

Sampling

References:

- Taggart, Handbook of Mineral Dressing, Wiley, 1945, Section 19
- Allen, Particle Size Measurement, Chapman and Hall, 1981
- Khan, Thesis, Bradford University, 1968

Introduction:

Sampling procedures cover the practice of selecting representative quantities of test material in the field, to evaluate bulk materials. Examples of the test materials are bulk granular solids, slurries, sludges, grains, and solid fuels. It is necessary to be able to sample bulk materials during shipment and during processing operations. Taggart (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).”

Thus with respect to the samples, it is very important that the sample be representative. In reality, the condition that the sample be completely representative of the whole as regards all aspects except bulk, is never fulfilled when heterogeneous materials are sampled. For instance, you can never get a perfectly representative sample of water from Lake Superior. 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 the purpose of the sample would be, and then plan the sampling procedure accordingly so that it will be sufficiently representative for your purposes.

Very often, when the material changes with time or with exposure to air, it is required that the sample also be fresh. Samples which are known to degrade, oxidize, or change on contact with air or water should be sealed in a tank, kept in an inert, atmosphere, and/or refrigerated or frozen. However, it should be remembered that all we are doing is slowing down the degradation process

Methods of Sampling from a Conveyor Belt:

Several methods for sampling material from a conveyor belt are available, and the method selected depends upon the accuracy desired, manpower 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
3. The cutter must have an opening width that is at least three times larger than the largest particle to be sampled, and 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.

There are three basic methods for collecting samples:

Linear Traversing Cut

The cutter moves across the stream in a straight-line path. Usually the path is perpendicular to the direction of the flow, but can be in the direction of the flow or against the direction of the flow. If adjusted properly, this is the most accurate type of sampler as it follows all six of the principles given above, but it is also the most expensive and hardest to maintain.

Rotational Traversing Cut

The cutter is mounted on a rotating axle and moves in an arc through the stream. These cutters are mechanically simpler and cheaper than linear traversing cut samplers, but they violate principle #2 (constant speed) and so the sample is not as high-quality.

Stationary Cut

The cutter remains stationary and cuts a particular part of the stream at all times. These cutters are very cheap, and are used very often. However, they violate both principle #1 (cut entire stream) and principle #2 (constant speed). As a result, the samples they collect are inherently less representative and less reliable than either of the other two cutter types.

A typical cross-chute sampler, which is a linear traversing cut type, is shown in Figure 1.

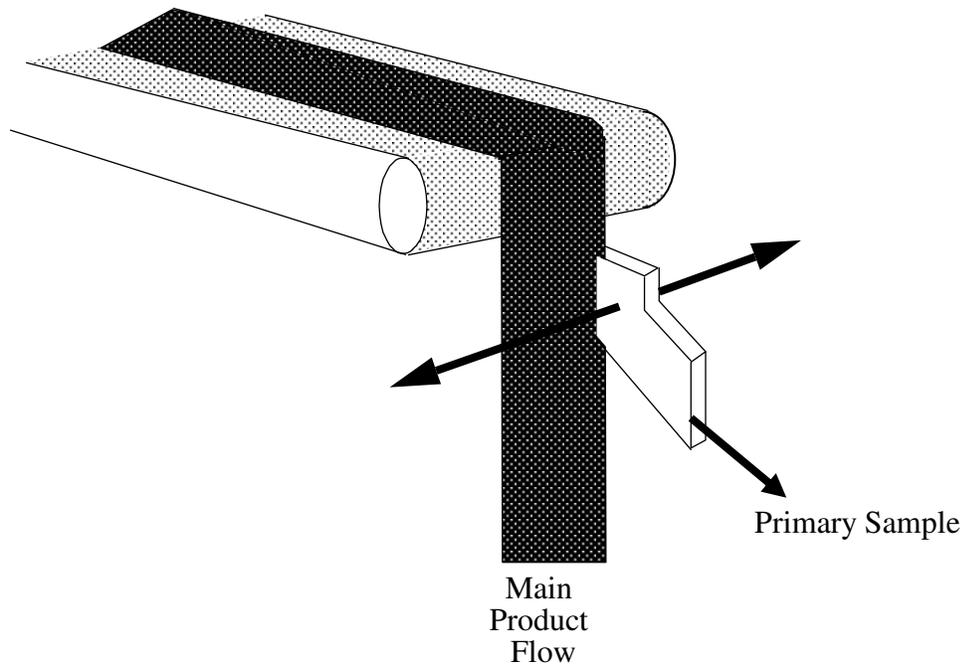


Figure 1. A cross-chute sampler for dry bulk solids. The primary sample is diverted from the main product flow onto a conveyor or into a collection bin.

Methods for Sampling Material in a Laboratory

In a laboratory before performing a size analysis, chemical analysis, or any other determination of material properties, it is necessary to take a representative sample from the lot being analyzed. A very detailed discussion of procedures for laboratory sampling is given by Allen (1981). For most metallurgical or environmental operations, the methods discussed in the following sections are adequate.

Grab Sampling

This 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 however many samples are desired. The material is first homogenized by thorough mixing on a rolling mat, as shown in Figure 2. 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, as shown in Figure 3. 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.

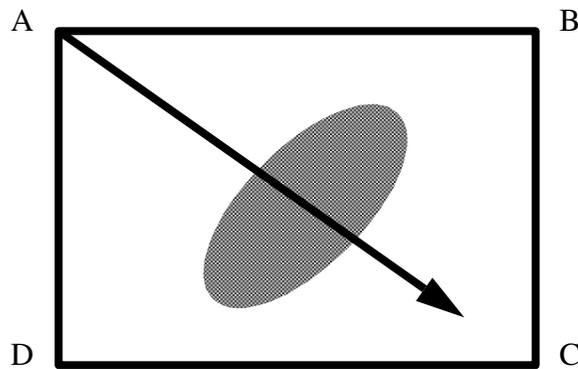


Figure 2: Mixing a sample on a rolling mat. Mix by first drawing corner A so that the sample rolls towards C, then drawing corner B to corner D, then drawing corner C to corner A, then corner D to corner B, then repeat.

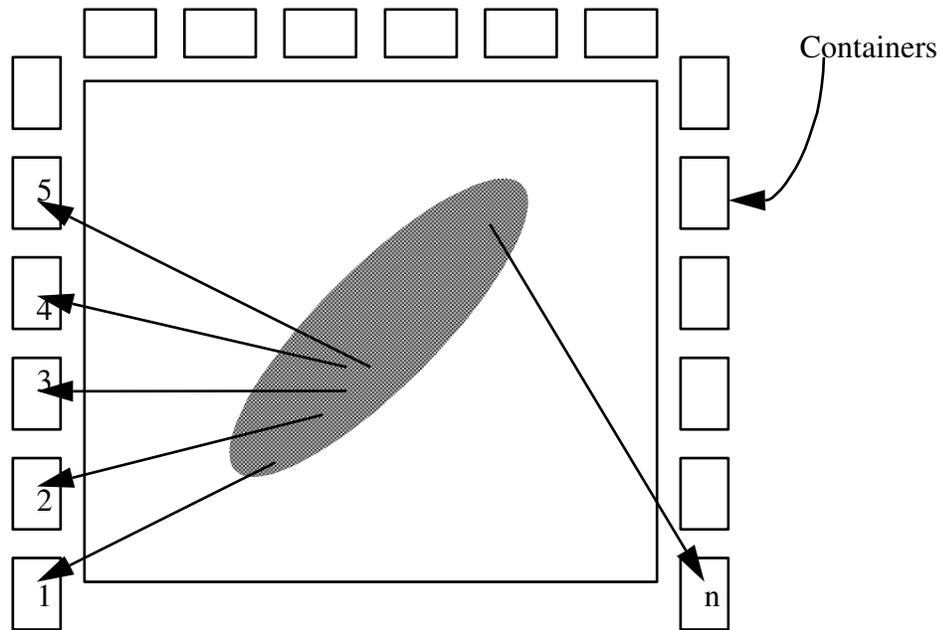


Figure 3: Dividing the mixed sample into subsamples, by incremental division with a spatula.

Coning and Quartering

This method is well-suited for large lots of material, and can be conveniently done using shovels, or even front-end loaders for very large samples. First, the material is mixed and shoveled into a uniform conical pile, as shown in Figure 4a. 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 4b. This disk is then divided into quarters using perpendicular boards, as shown in Figure 5. One pair of opposite quarters is removed, and the other pair is used as the sample. If the sample is too large, then it can be coned and quartered again until the desired sample size is obtained. This procedure is also prone to human error.

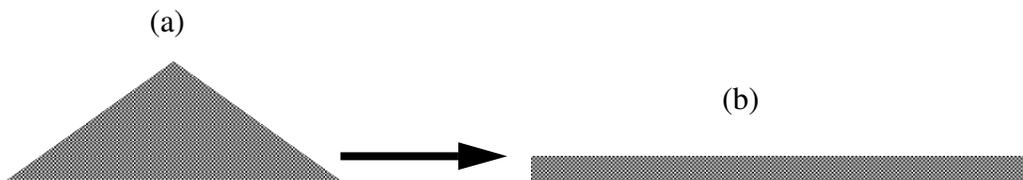


Figure 4: Side view of sample (a) in conical heap, and (b) after flattening

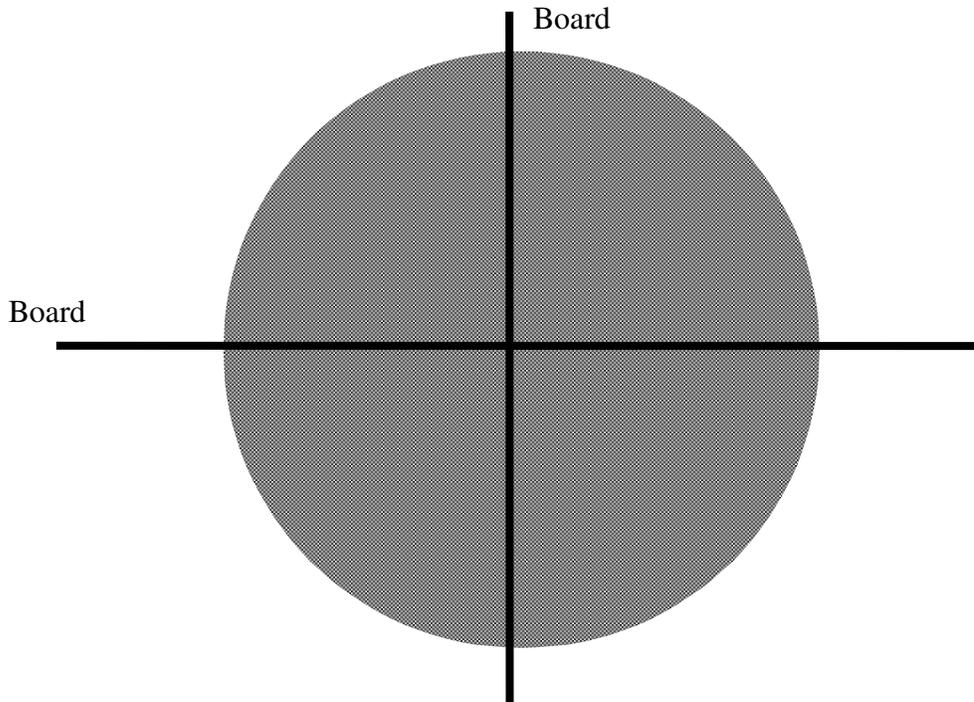


Figure 5: Divide the sample into quarters. Use boards to delineate them so that the sample can be divided precisely.

Sample Splitters

These are often called riffle or chute splitters, and a typical splitter is shown in Figure 6. These 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. One of the fractions can then be split again, and the procedure can be repeated until a sample of the desired size is obtained. In order to work properly, these splitters must be fed using a special pan that is exactly 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 be incorrect. 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.

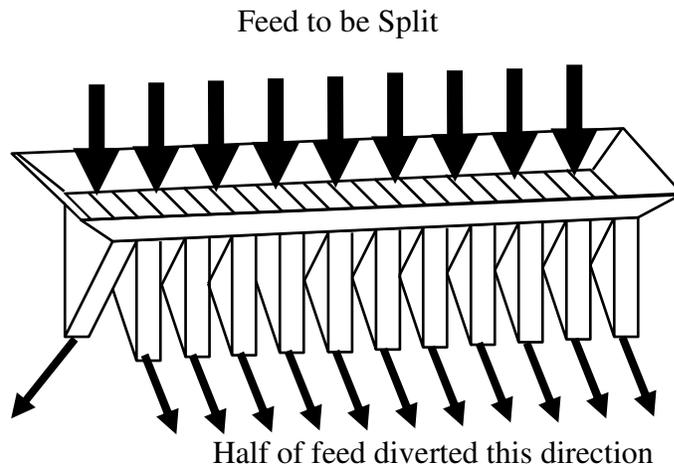


Figure 6. Chute-type sample splitter. The material flows through the alternating-direction chutes, and is split in halves.

Rotary Riffle.

The rotary, or spinning, riffle is the best method to use for dividing material into representative samples, because it produces the lowest variance between samples, and can produce a large number of samples in a single operation. In these riffles, the material to be sampled is fed from a feed hopper to a feeder (usually a vibratory feeder, although a screw feeder or small conveyor would work as well). The feeder drops the material at a uniform rate into a series of bins on a rotating table, as shown in Figure 7. 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).

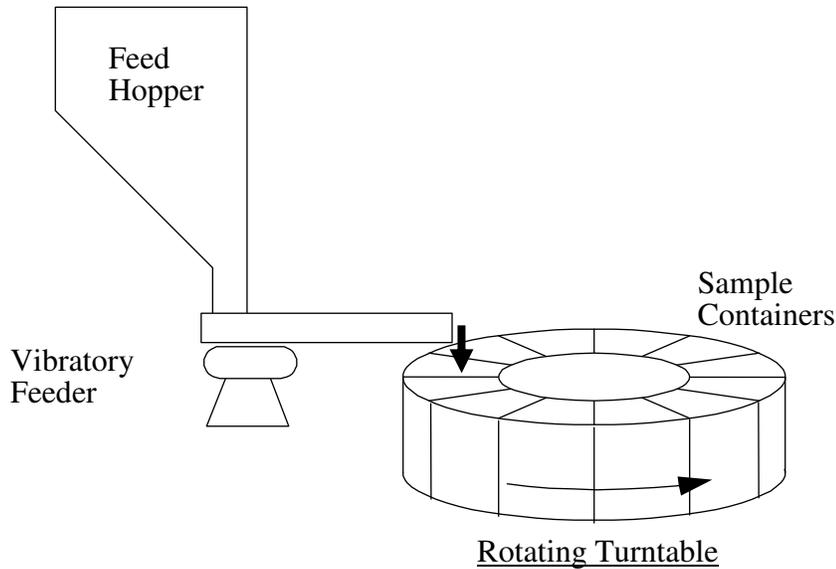


Figure 7. Rotary riffle splitter. Material is split into multiple samples in a single operation. In the example shown, the material is split into 12 identical fractions, which allows easy division of the bulk material into halves, thirds, quarters, sixths, or twelfths.

Comparison of Laboratory Sampling Devices

A comparison of the relative standard deviations of samples made by the above methods is given in Table 1. It can be clearly seen that rotary riffling is by far 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 & 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. Chute-type sample splitters give intermediate performance, indicating that they are suitable for routine, non-critical work.

Table 1: Standard deviations of samples produced from a 60/40% mixture of fine and coarse sand (Khan, 1968)

Sampling Method	Standard Deviation of Samples, %
Cone & Quarter	6.81
Grab Sampling	5.14
Chute-Type Sample Splitter	1.01
Rotary Riffle	0.125
Random variation for a Theoretically Perfect Sampler	0.076

Sampling Calculations Using Gy's Method

References:

- Gy, P. M. (1979), Sampling of Particulate Materials: Theory and Practice, Elsevier
- Wills, B. A. (1992), Mineral Processing Technology, 5th edition, Pergamon Press, pg. 79

Use:

This 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 limits.

Before using, approximate estimates of the following will be needed:

- The content of the species of interest in the lot (assay)
- 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 Equation:

$$\frac{WM}{W - M} = \frac{Cd_{\max}^3}{s^2}$$

Where:

- M= Minimum sample weight needed, grams
- W= Weight of the entire lot being sampled, grams
- C= Sampling constant for the material being sampled, g/cm³
- d_{max}=Dimensions of the largest pieces in the sample, cm
- s= value of the standard deviation that will be needed to give the desired level of assurance (assay units, such as % wt, ounces per ton, etc.)

When W is much larger than M, this equation can be simplified to:

$$M = \frac{Cd_{\max}^3}{s^2}$$

To calculate the desired value for s, first select the desired certainty range (for example, you might want a copper assay to be accurate to within +/- 0.1% copper), then divide this value by the mean probable assay value (so for a 5% copper sample, you get 0.1/5 = 0.02). Then, select the number of standard deviations that will be needed to give the desired certainty. For a probability of 95%, this is 1.96 standard deviations, so 1.96s=0.02, and so s=0.0102. If the desired certainty is 99%, then 2.576 standard deviations are needed, and so for this case s = 0.02/2.576 = 0.0078

Calculating C is more complicated, as follows:

$$C = fglm$$

where: f= Shape factor
 g= Size distribution factor
 l= Liberation factor
 m= Composition factor

The shape factor, *f*, is taken to be equal to 0.5 for most common natural materials, but in cases where the material of interest is highly flattened and elongated, the shape factor is set at 0.2.

The size distribution factor, *g*, is determined by estimating the 95% passing size (*d*) and the 5% passing size (*d'*), and using the following guidelines:

- If $d/d' > 4$, then $g=0.25$;
- If $2 < d/d' < 4$, then $g=0.5$;
- If $d/d' < 2$, then $g=0.75$;
- If $d/d' = 1$ (monosized particles), then $g=1$.

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. It is calculated from the expression:

$$l = \sqrt{\frac{L}{d}}$$

where: L= the size where the values are essentially completely liberated (grain size), cm.

The composition factor, *m*, is calculated from the formula:

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

where: r= specific gravity of the valuable component
 t= specific gravity of the remainder of the material
 a= fractional average assay of the valuable substance (for example, if an iron-bearing slag is being analyzed, and the iron is present as Fe₂O₃, use the fraction of the material that is Fe₂O₃, not the fraction that is iron, and it would be expressed as a decimal fraction, not as a percent)

Calculation with Incomplete Information

For a truly unknown sample, most of the parameters that go into calculating *C* will be unknown, and may even be impossible to guess with any accuracy. In this case, make the following conservative assumptions:

$f=0.5$ (normal blocky particles);

$g=0.75$ (narrow size distribution. Use $g=1$ if the sample is obviously monosized);

$l = 1$ (grains are as large as the particles)

The value of *m* will still need to be calculated, based on your best estimate of the assay of the sample and the densities of the components of interest. This initial sample will be larger than necessary, but can be analyzed and used to refine the calculations for collection of any subsequent samples.

Example Calculation:

Bulk Material Parameters:

Material of Interest: CuFeS_2 in a silica matrix, 1.5% Cu (4.3318% CuFeS_2); Top Size= 1.5 cm;

CuFeS_2 grain size= 0.01 cm

Desired Sampling Accuracy: +/- 0.02% Cu, certainty of 0.99 (2.576 standard deviations)

CuFeS_2 specific gravity= 4.2; Overall specific gravity: 2.8; Broad size distribution

Calculations:

(Calculations were done using a spreadsheet, and were carried out to more digits than are shown here. Carrying out the calculations with the values as shown will give slightly different results due to roundoff variations.)

$f = 0.5$ (regular shape)

$g = 0.25$ ($d/d' > 4$ for a broad size distribution)

$$l = \sqrt{\frac{L}{d}} = \sqrt{\frac{0.01}{1.5}} = 0.081650$$

$a = 0.043318$, $r = 4.2$, and $t = 2.8$, and so:

$$m = \left(\frac{1 - 0.043318}{0.43318} \right) ((1 - 0.43318)4.2 + (0.043318)2.8) = 91.4177$$

$$C = fglm = (0.5)(0.25)(0.081650)(91.4177) = 0.933028$$

$$2.576s = \frac{0.02}{1.5} = 0.01333, \text{ so } s = 0.0051758$$

$$M = \frac{Cd^3}{s^2} = \frac{0.933028(1.5^3)}{0.0051758^2} = 117,539 \text{ grams of sample needed for 99\% certainty that the assay}$$

will be within +/- 0.02% Cu of the true assay.

Since the original data was only carried to 2 significant digits, this should be rounded to 120,000 grams of sample, or 120 kg.

Sampling Calculations using Gaudin's Method

Reference:

- A. M. Gaudin, Principles of Mineral Dressing, McGraw-Hill, 1939

Use:

This method is limited to materials where the species of interest is a small fraction (a few percent or less) of the total volume. It does not allow for selecting the degree of certainty desired in the results, and it is not as generally useful or as precise as Gy's method. It would probably be most useful for calculating the size of a preliminary sample where minimal information about the material characteristics is available.

Basic Equations:

$$n = 0.45 \frac{x}{y^2}$$

$$n' = \frac{6}{\rho_s a^3}$$

$$S = \frac{n}{n'}$$

where:

- x = volume fraction occupied by the species of interest
- y = variation in volume fraction that would correspond to the maximum allowable error
- n = number of particles that will be needed in the final sample
- n' = estimated number of particles per gram
- ρ_s = density of the bulk sample, in grams/cm³
- a = top size of the material being sampled, in cm
- S = total sample weight needed, in grams.

Example Calculation:

Bulk Material Parameters:

Same material as for the previous example:

CuFeS₂, 1.5% Cu (4.3318% CuFeS₂); Top Size= 1.5 cm;

Desired Sampling Accuracy: +/- 0.02% Cu (+/- 0.0578% CuFeS₂)

CuFeS₂ specific gravity= 4.2; Overall specific gravity: 2.8

a = 1.5 cm

ρ_s = 2.8 gm/cm³

(Calculations were done using a spreadsheet, and were carried out to more digits than are shown here. Carrying out the calculations with the values as shown will give slightly different results due to roundoff variations.)

$$x = \frac{\left(\frac{4.3318}{4.2}\right)}{\left(\frac{100}{2.8}\right)} = 0.028879$$

$$y = \frac{\left(\frac{0.0578}{4.2}\right)}{\left(\frac{100}{2.8}\right)} = 0.00038505$$

$$n = 0.45 \left(\frac{0.028879}{0.00038533^2} \right) = 87650$$

$$n' = \frac{6}{(2.8)(1.5^3)} = 0.63492$$

$$S = (87650)/(0.63492) = 138,050 \text{ grams}$$

(compared to 117,599 grams using Gy's method)

Again, this should be rounded to 2 significant digits, and so 140,000 grams, or 140 kg, of sample is required.

Comparison of Gaudin's sampling equations and Gy's sampling equation

Gy's equation is a general-purpose sampling equation,

$M = \frac{Cd_{\max}^3}{s^2}$, where M = minimum sample mass needed, d_{\max} = maximum particle size present, and s^2 = allowable sampling variance. C is a sampling constant, which is calculated for any given material based on particle shape, size distribution, degree of liberation, density, and mineralogy.

Gaudin's equation is specifically intended for use in calculating sample sizes for precious metals, and 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).

The equation is normally written as two parts: a calculation of n , the number of particles needed in the sample, and of n' , the expected number of particles per gram:

$n = 0.45 \left(\frac{x}{y^2} \right)$, where x = ore grade expressed as volume fraction, and y = allowable sampling error expressed as volume fraction.

$n' = \frac{6}{\rho_s a^3}$, where ρ_s = bulk ore density, and a = maximum particle size present.

The mass of the sample needed, S , is then $S = \frac{n}{n'} = \frac{0.45 \left(\frac{x}{y^2} \right)}{\frac{6}{\rho_s a^3}} = (0.075)(x\rho_s) \frac{a^3}{y^2}$

Comparison: We can put Gaudin's equation into the same terms as Gy's equation by using the same symbols for equivalent values in each: $a = d$, $S = M$. The value of y (a volume fraction) can be converted into σ (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 $y = \sigma/k$. If we make these

substitutions, then Gaudin's equation becomes $M = (0.075)(x\rho_s k^2) \frac{d^3}{\sigma^2}$

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

Sampling Calculations Using Hassialis' Method for Increment Sampling

Reference:

- Taggart, Handbook of Mineral Dressing, Wiley, 1945, Section 19

Use:

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. The sample cutter is used to collect a series of test increments over a particular size 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 level of certainty in the results.

Basic Equation:

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

where

- n = number of increments that will be required to reach the desired level of certainty
- h = number of standard deviations that correspond to the desired level of certainty (for example, 95% certainty corresponds to 1.96 standard deviations, see Table 2)
- σ_s = experimental standard deviation of the assays of the test increments collected by the mechanical sampler
- \bar{x} = experimental mean of the assays of the test increments collected by the mechanical sampler
- z = allowable sampling error, expressed as a fraction of the assay value (for example, if the final sample is 15% Fe and z = .04, then the allowable variation in analysis of the final sample will be (0.04)(15) = 0.60% Fe. If the final sample is 30%Fe, then the allowable variation would be (0.04)(30) = 1.2%Fe).

Example Calculation

(Calculations were done using a spreadsheet, and were carried out to more digits than are shown here. Carrying out the calculations with the values as shown will give slightly different results due to roundoff variations.)

The following 21 test increment assays were obtained from increments collected at 15 minute intervals, and took a total of 5 hours to collect. Values are weight % Fe.

10.6	10.9	10.2	10.8	9.4	9.6	10.0	10.1	9.9	9.8	9.5
9.2	9.1	9.7	8.8	10.2	10.4	10.2	9.8	9.2	9.3	

Calculate the number of increments that must be collected over a 5 hour interval so that the probability of the assay of the final composite sample being accurate to within 2% of the true assay will be 99%

Standard Deviation: $\sigma_s = 0.57234$

Mean: $\bar{x} = 9.8428$ % Iron

Allowable variation: $z = 0.02$ (that is, the analysis of the sample will be accurate to within 0.02 of the mean, and so the analysis will be $9.8428 \pm 0.1968\%$ Fe)

Number of standard deviations needed: $h = 2.576$

Table 2: Values of h corresponding to various levels of probability that a particular sample of a lot will have an assay value within the desired error limits for the analysis.

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

Number of increments needed to make a composite sample of the desired degree of accuracy:

$$n = \left(\frac{h \cdot \sigma_s}{\bar{x} \cdot z} \right)^2 = \left(\frac{(2.576)(0.57234)}{(9.8428)(0.02)} \right)^2 = 56.09128$$

Since it is not possible to collect a fraction of an increment, this should be rounded up to 57 increments per composite sample, and these increments should be collected over a 5-hour interval.