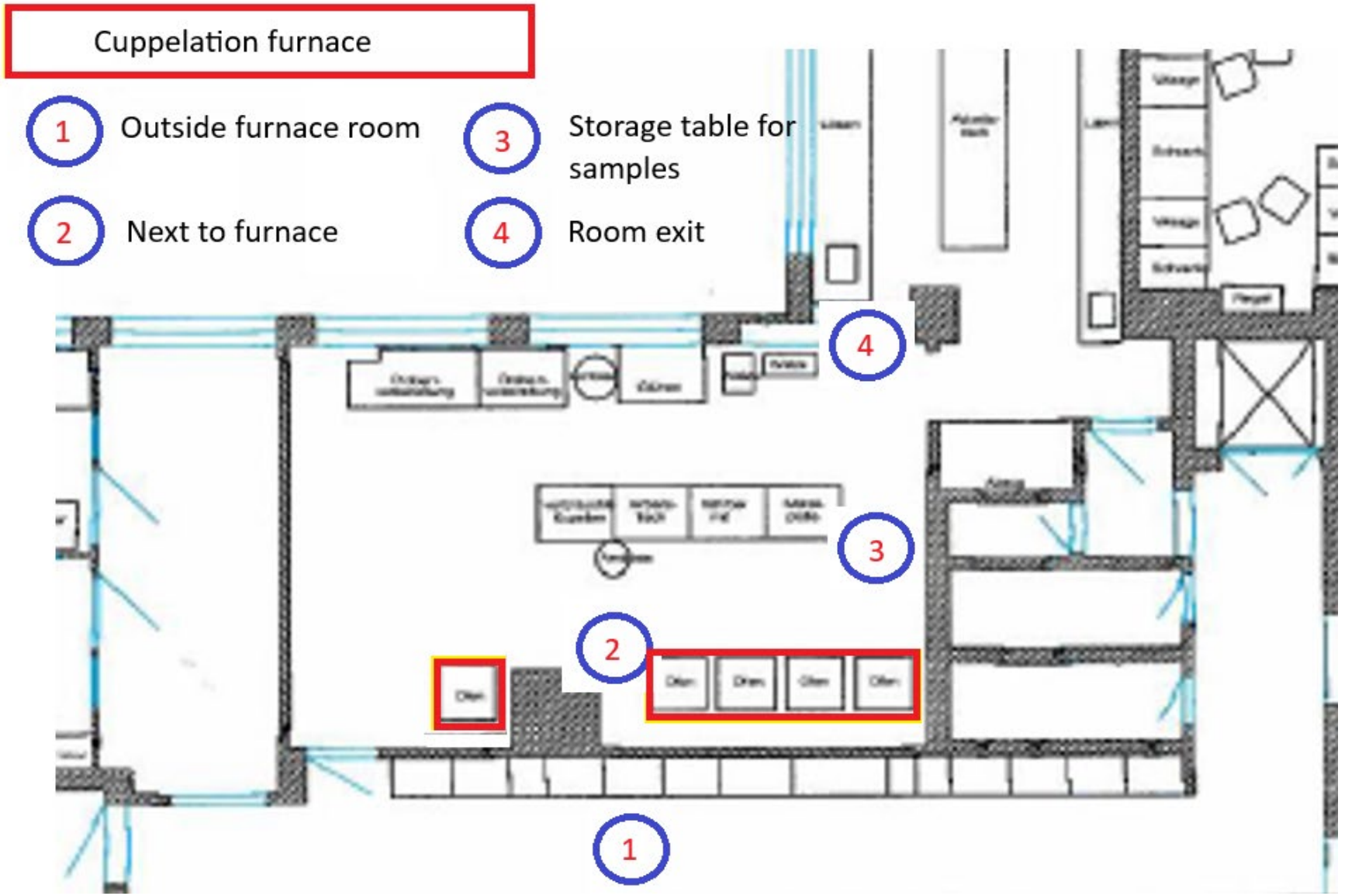
example for sampling Lead in Fire Assay lab

Lead emissions can occur during the cuppelation process

Sampling time : over the complete shift

The IOM sampler with foam inlet is used in Agosi

Measuring stations for stationary measurements



example for sampling chlorinated platinum salts in the aqua regia lab

The dissolution of platinum in aqua regia may lead to the release of chlorinated platinum salts.

According to the MAK commission, the permissible limit is set at 0.002 mg/m³ in 8-hour work period. Internal threshold is 0.0001 mg/m³.

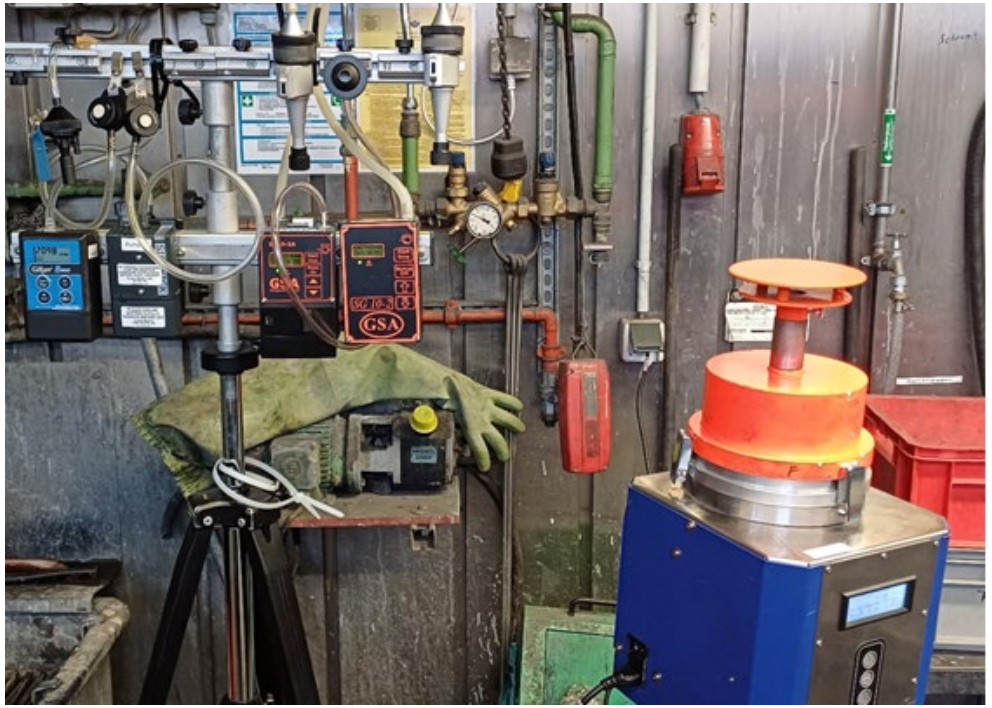
Sampling should occur throughout the entire shift.

An IOM sampler without a foam inlet is used at Agosi.

Comparative measurements with an official measuring institute

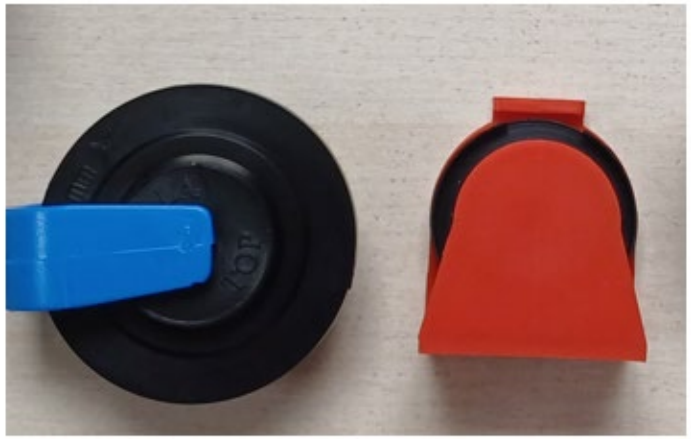
For validation, we measured together with an official insitut at the same time

Good agreement of the results according to information from the institute. The exact evaluation is still pending.

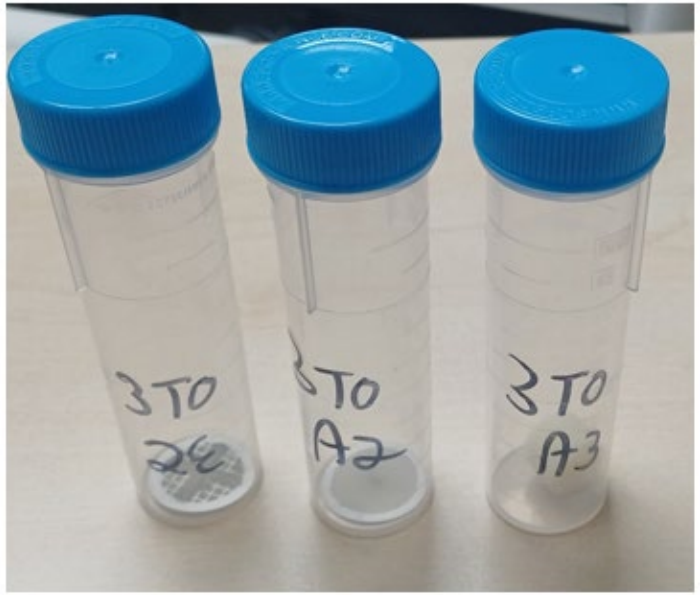


Sample preparation according to ISO 15202-2

the filters must be secured and sealed immediately after sampling

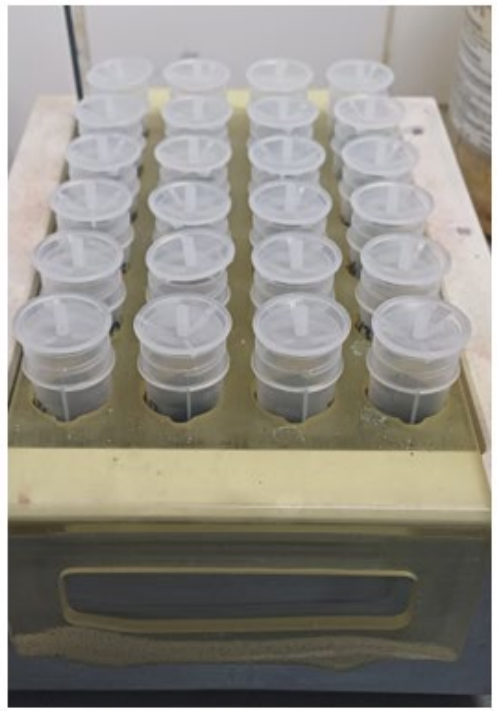


In the laboratory, the filters are transferred into vials



Element determination according to 15202 – 2 - 3

The process for dissolving the filters and measuring them using ICP-OES is detailed in ISO 15202 – 2 and ISO 15202 - 3.



Dissolving depends on the metal of interest. (Pb = aqua regia, Pt salt = 0,07 mol/L HCl)

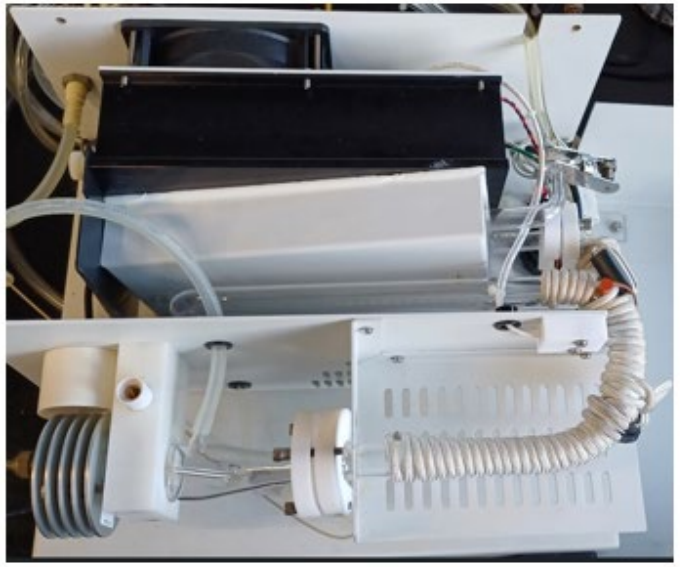
Measuring the samples with ICP-OES and ultrasonic nebulizer

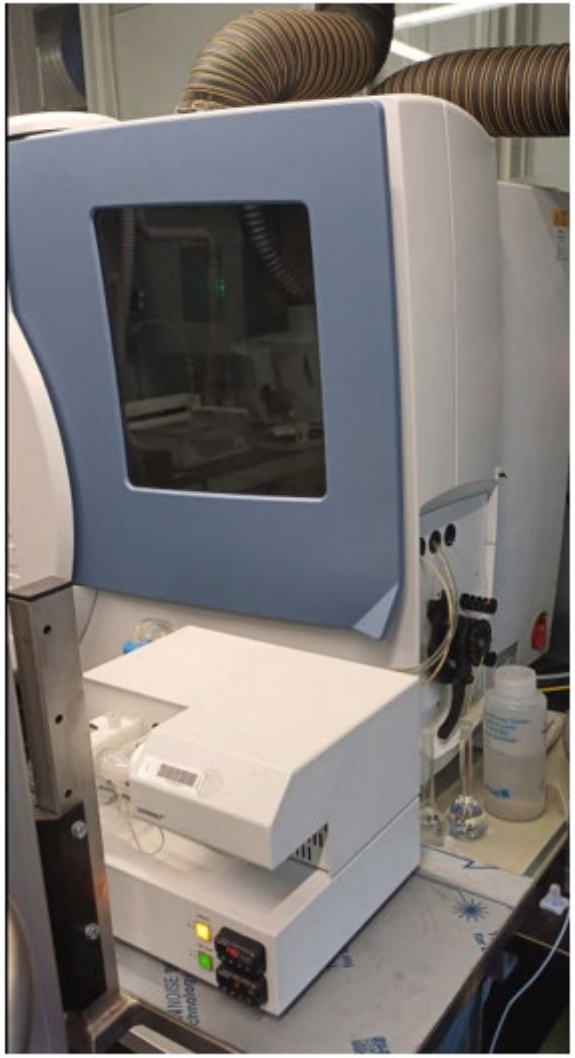
ISO 15202-3 Ultrasonic nebulizer

Certain elements like Pt or In require extremely low detection limits (less than 0.005 mg/L).

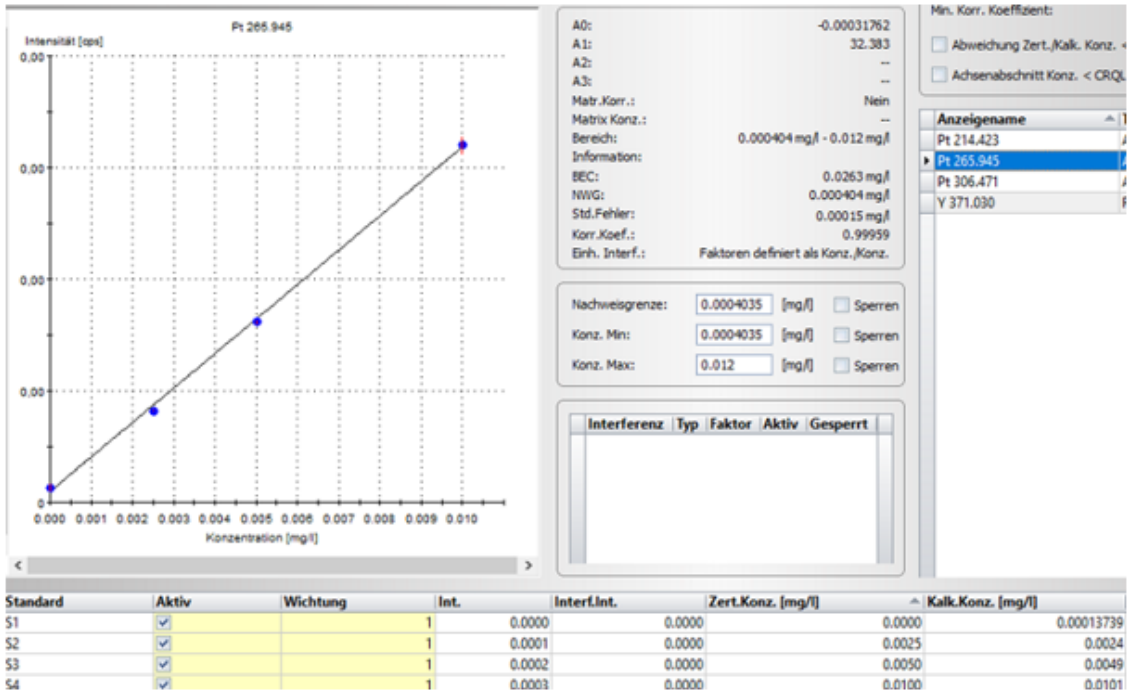
USN enhances the detection limit by a factor of 10 to 20.

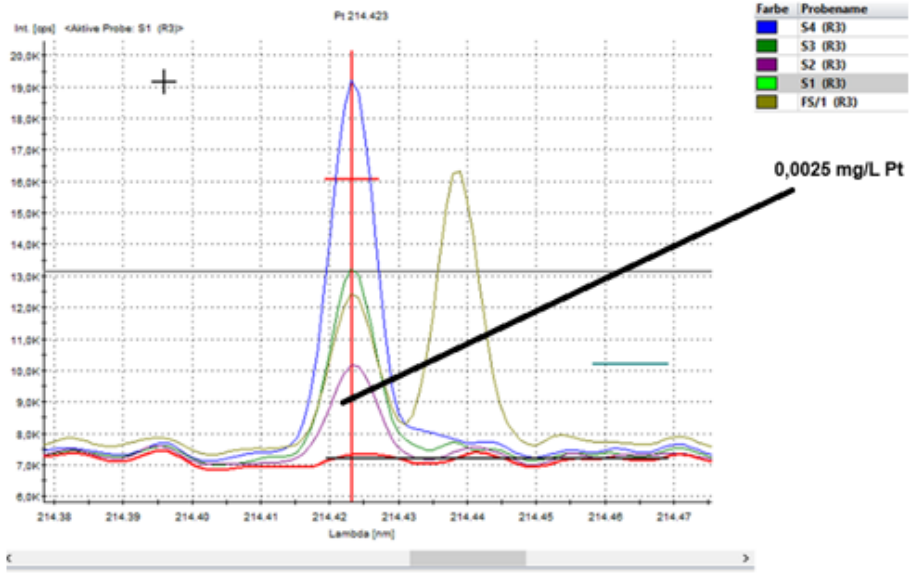
Used with an axial ICP-OES System.





Ultrasonic Nebulizer_Platinum

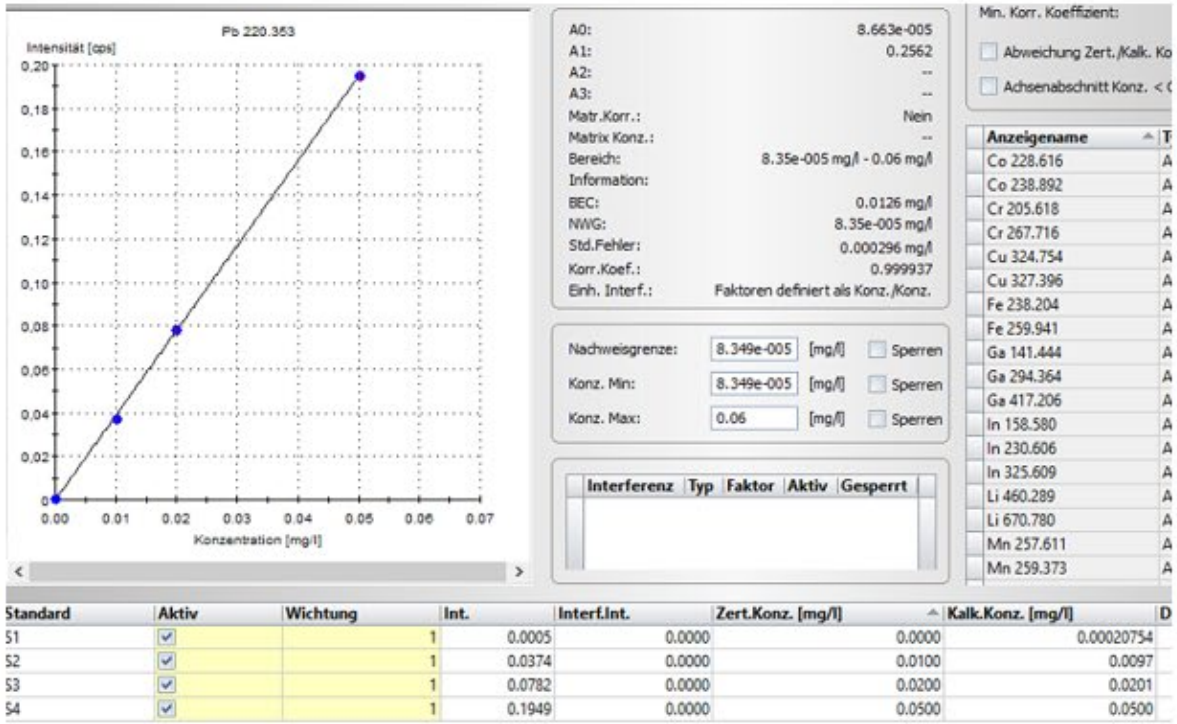




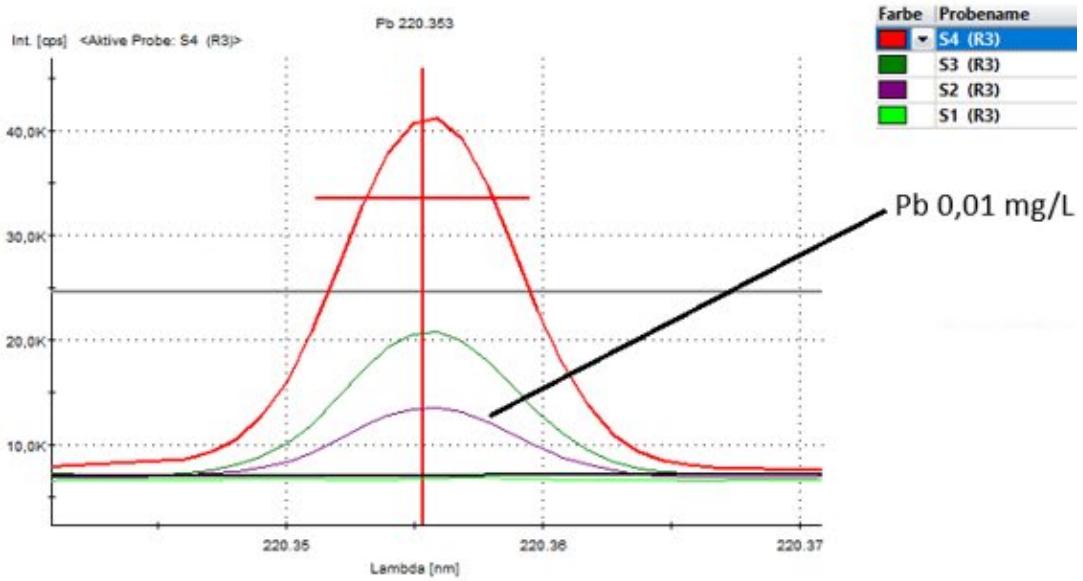
These images illustrate a platinum calibration using standards of 0.000 mg/L, 0.0025 mg/L, 0.005 mg/L, and 0.01 mg/L. They also display the peaks and provide a comparison between ICP-OES with USN and ICP-MS.

Probe	ICP-OES with USN	ICP-MS
	mg/L	mg/L
Sample 1	0,0036	0,0040
Sample 2	0,0000	0,0000
Sample 3	0,0325	0,0354
Sample 4	0,0019	0,0024
Sample 5	0,0107	0,0095
Sample 6	0,0012	0,0010
Sample 7	0,0120	0,0120
Sample 8	0,0003	0,0003
blanc	0,0000	0,0000

Ultrasonic Nebulizer_Lead



Standard	Aktiv	Wichtung	Int.	Interf.Int.	Zert.Konz. [mg/l]	Kalk.Konz. [mg/l]
S1	☑	1	0.0005	0.0000	0.0000	0.00020754
S2	☑	1	0.0374	0.0000	0.0100	0.0097
S3	☑	1	0.0782	0.0000	0.0200	0.0201
S4	☑	1	0.1949	0.0000	0.0500	0.0500



These images illustrate a lead calibration using standards of 0.000 mg/L, 0.01 mg/L, 0.02 mg/L, and 0.05 mg/L. They also display the peaks on Lead 220 nm

Ultrasonic Nebulizer_Indium

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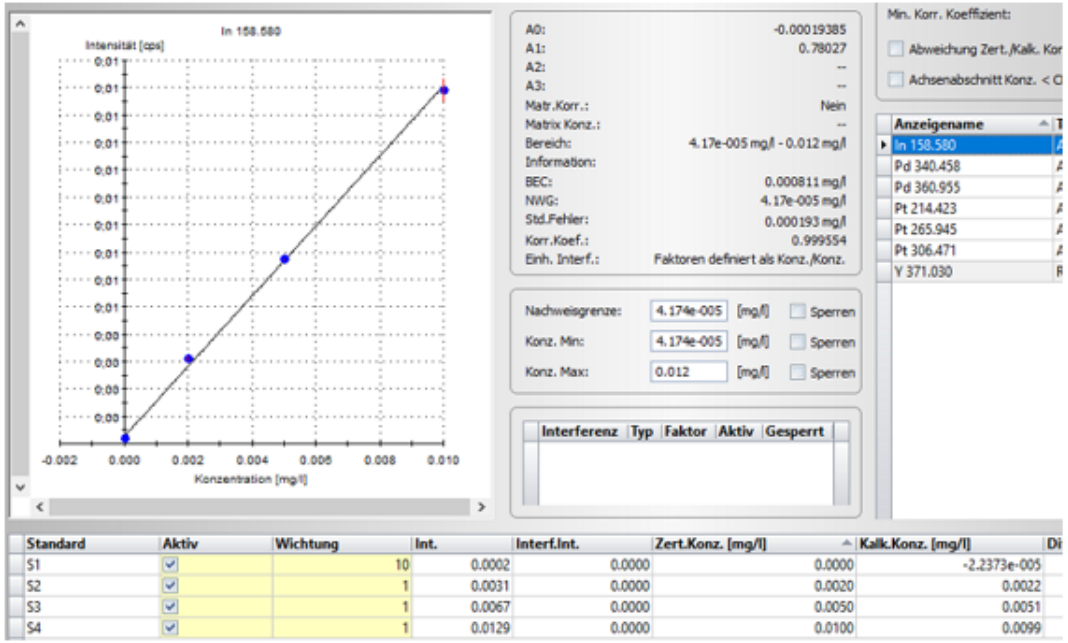
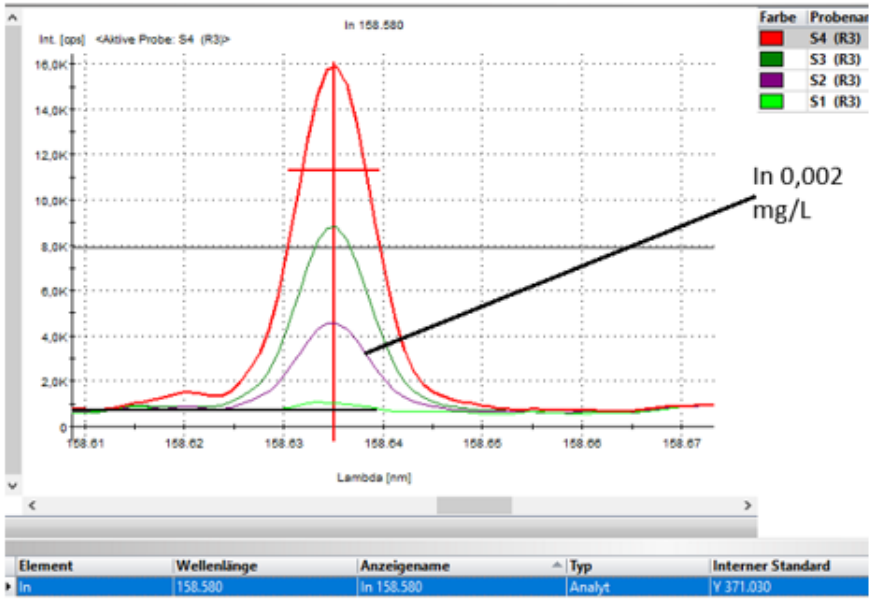
According to the German TRGS 900, the element indium has a limit value of 0.0001 mg/m³ → ICP must be able to measure 0,003 mg/L Indium for a 6 h sampling time

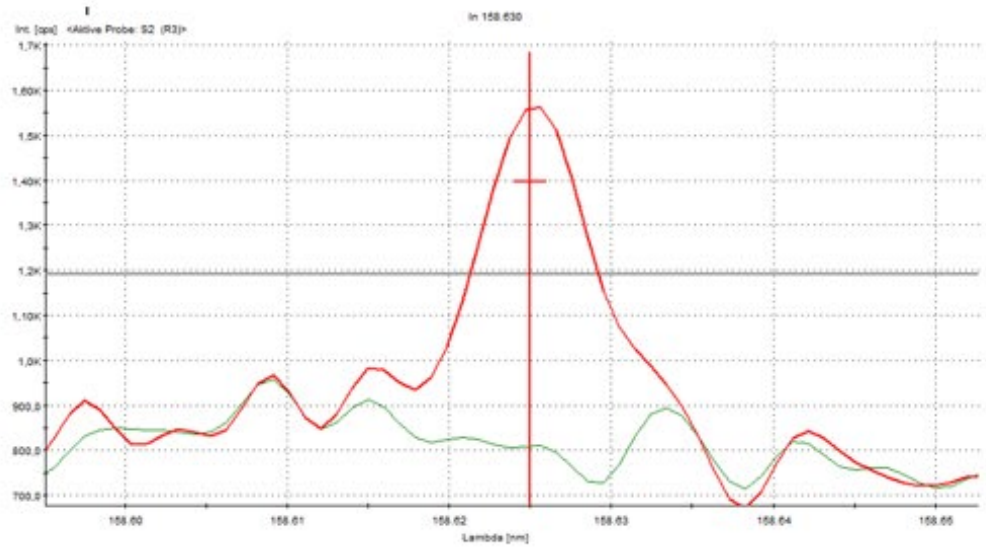




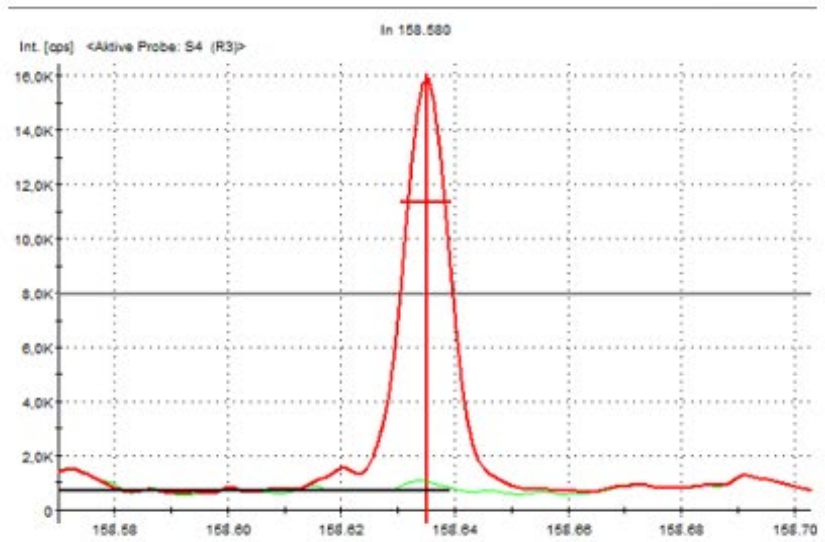
These images illustrate a Indium calibration using standards of 0.000 mg/L, 0.002 mg/L, 0.005 mg/L, and 0.01 mg/L. They also display the peaks in Indium 158 nm

Cross Flow and Ultrasonic Nebulizer comparison

This two spectra shows 0,01 mg/L Indium with Cross Flow nebulizer compared to an USN



EOP Plasma, Crossflow nebulizer and Scott chamber



EOP Plasma, Ultrasonic nebulizer

Calculation with ICP-OES Software

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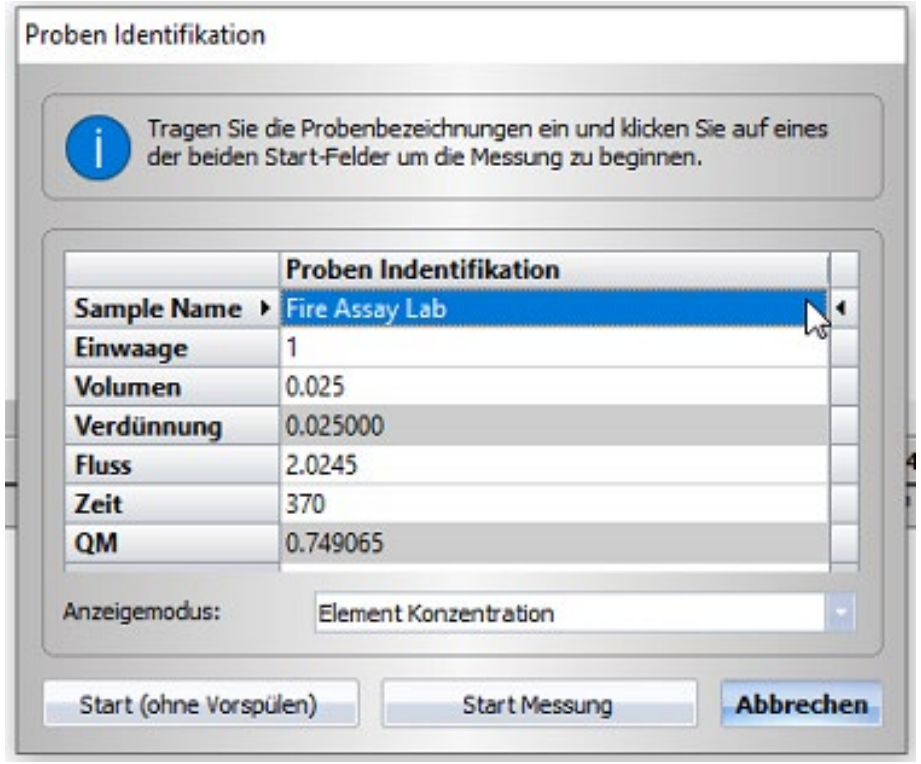
Input of all sampling data into the ICP-OES software

sampling data

- Sample name
- Volume sample
- Flow of the pump L/min
- Sampling time in min



Calculation with ICP-OES Software

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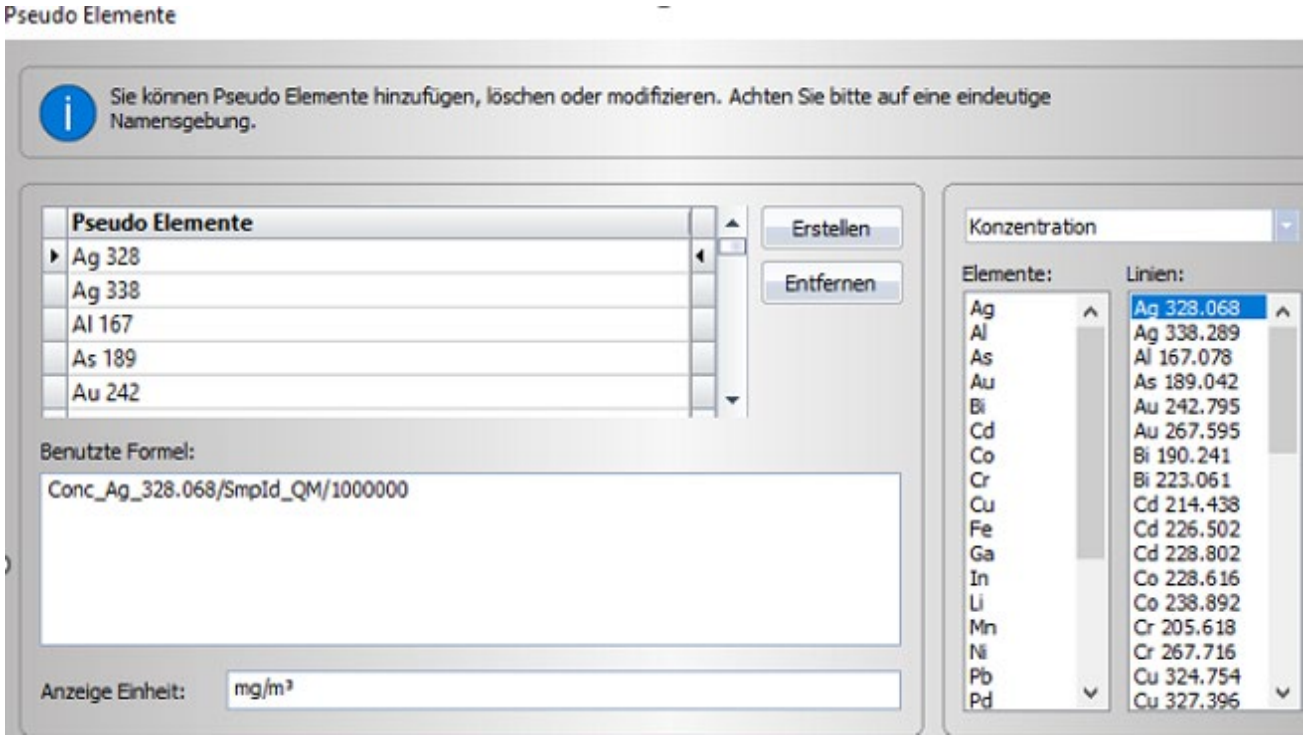
Calculation of values directly in mg/m³ with the ICP software by creating virtual elements



Probe	Typ		Pb 220
			mg/m³
Probierlabor A1	rep	!	0.00336
Probierlabor Z1	rep	!	0.00351
Probierlabor A2	rep	!	0.00335
Probierlabor Z2	rep	!	0.00338
Probierlabor A3	rep	!	0.00265
Probierlabor Z3	rep	!	0.00359

Pb results for the fire assay lab

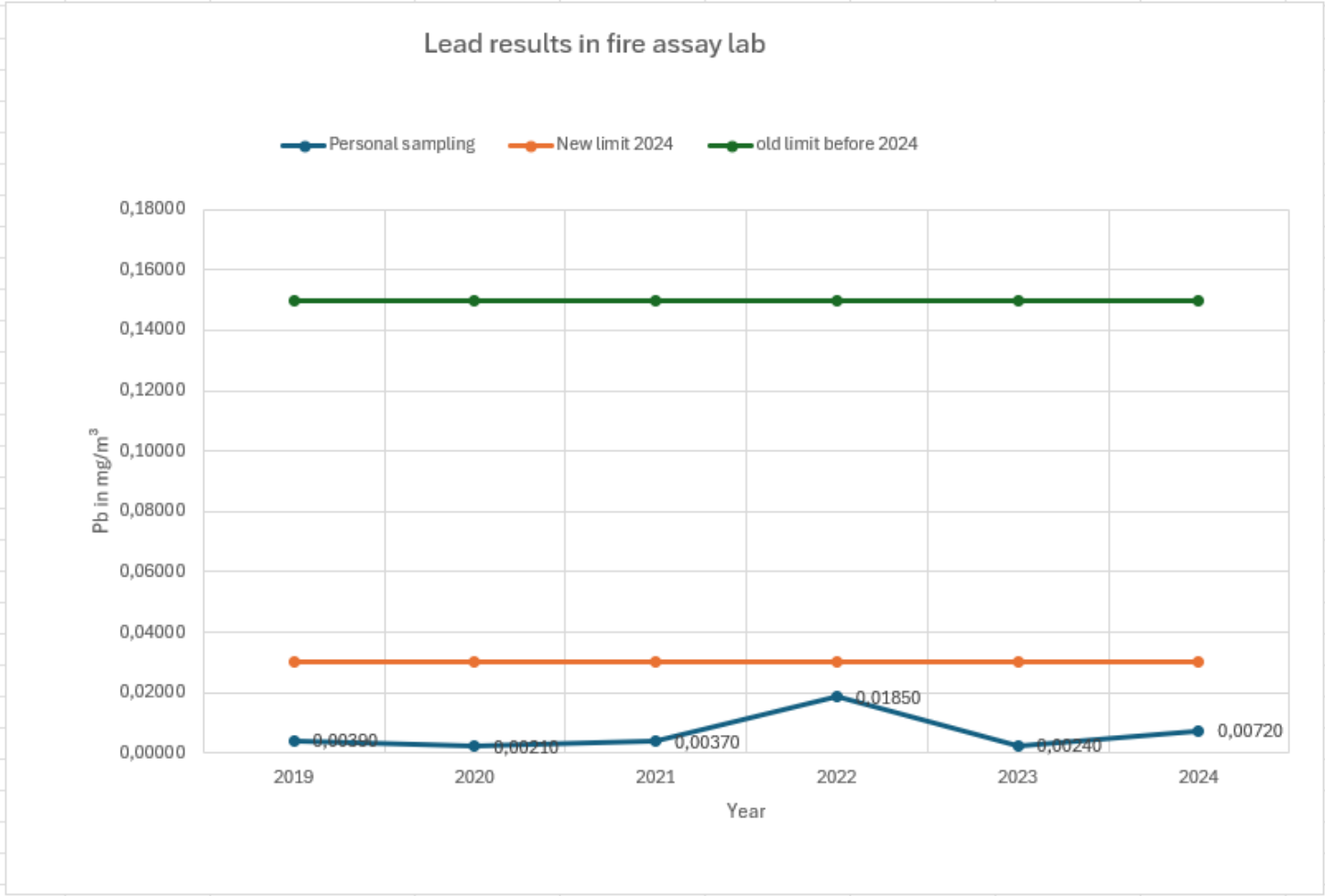
Results Pb in the fire assay lab

	respireable fraction	inhalable fraction	total dust
personal measurement	0,0033 mg/m³	0,0015 mg/m³	0,0048 mg/m³
personal measurement	0,0033 mg/m³	0,0039 mg/m³	0,0072 mg/m³
stationary measurement in the furnace room	0,0026 mg/m³	0,0014 mg/m³	0,0040 mg/m³
stationary measurement in the balance room	0,0006 mg/m³	0,0001 mg/m³	0,0007 mg/m³

according to the Guidline (EU) 2024/869 , the limit value is 0,03 mg/m³ total dust for 8 hours work

New directive for lead entered into force on 2 April 2024 and will apply from 9 April 2026.

Results lead 2019 – 2024 in fire assy lab



Personal sampling	
Year	**Pb max in mg/m³**
2019	0,00390
2020	0,00210
2021	0,00370
2022	0,01850
2023	0,00240
2024	0,00720
limit since 2024	0,03 mg/m³
limit before 2024	0,15 mg/m³

New directive for lead entered into force on 2 April 2024 and will apply from 9 April 2026.

Chlorinated platinum salts results for the Aqua Regia lab

Results Pt salt in the aqua regia lab

	Pt result
personal measurement	0,00006 mg/m³
personal measurement	<0,00005 mg/m³
room measurement	<0,00005 mg/m³
room measurement	<0,00005 mg/m³

Umicore limit 0,0001 mg/m³

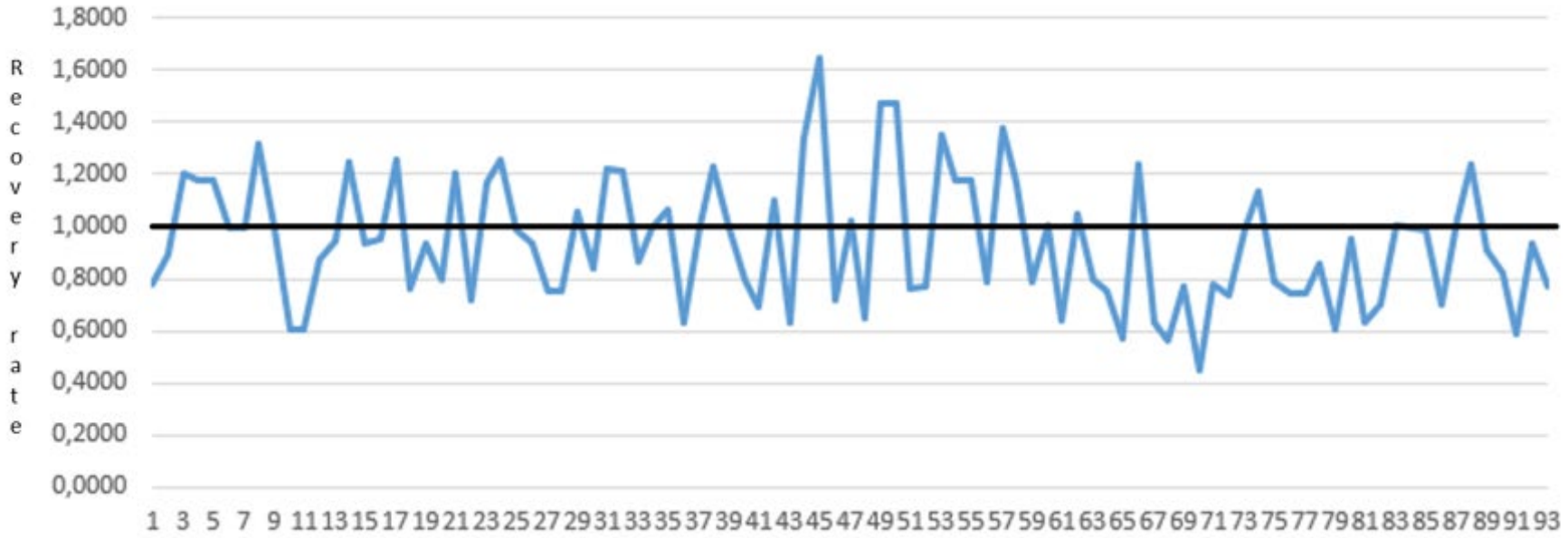
Detection limit of the method according to ICP-OES Software

0,00001 mg/m³

Comparison of IOM and Cyclon sampler for resireable dust

	Measure 1		Measure 2		Measure 3		
	IOM	Cyclone	IOM	Cyclone	IOM	Cyclone	Impaktor VC25-A
Cd	0,000471 mg/m³	0,000440 mg/m³	0,000470 mg/m³	0,000590 mg/m³	0,000460 mg/m³	0,000440 mg/m³	0,000367 mg/m³
In	0,001470 mg/m³	0,000930 mg/m³	0,003070 mg/m³	0,002420 mg/m³	0,003350 mg/m³	0,002150 mg/m³	0,004900 mg/m³
Ag	0,036100 mg/m³	0,033850 mg/m³	0,032480 mg/m³	0,040370 mg/m³	0,031910 mg/m³	0,032770 mg/m³	0,025800 mg/m³
Cu	0,008197 mg/m³	0,006070 mg/m³	0,004780 mg/m³	0,002690 mg/m³	0,004930 mg/m³	0,003830 mg/m³	0,004800 mg/m³

Recovery rate Cyclon to IOM sampler



Average Recovery Rate 94,1 %

T-test shows now siginificant differences between samplers

Summary

- Air sampling improves employee health
- Sources of danger are detected
- Base is the ISO norm 15202
- Determination of dust fractions possible using ICP-OES
- Different equivalent samplers are available

Questions ?

Thank you for your attention