# Sampling for Particle Size Analysis

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# Particle Sizing Workflow

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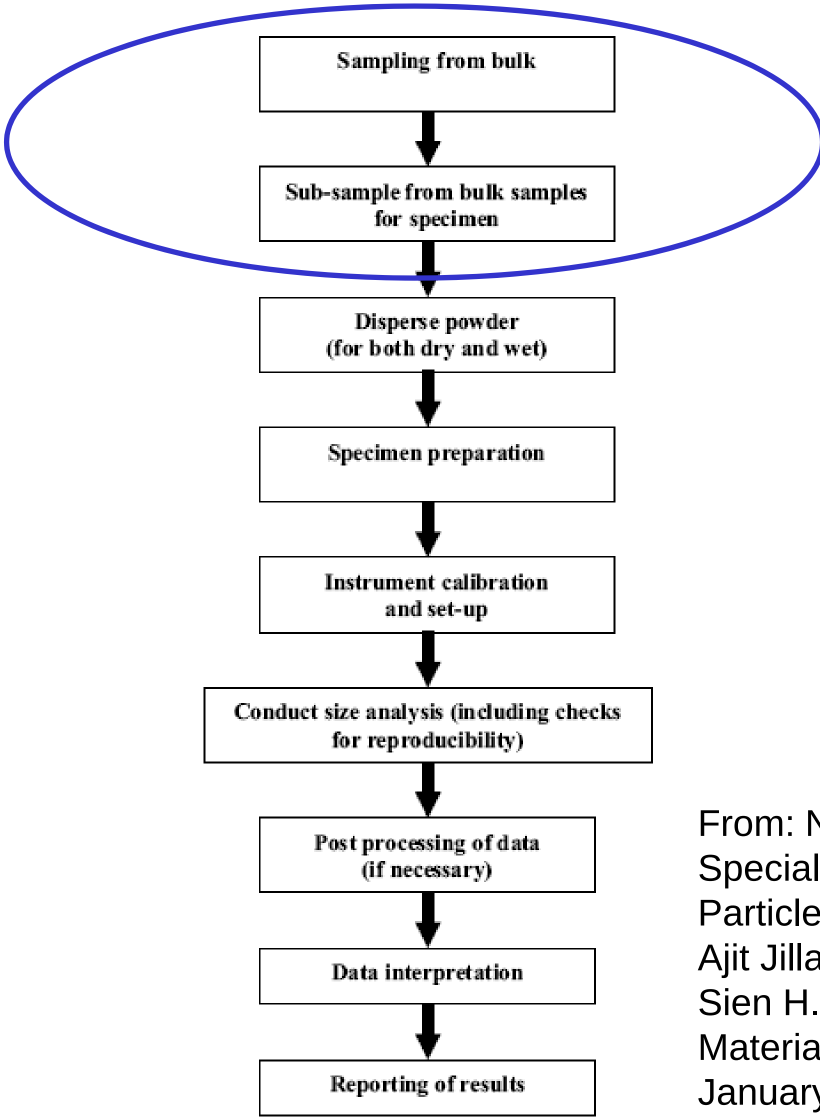
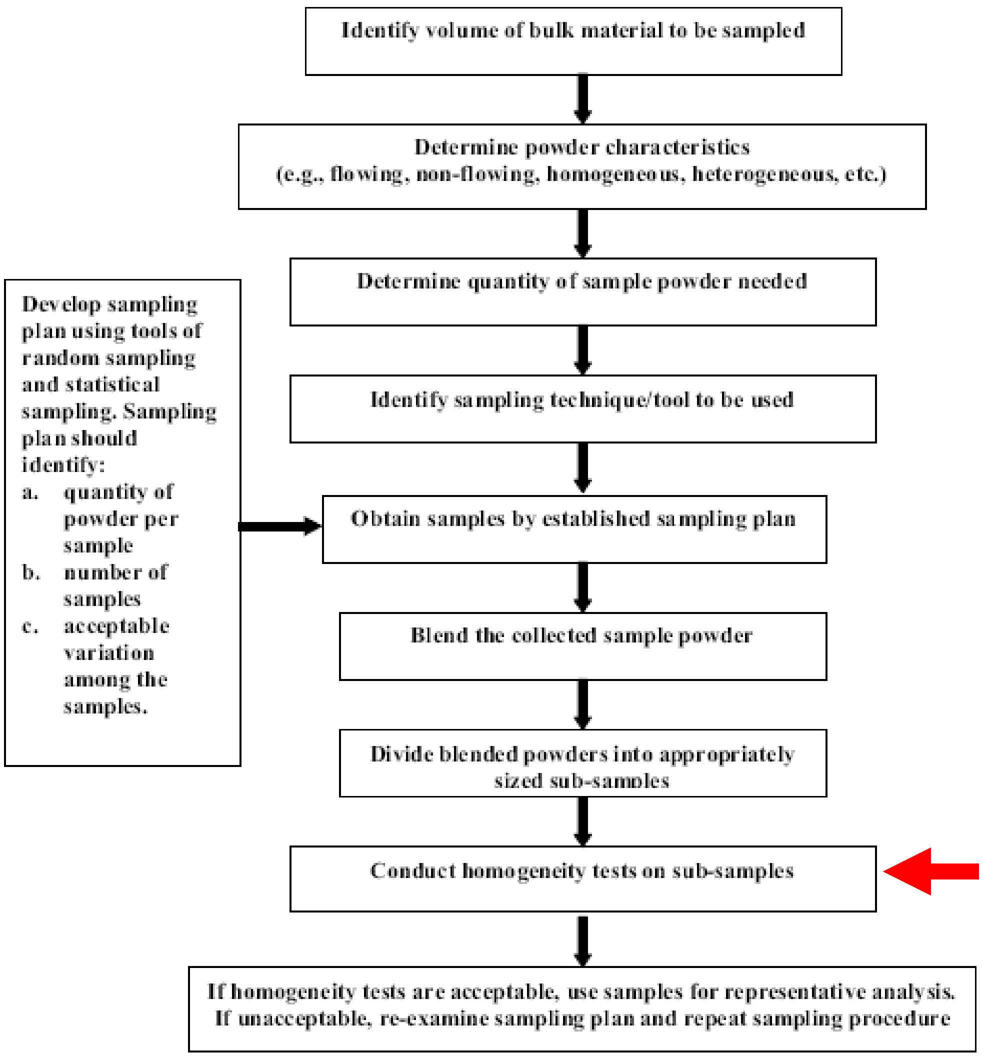



Today’s subject area

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

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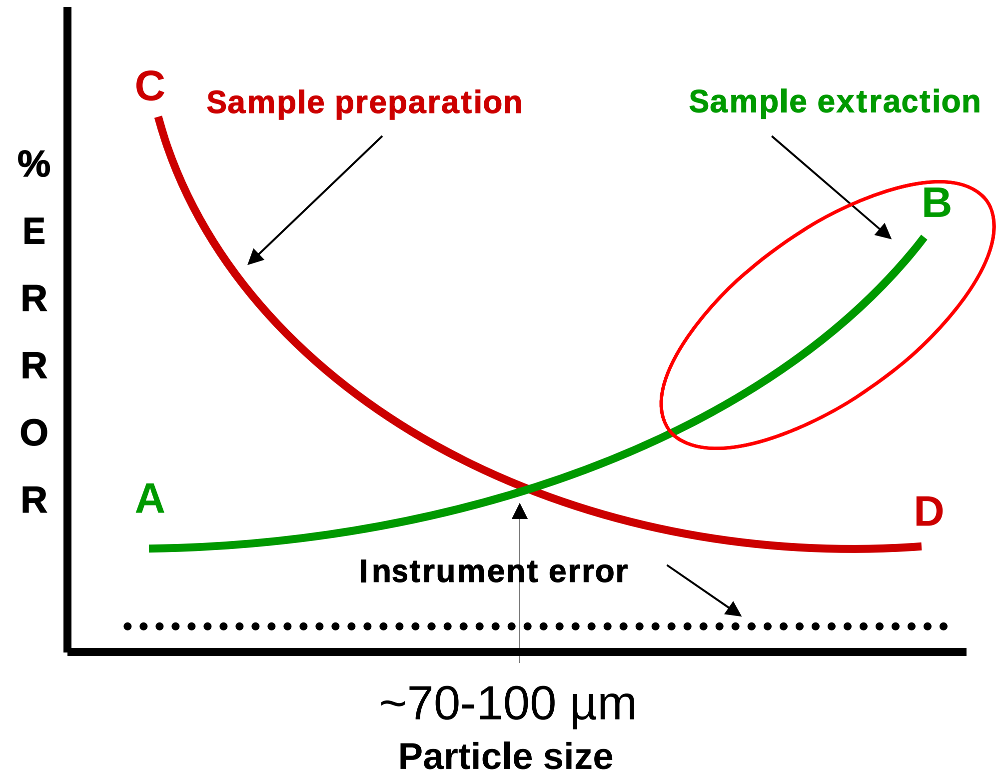
## Small particles

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

## Large particles

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



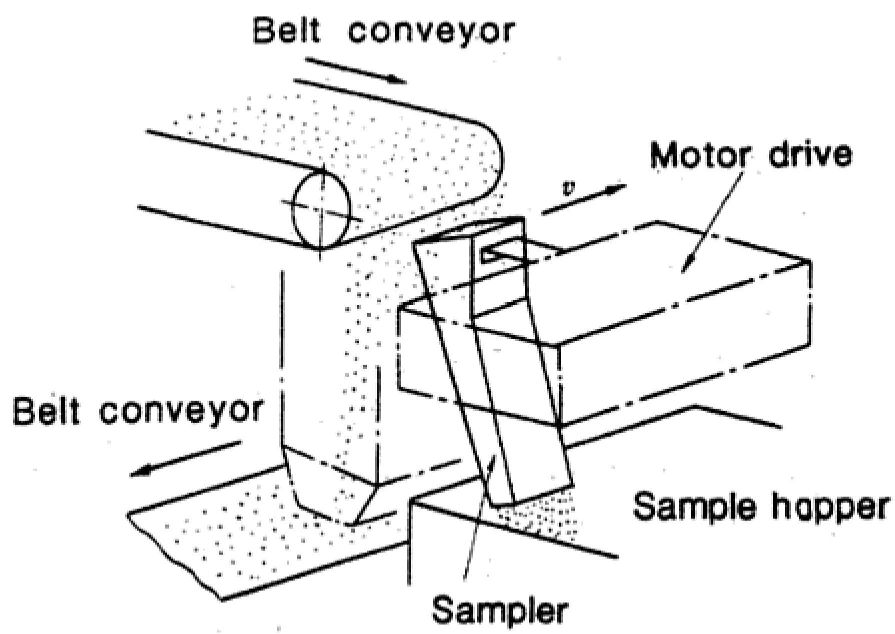

Instrument error relatively small

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

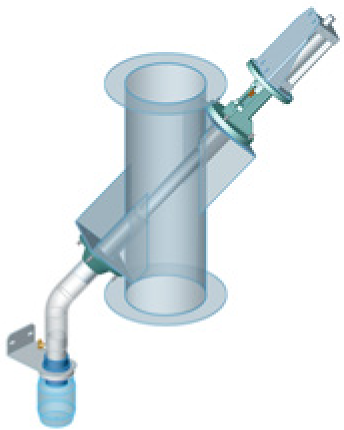
# Errors vs. Errors

- 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 sampler

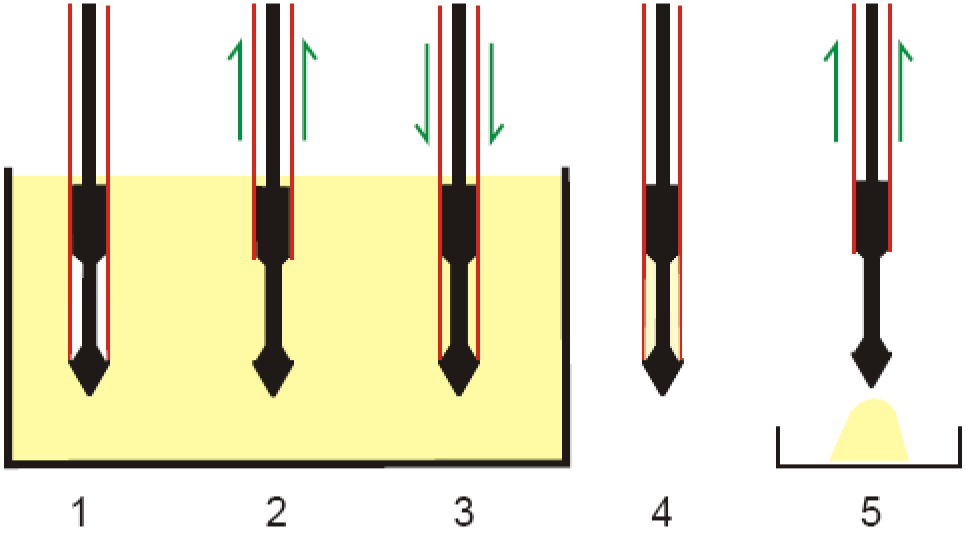
Cross-cut sampling: $w = \frac{L}{\nu}\frac{W}{L} b = \frac{Wb}{\nu}$

$\nu$ = sampling speed (m/s)
W = sample mass
L = width of powder stream
b = sample cutter width*

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

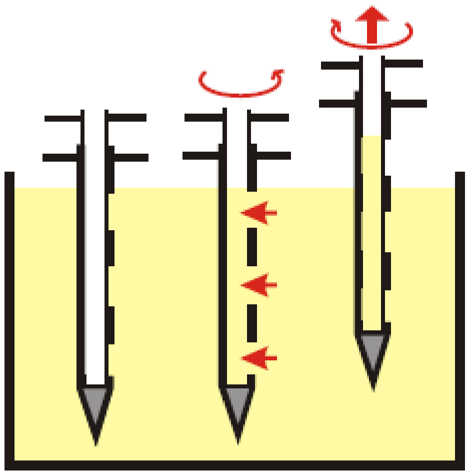
# Sampling from Drums

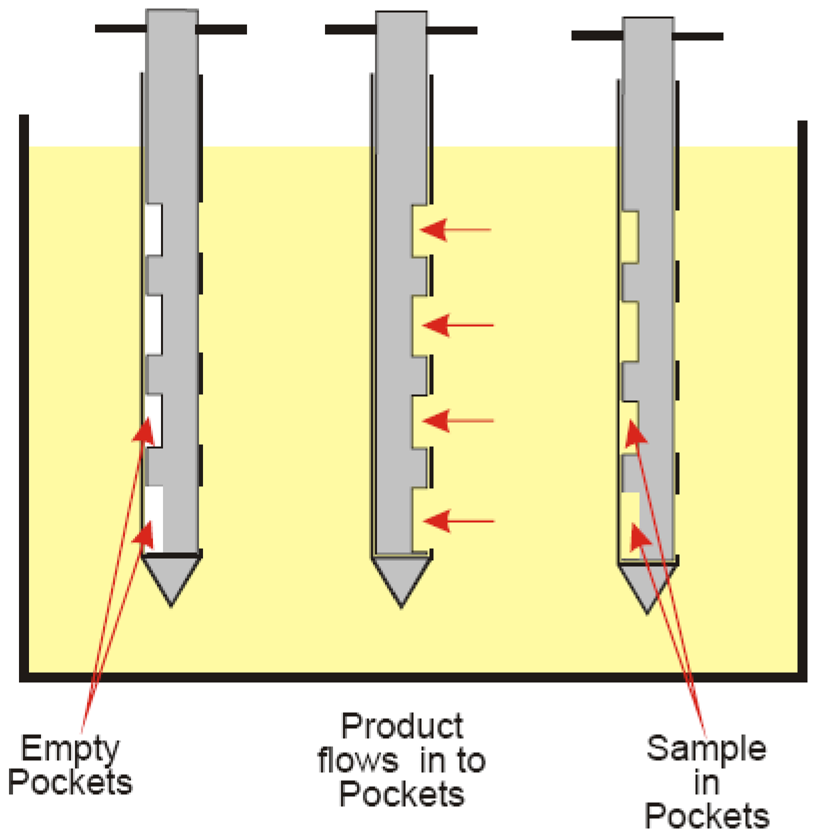
Powder Thief


www.samplingsystems.com

# Sampling from Drums






www.samplingsystems.com

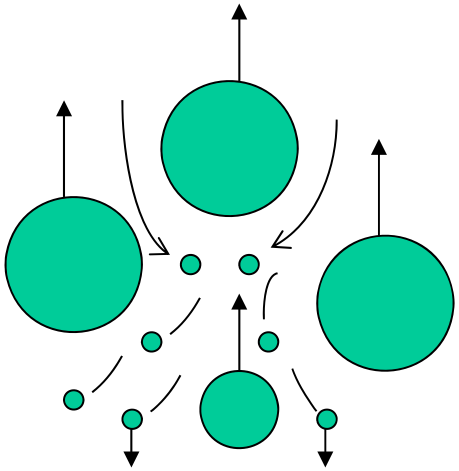
# Sub-sampling for Measurement

| bulk or process stream | gross sample | laboratory sample | test sample | measurement sample |
|---|---|---|---|---|
| ($10^n$ 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

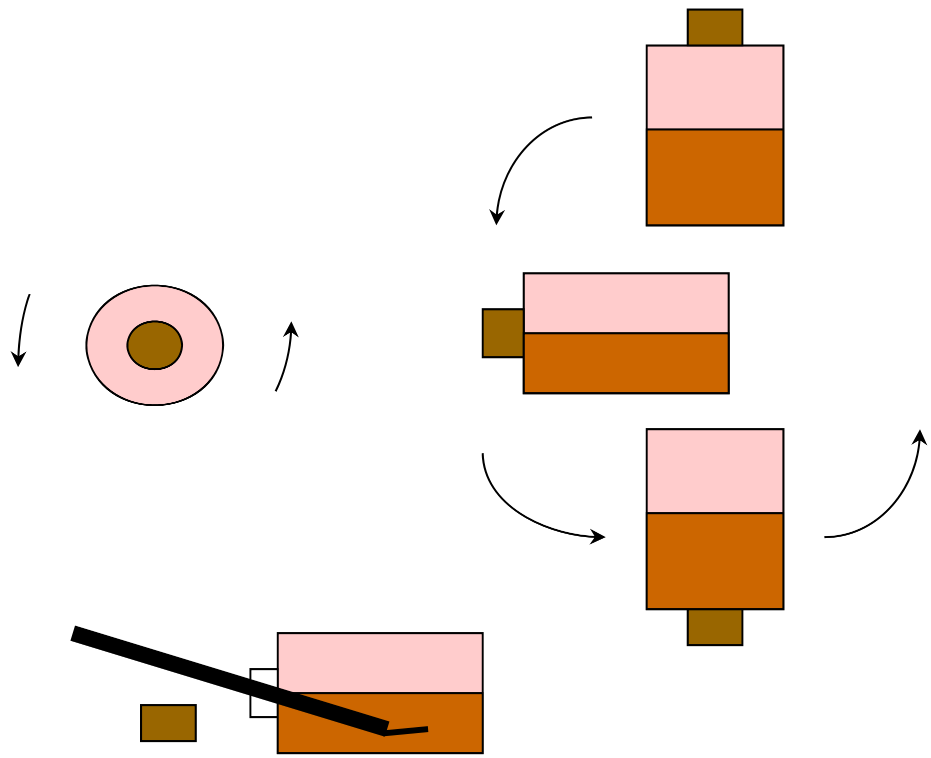
# Technique: Grab Sampling

- 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

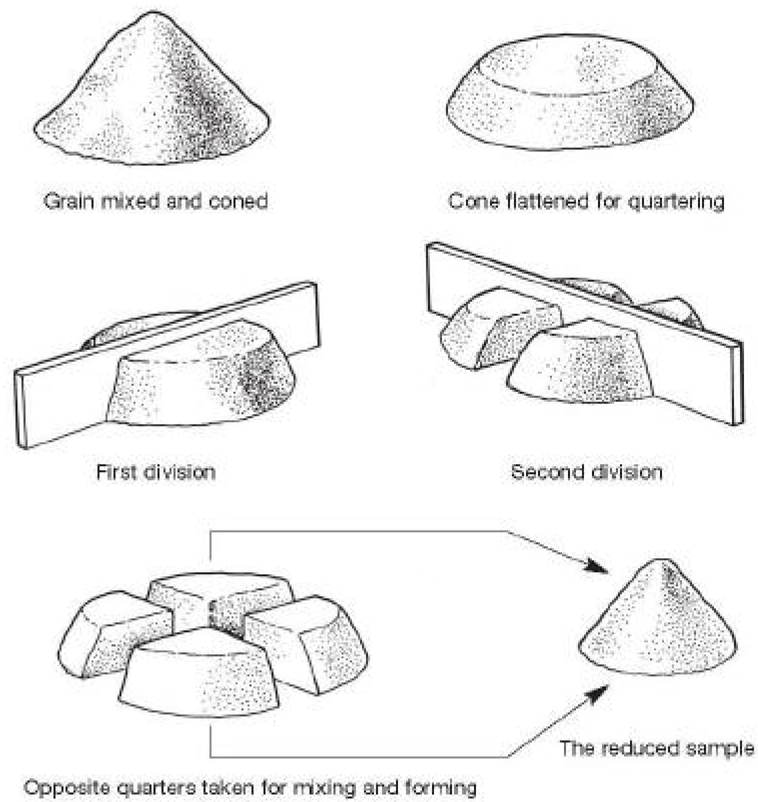
When a powder is stored in a container, it can be mixed by rolling and tumbling the container. The container should <u>not be more than half to two-thirds full</u>. It is important to perform this action before “grabbing” a sample with a spatula.



Then pull sample with a spatula…..

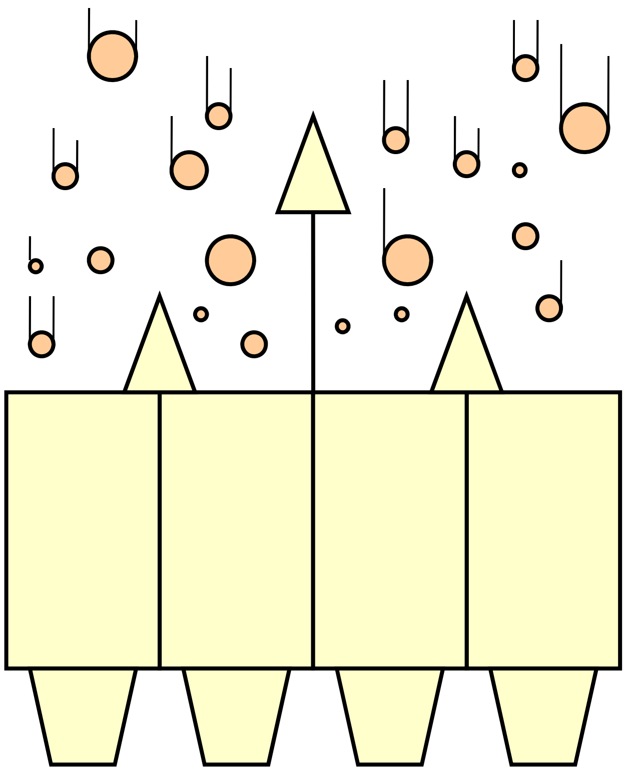
# Technique: Coning & Quartering

- 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 Riffling

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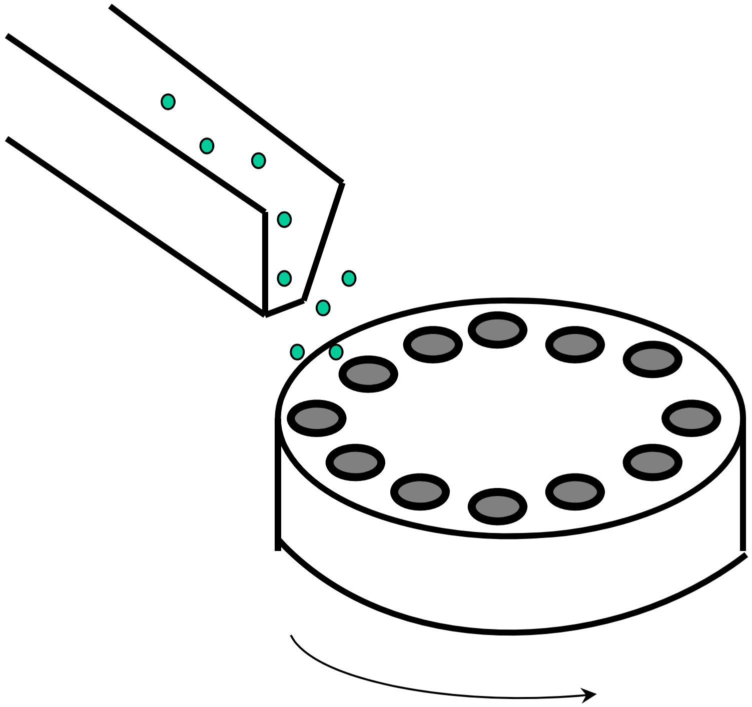
The best method of representative splitting of powders is the ROTARY RIFFLER. The complete sample to be split is directed down a chute into open containers. Each container will receive a sample which is representative of the original bulk material because the distribution of material is averaged over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

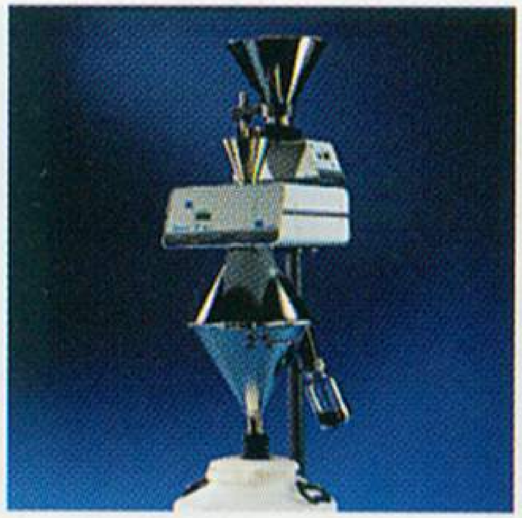
See: www.retsch.com

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*

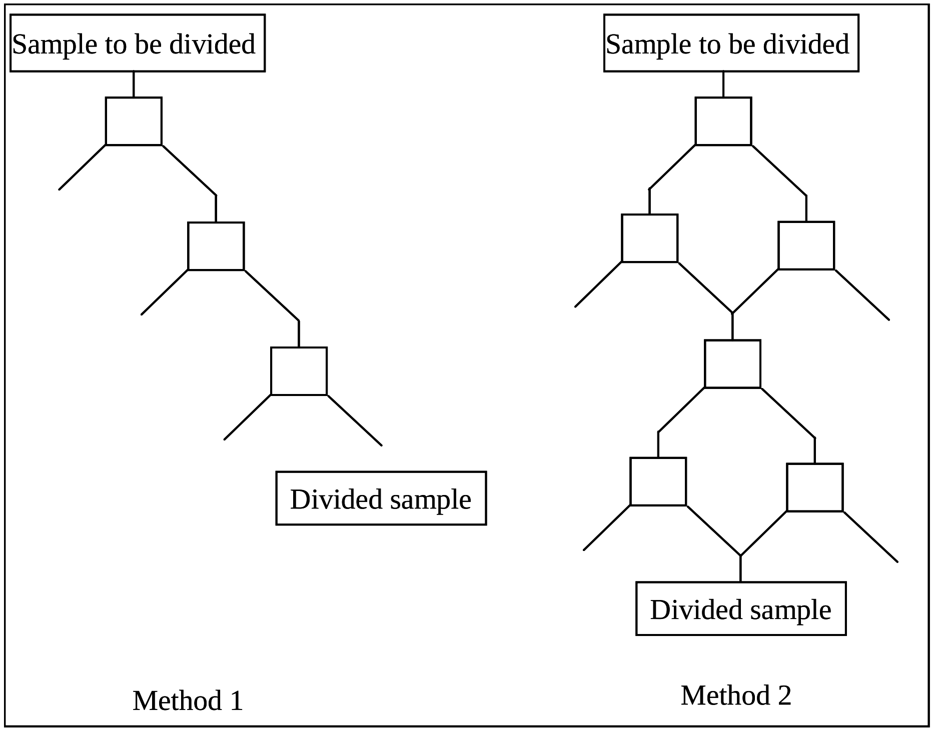


Laboratory rotary tube sample divider PKZ 1000
- *for pourable powders and granules*
- *feed size up to 10 mm*
- *various division ratios*

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

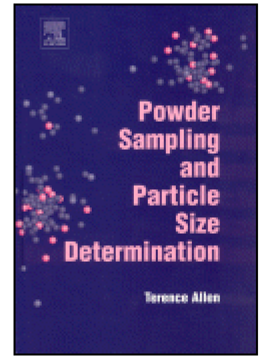
# Practical Concerns

- 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 |

| 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 |

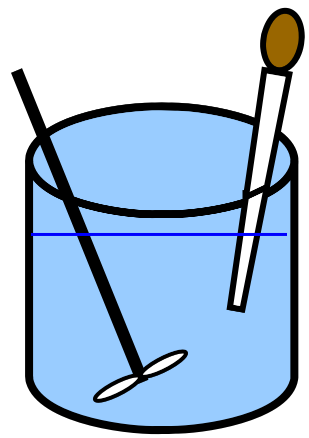
Sample riffled
All of sub sample dispersed and measured as a suspension

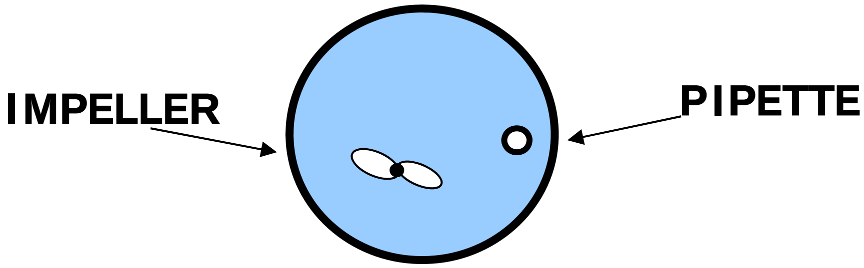
# Technique: Sampling from Beaker

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





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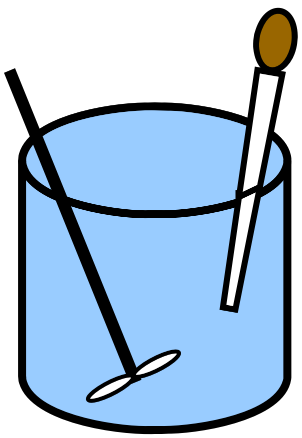
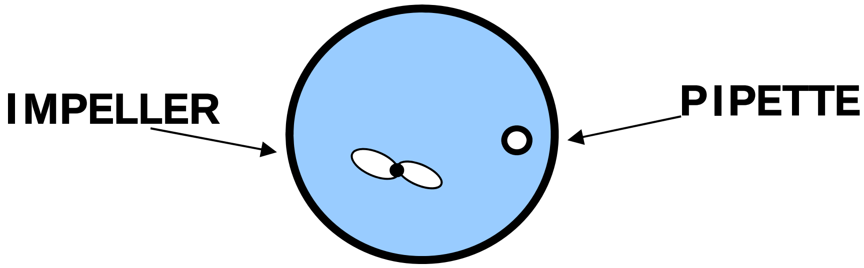
# Sampler Error w&w/o Mixing

| 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

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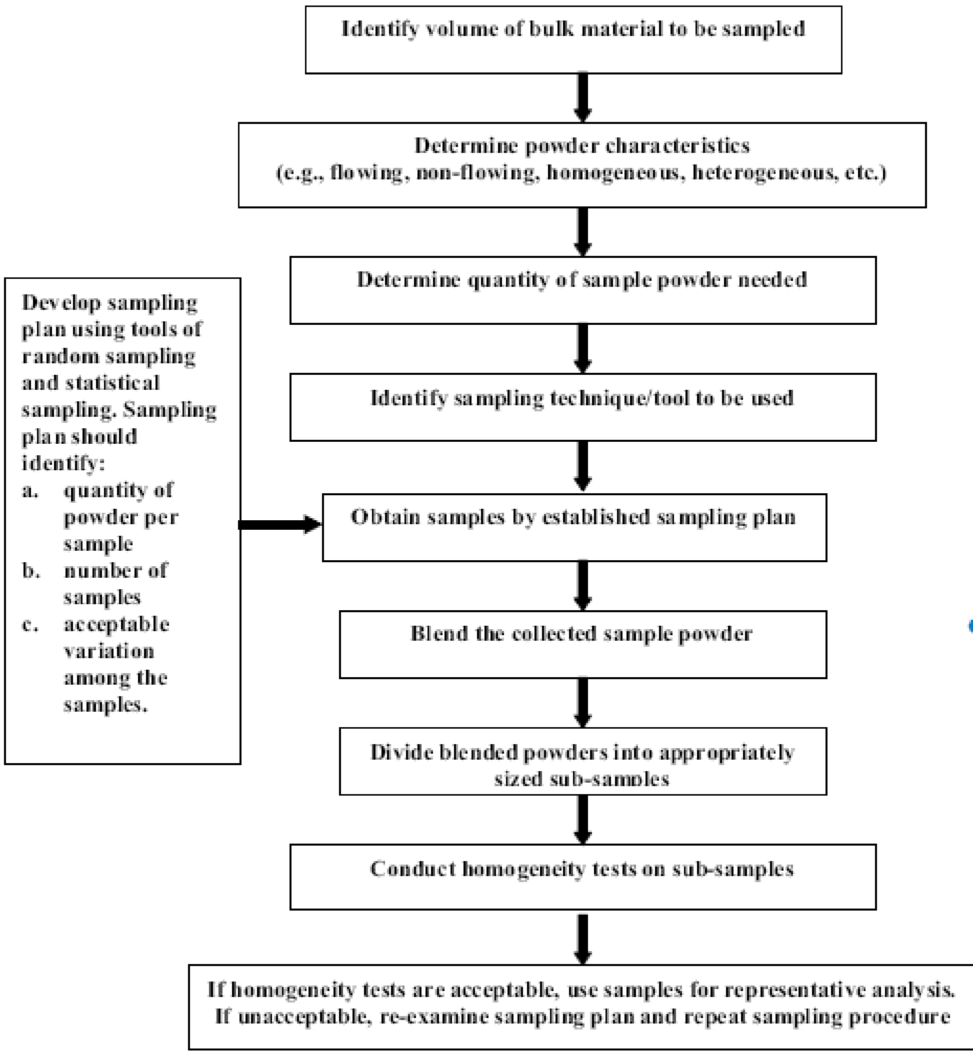


From: NIST Recommended Practice Guide
Special Publication 960-1
Particle Size Characterization
A. Jilla et.al.
Materials Science and Engineering Laboratory
January 2001

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