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PAPER 65-7

A RAPID AND SENSITIVE METHOD FOR  
DETERMINING GOLD IN ROCKS AND  
OTHER GEOLOGICAL MATERIALS

(Report and 7 figures)

R. A. Washington and R. H. C. Holman



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### ABSTRACT

Preliminary tests have indicated the feasibility of using the classical method of fire assay to concentrate gold in a silver bead from silicate rocks for subsequent neutron activation analysis using instrumental techniques. Radiochemical separation of gold following activation is not required. The detection limit, using silver as a collector, is about 0.01 ppm Au for a sample weight of one assay ton (29.2 g). The precision, for homogeneous samples, is likely to be about 1 to 2 per cent at 1 ppm falling to around 50 per cent near the detection limit. The proposed method is essentially simple and it is estimated that the probable productivity would be about 25 to 30 determinations per man day. Improved techniques are suggested that should lower the detection limit by a factor of about ten.

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# A RAPID AND SENSITIVE METHOD FOR DETERMINING GOLD IN ROCKS AND OTHER GEOLOGICAL MATERIAL

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## INTRODUCTION

Advances in geochemistry are often closely related to improvements made in techniques of chemical analysis, and a marked paucity of geochemical data for particular elements can often be ascribed to difficulties of analysis. A typical example is gold. Our geochemical knowledge of this metal is relatively poor when compared with the considerable geochemical data available for many other metals, such as copper, lead and zinc, that have been in common use since early times.

The need for a better knowledge of gold geochemistry became especially evident during recent studies of the Kirkland Lake, Ontario, gold mining district undertaken as a joint project by the Geological Survey of Canada and the Ontario Department of Mines. An important aim of these studies has been to provide basic geologic and geochemical information from which new techniques for gold exploration might be developed. Preliminary investigations into the nature of the glacial till occurring in the district (Lee, 1963) and the geochemistry of the rocks (Holman, 1963) indicated a pressing need for (i) an adequate method for determining gold and (ii) reliable information on the distribution and amount of gold occurring both in the rocks and the overlying surficial deposits of the area.

One of the prime difficulties in the analysis of geological materials for gold arises from the very low concentrations of this metal normally encountered. For example, a gold content as high as 10 ppm is extremely rare in silicate rocks and could in fact be regarded as ore grade. Normal concentrations usually lie well below this figure and within the range of 0.1 ppm down to 0.001 ppm (Vinogradov, 1962).

A comprehensive review of spectrographic and colorimetric methods for gold analysis made by Beamish in 1961 and Beamish, Sen Gupta and Chow (1964), suggested it was unlikely that the detection limit of these methods could be increased much beyond 0.01 ppm without laborious concentration of the gold. This limit of detection is only about 1 order of magnitude better than the sensitivity of the fire assay technique using a single fusion. Further work on the analysis of gold by either colorimetric or spectrographic methods seemed unpromising, therefore, and the use of activation analysis was considered.

From theoretical considerations a detection limit of the order of 0.0001 ppm Au could be expected for the direct irradiation of a 100 mg



sample of silicate rock in a flux of  $5 \times 10^{12}$  neutrons per  $\text{cm}^2$  per second. It was realized, of course, that this theoretical sensitivity might be reduced somewhat by such factors as interference from other elements and certain practical limitations.

Although direct irradiation of a powdered sample would at first sight appear to be the most attractive technique (e.g. Vincent and Smales, 1956) as it permits full use of the advantages of activation analysis, such as freedom from reagent blanks and the possibility of using only very small amounts of sample, this simple approach raises two important objections. Direct irradiation necessitates the separation of gold from interfering elements before counting. Although radiochemical separations are generally more rapid than the more conventional separation techniques, they are nevertheless sufficiently time-consuming to be avoided whenever possible. The second objection arises from the problem of adequately sampling a material in which the trace element to be determined is not distributed uniformly but is present in rare mineral grains as discussed in detail by Wilson (1964). This is very likely to be the case with gold and in silicate rocks it can be expected to occur as minute blebs of the metal, or its tellurides, of usually less than 50 microns in size<sup>1</sup>. Fine grinding may be expected to increase the homogeneity of a sample, but this treatment may have only a limited effect on the dispersion of very small rare grains, especially if they consist of a non-friable and malleable material such as gold.

This particular sort of sampling problem probably accounts for our own experience that the use of a small sample (of the order of 0.1 g) for direct irradiation is likely to give poorly reproducible results, that become quite unreliable with concentrations below 0.05 ppm Au. The only practical solution to this difficulty appeared to be an increase in the size of the sample. Problems in the handling of large radioactive samples and the high cost of neutron activation severely limit the weight of sample that can be used for routine analysis and it became necessary, therefore, to search for a convenient way of concentrating gold from a relatively large mass into a small weight of a medium suitable for irradiation and subsequent counting.

The general requirements of the method envisaged were (1) use of a large, finely ground sample; (2) concentration of the gold from this into a small weight of medium; (3) irradiation of the concentrate, and (4) a direct count of the gold activity without preliminary chemical separation.

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<sup>1</sup>Personal communication from S. Kaiman, Mines Branch, Department of Mines and Technical Surveys.

For the concentration stage of the method two possibilities seemed to merit serious attention. The digestion of the finely-ground rock sample with aqua regia and concentration of the dissolved gold by coprecipitation, ion exchange resins or other collectors, although not tested, appeared to be feasible; Safronov et al.(1960), for example, report the successful use of activated charcoal as a collector prior to the spectrographic determination of gold. A disadvantage of the aqua regia attack, however, is that it can only give, at best, that gold exposed to the acid during digestion, and this might vary considerably with the fineness of grinding.

The second method of concentrating gold considered was the classical fire assay technique. This offered several advantages and was consequently used for this work. An important advantage of this method was its wide acceptance by assayers and the fact that a great deal was known empirically about it. It would also be relatively cheap, and therefore acceptable if found satisfactory, since many assay laboratories are already well equipped to do this sort of work.

Certain difficulties were anticipated in the use of the fire assay method and part of the experimental work was directed towards clarifying the following problems. The standard fire assay technique (using a single sample of 1 assay ton weight or 29.2 g) is sensitive only to about 0.003 oz per ton or 0.1 ppm Au. No information was available on the performance of the method as an efficient collector of gold at the very low concentrations of much less than 0.1 ppm that were to be investigated. It seemed possible, also, that at these very low concentrations serious contamination from vessels and from the constituents of the charge might be encountered. An estimation of these factors was attempted to see whether the use of gold-free reagents might be required, since a considerable blank might well impose a practical limit to the sensitivity of the method. It seemed likely, also, from preliminary calculations that during the irradiation of the silver beads large amounts of  $\text{Ag}^{110\text{m}}$  would be generated for which a correction would be required if direct counting were to be used.

An account is given of the experimental work that has been completed on the proposed fire assay-neutron activation method and an assessment is given of its feasibility for further development. For a discussion of the geochemistry of gold based on results obtained by activation analysis see Vincent and Crocket (1960) and Shcherbakov et al. (1964).

## EXPERIMENTAL PROCEDURE

To investigate the problems discussed briefly in the Introduction two sets of silver beads containing gold were prepared.

The first set was made by adding measured volumes of a standard solution of gold to fire assay charges and proceeding with fusion and cupellation in the usual way to produce silver beads. A charge consisting of 90 g of litharge (PbO), 30 g of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), 20 g of silica (SiO<sub>2</sub>), 15 g of borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>2</sub>O) and 3 g of flour was used throughout.<sup>1</sup> To avoid possible loss of gold solution to the pot walls the following precautions were adopted. Half the charge was placed in the fusion pot and the remaining space above it lined with a sheet of thin plastic wrap. Most of the remaining charge was then added. A small depression was formed in the surface of the charge and measured volumes of gold solution were added.<sup>2</sup> The remainder of the fusion charge was now added and the pot dried for 2 hours at 100°C. The dried charge was then mixed thoroughly and returned to the pot together with the plastic wrapping. The fusions were carried out at 1000°C for 75 minutes, after which lead buttons weighing between 25 and 30 g were separated from the slag. The lead buttons, together with about 10 mg of pure silver foil, were each cupelled in magnesia cupels at 850 to 900°C to produce silver beads weighing about 10 mg. These are subsequently referred to as F.C. (fusion charge) beads.

The second set of beads, referred to as D.C. (direct cupellation) beads, was made by forming sheets of lead foil weighing about 25 g into boats. Measured amounts of gold solution were then added and evaporated to dryness on a hot plate. About 10 mg of silver foil was now added to each and cupelled in the same manner as previously described for the first set of beads.

Several blank silver beads containing no added gold were also prepared using both techniques in order to estimate contamination from reagents and possible losses.

The beads were weighed, encapsulated by crimping in known order in aluminium tubes (0.0625" I.D. x 0.0015" wall thickness: 5025 alloy), and each tube marked for later identification. The tubes were irradiated for about 3 days in a "self serve" position of NRX reactor at Chalk River in a thermal neutron flux of about  $8 \times 10^{12} \text{ cm}^{-2} \text{ sec}^{-1}$ . After the irradiation the aluminium tubes were removed by dissolving them in 6M potassium hydroxide solution. The beads were then washed with water and transferred to small screw-capped vials for counting.

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<sup>1</sup>Reagents supplied by Assay Supplies Ltd., Kirkland Lake, Ontario.

<sup>2</sup>The gold solutions were freshly prepared by dissolving weighed amounts of spectrographically pure gold (Johnson, Matthey and Co. Ltd.) in aqua regia and diluting to required volumes with water.

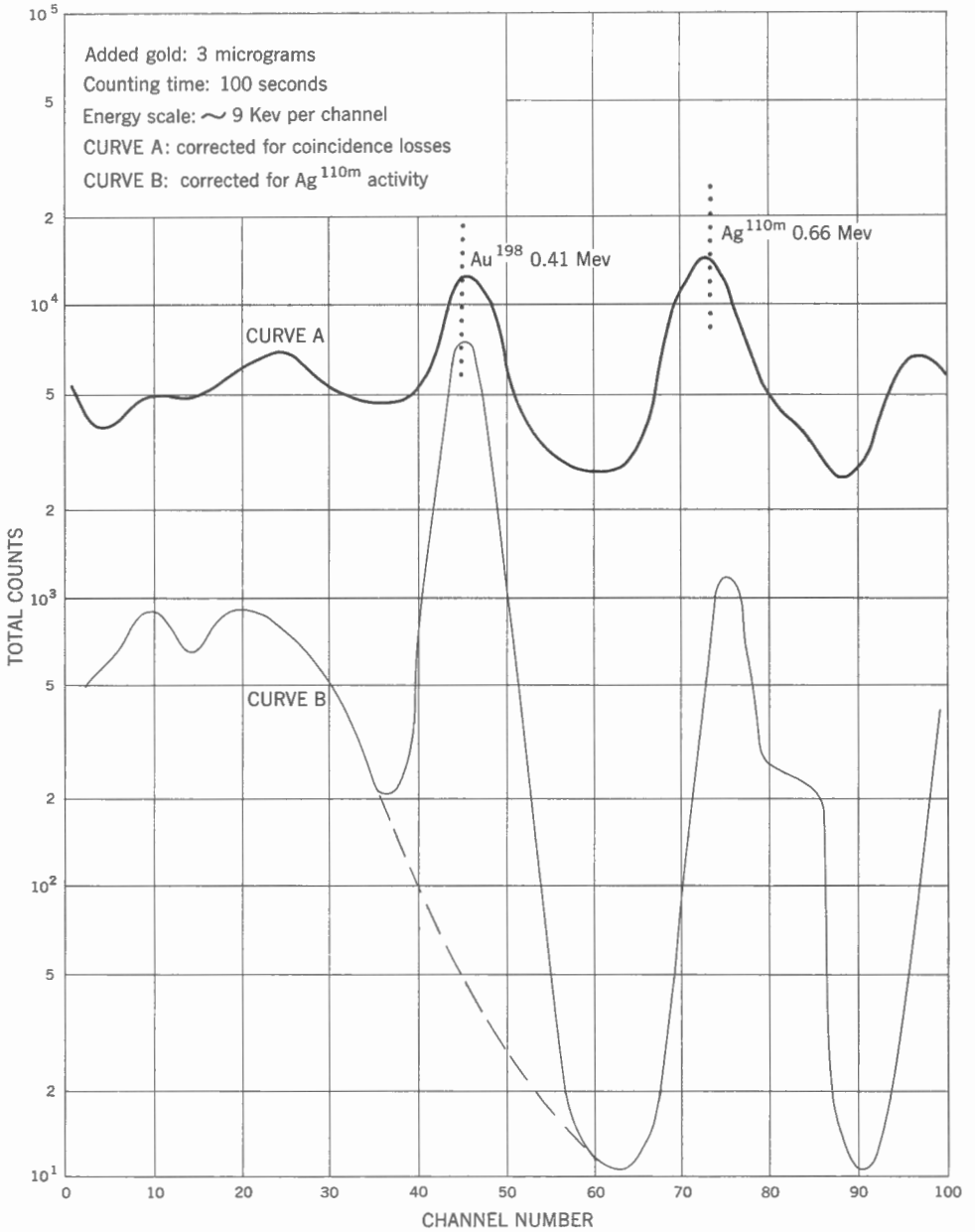


Figure 1. Typical gamma-ray spectrum from silver beads made by the fusion charge (F. C.) technique with 3 micrograms of added gold.

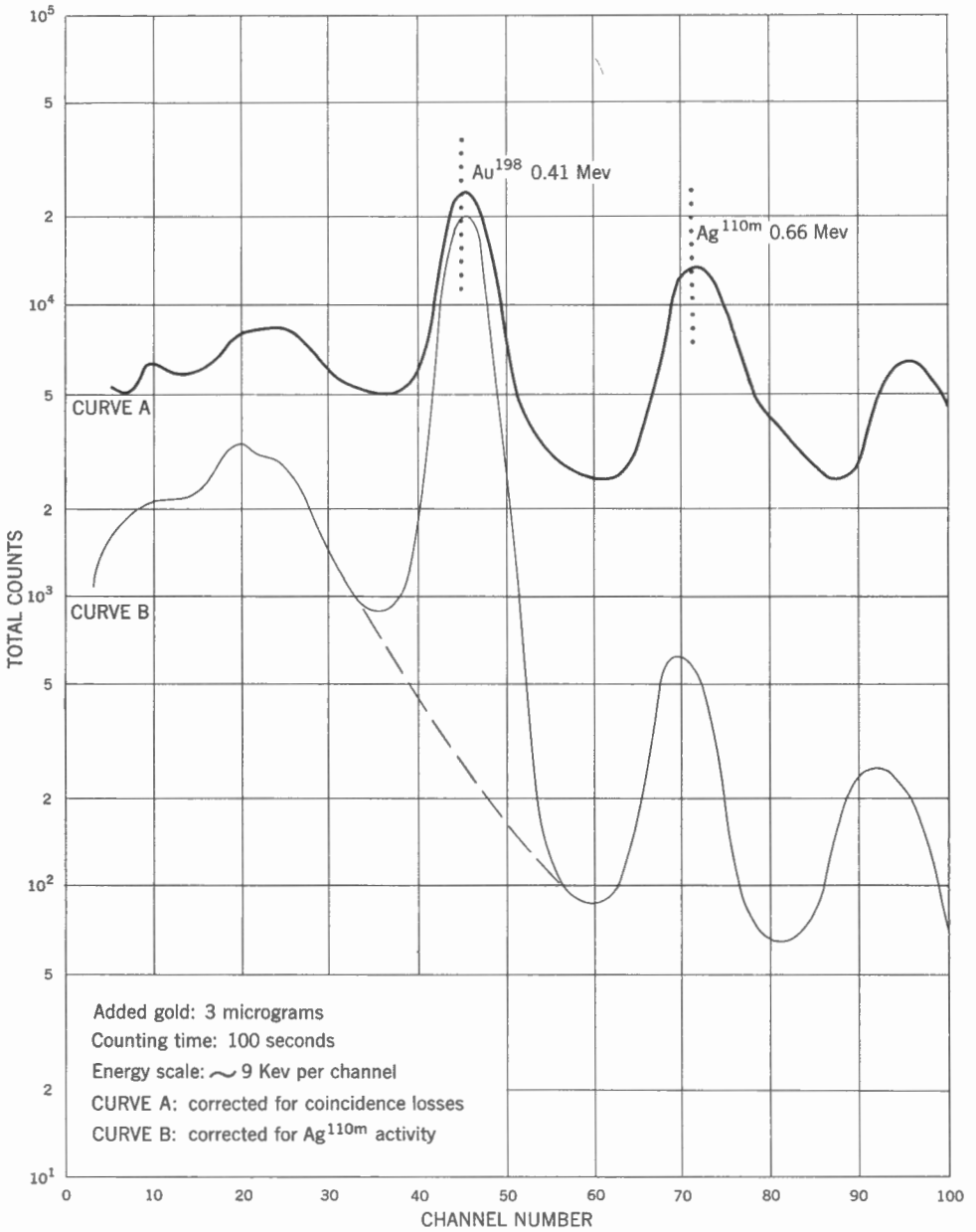


Figure 2. Typical gamma-ray spectrum from silver beads made by the direct cupellation (D. C.) technique with 3 micrograms of added gold.

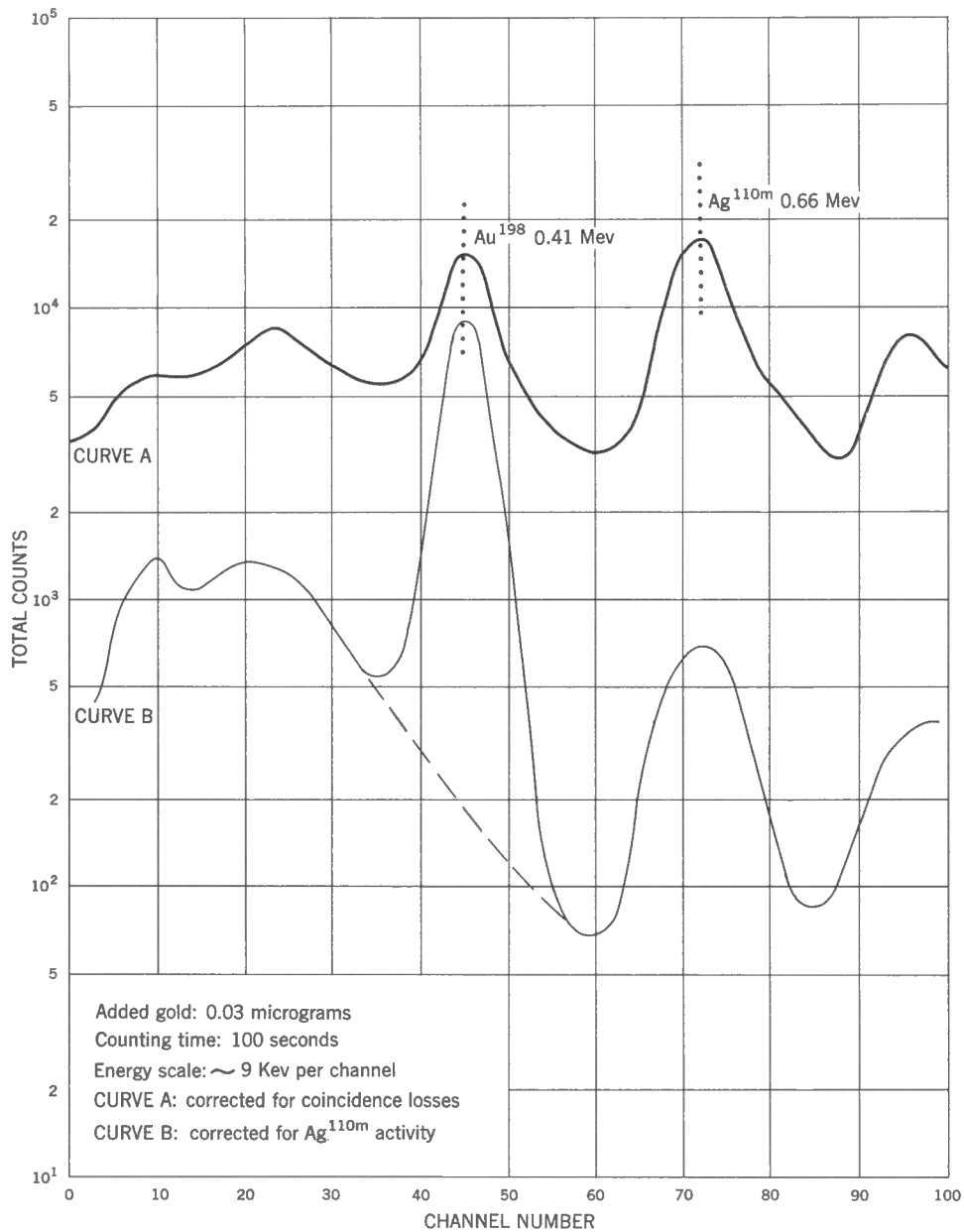


Figure 3. Typical gamma-ray spectrum from silver beads made by the direct cupellation (D. C.) technique with 0.03 micrograms of added gold.

The gamma-ray spectra of the samples were measured using a 3" x 3" thallium-activated sodium iodide crystal and a Computing Devices of Canada 100 channel pulse height analyzer. The samples were mounted at a distance of about 1 metre from the detector because of the large amounts of  $\text{Ag}^{110\text{m}}$  produced in the sample matrix. All the samples were counted for 100 seconds. The gamma-ray spectra were recorded graphically after corrections for coincidence losses in the electronic circuits and for the activity of  $\text{Ag}^{110\text{m}}$  produced in the silver matrix had been made, as described later.

## RESULTS AND DISCUSSION

### Coincidence losses

A correction for coincidence loss was made for each sample counted by multiplying the observed spectrum, i.e. the recorded number of counts in each of the 100 channels, by a factor  $\frac{100}{L}$ , where L is the measured coincidence loss in per cent for the sample, as indicated by the pulse height analyzer.

### Corrections for $\text{Ag}^{110\text{m}}$ activity

The spectra were further corrected for  $\text{Ag}^{110\text{m}}$  activity in the following way. The three spectra for the F.C. blank beads (e.g. Fig. 4, curve b) were averaged<sup>1</sup>, and a mean sample weight for the three beads calculated. The corrected spectrum for each sample,  $f(E_\gamma)_{\text{CORR}}$ , was then obtained from the following expression,

$$f(E_\gamma)_{\text{CORR}} = f(E_\gamma)_{\text{OBS}} - \overline{f(E_\gamma)_B} \times W_s / \overline{W}_b$$

$f(E_\gamma)_{\text{OBS}}$  is the observed spectrum (corrected for coincidence losses, e.g. Fig. 1, curve A)

$\overline{f(E_\gamma)_B}$  is the average spectrum of the F.C. blank sample

$W_s$  is the weight of the sample bead,

$\overline{W}_b$  is the average weight of the F.C. blank sample beads.

---

<sup>1</sup>By summing the counts obtained in each channel on the three samples (after normalizing to unit sample weight) and dividing by three.

Typical spectra resulting from this correction for silver  $f(E_\gamma)_{\text{CORR}}$  are shown in Figures 1 to 3, curves B. The curves drawn with a continuous line represent a visual estimation of the corrected spectra based on plotted points and previous experience of these particular spectra. The effect of this correction for silver is clearly seen in the marked enhancement of the  $\text{Au}^{198}$  0.41 Mev peak relative to the  $\text{Ag}^{110\text{m}}$  peaks.

#### Explanation of corrected spectra

The chief features of the corrected spectra are:

1. A peak in, or near, channel 45, due to the 0.41 Mev gamma-ray of  $\text{Au}^{198}$ .
2. Peaks in, or near, channels 71 and 95, due to the 0.66 Mev and 0.88 Mev gamma-rays of  $\text{Ag}^{110\text{m}}$  respectively.
3. A shoulder that is frequently seen on the high energy side of the 0.66 Mev peak near channel 80. This can be attributed to the  $\text{Ag}^{110\text{m}}$  gamma-ray at 0.76 Mev.
4. A slight broadening of the peak at 0.88 Mev is often noticeable and this is probably due to the 0.94 Mev gamma-ray of  $\text{Ag}^{110\text{m}}$ .

The major features of the observed spectra can thus be fully explained in terms of silver and gold activities and the presence of other elements in amounts sufficient to interfere with the measurements is not evident.

#### Estimation of gold activity

The amount of gold activity ( $\text{Au}^{198}$ ) was estimated by summing the counts in the corrected spectra,  $f(E_\gamma)_{\text{CORR}}$ , (Figs. 1 to 3) for channels 39 to 51 inclusive and subtracting the total 'background' counts for these same channels. The total 'background' count was estimated graphically from the curves as shown by the dotted lines in Figures 1 to 3. The results for gold activity obtained in this way for each silver bead are given in Tables 1 and 2. The relationships between these gold activities and amounts of gold added to the two series of beads are shown as graphs in Figure 5.

The curve C, Figure 5, for the F.C. beads indicates a linear relationship for amounts of added gold down to 0.3 microgram. Below this the response is weak and the curve flattens abruptly.



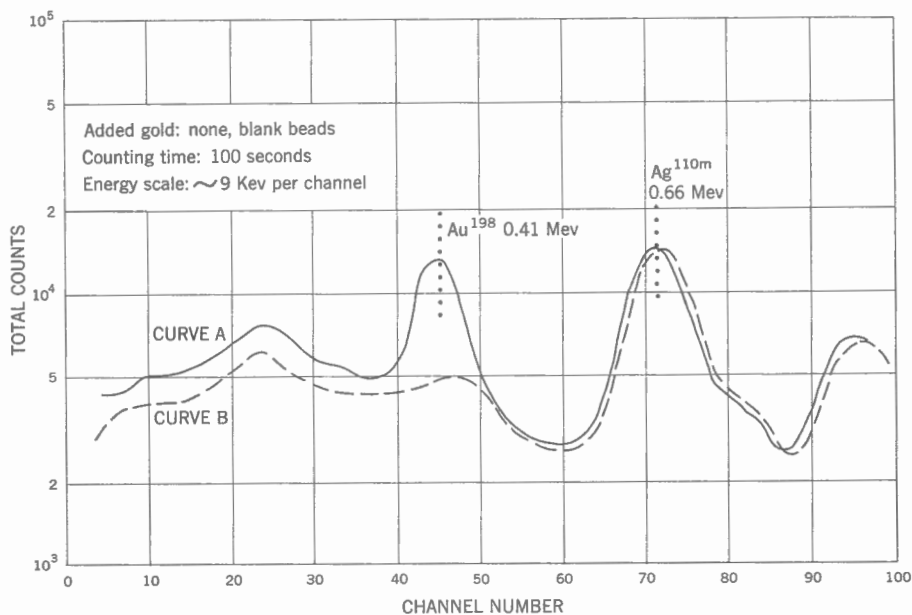


Figure 4. Typical gamma-ray spectra for blank silver beads: a, by direct cupellation (D. C.) technique; b, by fusion charge (F. C.) technique.

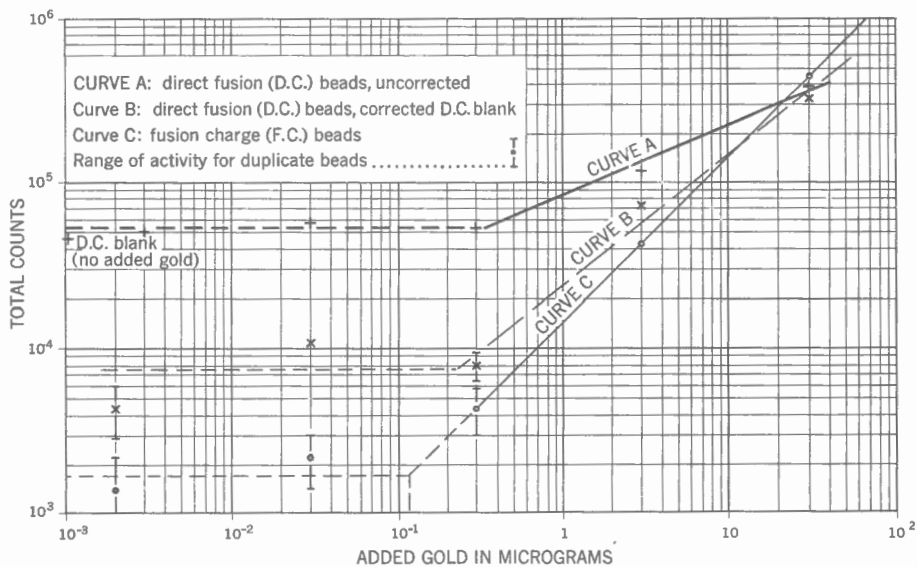


Figure 5. Gold activity produced by known amounts of gold.

The curve A for the D.C. beads shows a similar activity to the F.C. curve for 30 micrograms of added gold but a much flatter response for lower amounts. The activity is a minimum at 0.3 microgram and remains unchanged down to 0.003 microgram.

The differences between the curves obtained for the two sets of beads is best discussed by first considering the effect of blanks on the response.

#### Gold content of the blanks

The spectra for the D.C. blank beads (Fig. 4, curve a) show an appreciable peak at channel 45 and this suggests the lead foil used to make the boats carries a significant quantity of gold.

The effect of this gold blank is clearly seen in the flat response and abrupt change in slope of curve A, Figure 5, for the D.C. bead gold activities. When curve A (Fig. 5) is corrected for this blank, curve B is obtained with characteristics more closely resembling that of curve C for the F.C. beads.

The gold content of the D.C. blank beads estimated from the corrected curve B, Figure 5, is about 2 micrograms. This corresponds to approximately 0.07 ppm gold in the lead foil used to make the boats. The high gold content of the lead foil very probably accounts for the discrepancies between curves B and C.

The high blank in the D.C. beads effectively prevents their use as reliable gold standards as was originally intended. Nevertheless they may still be used by means of the corrected curve B to obtain an estimate of the gold in the F.C. beads. Using curve B, Figure 5, as a reference it would seem that amounts of gold down to about 0.3 microgram could be estimated by the F.C. technique with an accuracy of about 25 to 50 per cent. This would correspond to a concentration of approximately 0.01 ppm Au in an original sample of 1 assay ton weight.

The gold blank for the F.C. bead technique can be estimated roughly from curve C, Figure 5. Curve C shows that beads containing less than 0.3 microgram of added gold give similar gold activities, allowing for experimental error. The mean gold activity for these beads is about 1800 counts and this corresponds to 0.04 microgram of gold, or less, if the value given by extrapolating curve B is regarded as valid. The source of this gold is uncertain. Most likely it is a reagent blank and probably derived from the litharge used. If this is so, then it indicates a gold content for the litharge of about 0.001 ppm or less.

Table 1

Experimental data for gold in silver beads produced by direct cupellation (D.C.) technique

Bead No.	Added gold micrograms	Total counts in peak	Net counts (Peak area-average blank)
1	Blank	46,400	-
2	Blank	44,400	-
3	Blank	45,000	-
		Mean =	
		45,200±1000	
4	0.003	49,500±1000	4,300±1500
5	0.03	56,000±1000	10,800±1500
6	0.3	53,000±1000	7,800±1500
7	3.0	118,000±2000	73,200±2000
8	30	376,000±5000	330,000±5000

Table 2

Experimental data for gold in silver beads produced by the fusion charge (F.C.) technique

Bead No.	Added gold micrograms	Corrected mean activity for duplicate determinations	Precision per cent	Gold found micrograms (based on D.C. beads as standards)
1	0.003	1,300± 800	±62	less than 0.2
2	0.003			
3	0.03	2,200± 800	±36	less than 0.2
4	0.03			
5	0.3	4,300±1400	±33	less than 0.2
6	0.3			
7	3.0	44,000±2200	±5	2.1
8	3.0			
9	30	445,000±3000	±0.6	39
10	30			

### Accuracy and precision

A reliable measure of the accuracy to be expected from the F.C. technique is not possible because of the high blank in the D.C. beads, but a comparison of the gold activities as shown by curves B and C, Figure 5, allows some comment on the general performance of the technique proposed.

The discrepancy between the gold activity curves B and C, Figure 5, has already been noted and attributed partly to a high gold content of the lead foil used to prepare the D.C. beads. Differences between these two curves may be due, also, to the failure of the fire assay fusion process to collect all the added gold, especially at the lower concentrations. But curve C, Figure 5, shows a constant proportionality between gold activity and weight of added gold over the range 0.3 to 30 micrograms, which suggests that the loss of added gold over this concentration range is insignificantly small. How efficient the recovery of gold would be at lower concentrations is not known but there is no reason to suppose that it would change abruptly for the worse.

The precision of the F.C. technique in recovering added gold is indicated in Figure 5. Curve C represents the mean gold activity for duplicate F.C. beads; the deviations from these means at the various concentrations are indicated by vertical bars. A plot of the estimated precision against added gold, Figure 6, shows a fairly constant precision of between 30 and 60 per cent for amounts of added gold below 0.3 microgram. Above 0.3 microgram the performance improves markedly, giving a precision of 1 to 2 per cent, or better, at 30 micrograms per assay ton (1 ppm Au).

### Improvements and suggestions for further work

Greater precision would be obtained by using a longer counting time to obtain a greater number of counts. This would improve the accuracy in estimating the spectra after correcting for the blank  $\text{Ag}^{110\text{m}}$  activity. A counting time longer than 100 sec could not be used in these preliminary tests due to the limited capacity of the equipment for recording numbers of counts per channel.

The use of gold-free litharge and other reagents in the fire-assay fusion might be expected to lower the detection limit by decreasing the blank, but the present limit of about 0.3 microgram of gold is controlled mainly by the high gold blank in the D.C. or reference beads, the activity of the silver, and the techniques used for irradiating and measuring gold

activity in the beads. Attention to these latter factors is expected to provide the best possibilities for improving the method.

From the results of this study, and theoretical considerations, an estimate was made of the optimum conditions for irradiation and measurement of small amounts of gold in the presence of silver. It can be shown, according to Ordögh and Upor-Javanez (1961), that the ratio of Au<sup>198</sup> activity to Ag<sup>110m</sup> activity,  $A_{Au}/A_{Ag}$ , generated by thermal neutron irradiation, is given by,

$$\frac{A_{Au}}{A_{Ag}} = f(t) \frac{W_{Au}}{W_{Ag}}$$

$$\text{where } f(t) = 37.5 \frac{(1 - e^{-0.693t/65*})}{(1 - e^{-0.693t/6500})}$$

t = the irradiation time in hours,

\*values 65 and 6500 are the approximate half-lives, in hours, of Au<sup>198</sup> and Ag<sup>110m</sup> respectively (Koch, 1960).

For a given weight of gold to silver the factor f(t) is therefore a measure of the ratio of the gold activity to silver activity after time t.

A curve giving values of f(t) for irradiation times of 1 hour to 1000 hours is shown in Figure 7. It will be seen that the ratio of gold activity to silver activity decreases markedly for times greater than 100 hours. Some improvement in sensitivity for gold could be expected, therefore, by reducing the irradiation time of about 3 days that was used for this work to an irradiation of about 10 hours or less.

Consideration was given to the possibility of improving the method by using beta-gamma coincidence counting techniques to selectively count Au<sup>198</sup> in the presence of Ag<sup>110m</sup>. It can be easily demonstrated that, because the peak to total ratio of a 3" x 3" NaI detector for 0.41 Mev gamma-rays is 0.72 (Bell, 1955), the probability of observing a coincidence between a Au<sup>198</sup> beta-particle and a gamma-ray is about 0.72 G, where G is a factor that includes the geometry factors of both the beta and gamma detectors and their respective detection efficiencies and where the gamma-ray peak is assumed to occur in the energy range 0.41±0.05 Mev. It can also be shown

that the probability of the detection of a silver Compton event within the same energy range is only  $0.11T$ , where  $T$  is the total probability of detecting a  $\text{Ag}^{110\text{m}}$  gamma-ray.

From the foregoing facts it can be shown that the probability of observing a coincidence between a  $\text{Ag}^{110\text{m}}$  beta-particle and such a Compton event is only about  $0.053G$ . The use of a beta-gamma coincidence counter should, therefore, improve the probability of detecting  $\text{Au}^{198}$  relative to that of detecting  $\text{Ag}^{110\text{m}}$  by a factor of 13 or 14. At the time of these tests a beta-gamma coincidence counter was not available and this suggested improvement has not been tested.

The problems of counting gold activity in the presence of a large excess of silver prompted a consideration of the possibility of substituting for this carrier some other metal, e. g. tin (Faye and Inman, 1961). Consideration of the radioactive properties of isotopes of various metals indicated that the most promising carrier would be lead. A second advantage of lead was the possibility of shortening the fire assay procedure by taking it only as far as the fusion and the formation of a lead button. Irradiation would then be carried out on either a small portion of the button, or after reducing the weight of lead considerably by cupellation without the addition of silver. Several advantages arise from the use of these techniques:

1. Elimination or shortening of the cupellation stage would simplify and shorten the pre-irradiation manipulation required.
2. The cross-section for the activation of lead is less than silver by a factor of about 1000, so that far less activity due to the matrix would be produced using lead as a carrier.
3. The primary radioactive product of activation would be lead  $\text{Pb}^{209}$ . This has a half-life of about 3.3 hours only so that by allowing a short cooling period after irradiation most of the lead  $\text{Pb}^{209}$  activity would decay.
4. The lead  $\text{Pb}^{209}$  is a pure beta-emitter and no gamma-ray radiation is emitted that would interfere with the detection of the gold  $\text{Au}^{198}$  0.41 gamma-rays.
5. The use of lead would permit the minimum manipulation both during the fire assay and radiochemical stages and a probable productivity of about 25 samples per man-day.

Further experiments, in cooperation with the Ontario Department of Mines, are in progress to test the technique outlined and will be reported later.

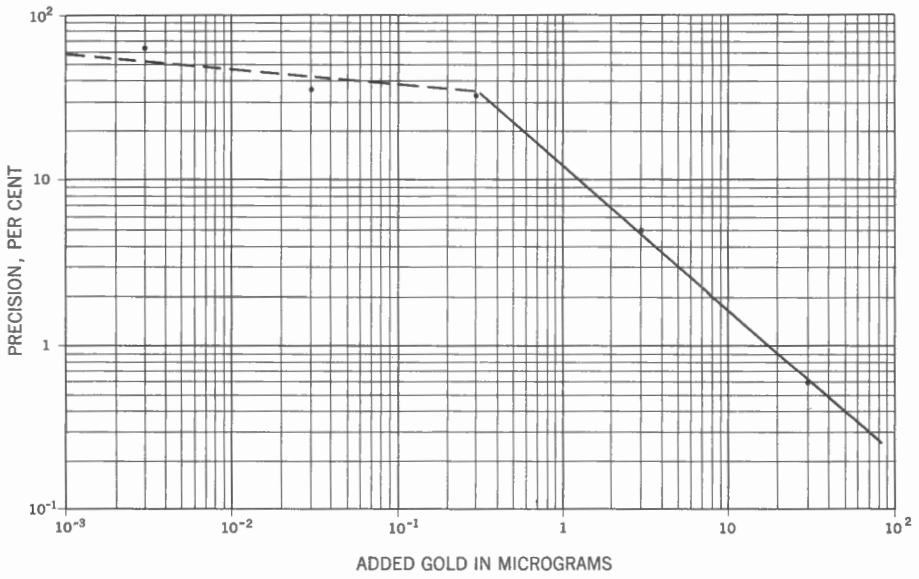


Figure 6. Precision of fusion change (F. C.) technique.

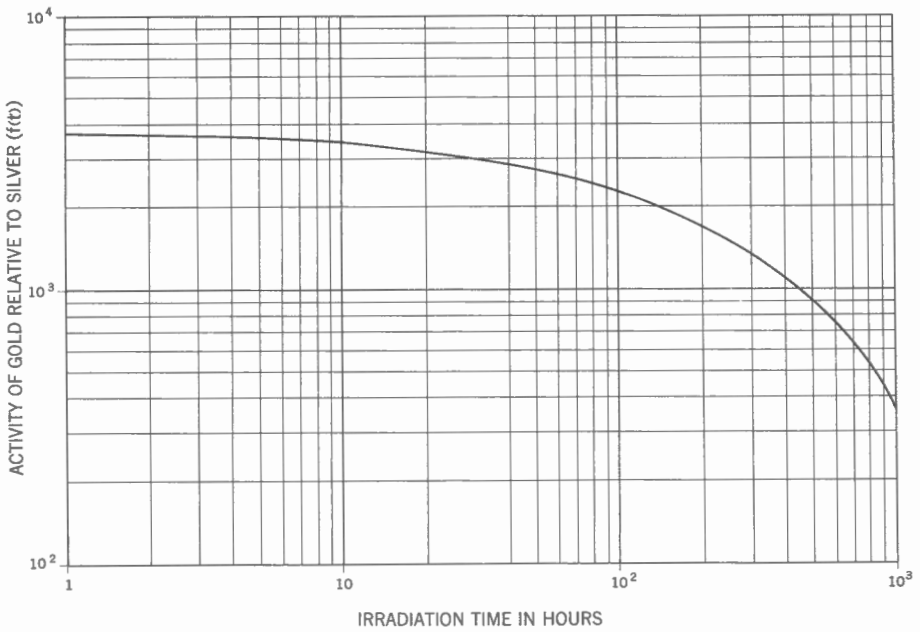


Figure 7. The effect of irradiation time on the activity of gold relative to silver.

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