## Effects of Platinum group metals (PGM) on the gold assay by the cupellation method

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### The aim of the study

• Iran is among the top 10 countries in the world in consuming gold articles



- About 5000 factories and gold articles production units operate in Iran.
- Most of manufacturers use scrap gold to produce gold articles. PGM elements are mixed with gold alloys for various reasons including economic profits while many laboratories are not still equipped with XRF method tools for assaying gold karat.
- Therefore, the present study aimed at providing a simple, commercially available, and inexpensive method for detecting the presence of Ir in gold alloys during the cupellation process for the laboratories without XRF equipment to avoid errors in gold assays.

#### How did this idea was come up with for the study(2017)?

- The gold assay laboratories were confused about the inhomogeneity of the gold articles samples they received; there was a big difference between the karat of test portions of a sample.
- **Gold factories** were concerned about the damage to production equipment such as lathes due to PGM (Platinum Group Metals) impurities in gold alloys.
- **Gold refiners** were worried about the significant reduction in the predicted weight of pure gold after extraction compared to the scrap gold and its karat. This difference was caused by PGM elements such as iridium.

#### Economic reasons for the addition of **Iridium to gold alloy**

Two reasons contributed to deliberate adding of Iridium to gold alloy:

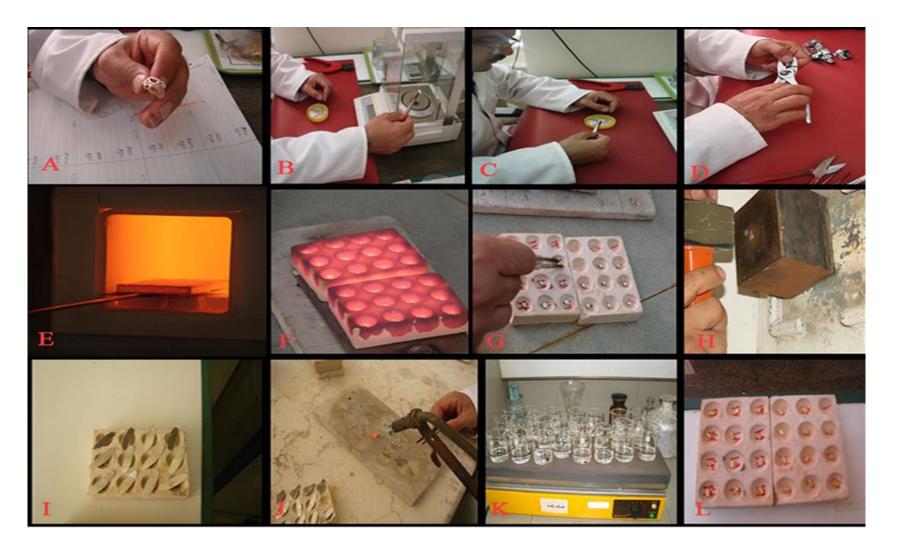
- The lower price of iridium compared to gold in March 2017.
- □Ir remains in gold after cupellation method is measured as gold karat in assaying procedure.

1,811.06 <b>-12.27 -0.67%</b>		Instrument: Gold Price, USD Spot Timescale: 1 hour						
1825 1823 1823 1823 1817 1815 1812 1810 Feb 2 <sup>2</sup> , 2023 at 17:15 NV	oz v 1811.06 v 12.27 v 0.67% 1811.06 v 12.27 v 0.67% 1811.06 v 12.27 v 0.67% oz v 1 Day v	and the second sec	stamp: :		3, 201 7.67 3.77 3.80 7.19	7 20	30:0	· · · ·
MCI 🗢 VPN 23:46	<b>@</b> 48% ■	Johnson Matthey					Q, Search	go
<sup>ate</sup> ebruary 2023	Ċ	HOME PRICES	SERVICES	ABOUT PGM	ABOUT US			A 🛛 4 Y
etal Current price (\$/troy oz)		Prices Price charts	charls					
O Platinum	928.00	Price tables				R	Pd Rn	n Ir Ru
O Palladium	1,396.00	Monthly price reports			ong Kong	London	New York	
	1,000,000	Weekly price bulletins archive		08:		09:00	09:30	15:00
Rhodium	10,750.00		Wed	1 <sup>st</sup> 700	0 700	700	700	•
Iridium	4,600.00		Thu	2 <sup>nd</sup> 700	0 700	700	700	-
C Ruthenium	465.00							

Last update : 24 Feb 2023/New York



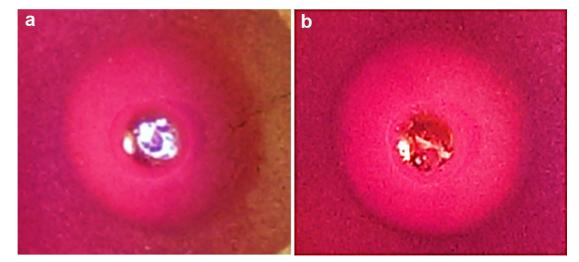
### **Cupellation method according to ISO 11426**



### **Preparing the Samples and Comparing Ir-free bead (R) and Ircontaining bead (A)**

 For the purpose of this study, one typical gold alloy (750‰ karat) without Ir and three gold alloys with different Ir concentrations (10, 12, 14) ‰ were used.

- The Ir-free beads (R) obtained from the cupellation stage were brighter in color than those containing Ir (A).
- the brightness of beads were measured by colorimeter in terms of L\* value.
- L\*R = 72.3 (higher brightness)
- $L^* A average = 65.3$  (lower brightness)

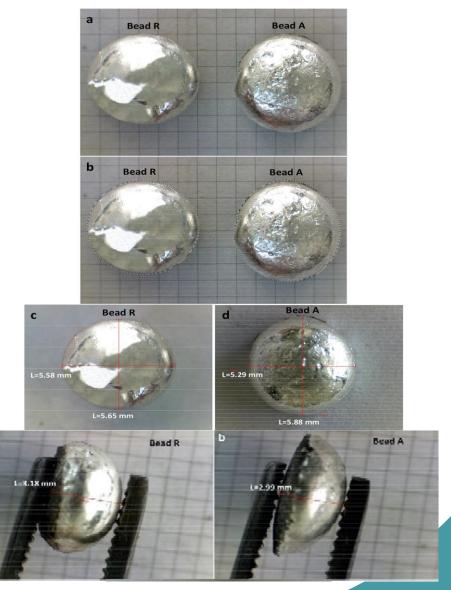


Beads obtained from sample R (a) and sample A (b)



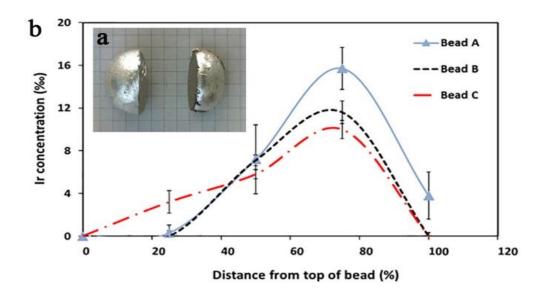
### **Comparison of Ir-free bead (R) and Ir-containing bead (A)**

- Bead A had a very coarse and rough upper surface, while bead R had a smooth one. It interpreted as an indicator of PGM such as Ir present in the gold alloy.
- Bead R had a circular shape while bead A was of an oval-like shape which led to a difference in their height. the presence of Ir in the gold matrix must have altered the melting and cooling conditions during the cupellation process, which ultimately influenced the geometric structure and shape of the bead.



## The evaluation of Ir-containing beads with different concentrations through XRF method

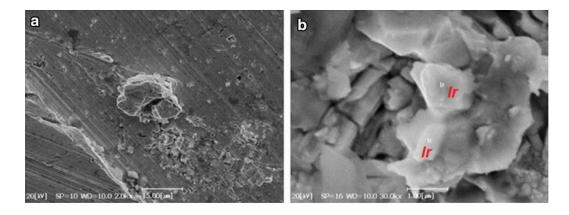
More specifically, Ir concentration in the three test portions was negligibly low at the top of the bead but increased at the 25 % of its height to the bottom. This indicates that Ir does not alloy with the other metals present in the gold alloy at the cupellation temperatures and concentrates near the bead bottom.



Vertically sectioned bead (a) and XRF results for the beads

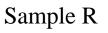
# Morphology of the beads (SEM) images from the vertical section of bead A

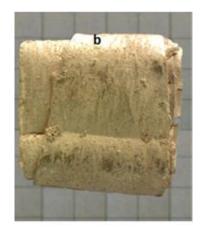
The inhomogeneous parts are formed because of the low solubility of solid Ir in gold and Ag which appear black and white floating particles on the surface of bead. It seems that the presence of Ir with a higher melting point (> 2000 °C) than those of Au (1063 °C) led to the crystallization of discrete phases of Ir within the matrix of the bead.



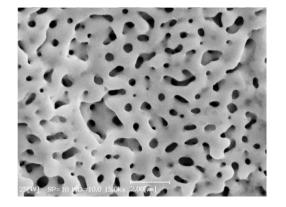
### **Comparison of SEM images of the cross section of the final cornet** after annealing







Sample A





Images of cornet of sample R (a) and sample A (b) after the parting stage.

- □ Blister-like appendages were observed on the surface of cornet A, while they were absent on cornet R because:
- 1. Ir does not react with acids so it remains in the cornet during the parting stage.
- 2. Ir cannot enter the gold matrix because of its higher melting point than gold and its low solid solubility in gold.
- Sponge-like (granular) nanostructures with a uniform granule size are clearly seen in R sample
- □ Sample A surface seems to be rougher and its shape is more irregular because Ir plates are surrounded by these spongelike structures remained in place.

### **Cupellation method for gold alloys with other platinum group metals (PGM)**

- Palladium, Platinum and all Silver are dissolved in nitric acid in the parting stage.
- Osmium is separated from gold during the melting process in the cupellation furnace.
- Most of the Ir, Ru and Rh are not separated from the gold during the cupellation test process.

## Conclusion

- Results showed that Ir-containing gold alloys were easily distinguishable with the naked eye from Ir-free ones.
- The beads obtained from the gold alloy containing Ir are matte, coarse, and full of black rough cavities with irregular oval-like shapes. However, a negligible number of such dark spots and lines were observed on the surface of Ir-free gold alloys.
- Microstructural investigations revealed that a major portion of the Ir content was concentrated as small discrete platelets at a distance of about 75% of the total height from the bead top.

## **Further Study**

- A Further study was conducted to separate Ir from gold in the final stage of cupellation method.
- The experiments can be replicated for gold alloys containing Ru and Rh to distinguish the presence or absence of these elements in gold alloys as reliable methods to report correct gold karats.

### **Thanks for your attention**

