Tips for taking samples from powder blends

You take samples during batch blending to determine whether the batch is blended properly to meet your customer’s specifications or the requirements of a downstream process. This article discusses sample size, frequency, and methods.

Some pharmaceutical manufacturers don’t need to sample because doing so was not part of the manufacturing process submitted to the FDA when their product was approved. Others—including manufacturers of dietary supplements—include blend sampling in their approved procedures and/or standard operating procedures. The goal of sampling: To determine whether the batch is blended properly to meet your customer’s specifications or the requirements of a downstream process.
Sample size

Sampling demonstrates that once a batch is blended, each portion taken from the batch will contain the proper proportion of each ingredient. To ensure this, the samples you take must be the same size as the portions of the blend that your customer requires or that will be used in your downstream process.

If you have a 100-pound blend and the entire 100 pounds is going to be used in the next process, you don't need to sample because all the required ingredients are in the batch in the designated proportions. But if the next process requires 1-ounce portions of your 100-pound batch, your sample size must be 1 ounce. Likewise, if you're making capsules weighing 5 grams, then your sample will be 5 grams.

To understand why your sample must be the same size as the batch portion to be used, think of a 100-pound pharmaceutical powder batch consisting of 99 pounds of various excipients and 1 pound of the active pharmaceutical ingredient (API) that's going to be filled into 3-gram capsules. If the blending isn't done properly, some capsules might contain 3 grams of the API, while others might contain no API and still others might contain some unknown ratio of excipients-to-API. But if you blend properly, every 3-gram portion of the batch will contain 1 percent API and 99 percent excipients. That's why you sample: If your sample has 1 percent API and 99 percent excipients, you can be fairly certain that all of the capsules in the batch will contain the proper proportions. But if your sample has some other ratio of API-to-excipients, you know that your blend isn't finished, continue blending and take additional samples until you have the right blend.

Practically speaking, this isn't as time-consuming as you might think. Once you've blended a correct batch of a material—and kept detailed records of how and when the ingredients were added and for how long they were blended—your first sample should turn out to be right on with every subsequent batch. (If conditions change—for example, you begin using a new ingredient supplier or humidity changes in your plant affect your ingredients—you may have to do additional sampling to ensure a correct batch.) While small portions require small samples, it's also true that the smaller the portion, the better the blend must be. So why must you take a large sample for large portions? After all, if you use a small sample and the proportions are correct, they'll certainly be right in the larger portion you or your customer will actually be using. But smaller portions mean finer blending, which generally takes more time and energy. If your portions are 10 pounds and you're taking a half-pound sample, you may be blending longer than needed.

When to sample

The sample tells you when the batch is done and, ideally, you want to blend until the blend is right and no longer. The longer you blend, the more it costs in time and energy. In addition, the longer you blend, the more heat you'll add to the batch, so additional time and possibly energy (depending on what method you use) will be required to cool the blend. In some cases, adding more heat than necessary can actually damage the materials in your blend. Another drawback of overblending is that demixing (segregation) can occur. Blending longer isn't usually a good thing. So when should you sample? If the blending cycle is 5 minutes, you should sample at the 5-minute point. If the blending cycle is 20 minutes, you should sample at the 20-minute point. You want to sample at the point you believe your blend should be finished based on your experience with similar blends.

Where to sample

Depending on your product's specifications, you can sample in the blender, in the container the blender discharges to, or both. Most commonly, companies sample in the blender. However, if you suspect that your material is segregating as it leaves the blender, you'll want to sample in the container the blender discharges to. To discover whether segregation occurs in the blender or during discharge, sample in both places. When you sample in the blender, theoretically you should be able to take a sample anywhere in the batch. But it's better to sample away from the impellers and the blender shell, where there's substantial drag on the material, and away from the surface, where the blend isn't representative. You'll get a more representative sample from the center of the material mass.

Number of samples

How many samples should you take? If your blend must meet a very tight specification, take more than one sample. Likewise, at the beginning of a production run, you may want to take more than one sample, but once the run is underway and you know that the blending operation is controlled as needed, a single sample at the end of each batch may suffice.

How to sample

Common ways to sample are to scoop material or use a sample thief. If you're using a scoop, make sure the scoop is the proper size to obtain the sample size you need, and take the sample from the center of the material mass, not from the surface. A sample thief is a device constructed of two tubes, one inside the other. Each tube has a hole in its sidewall. After the thief is adjusted so that the two holes don't align, it's inserted into the blend. Once in the blend, the inner tube is twisted so that both tubes' holes line up. Material enters the inner tube, which is twisted to close the holes and then removed from the blend. This device can capture a representative sample without disturbing the blend.

Sampling will help ensure that your blends are consistent from batch to batch. The more consistent your batches, the fewer process modifications you or your customers must make to use the blend or product. This will save them time and reduce scrap, making your blend or product less troublesome and less expensive to use.

Peter R. Holman, P.E., has more than 25 years' experience developing mixing systems for the chemical, pharmaceutical, cosmetics, biotech, and food industries. Before his retirement, he led mixing seminars at The Coating Institute and Eastern Michigan University. A version of this article originally appeared in Powder and Bulk Engineering.