Statistical Evaluation of Geochemical Au Sample Quality *

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Abstract

Exploration and mining activities are based on anomalous occurrence of minerals. The basic concept governing this high risk venture is '*no ore, no mining'*. Hence, the quality of anomalous sample or its accurate determination is of great concern to exploration and mining operations. Regarding erratic mineralisation such as gold (Au), anomalous pattern of no significance whatsoever may appear in geochemical sample data as a result of poor sampling, improper sample handling or error in analytical techniques among other causes. To prevent the frequency of these occurrences, quality control checks coupled with classical statistical probe can form an integral part of the checklist to eliminate these errors. Although duplicate results have often accompanied original Au assays in most analytical reports submitted by laboratories, it is not immediately known upon what basis the results need to be accepted or rejected. Often, some geologists accept results upon quick sight comparison.

A total of three hundred and ninety (390) geochemical soil samples from the Sefwi-Bibiani belt of Ghana together with some blanks and standards were subjected to statistical analysis after following rigorous quality control sampling protocols. The statistical models employed include outlier test, distribution and correlation analysis. The original and duplicate samples were then statistically compared using simple nested One –Way Analysis of Variance (ANOVA), the *Chi Square* Test and the Student's *t –Test.*

The ANOVA and the *t*–Tests revealed no significant analytical error. However, the other tests indicated multimodality of the populations as well as *batch effect* which culminates into significant procedural error. The investigation concludes that these systematic procedural errors if unchecked could mask true geochemical distribution.

1 Introduction

Analytical sample accuracy is of great importance to all exploration and mining activities since this forms the basis on which major decisions are made. Geochemical soil sampling programs sometimes encounter challenges concerning effective delineation of the orebody, partly due to the inaccuracy on the part of laboratory personnel responsible for assaying or the field crew. For this reason, quality control and assessment (QC/QA) protocols as well as sound statistical analysis of sample data may have to be adopted to establish precise geochemical signatures or guide exploration efforts (Bond, 2008).

Quality Control (QC) refers to standards, blanks, duplicate samples and repeats of previously prepared pulps that are all submitted to the laboratory with the geochemical samples of interest for analysis (Shaw, 1997). This is a system of routine technical activity to measure and control the quality of the inventory as it is being deployed to the laboratory. For such QC data to be accepted by an independent auditor, it is usually a requirement that such *blinds* be package in such a way that the laboratory cannot identify them.

Quality Assurance (QA) practice includes a planned system of review procedures conducted by personnel not directly involved in the inventory compilation or development process. It may also be defined as a written programme which describes the steps being taken to minimise sampling errors (Shaw, 1997). Written protocols which are usually defined include the sampling programme, the preparation of sub samples, the assaying procedures and the procedures and criteria for quality control. QC/QA protocols standardize procedures for collecting samples and obtaining related information useful for implementation. By employing these QA/QC procedures, the precision and accuracy of assay results from the laboratory can be guaranteed.

The difference between the original value and the duplicate value for any sample assay determination (or prediction) is termed as an error. The interest is in two aspects of this error. The consistent components of this error are termed *bias* and reflect the accuracy of the determination method. The random components of this error are termed *precision* and reflect the repeatability of the method. This is commonly expressed as standard deviation, relative standard deviation, and coefficient of variation.

Often geologists working on exploration and mine concessions for want of time and pressure often do not get enough time to crosscheck sample results from laboratories before values are used in computa-

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tion. In most cases little statistical evidence is generated to refute or accept assay results (Fletcher, 1981). Thus, if initial data are unchecked they may introduce errors that may seriously affect the final results. These errors could seriously influence the decision making process such as ore delineation and reserve calculation and could mar the viability of the project.

The purpose of this paper therefore is to evaluate statistical techniques and QA/QC protocols on geochemical samples from the Sefwi-Bibiani gold belt of Ghana as a case study. It is intended to assess the procedure for sample collection and statistically assess the original and duplicate samples from laboratory 'K' (to maintain anonymity), to appraise the quality of soil geochemical sample results.

2 Geological Settings

The rocks of the mining district are of the Birimian Supergroup, made up of metasedimentary and metavolcanic series that are associated with felsic to mafic intrusions (Fig. 1). The metasedimentary rocks consist predominantly of phyllite, tuffs and greywackes.

The phyllites are mostly lustrous and occasionally interbed with the greywackes. The metavolcanic series contain pyroclastic rocks and felsic lavas (Hirst & Junner, 1946). A little hornstone, generally accompanied by epidorite, occur in isolated places. Aplite pegmatite and porphyry veins are common in the metasedimentary rocks (Leube *et al*., 1990). The quartz veins are often developed in shear zones which trend parallel to the belt with dip at slightly flatter angles than the host rocks. Birimian greenstones crop out in the hills about a third of a kilometer west of the main reef channel.

Dykes of porphyry are common in the rock adjacent to the main reef zone and few occur within the reef zone. Characteristic minerals in the gold-bearing deposit include euhedral arsenopyrite, pyrite and trace of chalcopyrite, among others. Gold mineralisation often shows a close spatial relationship with zones of alteration such as carbonatisation, sericitisation and silicification.

3 Sample Classifications

(i) Original Sample

Original sample is the first pass or primary samples material from the soil location, outcrop exposure, core or RC cuttings. All initial analysis tests are often performed on the original samples and check analysis of randomly selected samples can also be carried out on them. Original field samples typically reflect a higher geochemical variability.

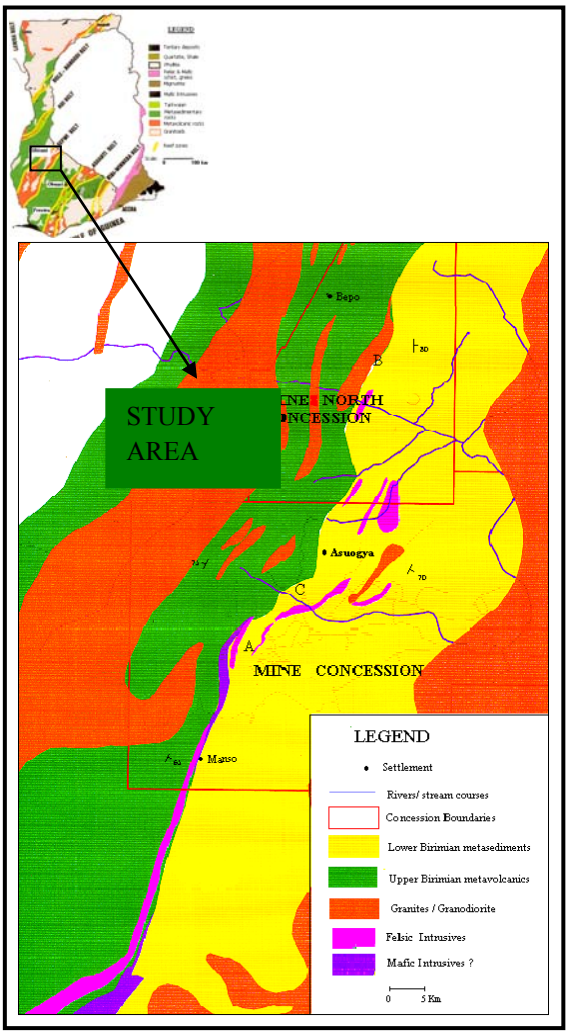

Fig.1 Geology of the Study Area (Modified after (Anon, 2000)

(ii) Duplicate Sample

Collecting additional independent sample from the same location or sample pulp is referred to as duplicate sample. This may come as field or laboratory duplicates. It gives an indication of the randomness or homogeneity of mineralisation that is naturally occurring.

Laboratory duplicates are made when the laboratory makes two separate pulps independently from the same sample. Comparison of original and duplicate samples gives an indication of valid laboratory preparation procedures and sample variability. It is desirable that at least 50 duplicate samples be collected in order to estimate sample error related to metal distribution within any specific deposit.

(iii) Blanks

Blanks or samples without mineralisation are often submitted with each batch of samples sent to the laboratory. The blank materials are collected from a

location known to be devoid of any mineralisation or purchased from a reputable supplier. Results from these samples indicate if there is any contamination introduced during the sample preparation or analytical procedures. It is appropriate that there should be approximately one blank for every 50 samples submitted to the laboratory. If any significant contamination is noted the analytical laboratory will be notified and corrective measures taken to resolve the error.

(vi) Certified Standards

Certified standards are sample pulps prepared, packaged and certified to contain known values of certain elements. The standards are prepared by a reputable laboratory that has validated their content. It is recommended that at least two standards be used with metal content that are representative of the deposit. Ideally, one standard should represent the expected mine grade and another that may represent a lower cut-off grade. In the early stages of exploration it may be more practical to prepare a high, medium, and low grade standard based on the known data at that time.

4 Quality Assurance and Quality Control Protocols

Standard reference materials otherwise known as quality control (QC) samples, is a collective name for standards, blanks and duplicate samples. Quality assurance (QA) is the documented procedures which describe the steps to be taken to minimise sampling errors (Shaw, 1997) and the sum total of sampling may be defined as sampling protocol.

QC/QA activities include general methods such as accuracy checks on data acquisition and calculations, measurements, estimating uncertainties, archiving information and reporting. It provides routine and consistent checks to ensure data integrity, correctness, and completeness. It identifies and addresses errors and omissions.

Some factors that cumulatively affect the precision of any assay result and often times the field data include the mass of material, the homogeneity of the material being assayed, the concentration of the component of interest, matrix effects due to other elements in solution, instrument calibration and drifting. Hence QC/QA system is designed to:

- ensure high samples quality and result reliability
- check both field and laboratory personnel responsible for sample collection and assaying
- standardize the processes of sample collection

5 Field Sampling

Geochemical original samples were taken on a rectilinear soil grid from the Sefwi-Bibiani concession with the beds striking 020°. Cross Lines for the grid were at 100m separation where sample points along the lines were spaced at 20m generally (Fig. 2). Thus, the sample interval was fixed by the possible width of the expected anomaly. The spacing was such that every economic anomaly could be intersected by at least two lines. Care was taken to ensure that the lines were not more than one-third of the minimum economic strike. The duplicate samples were taken from the 10th pit on every line. The assayed Au sample are in parts per billion (ppb).

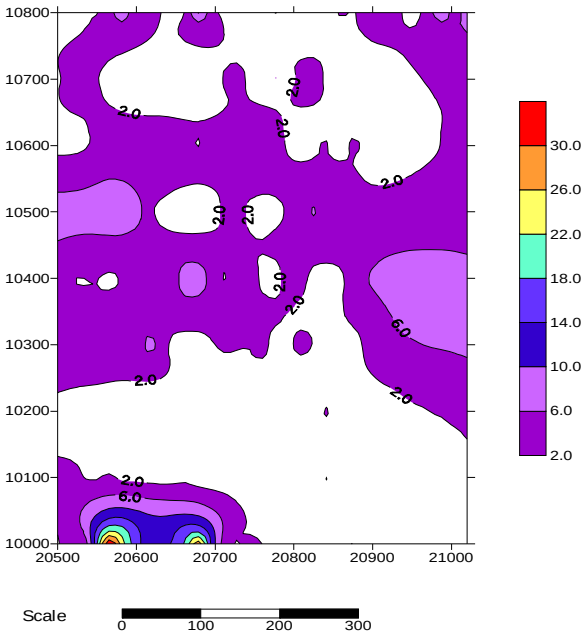

Fig. 2 Contour Map of Au Samples from the Study Area

6 Original and Duplicate Assay Data

The original and duplicate assay results of soil samples from the study area were analysed by a standard laboratory "K" (to maintain anonymity) (Table 1).

The assayed results were made available for the statistical investigation. One hundred and ninety five (195) sample data each from original and duplicate assay results, totaling three hundred and ninety (390) samples were used. Data validation was performed by plotting the sample points to ascertain reliability of assay values.

7 Results of Routine Statistical Methods

Various statistical methods could be used to analyse geochemical sample results. However, in this research the samples were first validated by testing for normal distribution. Showing positively skeweness, the samples were therefore log-transformed in an attempt to normalise them. Further, they were sub

Table 1 A Section of Analytical Geochemical Sample Results

Northings	Eastings	Original (ppb)	Duplicate (ppb)
20700	10260	7.64	7.36
20700	10280	8.05	10.03
20700	10300	8.33	6.45
20700	10320	8.87	7.87
20700	10340	9.42	9.89
20700	10360	9.69	8.26
20700	10380	9.70	10.12
20700	10400	10.00	9.89
20700	10420	10.20	10.40
20700	10440	10.80	10.50
20700	10460	11.90	10.19
20700	10480	13.20	10.81
20700	10500	14.90	11.95

jected to outlier test and correlation analysis. The original and duplicate samples were then statistically compared using simple nested One–way Analysis of Variance (ANOVA) and the student's *t –Test.*

(i) Detection of Outliers

The construction of scatter plot is the first step in the investigation of bivariate relations. It involves plotting the two variables on orthogonal axes. It is used to make subjective judgment about the general nature of the relation between the two variables. The outlier test was conducted at 5% level of significance using Moroney's t-factor diagram (Wellmer, 1998) .This gives perfect, weak or no linear relation. From this plot, isolated outlying values otherwise known as *lever effect* or *outliers* were detected but this was redressed by outright removal (Fig. 3).
The *outlier test* is also governed by the formula (Doerffel, 1967):

$$|X_a| \geq X_{diff} + S_{diff.} \cdot g \qquad \textit{Eq. (1)}$$

where 'X_{diff}' is the mean, 'S_{diff}' is the standard deviation and 'g' is the outlier threshold which is a function of the number of sample values.

$$|X_a| \geq 0.085 + (0.51 \times 3.75) = 2.0$$

As indicated, the abnormally high samples generating the *lever effect* were removed and the data was passed for normal distribution and correlation analysis.

(i) Correlation Analysis

Correlation is the measure of similarity between paired data. Two conceptually different categories form the basis for this statistical treatment. The first is the *R*-mode, which is the more traditional approach, deals with correlation between pairs of variables. The simple linear coefficient (*r*) lies between (-1) and (+1), where the absolute value of (1) means

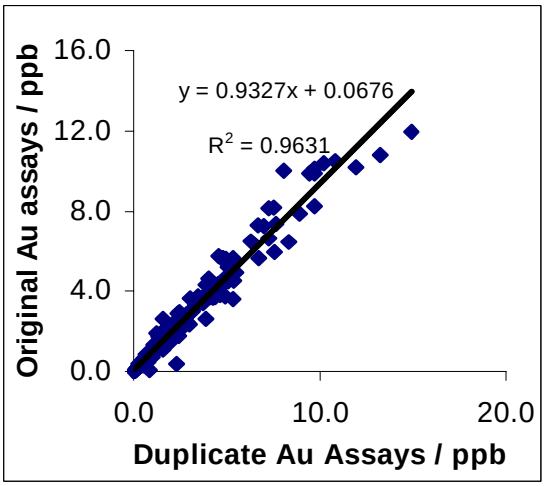

Fig. 3 Scatter Diagram Aiding Outlier Detection

perfect correlation and zero (0) mean no correlation. The other computational definition for correlation coefficient includes the Pearson and Spearman rank correlation. This analysis however uses the Pearson formula and gives a near perfect correlation as follows:

$$r = \frac{\sum xy - \frac{\sum x * \sum y}{n}}{\sqrt{\left[\sum x^2 - \frac{(\sum x)^2}{n}\right]\left[\sum y^2 - \frac{(\sum y)^2}{n}\right]}} \qquad \textit{Eq (2)}$$

$$r = \frac{86.67 - \frac{(20.94)(17.39)}{195}}{\sqrt{(85.76 - \frac{438.48}{195})(87.83 - \frac{302.41}{195}}} = 0.998$$

where *r*, being the Pearson's coefficient is calculated from the variables *x, y,* x^2 ,y^2 ,*xy* and *n*

(ii) Distribution Analysis

The most fundamental aspects of statistics lie in measure of central tendency and dispersion of unbiased samples. These parameters define the important characteristic of the probability density function of a data set. If unbiased estimate is observed, the comparable parameters of the population should show equal distribution pattern (Sinclair, 1976).

Histogram of the distribution of samples was therefore drawn. The samples appeared non-normal and apparently positively skewed. The sample data was thus log-normalised. There resultant histograms are shown in Fig 4a-b. The distribution showed multimodal pattern indicating that there was a possible mix of two or more sample populations.

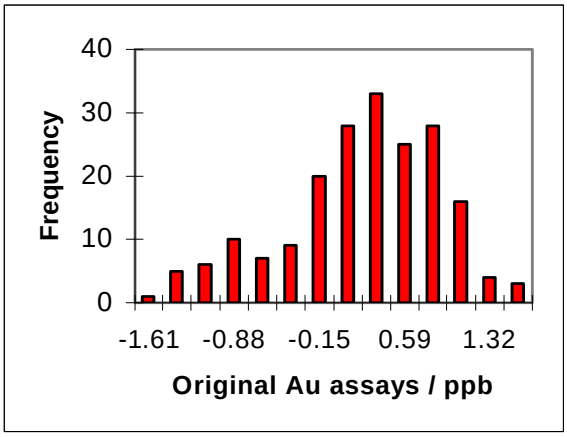

Fig. 4a Histogram Distribution of Sample Data

(iii) Analysis of Standard Reference Material

The result obtained from the sampling exercise was checked for precision and accuracy with the aid of a QA/QC protocols on the standards materials and scatter plot for the duplicate samples. Basically, +/-2SD and +/-3SD are considered as acceptable limits. The standards that fall beyond +/-3SD give a cause for re-assay. The results obtained indicated that the standards were largely below the expected value (Fig. 5).

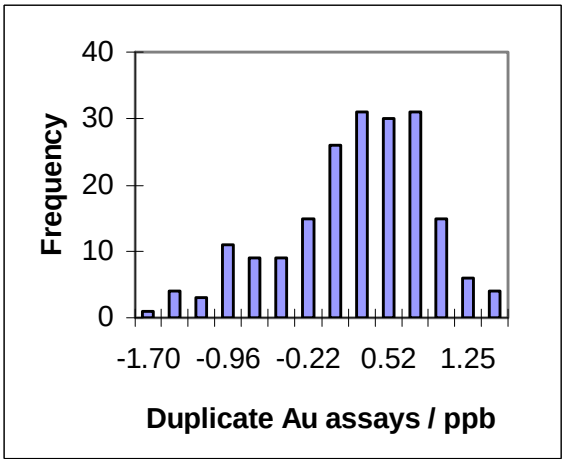

Fig. 4b Histogram Distribution of Sample Data

8 Results of Analytical Statistical Techniques

(i) Analysis of Variance (Anova) and Student t-Test

Analysis of variance (ANOVA) is used in verifying the similarity of population means using variance. It is a statistical technique which allows the decomposition of the total variance of a measurement into its constituent variances. In mineral exploration it is useful for quantifying the procedural error associated with measurement.

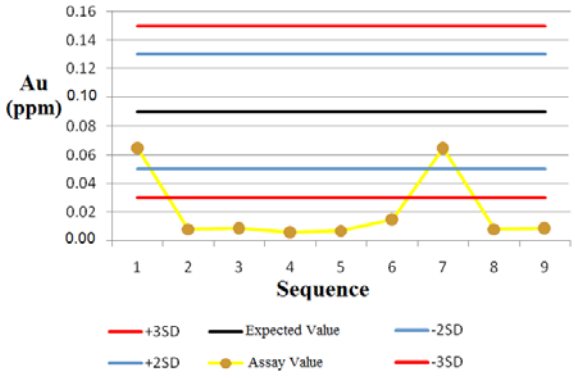

Fig. 5 Graphical Analysis of Standard Reference Material

Since variance and their estimators are additive, the variance estimate of the measurement (MS_t) is made up of (Davis, 1986):

$$MS_t = MS_b + MS_w \qquad Eq.\ (3)$$

where MS_w is the variance estimator within-group mean squares and (MS_b) is the variance estimator between–group mean squares.
The mean square terms are calculated via the corresponding 'sums of squares' as follows:

$$SS_t = \sum x^2 - \frac{(\sum x)^2}{n}$$

$$SS_b = \frac{\sum_1^i (\sum_1^j x)^2}{j} - \frac{(\sum x)^2}{n}$$

$$SS_w = SS_t - SS_b$$

$$MS_b = \frac{SS_b}{i-1}$$

$$MS_w = \frac{SS_w}{i(j-i)}$$

where i = the number of groups, j = the number of observations within each group and $n = ij$

It is then possible to perform the F-test to determine whether the populations from which the samples are drawn are significantly identical or otherwise using equation four.

$$F = MS_b / MS_w \qquad Eq.\ (4)$$

This is then compared with the critical F, (i-1) and {i (j-1)} values at 5% significant level.

Decision rule:

H_o: $\mu_o = \mu_d$

H_1: $\mu_o \neq \mu_d$

Accept if $F \leq F_c$, Reject if $F > F_c$

where F_c is the theoretical value.

Using the formula, the variance estimate of the measurement is 0.101

(iii) t-Test

The uncertainty introduced into estimate based on samples may be accounted for by using a probability distribution which has a wider *spread* than the normal distribution. One of such distribution is the *t-distribution* which is similar to normal distribution. The student *t*-test is another useful method for assessing whether there is significant difference (error) between the means of two samples. It establishes the likelihood that a given sample may be a member of a particular population with specific characteristic for testing hypothesis about the equivalency of two sample populations.

Two types of errors are distinguished:

- Random errors
- Systematic errors

Random errors cannot be avoided but is important to determine if systematic error is present (Wellmer,1998). The *t*-test therefore helps to determine if there is a difference between the two sample populations and whether the error is significant. The formula for calculating the *t*-factor is given by (Moroney, 1970):

$$t = \frac{|x_o - x_d|\sqrt{n-1}}{S_{diff}} \qquad Eq.\ (5)$$

where x_o and x_d are the means of original and duplicate samples respectvely, s_{diff} is the standard deviation and n is the number of observations.

Decision rule:

Ho: $\mu_o = \mu_d$

H_1: $\mu_o \neq \mu_d$

Reject if : $t > t_{\alpha/2}$

Accept if: $t \leq t_{\alpha/2}$

where $t_{\alpha/2}$ is the theoretical value.

Computed results from the available sample data using the ANOVA and the *t*-test empirical formula gave (Table 2):

Table 2 Computed ANOVA and *t*-Test Results

Parameter	Calculated Results	Critical value@95%
ANOVA	0.101	1.000
t-Test	2.120	1.960

(ii) The Chi-Square Test

The *Chi-Square* test (X^2) is non parametric in nature. It means it application is not dependent on the particular probability density function for the variable being tested. One of its principal application is to test whether or not a sample might have been drawn from a population with degree of freedom of a particular form. It is applied to test the model for *good fit*. The advantage of this distribution is that it can be used to test for nominal and ordinal data. To show a model with a good fit (Tables 3a and 3b), the difference between o_i and e_i should not look too large.

Thus,
$$X^2 = \frac{\sum_{i=1}^{n}(o_i - e_i)^2}{e_i}$$

where e_i is the estimated value for the slot at intersection of row '*i*' and o_i is the observed value for the slot of intersection of row '*i*'

Table 3a X^2 Test Results for Original Samples

Upper Class (Vcl)	Vcl - V	Score (Z)	Area under curve (%)	e_i %	e_i N = 195	o_i	$o_i - e_i$	$(o_i - e_i)^2$	$(o_i - e_i)^2 / e_i$
-1.611	-1.718	-2.62	49.41	0.59	1.15	1	-0.15	0.02	0.02
-1.367	-1.474	-2.25	48.78	0.63	1.23	5	3.77	14.22	11.58
-1.122	-1.229	-1.87	46.93	1.85	3.61	6	2.39	5.72	1.59
-0.878	-0.985	-1.50	43.32	3.61	7.04	10	2.96	8.76	1.25
-0.634	-0.741	-1.13	37.08	6.24	12.17	7	-5.17	26.71	2.19
-0.389	-0.496	-0.76	27.64	9.44	18.41	9	-9.41	88.51	4.81
-0.145	-0.252	-0.38	14.80	12.84	25.04	20	-5.04	25.38	1.01
0.099	-0.008	-0.01	0.40	14.40	28.08	28	-0.08	0.01	0.00
0.343	0.236	0.36	14.06	14.46	28.20	33	4.80	23.07	0.82
0.588	0.481	0.73	26.73	12.67	24.71	25	0.29	0.09	0.00
0.832	0.725	1.11	36.65	9.92	19.34	28	8.66	74.93	3.87
1.076	0.969	1.48	43.06	6.41	12.50	16	3.50	12.25	0.98
1.321	1.214	1.85	46.78	3.72	7.25	4	-3.25	10.59	1.46
			50.00	3.22	6.28	3	-3.28	10.75	1.71
Total					195.00	195			31.29

Table 3b X^2 Test Results for Duplicate Samples

Upper Class (Vcl)	Vcl - V	Score (Z)	Area under curve (%)	e_i %	e_i N = 195	o_i	$o_i - e_i$	$(o_i - e_i)^2$	$(o_i - e_i)^2 / e_i$
-1.7	-1.789	-2.68	49.63	0.37	0.72	1	0.28	0.08	0.11
-1.454	-1.543	-2.31	48.96	0.67	1.31	4	2.69	7.25	5.55
-1.208	-1.297	-1.94	47.38	1.58	3.08	3	-0.08	0.01	0.00
-0.962	-1.051	-1.58	44.29	3.09	6.03	11	4.97	24.75	4.11
-0.715	-0.804	-1.21	38.69	5.6	10.92	9	-1.92	3.69	0.34
-0.469	-0.558	-0.84	29.96	8.73	17.02	9	-8.02	64.38	3.78
-0.223	-0.312	-0.47	18.08	11.88	23.17	15	-8.17	66.68	2.88
0.023	-0.066	-0.10	3.98	14.1	27.50	26	-1.49	2.24	0.08
0.269	0.180	0.27	10.64	14.62	28.51	31	2.49	6.21	0.22
0.515	0.426	0.64	23.89	13.25	25.84	30	4.16	17.33	0.67
0.762	0.673	1.01	34.38	10.49	20.46	31	10.54	111.19	5.44
1.008	0.919	1.38	41.62	7.24	14.12	15	0.88	0.78	0.06
1.254	1.165	1.75	45.99	4.37	8.52	6	-2.52	6.36	0.75
			50.00	4.01	7.82	4	-3.82	14.59	1.87
Total					195.00	195			25.84

The routine statistical results obtained from the original and duplicate samples show close correlation with respect to distribution and characteristics passing the 95% and 99% significant level tests. The shape of the histograms of the actual data being asymmetrical, approached Gaussian distribution after log-transformation. The Pearson's correlation coefficient '*r*' gave a value of 0.998. The coefficient of only 0.002% bias shows a very high positive correlation of the original and duplicate samples.

From Fig. 3, the scatter plot indicates the likelihood

of *lever effect*. To address this, the high sample values were rejected and the data taken into further analytical considerations. Thus, applying the Doerffel (1967) empirical formula, the data was subjected to *outlier* test where the mean 'X_{diff}' and the standard deviation 'S_{diff}' were calculated without the high values causing the lever effect, the observed outlier value was 2.39 and this was still higher than critical value of 2.0. Hence, the sample data could be rejected, re-sampled, re-analyse or given undoubtedly convincing reason why the response was such.

Again, the distribution analysis from Fig. 4 shows multi-modality of the populations. This implies a procedural error. Several reasons for the procedural error could be assigned but the critical one is *batch effect, (*i.e., samples may have been taken from different formations or lithological units and lumped together as one sample batch to the laboratory.) It could have risen from sporadic high grade values amidst excess of low grades or vice versa. It could also have resulted from mix up of samples from different lithological units, sub-sampling or poor sample packaging and labeling. Analysis of standard reference material also revealed that the expected standards fell beyond +/-3SD and this gives cause for re-sampling or re-assay. It is clear from the discussions that the multiple populations, as well as the lever effects detected in the data are at best procedural systematic errors rather than analytic errors.

The summary of the statistical evaluation as presented in Table 2 gives ANOVA values of 0.101. The significance of this ratio is assessed by means of *F*-ratio at selected significance levels. At the 5 % significance level, the critical *F*-values were 1.0. This value exceeds the calculated of 0.101. This implies therefore that, there is no significant difference between the means of original and duplicate samples. The *null* hypothesis can be accepted. It implies therefore that there is no significant analytical error.

The *t-test* gave a calculated t-factor of 2.12 (Table 2). This value exceeds the critical range at 5% significant level which is 1.90. The null hypothesis is thus rejected. The implication is that there is a significant difference between the sample means at 5% level of significance. This means there is a possible systematic (procedural) error associated with the sample populations.
From the X^2-test, the distribution of original and duplicate data sets gave values of 31.29 and 25.84 respectively. At 95% confidence level, the value was 18.3 *(*X^2_{95}= 18.3) and at 99% confidence level, it increased to 23.2 (i.e. X^2_{99} = 23.2). Since the essence of the test is to show good-fit, the estimated result is expected to be equal or preferably lower than the critical value of 95% and 99% confidence levels. There is no reasonable agreement between the estimated percentile frequencies (e_i) and the observed frequencies (o_i). This may be attributed to a batch effect resulting from the sporadic high Au assay values amidst excess of low grade values. However, this does not mean that there is no correlation, it simply means that the test for *good-fit* has failed.

9 Conclusions and Recommendations

9.1 Conclusions

The following conclusions are thus drawn from the analysis of statistical discussions:
The data failed the goodness-fit test. The double fit registered by the sample data revealed a possibility that certain factor might have played a role in affecting a good fit. This is likely to be a batch effect resulting from lumping high and low grades.

The magnitude of the means for the sample population showed rejection of the null hypothesis as indicated by the *t*-test. However, there was enough evidence to conclude that the two data sets (original and duplicate) came from similar population within the critical limits of 0.95% and 0.99% confidence levels. Since the data passed the correlation test registering positive correlation of 0.998, it implied that analytical error was insignificant in the data, thus, assay results received from the laboratory were reliable from statistical consideration.

9.2 Recommendations

The study recommends that:
Systematic procedural errors resulting from batch effect, sample mishandling, etc., should be checked since it can mask true geochemical distribution in the field.

Samples from high and low anomalous zones should not be lumped together and submitted to analytical laboratory. They should be separated because effect due to smearing from other samples may generate *false* hope.

In order to check laboratories against unwarranted analytical bias, routine analysis of limited samples should be selected at random from previous batch and added to the batch being submitted to the same laboratory for re-assay.

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