Sample Preparation in the Laboratory

FRITSCH GMBH
Milling & Sizing
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Laboratory mills from the World Market Leader
Your Partner with more than 80 years of experience

New production premises Werk 2
SAMPLE PREPARATION AND PARTICLE SIZING
– Products and brand name –

 Instruments from FRITSCH are known around the world under the brand names
  PULVERISSETTE
  ANALYSETTE
  LABORETTE

 Instruments for comminution
 Instruments for particle sizing
 Instruments for dividing and feeding

Sample Preparation

• exact, reliable and reproducible samples
• better, accurate performance from analytical instruments
• no poorly prepared samples
• great risk of errors
• proper selection of grinding technique and instrument
Errors in sample preparation

- **Contamination**
- **Material loss (recovery) and demixing**
- **Changes to the compositions (decay) & and the matrix through thermal and physical forces**
  - Active ingredient analysis in the pharmaceutical industry
  - Quality parameters of polymers (colour, moisture, cluster destruction)
  - SiC preparation (decay)
- **Samples – surface**
  - e.g. X-ray analysis (mineralogical effect - particle size effect)
  - Rheology of polymer powders such as pourability

Sampling and sample division

- The full initial quantity of a substance to be analysed, for instance to determine its chemical composition, is generally significantly larger than the quantity that can be analysed. This makes sampling, sample division and preparation of the sample necessary.
- The result of an analytical process can only supply meaningful information if the analysed sample is representative of the material investigated.
  - Initial quantity → desired → Q(x)
  - Sampling (laboratory sample) ↑ ?
  - Sample division → analysis → Q*(x)
- Systematic, random and statistical errors can lead to deviations between Q(x) and Q*(x).
Theoretical aspects of sample preparation

The core problem of sampling and sample preparation is the inhomogeneity of the samples. Extremely pronounced in solid materials, but flowing gases and liquids are also inhomogeneous. Additional inhomogeneity is possible over time (e.g. demixing during sample preparation).

The max. achievable homogeneity during sample preparation is uniform, random mixing - true mixed state !!!!!!

Main Criteria for the selection of a Laboratory Grinder

- **Type of material**
  - Physical and chemical properties
- **Initial sample characteristics**
  - Feed size (pre-crushing required?)
  - Quantity, Number of samples
  - Milling time, Cleaning
  - Representative sample
- **Universal applicability**
- **Final sample characteristics**
  - Contamination, Abrasion
  - Final particle size, Particle size range
Criteria for selection of a laboratory mill

Additional usage options

- Wet grinding possible?
- Cryogenic grinding required?
  - E.g. plastics
- Grinding under inert gas required?

Cleaning

- Sample carry-over
- Duration

Comminution principles according to Rumpf

Pressure  Friction  Impact  Cutting  Shearing

Collision, pounding

$F = \text{Force, } v = \text{Speed}$
Action between two Crushing Tools

- **Pressure Action** (i.e. Jaw Crusher)

  ![Diagram](attachment:Diagram.png)

  **Mining and metallurgy**: Niobium-titanium, ferrovanadium, chrome vanadium, tungsten carbide, ores, coal, slag, coke

  **Chemicals**: wide variety of various raw materials in the chemical industry

  **Geology and mineralogy**: Rocks, granite, basalt, barite, silicates

  **Glass industry**: Frits, glass, raw materials

  **Ceramics**: Steatite, fire-clay, sintered ceramics, electroporcelain

  **Construction materials**: Bauxite, clinker, quartz, concrete

NEW Jaw Crusher PULVERISSETTE 1 premium line
Action between two Crushing Tools

• Combined Pressure and Shearing Action (i.e. Disk Mill)

Mining and steel industries: Ores, coal, coke, slags
Ceramics: Steatite, sintered ceramics, electrotechnical porcelain, fire-proof clay
Rocks and soils: Bauxite, slags, quartz, clinker, gypsum, chalk
Glass: Frits, glass types, raw materials
Soil research: Dried soil samples, sewage sludge, hydrological sediments, drilling cores, briquettes, coke, limestone, Thomas meal (potash), pumice stone

NEW Disk Mill PULVERISETTE 13 premium line
Vibrating Cup Mill PULVERISETTE 9 classic line

STRONGER, FASTER, BETTER

- Especially ergonomic the P9 grinding sets, which have been considerably reduced in weight, are placed at an optimised working height on a guide rail to be easily moved to the final position
- Especially safe Tensioned in seconds with one hand and one grip
- Especially intelligent a special detector automatically detects use of agate grinding set, for reducing rotational speed independently
- Especially well-arranged self-explanatory, multilingual menu navigation on the LCD display for adjustment of grinding parameters
- Max. feed particle size: 12 mm
- Useful capacity: 50, 100 or 250 ml
- Final fineness: 10 – 20 µm

FRITSCH features

- More power with new drive concept
- Extra-fast grinding with up to 1500 rpm
- Precise adjustment of rotational speed, grinding time and pause periods
- Programming and storage of grinding cycles
- Self-explanatory multilingual menu navigation
- Especially safe hood locking
- Complete soundproof lining
Grinding sets Vibrating Cup Mill PULVERISSETTE 9 classic line

1st step

PULVERISSETTE 9 Fixing grinding set

FRITSCH GMBH • Milling and Sizing • Idar-Oberstein • Germany
NEW Pellet Press

For fast and easy preparation of both: solid and highly permeable pellets for X-ray fluorescence analysis or infrared spectroscopy – manual, hydraulic, easy.

- Variable pressure force of up to 250KN
- Simple operation via hand lever
- Solid and compact with impact-resistant cladding
- Easy cleaning

Action between two Crushing Tools

➢ Combined Friction and Pressure Action (i.e. Mortar Grinder)

Mining and metallurgy: Ores, coal, coke, ashes, slags
Chemistry: Fertilisers, dyes, pesticides, salts, detergents, synthetic resins, paints
Geology and mineralogy: Minerals (up to and including a Moh’s hardness of 9), calcite’s, quartz, silicates
Glass: Sand, frits, glass, raw materials
Ceramics: Porcelain, fire-clay, sintered ceramics, clay
Agriculture: Soil samples, fertilisers, organic plant materials
Foodstuffs: Confectionery, gelatine, spices, yeast, pasta, sugar
Metallurgy: Bauxite, slags, additives
Pharmacy: dragées, drugs, tablets, pastes, raw materials
Rocks and soils: Gypsum, lime, clinker, sand, cement
Electrical Industry: Graphite, semi-conductors, insulating materials
Mortar Grinder PULVERISETTE 2 classic line

<table>
<thead>
<tr>
<th>Operating principle</th>
<th>Pressure, friction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. feed size</td>
<td>8 mm</td>
</tr>
<tr>
<td>Max. capacity</td>
<td>150 ml</td>
</tr>
<tr>
<td>Final fineness</td>
<td>10-20 µm</td>
</tr>
<tr>
<td>Hard (abrasive)</td>
<td>-</td>
</tr>
<tr>
<td>Medium-hard</td>
<td>++</td>
</tr>
<tr>
<td>Soft</td>
<td>+</td>
</tr>
<tr>
<td>Brittle</td>
<td>++</td>
</tr>
<tr>
<td>Tough</td>
<td>-</td>
</tr>
<tr>
<td>Fibrous</td>
<td>+</td>
</tr>
<tr>
<td>Temperature-sensitive</td>
<td>++</td>
</tr>
<tr>
<td>Moist</td>
<td>++</td>
</tr>
</tbody>
</table>

Chemistry, pharmacy, stones and soils, bauxite, gypsum, foodstuffs

FRITSCH - features

- 250W – motor
- Rimmed, protected mortar bowl
- Very robust construction
- Large range of grinding pressures
- Easy setting of scraper
- TÜV - Safety
**Principle of a Planetary Ball Mill**

1. Bowl with balls and sample fixed on the sun disk
2. Sun-disk rotates
3. Bowls rotate like planets around the center of the sun-disk
4. Milling by battering and friction

**Surface characteristics / roughness influencing the comminution**

- Silicon nitride
- Aluminium oxide
- Zirconium oxide
Planetary Mono Mill PULVERISETTE 6 classic line

Operating principle  Percussion
Max. feed size      10 mm
Max. capacity       1 x 225 ml
Final fineness      <1 µm
Hard (abrasive)     ++
Medium-hard        ++
Soft               ++
Brittle            ++
Tough              +
Fibrous            +
Temperature-sensitive +
Moist              ++

Laser spectral analysis, chemistry, pharmacy, glass, ceramics, construction materials, coal, coke, mineralogy, geology, foodstuffs

FRITSCH - features

- patented planetary ball
- easier adjustable counter weight
- bench top model
- RS232 interface
- programmable interval, break time and reversing
- TÜV - Safety
- GTM-System
- Safe-lock
Nano particles
"What do we actually mean by this?"

And what are the benefits?

- Improved catalytic properties with hot surfaces
- Immense boundary surfaces in plastics and paints
- Photocatalytic properties
"Frequently a problem"

Agglomeration !!!!!

Molecules → Oligomers → Nano particles → Agglomerate

Sol-gel, precipitation techniques; microemulsion, gas phase processes

New conception

- FRITSCH
- PREMIUM LINE
- Bowls embedded IN
- the sun-disk
Bowls embedded

Faster

Graph showing milling time vs. d50 for conventional milling and Premium Line.
SAMPLE PREPARATION
– Chopping wood e.g. –

Impact force
Variable Speed Rotor Mill

- **Soft materials** plants, wood, roots, drugs, herbs, textiles, grain, seeds, chalk, kaolin
- **Medium-hard samples** tablets, dragées, animal feed
- **Elastic samples** styrene, PVC, PP, rubber, etc.

Variable Speed Rotor Mill PULVERISETTE 14 classic line

<table>
<thead>
<tr>
<th>Operating principle</th>
<th>Impact</th>
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<tbody>
<tr>
<td>Max. feed size</td>
<td>10 mm</td>
</tr>
<tr>
<td>Max. capacity</td>
<td>5 l/h</td>
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<tr>
<td>Final fineness</td>
<td>0.08 - 6 mm</td>
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<tr>
<td>Hard (abrasive)</td>
<td></td>
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<tr>
<td>Medium-hard</td>
<td>+</td>
</tr>
<tr>
<td>Soft</td>
<td>++</td>
</tr>
<tr>
<td>Brittle</td>
<td>+</td>
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<tr>
<td>Moist</td>
<td></td>
</tr>
</tbody>
</table>

Foodstuffs, cereals, plants, textiles, wood, chemicals, plastics, animal feed, coal, soils
FRITSCH - features

- Motor speed 6,000 – 20,000 rpm, constant torque
- Digital display of rated speed
- Soft-touch keyboard
- Microprocessor controlled
- TÜV - Safety

New Variable Speed Rotor Mill PULVERISSETTE 14 premium line
Action between two Crushing Tools

➢ Combined Cutting and Shearing Action (i.e. Cutting Mill)

Materials: rubber, leather, paper, cardboard, tissues, plastics, wood, non-metallic waste, coal, animal feeds, maize, grain, confectionery, malt, farinaceous products, spices, dried meat, bones, horn, dragées, tablets, leaves, fibres, peat, roots, tobacco, cork, straw or film.

Furthermore, the Universal Cutting mill can also be used in the pharmaceutical or animal feed industries, in dental, medicinal and metallurgical technology as well as in Veterinary Institutes.

Table: Operating principle

<table>
<thead>
<tr>
<th></th>
<th>P-19</th>
<th>P-25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. feed size</td>
<td>70 x 80 mm</td>
<td>120 x 85 mm</td>
</tr>
<tr>
<td>Max. capacity</td>
<td>60 l/h</td>
<td>85 l/h</td>
</tr>
<tr>
<td>Final fineness</td>
<td>0.25-6 mm</td>
<td>1-10 mm</td>
</tr>
<tr>
<td>Hard (abrasive)</td>
<td>-</td>
<td>+</td>
</tr>
<tr>
<td>Medium-hard</td>
<td>++</td>
<td>++</td>
</tr>
<tr>
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<tr>
<td>Moist</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
FRITSCH - features

- Fast locking lid
- Easy access and very easy cleaning
- Rotors
- Less sample deposits
- Very stable and robust
- Cutting geometry
- Bearings
- Sample exhaust
- Motors
- Sieve cassettes
- TÜV - Safety

SAMPLE PREPARATION
– Learning to divide –

Comminution is always followed by taking of a representative sample
- either for the next stage of comminution or
- for the analysis

For this purpose, we offer the Rotary Cone Sample Divider LABORETTE 27.
Rotary Cone Sample Divider LABORETTE 27

- Representative division of dry solids or suspensions
- Dividing heads with ratios 1:8, 1:10 and 1:30
- Representative division of randomly segregated samples

Importance of sample dividing

Rotary Cone Sample Divider

Normal Sample Divider

Without sample dividing = Not representative
Light diffraction for particle sizing

- Illuminating particles with light produces diffraction rings.
- The size of the rings correspond with size of particle.

Small particles → wide rings
Large particles → narrow rings
Light diffraction

Two different theories:

- Fraunhofer Diffraction (large particles)
- Mie Scattering (large and small particles)

Laser-Particle-Sizer *ANALYSETTE 22*

Measuring Range 0,01 – 2000µm Nanotecplus
Measuring Range 0,08 – 2000µm Microtecplus

Simple
Flexible
Reliable

Free notebook with the purchase of a FRITSCH Analysette-22 MicroTec plus or NanoTec plus
MicroTec plus optical setup

**IR Laser used for large particles**

IR Laser: ON, Green Laser: OFF

- Distance between Measurement Cell and Detector is large
- → small diffraction angles are detected
- → large particles

**Green Laser**

**IR Laser**

**Measurement Cell**

**Detector**

MicroTec plus optical setup

**Green Laser used for small particles**

IR Laser: OFF, Green Laser: ON

- Distance between Measurement Cell and Detector is small
- → large scattering angles are detected
- → small particles

**Green Laser**

**IR Laser**

**Measurement Cell**

**Detector**
NEW Imagesizer Analysette 28 20µm – 20mm

Thank you