



DETERMINATION OF GOLD IN HEAVY-MINERAL CONCENTRATES: FIRE ASSAY AND ATOMIC ABSORPTION SPECTROSCOPY (FA-AAS) VERSUS INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS (INAA)*

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INTRODUCTION

Fire assay, followed by analysis of the resulting precious metal bead by atomic absorption spectroscopy (FA-AAS) or some other "finish", is widely used in determination of the total gold content of heavy-mineral concentrates. Direct instrumental neutron activation analysis (INAA) of the concentrate, which has the advantage of being a nondestructive multi-element technique, is also used. The objective of this study was to compare gold results obtained by the two methods on typical heavy mineral concentrates from streams in southern British Columbia.

METHODS

Bulk stream-sediment samples were collected from five streams draining gold occurrences in southern British Columbia and screened to give five size fractions between 50 and 270 mesh (ASTM). Heavy-mineral concentrates were then prepared for each fraction by density separation in methylene iodide (S.G. = 3.3). Full details of sample locations and laboratory procedures are given by Day and Fletcher (1986).

Concentrates were dried, weighed, loaded into vials and submitted to a commercial laboratory for analysis by INAA after irradiation in a flux of 5×10^{12} neutrons per square centimetre per second. Samples were then stored until their radioactivity had fallen sufficiently for them to be handled safely. Sixty-three samples, covering a concentration range of approximately 5 to 25 000 ppb gold, were then submitted to a second laboratory for analysis by FA-AAS. Samples weighing more than 10 grams were divided and weighed into two or more pots as required. After fusion, parting and cupellation, the precious metal beads for each sample were either treated individually or combined in pairs for digestion in aqua regia and determination of gold by flame atomic absorption. Gold content of the original sample was then calculated.

RESULTS AND DISCUSSION

In comparing gold values, 15 samples giving results close to or below the INAA detection limits (between 5 and 22 ppb

gold) have been omitted. Seven of these also gave concentrations below the FA-AAS detection limit of 5 ppb gold.

Results for the 45 remaining samples (after excluding three outliers) are summarized in Figure 5-5-1. It is apparent that despite the very strong correlation ($r = 0.99$) between the two sets of data, there is some scatter and results by FA-AAS tend to be somewhat higher than those obtained by INAA. Fitting a line to the data points by reduced major axis regression gives:

$$\log \text{gold}_{\text{FA-AAS}} = 0.1801 + 0.9799 \log \text{gold}_{\text{INAA}}$$

that is, a positive intercept and a slope very close to one. The difference between the two data sets thus appears to be translational rather than rotational or a combination of the two. [A rotational bias would give an intercept of zero and a slope greater or less than one (Thompson, 1982)].

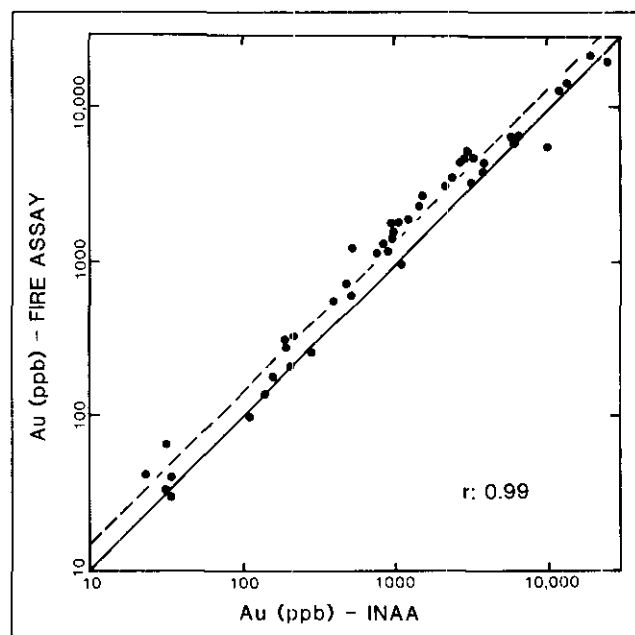


Figure 5-5-1. Log gold concentrations determined in 45 heavy-mineral concentrates by fire assay and atomic absorption (FA-AAS) and instrumental neutron activation analysis (INAA). The ideal line of equal values is shown by the solid diagonal with the reduced major axis regression to the data as the broken line ($\log \text{gold}_{\text{FA-AAS}} = 0.1801 + 0.9799 \log \text{gold}_{\text{INAA}}$).

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To investigate the discrepancy further, recovery of gold by INAA ($R\%_{\text{INAA}}$) where:

$$R\%_{\text{INAA}} = (\text{gold}_{\text{INAA}}/\text{gold}_{\text{FA-AAS}}) \times 100$$

was examined in relation to sample weight after arranging results in groups of nine by weight (Figure 5-5-2). It is apparent that recovery systematically decreases as weight increases. This is most obvious with weights greater than about 10 grams and leads to recoveries as low as 50 per cent with concentrates weighing 50 grams. There is also considerable variability in recovery within each weight group superimposed on this systematic trend. This variability is not obviously related to either sample composition (concentrations for 21 other elements are available from the INAA) or grain size. The latter, however, requires further investigation.

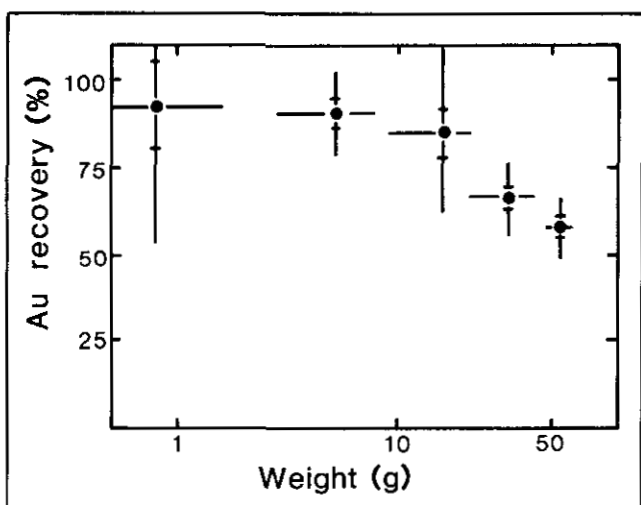


Figure 5-5-2. Per cent recovery of gold by INAA [$R\%_{\text{INAA}} = (\text{gold}_{\text{INAA}}/\text{gold}_{\text{FA-AAS}}) \times 100$] versus weight of heavy-mineral concentrate analysed. Each point represents the average weight and recovery of a group of nine samples. The vertical line through a point shows the one standard deviation limit for recovery and the horizontal line indicates the range of sample weights in the group. Small horizontal ticks on the vertical line indicate the standard error of the mean for recovery.

It is difficult to envisage an effect whereby the FA-AAS procedure could introduce a positive bias to gold values as sample weight increases. Low concentrations are therefore believed to reflect a systematic bias in the INAA method. This need not be a serious problem in exploration geochemistry, where relative rather than absolute concentrations may be acceptable (Fletcher, 1981), providing the bias remains constant. However, in routine surveys the yield of heavy minerals can vary considerably between samples from dif-

ferent bedrock sources and in sediment samples from adjacent sites on the stream bed (Day and Fletcher, 1986, 1987). In the determination of gold by INAA this variability could become an additional source of noise. Sample collection procedures should therefore be designed to keep variations in the weight of heavy-mineral concentrates to an acceptable minimum consistent with the goals of the survey.

Many parameters (for example, sample type and the INAA calibration procedures) may influence the magnitude of the bias between FA-AAS and INAA gold determinations. Results obtained in this study may not be typical. They do, however, indicate the need to evaluate weight-related effects in situations where there are likely to be large variations in the amounts of heavy-mineral concentrate available for analysis.

CONCLUSIONS

Instrumental neutron activation determination of gold content of heavy-mineral concentrates gave lower values than the fire assay and atomic absorption spectroscopy technique, with the difference increasing with increasing sample size. Where its multi-element nondestructive analytical capabilities make INAA the method of choice, survey design and sample collection procedures should attempt to minimize variations in heavy-mineral yield between sites as a source of unwanted variability in data for gold.

ACKNOWLEDGMENTS

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