Design of Optimum Sampling Plans: 1- Dry Powders

Part I-Operator's Guide

By

Hassan El-Shall
Materials Science and Engineering Department
University of Florida

Center for Particulate & Surfactant Systems (CPaSS)
University of Florida
Gainesville, Florida
and
Columbia University
New York City, New York

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Abstract

The Theory of Sampling (TOS) provides a description of all errors involved in sampling of heterogeneous materials as well as all necessary tools for their evaluation, elimination and/or minimization. This module elaborates on—and illustrates—selected central aspects of TOS. Most importantly, practical examples are given to illustrate these theoretical aspects. Sampling representativeness, perhaps, the most important aspect of sampling practice is emphasized in the module. Sampling strategies and equipment for various materials are discussed with links to related literature and sampling equipment manufacturers

Contents

Introduction

The following is a short write up as a guide for sampling dry powders. More details are given in the module itself (Part II). Even though the e-module is based on and/ or (in many instances) extracted from excellent literature sources, these references provide comprehensive information about sampling theory and practice. The cited literature is fully acknowledged for this information and their copy rights are reserved.

Things to remember

(Summarized from Allen 2003, Davies 2009, Gy 1998-2009, Minnitt et al 2007, and Petersen et al 2005)

- Sampling goal is to obtain "representative sample".
- Be aware of the suggested sample quantities for different streams
- Familiarize yourself with Sampling errors including: Compositional, Segregation and Statistical errors
- Most importantly, when sampling process streams or time streams, the influence of periodic fluctuations should be minimized
- Mix (homogenize) well before all further sampling steps
- Use composite sampling instead of premature focus on sample mass
- Use comminution (milling) whenever necessary for reduction in grain size)
- 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 as shown in the following table.
- 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.
- 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.
- 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.

- When sampling from a conveyor belt. It is best to sample material as it cascades over the end of the conveyor belt.
- Be aware of poor and good sampling techniques for moving belt conveyors
- 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.
- If the belt can be stopped, a frame the width of the belt can be used to remove all the material within the frame

Sampling Practice of Particulate Materials

Sample definition

In order to learn about a certain quality of a population, a small portion is extracted and analyzed for the desired property. That portion if extracted correctly, it supposed to provide information that can be used to describe the population. That portion of the population is termed "sample" or "representative sample".

Sample size

In powder production as in any material processing operation, sampling is very critical to the quality control and quality assurance purposes. Such sampling could be done on the feed stream, the intermediate products, and/ or the final products. 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. Suggested sample quantities for different streams include (Davies 2009):

Process Delivery	Gross Sample	Laboratory Sample	Measurement Sample
10^n Kg	5-10 Kg	100 gm	< 1 gm

Sampling errors

When sampling one wants to make what is called a 'correct' selection, where all the constituent elements of the lot have an equal probability of being taken into the sample and the increments and the sample are not altered in any way. The correct sampling plan can avoid some the following errors (Peterson 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.

Sampling Unit Operations and Strategies

For a good sampling strategy, the following unit operations should be closely observed by the operator (Allen, 2003, Minnitt et al, 2007, and Peterson et al., 2005).

- Always perform a heterogeneity characterization of new materials. Without knowledge of lot heterogeneity it will not be possible to fix a sample mass or choose a sampling plan.
- A bulk material or powder should only be sampled when in motion.
- Most importantly, when sampling process streams or time streams, the influence of periodic fluctuations should be minimized for both long and short term. 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.
- Mix (homogenize) well before all further sampling steps as illustrated below
- Use composite sampling instead of premature focus on sample mass as shown below
- Use comminution (milling) whenever necessary for reduction in grain size) as shown below
- Only use representative mass reduction as discussed below

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 samples, 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.

Sampling free flowing stored material

Scoop sampling as shown below by Peterson et al (2005)

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. Links to these companies' web sites are listed at the end of this module.

- Spear thrust into the bulk.
- Powder falls through holes into the spear.
- Rotate handle and extract spear with enclosed sample

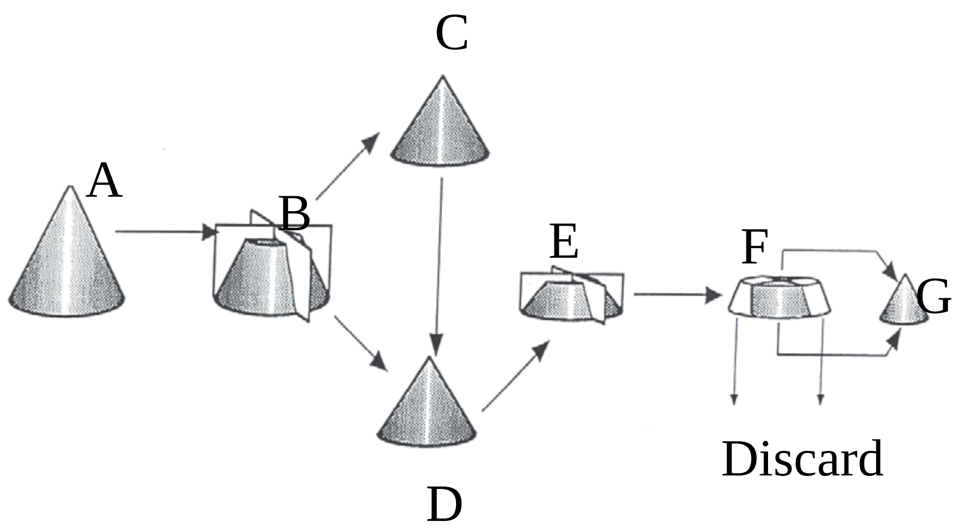
Click for Video

Courtesy of EET Corporate, www.eetcorp.com

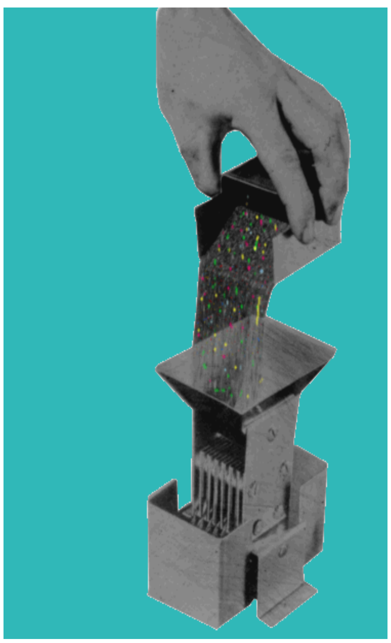
Coning and Quartering

As described by Davies, (2009) and illustrated below (sic)

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. 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.

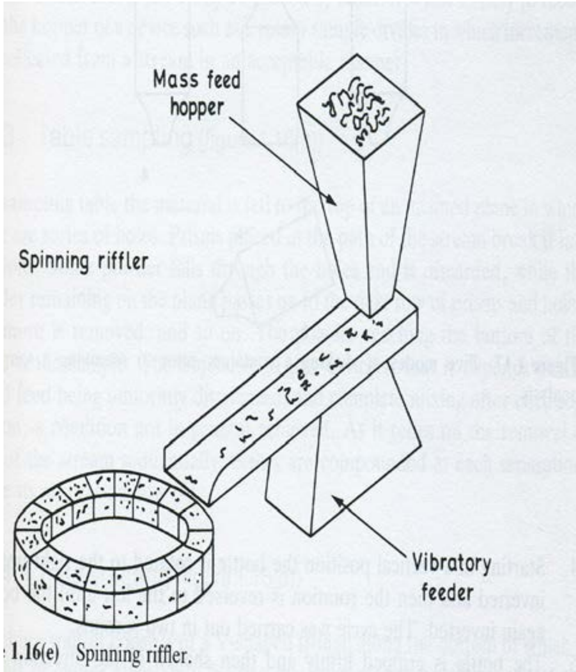


Chute Riffler



Courtesy of Sepor, www.sepor.com;

Laboratory spinning riffle



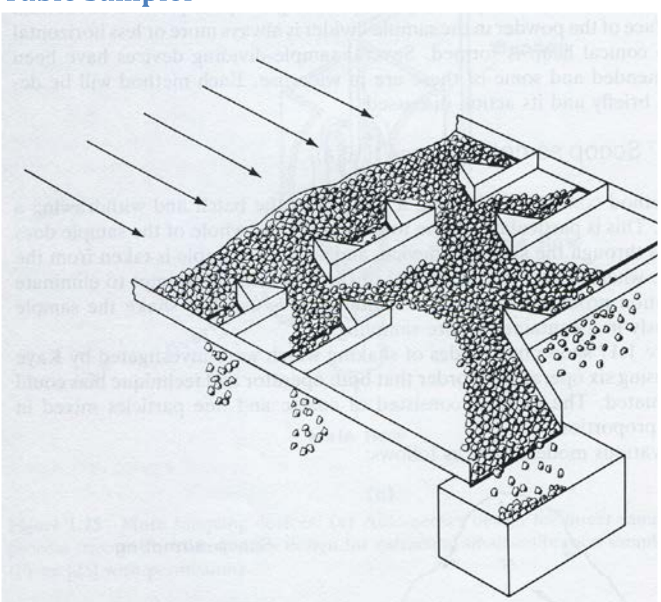
[Click to see the Micro Riffler in action]

[Click to see the Sieving Riffler in action]

Courtesy of Quantachrome Instruments, www.quantachrome.com

There are several companies producing such slitters. Among them is Quantachrome Instruments Company. There are a couple of videos on their web site showing the operation of such splitters.

Table Sampler



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

Reliability of some sampling/mass reduction techniques

Method	Estimated Maximum error (%)	Efficiency (%)
Cone & Quartering	22.7	0.013
Scoop Sampling	17.1	0.22
Table Sampling	7.0	0.13
Chute Riffling	3.4	0.56
Spinning Riffling	0.42	36.3

Sampling non-free flowing stored material

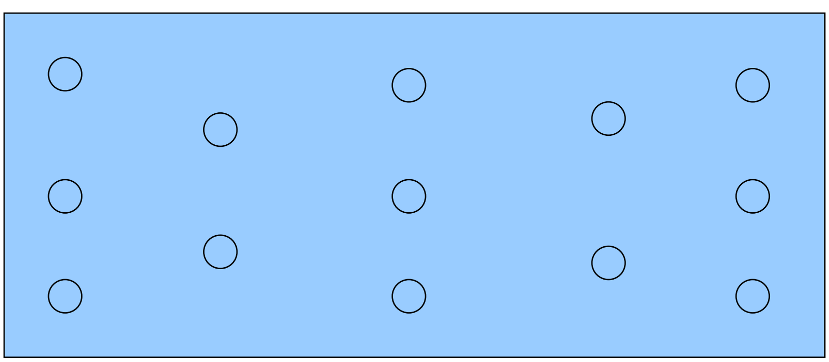
The following points should be taking into consideration before sampling takes place:

- 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.

Sampling from boxes or wagons

(Davies, 2009)

- Prepare a 3-dimensional pattern both in cross-sectional area and depth. Thief a sample from each designated location, and combine & mix them



- Subdivide the bulk sample to prepare several analytical samples. It will be statistically poor. Better to sample the flowing stream when the wagon or box is being filled.

Sampling from a hopper / silo

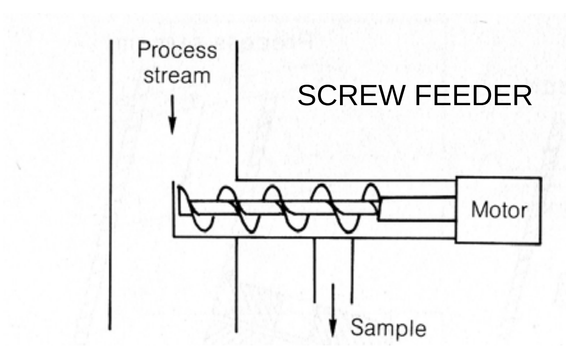
Examples of point samplers for free-flowing and non-free-flowing materials from gravity lines and hoppers are also given in the web site of Sentry Equipment Corporation, www.sentry-equip.com. A picture of this sampler is given below. A video clip of the sampler in operation is placed on the web site.

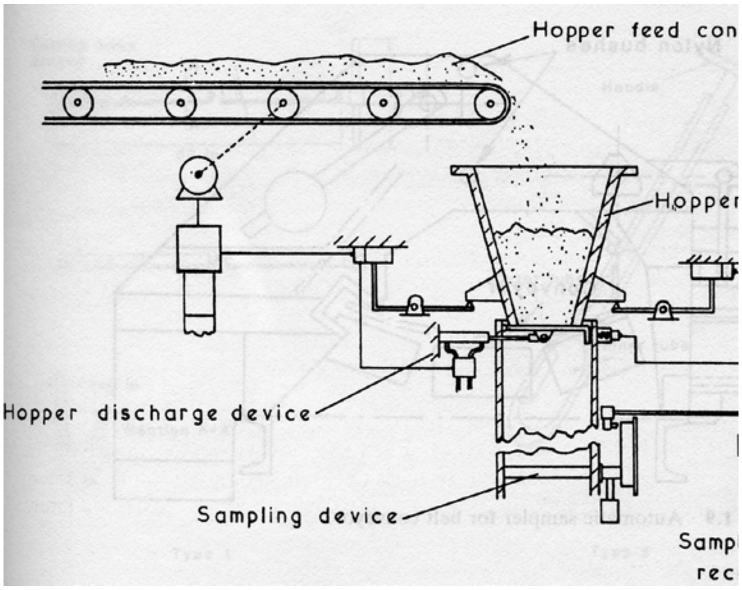
Click for Video

Click for Video

Courtesy of Sentry Equipment, www.sentry-equip.com

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





Sampling Flowing Streams

In this case, Terry Allen's Golden Rules of Sampling should be applied such 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 larges particle diameter) so that a biasaed sample deficient of coarse particles may be avoided.

An example is given below as adapted by Davies (2009) from Allen (2003)

Minimum Increment Weight

Example (sic):

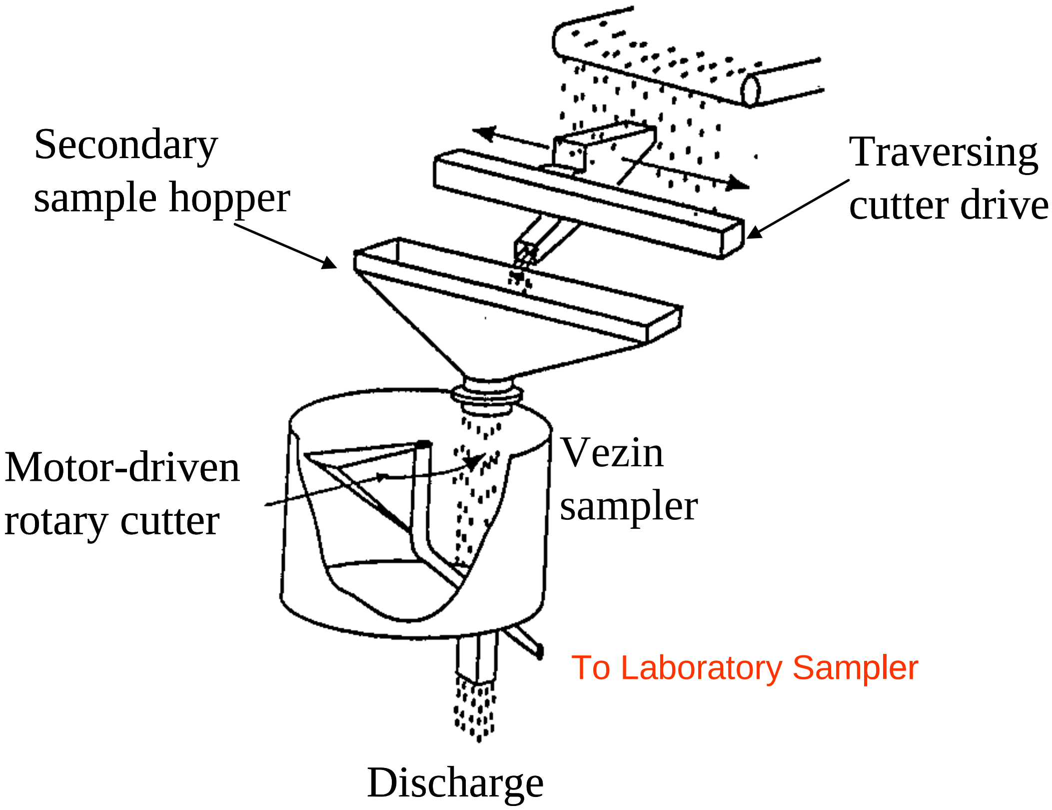
What is the minimum increment weight for a powder falling from a belt conveyor at a rate of 30 metric tons per hour if the size of the largest particle is 100µm, and the sampling cutter speed is 0.6m/s. Assume the cutter width is 20xd.

$$M = \left\{\frac{30\text{x}1000\ kg/hr}{3600 s/h}\right\} \left\{\frac{20\ \text{x}100\text{x}\ 10\ \ m}{0.60 m/s}\right\}$$

$$=28gm$$

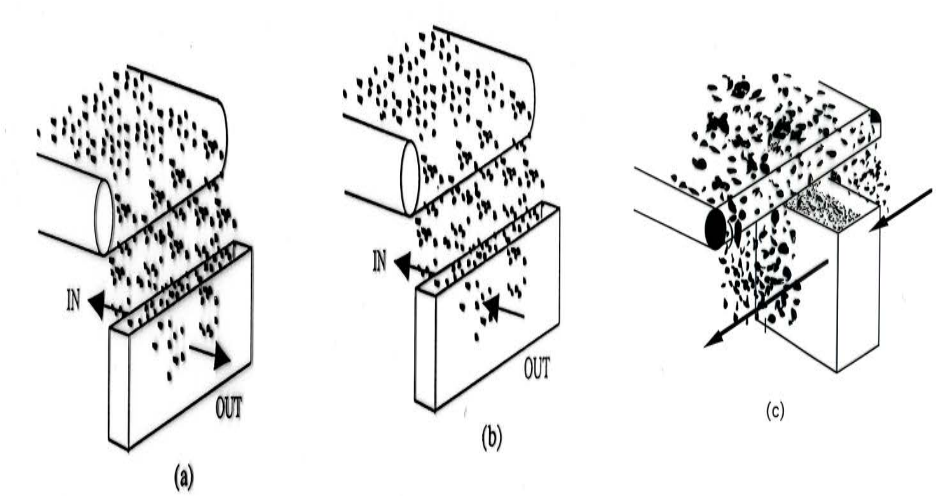
With a flowrate of 8.33 kg/s this is not a practical amount. The error will be too large. Hence a two stage sampler system is required. Sampler 1 will sample for 1second to generate 8.33 kg of powder which is fed to a hopper which will feed a second sampler at a rate of 8.33 kg/min. sampling for 1 second again yields 140 gm. therefore for a gross sample of 5kg, 35 increments will have to be taken.

Since M is proportional to d3 it rises rapidly with increasing particle size. The gross sample will then have to be reduced about 10 gm and finally by a rotary riffler to 1 gm as shown below:

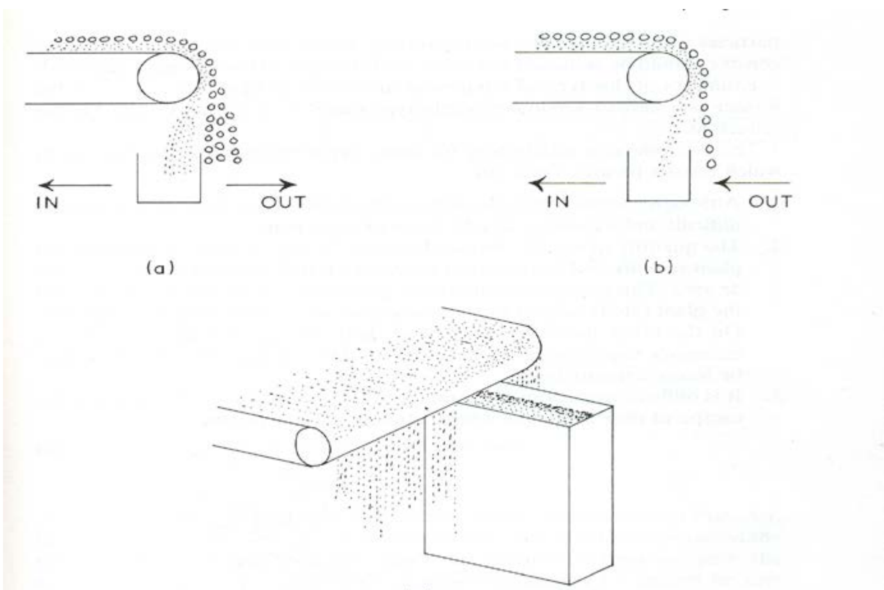


Reducing gross sample mass to laboratory sample weight

When sampling from a conveyor belt. It is best to sample material as it cascades over the end of the conveyor belt as shown below. 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 also shown to illustrate the reasons for such classification (courtesy of Reg Davies, 2009).



Sampling from falling conveyors: (a) bad sampling technique, (b) good sampling technique, (c) sampling procedure to be adopted for high mass flow rat (Courtesy of Reg Davies, 2009).



(c)

A schematic drawing explaining the reasons for good and bad sampling techniques (Courtesy of Reg Davies, 2009)

In figure (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

Click for Video

Sampling a moving belt (Courtesy of Sentry Equipment, www.sentry-equip.com)

If the belt can be stopped, a frame the width of the belt can be used to remove all the material within the frame as illustrated in the photograph below as published by Peterson et al (2005).

Sampling a stationary belt

Point samplers for sampling granules from a screw conveyor are also among the automatic sampling devices sold by various manufacturers. An example is the one sold by Sentry Equipment Corporation [www.sentry-equip.com] shown below. Their web site contains more details as well as a video of an operating sampler.

[Click for Video]

Sampling a screw conveyor

Point samplers for free-flowing products from pneumatic convey lines are also available from Sentry Equipment Corporation and other. An example of such equipment is given below and the Sentry web site contains more details as well as a video for its application.

[Click for Video]

Sampling pneumatic convey lines

Courtesy of Sentry Equipment, www.sentry-equip.com

This sampler is designed to eliminate the degradation of product, it takes a point sample of free-flowing material from dilute and dense phase pneumatic convey lines.

In addition, there are other automatic samplers that can be used with flowing material in pipes, chutes, or hoppers such as the one shown below.

[Click for Video]

Sampling flowing material in pipes, chutes, or hoppers

Courtesy of Sentry Equipment, www.sentry-equip.com

Detailed Information about Sampling Theory and Practice

Part II of this module contains more details about the theory and practice of sampling many industries. For any details please click on any connection (blue colored words / expressions).

References

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Minnitt, R. C. A., Rice, P. M., & Spangenberg, C. (2007). Part 1: Understanding the components of the fundamental sampling error: A key to good sampling practice. *Journal of the South African Institute of Mining and Metallurgy*, 107(8), 505-511.

Petersen, L., Minkkinen, P., & Esbensen, K. H. (2005). Representative sampling for reliable data analysis: Theory of sampling. *Chemometrics and Intelligent Laboratory Systems*, 77(1-2), 261-277.

Links to Literature

The Accuracy of Systematic Sampling from Conveyor Belts:

http://www.jstor.org/stable/pdfplus/2985832.pdf

Sampling equipment:

http://www.powderandbulk.com/analyzers/samplers.htm

Belt conveying equipment:

http://www.powderandbulk.com/conveying/belt.htm

http://www.directindustry.com/industrial-manufacturer/bulk-and-powder-handling-and-equipment-152/conveyor-belt-61357.html

http://www.ktron.com/products/pneumatic_conveyors/index.cfm

http://www.flexicon.com/us/products/PneumaticConveyingSystems/index.asp http://www.vac-u-max.com/pneumatic.html

Acknowledgements, Copyright statement, and disclaimer

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Pictures and videos are courtesy of following Companies. However, the author does not endorse specific equipment from any company. They are included to give the users examples of the available sampling equipment and their suppliers.

SENTRY EQUIPMENT CORP
966 Blue Ribbon Circle North
PO Box 127
Oconomowoc, WI 53066 USA
Phone: 262-567-7256
Fax: 262-567-4523
Website:
www.sentry-equip.com;

Intersystems
9575 North 109th Avenue
Omaha, NE 68142
USA
www.intersystems.com;

QUANTACHROME INSTRUMENTS
1900 Corporate Drive
Boynton Beach, Florida 33426 USA
www.quantachrome.com;

Sepor
718 N. Fries Ave.
Wilmington, CA 90744
www.sepor.com

Samplersdirect
www.samplersdirect.com;

EET Corporation
3106 Roane State Highway
Harriman, TN 37748
www.eetcorp.com

Sampling Systems Ltd.
2 Forge Mills Park,
Station Road,
Coleshill,
Warwickshire,
B46 1JH, UK
http://www.sampling.co.uk/