

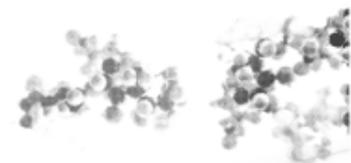


Sampling for Particle Size Analysis

Mark Bumiller

mark.bumiller@horiba.com

www.horiba.com/particle



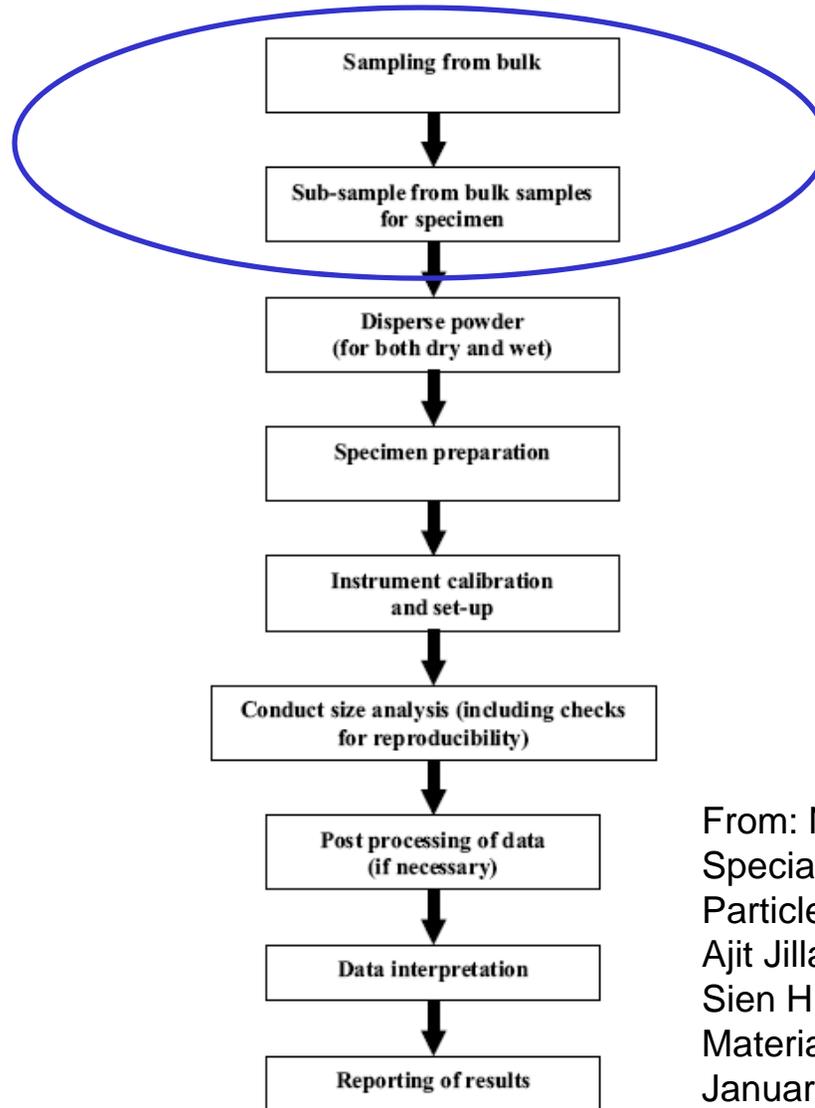
Explore the future

Automotive Test Systems | Process & Environmental | Medical | Semiconductor | Scientific

HORIBA

© 2012 HORIBA, Ltd. All rights reserved.

Particle Sizing Workflow

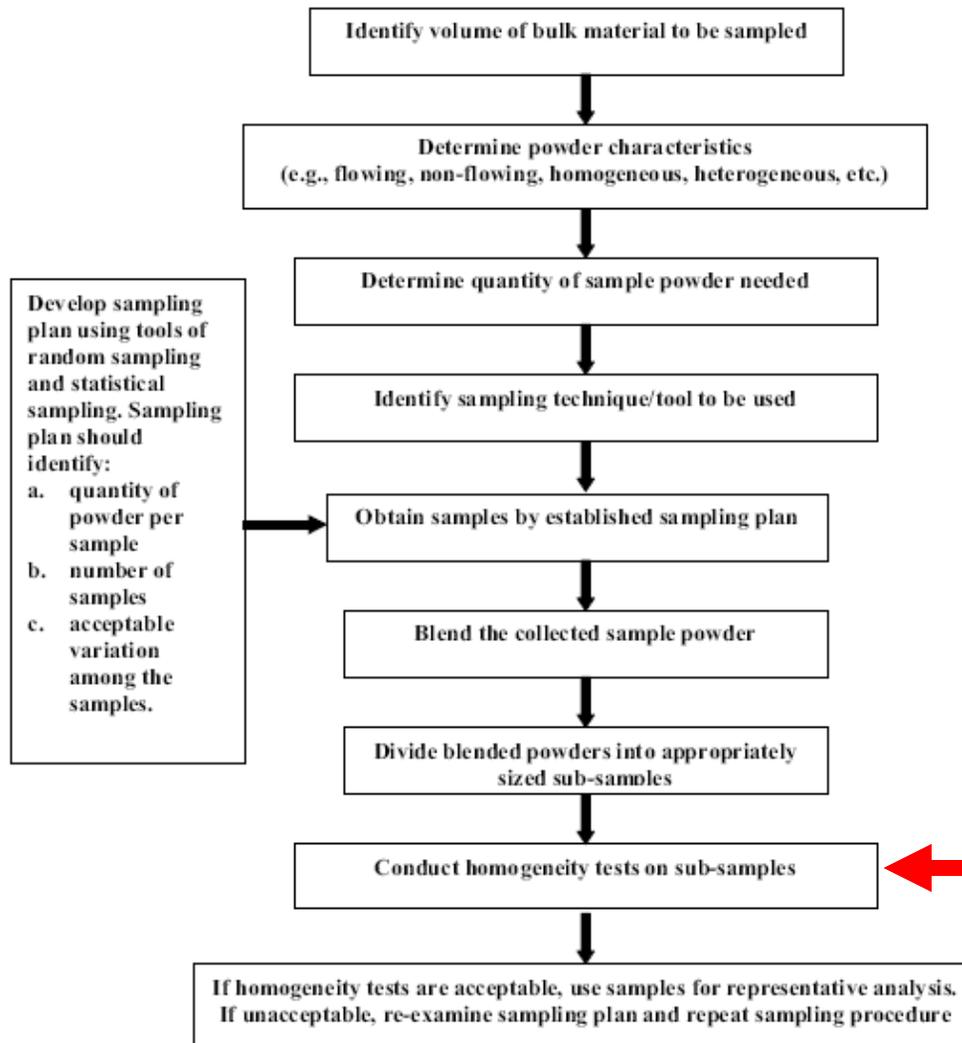


Today's subject area

If we don't get this right, why bother with the other steps?

From: NIST Recommended Practice Guide
 Special Publication 960-1
 Particle Size Characterization
 Ajit Jillavenkatesa, Stanley J. Dapkunas, Lin-Sien H. Lum
 Materials Science and Engineering Laboratory
 January 2001

Sampling Workflow



Excellent strategy
not sure how often followed

← Must do this

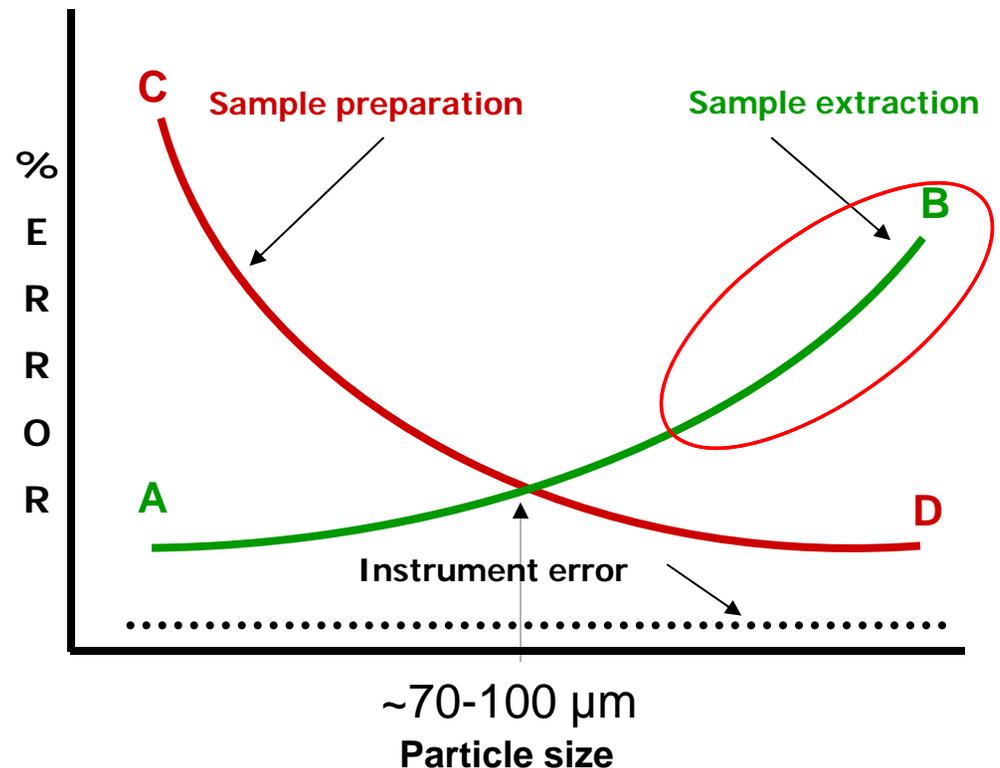
Measurement Error Sources

Small particles

- Smaller extraction errors (A)
- Larger sample prep errors (C)

Large particles

- Larger extraction errors (B)
- Smaller sample prep errors (D)



Instrument error relatively small

May increase w/decreasing particle size (less so w/LA-950)

Errors vs. Errors

■ Accuracy

- Is the size reported same as referee technique?
- RI, dispersion, method

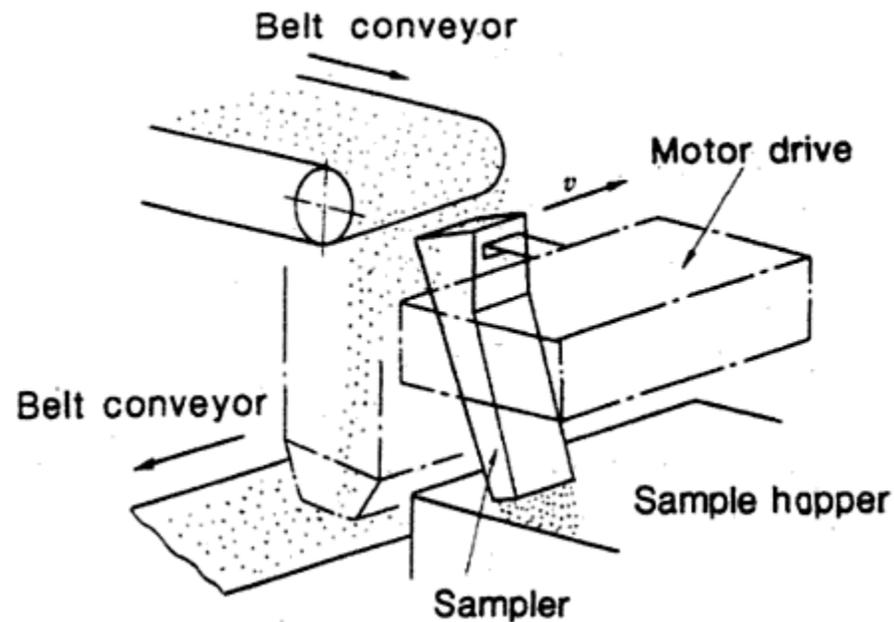
■ Repeatability

- As sample recirculates, get same result?
- Is sample stable

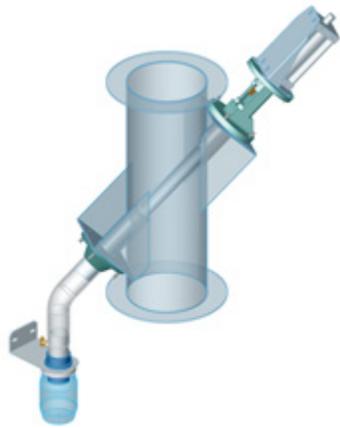
■ Reproducibility

- Sample, disperse, measure, clean, repeat
- Good sampling, dispersion, stability

Sampling from Flowing Powder



Sampling from Flowing Powder



Cross-cut sampling: $w = \frac{L}{v} \frac{W}{L} b = \frac{Wb}{v}$

v = sampling speed (m/s)

W = sample mass

L = width of powder stream

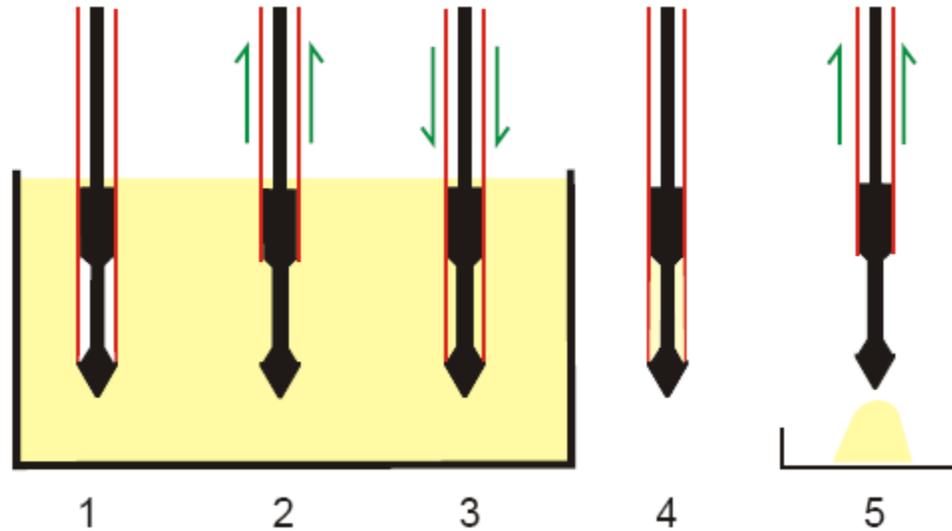
b = sample cutter width*

Cross-cut sampler

*Masuda, H, Powder Technology Handbook, CRC Press, p 771

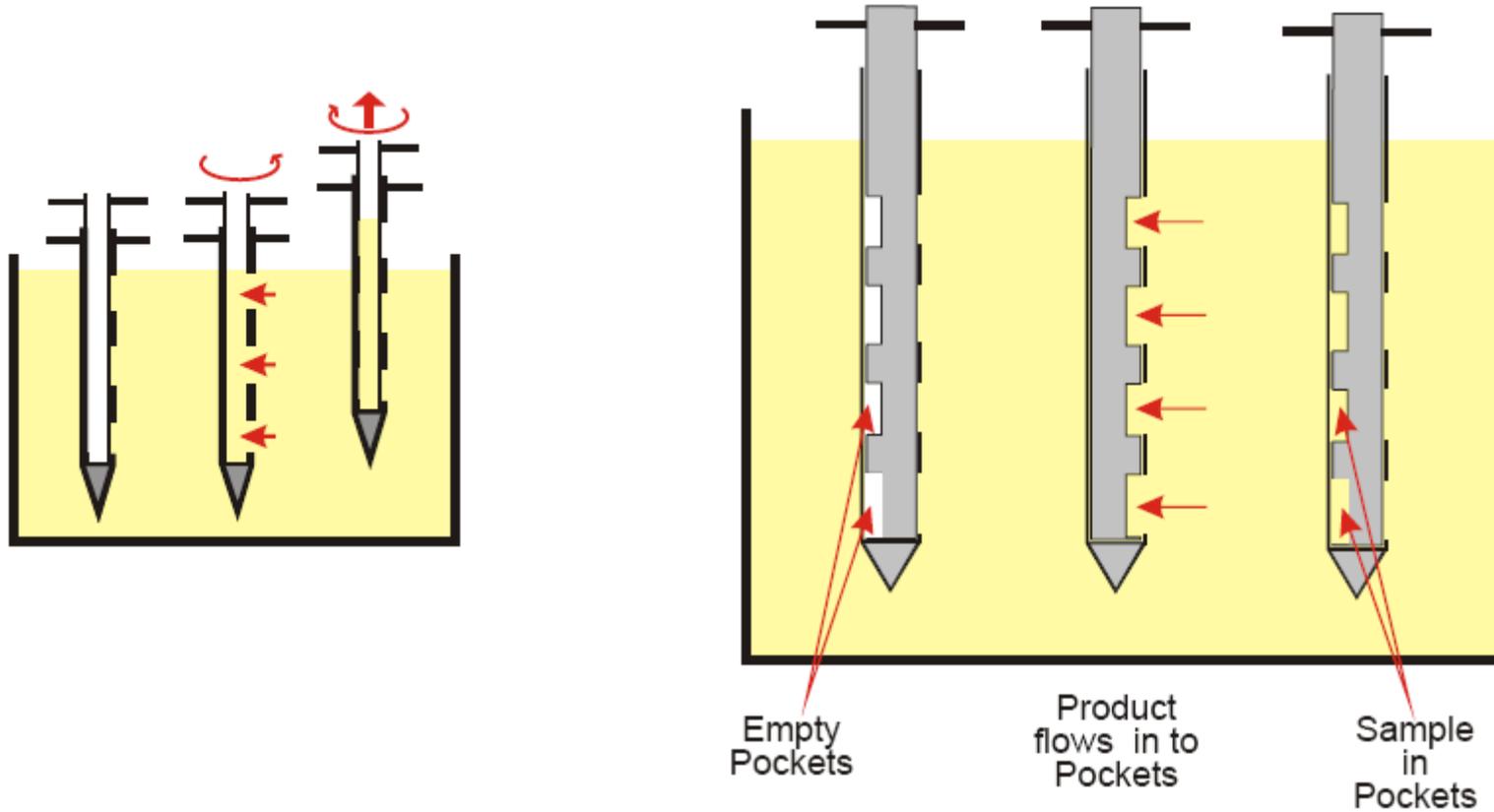
Sampling from Drums

Powder Thief



www.samplingsystems.com

Sampling from Drums



www.samplingsystems.com

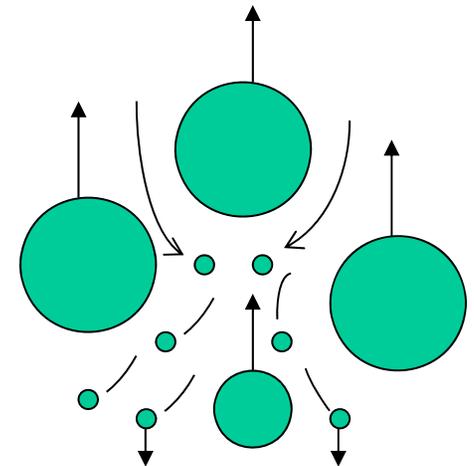
Sub-sampling for Measurement

bulk or process stream	gross sample	laboratory sample	test sample	measurement sample
(10 ⁴ kg)	(> kg)	(< kg)	(g)	(mg)

- Not all of sample brought to lab is analyzed
- Must sub-divide sample
- How to introduce representative sample into instrument
- Amount measured varies wet vs. Dry, choice of sample presentation unit

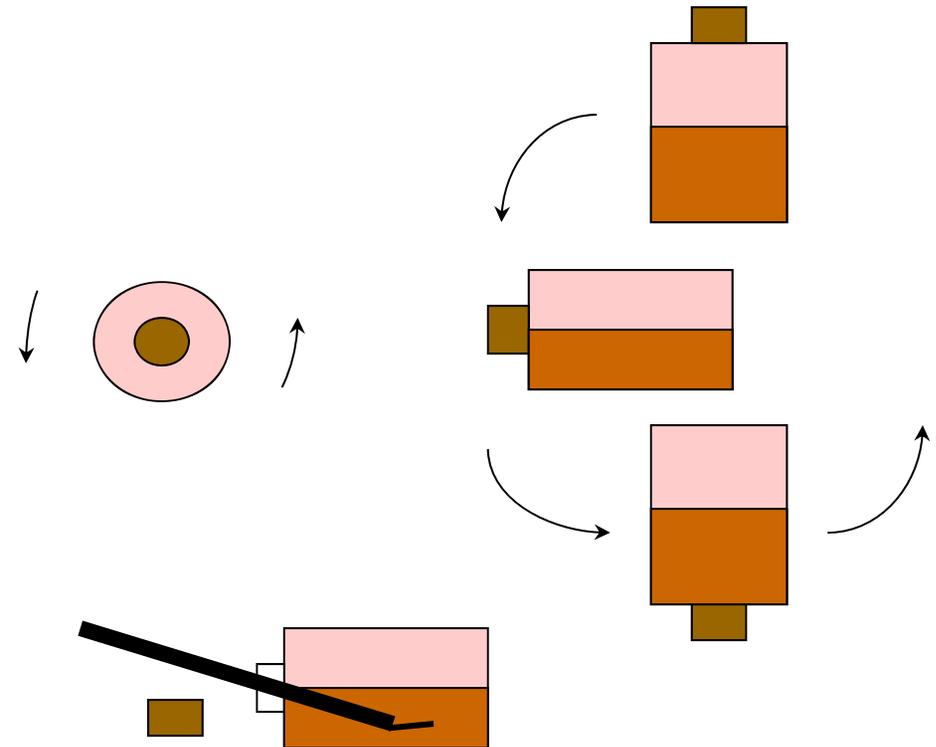
Technique: Grab Sampling

- Place spatula into powder, extract small amount for analysis
 - Easy, most used method
 - Maybe worse method
- May be acceptable for narrow distributions
- Problem: **segregation** of larger particles w/wide distribution
 - Large particles percolate upward
 - Small particles gravitate downward



Grab Sampling from Bottle

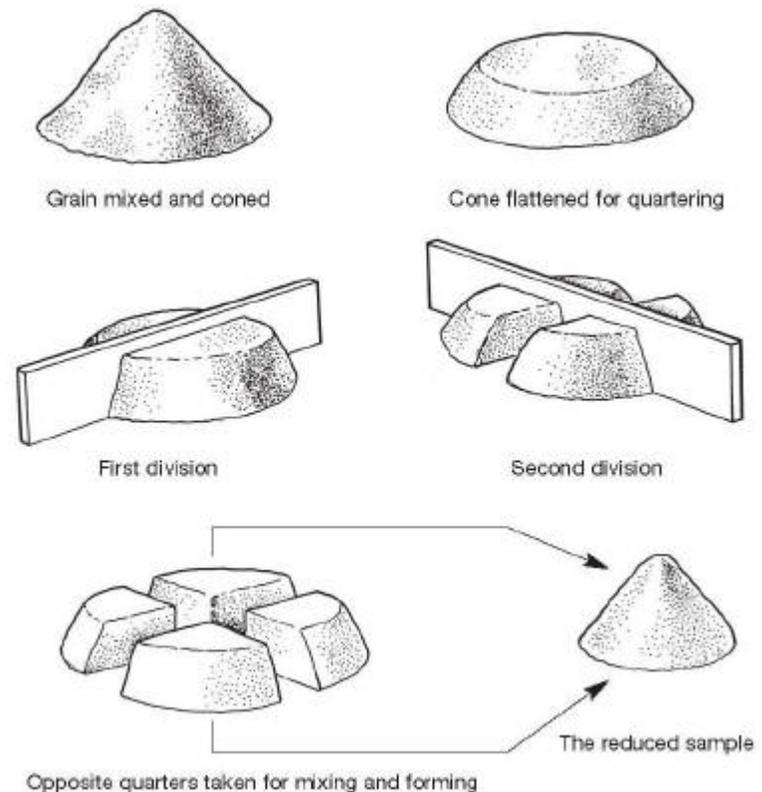
When a powder is stored in a container, it can be mixed by rolling and tumbling the container. The container should not be more than half to two-thirds full. It is important to perform this action before “grabbing” a sample with a spatula.



Then pull sample with a spatula.....

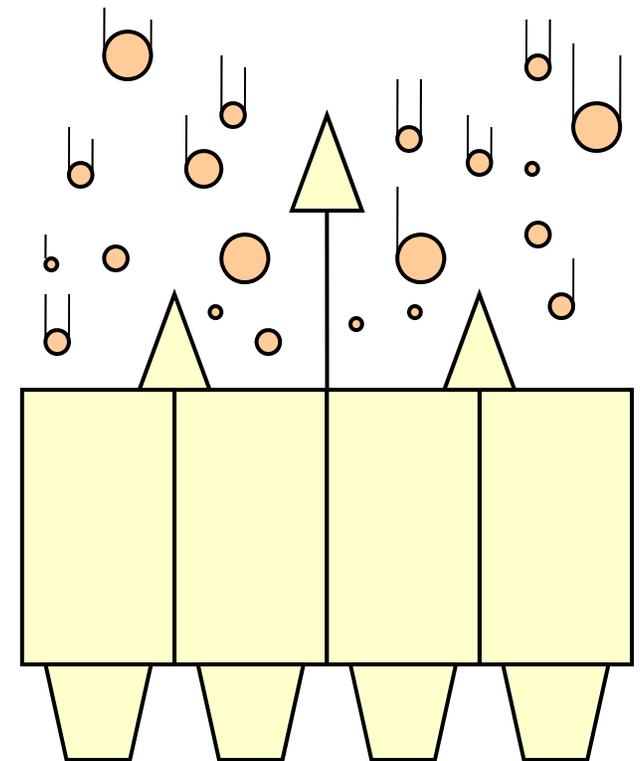
Technique: Coning & Quartering

- Pile of powder is divided into 4 sections.
- Two diagonal sections are discarded, and two are retained and mixed together.
- Mixture is again divided into 4 sections, and two diagonal sections are mixed.
- Process is repeated until remaining sample is correct amount for analysis.
- Can be carried out with very small sample amount or very large samples.



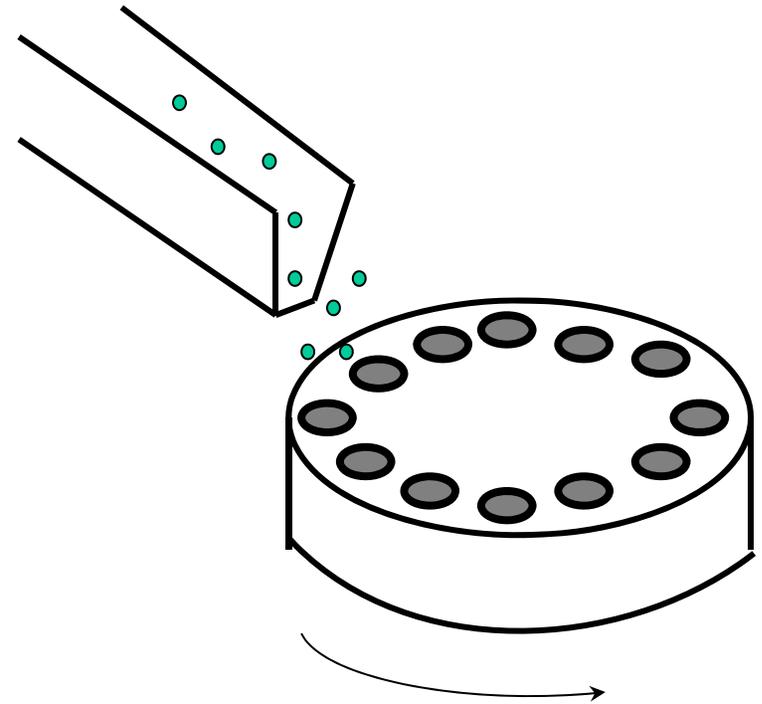
Technique: Chute Riffling

Chute splitting allows sample to vibrate down a chute to vanes which separate the mass into two portions. Each portion moves further where they each are divided into two parts, now giving four parts. This may be continued until usually 8 or 16 portions are obtained.



Technique: Rotary Riffing

The best method of representative splitting of powders is the ROTARY RIFFLER. The complete sample to be split is directed down a chute into open containers. Each container will receive a sample which is representative of the original bulk material because the distribution of material is averaged over time. The complete amount of the original bulk sample must be consumed.



These splitters are commercially available from companies that market various types of sample splitters.

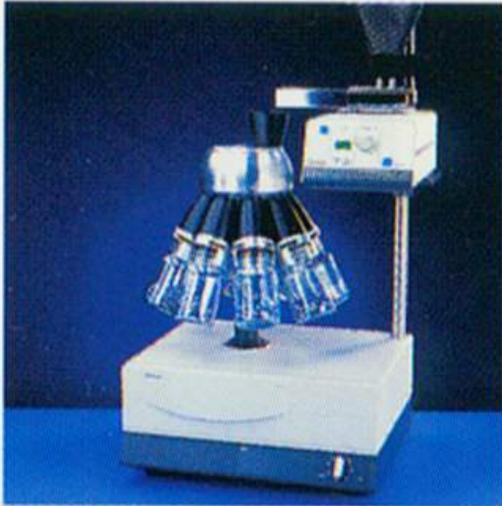
See: www.retsch.com

www.quantachrome.com

www.microscal.com



Sample Dividers



Laboratory sample divider PT 100

- *for pourable powders and granules*
- *feed size up to 10 mm*
- *division into 6, 8 or 10 representative samples*



Laboratory rotary tube sample divider PKZ 1000

- *for pourable powders and granules*
- *feed size up to 10 mm*
- *various division ratios*

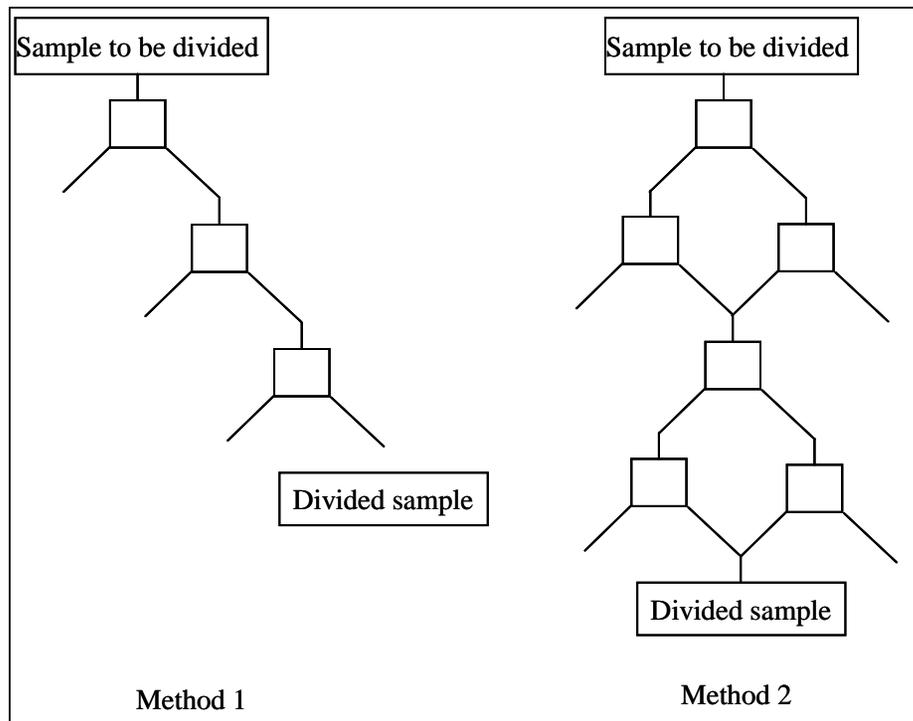


Sample splitter RT

- *for bulk materials*
- *feed size up to max. 50 mm*
- *division into 2 samples*

Sample Splitting

Particle max. size (mm)	Opening width (mm)
20-16	50
16-10	30
10-5	20
5-2,5	10
below 2,50	6



Measure the entire finally divided sample

Practical Concerns

■ Cleanliness

- Must clean splitter or riffler after use
- Main reason spinning riffler not used???
- Cross contamination must be avoided
- But not really so hard

■ Speed

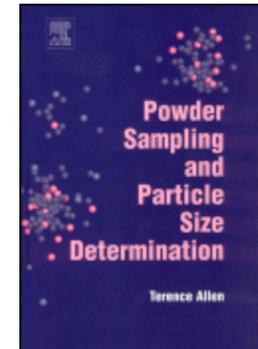
- Don't be in a hurry when using spinning riffler

■ Avoid exposure to dust

Sampling Technique Error Levels

Standard Deviation (σ) in % Sugar-Sand Mixture

SCOOP SAMPLING	6.31
TABLE SAMPLING	2.11
CHUTE RIFFLER	1.10
SPINNING RIFFLER	0.27



Density of sand and sugar respectively 2.65 and 1.64 g/ml

Reference: Allen, T. and Khan, A.A. (1934), Chem Eng, 238, CE 108-112

Method	Relative Standard Deviation (%)
Cone & Quartering	6.81
Scoop Sampling	5.14
Table Sampling	2.09
Chute Riffling	1.01
Spin Riffling	0.125

Sampler Error w&w/o Riffler

LA-950 WET without Riffler			
Sample Name	D(v, 0.1)	D(v, 0.5)	D(v, 0.9)
Run #1	3.080	38.018	203.416
Run #2	3.091	36.672	195.089
Run #3	2.915	35.762	200.610
Mean	3.029	36.817	199.705
Std. Dev.	0.099	1.135	4.237
COV (%)	3.255	3.083	2.121



Sample riffled
 All of sub sample
 dispersed and
 measured as a
 suspension

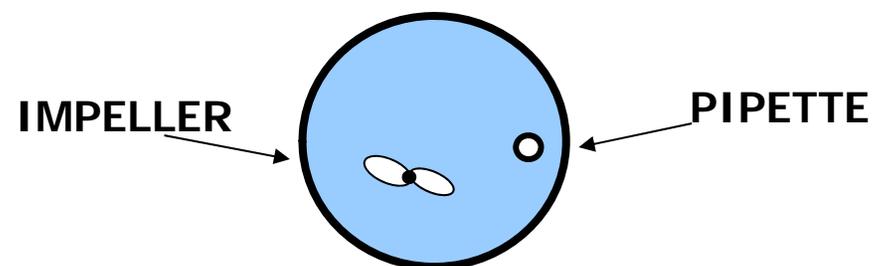
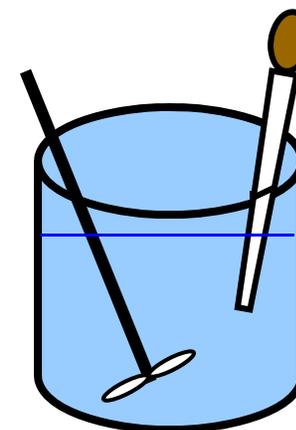
LA-950 WET Analysis with Riffler			
Sample Name	D(v, 0.1)	D(v, 0.5)	D(v, 0.9)
Run #1	2.796	36.848	202.660
Run #2	2.828	37.260	205.074
Run #3	2.895	35.998	200.843
Mean	2.840	36.702	202.859
Std. Dev.	0.051	0.644	2.123
COV (%)	1.779	1.753	1.046

Technique: Sampling from Beaker

Liquid should be in motion vertically and horizontally to insure good mixing.

Pipette should be about one-third of the way from the bottom when extracting sample.

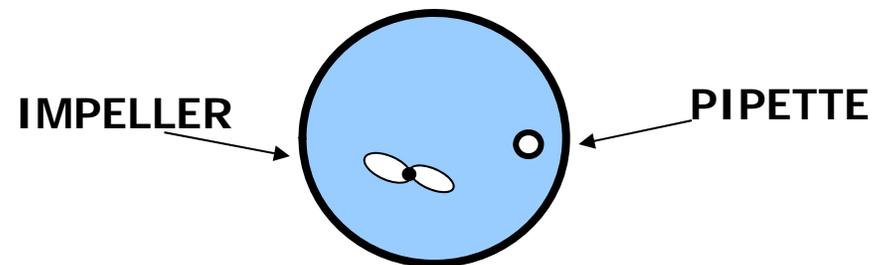
Alternative: When mixing powders into a slurry: make paste, pipette from paste



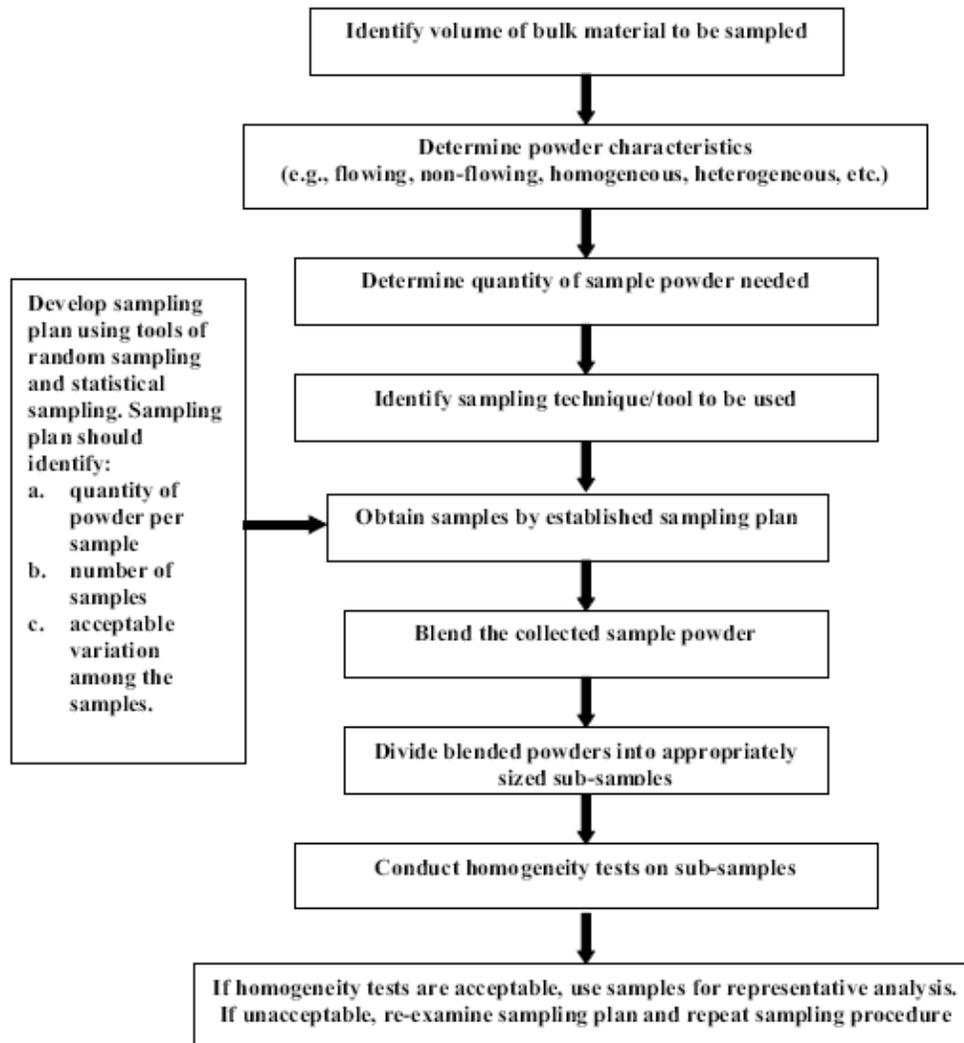
Sampler Error w&w/o Mixing

LA-950 WET Analysis without Mixing			
Sample Name	D(v, 0.1)	D(v, 0.5)	D(v, 0.9)
Run #1	8.365	43.867	92.267
Run #2	12.596	61.324	113.839
Run #3	14.722	76.164	156.757
Mean	13.659	68.744	135.298
Std. Dev.	1.503	10.493	30.348
COV (%)	11.007	15.264	22.430

LA-950 WET Analysis with Mixing			
Sample Name	D(v, 0.1)	D(v, 0.5)	D(v, 0.9)
Run #1	11.476	66.064	160.472
Run #2	12.296	65.121	152.838
Run #3	12.722	66.164	156.757
Mean	12.509	65.642	154.798
Std. Dev.	0.301	0.737	2.771
COV (%)	2.409	1.123	1.790



Summary



From: NIST Recommended Practice Guide
 Special Publication 960-1
 Particle Size Characterization
 A. Jilla et.al.
 Materials Science and Engineering Laboratory
 January 2001

Our own

..... WHITE PAPER
 Sampling for Particle Size Analysis

On www.horiba.com/particle

Resources: www.horiba.com/particle

HORIBA Scientific

Chisissez un pays ou une zone

Automotive Test Systems | Process & Environmental | Medical | Semiconductor | **Scientific** | All Segment Product Browser

Products | Markets & Industries | Service & Support | News & Events | Newsletter | About Us | Employment | Feedback

Particle Characterization

Home → Scientific → Products → Particle Characterization

Particle Characterization

HORIBA designs, manufactures, and supplies state of the art particle characterization instruments.

Every instrument across the five business segments must meet stringent requirements before the HORIBA name is attached. The Particle Characterization group of analyzers has incorporated this principle into each new design since entering the business in 1979. Relentless innovation united with high performance to attain the ultimate goal: a new standard in usability.

Particle Characterization Products

HORIBA offers instruments for particle size, particle shape, zeta potential, and surface area analysis. Measurable particle size range is from 1 nanometer to 30 millimeters, at concentrations ranging from 1 ppm to 50 vol% with shape determination available starting at 1 micrometer. A range of analytical techniques are employed including laser diffraction (Mie Theory), dynamic light scattering, acoustic and electroacoustic spectroscopy, and dynamic and static image analysis. (measuring both particle size and shape information).

HORIBA's advanced designs and powerful software, combined with flexible sample handling systems are available to meet every analysis need. These instruments can incorporate small volume pumping systems for precious materials, high throughput automation, dry powder dispersers and temperature controlled flow systems in order to provide the user with the best possible solution with none of the trade-offs that might otherwise be necessary.

Particle Size

- Laser diffraction
 - LA-950V2
 - LA-300
- Dynamic light scattering
 - SZ-100

Request Information

Quick Request

Particle Size Essentials

Newsletter

Download Center

Member login

Please enter your e-mail address and password in order to login on to www.horiba.com. We may ask you later to answer additional questions if you are the first time to this area.

E-Mail Address:

Password:

Login

← Receive news of updates

← View application notes, webinars, etc.

Thank-you