ED-XRF AS ALTERNATIVE REFERENCE TECHNIQUE FOR THE QUALITY CONTROL OF JEWELLERY & PRECIOUS METAL PRODUCTS.

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HALLMARKING - 'VIENNA CONVENTION' REQUIREMENTS

XRF testing permitted under 'Convention' is subject to meeting the following requirements:

1. XRF to be used as destructive techniques and surface must be prepared before the analysis.

2. Internal XRF method must be accredited to the latest version of ISO 17025.

3. The Assay Laboratory is obliged to have the reference testing (e.g. Fire Assay for gold / Potentiometric for silver / ICP-OES for Pt/Pd) and these reference methods must also be accredited by a national accreditation body (e.g. NABL/UKAS).

4. For an item to pass, **the result (Measured value) of the test sample to be higher than or equal to the standard of fineness after deduction of the measurement of uncertainty**; This is nothing but **'Decision Rule'** proposed in ISO 17025:2017.

5. A fit-for-purpose XRF instrument, reference material, qualified staff and written procedures to be in place.

MEASUREMENT OF UNCERTAINTY & CONFORMITY ASSESSMENT

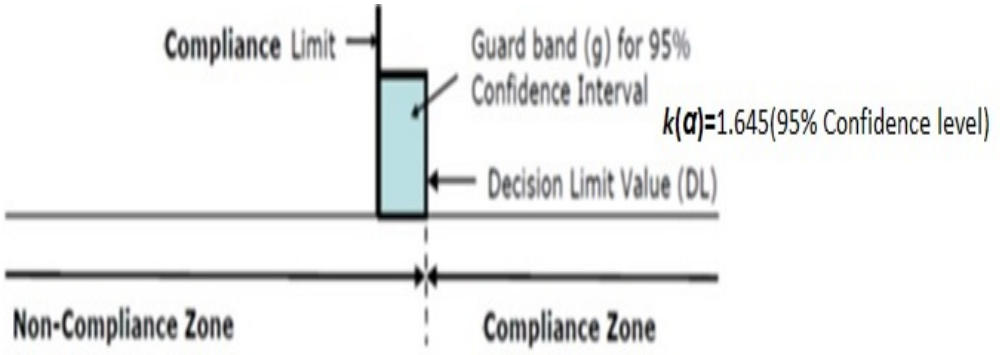
- It is clear from the 'Convention's XRF norms' that **measurement of the uncertainty** of XRF has a important role to play in the **conformity assessment** of jewellery items.

  (Note: We conduct 1 day MOU courses, next is scheduled in India, SEEPZ, Mumbai)

- 'Convention' also talks about implementation of the **'Decision Rule'** and accreditation of XRF method to ISO 17025.

  - ✓ Clauses (3.7/ 7.1.3/ 7.8.6.1) of ISO 17025: 2017 states that **'Decision rule'** must be applied when results are reported as a **'Pass'** or **'Fail'** and this rule must be agreed with the customer at the contract review stage.
  - ✓ Accreditation for any regulatory methods reporting results as a 'Pass' or 'Fail' will not be granted unless requirements of Clauses (3.7/ 7.1.3/ 7.8.6.1) of ISO 17025: 2017 is fulfilled.

DECISION RULE - CONFORMALITY ASSESSMENT - FORMULA



Carat	Regulatory Limit (%)	Decision Limit (%) (For MOU= 0.31% Relative)	Difference
9 ct	37.50	37.69	0.19%
14ct	58.50	58.79	0.29%
18ct	75.00	75.38	0.38%
22 ct	91.60	92.12	0.52%

ASSUME –

MOU for 18 ct gold = **0.31% relative** - this means its value for 18ct gold will be (0.31/75)*100= 0.23%

The total width of this **guard band/confidence interval** (green box above) = 1.645 * 0.23 =0.38%

(Note: Single tail, multiply by 1.645 (statistical factor) = Expanded Uncertainty)

Hence, Decision Limit = 75% + 0.38%=75.38% (for 18 ct gold, when uncertainty is 0.31% relative **– I have taken a worst case scenario**)

IF Measured Value ≥ 75.38% then item will be treated as **'Pass'.**

IF Measured Value < 75.38% then item will be treated as **'Likely Fail' (refer to reference testing).**

IT IS A FOOLFPROOF APPROACH, ALWAYS WORKS WITH A VERY HIGH LEVEL OF ACCURACY. But ……

FIRE ASSAY TECHNIQUE – REAL TIME UNCERTAINTY

- While analysing sample by the fire assay method, we run two or more proof /check samples of known purity; these reference samples are treated exactly in the same way as the other samples of the batch.

- Based on the results of proof/check samples, a correction factor for **GOLD** is calculated and it is applied on a proportionate basis to the gold results of all the samples to arrive at the **proof corrected results**. (**Note:** Correction factor must be within certain limit to ensure accuracy of the results, any value…. Not acceptable)

- The proof correction factor is nothing but it is a measure of the **DRIFT** in the process, or in other words, we can also call it a **DYNAMIC UNCERTAINTY OF THE PROCESS**. This dynamic uncertainty is **much smaller** than then the uncertainty calculated by the conventional method, but it is true uncertainty for that batch of the sample.

- If this concept of measuring drift/dynamic uncertainty of the process is true in case of fire assay results, then why can't it be true in case of testing by the XRF route? Let try a similar approach with the XRF results.

APPLYING FIRE ASSAY REAL TIME UNCERTAINTY CONCEPT TO THE XRF TECHNIQUE (ALTERNATIVE MODEL)

- Like fire assay, in XRF analysis too, we can calculate the process drift in the element of interest **(Au)** as a correction factor (Real time Uncertainty) by running a closely matched reference sample.

- **Condition to be met:**
  - ✓ The reference standard must contain all the elements present in the test sample and it should be analysed nearly at the same time as sample i.e. in the same environmental conditions.
  - ✓ `Measured value of reference standard must be within uncertainty range for that reference standard. If is not then machine must be re-calibrated and the process is repeated.

Drift in the element of interest (e.g. Au) = (Certified value – Measured value)

Decision Limit - Test Sample:

Decision Limit = Measured value of the **test sample** + **Drift** as per above

Decision Limit >=Standard of fineness =**Pass**

Decision Limit < Standard of fineness = **Likely Fail –** ***(sample to be quarantined for further testing by the reference method, usually we do not fail any sample by XRF route).***

Summary

- We have discussed two different models for calculating **Decision limit**:
  - Guard band by using measurement of uncertainty calculated by the Conventional Method.
  - Drift correction factor - a measure of measurement of uncertainty calculated on a real time basis.

- Either of the above two approaches may be used and applied when results are reported as **a 'Pass' or 'Fail'.** Both the approaches work for both **Indian & Western** type of jewellery and produce equally good results subject to **critical point below**:

❖ **The most important part here is the calculation of measurement of uncertainty and the drift/ correction factor;** if these two important parameters are **NOT** correctly estimated, then results will have some inaccuracy in them, and the Assay Centre will be putting more samples for reference testing (Fire Assay) than actually required.

- It is not possible for me to **divulge the full details of the test method and statistical principles applied to arrive at the correct results but I can confirm both the models work satisfactorily**. Western world is already following either of the two models. Second one is most popular.

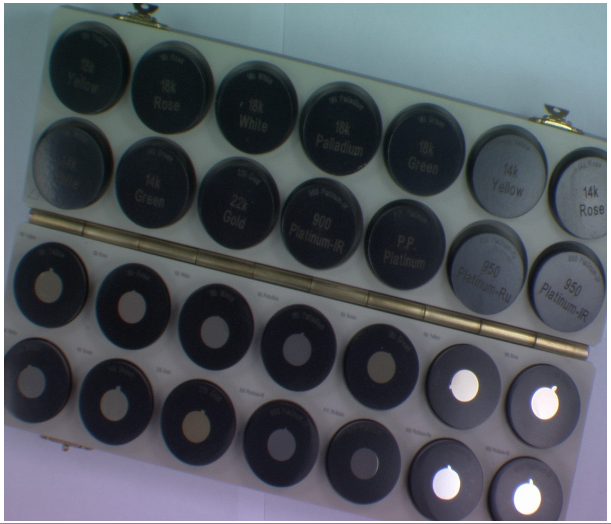
WAY FORWARD - INDIA

What India needs to implement XRF method for the hallmarking application:

- a standardised method (someone has already done the hard work☺);
- A set of tailored calibration standard samples with international traceability;
- A **statistical sampling plan (important)** along with a Technical and operational Manual;
- Training course at the end of the project;
- AND …… **an open mind** – don't form an opinion or make a decision until you know all the facts. Try before rejecting it.

BENEFITS

- It will create a common quality assurance system across the internal market.
- It will also improve the mutual acceptance of results between manufacturers, retailers, hallmarking centres, law enforcers and regulatory authorities.
- But…….. No single institution has all expertise.



Joining hands is the only way forward

UNDERCARATING ISSUES WITH HALLMARKED JEWELLERY WHO IS RESPONSIBLE?

Bangle Readings	Au%	Ag%	Cu%	Cd%
Edge of Bangle				
Positon 1 Left of item surface	89.5	1.9	6.9	0.5
Positon 2	88.0	3.7	7.9	0.5
Positon 3	90.6	1.7	6.8	0.5
Positon 4	90.0	3.0	6.3	0.9
Positon 5	85.3	3.5	10.0	0.8
Positon 6	27.5	34.5	26.5	6.0
Positon 7	21.3	40.1	26.2	7.1
Positon 8	29.3	35.1	23.9	7.0
Positon 9 Right of item surface	65.8	16.4	13.7	2.1

Sample Component Description	Fire Assay Results (%)	
Bangle (Samples were taken from three different locations)	Replicate 1	67.99
	Replicate 2	68.33
	Replicate 3	71.85
	Average	**69.39**

WHO IS RESPONSIBLE?

- If any Hallmarked product is found to be of the substandard quality and concerned retailer is charged for **'Product Liability '& Breach of Statutory Duty'** then as a first step - **BOTH** retailer & Hallmarking centre will have to demonstrate by documentary evidences that they took **all the reasonable steps under the law** ; and **exercised all due diligence** to avoid committing the offence.

  - **All reasonable steps –** They have systems/procedures of control.
  - **Exercising all due diligence –** Their system work (evidence required).

- Hallmarking centre can be punished only when they have failed to adhere to the approved procedure or when there is proven negligence on their part. This punishment will be limited to fine or the temporary/permanent suspension until they bring improvement to their quality system.

- The **ULTIMATE RESPONSIBILITY** lies with the **RETAILER** as they have a legal obligation to ensure that jewellery items meet the requirements of the Hallmarking regulation. **They must and should have exercised control at all levels to meet this obligation.**

- When it comes to compensating customer (victim of fraud), it will be the retailer's sole responsibility to compensate. I am advocating the same principle is the UK.

Thank you for your attention☺

Any Questions…

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