

Sampling Techniques

Introduction

Reliable powder sampling is obligatory for all particle characterisation procedures. The sample collected from the bulk must be representative of the entire physical and chemical characteristics of the system in order for derived data to contain any meaning. According to the 'Golden Rule of Sampling'¹:

1. A powder should be sampled when in motion
2. The whole sample stream should be taken over many short time increments, rather than part of the stream being taken for the whole of the time.

Bulk Sampling

Sampling issues arise due to powder segregation. Variation exists in different regions of a heap or bag and also between sample containers. If static sampling must be employed, samples should be taken from many random positions in different bags or containers, and later combined to form as representative a sample as possible. In the case of heap sampling, if possible the heap should be mixed prior to sampling.

Sub-sampling

There are a variety of different sub-sampling methods which can be used to turn a bulk sample into a more manageable laboratory sample.

Cone and quartering

The powder is formed into a cone shaped heap on a flat surface, before being flattened with a spatula and divided into four identical volumes. One portion is taken and the entire procedure is repeated until only 1/16th of the original volume remains. This method is heavily operator dependant and is only suitable for powders with poor flow behaviour and thus little segregation. Large particles will tend to roll to the outside edges of the heap as it is made, while fine material will adhere to the working surface, thus narrowing the final overall size distribution.

Scoop Sampling

The operator manually scoops a small portion of powder from the bulk sample. Only appropriate

for homogenous materials with poor flow characteristics.

Table Sampling

The sample moves down an inclined plane, prisms divert and control the flow so that aliquots of sample fall through holes in the table. The powder that is collected at the end of the table is the required sub-sample (figure 1). Initial feed should be representative and mixed thoroughly, to eliminate the main source of variation.

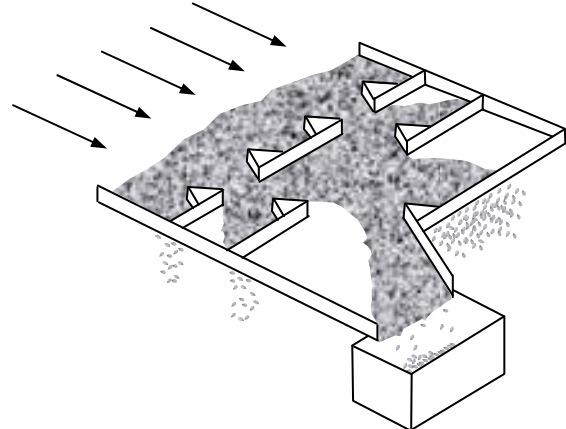


Figure 1: Table sampling

Chute

The sample is introduced into a V shaped trough, feeding through a number of chutes, and collected in two trays on either side of the trough. The procedure is repeated with the powder in one tray, until the desired amount is obtained. The trough must be loaded without causing sample segregation.

Spin Riffling

A spinning riffler (see figure 2) divides the sample into a series of glass containers. Powder is transported from the hopper, via a vibratory chute, into a series of containers rotating at a constant speed. The sample flows into the receivers at a constant rate causing them to be filled uniformly. The drawback to this method is that the sample volume is relatively small, handling powders in grams and milligrams.



Figure 2: Spinning riffler

For ease of comparison, a summary of the advantages and disadvantages of each sampling technique is given in table I.

Table I: Sampling technique comparison

Method	Advantages	Disadvantages
Cone & quarter	Good for powders with poor flow characteristics	Operator dependent
Scoop	Reliable for homogenous and non-flowing powder	Particle segregation
Table	Separates a large quantity of material	Initial feed dependant
Chute	Reduce powder sample by half after one pass	Operator bias
Spinning riffler	Reliable for free flowing powders	Inability to do large quantities efficiently

A paper by Allen and Khan² details a study regarding the reliability of each of the sampling devices mentioned in this application paper, and the results are given in table II. It is clearly seen that the spinning riffler is the most reproducible method of sub-sampling, with cone and quartering giving the largest standard deviation.

Table II: Sampling technique reliability

Method	Relative Standard Deviation (%)
Cone & quarter	6.81
Scoop	5.14
Table	2.09
Chute	1.01
Spinning riffler	0.125

Conclusions

The most reproducible method of sub-sampling is governed by the characteristics of the material. Using an incorrect method will result in a particle size distribution not representative of the bulk material.

References

- ¹ Allen, T., Particle Size Measurement, ed. R. Davies, 1996, Kluwer Academic Publishers, 824
- ² Allen, T. and A.A. Khan, Critical evaluation of powder sampling procedures, The Chemical Engineer, 1970, pp. 108 - 112

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