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REVIEW

Powder Sampling

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ABSTRACT

The factors involved when sampling powder mixes have been reviewed. The various methods are evaluated (manual, automatic, and sub-sampling) and the errors incurred are discussed. Certain rules have been applied to various samplers and their suitability for powder mixtures are described. The spinning riffler is apparently the most suitable, while the use of sample thieves should be avoided due to error and bias.

Key Words: *Automatic sampling; Bias; Error; Manual sampling; Powder mixing; Sampling; Sub-sampling*

INTRODUCTION

Sampling

Taking a large amount of powder for sampling is very expensive and wasteful to analyze, so it must be reduced (1). "Sampling in its strict sense is therefore a simple mass reduction" (1). To collect a manageable amount of the powder which is representative of the batch (2), theoretically, every particle should have an equal chance of being selected (3). This should be achieved with minimum disturbance to the system (4). The composition of the original powder must be retained (1). Powder characteristics change under an applied load and attrition (internal milling by the particles themselves) and segregation (separation of coarse from fine particles) may occur in transfer. Representative sampling is defined by the selection method and, to produce reliable samples, a correctly designed sampler is required (1). A review of sampling techniques is shown in Table 1.

Certain rules should be followed when sampling:

1. Sample from a moving stream of powder;
2. Sample the whole stream for equal periods of time, rather than part of the stream for all of the time (2).

The number, the size of each sample, and the method should all be considered. The acceptable error dictates the number of samples taken. With more

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Table 1

Powder Sampling

Apparatus	Type of Sampler	Method of Sampling	Scale	Advantages	Disadvantages
1. Sample thief	Manual	Tubular steel retains a core sample when inserted into powder	Large and small	• Good for free-flowing powders (10)	• Additional weight at bottom of bag may vary sample size • Can be hard to push into powder • Fines may lodge between tubes (12) • Particles can fracture • Fines compact, impeding flow (11) • Segregation may occur as fines percolate into sample more easily than coarse particles • A plug of powder can be pushed ahead of thief and surface material contaminates sample • Personal preference introduces bias for the area sampled (6)
2. Hand scoop	Manual	Cross-sectional sample from moving stream, bags, or barrels	Large	• Simple • Cheap (11)	• Thin layer may remain on belt, leading to bias (11) • Overfilling can lead to an excess of fines (14)
3. Shovel	Manual	Pits are drug in the powder bed and a shovelful taken from bottoms and sides	Large, up to several tons	• Simple • Cheap	• Cannot be used with particles of more than 5 cm diameter (14)
4. Cross-cut sampling	Manual/ semi automatic/ automatic	Material is shovelled from the conveyor belt	Large	• Simple	• Can leave a layer on the belt and result in bias (10)
5. Pneumatic lance	Semi-automatic	Air flow used on entry and exit of lance from powder bed	Large	• Disturbance of powder minimized over sample thief • Porous plate prevents too many fines due to strong air current	• Personal preference may bias sample (12)
6. Vacuum probe sampler	Semi-automatic	Powder extracted by vacuum	Large	• Simple	• Difficult to sample below surface without contamination • Personal preference leads to bias • Fines more easily extracted than coarse particles (10)

7. Gravity-flow auger sampler	Semi-automatic	Slotted tube in flowing powder rotates and worm screw carries out material	Large and small	• Easy to use	• Bias is still a problem here • Difficult to sample all of powder stream, therefore bias (10)
8. Sampling from a moving stream	Manual/ automatic	Powder is sampled as it falls off the conveyor	Large	• If carried out properly can be very good sampling technique • Easily designed into a new plant	• Segregation can occur on the conveyor belt • If overfilled, a greater number of fines than coarse particles are collected • Difficult and expensive to fit into an existing plant • Obtaining a fixed sample is difficult • Difficult to prevent dust escaping (11)
9. Full-stream trough sampler	Automatic	Samples powder as it falls off conveyor	Large	• Can be used to sample dusty material	• Must not overfill • Difficult and expensive to install into existing plant (11)
10. Arc path cutter	Automatic	Chute moves through powder stream, and collects sample	Large	• No operator bias	• Difficult to take more than one sample size • Must cover the whole of the stream to avoid bias (14)
11. Straight path cutter	Automatic	Rectangular chute moves through powder stream	Large	• Different sample sizes can be taken easily	• Must cover the whole of the stream to avoid bias (14)
12. Moving-flap sample divider	Automatic	A flap in the stream samples powder or allows it to be stored depending on its position	Large	• Efficient as covers the whole of the stream when sampling	• Resultant sample is large and sub-sampling needed. Bias may be introduced here (11) • Bias is present due to one side being sampled more than the other (14)
13. Integrated automatic sampling plant	Automatic	Primary sample is selected and repeatedly screened, resulting in the final sample	Large	• Quick	• Variations in materials can cause problems
14. Chute splitter	Sub-sampler	A series of chutes split sample repeatedly	Large	• Can be repeated until desired sample size is achieved	• If segregation occurs the result can be misleading • Prone to operator bias (11)

(*continued*)

Table 1. Continued

Apparatus	Type of Sampler	Method of Sampling	Scale	Advantages	Disadvantages
15. Cone and quartering	Sub-sampler	Powder poured through cone and divided into four equal parts. This is repeated until the desired sample size is reached	Small	• Simple	• Prone to operator bias as fine particles remain in the center of the cone (16) • Symmetry is difficult to achieve but essential for accuracy (11)
16. Spinning riffler	Sub-sampler	A steady stream of powder flows into a rotating basket of containers	Large/ small	• Good for sub-sampling large samples (14) • Good for powders with good flow properties (12) • Minimal bias (16) • More efficient than other samplers tested (17)	• Air currents may displace fines but can be avoided with a slower rotation speed • Expensive • Time-consuming • Segregation may be a problem (12)
17. Free-fall tumbler mixer	Sub-sampler	A ladle in the lid of the mixer collects a good represen-tative sample	Large/ small	• Can be used with fines present • Representative sample produced in a short time period (12)	
18. Hopper sample divider	Sub-sampler	Hoppersoscillatesandpowder falls into two containers. Only one of the contents is kept	Small	• Sample size can be controlled by monitoring time over each container	• Large number of incre-ments needed for accuracy (11)
19. Table sampler	Sub-sampler	Powder flows down inclined plane and prisms and holes split the powder	Small	• Simple	• Very low accuracy • After each separation should be a complete mix to avoid errors (11)

samples, the error becomes small (5). All analysts have a personal preference for sampling patterns and random number tables can eliminate this problem. "It is extraordinarily difficult to carry out any selection process without introducing individual preference or bias into the final selection" (6).

Two types of sampling error occur within a heterogeneous system:

1. Constitutional heterogeneity caused by differences in chemical and physical properties of the particles, which cannot be suppressed, even when the sampling is perfect.
2. Distribution heterogeneity caused by the non-random distribution of particles throughout the powder bed (7). This can be manipulated.

Scale of Scrutiny

When a mixture is examined closely, regions of segregation are often found. The smallest region to measure imperfections in a mix is the "scale of scrutiny" and can be a length, area, or volume (8). The scale of scrutiny is directly related to the finished product. If the product is a tablet, then the scale of scrutiny is the weight of the tablet. The lower limit is set by particle size (9). When the scale of scrutiny is large, or the particle size is small, a large number of particles are present in a sample and the mix appears uniform (8). Segregation can never be fully avoided and sampling the final product is therefore preferred (5).

Sampling can be undertaken in two ways:

1. By taking increments on flowing streams of powder;
2. By splitting, where the whole is handled (1).

MANUAL SAMPLING

Sample Thief

This tool can be useful for free-flowing powders. It consists of two concentric tubes. The outer tube is pointed, with holes cut in corresponding positions in inner and outer tubes. The holes are opened or closed through the rotation of the inner tube to capture material (10). The chamber can be full length, at the pointed end only, or there can be separate holes along the tube. The first is useful for spot sampling, the second to gain an average for many samples, and the third for segregation determination throughout a powder bed (Fig. 1) (11).

Another variation is a concentric, inner-slotted tube with a shoulder at the lower pointed end. The outer tube has no holes and sits on the shoulder of the inner tube in the closed position. The thief is pushed diagonally into the powder and the outer tube is raised to expose the slot, which must face upward. The material falls into the chamber and the tube is removed, slowly opening the tube to collect a representative sample from all parts of the powder bed (10).

The thief can sometimes be fairly hard to push into the powder bed and it is not a good idea to have too many close-fitting, moving surfaces when handling powders, because the fines clog and bind them (12). Particles may fracture and fine material compact by the motion of the sampler, impeding flow. Bias is introduced by personal preference for the area sampled. Segregation may occur by fines percolating into the sample more easily than coarse particles (11). A plug of powder can be pushed ahead of the thief and surface material will contaminate the sample (6).

An alternative technique, which reduces segregation, is a unit-dose compacting sample thief (Fig. 2). Bias is still likely due to the invasive nature of any sample thief (13).

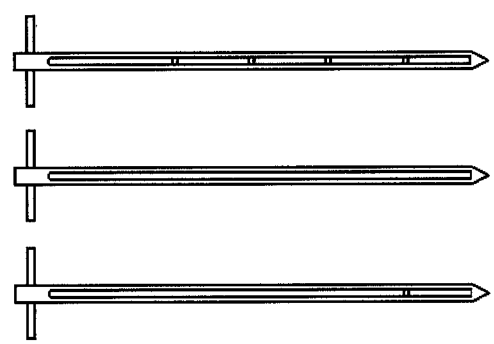

Figure 1. Sample thieves. (From Ref. 21.)

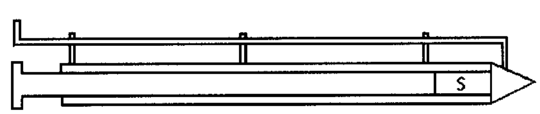

Figure 2. The unit-dose compacting sample thief. (From Ref. 13.) The sample is collected in the "S" section and the outer rod rotates to open and close the thief.

Hand Scoop

A scoop can take a cross-sectional sample from a flowing stream, bags, and barrels. From the powder stream, a single swipe of the scoop completely across the stream collects the sample. Opposite directions for each collection should be used (10). The scoop width should be at least 2.5–3 times the largest dimension of the particles to prevent overfilling leading to an excess of fines (14) and care must be taken not to leave a thin layer of powder on the belt (11). With fines, a larger scoop can minimize moisture loss, but the volume may be too large and using a shovel would be more convenient (14). This method is widely used in manufacturing industries as it is simple and cheap (11).

Sampling with a Shovel

In this method an imaginary grid is applied to a pile of material. At the intersections pits are dug, roughly 30 cm deep, and a shovelful is taken from the bottoms and sides of each hole (10). This method is only applicable to large amounts (up to several tons) of material, but cannot be used with particles larger than 5 cm in diameter (14).

Cross-Cut Sampling

Using a conveyor belt, the belt is stopped and a sample is taken by hand or by an automatic or semi-automatic device. A suitable size for sampling can be designated by markings on the conveyor belt and all material within these markings collected. If an automatic system is used, a mechanically-operated head moves across the material at a preset interval and moves a sample into the collection point (10).

SEMI-AUTOMATIC SAMPLING

Pneumatic Lance

This method is used for bulk powders. A gentle flow of air out of the nozzle allows the probe to move through the powder bed. At the site, air is very slowly reversed to draw up a sample, which is collected against a porous plate at the end of the probe. The porous plate prevents fines being present in the sample if the air current is too strong (Fig. 3). The pneumatic lance minimizes powder disturbance

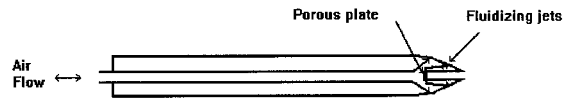

Figure 3. The pneumatic lance. (From Ref. 12.)

and is therefore advantageous over a sample thief, but bias is still a problem (12).

Vacuum Probe Samplers

Large samples can be extracted from bins using a vacuum-cleaner principle. Contamination is a problem when sampling below the surface and fines are extracted preferentially to coarse particles. Therefore bias is a problem (10).

Gravity-Flow Auger Sampler

A slotted tube is rotated in a flowing mass and the material is carried out of the tube by a worm screw. It is difficult to sample the entire stream and bias remains a problem (10).

SAMPLING FROM A MOVING STREAM

The powder is taken as it falls from the conveyor belt and can display two types of segregation, but great care should be taken to avoid the effect of segregation:

1. If the powder has been charged on the conveyor, fines concentrate at the center of the belt while coarse particles roll to the outer edges. Segregation occurs when sampling from a heap.
2. If the powder is exposed to vibration on the belt, percolation causes larger particles to rise to the top of the powder bed and fines remain at the bottom.

The whole of the powder sample must be collected for a short period of time and care must be taken in moving the sampler into and out of the stream (11).

Various methods exist for sampling from a moving stream and three are described here (Fig. 4):

a) This leads to an excess of coarse particles, if a longer time is taken whilst stationary than inserting the receiver and removing it, due

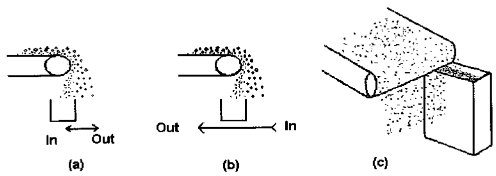

Figure 4. Methods of sampling from a moving stream. (From Ref. 11.)

to the surface usually being rich in coarse particles.

b) This shows good sampling technique. One movement samples the entire stream, but this is not always possible due to obstructions. The ratio of stationary time to moving time for the receiver should be as large as possible.
c) This method should be used only when it is not possible to collect the whole of the stream due to the sample being too large. A sample collector is passed through the powder stream (11).

With these methods (and all others that sample from a moving stream of powder) the dimensions of the sample collector are important. The width of the receiver should be greater for smaller particles than for larger ones, due to the diameter of the particles varying per sample. To minimize error, the ratio of box width to particle diameter should be at least 20 : 1. The depth should be large enough to prevent the receiver from ever becoming full whilst sampling. If it overfills, fines will percolate through the heap that forms, while coarse particles are lost. The receiver length should be sufficient to sample the full length of the powder stream (11). Disadvantages of this method include:

1. It is difficult and expensive to add to an existing plant but easily designed into a new one.
2. The sample quantity is proportional to (slit width + plant rate)/cutter speed. It is independent of stream shape or area. Plant rate can vary greatly and it can be difficult to obtain a fixed sample. Sample quantity may also be too large.
3. Enclosing the sampler to prevent dust escaping can be difficult (11).

Full-Stream Trough Sampler

This can be used for sampling dusty material. Sampling is only carried out on the return stroke (Fig. 5). The trough must not overfill so the constant volume sampler or the slide valve sampler should not be used for size-representative samples (11).

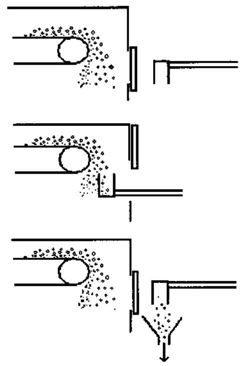

Figure 5. The full-stream trough sampler. (From Ref. 11.)

BULK SAMPLING

Bag Sampling

When tons of material need sampling, several sacks should be selected systematically, i.e., 100th, 200th, 300th, etc., by using a random number table, or $\sqrt{n} - 1$ (9 of 100). Each sample is individually assessed to determine the variation and whether acceptable, or combined to give an average. The thief sampler is recommended but this can lead to bias (15). It is preferable to use a spinning riffler, but this is expensive to install. It does, however, avoid rejecting good material and accepting poor material which would occur with the above, inexpensive technique (11).

Sampling from Wagons and Containers

This is impossible due to major segregation in the filling and vibrations in transport. Sampling should not be carried out in the top 30 cm to avoid segregation in the surface layer that occurs by vibration. When removing the samples, no surfaces on which particles can slide should be introduced. This can be achieved using a sample thief (11).

The extraction method involves sampling with a thief at eight points in the bed (Fig. 6). This method of sampling is not satisfactory but may have to be used in certain circumstances (11).

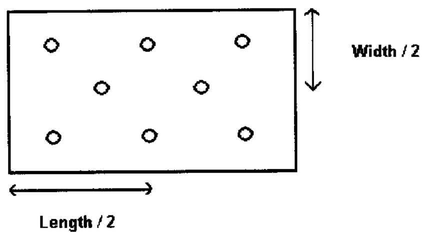

Figure 6. Locations for sampling from wagons and containers. (From Ref. 11.)

SAMPLING FROM HEAPS

This should not be carried out because marked segregation occurs, with fine particles concentrating in the center of the cone (Fig. 7). Sample from a moving stream of powder instead (11).

AUTOMATIC SAMPLING

A predetermined amount of powder is sampled at regular intervals or continuously. This may be subject to bias so regular inspections are needed. All the material should be sampled at a constant rate and clogging of the material should be avoided (14).

Arc Path Cutter

A chute at an angle is mounted on a vertical shaft, which rotates to periodically move the chute through the stream of powder. By increasing the speed of rotation, an increase in the number of increments is made, but this does not produce a larger sample (14).

Straight Path Cutter

A rectangular chute moves through the powder stream and rests outside it for a variable time period. This allows different sample sizes to be taken relatively easily. The whole of the stream must be covered to avoid bias (14).

Moving-Flap Sample Divider

A flap pivoted about a horizontal axis can rest in either of two positions. The powder stream goes to be stored or to be sampled and the time for the flap to be in either position can be controlled automatically (Fig. 8). If the sample is too large, the table sampler is used and the extra powder is returned to the store. This method would be efficient if it were not for the use of the table sampler, which causes error (11). A slight bias is involved as one side of the stream is sampled more than the other (14).

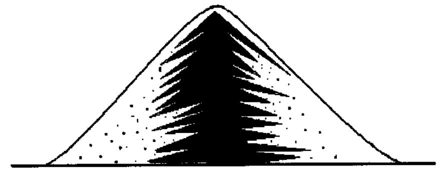

Figure 7. Segregation in powder heaps. Finer particles are illustrated in black, coarser particles are in white.

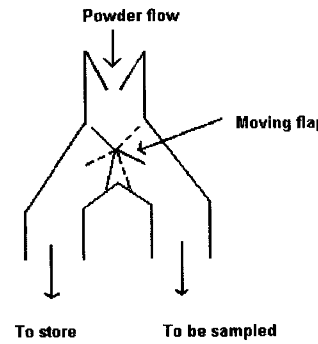

Figure 8. A moving-flap sample divider. (From Ref. 14.)

Integrated Automatic Sampling Plant

This apparatus selects the primary sample, which is then screened repeatedly to produce the final sample. The method is quick and convenient. Variations in materials can cause problems, however, and bias in sampling increases as the plant becomes worn (14).

SUB-SAMPLING

This sampling process results in a representative small sample from a large one (14).

Cone and Quartering

Powdered material is poured into a cone and divided into four equal parts. One quarter is recovered and the process repeated until the required weight of sample is obtained. This method is prone to operator bias, as finer particles tend to remain in the center of the cone due to size segregation (16). Symmetry of the cone is also quite difficult to achieve, but necessary for accuracy, so this method is not recommended (11).

Chute Splitting

This apparatus is a V-shaped trough, at the bottom of which is a series of chutes feeding two trays on either side of the trough. The sample enters the chute and is halved repeatedly until the desired sample size is achieved (16). The distance between the slots should be at least three times the size of the largest particle diameter (14). If segregation occurs, the results may be misleading. This method is prone to operator bias, due to unequal splitting of the sample. It has been found that an increase in the number of chutes can increase the overall efficiency (11).

The Spinning Riffler

A spinning riffler works by running a steady stream of powder into a rotating basket of containers (Fig. 9) (12). Two or more portions can be collected and it is a continuous dividing process (14). If sampling time divided by the time taken for one revolution is a large number, then each basket contains a representative sample of powder (12). Large samples are more difficult to sub-sample and rifflers are very useful in this situation (14).

If a powder has good flow properties, then a spinning riffler works very well. However, there should be only a small percentage of fines because air currents displace them and the sample is not representative. It is useful for a single powder ingredient but segregation can be a disadvantage. It is also fairly expensive and quite time-consuming (12). However, the spinning riffler has been found to be more efficient than cone and quartering or the chute splitter and is less prone to operator bias with free-flowing powders (16).

The efficiency was found to be dependent on the relative proportions of the mixture, with a mixture of quartz and copper sulfate crystals. Efficiency increases with increasing particle size, is reproducible under similar experimental conditions, and is not affected by the number of volume units (where a volume unit is the single presentation of a sample to the feed; the minimum was 100 in this experiment set, so it applies only to larger values than this). These conclusions may not apply where size segregation occurs (11).

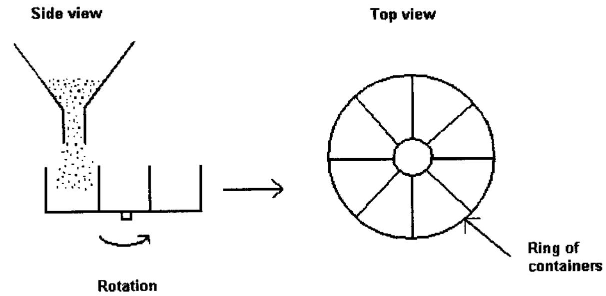

Figure 9. The spinning riffler. (From Ref. 15.)

In another set of experiments, the relative efficiencies of different samplers were compared and it was concluded that very little confidence could be placed in cone and quartering, scoop sampling, or table sampling. The spinning riffler emerged as so superior to the others that it should be used as often as possible. With the riffler, a minimum of 35 presentations are needed for optimum results to be obtained and if the speed of rotation is too great, the efficiency falls due to air currents set up (17).

Free-Fall Tumbling Mixer

This can be used if fines are present in a sample for prehomogenization. A sample container is mounted with a ladle in its lid and, after a short period of tumbling, contains a representative sample of the powder, due to the chaotic conditions created inside the mixer (12).

Hopper Sample Divider

The hopper feed oscillates on a horizontal axis and the powder falls into two collectors. Only one of the contents is kept. Monitoring the amount of time over each container can control the sample size. A large number of increments are needed for accuracy (11).

Table Sampling

The material is placed on top of an inclined plane containing a series of holes. Prisms in the path of the stream fraction the powder; some falls into the holes and is discarded while the rest carries on to the next set of prisms and holes and the process is repeated. The powder that remains at the bottom of the plane is the sample. The initial feed must be uniformly distributed and after each separation must be a complete mix, due to errors occurring. There is very low accuracy with this technique (11).

SAMPLING A SAMPLE

Having taken great care to extract a sample from a batch of powder, it is pointless to then take a small amount of this sample for analysis with little care for the technique used. By taking a sample from the bulk equivalent to the scale of scrutiny this will minimize problems associated with powder handling. Analytical chemists tend not to realize that they analyze the end product of an intricate sampling plan (18).

> "They (chemists) are content to be mere mechanics, material grabbers. One wonders how many analysts there are that do not realise that taking the analysis sample from a bottle of pulp is a sampling process whose extraction error may be a magnitude greater than the analytical error" (19).

Sampling is a stepwise process. The whole batch is represented by the first sample, the first by the second sample, and so on until the portion for analysis is produced. This is summarized in two main stages:

1. Primary sampling. This involves all steps outside the analytical laboratory resulting in a laboratory sample.
2. Secondary sampling. This includes all steps carried out within the analytical laboratory (18).

Table 2 illustrates errors involved in the sampling process.

In the analytical laboratory, seemingly small issues can have large effects on the accuracy of sample analysis. When weighing a sample:

1. Powder may be left on the tray, but weighing by difference does not solve the problem fully. The powder remaining on the tray will most likely be cohesive fines, leading to bias due to exaggerated concentrations of coarse particles analyzed.
2. Powder may be "washed in", leaving none behind. This is preferable but the choice of solvent is critical.

Table 2

Errors Involved in the Sampling Process

Primary sample	Up to 1000% bias
Secondary sample	Up to 50% bias
Analysis	0.1–1% bias

Source: Ref. 1.

CONCLUSION

Sampling undertaken correctly prevents the losses that the company may suffer from if it were done incorrectly. Even though sampling is not revenue-producing, it is very important economically and heavy losses can easily be avoided through the use of correct sampling.

> "Overall analytical reliability depends on sampling accuracy (negligible bias) and reproducibility (acceptable variance)" (18).

Certain rules of sampling are to sample from a moving stream of powder and to sample the whole of the stream for many equal periods of time. This ensures all parts of the powder are equally accessible and maximizes accuracy (2). The sample thief does not meet these criteria and therefore should not be used if possible. Despite being one of the most popular methods currently available it suffers from many disadvantages, which lead to bias and error. Most other methods discussed are immediately ruled out as suitable if these guidelines are followed.

The automatic sampling methods discussed are subject to bias, sampling some parts of the stream more than others. Sampling directly from a moving stream involves great care to avoid segregation, but method (b) in Fig. 4 can avoid this problem.

The two important methods are the spinning riffler and the free-fall tumbling mixer. The first has been proven to be the most superior method of sampling in experiments and should be used whenever possible. The second has not been investigated fully but deserves further study. Methods that rely on chaotic conditions created in the mixing process should be perfect for sampling. The powder is in motion and all is sampled, following the rules. The problem of losing fines into the surrounding atmosphere is also avoided (12).

Companies should take great care when sampling. The important rules should be followed at all times, awareness should be raised to minimize bias and other errors, and the importance of sampling should not be underestimated.

> "The accuracy of many analytical data reports is a mirage because unwitting negligence and false cost consciousness have ensured that a sample of powder taken with cursory swiftness has been examined with costly precision" (20).

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