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FIRE ASSAY SL - CARRER DE NOVELL, 54 - 2nd FLOOR ES-08014 BARCELONA - SPAIN - EU

WARE-HOUSE TSM Log. – B-682, CTRA. ACCÉS COSTA BRAVA km 1,4 ES-08389 **PALAFOLLS** – BCN – SPAIN – EU

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3.1.1. GOLD JEWELLERY ASSAY METHODS

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REVIEW OF METHODS FOR MEASURING GOLD CONTENT



1. FIRE ASSAY (CUPELLATION)

The most accurate method, with an accuracy of 2 - 3 parts per ten thousand (0.02%), is the <u>Fire Assay (Cupellation)</u> method.

This involves taking a small scraping from the article, typically about 250 mg, weighing it accurately, wrapping it in <u>lead foil</u> with some added silver (known as '<u>inquart</u>'), <u>cupelling</u> it in a furnace at about 1100°C to remove all base metals.

Then placing the resulting gold-silver alloy <u>bead</u> in <u>nitric acid</u> to dissolve out the silver (known as 'parting') and re-weighing the resulting pure gold.

This is the *standard reference technique* used <u>by the national Assay laboratories worldwide</u> for Hallmarking and is covered in the international Standard, ISO 11426:1997



2. INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY (ICP-MS)

Fire Assay is closely followed for accuracy by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) which involves taking a smaller sample of about 20 mg, dissolving in acid and subjecting a sample to analysis in an ICP Mass Spectrometer - an expensive instrument.

This technique has an accuracy of 1 ‰ but requires use of comparative <u>standard reference</u> <u>alloy samples</u> of known composition.

This technique is accepted for <u>Hallmarking</u> purposes and has the advantage in that it also measures the other alloying constituents.

It is a quick technique, an assay taking about 3 min., and the results can be automatically printed out by the computer. It also measures the content of the other alloying metals present



3. X-RAY FLUORESCENCE - (XRF)

X-Ray Fluorescence (XRF) is a non-destructive technique suitable for normal assaying requirements such as in-house quality control in manufacturing or for 'certifying' gold content in retail outlets.

It has an <u>accuracy</u> of, typically, 2-5 ‰ under good conditions, i.e. where the surface of the jewellery being measured is relatively flat and sufficiently large.

On curved surfaces, the gold X-rays generated and measured are scattered and accuracy is reduced significantly.

However, it only measures the gold content of a *thin surface layer*, so accuracy is severely compromised where the jewellery article has had a chemical surface treatment (to enhance colour) or has been electroplated with a layer of pure gold.

The more accurate XRF instruments measure the *intensity* of the generated gold X-rays by wavelength dispersion analysis. The use of energy dispersive analysers results in cheaper instruments but reduced accuracy. <u>Reference alloy standards</u>, of known composition close to that of the test piece, are needed if accuracy is required in XRF testing.

There are several instruments appearing on the market developed specifically for gold jewellery assaying, such as the X-tester, and these are more reasonably priced.

A major jewellery retailer in India has equipped each of their stores with such instruments. The gold content of each piece is measured as it is sold, printing off a Certificate, guaranteeing caratage conformance and providing consumer confidence in a country where national <u>Hallmarking</u> regulations do not exist.









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3.1.1. GOLD JEWELLERY ASSAY METHODS

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REVIEW OF METHODS FOR MEASURING GOLD CONTENT (cont.)



4. Touchstone testing

<u>Touchstone</u> testing is an ancient method for measuring gold content whereby a rubbing of the jewellery is made on a special touchstone alongside rubbings of known reference samples and treated with acids

The colour of the reacted area is compared to that of the reference sample.

It is not sufficiently accurate (about 15 ‰ at best) and is only useful as a sorting test to differentiate between different caratages.

It is less accurate at high caratages and with white golds.



5. GOLDEN PEN

The *Electronic Gold Tester* (or the so-called <u>Gold Pen</u>) is a cheap, although portable technique based on the *capacitance decay* principle.

Accuracy is poor, being correct to only 1 - 2 carats (4 - 8%) and is compromised if the surface is gold-plated, for example.

It is useful only as a sorting test.



6. DENSITY

The density of carat golds reduces as caratage is lowered and this gives rise to <u>density</u> <u>measurement</u> as a possible method of measuring gold content, using Archimedes principle.

However, density is also influenced by the other alloying constituents and so the accuracy of the method is poor.

Jewellery containing defects such as porosity would further reduce the accuracy of density measurement. It is *not recommended*.

7. Summary

- 7.1. For **high accuracy**, consistent with marking / Hallmarking regulations:
 - Only Fire Assay and ICP Mass Spectrometry are sufficient.
 - Both techniques involve taking a physical sample (a scraping) from the jewellery item.

7.2. For good accuracy,

- X-ray Fluorescence (XRF) analysis is suitable.
 - Accuracy depends on the shape (geometry) of the item; it is best on flat surfaces.
 - This technique is suitable for quality control in production.
 - It is a quick technique (3 4 min.) and does not require technical expertise to operate.
 - Results are automatically displayed / printed out by the computer control.
 - Suitable bench-top instruments developed specifically for jewellery use are available on the market.
 - Reference standards are necessary.
- 7.3. For **sorting jewellery** into different caratages:
 - Touchstone and electronic Gold Pens are suitable cheap, quick techniques

Information extracted from: World Gold Council website

► <u>Dr. C. W. Corti article -</u>

"Assaying of gold jewellery choice of technique"

► Roland Young article
"Analysis for Gold - Review of Methods"







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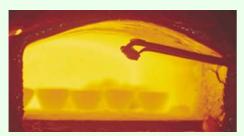
3.1.1. COMPARISONS OF METHODS

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1. Position of Fire Assay (Cupellation) Method

IN RELATION TO ALTERNATIVE ANALYSIS TECHNIQUES

FROM FEW SAMPLES ANALYSIS



Cupellation Furnace



Cupellation: single cupels

TO MASS SAMPLES ANALYSIS



Cupellation: Cupel Blocks



Parting & Annealing

ADVANTAGES

1. HIGH ACCURACY

o Five times better than next best method (ICP)

2. GREAT FLEXIBILITY

o Suitable for all carat range of alloys

3. Low Detection Limit

o Important for ore / residues analysis

4. MANY SAMPLES ASSAYED AT ONCE

- o Batch assay method allows many samples to be simultaneously processes in parallel,
- o all other methods of Gold Analysis are sequential (samples are processed 1 by 1)

5. FULL SCALABILITY

- Same Universal method from few to mass analysis
- o using same equipment
- only consumable / tools change

6. WITHOUT REFERENCE STANDARDS

o Other methods need alloys close to that of test piece

7. LEGAL STANDARD METHOD

o All Assay Offices / Hallmarking Labs employ it

8. STANDARD REFERENCE TECHNIQUE

o Against which all other methods are compared

9. MICRO CUPELLATION TECHNIQUES

o Allow smaller samples / shorter PROCESS time

10. Low Capital Costs

Low Investment necessary for implementation

DISADVANTAGES

1. DESTRUCTIVE OF THE SAMPLE

o (slightly better with micro-cupellation)

2. NOT VERSATILE

o Only gold is determined

3. HEALTH HAZARDS

o associated with lead









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3.1.1. COMPARISONS OF METHODS

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2. QUANTIFIED COMPARISONS OF ASSAYING TECHNIQUES

TECHNIQUE	VERSATILITY	SAMPLE SIZE (mg)	ACCURACY (%)	LIMITATIONS	EQUIPMENT Cost (\$)
1. FIRE ASSAY	Only gold	~ 125 - 250 ~ 10 mg (*) (*) micro-cupellation	0.02	Modifications for Ni & Pd	Moderate \$50,000
2. <u>ICP</u>	Complete analysis	~ 20	0,1	-	High \$150.000
3. <u>XRF</u>	Complete analysis	Non- destructive	0.1 - 0.5	Surface layer, flat samples	Moderate \$25,000 +
4. Touchstone	Only gold	Almost non- destructive	1 – 2	Unsuitable for high carat & white golds	Low \$100
5. GOLDEN PEN	Only gold	Non- destructive	4 – 8	Not consistent	Low \$200
6. DENSITY	Only gold	Non- destructive	Poor	Only for binary alloys	Low \$500

Information extracted from: World Gold Council website

► Jewellery Technology Pages (WGC) Detailed review of Assaying Methods for Gold Alloys in ▶ Dr. C. W. Corti article

For Methods of Gold Analysis in **Auriferous Ores / Residues** ► ALS Laboratory Group

MANY SAMPLES ASSAYED IN PARALLEL (BATCH PROCESSING)



4 Bullion Block 12 HB with Fork BF 12-48 48 probes cupelled & handled in parallel



Quartz Tray QBR 25 25 probes in parallel



Quartz Tray QBR 49 49 probes in parallel Batch Parting & Annealing



Seven Fire assay crucibles FC 55 unloaded in a row



Parting 25 samples at same time



Fork load 30 cupels # 7B (*)

(*) Fork MF 7 takes 30 cupels # 7B (array 6 x 5) directly from carton to furnace in a single movement