

## **Use of Bulk Sample and Hot Acids in Stream Sediment Survey for Gold**

**K. Bheemalingeswara**

Department of Earth Science  
College of Natural and Computational Sciences  
Mekelle University, P. O. Box 3066, Mekelle, Ethiopia  
Email: kbheema@hotmail.com

### **Abstract**

Fifteen bulk stream sediment samples (minus 0.59mm size) from an area known for gold mineralization in Ramagiri Gold Fields, India are tested for gold. In addition, different size fractions (-0.590 + 0.259; -0.259 + 0.177 and -0.177) of the stream sediment are also analysed for gold. The data when compared suggests that the bulk samples data not only tally well with the previous stream sediment survey data but also provide additional anomalies for gold. The method suggested in this paper is bulk leach extractable gold (BLEG) using hot aqua regia and methyl isobutyl ketone solvents (MIBK-AR). The bulk leach extractable gold data of the new method seems do not suffer from iron oxide coatings, nugget effects, invisible gold etc as compared to the data of different size fractions (coarse/ fine size fractions) of stream sediment sample. Hence it provides near total values for gold present in the sample. The proposed method differs from the widely used BLEG in terms of hot aqua regia treatment in place of cold cyanide solution. The procedure suggested in this paper is simple to use and it provides an alternative to the commonly used coarse/finer fractions or BLEG (using cyanide) methods in stream sediment surveys for Au particularly in areas where oxide/hydroxide coatings are conspicuous.

**Key words:** Iron oxide/hydroxide coating, Bulk sample, Bulk leach gold, Stream sediment survey.

### **Introduction**

Geochemical surveys these days have become an integral part of mineral exploration particularly in the search for different metallic deposits of Cu, Pb, Zn, Au etc. Sophistication in technology, which resulted in the development of advanced instrumentation techniques in recent years, has further helped to improve and modify the geochemical survey methods to a great extent. Of course, the need to look for subsurface or blind deposits also has provided impetus to this development.

Depending on the nature of the geochemical survey- regional or local and type of deposit under consideration, the sampling scheme is planned. Stream sediment, soil or rock sampling either one or in combination is a common practice. In the case of stream sediment and soil samples, it is important to decide whether a particular size fraction of the sample or the bulk sample is used for analysis. A brief account on the advantages and limitations of the commonly used geochemical survey methods for gold, like panning in the field, stream sediment or soil sampling using coarse/ finer fractions or bulk (for bulk leach extractable gold (BLEG)) is provided here so as to understand the scope of the proposed method in the paper.

Panning method is in practice from time immemorial. The nuggets are recovered by panning under water. This field method on the basis of number of visible gold nuggets present in the panned concentrates helps to obtain information related to the source, resource size, type and extent of the deposit. Reliability of the data of panning method has been improved significantly by shifting from field to laboratory and adopting a systematic and scientific approach. In the laboratory, heavy mineral fractions are separated from the bulk sample using heavy liquids and run in Isodynamic separator to separate magnetic and non-magnetic components. Gold bearing non-magnetic components are analysed for gold after pre-concentration using organic solvents like isobutyl methyl ketone (MIBK). Further developments in the panning methodology have helped to modify this method with regard to secondary iron oxide/hydroxide coating over gold grains which was hampering identification of visible gold and separation from magnetic fractions in Isodynamic separation (Bheemalingeswara 1995a, 1995b). Furthermore, secondary iron oxide-hydroxide coating being common in the secondary environment, there were many efforts by different workers to understand the possible relationship between the coatings and metal ions including gold in the secondary environment (Schooven *et al.*, 1992; Enzweiter and Joekes, 1991; Crone *et al.*, 1984; Bheemalingeswara, 1996; Chao *et al.*, 1976). Limitations in this method are the nugget effects, intensity of the secondary iron oxide coatings and sample processing problems.

Finer fractions of stream sediments or soil samples are used in place of coarse to avoid processing problems and nugget effects and there-by improving reliability of the data (Bheemalingeswara, 1997; Xie and Wang, 1991; Fletcher and Joanes Muda, 2003; Cohen *et al.*, 2005). Hot acid treatment is preferred to leach total gold present in the finer fraction of the sample *i.e.* 100% recovery. Limitations in this method are: 1) finer fractions generally will be <1% in the bulk sample, hence sieving of large quantities of bulk is required; 2) sieving should be done in the field itself; 3) concentrations will generally be in ppb ranges and 4) in the case of intense coating proper separation of the fraction may be difficult.

Another important method recently came into practice is bulk leach extractable gold (BLEG). Significant change in this method is use of the bulk sample instead of a particular size fraction and leaching of gold (irrespective of size) with cyanide solution. It does not involve any sample processing. Data becomes more reliable and reproducible. This method has helped to locate many gold deposits including low grade in different parts of the globe (Beeson, 1995). Limitations or disadvantages in this method are:

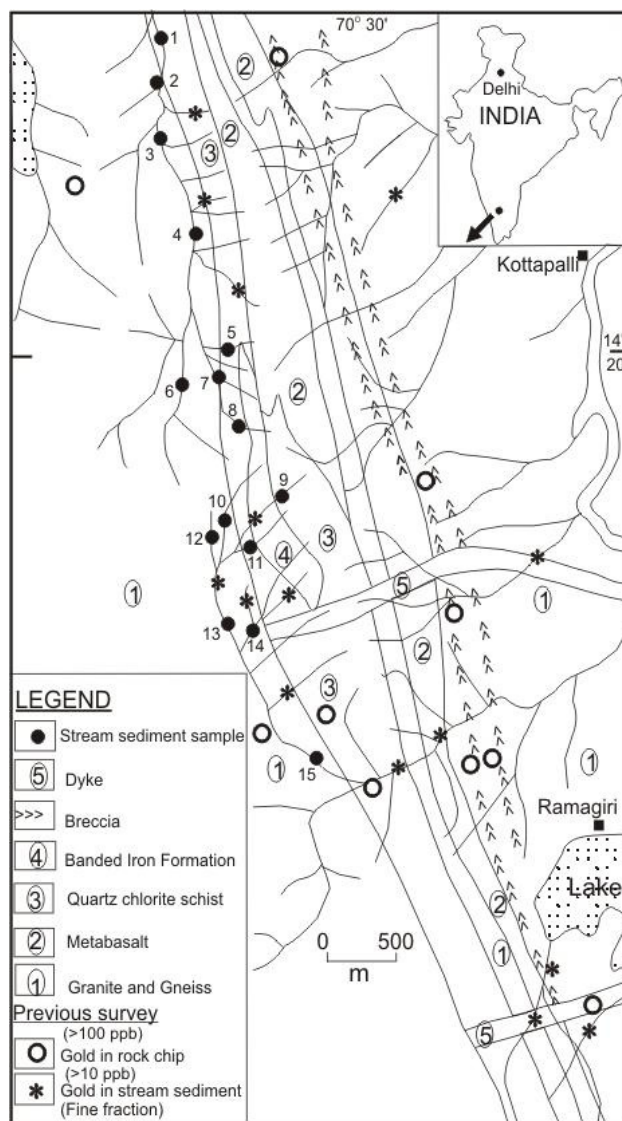
- a) The dilute cyanide solution react only the top layers of the gold grains not the complete grain.
- b) Leaching of gold in the presence of other metals like iron etc. may not be effective.
- c) It may not be effective in the case of invisible gold (Spry and Thieben, 2000) or trapped gold (Liang *et al.*, 1980) and
- d) Cyanide being highly poisonous, it has to be handled with care.

Keeping in view the above mentioned shortcomings, a new method is proposed in this paper. That is bulk leach extractable gold using hot aqua regia acids. It is denoted as BLEG-AR to differentiate it from BLEG (using cyanide). It is tested on 15 stream sediment samples collected from an area known for gold mineralization near Ramagiri, Anantapur district, Andhra Pradesh, India. The study area (about 15km<sup>2</sup>) is covered earlier by a regional stream sediment survey (about 100km<sup>2</sup>) covering the Ramagiri Gold Fields for gold by the author using minus 0.177 mm size fraction.

Geologically, the study area forms part of Ramagiri schist belt, locally and Ramagiri-Penakacherla schist belt, regionally. It is one of the greenstone belts in Precambrian Dharwar Craton. The belt forms a Y-shaped outcrop near Ramagiri village and surrounded on all sides by Peninsular Gneiss. The quartz bodies in the form of veinlets, pods, reefs etc., occur along foliation planes in the area. Three types of quartz

veins observed in the area are related to different generations and vary in color from grey to white. The gray fine-grained dull variety is generally considered to be gold bearing (Ghosh *et al.*, 1970).

Present study is confined to the western limb of the schist belt and the western part of the Kottapalli Block of Ramagiri Gold Fields. It is about 1-2 km north of Ramagiri town (Fig.1). The study area is dominated by granites, breccia, quartz-chlorite schist (with sericite) and Banded Iron Formation (BIF). The iron formation varies in thickness (about 100m) may be due to repeated folding.



**Fig.1.** Geological and stream sediment sample location map, Ramagiri Gold Fields, Anantapur district, Andhra Pradesh, India (after Ghosh *et al.*, 1970).

## Previous Survey

A stream sediment survey and a follow-up lithogeochemical survey were carried out in the Ramagiri Gold Fields covering both eastern and western limbs of the Ramagiri schist belt from Jibutal, in the south, to Chennabhavi (beyond Kottapalle, Fig.1) in the north. The stream sediment survey results (minus 0.177mm size fractions, hot aqua regia and MIBK treated) confirmed the presence of an ore body in both eastern and western limbs. The old workings at Jibutal, Chennabhavi, Kottapalle and working mine at Yappamana (all lie in the eastern limb) (not shown in the figure) correlate well with the anomalies recorded in the survey. Interestingly, the survey has also noted anomalies for gold in the western limb (passing Ramagiri village, Fig.1) which were not known earlier. The follow-up lithogeochemical survey further confirmed presence of gold mineralization in association with breccia, quartz-chlorite schist (Bheemalingeswara *et al.*, 1996) (Fig.1). Ore petrographic investigation indicated presence of pyrite, chalcopyrite, pyrrhotite, sphalerite and minor arsenopyrite in the auriferous zone in the Kottapalle Block of the Ramagiri ore zone (Deb and Bheemalingeswara, 2005). Among sulfides, pyrite has been the dominating sulfide mineral.

Selection of the area for the present study to test BLEG-AR method is quite appropriate because a) it is known for gold mineralization; b) iron hydroxide coating is quite conspicuous (as indicated in the previous geochemical survey); c) banded iron formation (BIF) is quite prominent; and d) pyrite is prominent among sulfide minerals.

## Sampling and Analysis

About 5-6 kilograms of -30 (-0.59mm) mesh size stream sediment samples (n=15) were collected after removing the top layer from each site (Fig.1). About 2 kg of the bulk sample was further sieved to obtain -30+50 (-0.59+0.297mm) and -80 (0.177mm) mesh size fractions. Later, the bulk (-0.59 mm), coarse (-0.59+0.297mm) and finer (0.177mm) fractions were further processed and analysed. Following is the procedure adopted to obtain the solutions.

- I. The bulk samples of -0.59 mm size were treated initially with 1+1 HCl, later with concentrated aqua regia and iso- butyl methyl ketone (MIBK). The purpose of this scheme is to eliminate the mineral phases like carbonates, remove iron hydroxide coating and liberate coating- bounded gold grains using 1+1 HCl and dissolve the liberated and other types of gold using aqua regia and transfer gold solution into MIBK organic solvent so that gold is transferred to MIBK. Since, all the available gold is finally transferred into the organic solvent, the volume of MIBK used becomes important rather than the volume of the hot acids.

The sample weighing 2 kg, was transferred into a glass beaker and treated initially with 250-500 ml of 1+1 HCl and kept on hot plate till the acid volume was reduced approximately to half. 150-250 ml of aqua regia was added and heated till the volume of the acid reduced to minimum. After cooling, about 200-300 ml double distilled water (DDW) was added to the beakers and mixed well and filtered into a 500ml volumetric flask. The flask was filled with DDW close to the mark after adding 10ml of MIBK organic solvent and mixed well. The top gold bearing MIBK layer was then transferred into a 100ml volumetric flask, denoted as **BULK**, for analysis. The organic layer which was showing intense red color for some of the samples, possibly due to high iron content, was repeatedly washed with dilute HCl and DDW and removed iron before analysis

- II.** The -30+50 (-0.59+0.297mm) mesh size fraction, weighing about 1kg was treated with bromoform to separate heavy mineral components (HMC) and run in Iso-dynamic separator, at standard settings (Bheemalingeswara, 1995a) to separate magnetic (M) and non-magnetic (NM) components. 100 gm of NM components was treated with aqua regia and MIBK. The solution thus prepared was denoted as **NM**.
- III.** The finer fractions (-0.177mm) weighing about 50gm were treated with aqua-regia and followed by MIBK and the solutions were denoted as **FINE**.
- IV.** All the solutions were analysed using Atomic Absorption Spectrophotometer (AAS) (Perkin-Elmer, 2380) in the Department of Geology, Delhi University. One gram of analytical grade chloroauric acid (E-MERCK) was dissolved using dilute hydrochloric acid and made the volume to 1 litre. This solution was used as standard/reference for gold analysis. From the stock solution of gold, different standards, 1, 2, 3, 4 and 5ppm were prepared and together with blank used to set AAS and suggested standard analytical conditions. Both standard and stream sediment solutions were treated with MIBK. In the case of gold standard (reference) one ml of 10 ppm gold was added to 100 ml MIBK to prepare 0.1 ppm gold standard. Likewise gold standards with different strengths were prepared and used to construct the working curve against which the unknown sample solutions were run. Since the standards fed were in ppm, the values thus obtained for samples were also in ppm. Known standards (as unknown) and stream sediment samples were re-run many times to check the reliability and reproducibility of the data. The precision of the data has been within the acceptable limits ( $\pm 10\%$ ). Data generated for fifteen stream sediment samples in the present study and data recorded in the previous survey are given in Table-1.

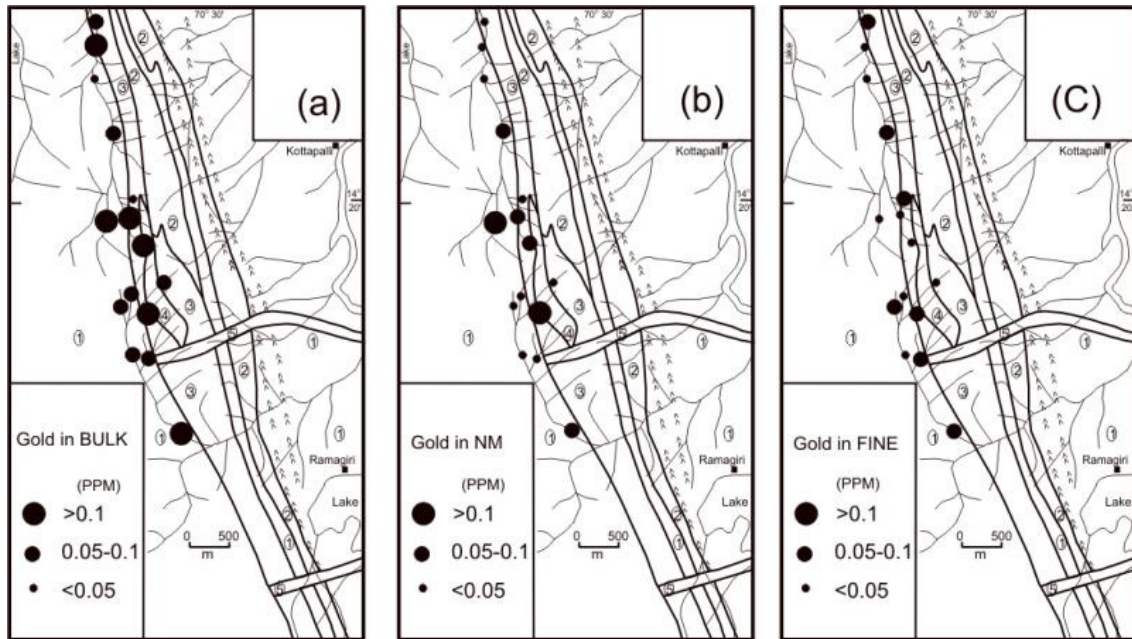
**Table-1:** Gold concentrations (ppm) in BULK, NM and FINE fractions stream sediment samples, Ramagiri Gold Fields (previous data is only for comparison).

S. No.	Bulk sample (BULK) (-0.59mm)	Magnetic component (-0.59+0.297mm)	Non-magnetic component (NM) (-0.59+0.297mm)	Fine fraction (FINE) (- 0.177mm)	Previous survey data (-0.177mm)
1	0.08	<0.01	<0.01	0.06	No data
2	0.16	0.09	0.03	<0.01	0.06
3	0.04	0.02	<0.01	0.02	<0.01
4	0.08	<0.01	0.03	0.06	0.02
5	0.04	<0.01	<0.01	0.06	0.04
6	0.45	0.11	0.20	< 0.01	0.03
7	0.12	0.08	0.06	0.05	0.04
8	0.14	0.03	0.08	< 0.01	<0.01
9	0.05	<0.01	0.02	< 0.01	<0.01
10	0.08	<0.01	0.04	< 0.01	<0.01
11	0.22	0.06	0.11	0.07	0.06
12	0.19	0.05	0.03	0.11	0.10
13	0.05	0.02	0.02	0.05	<0.01
14	0.08	0.05	<0.01	0.04	0.05
15	0.17	0.06	0.08	0.04	0.04

## Discussion

Gold data generated for BULK (-0.59mm), NM (-0.59+0.297mm) and FINE (-0.177mm) indicate presence of gold anomalies. They not only tally well with the earlier data (Bheemalingeswara *et al.*, 1996) and anomalies (Fig.1) but also show additional new anomalies compared to the previous survey (Fig. 2). Gold concentrations (maximum) vary significantly in bulk and size fractions *i.e.* 0.45 ppm in BULK, 0.20 ppm in NM and 90 ppb in FINE in the present survey. The values of FINE are comparable with the data of the finer fractions (100 ppb) of the stream sediments in the previous survey. In the same area, maximum value recorded for gold in rock samples is 2.47 ppm (Bheemalingeswara *et al.*, 1996).

Distribution of gold in the three different fractions/components is quite conspicuous. Bulk samples (BULK) show higher values for gold compared to both coarse (non-magnetic component) and finer fractions. Gold data obtained for finer fractions in both present and previous surveys, which tally well, amply indicate the reliability of the method. Gold values in the bulk sample and different fractions vary in the order of BULK>NM>FINE. Reproducibility of the data though a problem particularly in the case of coarse fractions, present data (with limitations) suggests that the values vary within 10-15%. Among the three fractions, both BULK and FINE seems to be more reliable compared to NM, where the values fluctuate significantly due to nugget effects.



**Fig.2:** Gold concentrations (ppm) in a. BULK; b. NM and c. FINE fractions of stream sediment samples, Ramagiri Gold Fields.

Iron oxide/hydroxide coating is quite significant on the grains in the study area. Its intensity is quantified approximately as 20%. Though it is difficult to say whether the coating is uniform or partial or selective, its effect or influence on the sample seems to be about 20%. The effect of these secondary iron oxide/hydroxide coatings will be in the form of 1) accumulation of small grains of magnetite, hematite, goethite etc, (Liang Pan

*et al.*, 1980); 2) fine to coarse grained particles of gold to get attached to these grains; and 3) growth of gold nuggets. The coatings also act like adhesive and influence different grains particularly small/fine grains including gold to stick together and attain bigger sizes. Higher the intensity of coating bigger the grain sizes and reduction in finer sizes. This affects proper recovery of different size grains during sieving and Iso-dynamic separation and thus causing errors in the data (Bheemalingeswara, 1995b). In addition, higher the intensity of coating over the coarse detrital grains, higher will be its ability to adsorb small/fine size detrital mineral grains (ferromagnetic or paramagnetic) on to its surface including gold. This is noticed when the samples were treated with dilute HCl to remove the coatings. Such fine magnetic or paramagnetic grains actually influence the separation process not the coating itself while running in Isodynamic separation. Once coating is removed, the grains become liberated and behave accordingly and achieve better separation.

Another problem that may still persist is the mineral inclusions like magnetite, ilmenite etc. in gold nugget or as nucleus for the growth of gold (nugget) (Chapman *et al.*, 2000; Bheemalingeswara, 2004). This kind of association is more common with dark colored minerals compared to the light colored minerals like quartz. Further, gold can also stretch into thin plates and fold around different mineral grains (Vasconcelos and Kyle, 1991) including magnetite, ilmenite etc. Dilute acid treatment in this case does not help. The survey programs, which intend to use coarse (particulate gold) or finer fractions particularly in areas where coating is conspicuous, need to take into account the above mentioned problems to achieve better results. Use of bulk leach extractable gold (BLEG) method (Beeson, 1995) and finer fractions (Xie and Wang, 1991) in place of traditional NM method though could overcome the problems like nugget effects, the question of total recovery of gold in the bulk or a particular fraction of interest still remained unanswered particularly where gold is invisible or secondary coatings are conspicuous.

In routine surveys, sample volumes in the case of coarse (-0.59+0.297mm) and fine (-0.177mm) will be generally about one kg and 200 gm respectively collected in the field after sieving the bulk sample. The proportion of these fractions is generally expected to be about 50 and 10% respectively in the bulk sample. This of course depends on the nature of the geological condition locally. This is an average figure obtained using the data from various case studies conducted by the author, predominantly the areas experiencing semi-arid conditions (central, west and south India). In all the cases, the sample volume used in the analysis was about 50 or 100gm. Since the volume of bulk sample used in the present analysis is about 2kg or more, the volumes of these fractions will also become more and the final solution prepared for analysis is 10ml. It means that gold available in BULK (out of 2kg) which includes all the fractions will be much higher compared to NM (100gm) and FINE (50mg) data. It is also important to note here that the BULK values are free from the secondary iron oxides and nugget effects and the recovery of gold is almost total and the reliability and reproducibility of the data is also better.

Bulk sampling and cyanide treatment (BLEG), though is a proven method and widely used to recover total gold in the sample, the question of its ability to bring gold into solution in the case of coatings, invisible gold etc., has remained unanswered. In the case of new hot aqua regia method suggested here, the problems related coatings, size of the grains, inclusions, nuggets, and invisible gold etc do not affect the gold recovery. Gold, irrespective of its type is brought into solution and the data reflects the total gold present in the sample.

Present method (MIBK-AR) of using bulk sample and treating with aqua regia though tested on only 15 samples, it has the potential to be an alternative to the existing methods in terms of total recovery of gold and reliability of the data. It will also take care of the problems related to the size fraction, processing, nugget effect, invisible nature, recovery and secondary iron oxide/hydroxide coatings etc. It may be best suited for orientation surveys where it can provide information to set parameters required for detailed survey.

## Conclusions

- BULK data shows anomalous values for gold and tally well with the previous survey data. Better contrast for gold is achieved for bulk (-0.59mm) compared to finer fractions.
- Data suggests that the selection of the size fraction in areas where iron oxide/hydroxide coating is conspicuous needs caution.
- Since BLEG-AR method does not suffer from nugget effects, coating, invisible, nucleus and mineral inclusion effects, the data reflects near total concentration for gold present in the sample irrespective of its forms.
- BLEG-AR method seems to provide better results than BLEG (using cyanide) at least in some cases.
- The method may also be tested using other sizes like -10 or -20 mesh.

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#### About the author



**Dr. K. Bheemalingeswara** is presently working as Associate Professor in Department of Earth Science, Mekelle University, Mekelle, Ethiopia. He has obtained his doctoral degree in 1988 from Delhi University, Delhi, India and involved in teaching and research in the fields of economic geology, ore geology, geochemical exploration, hydrogeochemistry, analytical geochemistry, mineralogy and crystallography. Since 2005 he is working in Mekelle University, Ethiopia. He published about 30 papers related to exploration geochemistry. He has received M. K. Raychaudhury memorial medal (jointly with Prof. Mihir Deb, Delhi University) for best paper of the year 1994 published in Indian Journal of Geology by Geological, Mining & Metallurgical Society of India.