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Comparison Assessment of Laboratory Subsampling Methods

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Abstract

This work evaluated the performance of the Rotary sample splitter (RSS) and Jones riffle splitter (JRS). Binary mixture of iron chips and sand was used as the test sample and four different tests were conducted to produce four subsamples. For each case, the iron chips were separated from the subsample with a magnet and the percentage weight was determined. The results obtained was analyzed statistically and it was observed that the mean value for the RSS is 33.2% and that of JRS is 28.7%, the variance for the RSS is 1.26 and that of JRS is 2.44. The standard deviation for RSS and JRS is 1.1225 and 1.5621 respectively, the coefficient of variation for RSS and JRS is 3.38 and 5.44 respectively. The bias for the RSS and JRS are the same (0.1%). The bias obtained in this study is lower than the maximum acceptable error for the two devices which indicated that the Rotary splitter and the Jones splitter used in this study produced representative subsamples. The Rotary sample splitter produced lower variance between subsamples compared to the Jones riffle splitter thus, it is prefer due to its accuracy, precision and efficiency.

***Keywords**:* Bias, Precision, Representative, Sample, Subsampling, Variance

1. INTRODUCTION

Sampling takes place successively to obtain representative smaller masses from larger masses of material. A correct sample of the lot is a subset of the original mass collected using correct sampling practices with the intent of selecting a representative sample that mimics the lot in every way. Subsampling is simply a repetition of this selection process whereby the sample now becomes the new lot and is itself sampled. A subsample is simply a sample of a sample. Generally, subsample is the smaller mass that is taken from the larger mass during the sampling step (Gerlach and Nocerino, 2003).

Sampling in the laboratory, commonly called laboratory sub-sampling, has the same issues as field sampling (Ramsey and Suggs, 2001). If the sub-sampling is not performed in a representative manner, a representative field sample will be rendered non-representative in the laboratory and yield non-representative data resulting in incorrect decisions (Walsh *et al*., 2002). It is often found that divided solids (powders, granules, etc) segregate by size when they are agitated or shaken. Small particles tend to collect towards the bottom of the container while larger ones on top, this is known as "*Brazil Nut Effect*". Heaps tend to accumulate larger particles towards the outside as they roll over smaller particles. Therefore, when taking sub-samples it is important to eliminate the effects of such segregation. If this precaution is not taken, the results of particle sizing, surface area measurements, porosity measurements and other analytical results can be severely biased. When inaccurate results are scaled up to industrial sized applications, the entire processes can fail to achieve their desired end results.

The "Golden Rules" for sub-sampling put forth by Allen (1981) simply state that the sample(s) should be taken when the powder is in motion (i.e. a powder stream), and the entire cross section of the entire stream shall be sampled many times. If this is not done when the container is loaded*,* it should surely be done when the container is emptied*.*

Some of the common methods of sample division include: Grab sampling, Coning and Quartering, Chute-type sample splitters and Rotary riffles. The performance of different powder sub-sampling methods is demonstrated by the data below:

Table 1: Estimated Maximum Error of various sub-sampling methods (Allen, 1981)

Sub-sampling Devices	Estimated Maximum Error %
Cone and Quarter	22.7
Scoop	17.1
Table splitter	7.0
Chute splitter	3.4
Rotary riffler	0.42

The Jones riffle splitter (also called a chute splitter, Jones splitter or sample splitter) is one of the common mechanical methods of subsampling. It is a device having an equal number of narrow sloping chutes with alternate chutes discharging the sample in opposite directions into two collection bins (Figure 1). The use of the riffle splitter as a sub-sampling device is done by pouring of materials through the splitter, after the sample is passed through the splitter, one collection pan is replaced with a clean pan. The material in the 'replaced" pan, which contains about one-half of the original sample, is then passed through the riffle splitter again thereby reducing the volume in the clean pan to one-quarter of its original sample volume. This process of sample reduction is repeated until the desired weight or sample size is obtained (Schumacher *et al*, 1990).

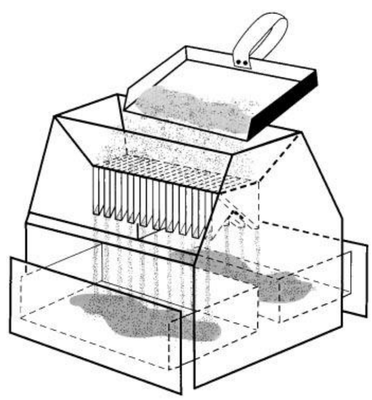

Fig. 1. The basic components of a Jones riffle splitter (Gerlach and Nocerino, 2003)

Another subsampling approach is to use a vibratory feeder to distribute the bulk sample into a number of wedge shaped containers as shown in Figure 2. This device is sometimes called a "spinning riffle" or "rotating sampler" and it is used for extracting representative samples from dry granular or powdered material (Wills, 2006). The preferred method of using this device is to fill a mass flow hopper, in such a way that little segregation occurs, by avoiding the formation of a heap. The table is then set in motion and the hopper outlet opened so that powder falls into the collecting boxes. The use of a vibratory feeder is recommended to provide a constant flow rate.

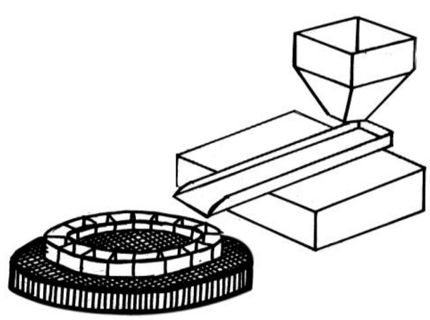

Fig. 2. Rotary sample splitter

This study is aimed at comparing the performance of the Rotary sample splitter to that of the Jones riffle splitter. The outcome will assist a mineral analyst to choose an appropriate laboratory sub-sampling technique that will produce samples that are homogeneous and representative of the bulk sample.

2. MATERIAL AND METHODS

2.1 Materials and Equipment

The materials and equipment required for this work are: iron chips sample, sand sample, bar magnet, Rotary sample splitter, Jones riffle splitter, weighing balance, sample container, recording book and pen

2.2 Sample Preparation

Binary mixture of iron chips and sand were prepared, the sample was thoroughly mixed to produce a homogenous mixture which was used as the test sample.

2.3 Experimental Procedure for Rotary Sample Splitter

The Rotary sample splitter was switched on and the binary mixture of iron chips and sand was fed into the machine through the hopper. The materials were distributed into the rotating sample containers until the hopper was emptied, then the machine was switched off. One of the sample containers was removed and the content spread on a plane surface, a magnet was then used to separate the magnetic constituents of the subsample. Iron chips were attracted to the magnet while the sand was left behind; the two materials were collected separately in different containers. The quantity of iron chips and sand collected were then weighed and recorded. The other subsample containers were also removed and the constituents were separated and weighed separately. The experiment was repeated four times to have a total of four different tests (Test 1, 2, 3 and 4).

2.4 Experimental Procedure for Jones Riffle Splitter

The mixture of the sand and iron chips was poured evenly into the hopper of the Jones riffle splitter. The samples then flowed through the alternately arranged passages in the opposite directions (chute/riffle bank) into the two collecting pan under the dividing head outlet. The feed sample was then divided into two representative sub-samples. One of the sub-samples was then spread on a plane surface and the bar magnet was placed very close to it in order to separate the iron chips from the sand. The iron chips that were collected by the magnet was placed inside a container and the sand was placed in another container. The two samples were thereafter weighed separately as earlier done and the results were recorded. This procedure was also repeated thrice in order to re-split the other portion of the splitted sample. All the results obtained were recorded.

2.5 Determination of the Percentage Composition of the Sample Constituents

The binary mixture of iron chips and sand has a true value of 33.3% iron chips in the case of Rotary Sample Splitter and 28.6% iron chips for Jones Riffle Splitter.

The percentage composition of the constituents in the parent sample and subsamples were calculated using equation 1 and 2.
Percentage (%) of iron in the parent sample or subsample = $\frac{Mi}{M} X\ 100\%$ (Eq. 1)

Percentage (%) of sand in the parent sample or subsample = $\frac{Ms}{M} X\ 100\%$ (Eq. 2)

Where, M_i = mass of iron in the parent sample or subsample
M_s = mass of sand in the parent sample or subsample
M = mass of the mixture of sand and iron chips in the parent sample or subsample.

The results obtained were further examined statistically to estimate the arithmetic mean of the percentage weight of iron in the subsample, the variance, standard deviation (S.D.), coefficient of variation (C.V.) and bias (sample error). The results are presented on Table 2 – 6.

Equations 3 – 7 were used in the statistical calculations:

Arithmetic mean, $xa = \frac{\sum x\,i}{n}$ (Eq. 3)

Variance, $V = \frac{\sum(xa - x)2}{n-1}$ (Eq. 4)

Standard deviation (S.D.), $\sigma = \sqrt{Variance}$ (Eq. 5)

Coefficient of variation (C.V.) $= \frac{100\sigma}{xa}$ (Eq. 6)

$Bias, E = Mean\ value - True\ value$ (Eq. 7)

Where, n = number of measurements
x = percentage weight of iron in the subsample
xa = arithmetic mean of the percentage weight of iron in the subsample

3. RESULTS AND DISCUSSION

The results obtained when four subsamples taken from four different tests performed using the Rotary sample splitter (RSS) and the Jones riffle splitter (JRS) were evaluated are presented on Table 2. From the subsamples taken, the weight of iron chips present was obtained; this was then used to calculate the percentage weight of iron chips in the subsamples for the two subsampling methods.

From Table 2, the percentage weight of iron chips in the subsamples produced by the Rotary sample splitter for Test 1 is 32%, Test 2 is 34.7%, Test 3 is 32.9% and that of Test 4 is 33.3%. The value ranges between 32% - 34.7%. This implies that the variation from the true value of 33.3% is low. The percentage weight of iron chips in the subsamples produced by the Jones riffle splitter can also be deduced from Table 2. The value for Test 1 is 27.1%, Test 2 is 28.1%, Test 3 is 28.7% and Test 4 is 30.8%. The range of value is between 27.1% - 30.8%. This also shows that the variation from the true value of 28.6% is also low.

Table 2: Percentage weight of iron chips in the sub-sample from the Rotary splitter and Jones splitter

Test No.	% Wt. of iron chips in RSS subsample	% Wt. of iron chips in JRS subsample
1	32.0	27.1
2	34.7	28.1
3	32.9	28.7
4	33.3	30.8

The summary of the results obtained from the statistical calculations conducted for both the RSS and the JRS is presented on Table 3. The mean value of the percentage weight of iron chips in the subsamples obtained from RSS is 33.2% while that of JRS is 28.7%. The variance in the case of RSS is 1.26 and that of JRS is 2.44. This implies that the JRS gives higher variance compared to that of RSS.

The standard deviation of the subsamples produced by the RSS is 1.1225 while that of JRS is 1.5621. For an ideal Rotary riffle and chute-type sample splitter, the standard deviation is 0.125% and 1.01% respectively (Khan, 1968). For both cases the standard deviation is higher than that of the ideal device.

Table 3: Summary of statistical results obtained for the two subsampling methods

Subsampling methods	Arithmetic Mean xa (%)	Variance V $(\%)^2$	Standard deviation, σ (%)	Coefficient of variation C.V. (%)	Bias (Error) E (%)
R S S	33.2	1.26	1.1225	3.38	0.1
J R S	28.7	2.44	1.5621	5.44	0.1

The coefficient of variation for the RSS and JRS are 3.38% and 5.44% respectively. This means that if the test is repeated several times, the variation in the results will be minimal although the coefficient of variation for JRS is higher than that of RSS. The variance and the coefficient of variation is a measure of precision or reproducibility, they are also the most important measures for variability between test results. A series of measurements can be precise but not adequately represent the true value. If there is no bias in the sampling regime, the precision will be the same as the accuracy (Gupta and Yan, 2006).

The bias for the two subsampling method is the same (0.1%) and on comparing this with data presented on Table 1, where the estimated maximum error for chute splitter and Rotary riffler are 3.4% and 0.42% respectively. It can be deduced that the bias obtained in this study is lower than the maximum acceptable error for the two devices. This is an indication that the Rotary splitter and the Jones splitter used in this study produced representative subsamples. The presence of bias is a major problem in sampling, because it does not average out over time. In contrast to poor precision, which can be improved by calculating the average of the results from replicate sampling, sample preparation and analysis, no amount of replicate sampling, sample preparation and analysis will eliminate bias once it is present. The final result is just a more precisely incorrect analytical value. Consequently, minimizing or preferably eliminating biases is in many respects more important than improving precision.

4. CONCLUSION

For any analytical work, sub-samples must be both accurate and precise, minimizing or preferably eliminating biases is more important than improving precision. The sub-sampling equipment used in this study was locally constructed; the Rotary sample splitter and the Jones sample splitter are very suitable methods for dividing material into representative samples. However, the Rotary sample splitter is more prefer due to its accuracy, precision and efficiency, it produces lower variance between samples, and can produce a large number of samples in a single operation. The two sub-sampling devices gave an intermediate performance indicating that they are suitable for routine and non-critical works.

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