



The Importance of Using Correct Sampling Procedures in Obtaining Representative and Meaningful Analysis Results

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Reliable analysis results require many things including a good analyzer, a good analyst, and what is often overlooked, a good sample. For solid samples, the sample presented to the analyzer should represent as closely as possible the characteristics of the initial bulk sample.

The Challenge

Representative sampling of solid materials is a critical first step in analysis because mistakes made at this point cannot be corrected. The goal is to develop a protocol that allows a small amount of material to be extracted from the bulk material while at the same time preserving the physical and chemical characteristics of the entire bulk material. While it may be impossible to achieve 100% accurate sampling, a method can be established that provides the maximum possible accuracy and is both practical and simple to perform.

Size Reduction

If we take calcium carbonate (limestone) as an example, initial size reduction would be made with a jaw crusher large



Figure 1: Retsch PT 100 – working principle.



Figure 2: Retsch ZM 200 ultracentrifugal mill and BB 200 Jaw Crusher.

enough to handle the largest feed size present in the material. A popular choice is the Retsch BB 200, which can take material up to 90 mm and reduce it to less than 2 mm, although for limestone, the target is kept at 10 mm to avoid material sticking in the grinding chamber. While this will produce material small enough for the fine grinding device, a sampling step is often necessary to maintain the representative nature of the material.

Laboratory size sample dividers are available for dividing the material into small enough aliquots for fine grinding. Examples include the benchtop PT 100 sample divider, illustrated below, and PK 1000 tube divider for larger bulk samples.

There are various mills for fine grinding of hard, brittle materials including ball

mills, ring, and disc mills. Limestone, which is much softer (with a Mohs hardness of 3), can be quickly and easily ground to the required size using the world-renowned Retsch ZM 200 ultracentrifugal mill.

The limestone example illustrates the issues that need to be addressed in developing effective sampling programs. As with size reduction, there are many choices for sampling methods, but in this case, the decision is easier. Studies have been conducted comparing the sampling techniques and their errors. One performed by Allen and Kahn in 1970 showed the following results:

Table 1: Relative Standard Deviation in Sugar-Sand Mixture

Cone & Quartering	6.81
Table Sampling	2.09
Chute Riffing	0.125
Spin Riffing	1.01

Conclusion

Sample preparation is not an exact science, but there are theoretical approaches that address sampling errors. However, their implementation must be tempered by real-world experiences and practicalities. By applying a common sense approach, sampling methods can be developed that will maintain the integrity of the sample from the bulk material all the way to the analysis.

References

- (1) Ajit Jillavenkatesa et al., *Particle Size Characterization*, NIST Special Publication 960-1. January 2001.
- (2) T. Allen, *Particle Size Measurement*, 4th ed. Chapman & Hall, London (1993).

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