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Determination of purity of 24 carat (5N gold) using inductive coupled plasma-optical emission spectrometer

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ABSTRACT

Hallmarking centers determine the purity of 22 carat usually by cupellation (Fire assay) method as per IS 1417. In this method they are using 24 carat gold as an additive and the purity of 24 carat gold is also certified. The determination of purity of 24 carat gold involves the estimation of impurities present in it using Inductive Coupled Plasma-Optical Emission Spectrometer (ICP-OES) quantitatively. The general impurities are Ag, Cu, Pd, Fe, Pb, Mg, As, Bi, Sn, Cr, Ni, Mn, Ru, Ir, Pt, Os, Rh, Cd, Zn and Te as per STD ASTM B- 562. Upon adding the impurities present in gold, rest is purity by difference. Based upon the results, gold can be classified into different grades which are 99.500, 99.950, 99.990 and 99.995 % fineness. This analytical technique is most important for Hallmarking centers for ascertaining their 24 carat Gold.

KEYWORDS

Purity of gold;
Determination;
ICP-OES;
Cupellation;
Carat;
Grades of gold.

INTRODUCTION

The jewelry industry still makes up for the majority of the world's total demand for gold, one area that has seen significant growth is its use in electronics, particularly within telecommunications, information technology, and safety-critical applications. The unique physical, electrical and chemical properties of gold make it a valuable material in the manufacture of semiconductor devices, including printed circuit boards, automotive electronics, and smart card technology, to name a few. Wire bonding is the method used to attach very fine gold wire (typically thinner than a human hair at 10-200 μm) from one connection pad to another, completing the electrical connection in an electronic device. Nowadays literally billions of wires are bonded every year in the world and most are used in the integrated circuits (ICs) we take for granted in all manner of electronic goods. Gold has many advantages as the material of choice for bonding wire including good corrosion resistance, high electrical conductivity, and the ability to be bonded into position in an ambient environment. It remains the most popular material for bonding wire and is specially refined to high purity (99.999% gold). For this analysis, gold wire samples have been received from a supplier that specializes in refining, manufacture, and trading of high-purity precious metals as part of their portfolio. This includes the determination of contaminants in 5N-gold wire, which, even at surprisingly low levels, can impact manufacturing processes, resulting in hard spots, embrittlement, blistering, and discoloration.

A new method that involves nebulisation ICP-OES

TABLE 1 : Calibration

S.No	Elements	Concentration (mg/L)
1	Bi, Cd, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Sn and Zn	0.05, 0.1, 0.5, 1.0 and 2.0
2	As	0.1, 0.5 and 1.0
3	Ir, Pd, Pt, Rh, Ru and Te	0.1, 0.5 and 1.0

TABLE 2 : Instrument operating conditions

S.No	Parameter	Setting
1	Spraychamber type	Double-pass, glass cyclonic
2	Nebulizer type	Sea Spray
3	Nebulizer flow	0.75 L/min
4	RF Power	1.25 kW
5	Plasma gas flow	15 L/min
6	Auxiliary gas flow	1.5 L/min
7	Uptake delay	20 s
8	Stabilization delay	10 s
9	Rinse time	15 s
10	Integration time	60 s
11	Replicates	3

analysis and accurate estimation of Ag, Cu, Pd, Fe, Pb, Mg, As, Bi, Sn, Cr, Ni, Mn, Ru, Ir, Pt, Os, Rh, Cd, Zn and Te in 24 carat gold. High resolution inductively coupled plasma mass spectrometry (HR-ICP-MS) is also useful in the study of trace elements in high purity gold[1]. Inductively coupled plasma mass spectrometry with prior matrix removal by electro deposition studies is also used in the determination of trace impurities in high purity gold[2]. The investigation of trace elements in pure gold by using ICP-AES based on matrix element reductive separation followed by spectrometric determination is also studied[3].

24 carat gold jewelry where 24 of the 24 parts are gold that is 99.995% fine gold. There is a technique called X-ray fluorescence instrumentation available to test the carat measurement of precious metals and finished jewelry without any damage. In alternative and accurate way of determination of purity of gold is the determination of impurities present in it. Generally the impurities are Ag, Cu, Pd, Fe, Pb, Mg, As, Bi, Sn, Cr, Ni, Mn, Ru, Ir, Pt, Os, Rh, Cd, Zn and Te as per STD ASTM B- 562 are to be detected using ICP-OES[4]. Nadkarni and Agarwal[5] classified gold impurities into three categories: metallic or elemental, non-metallic and radioactive. Radioactive impurities in gold, such as uranium and thorium, are important to industrial users because of the effects of small amounts of radiation on electronic components but are not generally a concern to jewelry manufacturers. Similarly, non-metallic impurities, for example oxide particles containing chromium or magnesium that cause problems when gold is formed into very thin wires or strips for industrial products. Elemental impurities, fortunately the easiest to detect, form the most important class of impurities with regard to jewelry fabrication. The impurities in other components like food and drugs have also been studied by various techniques which are compared[6].

EXPERIMENTAL

Instrumentation

Varian 720-ES

The Varian 720-ES with axial torch configuration is a truly simultaneous ICP-OES with solid-state, Charge Couple Device (CCD) detection system. The custom-designed and patented CCD detector incorporates IMAP technology, whereby pixels are arranged in continuous angled arrays matched exactly to the image produced by the echelle optics. This provides true simultaneous measurement and full wavelength coverage from 167 nm to 785 nm.

Sample preparation

Approximately 5 g of 5N-gold wire was obtained for analysis. To reduce the possibility of contamination of the sample by the wire cutters, the gold was wrapped with parafilm. After cutting, the parafilm was removed from the gold wire. To pre-clean the sections of gold wire, the piece was placed into a beaker containing 20 mL of 25% aqua regia solution for about 15 min. The sample was then rinsed with deionized water. After drying, the weight of gold piece was measured. The gold piece was placed into separate 200-mL beakers and 20 mL of aqua regia was added to each container. The beakers were heated gently on a hot plate until the gold wire dissolved completely, taking about 30 min in total. The solutions were heated for a further 15 min to reduce the volume of the solution. Once the volume of aqua regia was reduced down to about 5 mL, the beaker was removed from the hot plate and left to cool down. After equilibrating to room temperature, the gold

TABLE 3 : Results

S. No	Element	Wavelength	Correlation coefficient	24 Carat gold concentration, mg/kg
		Sample No. 1		
1	Palladium as Pd	340.458	0.999914	BD L (DL : 1 mg/kg)
2	Ruthenium as Ru	240.272	0.999753	BD L (DL : 1 mg/kg)
3	Iridium as Ir	204.419	0.999969	BD L (DL : 1 mg/kg)
4	Platinum as Pt	214.424	0.999996	BD L (DL : 1 mg/kg)
5	Rhodium as Rh	343.488	0.999960	BD L (DL : 1 mg/kg)
6	Bismuth as Bi	223.061	0.999169	BD L (DL : 1 mg/kg)
7	Lead as Pb	182.143	0.999757	8.44
8	Silver as Ag	328.1	1.0000	BD L (DL : 1 mg/kg)
9	Copper as Cu	327.395	0.999860	4.12
10	Cadmium as Cd	226.502	0.999675	BD L (DL : 1 mg/kg)
11	Nickel as Ni	227.021	0.999777	1.96
12	Zinc as Zn	213.857	0.999578	6.28
13	Iron as Fe	238.204	0.998788	3.24
14	Magnesium as Mg	279.553	0.997762	18.8
15	Chromium as Cr	205.560	0.999796	4.12
16	Manganese as Mn	259.372	0.999892	3.14
17	Arsenic as As	193.696	0.999565	BD L (DL : 1 mg/kg)
18	Tin as Sn	283.998	0.999294	1.77
19	Tellurium as Te	214.282	0.999918	BD L (DL : 1 mg/kg)
Σ Impurities (mg/kg)				51.87
% Purity of Au				99.9948
		Sample No. 2		
1	Palladium as Pd	340.458	0.999914	BD L (DL : 1 mg/kg)
2	Ruthenium as Ru	240.272	0.999753	BD L (DL : 1 mg/kg)
3	Iridium as Ir	204.419	0.999969	BD L (DL : 1 mg/kg)
4	Platinum as Pt	214.424	0.999996	BD L (DL : 1 mg/kg)
5	Rhodium as Rh	343.488	0.999960	BD L (DL : 1 mg/kg)
6	Bismuth as Bi	223.061	0.999169	BD L (DL : 1 mg/kg)
7	Lead as Pb	182.143	0.999757	2.45
8	Silver as Ag	328.1	1.0000	BD L (DL : 1 mg/kg)
9	Copper as Cu	327.395	0.999860	7.85
10	Cadmium as Cd	226.502	0.999675	BD L (DL : 1 mg/kg)
11	Nickel as Ni	227.021	0.999777	BD L (DL : 1 mg/kg)
12	Zinc as Zn	213.857	0.999578	11.1
13	Iron as Fe	238.204	0.998788	3.11
14	Magnesium as Mg	279.553	0.997762	1.96
15	Chromium as Cr	205.560	0.999796	1.96
16	Manganese as Mn	259.372	0.999892	1.15
17	Arsenic as As	193.696	0.999565	BD L (DL : 1 mg/kg)
18	Tin as Sn	283.998	0.999294	BD L (DL : 1 mg/kg)
19	Tellurium as Te	214.282	0.999918	BD L (DL : 1 mg/kg)
Σ Impurities (mg/kg)				29.58
% Purity of Au				99.9970

S. No	Element	Wavelength	Correlation coefficient	24 Carat gold concentration, mg/kg
		Sample No. 3		
1	Palladium as Pd	340.458	0.999914	BD L (DL : 1 mg/kg)
2	Ruthenium as Ru	240.272	0.999753	BD L (DL : 1 mg/kg)
3	Iridium as Ir	204.419	0.999969	BD L (DL : 1 mg/kg)
4	Platinum as Pt	214.424	0.999996	BD L (DL : 1 mg/kg)
5	Rhodium as Rh	343.488	0.999960	BD L (DL : 1 mg/kg)
6	Bismuth as Bi	223.061	0.999169	BD L (DL : 1 mg/kg)
7	Lead as Pb	182.143	0.999757	3.27
8	Silver as Ag	328.1	1.0000	BD L (DL : 1 mg/kg)
9	Copper as Cu	327.395	0.999860	14.4
10	Cadmium as Cd	226.502	0.999675	BD L (DL : 1 mg/kg)
11	Nickel as Ni	227.021	0.999777	BD L (DL : 1 mg/kg)
12	Zinc as Zn	213.857	0.999578	10.7
13	Iron as Fe	238.204	0.998788	4.15
14	Magnesium as Mg	279.553	0.997762	3.93
15	Chromium as Cr	205.560	0.999796	5.24
16	Manganese as Mn	259.372	0.999892	1.31
17	Arsenic as As	193.696	0.999565	BD L (DL : 1 mg/kg)
18	Tin as Sn	283.998	0.999294	BD L (DL : 1 mg/kg)
19	Tellurium as Te	214.282	0.999918	BD L (DL : 1 mg/kg)
Σ Impurities (mg/kg)				43.0
% Purity of Au				99.9957
		Sample No. 4		
1	Palladium as Pd	340.458	0.999914	BD L (DL : 1 mg/kg)
2	Ruthenium as Ru	240.272	0.999753	BD L (DL : 1 mg/kg)
3	Iridium as Ir	204.419	0.999969	BD L (DL : 1 mg/kg)
4	Platinum as Pt	214.424	0.999996	BD L (DL : 1 mg/kg)
5	Rhodium as Rh	343.488	0.999960	BD L (DL : 1 mg/kg)
6	Bismuth as Bi	223.061	0.999169	BD L (DL : 1 mg/kg)
7	Lead as Pb	182.143	0.999757	1.88
8	Silver as Ag	328.1	1.0000	59.0
9	Copper as Cu	327.395	0.999860	7.47
10	Cadmium as Cd	226.502	0.999675	BD L (DL : 1 mg/kg)
11	Nickel as Ni	227.021	0.999777	BD L (DL : 1 mg/kg)
12	Zinc as Zn	213.857	0.999578	4.02
13	Iron as Fe	238.204	0.998788	6.28
14	Magnesium as Mg	279.553	0.997762	4.40
15	Chromium as Cr	205.560	0.999796	BD L (DL : 1 mg/kg)
16	Manganese as Mn	259.372	0.999892	BD L (DL : 1 mg/kg)
17	Arsenic as As	193.696	0.999565	BD L (DL : 1 mg/kg)
18	Tin as Sn	283.998	0.999294	BD L (DL : 1 mg/kg)
19	Tellurium as Te	214.282	0.999918	BD L (DL : 1 mg/kg)
Σ Impurities (mg/kg)				83.05
% Purity of Au				99.9917

TABLE 3 : Results

S. No	Element	Wavelength	Correlation coefficient	24 Carat gold concentration, mg/kg
		Sample No. 5		
1	Palladium as Pd	340.458	0.999914	BD L (DL : 1 mg/kg)
2	Ruthenium as Ru	240.272	0.999753	BD L (DL : 1 mg/kg)
3	Iridium as Ir	204.419	0.999969	BD L (DL : 1 mg/kg)
4	Platinum as Pt	214.424	0.999996	BD L (DL : 1 mg/kg)
5	Rhodium as Rh	343.488	0.999960	BD L (DL : 1 mg/kg)
6	Bismuth as Bi	223.061	0.999169	BD L (DL : 1 mg/kg)
7	Lead as Pb	182.143	0.999757	BD L (DL : 1 mg/kg)
8	Silver as Ag	328.1	1.0000	39.0
9	Copper as Cu	327.395	0.999860	9.52
10	Cadmium as Cd	226.502	0.999675	BD L (DL : 1 mg/kg)
11	Nickel as Ni	227.021	0.999777	BD L (DL : 1 mg/kg)
12	Zinc as Zn	213.857	0.999578	2.97
13	Iron as Fe	238.204	0.998788	10.8
14	Magnesium as Mg	279.553	0.997762	21.9
15	Chromium as Cr	205.560	0.999796	BD L (DL : 1 mg/kg)
16	Manganese as Mn	259.372	0.999892	BD L (DL : 1 mg/kg)
17	Arsenic as As	193.696	0.999565	BD L (DL : 1 mg/kg)
18	Tin as Sn	283.998	0.999294	BD L (DL : 1 mg/kg)
19	Tellurium as Te	214.282	0.999918	BD L (DL : 1 mg/kg)
Σ Impurities (mg/kg)				84.19
% Purity of Au				99.9916

solutions were quantitatively transferred into 100-mL volumetric flasks and diluted to the mark with deionized water. In 5% aqua regia, the gold digest solutions will have total dissolved solids (TDS) content of around 1% W/V. A preparation blank solution was also prepared with the samples. All samples were diluted to 100 mL in plastic disposable tubes and filtered with 2 micron teflon filter. This high dirt trapping filter is especially suitable for trace level analysis. The filtered samples were transferred directly to the ICP-OES.

Calibration solution preparation

The calibration summary is listed in TABLE 1.

Conditions

Instrument operating conditions are shown in TABLE 2.

RESULTS AND DISCUSSION

The results are summarized in TABLE 3 which lists the average concentration values of trace metals determined from triplicate measurements for the sample, after weight and volume corrections. The total average concentration for each analyte and corresponding correlation coefficient are also shown.

The resulting total trace metal impurities (Σ Impurities) found in the 5N-gold samples and the calculated percentage (%) purity has been calculated. On the basis of the ICP-OES and AAS results the purity of this 5N gold wire is found to be greater than 99.99%. From the above results it was observed that the platinum group metals (Pd, Ru, Ir, Pt and Rh) were found to be almost nil. Lead was found to be around 8 mg/kg in the first sample and the remaining samples it was less than 5 mg/kg. Silver was found to be the greatest by considering all other elements, and in the 4th sample it was 59.0 mg/kg analysed by using Atomic Absorption Spectrophotometer. By considering all the samples copper was found to be below 15 mg/kg. Cadmium and nickel were present in some of the samples below 2 mg/kg and they were not found in other samples. Zinc was found to be changed from 3 to 12 mg/kg by comparing the above samples. Iron was maximum for the 5th sample (around 11 mg/kg). Mg was maximum for the 1th sample (around 19 mg/kg). Chromium ranged from 0 to 5 mg/kg. Manganese ranged from 0 to 3 mg/kg. Arsenic, tin and technetium were present well below the limit.

Various grades of gold in respective of its fineness are classified as,

Grade 99.500: gold having a minimum fineness of 995.00

Grade 99.950: gold having a minimum fineness of 999.50

Grade 99.990: gold having a minimum fineness of 999.90

Grade 99.995: gold having a minimum fineness of 999.95

The above results indicate that the 5N i.e 24 carat gold sample numbers 1, 2 and 3 come under the grade of 99.995 having the minimum fineness of 999.95 and sample numbers 4 and 5 come under the grade of 99.990 having the minimum fineness of 999.90.

CONCLUSIONS

The Varian 720-ES and AA 240 have been successfully used for the determination of trace impurities in pure 5N-gold. This work demonstrates the benefits of high sensitivity and to measure gold purity to five-nines (5N). In fact, routinely achieving detection limits to determine contaminates in gold of six-nines (6N) purity is not far out of reach and would only require slightly cleaner working conditions.

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REFERENCES

[1] H.Xie, K.Huang, X.Nie, L.Fu; J.Wuhan Uni.Technol- Mater.Sci.Ed., **24**, 608 **(2010)**.

[2] Y.C.Sun, C.H.Hsieh, T.S.Lin, J.C.Wen; Spectrochim.Acta Part B: Atomic Spectroscopy, **55**, 481 **(2000)**.

[3] I.Karadjova, S.Arpadjan, L.Jordanova; Fresenius J.Anal.Chem., **367**, 146 **(2000)**.

[4] American Society for Testing and Materials (ASTM); Standard Specification for Refined Gold Designation: B 562-95 **(2005)**.

[5] R.M.Nadkarni, D.P.Agarwal; Int.Precious Metals Inst., Allentown, PA, 283 **(1988)**.

[6] D.R.Abernethy, A.J.DeStefano, T.L.Cecil, K.Zaidi, R.L.Williams; Pharm.Res., **27**, 750 **(2010)**.