



# FIRE ASSAY



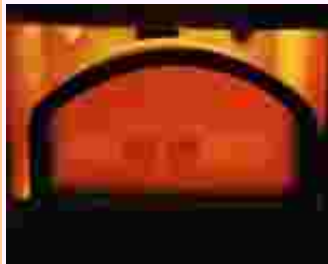
Passeig Marià Estrada, 12  
ES-08328 ALELLA – BARCELONA – SPAIN  
+34 934 181 310  
+34 628 094 398 (Mob)  
fireassay@fireassay.eu – www.fireassay.eu

Review 1  
Comparison 1 ◀ ▶ Review 2

## GOLD JEWELLERY ASSAY METHODS

6-Feb-18  
GoldAnalysis\_Review\_1.doc

### 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 Fire Assay (Cupellation) method.

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

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

This is the standard reference technique used by the national Assay laboratories worldwide 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 standard reference alloy samples of known composition.

This technique is accepted for Hallmarking 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 accuracy 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. Reference alloy standards, 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 Hallmarking regulations do not exist.

**Fire Assay** is the **Standard Reference Technique** against which the other methods are compared

JEWELLERY ASSAYING TECHNIQUES: ▶ Review of Methods (1) ▶ Review of Methods (2) – COMPARISONS: ▶ pros / cons (1) – ▶ Quantified (2)

TERMS: ▶ Cupellation ▶ Fire Assay ▶ Reagents ▶ Other Methods ▶ Metals – SHEETS: ▶ Cupels ▶ Crucibles – INDEX: ▶ Programme



# FIRE ASSAY



Passeig Marià Estrada, 12  
ES-08328 ALELLA — BARCELONA — SPAIN  
+34 934 181 310  
+34 628 094 398 (Mob)  
fireassay@fireassay.eu — www.fireassay.eu

Review 2  
Review 1 ◀ ▶ Comparisons

## GOLD JEWELLERY ASSAY METHODS

6-Feb-18  
GoldAnalysis\_Review\_2.doc

### REVIEW OF METHODS FOR MEASURING GOLD CONTENT (cont.)



#### 4. TOUCHSTONE TESTING

Touchstone 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 Gold Pen) 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 density measurement 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

▶ 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

**Fire Assay** is *the Standard Reference Technique* against which the other methods are compared

**JEWELLERY ASSAYING TECHNIQUES:** ▶ Review of Methods (1) ▶ Review of Methods (2) – **COMPARISONS:** ▶ pros / cons (1) – ▶ Quantified (2)

**TERMS:** ▶ Cupellation ▶ Fire Assay ▶ Reagents ▶ Other Methods ▶ Metals – **SHEETS:** ▶ Cupels ▶ Crucibles – **INDEX:** ▶ Programme



# FIRE ASSAY



Passeig Marià Estrada, 12  
ES-08328 ALELLA — BARCELONA — SPAIN  
+34 934 181 310  
+34 628 094 398 (Mob)  
fireassay@fireassay.eu — www.fireassay.eu

Comparisons 1  
Review 2 ◀ ▶ Quantif. Comp. 2

## COMPARISONS OF METHODS

6-Feb-18  
GoldAnalysis\_Comp\_1.doc

### 1. POSITION OF **FIRE ASSAY** (CUPELLATION) METHOD IN RELATION TO ALTERNATIVE ANALYSIS TECHNIQUES

#### FROM FEW SAMPLES ANALYSIS



Cupellation Furnace

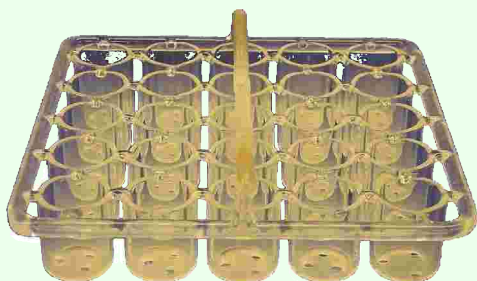


Cupellation: single cupels

#### TO MASS SAMPLES ANALYSIS



Cupellation: Cupel Blocks



Parting & Annealing

#### ADVANTAGES

- 1. HIGH ACCURACY**  
Five times better than next best method (ICP)
- 2. GREAT FLEXIBILITY**  
Suitable for all **carat range** of alloys
- 3. LOW DETECTION LIMIT**  
Important for **ore / residues** analysis
- 4. MANY SAMPLES ASSAYED AT ONCE**  
Batch assay method allows many samples to be simultaneously processed **in parallel**, all other methods of Gold Analysis are **sequential** (1 by 1)
- 5. FULL SCALABILITY**  
Universal method from **few** to **mass analysis** using same equipment (only consumable / tools change)
- 6. WITHOUT REFERENCE STANDARDS**  
Other methods need alloys close to that of test piece
- 7. LEGAL STANDARD METHOD**  
All Assay Offices / Hallmarking Labs employ it
- 8. STANDARD REFERENCE TECHNIQUE**  
Against which all other methods are compared
- 9. MICRO CUPELLATION TECHNIQUES**  
Allow **smaller samples** / shorter process time
- 10. LOW CAPITAL COSTS**  
**Low Investment** necessary for implementation

#### DISADVANTAGES

- 1. DESTRUCTIVE OF THE SAMPLE**  
(slightly better with microcupellation)
- 2. NOT VERSATILE**  
**Only gold** is determined
- 3. HEALTH HAZARDS**  
associated with **lead**

**FIRE ASSAY** is *the Standard Reference Technique* against which the other methods are compared

**JEWELLERY ASSAYING TECHNIQUES:** ▶ Review of Methods (1) ▶ Review of Methods (2) – **COMPARISONS:** ▶ pros / cons (1) – ▶ Quantified (2)  
**TERMS:** ▶ Cupellation ▶ Fire Assay ▶ Reagents ▶ Other Methods ▶ Metals – **SHEETS:** ▶ Cupels ▶ Crucibles – **INDEX:** ▶ Programme



# FIRE ASSAY



Passeig Marià Estrada, 12  
ES-08328 ALELLA – BARCELONA – SPAIN  
+34 934 181 310  
+34 628 094 398 (Mob)  
fireassay@fireassay.eu – www.fireassay.eu

Comparisons 1  
Comp. Position 1 ◀...▶ Review 1

## COMPARISONS OF METHODS

6-Feb-18  
GoldAnalysis\_Comp\_2.doc

### 2. QUANTIFIED COMPARISONS OF ASSAYING TECHNIQUES

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

(\*) MICROCUPELLATION: ~ 10 mg

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)



Quartz Tray  
20 probes in parallel



Quartz Thimbles

Batch Parting & Annealing



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

**FIRE ASSAY** is *the Standard Reference Technique* against which the other methods are compared

**JEWELLERY ASSAYING TECHNIQUES:** ► [Review of Methods \(1\)](#) ► [Review of Methods \(2\)](#) – **COMPARISONS:** ► [pros / cons \(1\)](#) – ► [Quantified \(2\)](#)  
**TERMS:** ► [Cupellation](#) ► [Fire Assay](#) ► [Reagents](#) ► [Other Methods](#) ► [Metals](#) – **SHEETS:** ► [Cupels](#) ► [Crucibles](#) – **INDEX:** ► [Programme](#)