Design of Optimum Sampling Plans for Dry Powders and Slurries†

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Abstract

Generally, in order to learn about a certain quality of a population, a small portion (sample) is extracted and analyzed for the desired property. In order to obtain accurate information, the sample has to represent the stream it is taken from (plant feed, intermediate product, and/or final product). In materials processing sampling of materials (powders or slurries) is very important to the quality control and quality assurance purposes. This sample can be too large and has to be further subdivided, or too small and a two-stage sampler has to be introduced. In most cases, the desired property is determined by analyzing a sample as small as a few milligrams. In this regard, obtaining a representative sample is not as straightforward as it sounds. For homogeneous materials, it is easy to use statistical probabilities to estimate numbers and sizes of samples that accurately represent the whole population. However, this is not so easy in the actual practice especially dealing with inhomogeneous materials. Therefore, sampling representativeness, perhaps the most important aspect of sampling practice is emphasized in this paper. Sampling strategies and equipment for dry materials and slurries are discussed with links to related literature and sampling equipment manufacturers.

Keywords: sampling, representative samples, dry powders, slurries, sampling errors, sampling equipment, sampling rules

1. Introduction

In order to learn about a certain quality of a population, a small portion is extracted and analyzed for the desired property. If the sample is extracted correctly (representative of the population), it can provide information that is used to describe the population. In powder production, as in any materials processing operations, sampling is very critical to the quality control and for quality assurance purposes. Such sampling could be done on the feed stream, the intermediate products, and/or the final products. If this sample is too large then it has to be further subdivided, and if it is too small then a two-stage sampler has to be introduced. In most cases, the desired property is determined by analyzing a sample as small as a few milligrams. Using a defined statistical parameter, we can determine if the sample is representative of the larger quantity. We can utilize automatic analysis equipment in process-control sampling to minimize detrimental time delays. The sampling process is as follows:

1. Take a gross sample from bulk material.
2. Divide the sample into smaller weights as required by the subsequent processing step. Sample crushing and/or grinding may be needed during such step.
3. Conduct required tests.

2. Sample Weight

Variations in materials quality dictate sampling techniques. For example, materials containing wide size distributions will require more rigorous sampling than those of close size distributions. In addition, coarse material needs to be sampled more carefully as the desired valuable mineral could be non-uniformly distributed in all the particles. Thus, finer materials may require less sampling than course ones as shown in Table 1.

To specify minimum sample size for estimating particle distributions within allowed variance, we use a simple approach based on a screening process in terms of binomial distribution. The particles will either pass through the screen or not. This is clear from examining the following example given in Perry’s Chemical Engineering handbook (1997). The sample weight needed for screen analysis can be calculated as follows:
Where:

\[ G = \% \text{ weight did not pass the desired screen} \]
\[ w = \text{weight of one particle remaining on that screen} \]
\[ V = \text{variance} = (\text{Standard error})^2 \]

Another example is given by Allen (2003) and summarized by Davies (2009) relating a minimum weight for the sample depending on the particle size and the variance of the tolerated sampling errors as expressed in the following equation:

\[ M_s = 1/2 \{\rho/\varphi^2\} \{1/W_s^2 - 2\}d_i^3 \times 10^3 \] (2)

Where:

- \( M_s \) is the limiting weight in grams
- \( \rho \) is the powder density in gm/cm\(^3\)
- \( \varphi \) is the variance of the tolerated sampling error
- \( W_s \) is the fractional mass of the largest size class in the bulk
- \( d_i \) is the arithmetic mean of the cubes of the largest diameter in the size class in cm\(^3\)

### 3. Number of Samples

In practice, there will always be some differences between the sample and the population. Repeated samples from the same population may also yield different results. Therefore, it is important to answer questions such as “What are the sources of such variations?”, “What is the optimum number of samples?”, and “How to minimize the errors?” The answers to these questions are very important so that cost effective and efficient sampling plans can be designed to achieve the desired information. These sampling principles are covered by the Theory of Sampling (TOS) which, if properly applied, can lead to information gathering at a minimum cost and in the shortest possible time. For materials processing, there are several challenges encountered due to the nature of the material and/or the processing methods (Gy, 1998). Various sampling errors are discussed in details by Petersen et al. (2005) and will be summarized later in this paper.

In statistics it is convenient to use the term population for the aggregate of all possible measurements or observations of a given type. Of course, one sample \((n = 1)\) drawn from a population would not give enough information. However, more samples \((n = 100 \text{ for example})\), if properly drawn, could be very informative about that population. Important statistical information can be derived from the observations. For example, the average and the variance can be calculated as shown below.

Usually, the population is characterized by value of the mean \(\eta\), and the variance \(\sigma^2\). The information in the sample is unfolded by calculating the average, \(y_{av}\),

\[ y_{av} = (y_1 + y_2 + ... + y_n)/n = [\Sigma y_i]/n \] (3)

\[ i = 1 \]

In practical situations we have a sample, from which we estimate the average, as mentioned above, and the variance as:

\[ s^2 = [\Sigma (y_i - y_{av})^2]/(n - 1) \text{ or} \]

\[ s^2 = [\Sigma y_i^2 - (\Sigma y_i)^2/n]/(n - 1)s^2 \] (4)

Where:

- \( \Sigma y_i^2 = \text{Crude Sum of Squares} \)
- \( (\Sigma y_i)^2/n = \text{Correction Factor} \)
- \( [\Sigma y_i^2 - (\Sigma y_i)^2/n] = \text{Corrected Sum of Squares} \)
- \( n - 1 = v = \text{degrees of freedom} \)
- Thus, \( s^2 = \text{Corrected Sum of Squares/degrees of freedom} \)

\[ \sqrt{s^2} = \text{standard error (s.e.)} \]

From the Central Limit Theorem, if \(y_1, y_2, ... y_n\) is a random sample of size \(n\) from a distribution with mean \(\eta\) and variance \(\sigma^2\), then \(y_{av} = \eta\) and the variance \(V = \sigma^2/n\). It should be noted that increasing the number of observations (samples) can lead to reduction in the variance. The implications related to sampling materials will be clarified later.

Variation in a particular result may be contributed by a number of different sources.

For example, the yield of a chemical process on a particular day might be determined by taking one small sample of the product and submitting that sample to a single laboratory test. Variation in the resulting observed yield would be contributed by:

1) the intrinsic day-to-day variability of the process,
2) the sampling error, and
3) the analytical error.

<table>
<thead>
<tr>
<th>Particle Diameter In Inches</th>
<th>Minimum Weight of Sample (pounds)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.04</td>
<td>0.0625</td>
</tr>
<tr>
<td>0.08</td>
<td>0.5</td>
</tr>
<tr>
<td>0.16</td>
<td>4</td>
</tr>
<tr>
<td>0.32</td>
<td>32</td>
</tr>
<tr>
<td>0.64</td>
<td>256</td>
</tr>
<tr>
<td>1.25</td>
<td>2048</td>
</tr>
<tr>
<td>2.5</td>
<td>16348</td>
</tr>
</tbody>
</table>
Before efforts can be directed at reducing the over-all variation it is necessary to know the relative importance of these different potential sources of variation.

To estimate these sources of variation a special hierarchical (or nested) design may be run in which two or more samples are taken on each of a number of days, and two or more chemical analyses are run on each sample.

An analysis of variance of the results (ANOVA) makes it possible to isolate and estimate the separate components of variance associated with:
1) variation of the process alone,
2) the sampling error alone, and
3) the analytical testing error alone

As a result of such a special study, the researcher who is interested in reducing over-all variation can direct her/his inquiries to the process itself, to the sampling technique, or to the analytical method, as appropriate. Alternatively, she/he may increase the number of samples, or the number of analytical tests, so that through the process of averaging the variance, contribution by any component may be reduced.

Therefore, it is important to be aware of the sources of errors in any sampling plan. These errors are summarized below and explained in details by Petersen, et al. (2005).

4. Sampling Errors

In addition to particle size, all the constituent elements of the stream should have an equal probability of being represented in the sample. Thus, good sampling plans are designed to avoid the following errors (Petersen et al. 2005):

- Compositional error: variations caused by temporal differences in the chemical nature of the bulk material give rise to this error. This error is usually reduced by milling the sampling stream and taking many sample increments from the resulting production cycle.

- Segregation error: this depends on the nature of the material and the ranges of size, shape and density distributions present. Thus, it is directly related to the amount of segregation in a lot. It can be minimized a) by mixing or building up a well-mixed composite sample from a large number of increments and b) by a correct design of the sampling system.

- Statistical error: it is the only sampling error that cannot be suppressed and occurs even in ideal sampling. It can be estimated beforehand and reduced by increasing the sample size (number of samples) as explained above.

In this paper, we will focus on mechanical extraction of representative samples of proper weight and numbers. Manual sampling methods are also used in practice; however mechanical sampling techniques should be used as much as possible to obtain best results with minimum errors. In addition, a good sampling strategy should involve important unit operations as emphasized by several authors (Allen, 2003, Minnitt et al., 2007, and Petersen et al., 2005) including:

- Heterogeneity characterization of new materials
- Sampling bulk material or powder only when in motion.
- Sampling the whole of the moving material stream in many short increments, rather than part of the stream for the whole of the time.
- Mixing (homogenization) well before all further sampling steps
- Composite sampling
- Particle size reduction (comminution or milling) whenever necessary
- Representative mass reduction

5. Sampling Practice of Particulate Materials

5.1 Sampling/mass reduction tools and methods

There are several sampling tools and methods that may be used to extract the primary sample. The choice of particular method depends on many factors including the relative reliability, type of lot to be sampled, etc. Mass reduction is often done via scooping devices or using specific mass reduction devices. Some common mass reduction techniques and devices include: grab sampling, alternate shoveling, fractional shoveling, spoon method, riffle splitters, and rational splitters. Examples of these tools are given below for free and non-free flowing materials.

5.2 Thief or spear sampling

- There are several companies that produce and/or sell such equipment including Samplers direct, Sampling Systems Ltd. UK, EET Corporation, etc. (Specific sampling equipment supplier names are provided for information purposes only and do not necessarily imply endorsement of the equipment by the author) Links to these companies’ web sites are given at the end of the paper. Sampling steps generally include,
  - Thrusting the spear into the bulk (Fig. 1).
  (Powder falls through holes into the spear.)
  - Rotating handle and extracting spear with enclosed sample

5.3 Coning and Quartering

Sampling of small heaps is frequently done by coning
and quartering. The heap is flattened and separated into four equal parts by a sharp edged tool, opposite quadrants are recombined, half the heap is discarded, and the remaining half is quartered again (Fig. 2). This process is repeated until the desired sample mass is achieved. Quartering should not be used for the sampling of free flowing powders. It is more accurate to sample the powder while it is being poured into a heap rather than after. It is not recommended to sample stationary free-flowing powder due to segregation. However, if it must be done, samples should be taken and analyzed separately to determine the degree of segregation. Fine particles will be concentrated in the center of the heap while coarser particles will be located in the outer portions.

5.4 Laboratory Spinning Rifflers

There are several companies producing such splitters. Among them is Quantachrome Instruments Company. A picture of a Micro Riffler is given in Fig. 3. There are a couple of videos on the company’s web site showing the operation of such splitters. For particles of larger size and samples of larger mass, chute rifflers are used. A couple of these rifflers are shown in Fig. 4.

It should be noted that reliability of some of these methods is low as shown in the following Table 2.

5.5 Sampling non-free flowing stored material

The following points should be taken into consideration before sampling takes place:

- It is usually assumed that the material is well mixed, but this assumption is often incorrect and
biased sampling results.
- Surface sampling by scoop is unreliable.
- Better accuracy is obtained if samples are taken from the body of the material by the use of a sampling spear or thief.
- A template should be devised so that samples can be withdrawn from various parts of the material volume.
- Never take scoop samples if at all possible from heaps. If it must be done, use coning and quartering or chute riffling of the whole heap.
- Sampling materials in bags, bins, wagons, bottles should not be done using scoop or spatula samples. Try to sample the materials when the containers are being filled.
- Expect large sampling errors from stored material sampling.

5.6 Sampling from a hopper/silo

Examples of point samplers for free-flowing and non-free-flowing materials from gravity lines and hoppers are also given in the web site of Sentry Equipment Corporation. A picture of this sampler in operation is given in Fig. 5 below.

The sampler takes a sample when a gear motor-driven auger draws the product to the discharge point. A schematic of similar operation is shown in Fig. 6 below (After Davies 2009).

5.7 Sampling Flowing Streams

In this case, Terry Allen’s Golden Rules of Sampling should be applied as suggested by Davies (2009):
- A bulk material or powder should only be sampled when in motion.
- The whole of the moving material stream should be sampled in many short increments, rather than part of the stream for the whole of the time.
- The sampling cutter should be designed to introduce no bias in the sampling of the largest particles present, and the cutter must never be allowed to overflow.
- To sample a moving stream the gross sample is made up of a series of increments. In this case, the minimum incremental weight is given by multiplying the flow rate times the cutter width (for a traversing cutter) and the cutter velocity. The cutter width should be large enough (about 10 times the largest particle diameter) so that a biased sample deficient of coarse particles may be avoided.

<table>
<thead>
<tr>
<th>Method</th>
<th>Estimated Maximum error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cone &amp; Quartering</td>
<td>22.7</td>
</tr>
<tr>
<td>Scoop Sampling</td>
<td>17.1</td>
</tr>
<tr>
<td>Table Sampling</td>
<td>7.0</td>
</tr>
<tr>
<td>Chute Riffling</td>
<td>3.4</td>
</tr>
<tr>
<td>Spinning Riffling</td>
<td>0.42</td>
</tr>
</tbody>
</table>
When sampling from a conveyor belt, it is best to sample material as it cascades over the end of the conveyor belt as shown in Fig. 7. If this is not possible samples must be taken from the belt itself. Some automatic samplers have a moving arm that sweeps across the belt collecting all of the materials within a particular area. Sampling from a moving stream can be done continually or intermittently. Poor and good sampling procedures are shown below. A schematic drawing is shown in Fig. 8 to illustrate the reasons for such classification (Courtesy of Davies, 2009).

In Fig. 8 (a) the sample will not represent the whole size distribution as the course particles will not be collected together with the fine particles. However, this does not happen in part (b). There are several samplers that can be used for Belt Conveyor sampling such as the ones sold by Sentry Equipment Corporation [www.sentry-equip.com] and Intersystems Company [www.intersystems.com]. The picture of this machine is shown below. The company’s web site contains more details as well as a video for this sampler and others.

6. Slurry Sampling

Slurries (a mixture of liquid and solid) are generally easier to sample than solids. Since slurries can be pumped

![Fig. 7 Sampling from falling conveyors: (a) bad sampling technique, (b) good sampling technique, (c) sampling procedure to be adopted for high mass flow rate. (Courtesy of Reg Davies, 2009)](image7)

![Fig. 8 A schematic drawing explaining the reasons for good and bad sampling techniques. (Courtesy of Reg Davies, 2009)](image8)

![Fig. 9 Sampling a moving belt. (Courtesy of Sentry Equipment, www.sentry-equip.com)](image9)
through pipes and are in constant motion, we can almost get a perfect representative sample. However, getting a complete cross section can be difficult due to the pipes being round. In-pipe samplers do not adequately extract particles at the pipe walls and thus you must consider both the location and the type of sampler in order to obtain a representative sample.

For accurate primary sampling, it is recommended that you sample at a dumping point. Samples are taken at a pipe discharge point using a cross stream sampler along with a slurry cutter. For secondary sampling, the primary sample can be split using a vezin sampler discussed on the web site of Multotec Company, 2012 (www.multotec.com.au.). Fine particles can be reduced by using a 2% cutter, reducing the primary sample by 98%. Two or three vezins can be used to split larger samples to final lab size volumes for the quality control testing.

In order to assure a representative sample, all material in a process flow should have an equal probability of being part of the sample. Typical process streams require a turbulent flow to keep the slurry in suspension (speed >1.5 m/s) as turbulence keeps the slurry well mixed. Lighter and finer solids in slurry require a greater level of mixing. When particles are above 100 μm in size and/or of high specific gravity material, mixing still produces the desired effect in the horizontal direction. We can use several mechanical sampling equipment, as discussed below, to obtain representative samples.

6.1 Mechanical sampling equipment

For slurry that flows by gravity through launders and sloped pipes, samples are taken from the point of discharge. Samples can also be taken at an open discharge from a vertical gravity pipe. There are two basic mechanical sampler designs for sampling material from a gravity flow such as sampling with linear cutter motion and sampling with radial cutter motion. When equipment is properly designed and operated, both designs produce the same result. Based on flow quantity, we can determine the mechanical installation factors. Equipment suppliers should be consulted to provide the proper design for any particular application. For helpful information, you may consult Perry’s Handbook of Chemical Engineering (1999).

6.2 Sampling from Pipes

In Florida phosphate industry, sampling equipment is either home-custom made or obtained from suppliers such as in Fig.10 from Sentry Equipment Corp. The following is a general description of sampling slurries either flowing under pressure or by gravity.

6.2.1 Pressure Flow

We must determine the primary sample flow for process streams under pressure. This is done by finding the balance between sampler pressure head and sample pipe friction. Outokumputechnology.com (2011) offers a full description of primary samplers and criteria relating sampler specifications to pipe size and flow rates. In addition, sentry-equipment.com offers a wide array of sampling equipment that can be used for slurry sampling from pipes under pressure flow or gravity flow. Fig. 11 shows an example of such equipment (ISOLOK® SAA Automatic Fixed Volume Sampler).

6.2.2 Gravity Flow

Gravity flowing slurries either through in launders, sloped pipes or vertical gravity flow pipe, should be sampled at the point of discharge. A vertical sample cutter can be used for process streams at/around ambient pressure. In order to obtain a proper sample using this technique, you must ensure that the cutter used has an opening many times larger than the largest particle size of sample slurry (preferably greater than 20 mm, no less than 8 mm). You can use an adjustable cutter opening to adjust sample flow for pipe sizes up to 400 mm. Several sampling steps or innovative sampling designs are required.

Outokumputechnology.com (2011). In addition to Outokumputechnology (now Outotec), Sentry Equipment Corporation and other suppliers have several samplers that can be used for this purpose. These companies’ web sites are given below to guide you to choose the best sampler for your application. Another example of samplers that can be used for flowing streams either under pressure or under gravity is shown in Fig. 11 as published by Sentry Equipment Corporation (2011).

There are other types of samplers using linear and rotary traverse mechanisms. The following is an example of how to calculate amount of sample in every increment.
as mentioned in Perry’s Handbook of Chemical Engineering (1997).

6.2.3 Slurry Sampling by Rotary Traverse of Gravity Flow

Given the following information, the quantity of slurry extracted by one rotation, $S$, can be calculated.

$$ S = \text{increment volume (quantity of slurry extracted by one rotation)} $$

$$ B = \text{bulk slurry flow, in volume/unit time} $$

$$ R = \text{rotation of cutter, in rpm} $$

$$ D = \text{cutter angle opening, } D/360 = \text{extraction ratio for continuous cutter rotation} $$

$$ S = D \times B/360 \times R \tag{5} $$

In addition to mechanical sampling equipment, hand sampling can be used to obtain samples from gravity flowing material from chutes, vibrating screens, belts, etc. When sampling from such streams, it is best to sample material as it cascades over the end of the equipment as explained earlier in the case of belt conveyors.

Suppliers of sampling equipment are eager to help in choosing the proper samplers for specific applications. Another example of such equipment is presented in Fig. 12 as a courtesy of Sentry Equipment Corp. The ISOLOK® SAB operates when compressed air forces the plunger into the process line to capture a fixed volume of material. Compressed air then acts on the opposite side of the piston to retract the plunger to a position which allows the sample to drop by gravity into a container. The operator is isolated from the process at all times by the sampler’s seal design, and the sample captured in the container is “locked out” from external influences. The reader is advised to consult the manufacturer catalogue for further details.

6.2.4 Calculation of Sample Extraction Increments

Cooper in Perry’s Handbook of Chemical Engineering (1997) presents clearly several examples illustrating calculation of incremental weights obtained by three commonly used sampling methods. These methods include: (a) Linear Traversing Trajectory Cutter, (b) Slurry Sampling by Rotary Traverse of Gravity Flow as explained above, and (c) Cross-Belt Sampling of Solids from Conveyors

6.2.5 Processing Wet Samples

Collected samples are dried in air and/or ovens provided that drying does not affect the chemistry of the sample contents. The dried samples then will be treated as dry powder sampling procedure explained earlier.

7. Summary Highlights of Sampling Rules

Several authors including Allen 2003, Davies 2009, Gy 1998, 2004a, 2004b, Minnitt et al. 2007, and Petersen et al. 2005 stressed the importance of the following points:

There are several sampling tools and methods that may be used to extract the primary sample. The choice of particular method depends on many factors including the relative reliability, type of lot to be samples, etc. It should be noted that reliability of some of these methods is low.

It is usually assumed that the material is well mixed, but this assumption is often incorrect and biased sampling results.

Select general guidelines for obtaining reliable sampling results are as follows.

- Never take scoop samples if at all possible from heaps. If it must be done, use coning and quartering or chute riffling of the whole heap.
- Sampling materials in bags, bins, wagons, bottles should not be done using scoop or spatula samples.
Try to sample the materials when the containers are being filled.

- A bulk material or powder should only be sampled when in motion.
- The whole of the moving material stream should be sampled in many short increments, rather than part of the stream for the whole of the time.
- When sampling from a conveyor belt, it is best to sample material as it cascades over the end of the conveyor belt. If this is not possible, samples must be taken from the belt itself.

Acknowledgements, Copy right statement and disclaimer

Financial support of this work was provided by the Center for Particulate and Suspension Systems (CPaSS) a NSF I/UCRC (NSF Grant #0749481). Opinions expressed are those of the authors and do not necessarily represent the views of the NSF or the CPaSS Industry Members. Commercial suppliers of the various equipment are provided for information purposes only and do not necessarily imply endorsement of the specific equipment by the authors.

References


Links to Literature

- Sampling equipment: http://www.powderandbulk.com/analyzers/samplers.htm
- Belt conveying equipment: http://www.powderandbulk.com/conveying/belt.htm
http://www.directindustry.com/conveying/belt.html
http://www.vac-u-max.com/pneumatic.html
Author's short biography

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Dr. El-Shall is a professor emeritus at the Department of Materials Science and Engineering of University of Florida. He has been serving as an associate professor of Materials Science and Engineering and Associate Director for Research at The Particle Engineering Research Center of University of Florida since 1994. He also served as Associate Director for beneficiation research at the Florida Institute of Phosphate Research from 1986–1992. In the early 80’s, he has served as assistant professor of Mineral Processing at Montana Tech from 1980 to 1986. He posses thirty nine years of diverse experience in research (both basic and applied), management, as well as hands-on experience in the areas of Applied Surface and Colloid Chemistry, Mineral Processing, Chemical Metallurgical Engineering, and related environmental services. His work related to industrial minerals such as phosphate, gypsum, clay, etc is recognized by his peers all over the world. In addition, he has extensive and practical experience in the development of training and teaching programs for both undergraduate and graduate students in the above fields. His major research interests include interfacial phenomena and its applications in mineral industry, and waste treatment in various industries. He expanded his research efforts to include mineral modifications for medical applications.

Dr. El-Shall has published over 165 publications including: 11 patents, 9 edited books, one authored manual, 19 chapter contributions to books, over 50 Refereed articles in prestigious journals such as Powder Technology, Mineral and Metallurgical Processing, International Journal of Mineral Processing, and others, in addition to many industrial reports.

Dr. El-Shall is a dedicated teacher in the fields of Mineral processing Engineering, Materials Science and Engineering, and Chemical Engineering. He has served as chair, co-chair, or a member of over 50 graduate committees for M.S. and Ph.D. students including his own 15 M.Sc. and 13 Ph.D. students who are currently employed by various industries.

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