# Combination of Fire Assay and Modern Instrumental Techniques for Precious Metals Analysis

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# Institut für Materialprüfung Glörfeld GmbH (IMG)

- Precise analysis of different materials since 1977
- Specialized Laboratory for Precious Metals analysis
- Certified by DIN EN ISO 9001 (2008)
- Sworn expert for chemical analysis of commercial purpose
  (by the chamber of Industry and Commerce) (IHK)
- Mainly work for industry, recycling companies,
  research institues, universities and private customers
- Production of certified reference materials, development of new analytical methods in Cu and PMs
  Collaborate with German Association of Metallurgy and Miners)(GDMB)

# Chemical Analysis offered at IMG:

## Analysis of Precious metals

- Ores, concentrates, intermediates, metallurgical products, electronic wastes, dental scrap, catalytic converters, fine metals and alloys
- All kinds of recycling materials

**E-scraps**

## Analysis of Metals

- Ferrous and non-ferrous metals, ores, concentrates

## Trace elements analysis

- Pure metals, precious metals, semiconductors, geological samples, etc.

**Sampling in** Germany and abroad in cooperation with competent sampling companies.

# Sample Preparation (Pre-treatment of Samples)

**Homogenisation of raw samples**
**(Mechanical + Thermal processes)**

- shredding and crushing (1000 kg or more of raw materials)
- incinerating (700 – 800 °C)
- melting in a furnace (up to 1800 °C)
- casting in a mould
- cutting the metal samples
- grinding the brittle metals and non-metal samples to analytical fineness (grain size <160 µm)

**Shredder**

**Hammer mill**

**Induction furnace (up to 1800 °C)**

**pouring melted sample into a casting mould**

**Sample divider**

# Metal Analysis by Traditional Method

## Analysis by Fire Assay (Published by G. Agricola in 1550 A.D.)

- The most accurate method for determination of Au (99.9%).
- Flux is used to convert infusible matter into fusible compounds at high temp, collecting of the precious metals in Pb or other collecting materials.

  (Main reagents of flux: Litthargo (PbO), $Na_2CO_3$, borax, silica)
- Used for the analysis of Platinum group metals (PGMs)

  (PGMs: Pd, Pt, Rh, Ru, Ir, Os, Au)
- Modification of the method is made for analysis of PGMs.

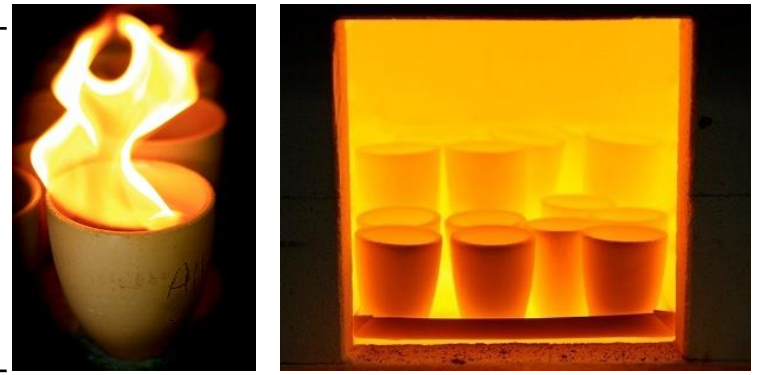
**Advantages of the method:**

- Elimination of interfering elements for the analysis.
- Efficiency of the recovery of PMs is ~100%.

# Collecting Methods for PMs in Fire Assay

| Collecting methods | Precious Metals |
|---|---|
| Lead collection | Ag, Au, Pd, Pt, (Rh)* |
| Nickel sulphide (NiS) collection | Pd, Pt, Rh, Ir, Ru, Os |
| Copper collection | Ag, Au, Pd, Pt, Rh, (Ir, Ru, Os)* |

* Under special conditions



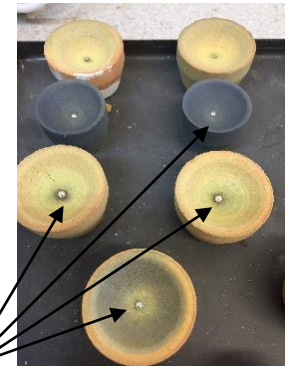
**Crucibles in furnace for fire assay at (1000 - 1100°C).**



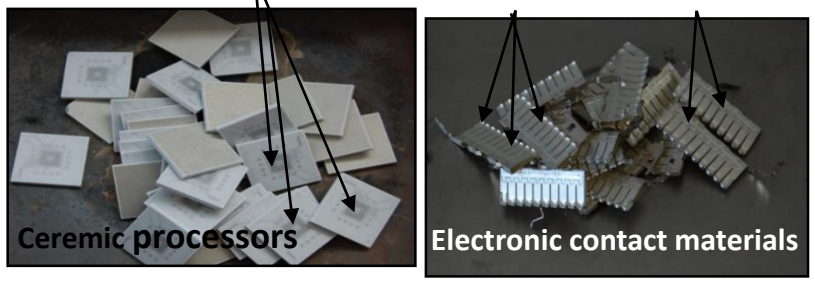


**PMs collected in Pb beads**


**Cupels in furnace (900 – 1000°C)**



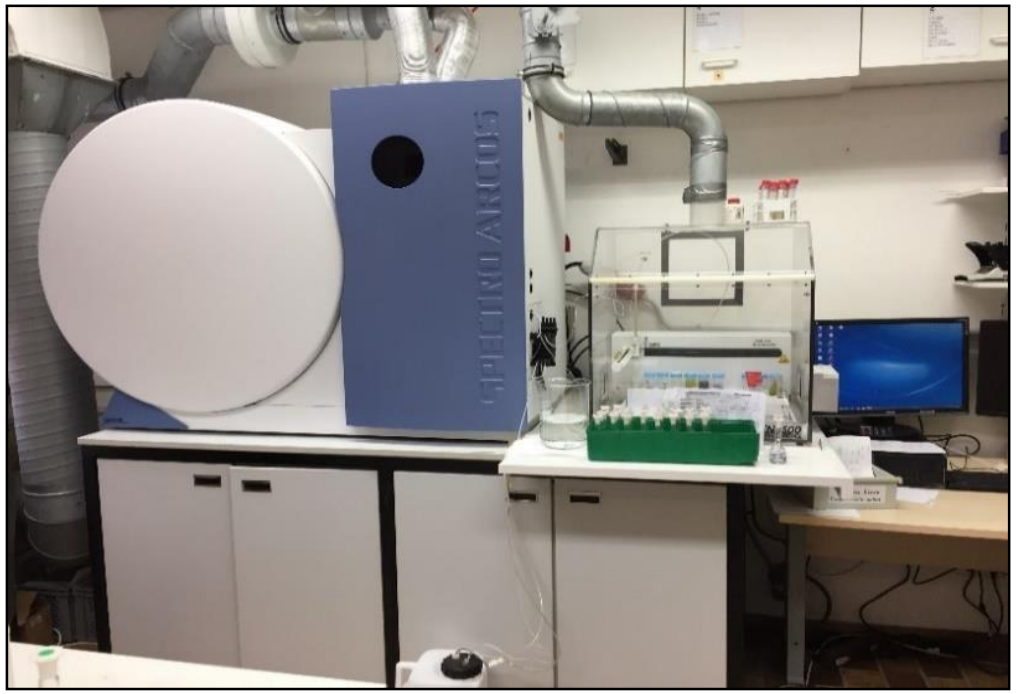
**Prills of PMs after cupellation (from lead fire assay)**



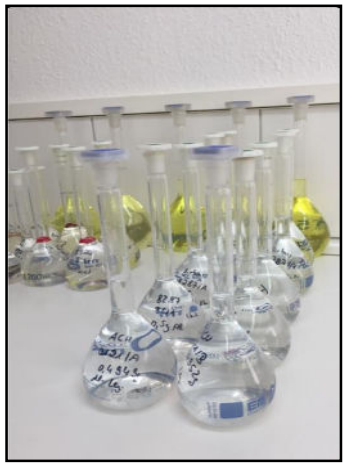

**Waste Electrical and Electronic Equipment (WEEE)**

# Modern Instrumental Techniques

## (A) Analysis by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES)




**Radial view plasma ICP-OES system (Arcos, SPECTRO)**



**Sample solutions for analysis by ICP**

**Elements present in WEEE**

| Element | g/t |
|---|---|
| Au | 10 – 300 |
| Ag | 500 - 5000 |
| Pd | 1 - 250 |
| Cu | 5 – 90 % |

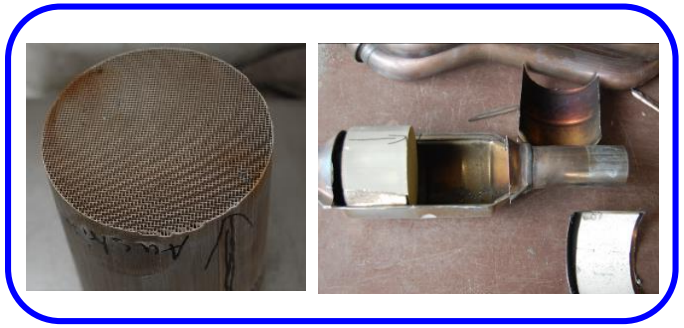
- Detection limit: < 1 ppm (mg/L)

- Destructive method
- Wide range of concentrations of PMs can be determined (depending on fire assay, sample material, amt. of sample)

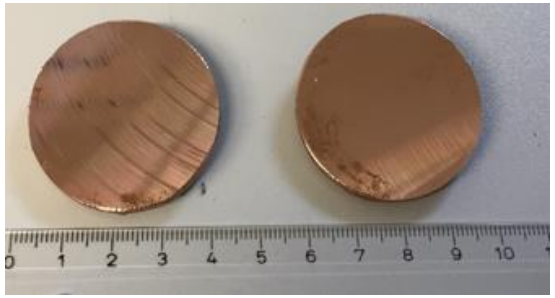
# (B) Analysis by X-Ray Fluorescence Spectrometry (XRF)

PMs in copper discs can be analysed

**Sample chamber of WD-XRF (4 KW), Axios, Panalytical**



**Automotiv catalytic converters**



**Copper discs (40 mm) for XRF analysis**

➢ Detection limit: 1 - 2 ppm

**Advantages of using XRF:**

A rapid and non-destructive technique

Reproducibility of the measurement

Typical applications:

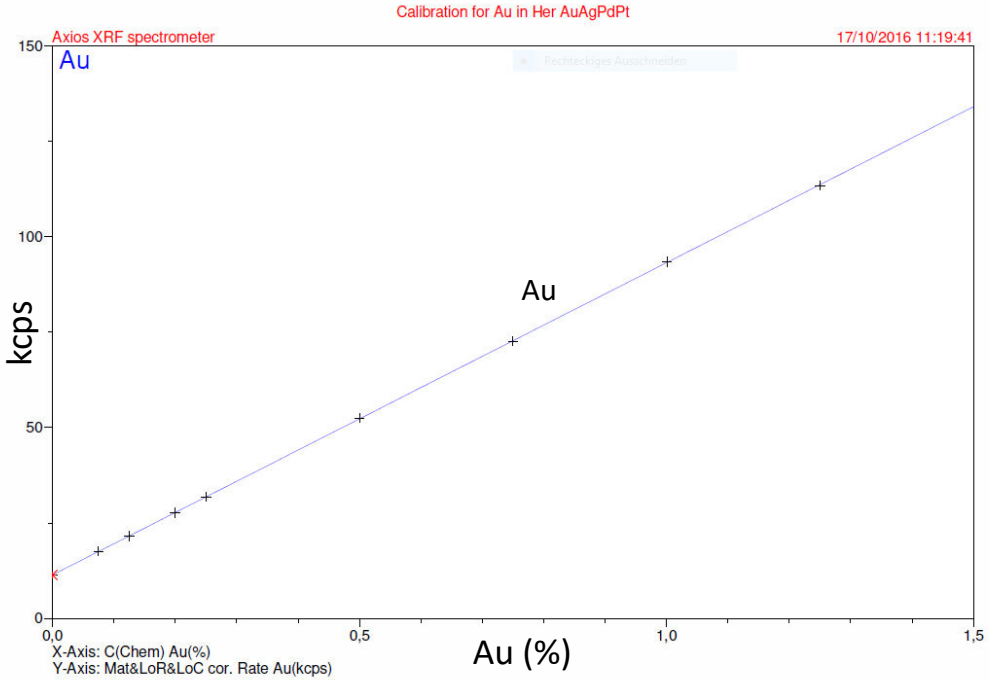
Analysis of

- reforming catalysts
- PGM concentrates
- PM containing ceramics
- automotiv catalysts
- alloys
- ashes, etc.

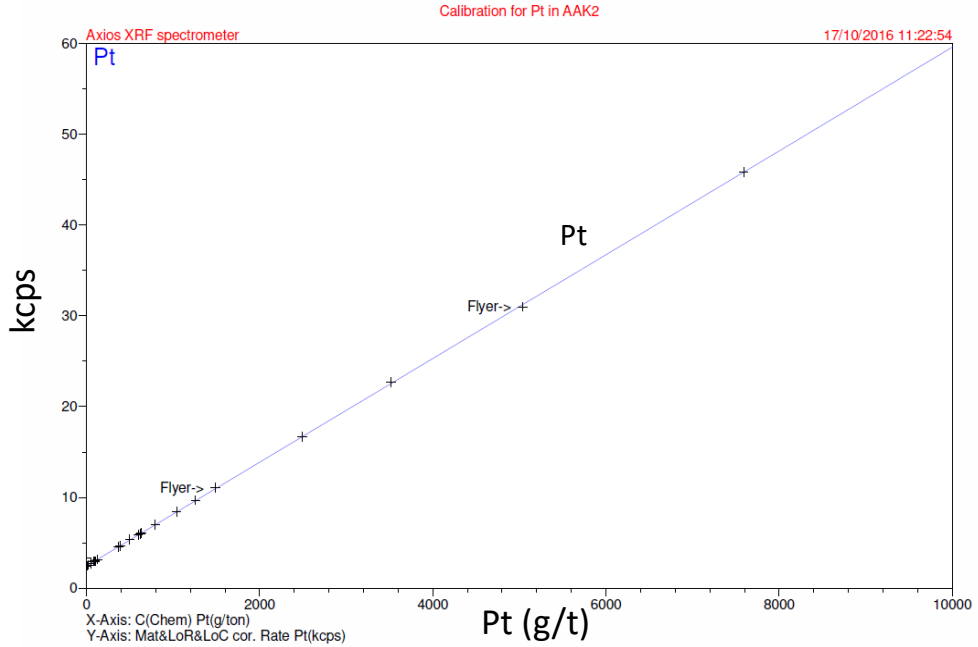
**Elements present in catalytic converters**

| Element | g/t |
|---|---|
| Pt | 500 - 2500 |
| Pd | 500 - 3500 |
| Rh | 50 - 500 |

# Linearity of the calibration curves in XRF




**Calibration curve for the analysis of gold present in sample prepared as copper disc.**

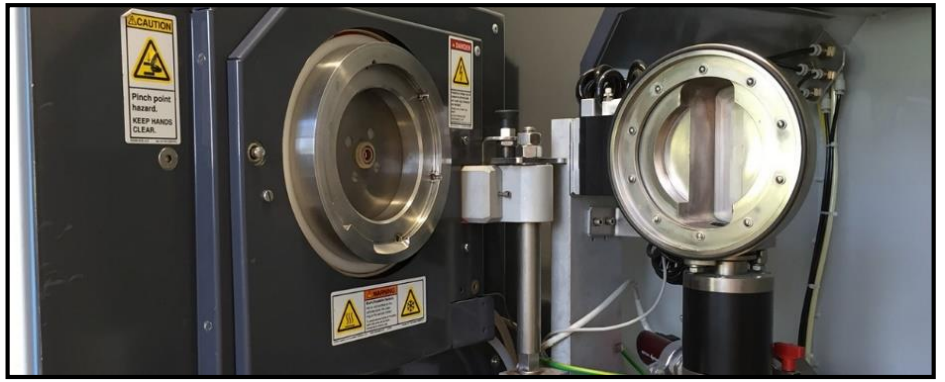



**Calibration curve for the analysis of platinum present in sample prepared as copper disc.**

# (C) Analysis by Glow Discharge Mass Spectrometry (GD-MS)

Analysis for PMs and impurities at background levels

**Thermo Scientific Element GD PLUS GD-MS (500 -1400 V), Thermo Fischer**

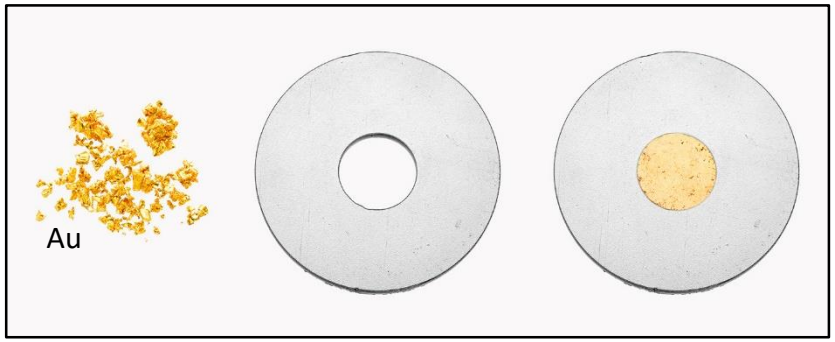



**Sample chamber with ion source in GD-MS PLUS.**

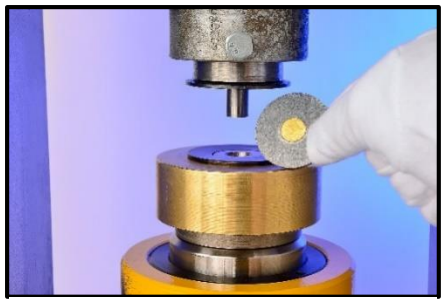
- Detection limit: 0.01 ppb (depending on analyte elements and sample matrix)

- Copper or lead or nickel discs from fire assay collection, after remelting step can be analysed
- Predestined for trace elements in pure metals
- High sensitivity
- Minimum calibration and less sample preparation
- Combination of XRF and GD-MS enables to determine from some ppb to 100%.

# Analysis of pure metal powders by GD-MS

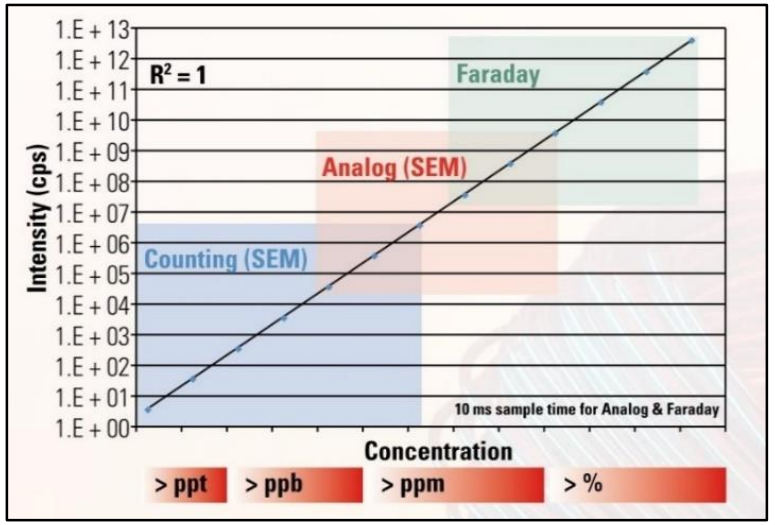



**Gold sample pressed into orifice for GD-MS measurement (direct analysis)**



**Hydraulic press**

**Sample mounted in ion source**




**Detector ranges for the ELEMENT GD-MS.**

**Metallic disc before and after GD-MS measurement.**

**Some trace elements in Au (99.99%) analyzed by GD-MS***

| Element | % |
|---|---|
| Pt | 0.0026 |
| Cu | 0.0011 |
| Fe | 0.0011 |
| Ag | 0.0005 |
| Zn | 0.0002 |
| Pd | 0.00013 |
| Rh | 0.00003 |
| Ca | 0.00002 |
| Bi | 0.000012 |

*measured by a dual mode SEM + Faraday collector

# Detection Ranges:

| Instrument | Concentration range |
|---|---|
| ICP-OES | <1 ppm – 100% |
| XRF | 1 ppm – 100% |
| GD-MS | 0.01 ppb – 100% |

# Conclusion

**Advantages from the combination of fire assay and modern analytical techniques**:

- industry standard process for obtaining Au, Ag and PGMs.
- efficiency of the recovery of PMs is nearly 100%.
- worthwhile to recycle different kinds of materials
  (E-scraps, automotive catalysts, chemical catalysts, electronic and galvanic industries production waste)
- analysis of PMs in the primary raw materials (ores, concentrates).

**Important to know the very precise content of PMs**:

for selling, buying or recycling to ensure the quality and value of the materials.

**Well experience with good background of understanding on the analysis in IMG**:

produces the accurate and precise assay results.

# Thank you very much for your attention!!!

Welcome to our booth

Welcome to IMG in Willich