Recent Advances in Measuring Precious Metals in Alloys

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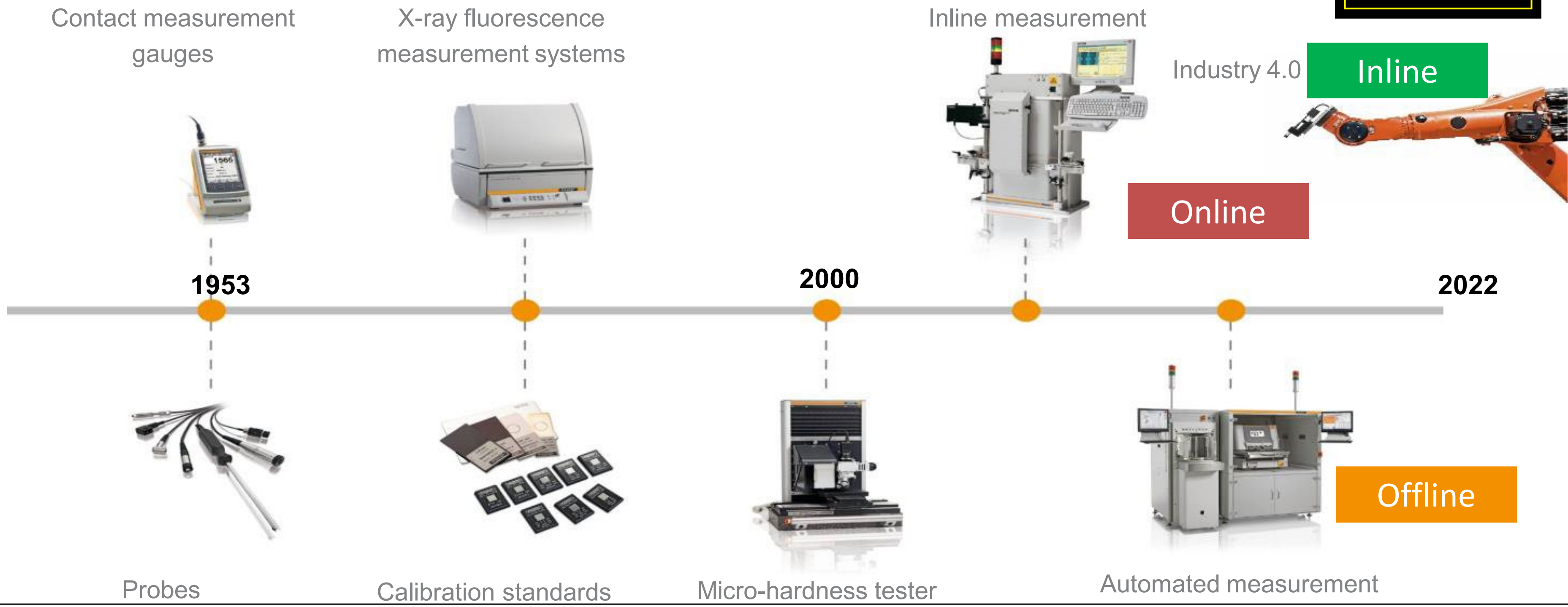
Helmut Fischer Group

- Head Quarter in Sindelfingen, Germany
- 25 subsidiaries worldwide, presence in more than 100 countries.
- Leading supplier of EDXRF instruments for precious metal analysis and decorative coating thickness measurement.

- Fischer India- 100% subsidiary established in Year 2006.

Offering Innovative Solutions for more than 70 years



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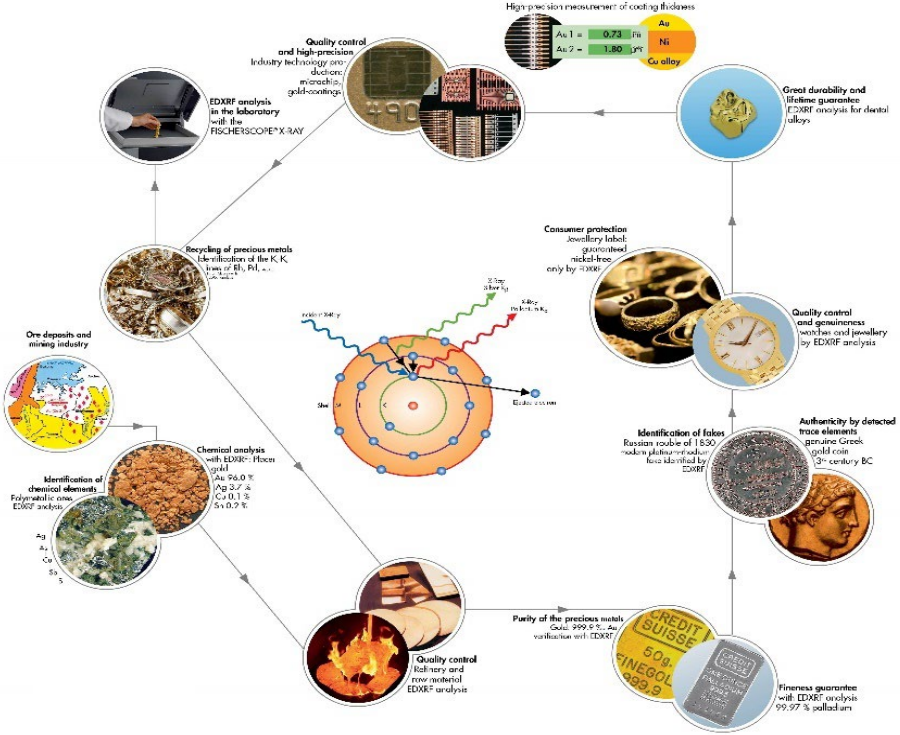
Stages of Gold Processing



Role of Analysis

- Verifying the purity of gold and other precious metals.
- Identification of various elements within the alloy and their composition.
- Identifying the impurities for measurement of adulteration within the alloy.
- Quality and Process Control.
- Measurement of decorative coating thickness.

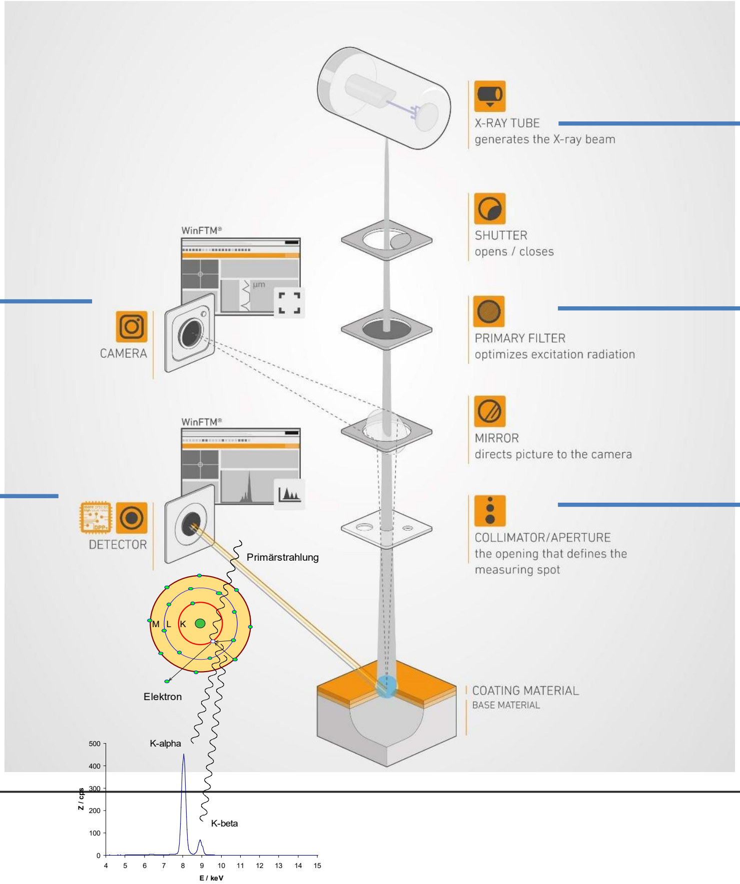
Analytical Methods of Precious Metal Analysis

- **Various analytical methods for gold and alloy analysis**
  - Acid Test
  - Fire Assaying
  - ICP-OES (requires sample to be atomized to enable excitation)
  - X-ray Fluorescence Analysis (XRF)

Why EDXRF Method ?

- Fast, Quick, Accurate and Non Destructive.
- Requires no or very less sample preperation.
- Measurement is done directly on solid sample and does not rely on acid digestion.
- Quantitative Analysis of gold alloys including Platinum Group Metals (Pt, Pd, Rh, Ru, Ir, Os).
- Easy to use and practically feasible.
- Quantification of all metals of interest in one single measurement.
- Advances in detector and signal processing sensitivity has improved precision and accuracy of the measurement comparable to fire assay method.

Major Components of an EDXRF



X-Ray tube:
Micro Focus tube with Beryllium Window

Camera:
High Resolution CCD Camera

Primary Filter:
Fixed or Multiple Changeable Filters like
Nickel, Aluminum , Mylar etc.

SDD Detector / Si PIN Detector/PC :
Resolutions : 140eV/ 180eV/ 800eV

Collimator:
Fixed or Motorised Changeable Collimators like -
Ø 0.2 mm (7.9 mils), Ø 0,6 mm (23.6 mils), Ø 1 mm (39.4 mils), Ø 3 mm (118 mils) others on request

Factors Affecting Accuracy and Reproducibility of XRF Measurements

Factors related to Instrument:

- Resolution of the Detector
- Pulse Processor
- Collimator
- Measuring distance
- Measuring time
- Calibration

External Factors:

- Sample Homogeneity
- Environmental Conditions
- Positioning of the Sample
- Focusing
- Measurement Approach

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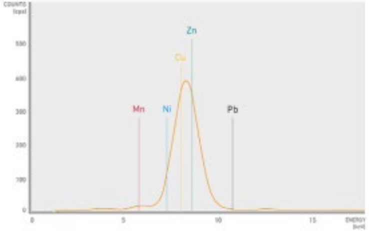
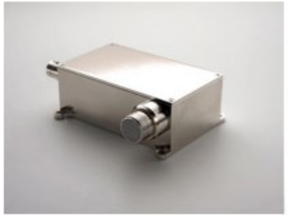
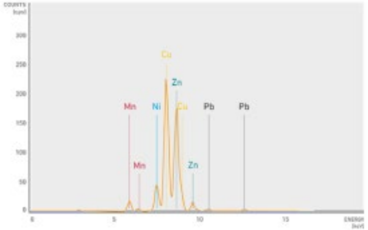
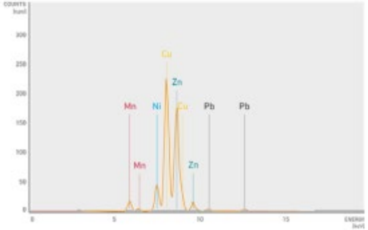
Detectors









- Proportional Counter Detector:
  - High count rates due to the big entrance window.
  - E-resolution ~ 900 eV (Mn-Ka)
  - Used for simple coating thickness applications.
- Silicon Pin Detector:
  - Lower cps due to a smaller entrance window.
  - E-resolution ~ 170-200 eV (Mn-Ka)
  - Advantages for „complex“ applications (e.g. thin layers, trace elements, composition analysis etc.
- Silicon Drift Detector (SDD):
  - Semiconductor device (functionality like a PIN-Diode, with a ring structure for the Si-Chip)
  - Better energy resolution: ~ 135 eV for 10mm² and 30mm² Chip
  - Higher count rates (optimal at ≤ 150’000 cps) while still having a good energy resolution and peak form (shape)
  - Better sensitivity for light elements (at low energies)
  - Applications: Thin layers, trace elements, light elements

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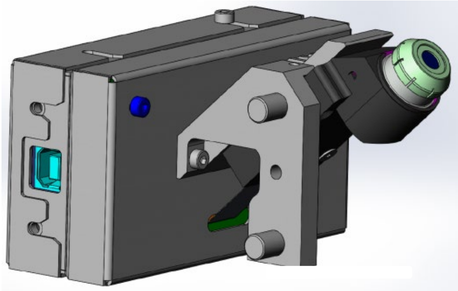
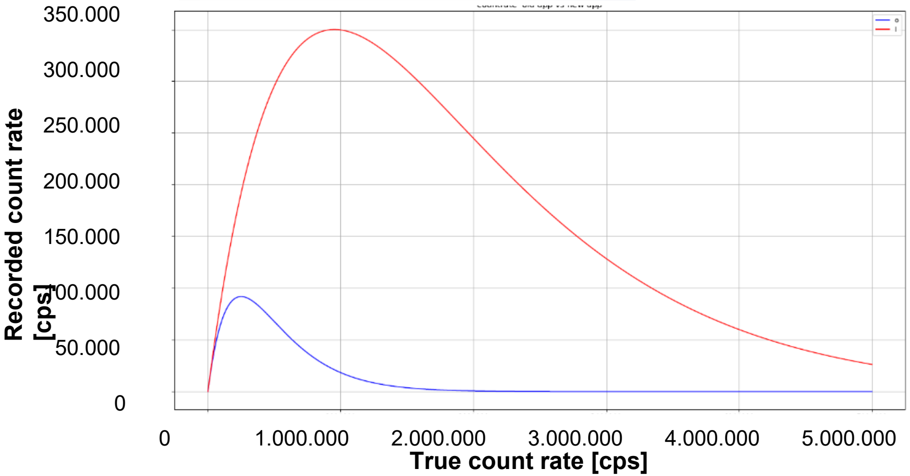
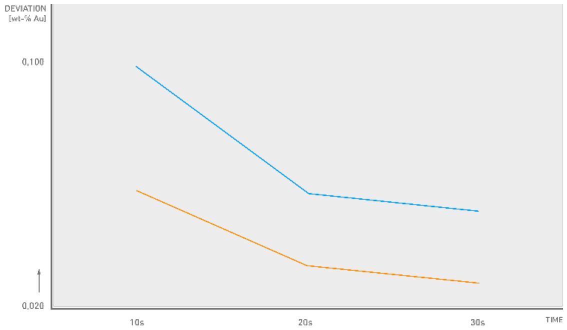
New Detector Technology - SDD with DPP+

Function	Existing Setup 20/30 mm² SDD + old DPP electronics	New Setup (SuperSense) SDD + DPP: DPP+
Max. energy resolution Fe_{55}	< 165 eV	< 140 eV
Time to peak	1.6 µs	0.4 µs
Top width	0.4 µs	0.125 µs
Count rate in cps	up to 100.000	up to 500.000
Diagnostic features	Bus Errors	• Tracking power supply • voltages and currents • BIAS voltage • cooler current • detector temperature • HT operation

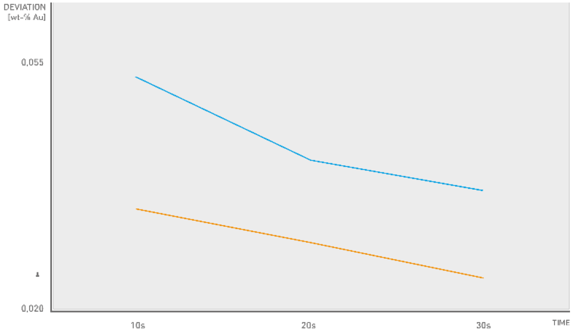




Better Absolute standard deviation (precision) with high countrate and good energy resolution

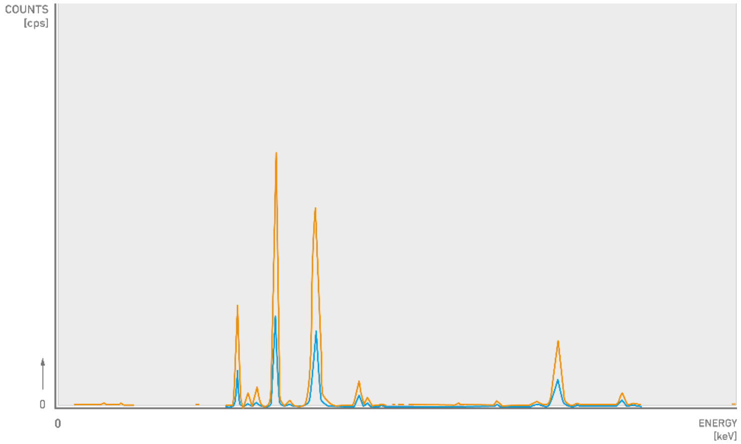


585 gold (14 carat)



950 gold (23 carat)

- New SDD Detector with DPP+
- Old DPP

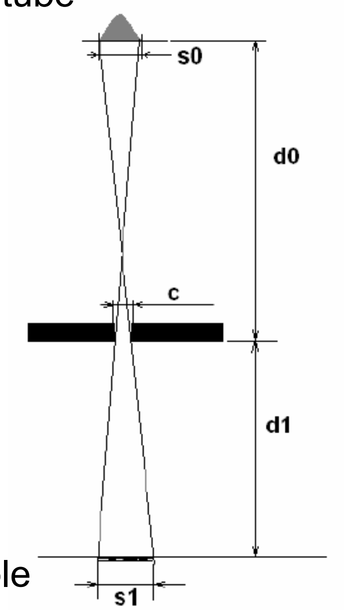


High countrate with good energy resolution

- New SDD Detector with DPP+
- Old DPP

Measurement Spot Size

X-ray tube



Sample

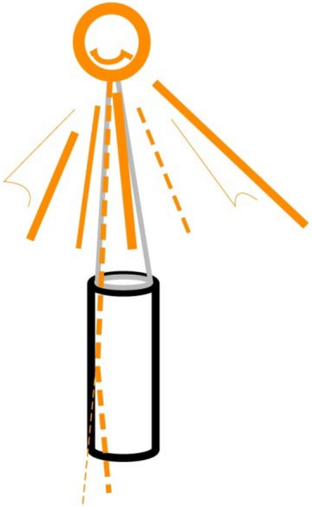
$$M = c \cdot (d0 + d1) / d0 + s0 \cdot d1 / d0$$

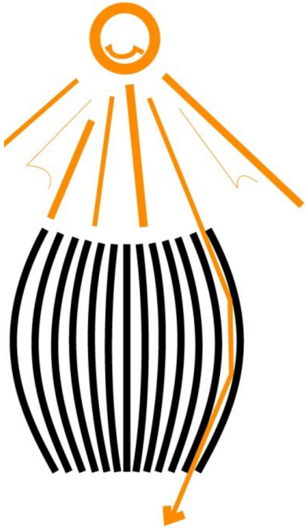
The size of the measurement spot depends on

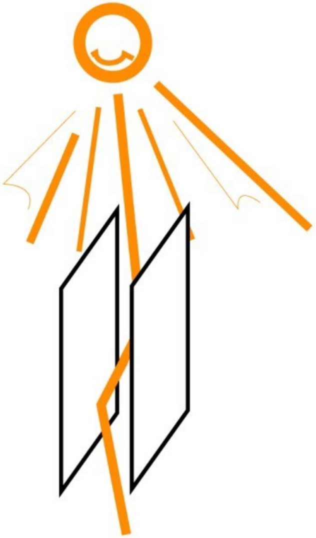
- size of collimator c,
- focal spot of the anode s0
- ratio d0/d1.

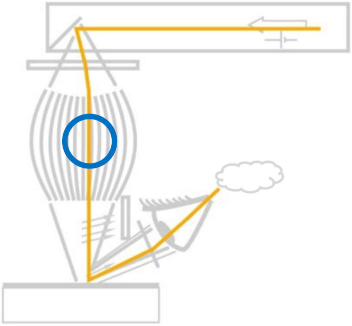
- Small Measurement spot allows for the best determination of inhomogeneity's
- Large Measurement spot allows for low influence of inhomogeneity

Advanced Optical Collimation Systems





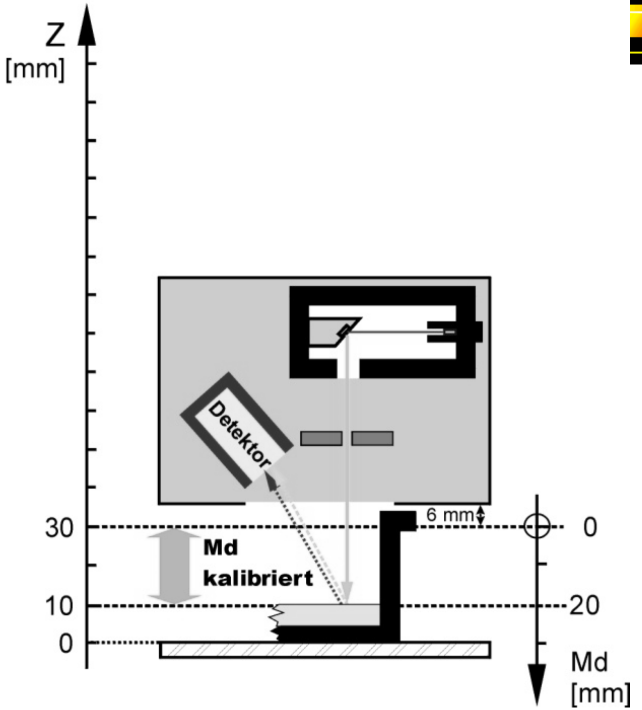




mono capillary	poly capillary	mirror optics	
100 – 300	~ 10 - 60	20 x 50	spot size [μm]
~ 10	~ 500	~ 10	amplification

Measuring Distance - Md

- Automatic correction of the reading
- Any md in the valid calibrated range can be chosen
- Signal (count rate) ~ 1 / (measuring distance)^2
  repeatability ~ √count rate
- → keep measuring distance small!



Measuring Time

- Repeatability: s ~ √measuring time
- Four times longer measuring time will improve the repeatability by a factor of 2.
- Sometimes very long measuring time will not further improve the standard deviation. It is better to utilise the mean value of several measurements
- With advances in signal processing electronics, very accurate results can be obtained with a measuring time of 15 - 30 secs.

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Calibration

- To ensure valid results, measuring equipment shall be calibrated against measurement standards traceable to international or national measurement standards.
- Ensures Traceability of measurement results
- Helps to minimize the Systematic Error
- Integrity of the calibration standards is very important

Thank You for Your Attention!

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