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QUANTITATIVE DETERMINATION BY THE STALLWOOD AIR-JET DC ARC TECHNIQUE

3. TRACE IMPURITIES IN HIGH PURITY GOLD

by

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SUMMARY OF RESULTS

By a combination of the Jaycox Dry Powder - dc Arc Method (ASTM E-2 SM 6-1) and the normal Stallwood Air-Jet dc Arc Method, trace impurities in two Mint samples of high purity gold were determined.

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INTRODUCTION

The purpose of the investigation was to determine the purity of gold, supposedly 99.98% pure, obtained from the Royal Canadian Mint.

OUTLINE OF METHOD

The sample was first analysed by the Stallwood semi-quantitative method to determine what impurities were present. The amount of the impurities found was then determined by a combination of the Jaycox dry powder - dc arc method (1) and the Stallwood air-jet dc arc method (2, 3, 4).

STANDARD SOLUTIONS

0.0806 g/ml standard gold solution: prepared by dissolving a 4.118 g Specpure gold rod, previously washed in hot hydrochloric acid, in aqua regia and making the volume up to 50 ml.

0.2 g/100 ml solutions of iron, magnesium, lead, copper and aluminium: prepared by dissolving the appropriate amount of Specpure Fe, MgO, Pb, CuO and Al in nitric acid and making the volume up to 100 ml.

0.02 g/100 ml solutions of bismuth, cadmium, silver and manganese: prepared as above from Specpure Bi_2O_3, CdO, $AgNO_3$ and MnO_2. In the case of manganese and aluminium a little hydrochloric acid had to be added.

1 g/100 ml solution of silicon: prepared by dissolving the appropriate amount of $Na_2SiO_3.9H_2O$ in 100 ml distilled water.

Stock solution A: containing 0.1 mg/ml Fe, Mg, Pb, Cu and Al plus 0.01 mg/ml Bi, Cd and Mn prepared by adding 5 ml of each of the standard solutions to a 100 ml volumetric flask and making the volume up to 100 ml.

Stock solution B: containing 0.1 mg/ml silicon and 0.01 mg/ml silver prepared by adding 5 ml Ag standard solution and 1 ml of silicon standard solution to a 100 ml volumetric flask and making the volume up to 100 ml.

PREPARATION OF STANDARDS

5 ml of the gold solution was introduced to a 25 ml porcelain casserole together with varying amounts of stock solutions A and B. The solutions were evaporated to dryness and baked in the oven at 400°C for half an hour. The gold residue was then scraped into an agate mortar and ground for 15 min.

SAMPLE PREPARATION

0.0806 g of gold was dissolved in aqua regia and the volume made up to 10 ml. 5 ml of the solution were introduced into a 5 ml porcelain casserole. It was then treated the same way as the standards. For details see Table 1.

TABLE 1

Preparation of Standards

	Stock Solution A			Stock Solution B		
Std. No.	Vol. added	%Fe, Mg, Pb, Cu	%Bi, Cd, Mn	Vol. added	%Si	%Ag
0	0	0	0	0	0	0
1	0.01	0.00025	0.000025	0.02	0.0005	0.00005
2	0.02	0.00046	0.000046	0.05	0.00125	0.000125
3	0.06	0.0015	0.00015	0.2	0.005	0.0005
4	0.2	0.00465	0.000465	0.5	0.0125	0.00125
5	0.6	0.0015	0.0015	2	0.05	0.005
6	2	0.0465	0.00465	5	0.125	0.0125

SPECTROGRAPHIC PROCEDURE

A modification of the Stallwood procedure was used.

20 mg of the sample or standard was mixed with 30 mg high purity graphite and loaded into an electrode cup 7/8 in. deep and with 1/8 in. diameter. Each standard and sample was exposed in duplicate using the following excitation conditions:

Spectrograph:	3 metre Baird grating spectrograph equipped with an air jet.
Wavelength range:	2300 - 3600 A
Slit:	25 μ
Sector:	3 step rotating sector with 1-10-100% transmission steps
Air jet pressure:	5 in. of mercury
Electrode system anode: (lower)	1/8 in. diameter high purity graphite cup
cathode: (upper)	1/8 in. diameter high purity graphite cone.
Electrode gap:	2.5 mm
Emulsion:	S.A. 2
Excitation:	6 amps dc arc
Exposure:	sample burned to completion

PHOTOMETRY

The following spectral lines were used for the determination of the metals.

Element	Spectral Line	Element	Spectral Line
Iron	3020.640	Manganese	2576.107
Copper	3273.962	Cadmium	2288.018
Magnesium	2802.692	Bismuth	3067.716
Aluminium	3092.713	Silicon	2516.123
Lead	2833.069	Silver	3280.683

The Sturrock Cathode Ray Densitometer was used to determine the intensities of the lines. Calibration curves were prepared by plotting the intensities of the lines against the percentage of metal in the standards. In each case the intensity of the blank was subtracted from the intensity of the respective standards. On using log-log graph paper straight lines were obtained for the calibration curves of all the elements.

RESULTS

The results of the determinations are tabulated in Table 2.

TABLE 2

Results

Sample No.	Mn %	Mg %	Fe %	Al %	Cu %	Ag %	Pb %	Cd %	Bi %	Si %	Au by difference
1	0.0002	0.0006	0.001	0.004	0.0003	0.006	Tr? <0.002	N.D.	N.D.	0.008	99.98
2	0.0002	0.0004	0.001	0.0004	0.00025	0.006	Tr <0.002	N.D.	N.D.	0.001	99.99

N.D. = not detected

REFERENCES

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4. R.F. Sturrock and F.A. Lang, "Semi-Quantitative and Quantitative Spectrographic Analysis with the Stallwood Air-Jet Source", 1st Ottawa Symposium on Applied Spectroscopy, Mines Branch, Ottawa, September 20, 1954.

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