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A study of the trace elements associated with gold from the Barberton District

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is intimately related to the surrounding minerals. In addition, minute inclusions of impurities seem to be constantly present in the actual crystals themselves. These observations agree with those of Poole (personal communication), who has also been studying the mineragraphy of Barberton gold, and of Crook (1939).

The ubiquitous presence of inclusions in gold must be considered when discussing the mode of occurrence of trace elements in native gold. Whereas the larger inclusions and adhering particles of quartz and sulphides can be removed from the gold grains by treatment with acids, it seems unlikely that any method can be devised to remove completely those minute inclusions which occur in the gold crystals, or possibly, even in the actual gold lattice. Therefore, while arguments can be advanced to suggest that certain trace elements occur in solid solution with gold, the possibility of them occurring as mineral inclusions must be taken into consideration. However, it is considered that the cleaning process devised was sufficiently effective to ensure a negligible amount of contamination by the inclusions.

(c) <u>The extraction of the gold from the samples</u>

The extraction of the gold had to be carried out in such a way that a maximum amount was obtained with a minimum amount of contamination by extraneous material. Chemical methods which might affect the trace element composition

..22/...

of the gold had also to be avoided. The normal methods of extraction of gold from the host rocks, such as amalgamation and cyanidation were considered but were not used for the following reasons. In amalgamation, the mercury dissolves the gold but, according to Seidell (1940), will also dissolve small amounts of silver, copper, and other base metals. Thus extraction by amalgamation would tend to have a refining effect on the gold.

According to Rose and Newman (1937), cyanide solutions will dissolve gold and silver and some of the other base metals which may be associated with the gold. Copper and zinc are readily dissolved by cyanide solutions; iron, lead and tellurium are much less soluble, while aluminium, arsenic and platinum are insoluble. In addition, the cyanide solutions may attack partly decomposed sulphides and certain minerals such as pyrrhotite, arsenopyrite and stibnite affect the dissolution process. Therefore, in view of the possible alteration of the composition of the gold during extraction by cyanidation or amalgamation, it was decided not to use them.

Sampling in polished section using a drill as described by Haycock (1931) was considered and rejected because of the possibility of contamination by the drill material. Moreover, the amounts of material which can be extracted from a polished section is limited and need not necessarily be representative of the gold in the rock.

The method decided upon was the following. To extract the

..23/...

maximum amount of gold the G samples were crushed in an iron percussion mortar of the type described by Wager and Brown (1960) to between -60 + 120 mesh (Tyler mesh sizes). The use of an iron mortar limited contamination to surface iron on the gold. Nylon mesh was used for sieving the samples. As soon as the larger particles of gold were liberated from the gangue material, they were removed from the mortar to prevent any gangue minerals being hammered into them. It was found that only a very small amount of gold passed through into the -120 mesh fraction.

The gold was then concentrated and extracted by panning on a superpanner. Only in those cases where very little gold was obtained from the +120 fraction was the -120 fraction also treated on the superpanner.

The extraction of the grains of gold from the S samples could not, however, be carried out as described above, because of the much larger size of the gold grains. It seemed obvious that if these samples were crushed, considerable contamination by hammering gangue material into the gold and wrapping gold around impurities, would occur. To overcome this, the gold was liberated by dissolving the remainder of the rock in hydrofluoric acid. This acid attacks quartz and the silicate minerals readily but has little effect on the noble metals and metallic sulphides (see table VII). To test the effect of hydrofluoric acid on the base metals present in gold, a gold alloy

..24/...

known to contain aluminium, antimony, lead, copper, iron, magnesium, manganese, nickel, silicon, tin and zinc, was placed in a 50% solution of hydrofluoric acid and heated at 60°C on a water bath for 4 weeks. After this treatment, the alloy was spectrographically analyzed and found to have the same qualitative trace element composition.

Before placing them in the acid, the samples were broken into pieces of half an inch to an inch in diameter by wrapping the rock in paper and striking it two or three times with a hammer. The fragmented samples were placed in polythene beakers which were then filled with a 1:1 mixture of 66% hydrofluoric acid and distilled water. The beakers were heated in a water bath at approximately 60°C until dissolution was complete. The time required to dissolve the rocks varied from one to two weeks, depending on the size of the sample. The gold-sulphide concentrate remaining after dissolution was washed thoroughly in running water for half an hour and then by decantation with distilled water and dried. The gold was sorted out from the sulphides by hand using a binocular microscope.

(d) The cleansing of the gold particles

Examination of the gold concentrates under a binocular microscope, showed that most of the gold grains had particles of sulphides, oxides and quartz adhering to them. This was especially the case for the larger grains from the S samples which seemed to enclose completely, sulphide crystals. Occasionally the gold had a dull, reddish surface due to a coating of iron oxide.

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Both Crook (1939) and Fer'yanchich (1959) have discussed the removal of these surface impurities adhering to the gold. Crook cleaned the gold particles by heating them in a platinum crucible in a solution of hydrochloric and hydrofluoric acids for 4 to 8 hours. Fer'yanchich used 6N hydrochloric acid to clean gold nuggets.

Following Crook's method, the gold from the samples was placed in a mixture of equal parts of concentrated hydrochloric and hydrofluoric acids and distilled water and heated in a water bath for 24 hours at 60°C. Crook's recommended time was found to be totally inadequate. This treatment removed all the silicates and some of the oxides and sulphides.

The majority of the sulphides, such as pyrite and arsenopyrite, are not attacked by hydrochloric acid but are readily soluble in nitric acid. However, this latter acid is also liable to attack silver and other metals alloyed with the gold. In the process of parting, which involves the removal of silver from a silver-gold alloy using nitric acid, the silver must predominate in the alloy and for complete parting, the ratio of silver to gold should be 5:2. However, if gold predominates in the alloy, only silver on or near the surface will be dissolved. Therefore, in native gold samples containing approximately 10% silver, the loss of silver due to parting by nitric acid, should be very small. Similarly, the loss of base metals in the gold should be negligible.

..26/...

To test the above premises as regards the attack of nitric acid on native gold alloys, portions of an alloy containing 42.5% silver were cut up into small pieces to increase the surface area and were then heated in 20 cc of boiling concentrated nitric acid, for 10 minutes. The amount of silver lost was determined by weighing the samples before and after parting. Tests were carried out on 15 different portions of the alloy and the loss was found to vary with the size of the sample and the surface area. The maximum amount of silver lost was 1.96% on a sample weighing 450 mgm. Only in two other cases was the loss greater than 1% and the average loss for the remaining samples was 0.6%. These results would seem to justify the above premises that the loss of trace elements and silver from the native gold would be negligible during the removal of the sulphides from the gold surface by nitric acid.

The majority of the samples were, therefore, treated with a boiling 1:1 nitric acid solution for 10 minutes. The acid was then poured off and the samples washed by decantation with distilled water. This treatment removed most of the remaining impurities and those grains which were still contaminated by adhering material as seen under the binocular microscope were discarded.

..27/...

The larger grains in some of the S samples, however, enclosed too many crystals of sulphides to be removed by nitric acid. In these cases, the grains were cut open using a stainless steel scalpel to expose the sulphides, which were removed mechanically, if possible. For the cleansing of these samples, a process of pressure oxidation of the sulphides was devised. The samples were placed in an autoclave at 150°C with a small amount of distilled water. Oxygen under pressure was allowed to pass through the autoclave for 24 hours. The total pressure in the autoclave was 200 lbs/sq. inch and the oxygen partial pressure 142.95 lbs/sq. inch. After removal from the autoclave, the coating of oxides remaining on the gold grains was removed with dilute hydrochloric acid.

Table VII was compiled to show the effect of the three acids used in the cleansing process on gold and some of its trace constituents. Unfortunately, much of the data on solubilities are contradictory and the table reflects the generally accepted solubilities as given in the references consulted. The solubilities given are for the native elements only and do not necessarily hold for alloy constituents. This is especially true in the case of the trace constituents, which may be protected from the acid attack by the gold. The solubility varies with the strength of the acid, the temperature of the solution and the physical state and purity of the metal. The results quoted in the table are for 1:1 mixtures of the acids and distilled water at a temperature of approximately 60°C.

..28/...

Table VII Solubilities of certain native elements in hydrochloric, hydrofluoric and nitric acids.

Element	HCl		HF		HNO_3	
	A	B	A	B	A	B
Aluminium	s	s	i	s	i	i
Antimony	i	i	i	i	i	i
Arsenic	i	i	i	?	s	i
Bismuth	sl	i	i	?	s	s
Cadmium	s	s	?	?	s	s
Calcium	s	s	?	?	s	s
Cobalt	s	s	?	?	s	s
Copper	sl	sl	i	sl	s	s
Gold	i	i	i	?	i	i
Iron	s	s	?	s	s	s
Lead	i	i	i	i	s	s
Magnesium	s	?	i	i	s	s
Manganese	s	s	?	s	s	s
Mercury	i	i	i	i	i	i
Molybdenum	sl	i	i	i	s	s
Nickel	sl	sl	?	?	s	s
Palladium	sl	sl	?	?	s	sl
Platinum	i	i	i	i	i	i
Silicon	i	i	s	s	i	i
Silver	i	i	i	?	s	s
Tellurium	i	i	i	?	s	s
Tin	d	sl	?	?	d	s
Titanium	s	s	?	s	s	s
Vanadium	i	i	s	sl	s	s
Zinc	s	s	?	s	s	s

N.B. s - soluble; sl - slightly soluble;
i - insoluble; d - decomposes;
? - uncertain due to lack of information.

A - solubilities from Hodgman (1954)

B - solubilities from Comey and Hahn (1921).

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(e) Homogenization of the samples

To obtain reproducible results for each sample, it was decided to melt the individual grains in the sample together to form one homogeneous bead. Before melting the grains, however, the samples were carefully re-examined under the binocular microscope to ensure the purity of the samples. The method used to form the beads was similar to that described by Esterhuizen (1962). The grains were placed in a large cup graphite electrode and an arc was struck on the edge of the electrode crater and allowed to wander for a time interval just sufficient to melt the grains. To avoid significant volatilization from the gold, the arc was not struck on the grains themselves. Fig. 13, page 74, shows the arrangement of the electrodes during the bead formation. The following table gives the arcing conditions used.

Spectrograph: A.R.L. grating spectrograph.
Excitation: Anode excitation, d.c. arc, 220 volts, 4 amps.
Electrode gap: 4 mm.
Slit width: 10 microns.
Wavelength region: 3250 - 4400 Angstrom units.
Emulsion: Kodak spectrum analysis No. 1 film.
Time: 5 - 8 seconds, depending on the size of the sample.

As is shown in table VIII, which summarizes the results of all the lines emitted during the bead formation, silver and copper were

..../29...

Table VIII. The spectral lines emitted during the homogenization of the G and S samples of native gold.

Line:	Silver			Copper		Iron		Others
	3280	3383	4055	3248	3274	3440	3441	
Intensity:	2000	1000	800	5000	3000	500	300	
G 1	W	W	-	VW	VW	-	-	Zn 3345 - S
G 2	W	W	-	W	W	-	-	
G 3	W	W	-	-	-	-	-	
G 4	W	W	VW	W	W	-	-	
G 5	VW	VW	-	-	-	-	-	
G 6	tr	tr	-	-	-	-	-	
G 7	M	M	-	W	W	-	-	
G 8	VW	VW	-	-	-	-	-	
G 9	W	W	-	VW	VW	-	-	
G 10	M	M	VW	W	W	VW	VW	
G 11	tr	tr	-	-	-	-	-	
G 12	VW	VW	-	VW	VW	-	-	
G 13	tr	tr	-	-	-	-	-	
G 14	VW	VW	-	-	-	-	-	
G 15	VW	VW	-	-	-	-	-	
G 16	M	M	VW	W	W	S	S	
G 17	M	M	VW	W	W	S	S	
G 18	W	W	-	VW	VW	-	-	
G 19	VW	VW	-	-	-	-	-	
G 20	VW	VW	-	-	-	-	-	
G 21	VW	VW	-	-	-	-	-	
G 22	W	W	-	VW	VW	-	-	

Table VIII. The spectral lines emitted during the homogenization of the G and S samples of native gold.

	Silver			Copper		Iron		Others
Line:	3280	3383	4055	3248	3274	3440	3441	
Intensity:	2000	7000	800	5000	3000	500	300	
G 1	W	W	-	VW	VW	-	-	Zn 3345 - S
G 2	W	W	-	W	W	-	-	
G 3	W	W	-	-	-	-	-	
G 4	W	W	VW	W	W	-	-	
G 5	VW	VW	-	-	-	-	-	
G 6	tr	tr	-	-	-	-	-	
G 7	M	M	-	W	W	-	-	
G 8	VW	VW	-	-	-	-	-	
G 9	W	W	-	VW	VW	-	-	
G 10	M	M	VW	W	W	VW	VW	
G 11	tr	tr	-	-	-	-	-	
G 12	VW	VW	-	VW	VW	-	-	
G 13	tr	tr	-	-	-	-	-	
G 14	VW	VW	-	-	-	-	-	
G 15	VW	VW	-	-	-	-	-	
G 16	M	M	VW	W	W	S	S	
G 17	M	M	VW	W	W	S	S	
G 18	W	W	-	VW	VW	-	-	
G 19	VW	VW	-	-	-	-	-	
G 20	VW	VW	-	-	-	-	-	
G 21	VW	VW	-	-	-	-	-	
G 22	W	W	-	VW	VW	-	-	

Table VIII, continued.

Line:	Silver			Copper		Iron		Others
	3280	3383	4055	3248	3274	3440	3441	
Intensity:	2000	1000	800	5000	3000	500	300	
G 23	W	W	-	W	W	-	-	
G 24	W	W	-	VW	VW	-	-	
G 25	W	W	-	VW	VW	-	-	
G 26	M	M	-	W	W	-	-	
G 27	W	W	-	W	W	-	-	
G 28	W	W	-	W	W	-	-	
G 29	W	W	VW	W	W	-	-	
G 30	W	W	-	W	W	-	-	
G 31	W	W	-	VW	VW	-	-	
G 32	M	M	VW	W	W	-	-	
G 33	W	W	-	VW	VW	-	-	
G 34	W	W	-	M	M	-	-	
G 35	W	W	-	VW	VW	-	-	
G 36	M	M	-	W	W	W	W	
G 37	W	W	-	VW	VW	-	-	
G 38	W	W	-	-	-	-	-	
G 39	W	W	-	W	W	W	W	Sn 3263 - M
G 40	VW	VW	-	W	W	-	-	
G 41	VW	VW	-	VW	VW	-	-	
G 42	W	W	-	W	W	-	-	
G 43	W	W	-	VW	VW	-	-	
G 44	W	W	-	VW	VW	-	-	
G 45	W	W	-	VW	VW	-	-	
G 46	W	W	-	W	W	-	-	

Table VIII, continued.

Line:	Silver			Copper		Iron		Others
Intensity:	3280 2000	3383 1000	4055 800	3248 5000	3274 3000	3440 500	3441 300	
S 1	W	W	-	VW	VW	-	-	
S 2	W	W	-	W	W	VW	VW	
S 3	M	M	-	W	W	-	-	
S 4	M	M	-	W	W	-	-	
S 5	tr	tr	-	-	-	-	-	
S 6	tr	tr	-	-	-	-	-	
S 7	M	M	-	W	W	-	-	
S 8	tr	tr	-	-	-	-	-	
S 9	tr	tr	-	-	-	-	-	
S 10	M	M	-	W	W	-	-	
S 11	M	M	-	W	W	-	-	
S 12	VW	VW	-	-	-	-	-	
S 13	W	W	-	W	W	-	-	
S 14	tr	tr	-	-	-	-	-	
S 15	W	W	-	VW	VW	-	-	
S 16	M	M	-	W	W	-	-	
S 17	W	W	-	W	W	VW	VW	
S 18	tr	tr	-	-	-	-	-	
S 19	M	M	-	VW	VW	-	-	
S 20	M	M	-	W	W	-	-	
S 21	W	W	-	W	W	-	-	
S 22	W	W	-	W	W	M	M	

S = strong; M = medium; W = weak; VW = very weak; tr = trace;
- = not detected.
N.B. Intensity measurements made by visual estimation.

the only elements lost from the majority of the samples. However, except perhaps for the loss of tin and zinc from G 39 and G 1, respectively, the losses are considered in all cases to be negligible. Silver is present in large amounts in native gold and the lines detected are of such great intensity, that it is not surprising that they are always present. The assumption of negligible loss of silver is further substantiated by the fact that the relatively intense silver line, 4055.264, is absent from more than 90% of the spectra. The copper lines are also very intense and could be expected to appear at very low concentrations.

After homogenization, the beads were all washed in boiling 2N hydrochloric acid to remove any surface oxidation layers or impurities and were, subsequently, re-examined under the binocular microscope to check their cleanliness. They were weighed, flattened and rolled into strips which could be cut up for spectrographic analysis, fire assay and atomic absorption analysis.

To check that the melting of the individual grains did homogenise the samples, a large sample of gold was formed into a bead and cut up into small pieces from which five further portions, each weighing 10 mgm, were taken. These 10 mgm samples were made into beads, as described above, and spectrographically analyzed under the following arcing conditions.

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Spectrograph: Large Hilger-Watts, Littrow type, spectrograph with quartz optics.

Excitation: Anode excitation, constant current d.c. arc, 220 volts, 4 amps.

Electrode gap: 4 mm.

Slit width: 20 microns.

Time: 20 seconds, the time required to burn the samples completely.

Emulsion: Ilford H 50 thin film half tone plates.

Wavelength region: 2360 A - 3200 A.

After developing the plate, the intensities of various silver, gold, copper and iron lines were measured using a microphotometer. A calibration curve for the plate was drawn on a Respectra spectro-calculating board and the log intensity ratios of the silver, copper, and iron lines to the gold lines were calculated. Table IX gives the results obtained for these ratios and the standard deviations and coefficients of variation for the five samples.

The results indicate that, within the limits of accuracy of the spectrographic analytical method, all five samples contain approximately the same amounts of copper, silver and iron. In other words, it can be argued that these three elements are uniformly distributed throughout the sample and that the method of homogenizing the gold grains is valid. If elements such as silver, copper and iron, which are present in the gold in relatively large amounts, distribute themselves uniformly throughout the sample during the bead formation, it seems likely that

..31/...

Table IX The intensity ratios, standard deviations and coefficients of variation calculated for five beads from one sample, to determine the homogeneity of the sample.

	1	2	3	4	5	$\bar{x}$	s	v(%)
$\log \frac{Ag\ 2437}{Au\ 2590}$	-0.69	-0.84	-0.89	-0.73	-0.71	-0.77	0.079	10.3
$\log \frac{Ag\ 2375}{Au\ 2590}$	-0.85	-0.90	-1.13	-0.71	-0.79	-0.88	0.142	16.2
$\log \frac{Cu\ 2961}{Au\ 2932}$	-0.73	-0.61	-0.71	-0.66	-0.81	-0.70	0.068	9.7
$\log \frac{Cu\ 2618}{Au\ 2590}$	-1.04	-0.77	-0.92	-0.82	-0.94	-0.90	0.095	10.6
$\log \frac{Fe\ 2599}{Au\ 2590}$	-0.45	-0.38	-0.32	-0.36	-0.40	-0.38	0.043	11.3
$\log \frac{Fe\ 3020}{Au\ 2932}$	-0.29	-0.15	-0.24	-0.16	-0.22	-0.21	0.058	24.8

$\bar{x}$ - mean value; s - standard deviation $= \sqrt{\frac{\sum (x - \bar{x})^2}{n}}$;

v - coefficient of variation $= \frac{100s}{\bar{x}}$

Table X The limits of impurities in the acids used for the cleansing of the samples, (from the manufacturer's specifications).

Element	HCl (30.5%)	HF (48%)	HNO_3 (55.5%)
Pb	0.00002%	0.001%	0.000009%
Fe	0.0001%	0.0008%	nil
As	0.000002%	nil	0.0000004%

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Table XI Limits of impurities in the high purity graphite electrodes and rods used for the spectrographic analysis, (from the manufacturer's reports).

Impurities	Electrodes			
	AGKSP L 3806	AGKSP L 3951	AGKSP L 4018	AGKSP L 4006
Aluminium	0.4 p.p.m.	0.1 p.p.m.	0.7 p.p.m.	x
Copper	0.9 p.p.m.	x	x	x
Iron	0.6 p.p.m.	0.2 p.p.m.	0.8 p.p.m.	1
Magnesium	0.8 p.p.m.	0.6 p.p.m.	1.0 p.p.m.	0
Silicon	1.6 p.p.m.	0.5 p.p.m.	0.9 p.p.m.	0
Sodium	x	0.2 p.p.m.	0.8 p.p.m.	x

x - complete absence; 0 - barely visible;

numbers indicate relative intensity.

Other elements sought: B, Ca, Pb, Mn, K, Ag, Sn, Ti, V.

Table XII The limits of impurities in the spectrographically pure copper oxide and iron sponge, (from the manufacturer's reports).

Element	Copper	Iron
Silicon	5 p.p.m.	1 p.p.m.
Calcium	2 p.p.m.	-
Iron	2 p.p.m.	-
Silver	2 p.p.m.	< 1 p.p.m.
Magnesium	1 p.p.m.	1 p p.m.
Sodium	1 p.p.m.	1 p.p.m.
Aluminium	< 1 p.p.m.	-
Manganese	-	3 p.p.m.
Nickel	-	2 p.p.m.
Copper	-	< 1 p.p.m.

Other elements sought: As, Au, B, Be, Bi, Cd, Co, Cr, Cs, Ga, Ge, Hf, Hg, In, Ir, K, Li, Mo, Nb, Os, P, Pb, Pd, Pt, Rb, Re, Rh, Ru, Sb, Se, Sn, Sr, Ta, Te, Ti, Tl, V, W, Zn, Zr.

the other minor elements present will also be evenly dispersed in the gold. This conclusion was confirmed when the reproducibility of the log intensity ratios of various trace elements was calculated for the spectrographic analysis of the samples (see table XXVI)

(f) Precautions taken to prevent contamination of the samples.

As the analytical methods used were very sensitive, being capable of detecting concentrations down to 1 p.p.m., great care was taken to prevent contamination of the samples. Crushing of the samples was carried out with an iron mortar only and the samples were sieved through a nylon mesh- perspex sieve. Only analar grade chemical reagents and distilled water were used for the cleansing of the samples. High purity graphite electrodes and rods, manufactured by National Chemical Products, were employed for the spectrographic analyses. Tables X, XI, XII list the limits of impurities in the acids, electrodes and metals used.

The gold beads were flattened and rolled on an anvil and roller, both highly polished and cleaned before use. For cutting up the large gold grains, a stainless steel scalpel was used and for the cutting of the rolled gold foil, a pair of dentist's pliers was used. The anvil, roller, scalpel and pliers were used only for their respective purposes.

..32/...

At each stage during the preparation of the samples for analysis, the gold was carefully examined under the binocular microscope to check the purity.

B. THE QUANTITATIVE DETERMINATION OF COPPER, IRON AND SILVER IN NATIVE GOLD

(a) Introduction

A great deal of work has been carried out by other workers on the quantitative determination of impurities in high purity gold. Two reviews, one by Beamish (1961) and other by Finkelstein (1962), dealing with the analysis of high purity gold have been published recently. Of the individual papers, those dealing with spectrographic methods of analysis include papers by Vorsatz (1957), Kuranov and Ruksha (1959), Kuranov, Ruksha and Sviridova (1959), Degtyareva and Ostrovskaya (1960), Esterhuizen (1962) and Strasheim, De Villiers and Brink (1962). Atomic absorption spectroscopy has been applied to the analysis of very fine gold by Schuler, Janson and James (1962) and Strasheim, Butler and Maskew (1962). Polorography, flame photometry and spectrophotometry have also been applied to the determination of impurities in gold.

However, very little work has been done on the quantitative analysis of native gold. Spectrographic methods were used by Raikhbaum et al (1940), Raikhbaum (1941) and Wilska (1952) for the quantitative determination of trace impurities in native gold. In the first paper,

..33/...

Raikhbaum and his co-workers determined silver, copper and tellurium in gold nuggests of 3 to 5 mgm, while in his later paper Raikhbaum described a method for the quantitative analysis of gold flakes of 0.5 to 2 mgm. Wilska determined the amounts of copper, iron, manganese and aluminium in gold.

A microanalytical chemical method for the quantitative analysis of gold nuggets was developed by Fer'yanchikh (1959). After cleaning, the nuggets were dissolved in aqua regia in the presence of tartaric acid and the precipitate of silver chloride and the filtrate were analyzed separately for the determination of the trace elements.

For the determination of the fineness of native gold, the fire assay technique has been in use for centuries and has recently been shown by Hanrahan (1962) to be extremely reliable. The specific gravity method for the determination of fineness has been discussed in detail by Caley (1949). The method is very simple but is, unfortunately, only reliable to 1%.

Of all the techniques mentioned above, the spectrographic, atomic absorption spectroscopic and fire assay methods seemed to be the most suitable for the quantitative determination of silver, copper and iron in the gold samples. As the silver content in the samples was too great to be determined spectrographically, the fire assay technique was used. Spectrographic methods were at first selected for the quantitative determination of copper and iron, though it was

..34/...

decided not to work in solution but to keep the gold in its native form. The risk of contamination during dissolution was considered to be high and it was thought that elements could be added to the gold from the reagents. However, as will be explained in the following section, suitable standards for spectrographic analysis could not be prepared and the copper and iron contents were eventually determined using atomic absorption spectroscopy.

(b) <u>The attempts to prepare homogeneous gold alloys for use as standard samples</u>

For quantitative spectrographic analysis, reliable standard samples are required and therefore an attempt was made to prepare gold alloys with varying amounts of silver, copper and iron. A study of the phase diagrams of gold-silver, gold-copper and gold-iron alloys, as given by Hansen (1958) showed that silver, copper and iron were capable of complete, or partial, solid solution with gold. The solubility curves for these metals in gold are shown in figures 2, 3, and 4. From the curves it is seen that silver and copper are both capable of complete solid solution in gold while up to 56% iron can enter the gold lattice. Therefore, it appeared that there should be no difficulty in the silver, copper and iron forming homogeneous alloys with gold.

Strasheim, De Villiers and Brink (1962) described a method for the preparation of gold alloys containing impurities of copper, iron,

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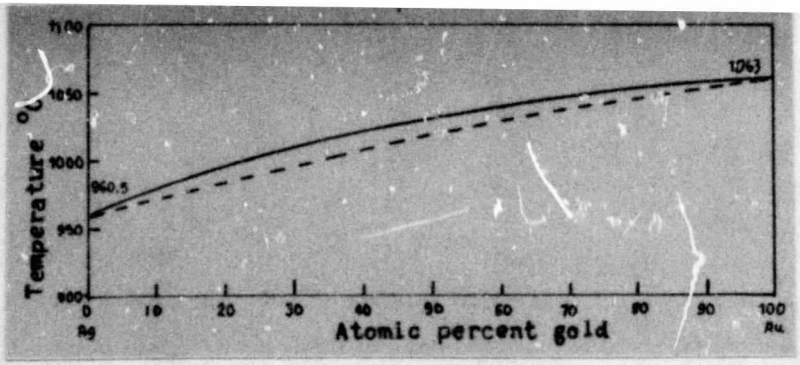


Figure 2 The gold-silver phase diagram showing complete solid solubility between gold and silver. (After Hansen, 1958).

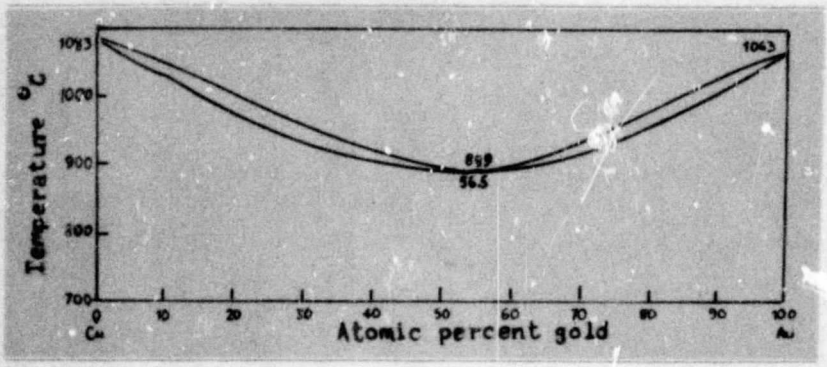


Figure 3 The gold-copper phase diagram showing complete solid solubility between gold and copper at elevated temperatures. (After Hansen, 1958).

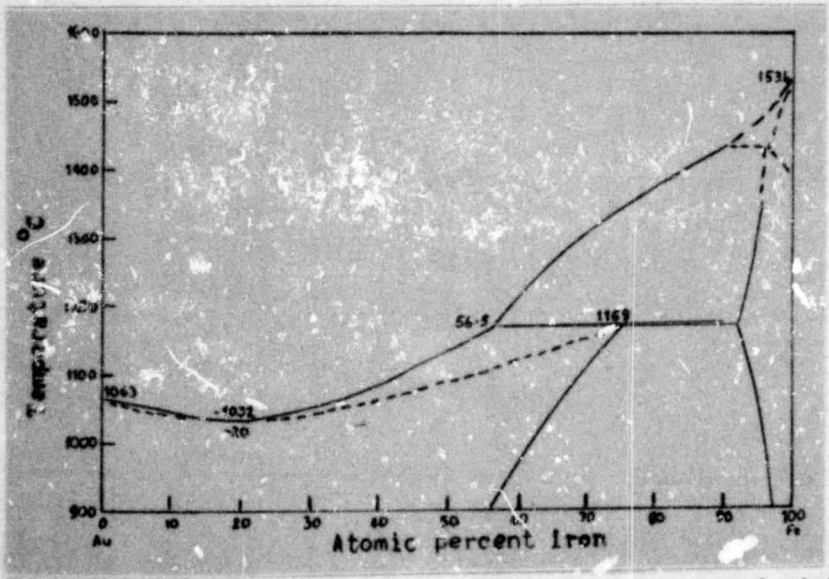


Figure 4 The gold-iron phase diagram showing approximately 56% solid solubility of iron in gold. (After Hansen, 1958).

silver, palladium, lead and zinc. The method involved the preparation of four preliminary standards by compressing the gold and impurities into pellets, sealing these pellets into evacuated silica tubes, heating the tubes until the pellets were molten and shaking them to ensure good mixing. The final set of standards was prepared by diluting the preliminary standards with proof gold. Owen and Roberts (1945) also described the preparation of gold alloys, their method being to shake the molten alloys, under vacuum, for periods ranging from a quarter of an hour to an hour. The alloys were allowed to cool slowly in the furnace or were else quenched rapidly to prevent segregation. The ingots were sampled to test their homogeneity and if they were found to be inhomogeneous, they were annealed for periods of twelve hours or more. Kuranov (1959) prepared standard alloys for the analysis of high purity gold by first making binary alloys of the impurities which he hoped to introduce into the spectrographically pure gold. In this way he lowered the melting points of the impurities to within the range of the melting point of gold. The impurities were added to the gold in a graphite crucible and were covered by charcoal. A high frequency furnace was used with an inductor to ensure intense mixing of the melt.

Initially it was decided to follow the method of Strasheim, De Villiers and Brink (1962) and the required amounts of gold, silver, copper and iron to give a gold alloy containing 20% silver, 0.5% copper and 0.2% iron were weighed out from spectrographically pure metals. The alloy constituents were then melted as described above.

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Author Gay N C (nicholas Gay)
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