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A METHOD FOR THE DETERMINATION OF THE GOLD CONTENT OF SAMPLES BY SOLVENT EXTRACTION AND ATOMIC ABSORPTION SPECTROPHOTOMETRY.

by

J.R. Beevers

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A METHOD FOR THE DETERMINATION OF THE GOLD CONTENT OF SAMPLES BY SOLVENT EXTRACTION AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (a)

by

J.R. Beevers (b)

Gold bearing samples can be assayed in many ways but the need exists for a rapid laboratory method of analysis for gold, arising particularly out of the recent increase in geochemical prospecting where both cost and speed are of paramount importance. The following method, which is rapid, cheap and of sufficient sensitivity to be useful in geochemical prospecting, is completely free from interferences; for instance as little as 0.2 ugm Au can be determined, and the presence of at least 0.1 gm of any of the following metals does not affect the result:

Al As Ba Be Ca Cd Ce(IV) Co Cr(III) Cu(II) Fe(III) Hg(II)
K Mg Mn Mo Na Ni Se Sn Sr Te Th U(VI) and Zn

In addition, this amount of gold can be determined in the presence of at least 0.01 gm of either Pt, Ru, Zr, or Pd. Saturation amounts of Ag, Tl, Ti or Pb in N HCl do not interfere either.

Anions do not interfere as shown by the fact that 0.2 ugm of Au can be determined in the presence of up to 0.5 gm of any of the following anions, added as their sodium salts:

PO_4^{3-} Br^- ClO_4^- NO_3^- SO_4^{2-}

REAGENTS AND APPARATUS

Aqua regio
H HCl

Organic Reagent - 0.03% solution of p - dimethylamino benzal rhodanine in amyl acetate, kept under refrigeration when not in use.

A commercial atomic absorption spectrophotometer (A.A.S.) was used with an air-acetylene burner fuel, a monochromator slit width of 100 microns and the 2428 A^o gold absorption line. A fully oxidising flame was used and an atomisation rate of 2 - 3 ml/min.

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(b) Present address: Bureau of Mineral Resources, Box 378, Canberra City, A.C.T., Australia.

EXPERIMENTAL

The sample is ground to 120 B.S.S. mesh, and 2.5 gm is digested for one hour with 10 ml of aqua regia in a covered beaker on the water bath. The dissolved sample is then taken to dryness, still in the water bath, and finally taken up with about 15 ml of N HCl. This is filtered, and the residue washed with two 5-ml portions of N HCl; the washings and filtrate are combined, the volume made up to 25 ml with N HCl in a standard flask and the contents thoroughly mixed.

10 ml of this solution is pipetted into a screw cap tube of 20 ml capacity, 1 ml of the organic reagent added, and the whole shaken violently for two minutes, after which the tube is allowed to stand in a vertical position to allow the two liquid phases to separate. The organic phase is then analysed for gold on the AAS using a calibration curve prepared by extracting 10 ml aliquots of N HCl containing respectively 1, 2, 4, 6, 8, 10 micrograms of gold with 1 ml of the organic reagent. If the sample contains so much gold that the absorption lies outside the range of the calibration curve, a repeat extraction is carried out using an aliquot of the sample extract and making up to 10 ml with N HCl before addition of the 1 ml of organic reagent. Variations of the relative volumes of the aqueous and organic phases should not be used as an alternative to this dilution procedure.

Direct flaming of the N HCl extract is not practicable in most cases. Firstly, the level of gold in most samples is too low to give a measurable absorption when using only the aqueous phase. Secondly, non-atomic absorption due to metal ions other than gold is too high, and is most severe at the lower levels of gold. Thirdly, when solutions containing large amounts of dissolved salts are aspirated, gold is trapped at the burner outlet where the salts are deposited.

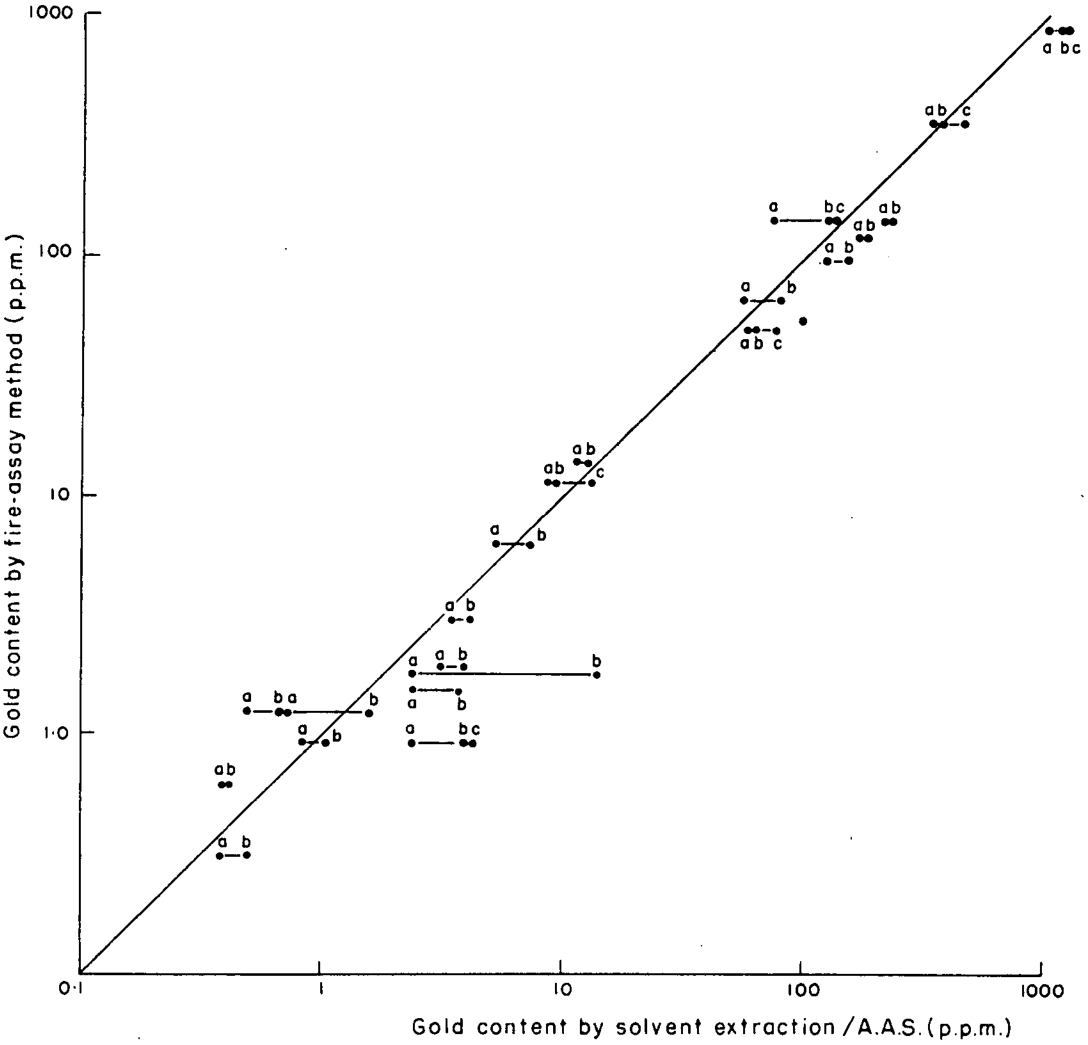
No difficulty or error due to gold in solution being reduced to the metal at the burner slit has been detected when using the solvent extraction technique.

The small quantity taken, compared to that for conventional fire assay, limits the accuracy of the method, but comparisons between the results of fire assays and the method described here show a good agreement (see Figure 1). Moreover, even when the samples contained very small amounts of gold as determined by fire assay (0-1 p.p.m.), gold was always detected by this method. By analysing different aliquots of the same crushed sample, considerable variation in the values of the gold content was found, and this was greater the smaller the amount of gold present. This variation is due to errors in sampling arising out of the particulate nature of the gold, and not due to analytical errors.

A further improvement in sensitivity has been gained by panning the very finely ground sample and assaying only the heavy material. In some cases, a hundred-fold increase in sensitivity can be effected by this technique.

A more detailed account of the method will be given in a subsequent paper.

Figure 1. Comparison of the gold content of twenty-one samples determined by the classical fire-assay method and by the solvent extraction/A.A.S. method. a, b, c are replicate samples.



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