Role of Sample Preparation in Spectroscopic analysis of Metal Samples

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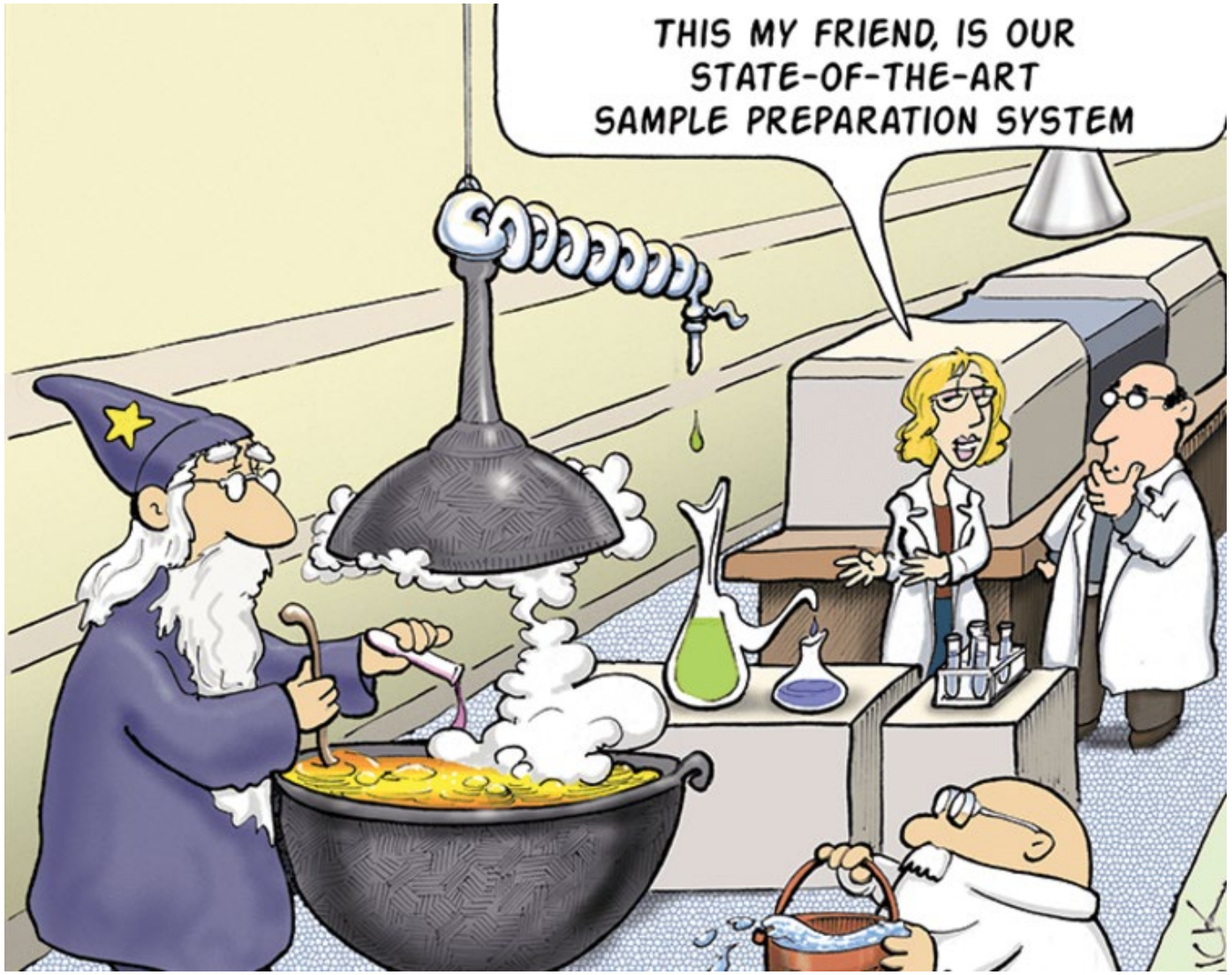
Introduction

Sample Preparation



Introduction

- One of the three major aspects
- Preliminary step in the analytical process
- Isolating and concentrating the analytes of interest while removing interferences.
- Essential for – Accuracy, reproducibility, sensitivity
- High-quality & reliable data in any analytical workflow.

Objectives

Objectives

- Importance of metal sample preparation for spectroscopy analysis.
- Problems associated with sample preparation
- Techniques involved in sample preparation
- Best possible practices to ensure reliable results.
- Conclusion

Challenges in Sample Preparation

Challenges

- General Metal sample preparation
- Two challenges-
  1) Dealing with sample variability
  2) Reproducibility issues.
- Quality and consistency of the sample
- Sampling plan and sample preparation
- Surface contamination
- Unstandardized sample preparation procedures

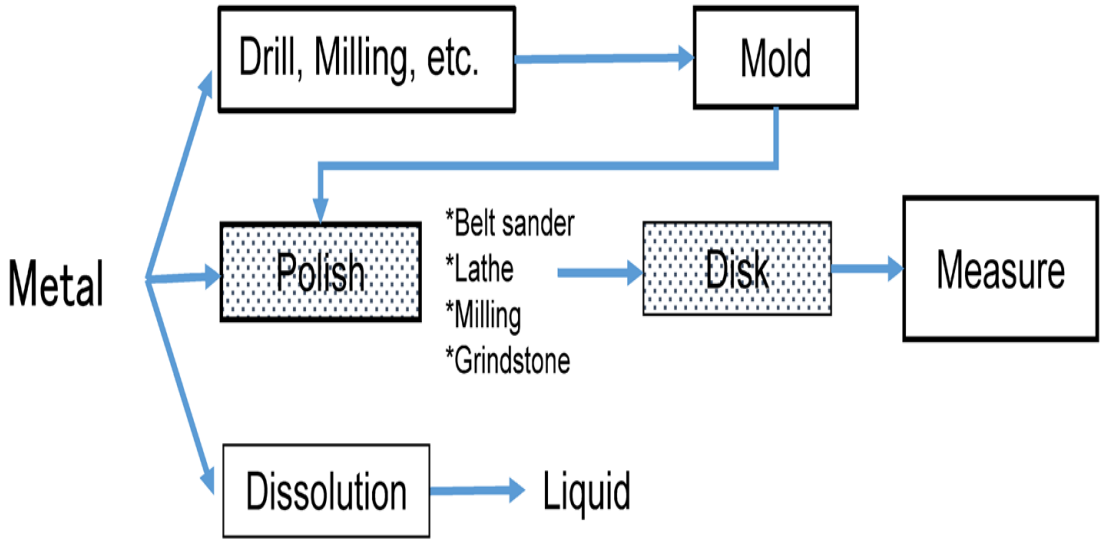


Fig-1

Challenges –Disadvantages of Sample preparation

- The physical and chemical changes that samples undergo during preparation.

- Key issues include :

- Contamination Risks
- Loss of Volatile Compounds
- Sample Degradation
- Compatibility for acid dissolution
- Thermal Effects
- Time and Labor Intensive
- Cost Implications: Environmental and Safety Concerns

Sample Preparation- Spark OES

Sample preparation in Spark OES

- The secret of perfect OES analysis: SAMPLE PREPARATION
- Homogeneous Samples
- Representative and with an even surface
- Morphology of the solidification and cast structure of the sample
- Use of Spark Stand and contamination of electrode

Pictorial presentation samples for Spark OES

Fig-1

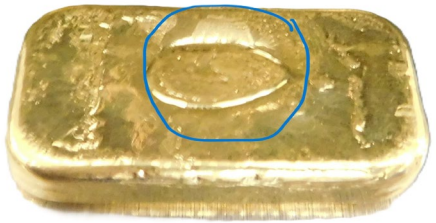
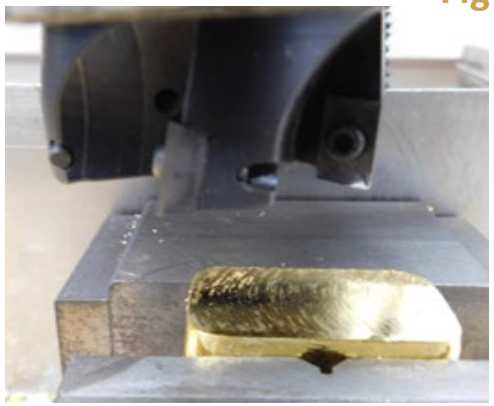


Fig-2

Fig-3



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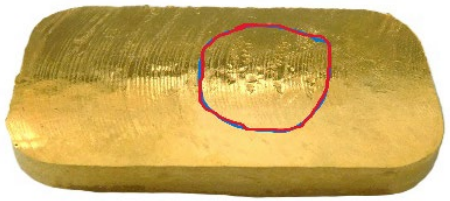


Fig-5

Fig-6

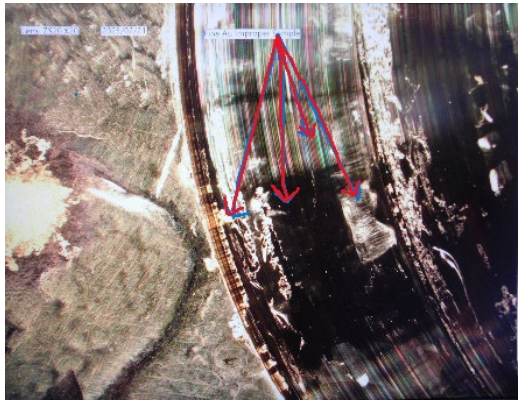
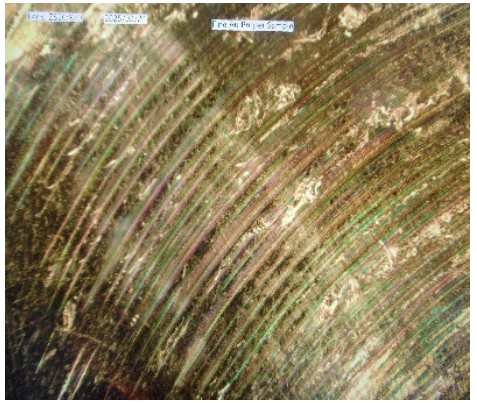


Fig-7

Fig-8

Fig-9



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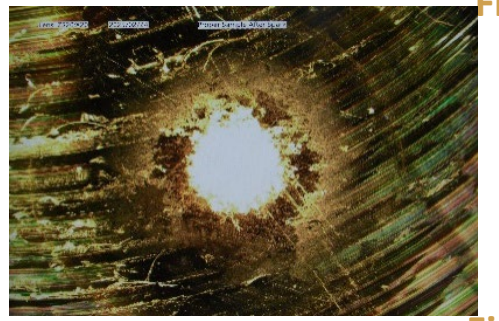
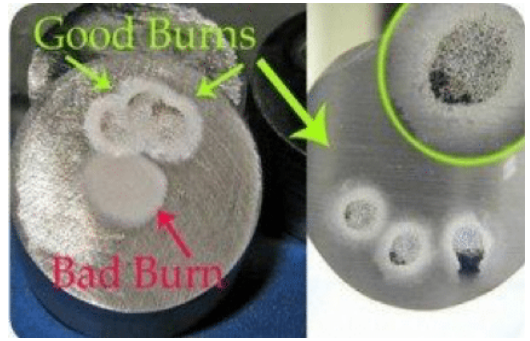


Fig-11

Fig-12

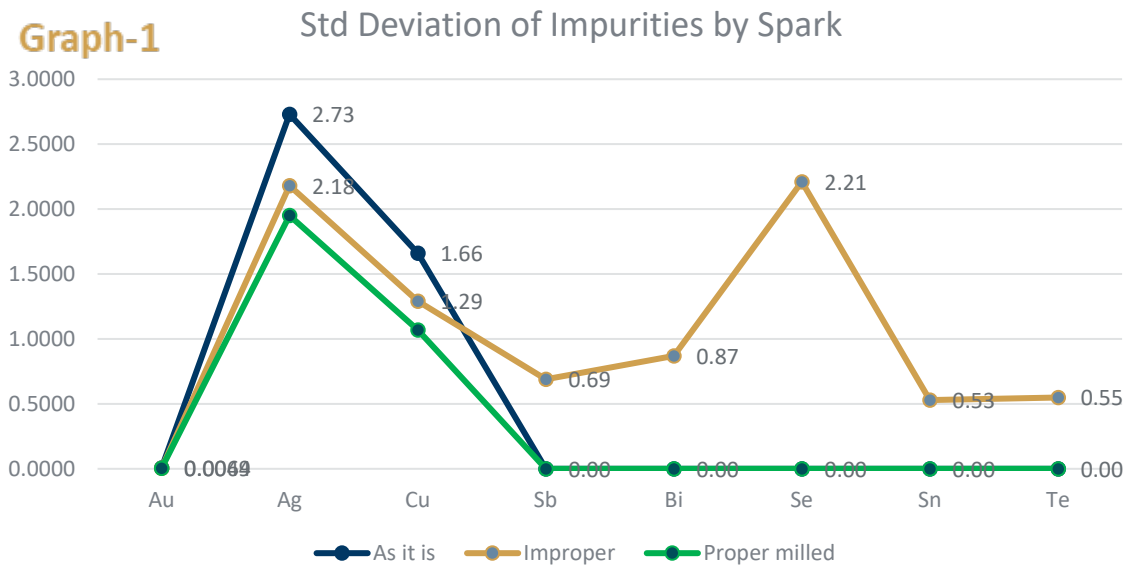


Graphical representation for differences

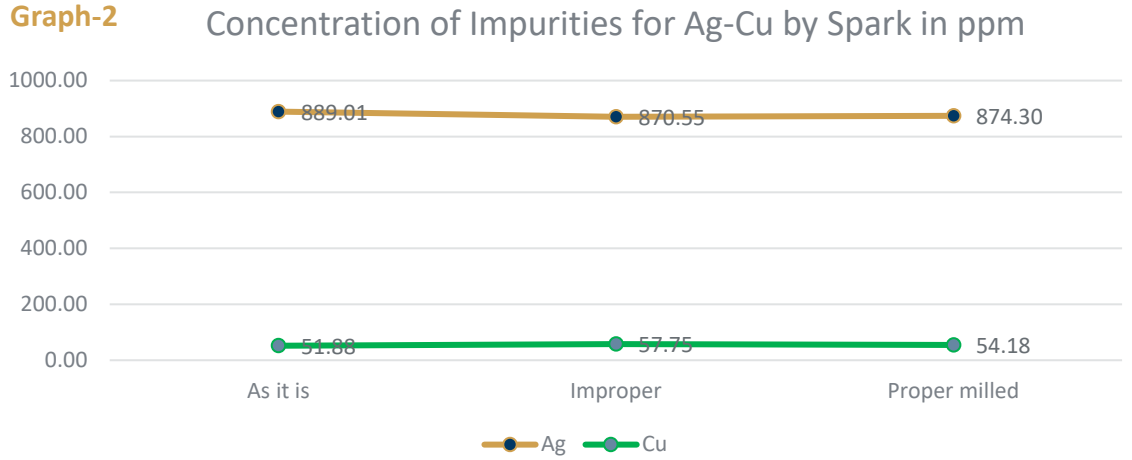
Standard Deviation for Impurities by Spark			
Elements	As it is	Improper	Proper milled
Au	0.0064	0.0049	0.0046
Ag	2.73	2.18	1.95
Cu	1.66	1.29	1.07
Sb	0.00	0.69	0.00
Bi	0.00	0.87	0.00
Se	0.00	2.21	0.00
Sn	0.00	0.53	0.00
Te	0.00	0.55	0.00

Concentration of Impurities in ppm by Spark				
Elements	Nominal	As it is	Improper	Proper milled
Ag	875	889.01	870.55	874.30
Cu	55	51.88	57.75	54.18
Au(‰)	999.07	999.053	999.034	999.073

Graph-1



Graph-2



Best practices for Sample prep for Spark OES

- The homogeneity
- Flat surface
- Smooth surface
- Surface pollution
- Physical evaluation at the time of sampling
- The best way-lathes and milling machines.

Sample Preparation- ICP OES

Sample preparation Techniques _ICP

- Crucial step

- Common sample preparation techniques include:

- Sample Digestion:
- Dilution and Filtration:
- Other Pre-concentration Methods:

- Factors Influencing Sample Preparation

- Sample Type and Matrix Composition:
- Desired Analytical Sensitivity:
- Sample Throughput and Automation:
- Safety Considerations:
- Instrument Compatibility:
- Matrix Interference: Sample Homogeneity: Contamination Control:

Pictorial presentation samples for ICP OES

Fig-1

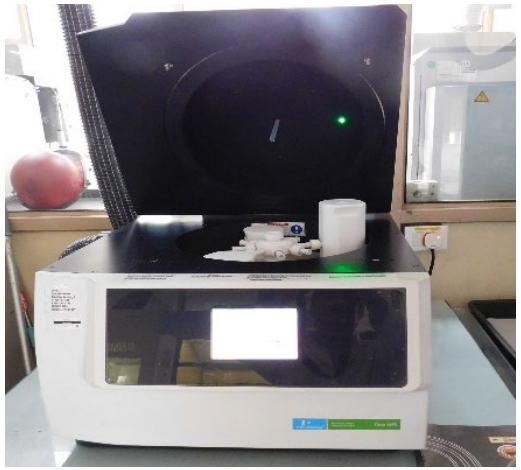
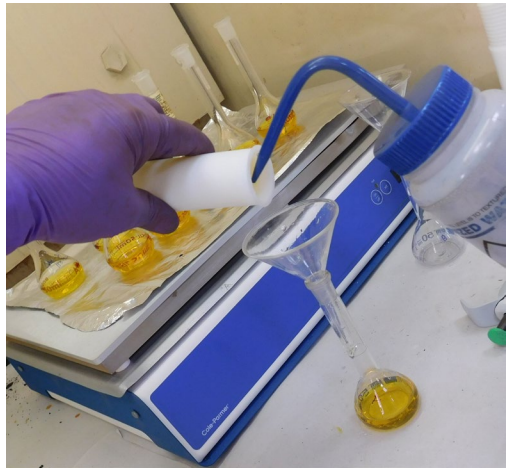


Fig-2

Fig-3



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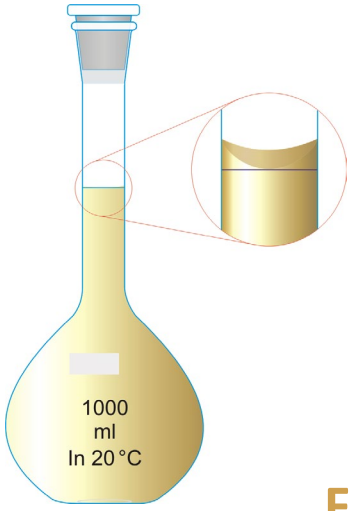

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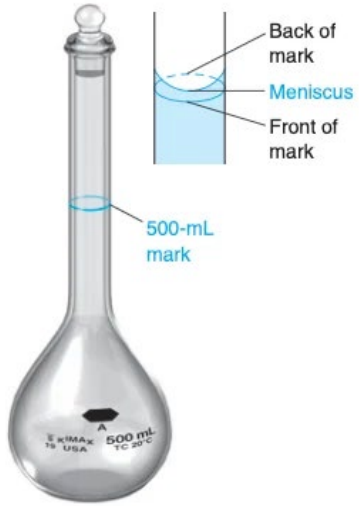

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Fig-7

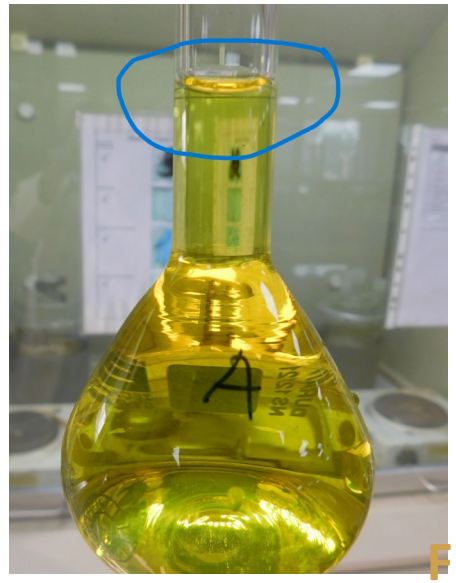
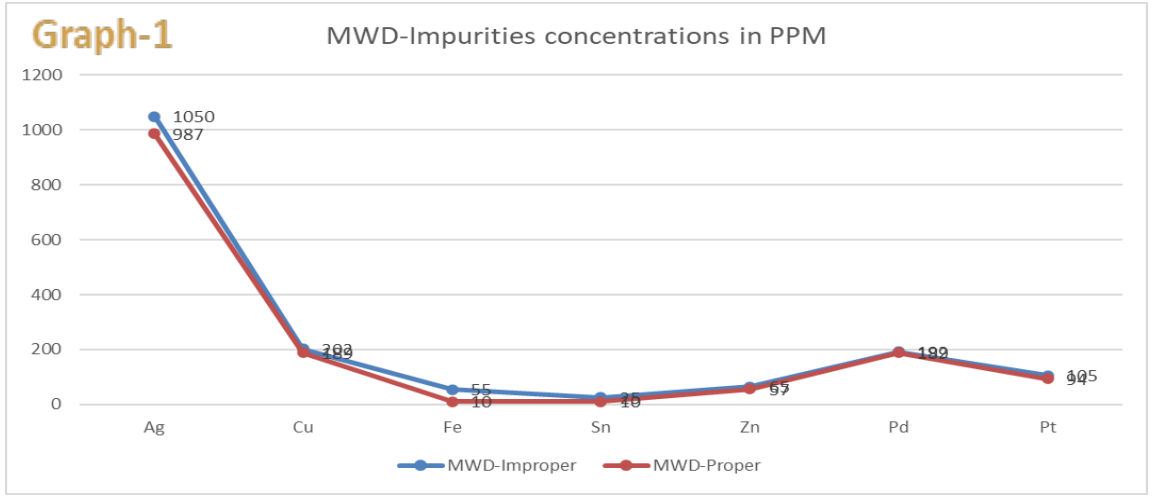


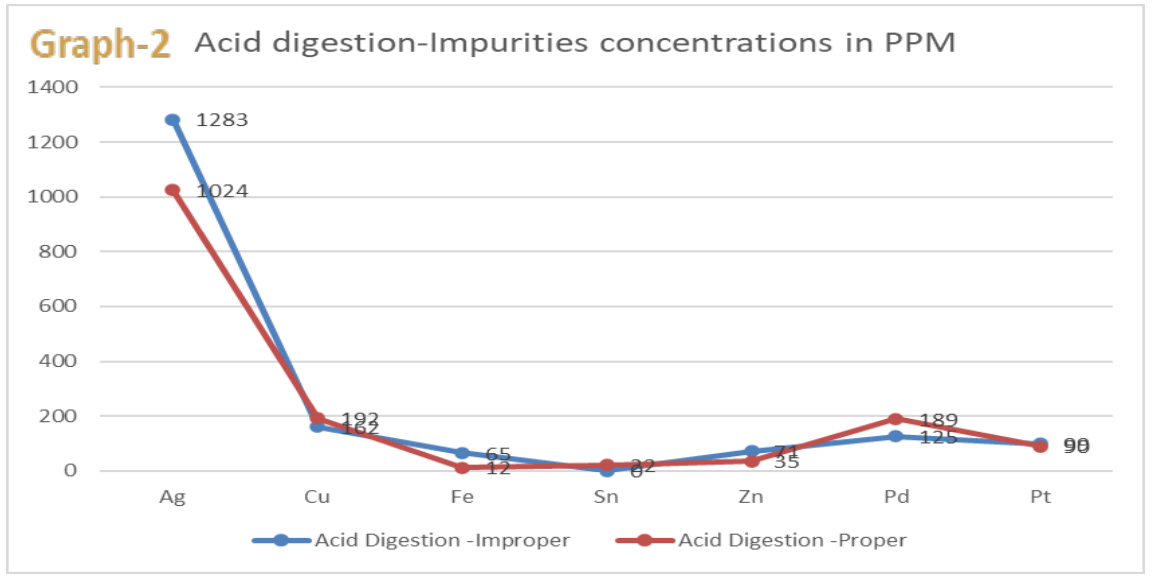
Fig-8

Graphical representation of impurities differences in ppm

Concentration of Impurities by ICP - MWD							
Sample prep method-Elements	**Ag**	**Cu**	**Fe**	**Sn**	**Zn**	**Pd**	**Pt**
MWD-Improper	1050	202	55	25	65	192	105
MWD-Proper	987	189	10	10	57	189	94
Difference(ppm)in both methods	63	13	45	15	8	3	11
Nominal Value(ppm)	1000	200	15	19	49	196	96

Concentration of Impurities by ICP-Acid Digestion							
Sample prep method-Elements	**Ag**	**Cu**	**Fe**	**Sn**	**Zn**	**Pd**	**Pt**
Acid Digestion-Improper	1283	162	65	0	71	125	99
Acid Digetion-Proper	1024	192	12	22	35	189	90
Difference(ppm)in both methods	259	-30	53	-22	36	-64	9
Nominal value(ppm)	1000	200	15	19	49	196	96





Best practices for Sample prep for ICP OES

- Critical aspect of ICP-OES analysis
- Sample integrity and control of contamination
- Handling & Storage
- Cross contamination
- Dilution & Filtration-matrix matching
- Sample homogeneity
- High-grade material and cleaning

Sample Preparation-XRF Spectrometer

Sample preparation in XRF

- Quality of the sample preparation technique

- Analysis errors of metal samples come from :

1) Internal segregation,
2) Defective surface,
3) Surface roughness and uneven surface,
4) Surface transformation,
5) Metallurgical history

- An ideal sample is prepared so that it is:

1) Representative of the material
2) Homogeneous
3) Thickness

Pictorial presentation samples for XRF Spectroscopy

Fig-1

Fig-2

Fig-3

Fig-4

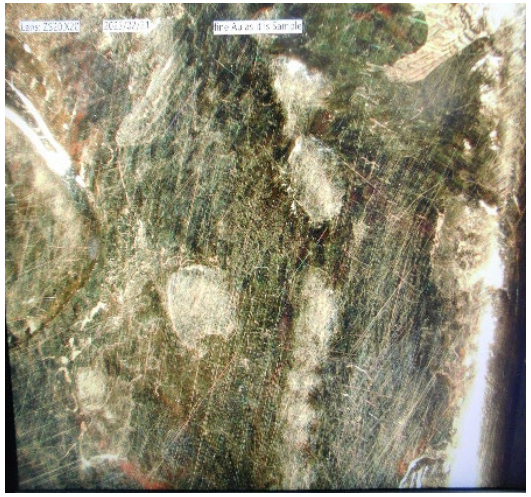


Fig-5

Fig-6

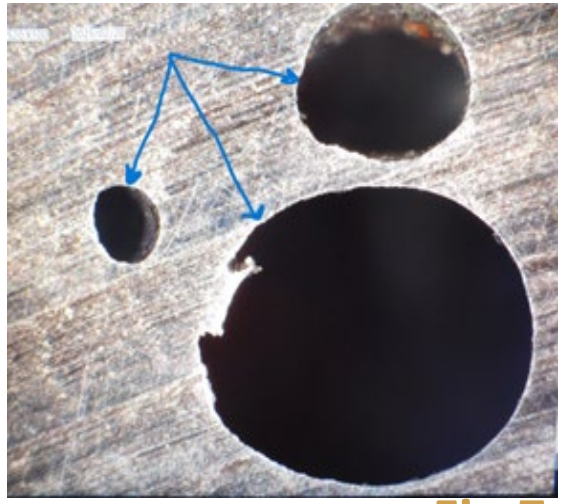


Fig-7

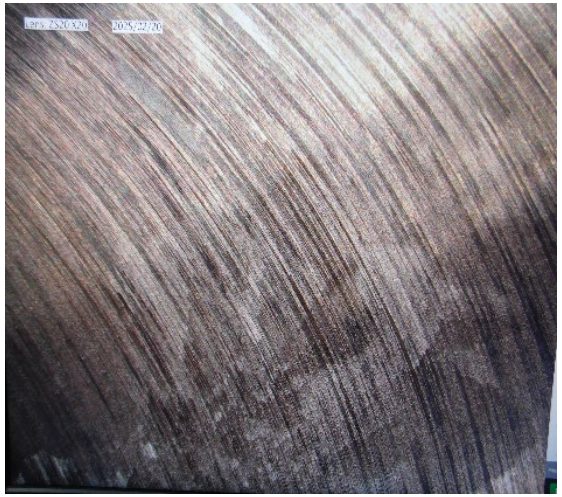


Fig-8

Why to have smooth flat surface?

- Imperfections in the surface - **shadow effect**
- Polishing striations give rise to the so-called **shielding effect**

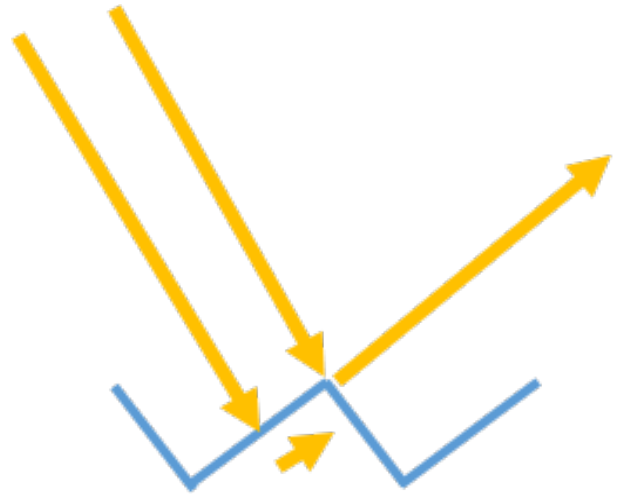


Fig.1

- **Surface Roughness**: Figure-2 shows different sample surface due to different grinding/milling conditions (A) is coarse, and(B) is fine
- X-ray intensity of (B) is stronger than (A) from the diagram

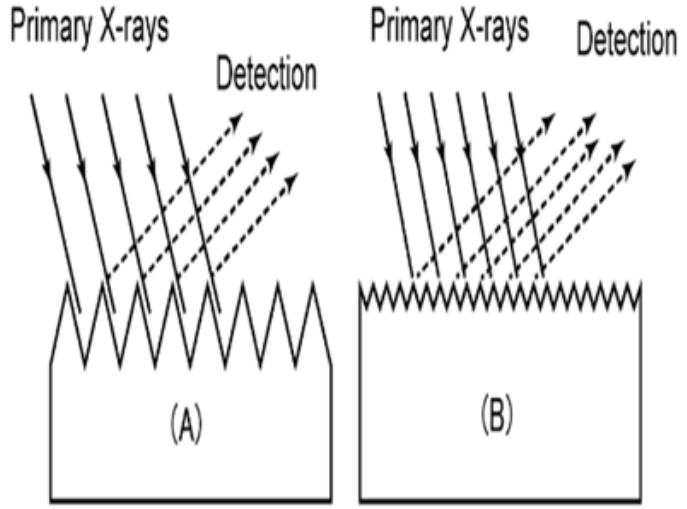
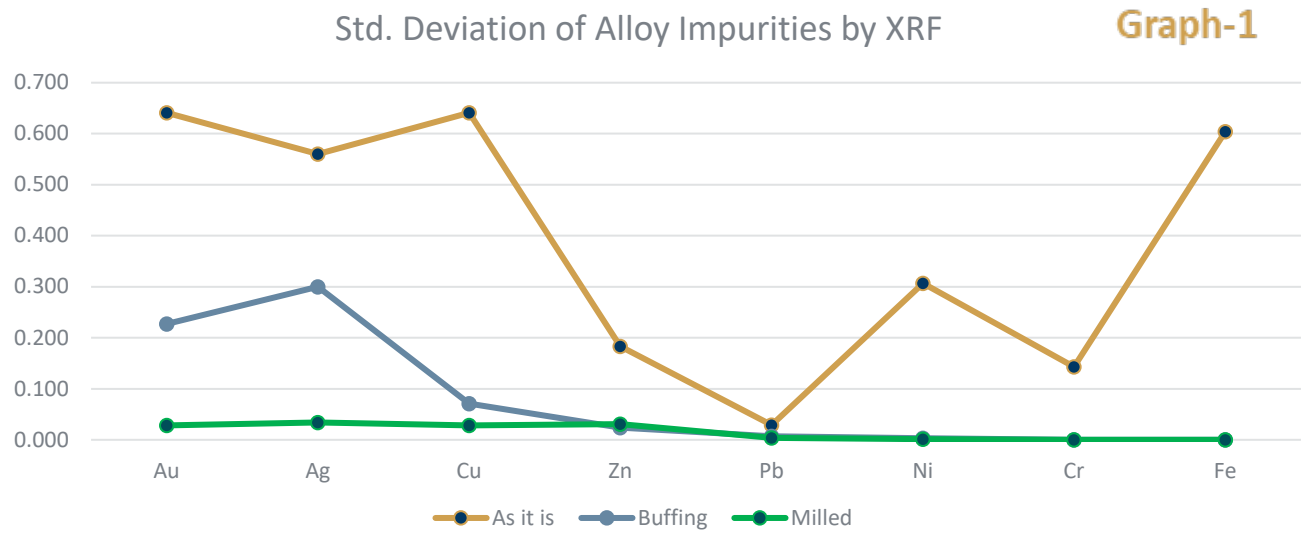


Fig.2

Graphical representation for differences

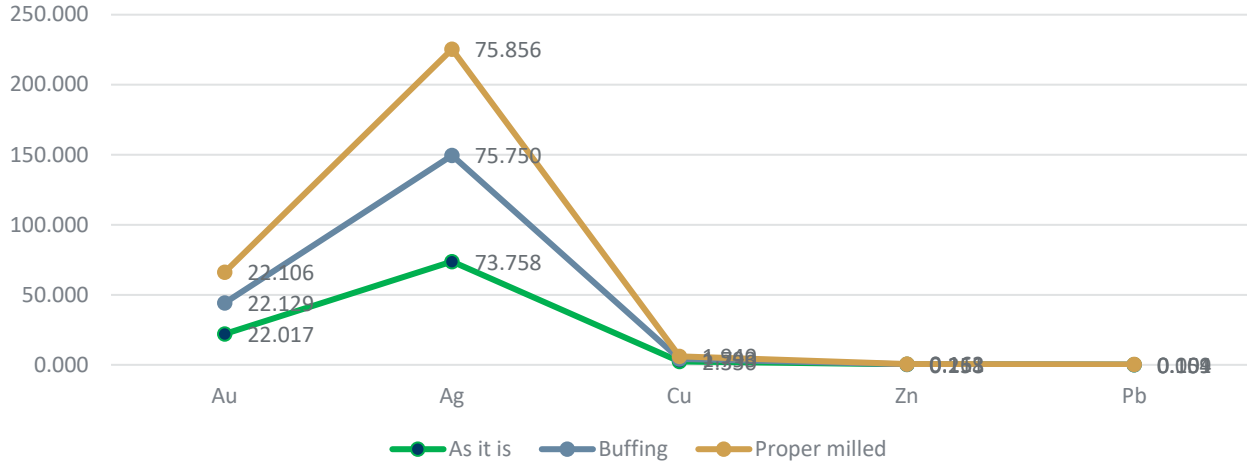
Standard Deviation for Alloy Impurities by XRF			
Element	As it is	Buffing	Milled
Au	0.641	0.227	0.028
Ag	0.560	0.300	0.034
Cu	0.641	0.071	0.028
Zn	0.183	0.024	0.031
Pb	0.029	0.007	0.004
Ni	0.307	0.003	0.001
Cr	0.143	0.000	0.000
Fe	0.604	0.000	0.000

Graph-1



Concentration Impurities in % for Alloy by XRF				
Elements	Nominal	As it is	Buffing	Milled
Au	22.09	22.017	22.129	22.106
Ag	75.60	73.758	75.750	75.586
Cu	1.96	2.330	1.793	1.940
Zn	0.15	0.258	0.113	0.161
Pb	0.10	0.054	0.101	0.109

Graph-2



Best practices for Sample prep for XRF

- Having a foreign deposit or a blot on the analysis surface is not suitable for accurate analysis.

- Surface treatment of metal sample:

1. Roughness of surface and surface grinding

2. Change of component and structure, and analysis position

3. Contamination

4. Storage of sample and time of surface renewal

Conclusion

Conclusion

- Sample preparation is a critical aspect.
- Significant impact on accuracy , precision and reliability of results
- Primary inspection is important
- Understanding and following best practices

Acknowledgment

Acknowledgment

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- Ms.Barbara Badiello, Head of Laboratory,MKS PAMP
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- Ms. Sakshi Bhagat – Instrumentation Lab-ICP OES
- Mr. Amit Solanki – Recovery and XRF Team

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- How to Prepare Metal Alloy Samples for Analysis by XRF by Kim Halkiotis

THANK YOU