CHAPTER 1.8

Sampling Practice and Considerations

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Taggart and Behre (1945) defined sampling as “The operation of removing a part convenient in size for testing, from a whole which is of much greater bulk, in such a way that the proportion and distribution of the quality to be tested (e.g., specific gravity, metal content, recoverability) are the same in both the whole and the part removed (sample).”

Sampling procedures cover the practice of selecting representative quantities of material in the field to evaluate bulk material properties. Examples of the test materials are bulk granular solids, slurries, sludges, grains, solid fuels, process liquids, and process gases. It is necessary to be able to sample bulk materials during shipment and during processing operations to ensure that the bulk material meets specifications.

When sampling a bulk material, it is very important that the sample be representative of the bulk material. In reality, the condition that the sample be completely representative of the bulk material in all aspects except amount is never fulfilled when heterogeneous materials are sampled. For instance, you can never get a perfectly representative sample of water from the Pacific Ocean. You may even ask what good it is or what purpose such a sample would serve. It is important, before collecting a sample, to decide what purpose the sample would be, and then plan the sampling procedure accordingly so that it will be sufficiently representative for one’s purposes.

PROPER SAMPLING

Proper sampling techniques ensure that measurements performed on a sample are representative of the material that was sampled. This is extremely important in all industries that process solid, liquid, and gaseous materials. To expand on this topic, the following key terms must be defined:

- **Lot:** A large (bulk) quantity of material from which a sample must be taken. If proper sampling is performed, analysis of the sample will be representative of the lot within a specified degree of certainty (or confidence interval).
- **Homogeneous material:** A material that is either one substance or a perfect mixture of multiple substances with constant thermodynamic properties throughout. By definition, perfectly homogeneous materials do not exist and cannot be created.
- **Heterogeneous material:** A material that has different properties depending on where the material is analyzed. Examples include slurries, immiscible fluids, and even tap water.
- **Component:** A particular substance within a lot of heterogeneous material. For example, silica, pyrite, and gold are typical components of gold ore.
- **Critical component:** The component of the heterogeneous material that is of interest. For example, gold is the critical component in gold ore.
- **Content:** The concentration of a component within a lot of heterogeneous material. For example, a gold ore may have a silica content of 65%.
- **Critical content:** The concentration of the critical component within a lot of heterogeneous material. For example, a gold ore may have a critical content of 0.01% gold.

Example of Poor Sampling: The Bre-X Gold Scandal

The Bre-X gold scandal is regarded as the biggest and most sophisticated scam ever in the history of mining. The scandal began when Canadian-based mining company, Bre-X Minerals Ltd., purchased the mineral rights to land in the jungle of Borneo, Indonesia, in 1993. Bre-X hired a geologist to take core samples of the site and assay the samples for gold. In just a few years’ time, Bre-X had discovered the largest gold reserve on the planet and stock soared from roughly US$1 to US$300. At this time, larger entities began to take notice, such as the Indonesian government. The Indonesian government then involved the American mining company Freeport-McMoRan Copper and Gold. To verify the claims made by Bre-X, Freeport sent in a group of their own to take core samples and assay for gold. Freeport reported that they were unable to find any significant gold reserves on-site. Four weeks later, it is believed that the lead geologist for Bre-X “jumped” to his death from a helicopter 800 feet in the air. So how did Bre-X fool everyone into believing it had discovered the largest gold reserve on the planet?

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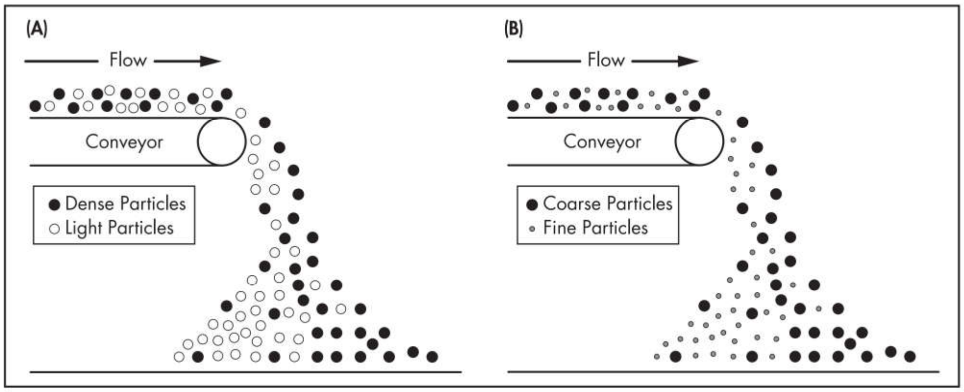


Adapted from Pitard 1993

Figure 1 Segregation of a heterogeneous material (A) of different density and (B) of different particle size when stockpiled from a conveyor belt

Bre-X was able to deceive everyone into believing it had discovered the world's largest gold reserve by conducting a fraudulent sampling campaign. It is believed that before the core samples were to be assayed, the geologist "salted" the samples with gold shavings from his wedding ring or the local river bed. With all the fervor over having found the largest gold reserve in the world, many of the early warning signs of fraud were ignored. For example, an independent contractor was brought on-site to examine the sampling procedure being carried out by the lead geologist. The contractor discovered that Bre-X was crushing and grinding the entire core sample for gold assay (it is typical to save half of the core for outside analysis). By doing this, Bre-X was able to destroy any evidence that may have disputed their claims to finding the world's largest gold reserve. When analyzing sampling results, it is important to always be skeptical of the findings, and remember: If it sounds too good to be true, it probably is.

SCOPE OF THIS TEXT

This chapter is designed to provide an overview of sampling methods without the cumbersome calculations and complicated error analysis of other works. The intended audience for this work is the technician, operator, or student that is interested in the most practical solutions to their sampling issues without complex calculations. For a more advanced and in-depth discussion of sampling and the errors involved, several fantastic and comprehensive books should be referenced:

- *Sampling of Particulate Materials: Theory and Practice* (Gy 1979)
- *Wills' Mineral Processing Technology*, 8th edition (Wills and Finch 2016)
- *Pierre Gy's Sampling Theory and Sampling Practice*, 2nd edition (Pitard 1993)
- *Principles of Mineral Dressing (*Gaudin 1939)
- *Handbook of Mineral Dressing* (Taggart and Behre 1945)
- *An Introduction to Metal Balancing and Reconciliation* (Morrison 2008)

SAMPLING A LARGE LOT

Obtaining a representative sample from a large (>1 metric ton) lot of material can be a very tedious and time-consuming task when done correctly. Many engineers and scientists make the mistake of merely shoveling a bucket of material from a stockpile, splitting it in a laboratory, and assuming that the properties of these splits are representative of the entire lot. Unfortunately, this is rarely the case with a heterogeneous material such as an ore. With a heterogeneous material, particles of varying particle size, density, particle shape, and friction tend to segregate when stockpiled, as indicated in Figure 1 (Pitard 1993).

Because of this segregation upon stockpiling, proper sampling techniques must be used to obtain a representative sample from a large lot of material. Several techniques can be used to split a large lot of material, some being better than others. Two common techniques are the "grab sample" and "coning and quartering."

Grab Sample

A grab sample is a convenient procedure for sampling large lots of material and can be conveniently done using shovels or even front-end loaders for very large samples. A grab sample involves removing equal-sized increments from a well-mixed lot, creating numerous smaller samples until the lot is eliminated, as shown in Figure 2. The sample for laboratory testing is then randomly selected from the increments. This procedure should be performed on a clean surface that will not contaminate the lot. Before incrementing the lot, it is advisable to use

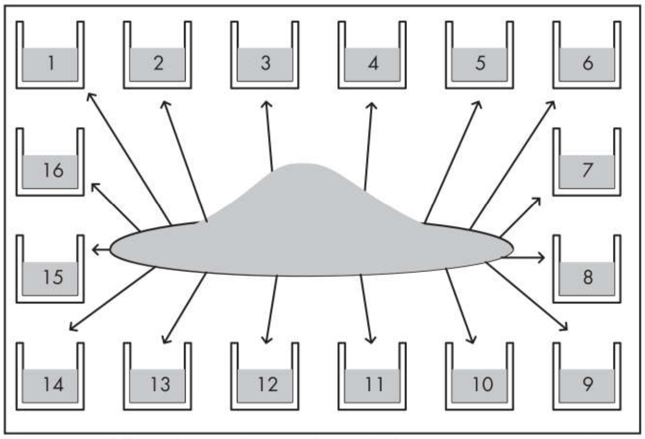
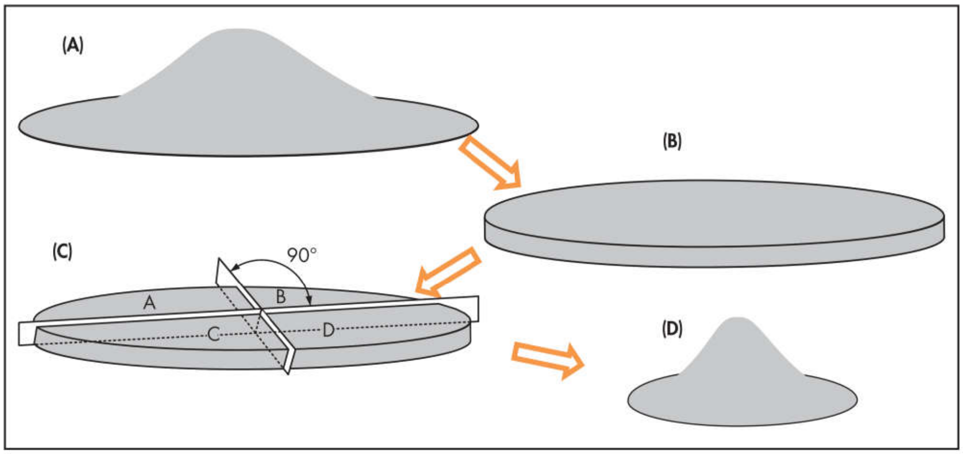


Figure 2 Using the grab sample technique to separate a lot into 16 representative increments



Source: Khan 1968; Pitard 1993

Figure 3 Coning and quartering a lot of material to acquire a sample that is one half the size of the entire lot (A+C or B+D)

an appropriately sized shovel (hand shovel, front-end loader, etc.) to mix the lot thoroughly by scooping from the sides of the lot and dumping the scoops in the center of the lot to create revolutionary homogeneity. Increments are removed from the lot and placed in a specified number of containers until the lot has been completely split. A container is selected at random and used as a sample. Grab sampling is a good way to obtain a representative sample from a large lot of material; however, this method is very time-consuming. Properly using the grab sample technique has been shown to give a standard deviation of ~5.14% between samples (Khan 1968).

Coning and Quartering

This method is well-suited for large lots of material and can be conveniently done using shovels or front-end loaders for very large samples. First, the material is mixed and shoveled into a uniform conical pile, as shown in Figure 3A. The pile must be made so that the natural segregation in the cone is radially symmetrical. The cone is then spread from the center to form a flattened disk of material, as shown in Figure 3B. This disk is then divided into quarters at exactly 90° using perpendicular boards, as shown in Figure 3C. One pair of opposite quarters (either A and C or B and D) is removed, and the other pair is used as the sample, as shown in Figure 3D. The choice between the two should be random (flip a coin). If the sample is too large, it can be coned and quartered again until the desired sample size is obtained. In addition to error generated from segregation, this procedure is prone to human error with a standard deviation of 6.81% between samples (Khan 1968; Pitard 1993).

SAMPLING A PROCESS FLOW

Heterogeneous process flows, including conveyor belts, slurry lines, liquid streams, and gas streams, tend to segregate based on the properties of the process flow. Sampling from a segregated process flow can present large errors if proper procedures are not followed. In general, the sample must be taken from a free-falling process stream, be it liquid, slurry, or solid, using a traversing or rotating cut. The cut must span the entire stream and minimize loss of material from dust generation or liquid splashing. Numerous cuts (increments) must also be taken to ensure that the overall sample is representative.

Rules of Cutting Flowing Liquid or Solid Material

Several methods for sampling material from a conveyor belt are available, and the method selected depends upon the accuracy desired, labor available, and the cost. However, the following basic principles should always be observed:

1. The cutter must cut the entire stream.
2. The speed of the cutter must be constant and lower than a specified maximum.
3. The cutter must have an opening width that is at least three times larger than the largest particle to be sampled, and it should be wide enough to prevent bridging (at least 1 cm for dry solids, and at least 0.5 cm for slurry streams).
4. The cutter opening must have parallel edges.
5. The flowing stream to be sampled must be in free fall.
6. The sample must pass quickly through the sampler to avoid blockage.

Sampling Flowing Solid Material (Conveyor Belt)

Linear (Traversing) Cut

A linear (traversing) cut sampler (or high cross-shear sampler) is the best way to cut a falling stream from a conveyor belt. The cutter moves across the falling stream in a straight-line path, as shown in Figure 4. Usually the path is perpendicular to the direction of the flow. The sample cutter travels linearly across the entire falling stream at a constant velocity. The primary sample is diverted from the main product flow onto a conveyor or into a collection bin. The cutter must travel across the entire falling stream because particle segregation occurs on the belt. If adjusted properly, this is the most accurate type of sampler because it follows all six of the principles given previously, but it is also the most expensive and hardest to maintain. These samplers typically use multiple identical cutters attached to a moving chain to take equal-sized cuts at a constant velocity.

The cutter must also be designed properly to ensure that each particle in the slurry has the same chance of being taken into the sample. The cutter opening width (or aperture) should be at least 10 mm or 3 times the size of the largest particle. Also, the speed the cutter traverses the falling stream must remain constant and below the maximum speed, given in the following equation:

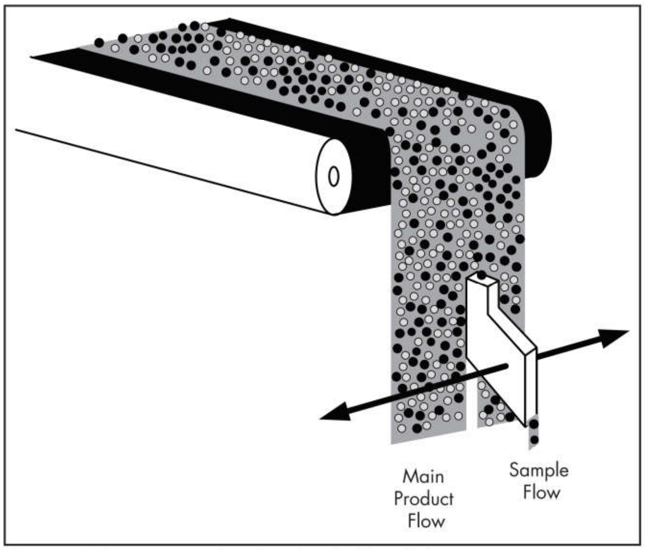


Figure 4 Linear sampler for dry bulk solids

$$V_C = 0.3\left(1 + \frac{w}{w_0}\right)$$

where

V_C = maximum cutter speed, m/s
w = cutter opening width (or aperture), mm
w_0 = minimum cutter opening width (or minimum aperture) as defined previously, mm

Cross-Belt Sampler

Collecting a sample from on top of a conveyor belt is possible using a cross-belt sampler, as shown in Figure 5. Representative sampling requires that the end plates are made of a rigid material that exactly conforms to the curvature of the belt. The type of cross-belt sampler shown requires that the belt be stopped while the sample increment is being taken. This limitation can cause issues in continuous processes but can work well when synchronized with belt filters that move by indexing incrementally. After the belt is stopped, a cross section of the material on the belt is scraped off the belt into a sample container. This type of sampler provides excellent representability of samples and is typically used to ensure that other automated samplers do not introduce bias.

Example of a Poor Solid Sampler: Stationary Cut

The stationary cut method, as depicted in Figure 6, represents a common but poor sampling method for sampling off of a conveyor belt. The cutting device is stationary and does not traverse across the entire material flow. Without the linear traversing motion, the segregation of particles on the belt causes the sample to not be representative of the entire process flow. This method ignores the heterogeneity and segregation of the solids being conveyed on the conveyor belt.

Sampling Flowing Liquids and Slurries

Sampling liquids and slurries presents inherent problems due to splashing and segregation of solids and liquids within process lines. Solid particles in slurries tend to settle in process lines unless the fluid flow is sufficiently turbulent. Nonhomogeneous liquids can also segregate or be segregated based on the miscibility of the liquid mixtures or insufficient mixing at reagent addition points. To sample these fluids, the rules outlined previously still apply. Most importantly, the fluid must be in free fall during sample collection to reduce any gravitational segregation. This poses issues when dealing with pressurized systems and requires specialized sampling devices, such as a Vezin sampler.

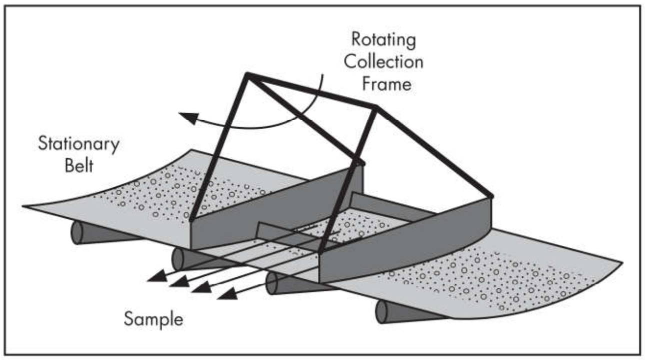


Figure 5 Example of a cross-belt sampler operating on a stationary conveyor belt

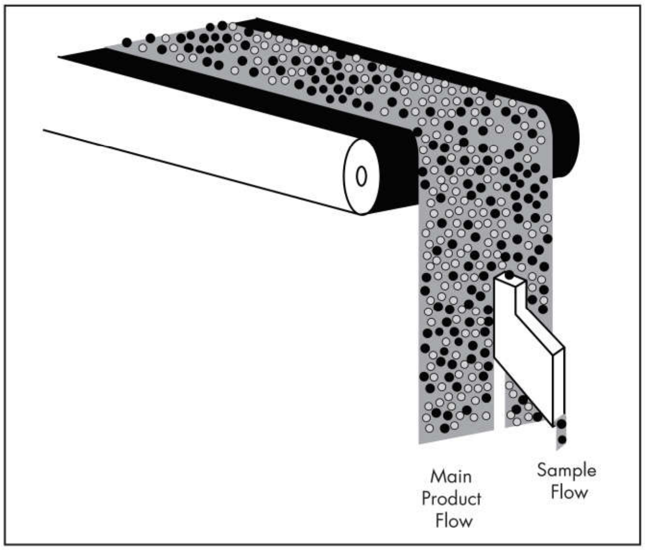
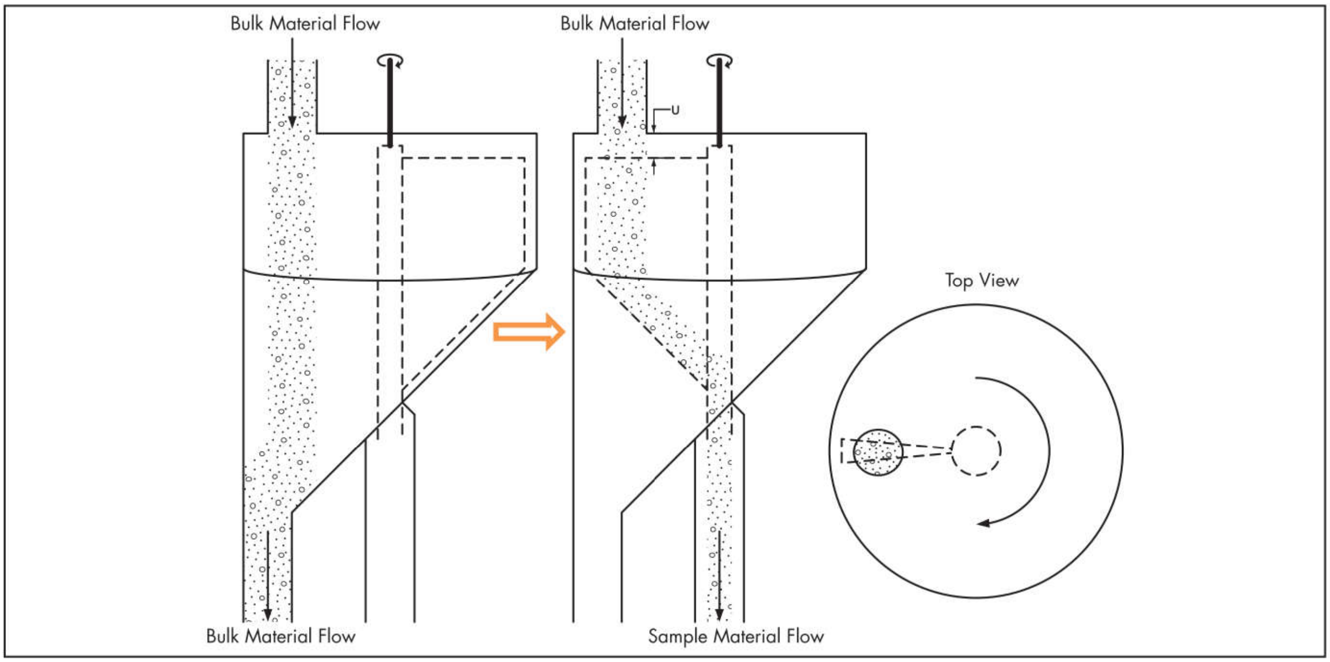


Figure 6 Example of a poor sampling technique: the stationary cut

Rotating Cut (Vezin Sampler)

A Vezin sampler consists of a sealed enclosure through which process liquids (or solids) flow vertically. A sample cutter rotates at a constant angular velocity to collect the sample increments and discharge them through a sample port, as shown in Figure 7. The key to correct sampling with a Vezin sampler is both the geometry of the cutter and the distance (u) between the top of the cutter and the liquid inlet. The cutter must be larger than the falling stream and be angled appropriately so that there is no bias in the part of the stream where the sample is being taken; see the top view in Figure 7. The distance, u, must be at least 3 times the size of the largest particle, or 0.5 cm for liquid streams. The sample cutter spins at a constant angular velocity and cuts increments from the process flow. The geometry of the cutter is important to obtain an unbiased cut; note the angular geometry of the cutter. If



Adapted from Wills 1992

Figure 7 Vezin sampler. The top view illustrates the geometry of the cutter.

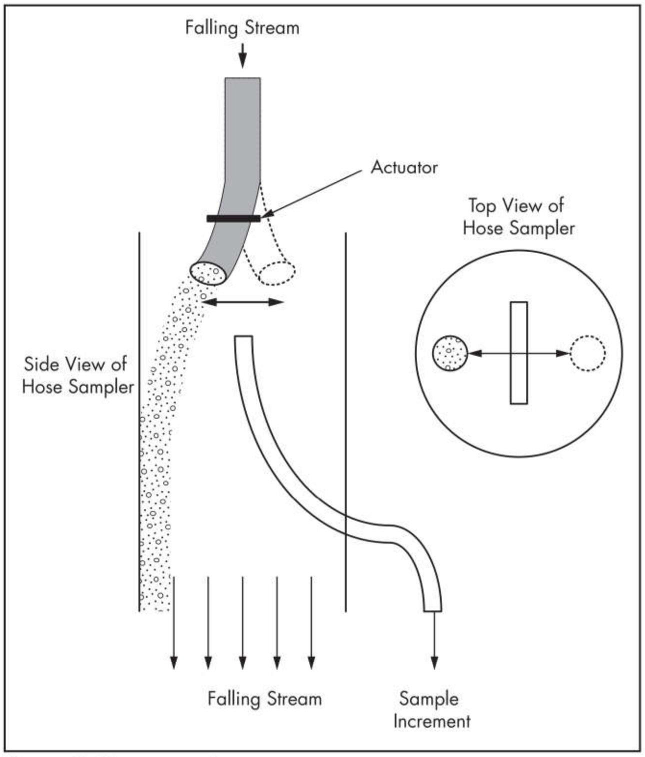


Figure 8 Hose sampler

using a Vezin sampler for sampling solids, this distance should be minimized to prevent dust buildup (Pitard 1993). Further design considerations can be found in Pitard's book, *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness, and Statistical Process Control*.

Hose Sampler

A hose sampler is a common method of taking representative samples with enhanced control over the time span between increments (Figure 8). A falling stream travels through a flexible hose. The trajectory of the falling material is changed using an actuator that moves the hose back and forth across the vessel at a constant velocity over the sample cutter. Each time the hose moves past the sample cutter, an increment is collected. Sampling by using this method offers greater flexibility than a Vezin sampler because the time interval between increments as well as the velocity of the stream crossing the cutter can be altered. With a Vezin sampler, only the angular velocity of the cutter can be altered.

Examples of Poor Liquid and Slurry Samplers

Many common types of sampling devices are available that attempt to collect a representative sample; however, they do not satisfy all the conditions outlined previously for process sampling. Two examples of in-stream sampling devices, an in-stream probe and a swing-gate sampler, can be seen in Figure 9A and 9B, respectively. The in-stream probe does not cut the entire stream and hence ignores radial heterogeneity. The swing-gate sampler opens on one side of the pipe and only captures a portion of the stream. This type of valve ignores any revolutionary heterogeneity. Both of these types of samplers work for a homogeneous or well-mixed process flow, if such a flow exists practically or statistically.

Sampling Process Gases

Taking a representative sample of a gas or liquid is a difficult task. This task is made even more difficult if the gas or liquid is flowing through a pipe. It is often difficult to obtain a representative cross-stream sample due to segregation of material caused by laminar flow. Laminar flow often causes the heavy fluid to settle out to the bottom of the pipe while the light fluid floats atop the heavy fluid (Figure 10).

There is currently no correct way to sample a gas or liquid flowing in a pipe, only recommendations to minimize bias. To minimize bias caused by segregation, it is important that the fluid be well mixed before sampling. Static mixers, as seen in Figure 10, can be used to mix gases and liquids. For a stream flowing horizontally, the sample probe should come out of the side of the pipe for liquids and out of the top for gases. The location of a sampling probe should be far enough downstream of a static mixer that the velocity profile of the fluid is well established.

When sampling flowing fluids, it is important to remember:

- Increasing the stream velocity does not ensure a well-mixed stream.
- A strainer is not sufficient for mixing; use a static mixer.

Sampling from Vessels

Sampling from vessels can be problematic because of the amount of material present and how the material may segregate over time. First, consider a large amount of *solids* being stored in a large vessel, as shown in Figure 11A. It is extremely time-consuming to cone and quarter a large quantity of material and then properly split it to achieve a representative sample. Therefore, tools have been designed that allow for easy sampling of bulk solids. One of the more common tools for bulk solids sampling is called a thief probe (Figure 11B). A thief probe is chiefly used for obtaining vertical core samples from bulk solids. The knob at the top is turned to open and close the sample windows along the length of the shaft. The pointed tip at the end is used to penetrate through the material. To use a thief probe, one must make sure the sample windows are completely closed before penetrating the material. Once the probe has been pushed through the material, the windows are opened to collect the sample. A thief probes work well for loosely packed solids.

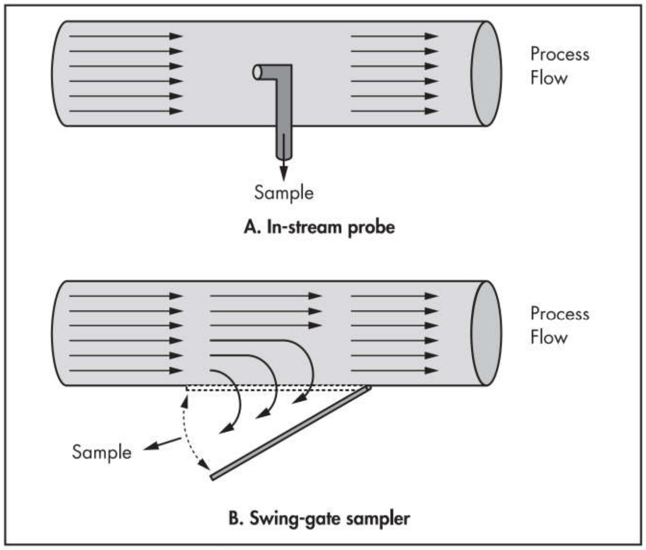


Figure 9 Examples of poor process sampling devices

Before using a thief probe, it is important to be aware of some of its limitations:

- The windows may not seal properly, causing sample leakage into the ports before the probe is fully inserted.
- Material may be too large to fall into the sample ports.
- If the solids are tightly packed, it will be difficult to push the probe through the material.

Now, consider obtaining a representative sample from a large amount of *liquid* being stored in a large vessel. As liquids are stored for long periods of time, the immiscible liquids tend to segregate from one another. Next, consider a mixture of oil and water. The water will eventually settle out on the bottom of the vessel and the oil will float atop the water, as shown in Figure 12A.

Obtaining a representative sample in this case is difficult because the immiscible nature of the liquids creates two distinct layers. If a sample were poured off the top, if would only contain oil; if a sample were taken from the bottom, it would only contain water. Therefore, a tool that utilizes the same principles as a thief probe is commonly used to obtain representative liquid core samples. The tool is called a coliwasa (composite liquid waste sampler), as shown in Figure 12B.

To operate a coliwasa, the instrument must be inserted to the bottom of the tank slowly with the bottom seal open. The coliwasa should be inserted as deep as possible to obtain a representative sample. Once the fluid from the tank has filled the coliwasa, the handle must be pulled to seal the bottom of the instrument. The coliwasa can then be slowly removed from the tank. The coliwasa must be sealed while pulling it from the tank or it will leak fluid back into the tank and the sample will not be representative.

Modern Sampling Devices

Slurries and liquids pose many issues to proper sampling, such as how does one split a continuously flowing stream down to a continuously flowing sample stream for online analysis? Recent developments in sampling technology have

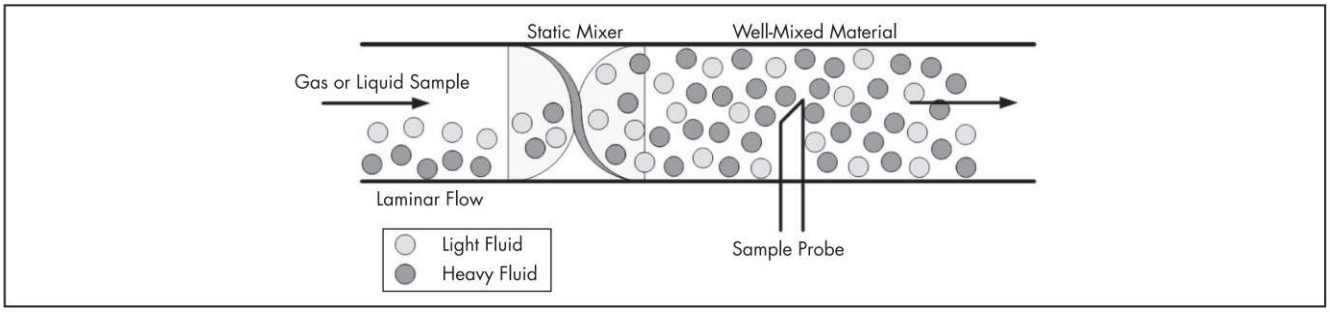


Figure 10 Sampling of gases and liquids from a pipe

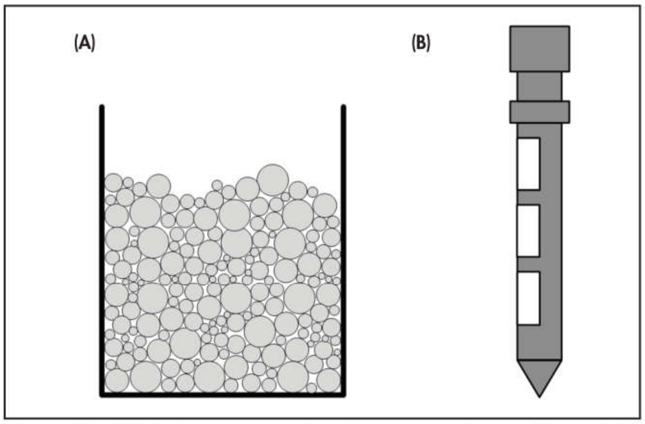


Figure 11 (A) Solids storage in a large vessel, and (B) a thief probe used for bulk solids sampling

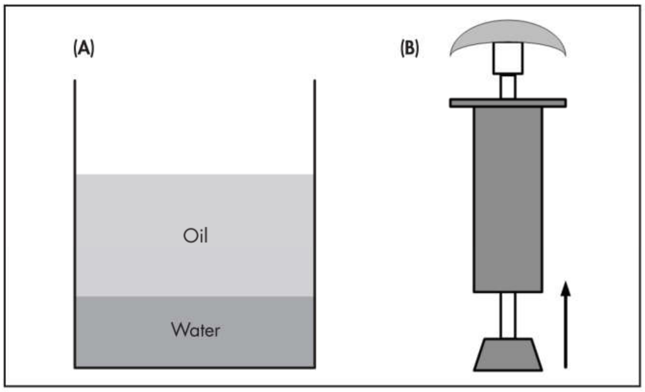


Figure 12 (A) Liquid storage in a large vessel, wherein the immiscible phases have separated over time. (B) The coliwasa is used to sample large quantities of liquid.

addressed some of these issues. One such sampling device is the Outokumpu slurry sampler depicted in Figure 13. It is used to sample high flow-rate slurries. This sampler reduces the flow rate of the slurry by taking multiple stages of cuts from the incoming process flow. Each stage has a lower incoming slurry flow rate, and the final stage is cut using a traditional linear traversing sample cutter (Alfthan 2003).

This type of sampler has two stages of stationary cutters. Although there are many stationary cutters per stage, it still violates the rules of sampling process flows. To remedy this issue, the first and second cut could be taken using a linear traversing cutter.

SPLITTING SAMPLES FOR LABORATORY ANALYSIS

Laboratory Grab Sample

A laboratory grab sample, otherwise known as the "four-corners method," is the simplest, quickest, and most flexible method, as it can be carried out on small quantities using spatulas, or on large quantities using shovels, and can divide the material into the number of samples desired. The material is first homogenized by thorough mixing on a rolling mat, as shown in Figure 14. The mat should be a smooth, flexible sheet that the sample will not stick to, such as glazed paper, hard vulcanized rubber, or smooth vinyl. The material is then divided into samples by randomly grabbing small amounts from the homogenized pile on the cloth, not unlike the method shown in Figure 2. This method uses the least equipment, but also is the most prone to human biases and has a higher variance between samples than other methods.

Laboratory Coning and Quartering Methods

Coning and quartering can be performed on small amounts of material in the laboratory as well. The method is the same as discussed earlier except with smaller equipment. This is typically carried out on a smooth, clean laboratory surface such as a lab bench or lab floor.

Riffle Splitters

Riffle splitters consist of a series of chutes that run in alternating directions, so that when material is poured into the top of the splitter, it flows through the chutes and is randomly divided into two equal-sized fractions. An example of a riffle splitter is shown in Figure 15. One of the fractions can then be split again, and the procedure can be repeated until a sample of the desired size is obtained. To work properly, these splitters must be fed using a special pan that is the same width as the top of the chutes, otherwise the amount of material entering the two end chutes will be different and the sample will not be representative. Also, if a material is repeatedly split into smaller fractions using a riffle, the errors from each stage of splitting will be added together, resulting in increasing variance between samples.

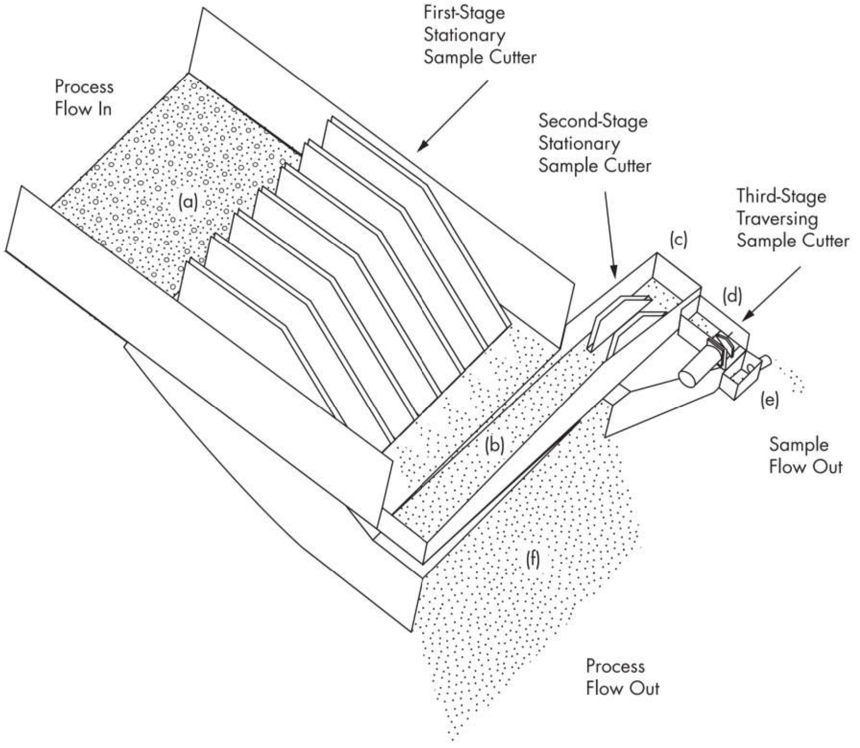
Rotary Riffle Splitters

The rotary, or spinning, riffle is the best method to use for dividing material into representative samples. It produces the lowest variance between samples of all sampling methods discussed thus far. It can also produce many samples in a single operation.

The material to be sampled is fed from a feed hopper to a feeder (usually a vibratory feeder, although screw feeders and small conveyors work as well). The feeder drops the material at a uniform rate into a series of bins (sample containers) on a rotating table, as shown in Figure 16. The turntable speed is set so that each sample container will pass under the end of the feeder numerous times before the feed hopper is emptied, but slowly enough that the bin edges do not strike the falling particles hard enough to bounce them into a different container or throw them out of the machine entirely (generally about 10 to 25 rpm). The turntable rotates at a constant angular velocity, providing representative increments.

Comparison of Laboratory Sampling Methods

A comparison of the relative standard deviations of samples made by the previously mentioned methods is given in Table 1. It can be clearly seen that rotary riffling is the best method of sample division and approaches the standard deviation that would be expected from an ideal sample divider where division of material into samples is perfectly random. Both coning and quartering and grab sampling perform relatively poorly, indicating that they should only be used when there are no other practical methods that will work with a given material. Riffle splitters give intermediate performance, indicating that they are suitable for routine, noncritical work.



(a) The feed slurry enters the sampler and is cut by several stationary cutters while the slurry is in free fall.
(b) The cuts are mixed in a trough and redirected toward a second stage of cutters. Again, the cuts are taken while the slurry is in free fall.
(c) The cuts are mixed in another trough.
(d) The cuts are redirected to a third stage where the slurry flow is cut in free fall using a traversing cutter.
(e) The resulting cut is the sample.
(f) The slurry that did not get cut in three stages of cutters is mixed in the outlet trough and sent to downstream processes.

Adapted from Alfthan 2003

Figure 13 Modern sampler

TYPICAL SOURCES OF ERROR IN SAMPLING

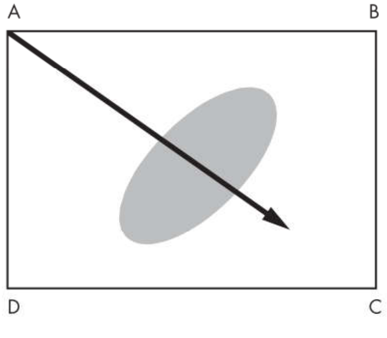
Poor sampling occurs because of poor technique selection, poor equipment maintenance, carelessness, haste, and lack of knowledge. When developing a sampling strategy, it is important to consider the following:

- Is the appropriate sampling equipment being used?
- Where could possible errors in sample handling occur?
  - Contamination
  - Loss
  - Unintentional mistakes

When sampling, always keep in mind the principle of correct sampling: Every part of the lot has an equal chance of being in the sample and the integrity of the sample must be preserved during and after sampling.

Appropriate Sampling Equipment

Choosing the appropriate equipment for sampling is important in obtaining a representative sample. Many times, equipment for sampling solids may not be suitable to sampling liquids and gases. For example, the rotary riffle splitter is great for



Note: Mix by first drawing corner A so that the sample rolls toward corner C. Then draw corner B to corner D, corner C to corner A, and corner D to corner B. This should be repeated at least 40 times to ensure proper mixing.

Figure 14 Four-corners method of mixing a sample for grab sampling

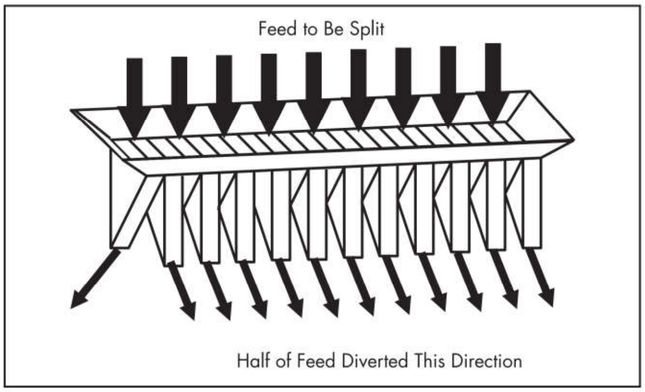


Figure 15 Riffle splitter, wherein the material flows through alternating-direction chutes and is split in half

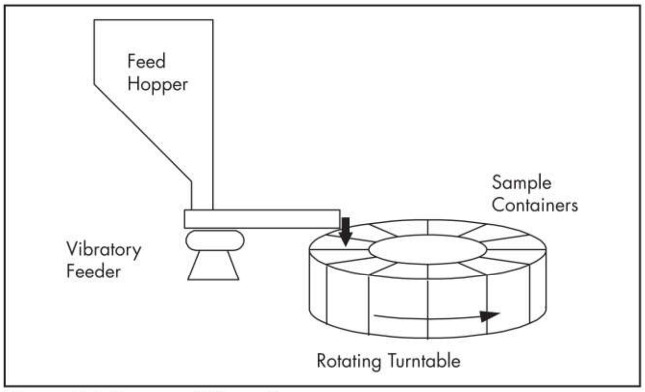


Figure 16 Rotary riffle splitter

Table 1 Standard deviations of samples produced from a 60%/40% mixture of fine and coarse sand

Sampling Method	Standard Deviation of Samples, %
Cone and quarter	6.81
Grab sampling	5.14
Riffle splitter	1.01
Rotary riffle splitter	0.125
Random variation for a theoretically perfect sampler	0.076

Source: Khan 1968; Allen and Khan 1970

splitting and sampling solids, but using a rotary riffle splitter for a liquid would be a poor choice of sampling equipment. Materials spills and splashing may occur, leading to errors in sample handling.

Sample Contamination

Sample contamination occurs when unwanted material is added to the sample after sampling but before chemical or physical analysis. The simplest source of contamination occurs because the sampling equipment has not been cleaned. If equipment is not cleaned, it is possible that material from a completely different product may contaminate the current sample. For example, because of static electricity, fine material often adheres to the sampling tool. If not cleaned properly after sampling, these fines may end up in the next sample. Sampling lines can become contaminated when they are not properly purged of old material. After using a sampling line, it must be purged the length of the line to avoid buildup of old material.

Chemical reactions between the sample material and sample container can also lead to unwanted atoms or molecules contaminating the sample. For example, if a sample is to be analyzed for sodium, it should not be stored in a glass container. When choosing a sample container, it is important to choose an inert environment for storage.

Sample Loss

Sample loss occurs when some of the sample mass, or a percentage of a specific component, is not retained after the sample is taken. Spills and splashes during or after sampling are the most common source of material loss during sampling. Material is also often lost during the crushing and grinding stages. Precious metals tend to smear onto the surfaces of crushing and grinding equipment, causing some material to remain behind and not become part of the sample. Care needs to be taken when transferring material from one storage container to another. When transferring material to another storage container, fines tend to be left behind. It is important that the number of transfers between containers be minimized to reduce the amount of fines lost.

Sample loss will also occur with materials that are susceptible to chemical reaction. For example, consider that the moisture content of a material is of interest. If the material is not stored in a humidity-controlled environment, moisture may be lost to the surrounding atmosphere, causing a loss in material.

Unintentional Mistakes

Unintentional mistakes are innocent errors that can severely compromise analytical results. Mislabeling of a sample is a common unintended mistake. For example, labeling a sample for ambient storage when it needs to be refrigerated may compromise the analytical results. Many times, unintentional mistakes can be avoided by simply paying more attention to what one is doing.

CALCULATIONS FOR DEVELOPING SAMPLING STRATEGIES

The best and most impractical way of determining the characteristics of a lot is to analyze the entire lot as a sample. However, this is not feasible in most situations. A smaller sample is typically analyzed and is assumed to be representative of the entire lot. So far, the various methods of taking these smaller samples have been discussed, but the amount of sample required to be considered representative has not yet been examined. This section details the calculations required to determine the amount of sample necessary to be statistically representative of the entire lot.

Gy's Method for Calculating Required Sample Size

Gy's method is a general-purpose calculation to determine the minimum size of sample needed to ensure that it will be representative of the whole lot, within specified statistical limits. Before using this method, approximate estimates of the following will be needed:

Table 2 The number of standard deviations away from the mean that can be expected at a given confidence interval

Confidence Interval	h
0.90	1.645
0.91	1.705
0.92	1.750
0.93	1.812
0.94	1.881
0.95	1.960
0.96	2.054
0.97	2.170
0.98	2.326
0.99	2.576
0.999	3.291
0.9999	3.890
0.99999	4.417
0.999999	4.892

- The content of the species of interest in the lot (critical content)
- The general shape of the particles
- The densities of the various species and phases present
- The particle size distribution
- The degree of liberation and the grain size

Basic Equations for Gy's Method

The basic calculation to determine the required sample weight, M, from a lot of total weight, W, is shown in Equations 1 and 2. This equation provides the mass of a sample required to be representative of the entire lot within a specific confidence interval, s.

$$s^2 = \left(\frac{1}{M} - \frac{1}{W}\right)Cd_{max}^3 \quad \textbf{(EQ 1)}$$

or

$$\frac{WM}{W - M} = \frac{Cd_{max}^3}{s^2} \quad \textbf{(EQ 2)}$$

where

s = value of the standard deviation that is needed for desired confidence interval
M = minimum sample weight needed, g
W = weight of entire lot being sampled, g
C = mineralogical factor for the material being sampled, g/cm^3
d_{max} = size of the largest particle in the lot (or top size), cm

When the weight of the lot being sampled, W, is much larger than M, this equation can be simplified to

$$M = \frac{Cd_{max}^3}{s^2} \quad \textbf{(EQ 3)}$$

Determining the Statistical Parameter: s

To calculate the desired value for s:

1. First select the *desired certainty range* (e.g., you might want a copper assay to be accurate to within ±0.1% copper).
2. Divide this value by the *mean probable assay value* (so for a 5% copper sample, you get 0.1/5 = 0.02).
3. Select the number of standard deviations, h, that will be needed to give the desired confidence interval (see Table 2).

$$s = \frac{\text{desired certainty range}}{\text{mean probable assay value}} \cdot \frac{1}{h} \quad \textbf{(EQ 4)}$$

Determining the Mineralogical Factor: C

The mineralogical factor for an ore body is a constant that depends on the physical characteristics of the ore particles. It can only be calculated if extensive information about the ore has been determined. In most circumstances, the variables used to determine the mineralogical factor are assumed from general observations and previous experience working with the particular ore. The mineralogical factor, C, can be calculated as follows:

$$C = f \cdot g \cdot l \cdot m \quad \textbf{(EQ 5)}$$

where

f = shape factor
g = size distribution factor
l = liberation factor
m = composition factor

Shape factor. The shape factor, f, corrects the mineralogical factor for particles that are not perfect cubes (f = 1). For most natural minerals, the shape factor is taken to be equal to 0.5. Flaky minerals, such as mica, have a shape factor of about 0.1. Materials that contain soft solids, such as gold, have a shape factor of about 0.2. Needle-like materials, such as asbestos, have a shape factor ranging from 1 to 10 (Pitard 1993).

Size distribution factor. The size distribution factor, g, is determined by estimating the 95% passing size, d_{95}, and the 5% passing size, d_5, and using the following guidelines:

- If $d_{95}/d_5 > 4$ (broad size distribution), then $g = 0.25$.
- If $2 < d_{95}/d_5 < 4$ (moderate size distribution), then $g = 0.5$.
- If $1 < d_{95}/d_5 < 2$ (narrow size distribution), then $g = 0.75$.
- If $d_{95}/d_5 = 1$ (mono-sized particles), then $g = 1$.

When size distribution information is not available, g should be estimated conservatively as a narrow size distribution having a value of 0.75.

Liberation factor. The liberation factor, l, is a measure of the degree of dispersion of the valuable material through the bulk, and of the homogeneity of the material. The liberation factor varies from 0 to 1, with 0 representing a perfectly homogeneous material and 1 representing a perfectly heterogeneous material. It is calculated from the following expression:

$$l = \sqrt{\frac{d_l}{d_{max}}} \quad \textbf{(EQ 6)}$$

where

d_l = liberation size, cm
d_{max} = size of the largest particle in the lot (or top size), cm

Generalizations can be made when insufficient data are provided to calculate an exact liberation factor. These generalizations are shown in Table 3.

Table 3 Generalizations for liberation factor when sufficient information is not available to calculate it

Degree of Heterogeneity of the Material	Approximate Liberation Factor
Very heterogeneous	0.8
Heterogeneous	0.4
Average	0.2
Homogeneous	0.1
Very homogeneous	0.05

Adapted from Pitard 1993

Composition factor. The composition factor, m, is calculated from the following formula:

$$m = \left(\frac{1-a}{a}\right)((1-a)r + at) \qquad \textbf{(EQ 7)}$$

where

a = fractional average assay of the critical component
r = specific gravity of the critical component, g/cm^3
t = specific gravity of the bulk material, g/cm^3

The fractional average assay of the critical component, a, is the estimated critical content of the bulk material in decimal format (not percent).

Example 1: Calculation Using Gy's Method

In this example, a nickel ore has a nickel sulfide (NiS) critical component containing 3.6% Ni (5.567% NiS) by weight. The top size of the ore particles is 1.2 cm, and the NiS grain size is 0.004 cm. The desired sampling accuracy is ±0.01% Ni (0.0155% NiS), with a 99% confidence interval (2.576 standard deviations). The specific gravity of the NiS is 5.3, the specific gravity of the bulk ore is 3.1, and it has a "broad" size distribution.

First, determine the shape factor, f. Nickel sulfide is a common natural mineral:

$$f = 0.5 \text{ (regular shape)}$$

Next, determine the size distribution factor, g. The problem statement claimed a broad size distribution:

$$g = 0.25 \text{ (broad size distribution)}$$

Determine the liberation factor, l. The liberation size and top size are given as 0.004 cm and 1.2 cm, respectively:

$$l = \sqrt{\frac{d_l}{d_{max}}} = \sqrt{\frac{0.004}{1.2}} = 0.05774$$

The critical content is given as 5.567% NiS:

$$a = 0.05567$$

The specific gravity of the critical component (NiS) and the bulk ore are given as 5.3 and 3.1, respectively:

$$r = 5.3 \text{ g/cm}^3$$

$$t = 3.1 \text{ g/cm}^3$$

With this information, the composition factor can be calculated as follows:

$$\begin{aligned} m &= \left(\frac{1-a}{a}\right)((1-a)r + at) \\ &= \left(\frac{1-0.05567}{0.05567}\right)((1-0.05567)5.3 + 0.05567 \cdot 3.1) \\ &= 87.826 \text{ g}/\text{cm}^3 \end{aligned}$$

All of the variables in the mineralogical factor, C, have now been determined. C can now be calculated as follows:

$$C = fglm = 0.5 \cdot 0.25 \cdot 0.05774 \cdot 87.826 = 0.634 \text{ g}/\text{cm}^3$$

The statistical parameter, s, can be calculated using the desired sampling accuracy, 0.0155% NiS, the critical content, 5.567% NiS, and the number of standard deviations correlating with the 99% confidence interval, 2.576:

$$s = \frac{0.0155\%}{5.567\%}\frac{1}{2.576} = 0.001079$$

All required information has now been calculated to predict a sample size that would be considered representative of the lot at a 99% confidence interval:

$$\begin{aligned} M &= \frac{Cd_{max}^3}{s^2} = \frac{0.634(\text{g/cm}^3)(1.2\text{ cm}^3)}{0.001079^2} \\ &= 940{,}572 \text{ g, or 0.9 metric tons} \end{aligned}$$

Gaudin's Method for Calculating the Required Sample Size

Gaudin's method is a derivation of Gy's method using assumptions typically found in ores with the critical component being a precious metal (gold, platinum, diamonds, etc.). This method is limited to ores where the critical content is a small fraction (a few percent or less) of the total volume (Gaudin 1967).

Basic Equations for Gaudin's Method

The basic calculation to determine the required sample weight, S, from a lot is shown in Equation 8. This equation provides the mass of a sample required to be representative of the entire lot within a specific confidence interval, y:

$$S = \frac{n}{n'} \qquad \textbf{(EQ 8)}$$

where

S = sample weight required for representativity, g
n = number of particles that are required for a representative sample
n′ = number of particles in 1 g of material in the lot, 1/g

Determining the Number of Particles Required: n

To determine the number of particles required for a representative sample, the approximate volumetric critical content, x, and the allowable volumetric error, y, must be known:

$$n = 0.45\frac{x}{y^2} \qquad \textbf{(EQ 9)}$$

where

x = approximate volumetric critical content of the lot, decimal fraction
y = allowable volumetric error, decimal fraction

Often, the critical content and allowable error is given as a weight percent instead of a volume fraction. These can be converted to volume fractions given the density of the critical component and the density of the bulk material in the lot.

$$x = \frac{\text{critical content, \%/wt}}{\text{critical component density}} \cdot \frac{\text{bulk material density}}{100\%}$$

$$y = \frac{\text{allowable error, \%/wt}}{\text{critical component density}} \cdot \frac{\text{bulk material density}}{100\%}$$

Determining the Number of Particles per Gram: n′

To make a conservative estimate of the number of particles per gram of material, n′, it is necessary to know the density as well as the top size of the bulk lot:

$$n' = \frac{6}{\rho_s \cdot d_{max}^3} \tag{EQ 10}$$

where ρ_s is the density of the bulk material, in g/cm^3.

Example 2: Calculation Using Gaudin's Method

In this example, a nickel ore has a nickel sulfide (NiS) critical component containing 3.6% Ni (5.567% NiS) by weight. The top size of the ore particles is 1.2 cm, and the NiS grain size is 0.004 cm. The desired sampling accuracy is ±0.01% Ni (0.0155% NiS), with a 99% confidence interval (2.576 standard deviations). The specific gravity of the NiS is 5.3, the specific gravity of the bulk ore is 3.1, and it has a "broad" size distribution.

First, the critical content and allowable error must be converted to volumetric fractions from weight percent:

$$x = \frac{\left(\frac{5.567}{5.3}\right)}{\left(\frac{100}{3.1}\right)} = 0.0326\%$$

$$y = \frac{\left(\frac{0.0155}{5.3}\right)}{\left(\frac{100}{3.1}\right)} = 0.000091\%$$

Next, the number of particles required to obtain a representative sample can be calculated:

$$n = 0.45\frac{x}{y^2} = 0.45\frac{0.0326}{0.000091^2} = 1{,}783{,}000 \text{ particles}$$

Using the provided lot density and top size, the number of particles per gram can be estimated:

$$n' = \frac{6}{\rho_s \cdot d_{max}^3} = \frac{6}{3.1 \cdot 1.2^3} = 1.12007 \text{ particles/g}$$

Finally, the sample size required for representativity can be calculated:

$$S = \frac{n}{n'} = \frac{1783000}{1.12007} = 1{,}591{,}617 \text{ g, or 1.6 metric tons}$$

This calculation was performed on a nickel sulfide ore, not a precious metal ore. The sample size predicted by Gaudin's method is much larger than necessary because of the assumption made when defining this method.

Comparison of Gy's and Gaudin's Methods

Gy's method is a general-purpose sampling equation. Gaudin's method is specifically intended for use in calculating sample sizes for precious metals, and it makes a number of simplifying assumptions based on the fact that precious metals make up a minute fraction of the mass of the ore (typically only a few grams per metric ton).

Gaudin's equation can be put into the same terms as Gy's equation by using the same symbols for equivalent values: S = M. The value of y (a volume fraction) can be converted into s (a weight fraction) by multiplying it by a constant, k, that depends on the relative densities of the ore and the valuable minerals, and so, y = s/k. If these substitutions are made, then Gaudin's equation becomes

$$M = 0.075 \cdot x \cdot \rho_s \cdot k^2 \frac{d_{max}^3}{s^2} \tag{EQ 11}$$

If we now take $(0.075 \cdot x \cdot \rho_s \cdot k^2) = C$, it can be seen that Gaudin's equation reduces to the same form as Gy's equation. Gaudin's equation is therefore simply a special case of Gy's equation.

Hassiali's Method for Development of Incremental Sampling Strategy

This method is intended for designing a mechanical sampling system that will account for normal variations in the characteristics of the process stream being sampled. A sample cutter is used to collect a series of test increments over a particular time interval, and then each increment is assayed separately. The variations in the assays between increments are then used to calculate the number of increments that the sample cutter needs to collect over that span of time to give the desired confidence interval in the results. Correct increment sampling should produce a normal distribution curve where individual increments are plotted against increment frequency.

Basic Equations for Hassiali's Method

To calculate the number of increments required from a process flow, n, to reach a desired confidence interval of the measured critical content, the mean, $\bar{x}$, and standard deviation, σ_s, between experimental measurements of the critical content of individual increments is necessary (Taggart and Behre 1945). The basic calculation for Hassiali's method is as follows:

$$n = \left(\frac{h \cdot \sigma_s}{\bar{x} \cdot z}\right)^2 \tag{EQ 12}$$

or

$$n = \left(\frac{\sigma_s}{\bar{x} \cdot s}\right)^2$$

where

n = number of increments required for desired confidence interval
h = number of standard deviations corresponding to desired confidence interval
σ_s = experimental standard deviation of the measured critical content
$\bar{x}$ = experimental mean of the measured critical content
z = allowable sampling error
s = value of the standard deviation that is needed for desired confidence internal as described by Equation 4

The number of standard deviations corresponding to the desired confidence interval, h, can be found in Table 2. The allowable sampling error, z, is expressed as a fraction of the critical content. The value of z is equivalent to the first fraction in Equation 4:

$$z = \frac{\text{desired certainty range}}{\text{mean probable assay value}}$$

Example 3: Calculation Using Hassiali's Method

For this example, the following 11 test increment assays were obtained from increments collected at 30-minute intervals (total elapsed time of 5.5 hours). Values are weight % Ni.

3.61	3.69	3.58	3.65	3.56	3.63	3.61	3.66	3.57	3.59	3.60

Calculate the number of increments that must be collected over this same time interval so that the probability of the assay of the final composite sample is accurate to within 1% of the true assay at a 99% confidence interval.

First, calculate the mean and standard deviation of the increment assays:

$$\bar{x} = \frac{\sum x_i}{N} = 3.614$$

$$\sigma_s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N - 1}} = 0.0403$$

where

x_i = assay value of each increment
N = total number of increments

Next, refer to Table 2 for h at a 99% confidence interval:

h = 2.576 for 99% confidence interval

The value of z is given as 0.01:

z = 0.01 for 1% accuracy

Finally, all of the information has been determined to calculate the number of increments required for a 99% confidence interval and ±1% accuracy:

$$n = \left(\frac{h \cdot \sigma_s}{\bar{x} \cdot z}\right)^2 = \left(\frac{2.576 \cdot 0.0403}{3.614 \cdot 0.01}\right)^2 = 8.26$$

Round up to 9 increments because one cannot take fractional increments.

TERMS AND SYMBOLS USED IN THIS CHAPTER

Term	Definition
ρ_s	Density of the bulk material (Gaudin's method)
σ_s	Experimental standard deviation of the measured critical content (Hassiali's method)
a	Fractional average assay of the critical component (Gy's method)
C	Mineralogical factor for the material being sampled (Gy's method)
Component	A particular substance within a lot of heterogeneous material. For example, silica, pyrite, and gold are typical components of gold ore.
Content	The concentration of a component within a lot of heterogeneous material. For example, a gold ore may have a silica content of 65%.
Critical component	The component of the heterogeneous material that is of interest. For example, gold is the critical component in gold ore.
Critical content	The concentration of the critical component within a lot of heterogeneous material. For example, a gold ore may have a critical content of 0.01% gold.
d_5	5% passing size of the material being sampled (Gy's method)
d_{95}	95% passing size of the material being sampled (Gy's method)
d_l	Liberation size of the material being sampled (Gy's method)
d_{max}	Size of the largest particle in the lot (or top size) (Gy's method)
f	Shape factor for the material being sampled (Gy's method)
g	Size distribution factor for the material being sampled (Gy's method)
h	Number of standard deviations corresponding to the desired confidence interval (Gy's method and Hassiali's method)
Heterogeneous material	A material that has different properties depending on where the material is analyzed. Examples include slurries, immiscible fluids, and even tap water.
Homogeneous material	A material that is either one substance or a perfect mixture of multiple substances with constant thermodynamic properties throughout. By definition, perfectly homogeneous materials do not exist and cannot be created.
l	Liberation factor for the material being sampled (Gy's method)
Lot	A lot is defined as a large (bulk) quantity of material from which a sample must be taken. If proper sampling is performed, analysis of the sample will be representative of the lot within a specified degree of certainty (or confidence interval).
M	Minimum sample weight needed for a representative sample of a lot (Gy's method)
m	Composition factor for the material being sampled (Gy's method)
N	Total number of increments (for calculating standard deviation)
n	Number of particles that are required for a representative sample (Gaudin's method) or number of increments required for desired confidence interval (Hassiali's method)
n′	Number of particles in 1 g of material in the lot (Gaudin's method)
r	Specific gravity of the critical component (Gy's method)
s	Value of the standard deviation that is needed for the desired confidence interval (Gy's method and Hassiali's method)
S	Minimum sample weight needed for a representative sample of a lot (Gaudin's method)

Term	Definition
t	Specific gravity of the bulk material (Gy's method)
W	Weight of the entire lot being sampled (Gy's method)
x	Approximate volumetric critical content of the lot, decimal fraction (Gaudin's method)
$\bar{x}$	Experimental mean of the measured critical content (Hassiali's method)
x_i	Assay value of each increment (for calculating mean)
y	Allowable volumetric error, decimal fraction (Gaudin's method)
z	Allowable sampling error (Hassiali's method)

REFERENCES

Alfthan, C.V. 2003. *Method and Apparatus for Taking Slurry Samples*. G01N 1/20 ed. Finland: Outokumpu.

Allen, T., and Khan, A.A. 1970. Critical evaluation of powder sampling techniques. *Chem. Eng.* 238:108–112.

Gaudin, A.M. 1939. *Principles of Mineral Dressing*. New York: McGraw-Hill.

Gaudin, A.M. 1967. *Principles of Mineral Dressing*. New York: McGraw-Hill.

Gy, P.M. 1979. *Sampling of Particulate Materials: Theory and Practice*. Amsterdam: Elsevier.

Khan, A. 1968. Critical evaluation of powder sampling procedures. Master's thesis, University of Bradford, West Yorkshire, United Kingdom.

Morrison, R.D. 2008. *An Introduction to Metal Balancing and Reconciliation*. Indooroopilly, Queensland: Julius Kruttschnitt Mineral Research Centre.

Pitard, F.F. 1993. *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness, and Statistical Process Control*, 2nd ed. Boca Raton, FL: CRC Press.

Taggart, A.F., and Behre, H.A. 1945. *Handbook of Mineral Dressing: Ores and Industrial Minerals*. New York: John Wiley and Sons.

Wills, B.A. 1992. *Wills' Mineral Processing Technology*, 5th ed. New York: Pergamon Press.

Wills, B.A., and Finch, J.A. 2016. Wills' *Mineral Processing Technology*, 8th ed. Oxford: Elsevier/Butterworth-Heinemann.