LUND UNIVERSITY

# Sampling and mixing



LUND UNIVERSITY

## Mixtures

- Type of mixtures
  - Positive mixtures- spontaneously homogenously - solutions
  - Negative mixtures-Spontaneously separate-emulsions
  - Neutral mixtures- neutral -powders
- Neutral mixtures
  - Homogeneity depends on handling and process conditions
  - Type of neutral mixtures
    - Random mix
    - “Perfect mix”
    - Ordered mix

## Sampling of powders

- Why do we take samples?
- How many samples?
- Where and with what technique?
- How much?





## Why do we take samples?

- To detect variation
  - validate processes
  - Product quality variations
- To describe the sample - gross sample “general prov”
- To control processes

## How many samples?

- Depends on what information you want
- Remember the statistics
  - Accuracy
  - Precision
  - Variance





## Variance

- Example
- 400g A with a weight of 0,05g
- 400g B with a weight of 0,1g
- Sample weight 50 g
- What is the Variance

- Effect of sample size on Variance

| Sample | $\sigma$ |
|---|---|
| 100 | 1,3% |
| 50 | 1,88% |
| 25 | 2,7% |
| 10 | 4,3% |

LUND UNIVERSITY

## Variance as a tool to estimates number of samples

- How representative is a sample

$$\textit{true}\ \text{value} = x_m \pm \frac{ts}{\sqrt{\text{n}}}$$

- Normal distribution
- 95% conf. Interval t=1,96
- t-distribution
- 95% conf. Interval t=2,14 for n=15-1

Number of samples

- Depends on wanted precision and standard deviations of samples

$$n = \left(\frac{ts_t}{E}\right)^2$$

7

LUND UNIVERSITY

## Where and with what technique?

- Discuss the following situations
  - A lorry comes in with grains to a mill
  - Validation of a tray dryer
  - Process sampling from a mill stream
  - Sampling from an oral dispersion in a bottle

8

## Golden rules of sampling

- A powder should be sampled when in motion
- The whole stream of powder should be taken for many short increments of time in preference to part of the stream being taken for the whole time



LUND UNIVERSITY

## How much?

- Best rule adjust to the situation (scale of scrutiny)
  - Product characteristic
  - Demand on accuracy
- However
  - To few particles will give to much variation
  - Low amount of one component increase the need of large samples for detection

LUND UNIVERSITY

## Effect of scale of scrutiny

| Sample number | N=1000 | N=10 000 | N= 100 000 |
|---|---|---|---|
| 1 | 1 | 7 | 108 |
| 2 | 0 | 10 | 91 |
| 3 | 2 | 8 | 105 |
| 4 | 1 | 15 | 116 |
| 5 | 0 | 13 | 84 |
| 6 | 1 | 10 | 93 |
| 7 | 1 | 6 | 113 |
| average /1000 | 0,85714286 | 0,98571429 | 1,01428571 |
| s | 0,69006556 | 0,32366944 | 0,12149858 |

True value 1/1000



LUND UNIVERSITY

## Mixing

- Purpose: To obtain as homogenous bulk as possible
- Common problems
  - Mix in a small amount of one substance in a large bulk
  - Segregation and over mixing
  - Mixing that effects the size of the particles

LUND UNIVERSITY

## Quality of a mix

- A mix is evaluated from standard mean and standard deviation (of appropriate kind)
- Problem
  - Segregated materials are not following a normal distribution
  - Is dependent on sample size
- Variance might give a better picture
- Mixing index also an alternative

$$M = \frac{\sigma_{randommix}}{\sigma_{sample}}$$

13

LUND UNIVERSITY

## Mixing mechanisms

- Convection
  - Due to circulating flow of powder during mixing
- Shear Mixing
  - The momentum exchange between the powder particles having different velocities
- Diffusion
  - The random motion of powder particles

14

LUND UNIVERSITY

## The mixing curve

From Powder technology handbook

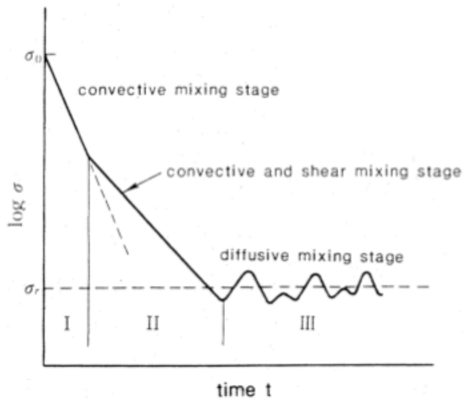



**FIGURE 6.2** Characteristic curve of mixing process (schematic).



LUND UNIVERSITY

## Mixing

- Standard equipment
  - High share mixers
  - Tumbling mixers
    - Y-cone
    - Rotating cube
    - Double cone
  - Fluidized bed
  - Agitator mixers
- Things to consider
  - Homogeneity
  - Risk for overmixing
    - Leading to demixing
    - Influence properties of particles
  - High share rates
    - Influence particle size for week powders
  - Scaling up
    - Not always linear

LUND UNIVERSITY

## Mixer types

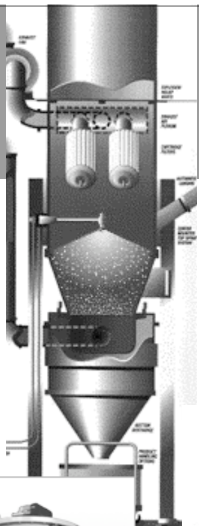
- Segregating
  - Rotating drum
  - V-blender
  - Double cone blender
  - Cubic blenders
- Non segregating
  - Ribbon blender
  - Nauta blender
  - Lödiger
  - Fluidizing blender (Forberg)

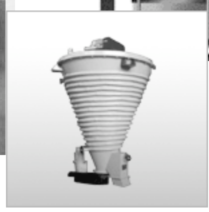
- Characterized by Froudes number
- Fr<1
  - Thrust mixers -Ribonblender, Nauta mixer
  - Free fall mixers -V-blender
- Fr≈1
  - Fluidized beds
- Fr>1
  - Centrifugal mixers
  - Intensive mixers

17

LUND UNIVERSITY

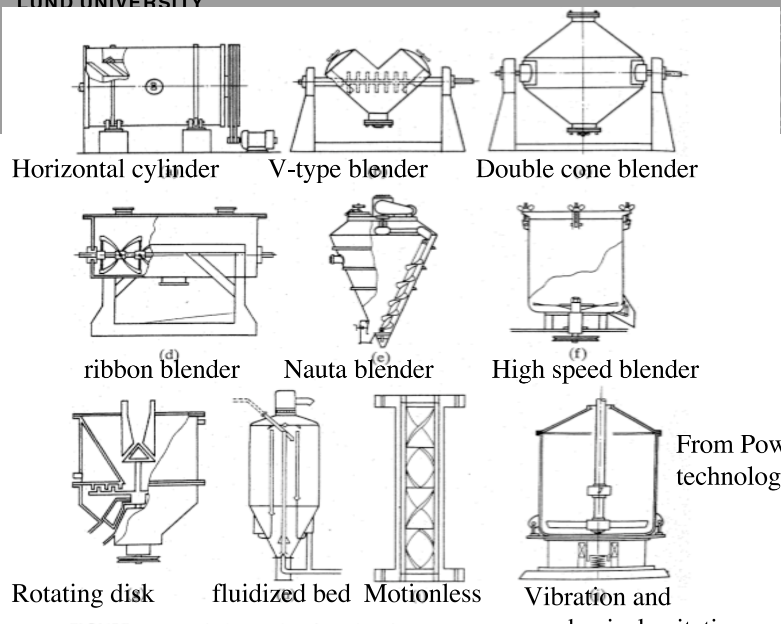
## How do they look




18

LUND UNIVERSITY




From Powder technology handbook

FIGURE 6.1 Typical examples of powder mixers.

19

LUND UNIVERSITY

## What to consider when designing a mixing process

- What's needed for homogenous mixer
- Mixing time
- Batch size
- Degree of filling
- Energy need
- Temperature
- Mild or shearing mixing
- Deagglomeration
- Segregation
- Handling of powder
- Cleaning
- Worker protection
- Explosion risks
- Prize
- Material
- Etc etc etc

20

LUND UNIVERSITY

## Segregation mechanisms

- Percolation-slip through the holes
- Trajectory Effect - size or density segregation due to air drag during filing or feeding

$$L = \frac{v_{\mathrm{h}} \rho d^2}{18\mu}$$

- Rolling Effect -Due to friction and gravity
- Stumbling Effect , Push-Away Effect
- Elutriation effects- dusting segregation-dust layer formed on top of particle bed
- Densification

21

LUND UNIVERSITY

## Segregation

- Can occur
  - During mixing
  - Storage
  - Transport
- Can be counteracted by
  - Narrow particle size
  - Ordered mixtures
  - Irregular and coehisive powder
  - Granulation
  - Ordered mixtures
  - Reduce vibrations

22