Five fundamentals for effective blend sampling

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Does your powder blend meet your uniformity requirements? Sampling can give you the answer. But poor sampling techniques can leave you more uncertain or even dead wrong about your blend's uniformity. This article describes five fundamentals for ensuring that your sampling process accurately demonstrates your blend quality. While this article is focused on blend sampling, the same principles apply in other sampling applications.

Blending dry bulk solid ingredients is a common processing step in many industries. Many powdered food products, such as dry beverage mixes, cake mixes, and seasonings, are made from custom-blended batches of dry ingredients. In pharmaceutical manufacturing, a small amount of a powdered or granular active drug is carefully blended with *excipients* (inactive ingredients), such as sugar, starch, cellulose, lactose, or lubricants, to form tablets and capsules. Blending is part of the manufacturing process for thousands of products, including specialty chemicals, explosives, fertilizers, detergents, glass, ceramics, and plastics. Achieving a highly consistent blend is often critical to a product's performance and quality.

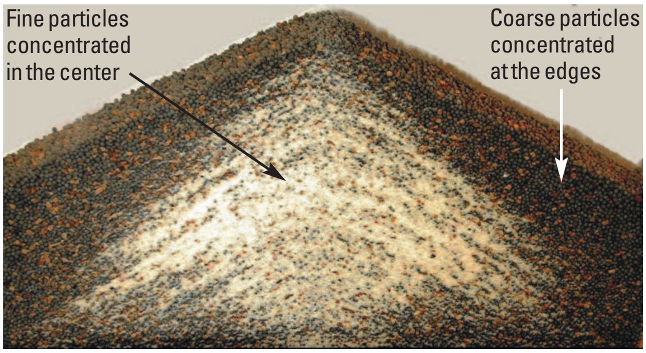
Achieving a "perfect" blend in your blender, however, doesn't guarantee you'll end up with a perfect or even acceptable final product. Blended ingredients often segregate during blender discharge or downstream processing. Common causes of segregation include sifting, fluidization, and dusting.[1,2] Depending on what causes the segregation, fine and coarse particles will concentrate in different locations in a downstream vessel. With sifting segregation, for example, fine particles will concentrate in the center, while coarse particles will concentrate at the edges, as shown in Figure 1.

Sampling can help you spot segregation and get an accurate picture of your blend quality. Improper sampling, however, can give you the false impression that your ingredients are adequately blended when they aren't or, conversely, that they aren't blended well when they are.

In any sampling discussion, it's important to keep in mind the two golden rules of sampling described by powder technology researcher Terence Allen: *Sample material when it's in motion,* and *sample the entire material stream during short intervals*.[3] But as you'll see, it's not always possible to strictly follow these rules, and sampling material at rest is often the only practical or cost-effective option.

Figure 1

Sifting segregation



To ensure that your sampling process reliably and accurately demonstrates your blend's overall quality, follow these five fundamental sampling principles.

1 Remember that perfect blending doesn't guarantee a uniform product.

Every time you transfer a blend from one vessel to another, it has the potential to segregate. You can't assume that because the blend is adequate in the blender it'll remain that way after it's discharged. Effective sampling can tell you your blend's characteristics *throughout* your process. To accurately demonstrate your blend's uniformity, take samples in the blender, during blender discharge, and from the final product.

Sampling in the blender. Sampling your mixture in the blender can't tell you if your final product will be well blended, but it can indicate a need for more or less blending time or a different blending method. Take samples at different locations in the blender, since the ingredients may not be consistently blended throughout. This *stratified* (or *nested*) sampling method (discussed in section 3) can not only improve your in-blender sampling reliability but also help identify dead zones in your blender.

Sampling during blender discharge. Sampling your blend during blender discharge is more challenging than sampling in the blender but is the only way to find out if your well-blended ingredients are segregating during discharge. The method tends to yield a larger collected sample, which you may have to divide into smaller subsamples for analysis (discussed in section 5). Also, dust release, material toxicity, or rotating blender components can make the method difficult or even dangerous.

Sampling the final product. Sampling the final product is the ultimate test because it reflects the blend's quality when it reaches your customer. Sampling at this stage alone, however, typically isn't adequate, because if the sample reveals that your product has segregated, you still may not know where the segregation occurred. In such a case, you'll need to sample at each piece of equipment your blend is transferred into to determine where it's segregating.

2 Beware of thieves.

A *sample thief* is commonly used to collect samples from stationary material in a blender, drum, or storage vessel. The thief is a metal cylinder with one or more recessed cavities that can be opened and closed by twisting a handle, as shown in Figure 2. An operator inserts the thief into the material bed and opens the cavities, allowing material to flow in. The operator then closes the cavities and removes the thief with the sample material inside.

Note that thief sampling violates the first golden rule of sampling because the material isn't in motion. Inserting the sample thief into the stationary material also disturbs the blend, so the operator should always extract samples with care. Studies have shown that thief-sampling results can vary depending on the operator's technique (such as thief insertion angle, penetration rate, and twisting technique). Also, some ingredients may not flow well into the thief cavity or may stick to the thief itself.

Though it can be problematic if not used properly, the sample thief shouldn't be abandoned. However, be sure to record any irregularities you observe in the thief cavity or extracted sample (such as static cling, agglomeration, or smearing) and carefully scrutinize the resulting data.

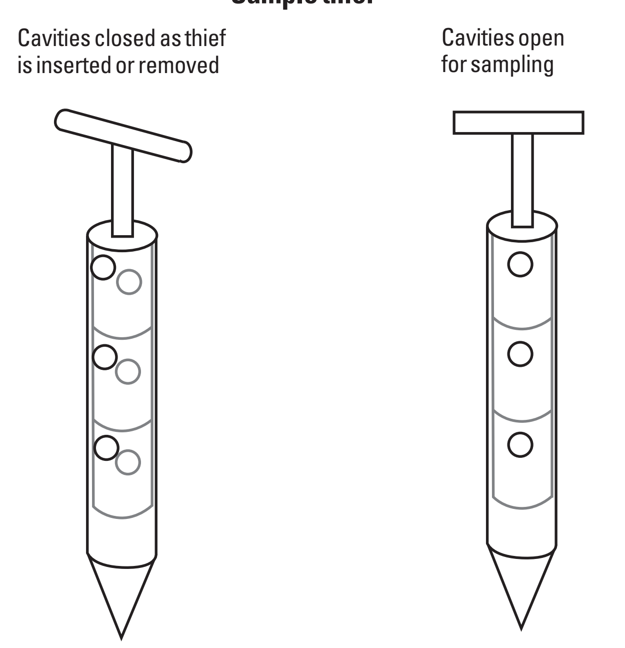
3 Use stratified sampling.

You can improve the quality of your thief sampling with a stratified approach, as shown in Figure 3. Instead of extracting only one sample from one location in a blender (or other vessel), extract multiple samples (a minimum of three, with five recommended) from the same location. Repeat this at several locations in the blender, especially in known dead zones, such as at the blender walls. Then analyze these samples and assess whether variations are the result of inadequate blending or sampling error (from the thief, collection method, or lab analysis).

For example, if the five samples collected at any one of the numbered locations in Figure 3 vary widely, it raises

Figure 2

Sample thief



questions about the thief or analysis method. If five samples collected from different numbered locations vary widely, then it's likely that blending isn't complete and additional time or agitation is required. It's also possible that overblending has caused the ingredients to segregate.

4 Collect a full-stream sample.

An alternative to thief sampling is full-stream sampling. This method follows the golden rules of sampling mentioned earlier, since it collects the material while it's in motion during discharge and collects the entire material stream for a brief interval. Full-stream sampling provides a true snapshot of the blend's uniformity as it exits the blender and also overcomes many pitfalls common to the sample thief.

Unlike with thief sampling, however, if your full-stream sample indicates that more blending is required, you can't simply continue the blending process, because you've already discharged the material from the blender. Another downside is that a full-stream sample is generally much larger than a thief sample. For instance, a 2-second full-stream sample could yield as much as 5 gallons of material, when you may only need a fraction of that for analysis.

5 Handle the collected sample carefully.

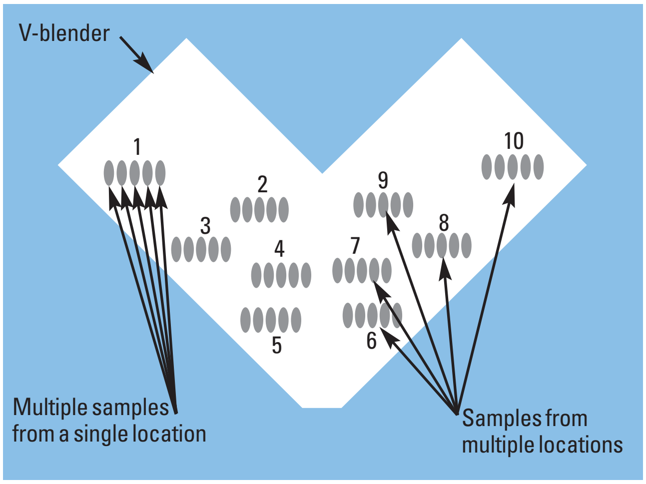
How you handle the sample after it's collected can affect how accurate your results are. Ideally, you should analyze the entire collected sample, but in many cases, you may have to split the sample into smaller subsamples for analysis (such as chemical assay, pH, or particle size). Do this carefully to avoid introducing error to an otherwise representative sample.

For example, a 500-gram sample collected from a hopper could potentially segregate in the sample container. If a lab technician collects a 5-gram subsample from this for analysis, the subsample may not represent the entire sample's true particle size distribution, resulting in error. To ensure that you accurately divide a large sample, you can use a *rotary splitter* (also called a *rotary riffler* or *spinning riffler*), which will properly distribute fine and coarse particles to the subsamples. Avoid using error-prone splitting methods like *coning and quartering* or *chute riffling*. [***Editor's note:*** Find more detailed information on sample-splitting methods in sources listed in the "References" and "For further reading" sections.]

If you collect samples over time and then combine them into a composite sample, be sure to blend the combined sample well to ensure accurate results. Also, be aware that this method will, at best, only tell you your blend's quality over that specific time period. **PBE**

Figure 3

Stratified sampling in a V-blender (10 blender locations with 5 samples per location)



References

1. Herman Purutyan and John W. Carson, "Predicting, diagnosing, and solving mixture segregation problems," *Powder and Bulk Engineering,* January 2007, pages 35-43.
2. J. C. Williams, "The segregation of particulate materials: A review," *Powder Technology,* Vol. 15, Issue 2, pages 245-251.
3. Terence Allen, *Particle Size Measurement,* 4th edition, Chapman and Hall, 1990, page 9.

For further reading

Find more information on blend sampling in articles listed under "Sampling" and "Mixing and blending" in *Powder and Bulk Engineering*'s article index in the December 2014 issue or the Article Archive on *PBE*'s website, www.powderbulk.com. (All articles listed in the archive are available for free download to registered users.)

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