CAMSIZER®

Particle Size and Shape using Dynamic Image Analysis
ISO 13322-2 conform

HORIBA  Scientific & Retsch Technology
CAMSIZER Fertilizer Webinar

November 16th 2011

Host:  Ian Treviranus, HORIBA Instruments
Speaker: Dipl.-Ing. Gert Beckmann
Retsch Technology GmbH
Content

* CAMSIZER measurement principle
* Intelligent matching of sieve results
* SGN, UI and other calculations
* Q & A session

Device

1. Measuring Principle
2. Measurement Results

Application

3. CAMSIZER Applications in the Fertilizer Industry
4. Competing Measuring Methods
Retsch Technology

Development of CAMSIZER & CRYSTALSIZER

1999

Announcement of Particle size analyzers in Europe

2001

New shape functions of CAMSIZER, first Online-Installations

2002

Development of AutoSampler System

2003

Development of CAMSIZER XT for agglomerating powders 1µm-3000µm

2011

Improvement of CAMSIZER® with AutoHeight, LED technology, Software, improved resolution, sharpness and contrast

2006
Comparison between Static ⇔ Dynamic Image Analysis

ISO 13322-1

ISO 13322-2
Content

Device
1. Measuring Principle
   - Two Camera-System
   - Resolution
2. Measurement Results

Application
3. CAMSIZER Applications in the Fertilizer Industry
4. Competing Measuring Methods
Two-Camera-System

Measuring Principle

Basic-Camera

Zoom-Camera
Detection of particles

One pixel is an element of a projection when at least half of the pixel is covered.
One Camera Setup

Drawback: tight dynamic range

Dynamic Range: $x_{\min}$ to $50 \cdot x_{\min}$
CAMSIZER Principle (Two Cameras)

**Advantages**

- Precise full-frame images
- Wide dynamic range: 30µm to 30mm

Dynamic Factor: \( \frac{X_{\text{max}}}{X_{\text{min}}} = 1000 \)
Content

Device
1. Measuring Principle
2. Measurement Results
   - Size
   - Shape

Application
3. CAMSIZER Applications in the Fertilizer Industry
4. Competing Measuring Methods
What is the size of this particle?
Measuring Principle:

- Sphere
- Stick
- Coin
Visualization
Cumulative distribution

Q₃ [%]
p₃ [%]

Particle size x[µm]

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a VERDER company
Visualization
Cumulative distribution

Q₃ [%]

Particle size x [µm]

P₃ [%]

Visualization
Cumulative distribution

Q₃ [%]

Particle size x [µm]

P₃ [%]
Visualization
Cumulative distribution

Q₃ [%]

p₃ [%]

Particle size x[µm]

45 50 63 100 140 250 500 1000 2000 6000

10 20 30 40 50 60 70 80 90

90 80 70 60 50 40 30 20 10
Visualization
Cumulative distribution

Q₃ [%]

p₃ [%]

Particle size x [µm]
Visualization
Cumulative distribution

\[ Q_3 [\%] \]

\[ p_3 [\%] \]

Particle size \( x [\mu m] \)

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Visualization
Cumulative distribution

Q₃ [%]

p₃ [%]

Particle size x[µm]

45 50 63 100 140 250 500 1000 2000 4000 6000

10 20 30 40 50 60 70 80 90
Visualization
Cumulative distribution

Q₃ [%]

P₃ [%]

Particle size x[µm]

Cumulative distribution chart showing the percentage of particles below a certain size range. The x-axis represents the particle size in micrometers, and the y-axis represents the cumulative percentage Q₃. The chart includes a histogram and a cumulative distribution line for p₃.
Visualization
Cumulative distribution

Q₃ [%]

Particle size x [µm]

p₃ [%]
Visualization
Cumulative distribution & Fractions

Q₃ [%]

Particles < 330 µm

Particles < 250 µm

250 µm  330 µm
Particle Size

\( x_{cmin} \)  “width”

\( x_{area} \)  “diameter over projection surface”

\( x_{Fe\ max} \)  “length”

CAMSIZER results are compatible with sieve analysis
Comparison of Size Definitions

Different size definitions

Different results

Measurement Results
Reproducibility

Calibration with traceable standard

=> Absolute accuracy
Whitehouse Standard XX030
Maintenance-Free by Venturi-Flushing
Content

Device
1. Measuring Principle
2. Measurement Results

Application
3. CAMSIZER Applications in the Fertilizer Industry
4. Competing Measuring Methods
Places of Installation

- laboratories
- next to manufacturing line
- optimized quality control of product and process
- research institutes / universities

Examples of Applications

- fertilizer
- salt
- crop protection
- pharmaceuticals
- food (human and animals)
Quality Control

Features

Reg. 56266; 10-20 mm

Company: Retsch Technology

Material: Limestone

Grades:

- 30
- 50
- 70
- 90
- 118

Table:

<table>
<thead>
<tr>
<th>Grade (µm)</th>
<th>0.063</th>
<th>0.086</th>
<th>0.106</th>
<th>0.125</th>
<th>0.16</th>
<th>0.20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Count (%)</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Spreading Width (µm)</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Size (µm)</td>
<td>0.063</td>
<td>0.086</td>
<td>0.106</td>
<td>0.125</td>
<td>0.16</td>
<td>0.20</td>
</tr>
<tr>
<td>Mass (g)</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
</tbody>
</table>

Characteristics:

- Q2 [%] = 50
- Spreader = 0.081
- LO = 1.246
- φa = 0.021 in/m

User:

a VERDER company
Specific Fertilizer Features

**UI and SGN values (Literature)**

**Potash Processing in Saskatchewan – A Review of Process Technologies.**

By

Carlos F. Perucca  
Sr. Metallurgist  
AMEC Engineering & Construction Services Limited

The uniformity index (UI) is the particle size at which 95% of the material is retained, divided by the particle size at which 10% of the material is retained, multiplied by 100.

The UI is a measure of the uniformity of the size distribution of the product, when all the particles are exactly the same size, the size distribution curve is flat and the UI is 100%. Usual values of UI for premium products are about 40%.

Figure 1 describes the calculation of the SGN number and UI index.

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**Table 2: Fertilizer Grade Potash Specifications**

<table>
<thead>
<tr>
<th>Tyler Mesh</th>
<th>Opening [mm]</th>
<th>Granular (% cum.)</th>
<th>Coarse (% cum.)</th>
<th>Standard (% cum.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>3.360</td>
<td>25.0</td>
<td>17.5</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>2.380</td>
<td>73.0</td>
<td>53.0</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>1.680</td>
<td>97.0</td>
<td>88.0</td>
<td>7.0</td>
</tr>
<tr>
<td>14</td>
<td>1.190</td>
<td>99.5</td>
<td>98.8</td>
<td>29.0</td>
</tr>
<tr>
<td>20</td>
<td>0.841</td>
<td>99.5</td>
<td>98.8</td>
<td>50.0</td>
</tr>
<tr>
<td>28</td>
<td>0.600</td>
<td>99.7</td>
<td>98.8</td>
<td>78.0</td>
</tr>
<tr>
<td>35</td>
<td>0.420</td>
<td>99.7</td>
<td>98.8</td>
<td>87.0</td>
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<tr>
<td>65</td>
<td>0.210</td>
<td>98.9</td>
<td>98.8</td>
<td>98.0</td>
</tr>
<tr>
<td>100</td>
<td>0.149</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SGN</td>
<td>285</td>
<td>245</td>
<td>245</td>
<td>49</td>
</tr>
<tr>
<td>UI</td>
<td>49</td>
<td>38</td>
<td>20</td>
<td></td>
</tr>
</tbody>
</table>

**Figure 1**: Calculation of the SGN number and UI index.

\[ UI = \frac{X_{5}}{X_{90}} \times 100 \]
SGN and UI values also with CAMSIZER

Specific Fertilizer Features

Characteristics

Basic characteristics

<table>
<thead>
<tr>
<th>x(Q) values:</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q1</td>
<td>10</td>
</tr>
<tr>
<td>Q2</td>
<td>50</td>
</tr>
<tr>
<td>Q3</td>
<td>90</td>
</tr>
</tbody>
</table>

- Span value
- Nonuniformity

- Q(x) values:
  - 1-Q(x) values:
    - x1 = 1 mm
    - x2 = 2 mm
    - x3 = 4 mm

Further characteristics

Shape characteristics

Based on number

- Q3 (SPHT): SPHT < 0.9
- Q3 (Symm): Symm < 0.9

Based on volume

- Mean value over all particles:
  - Mean value SPHT3
  - Mean value Symm3

Print in report

- Q3(shape)

RRSB characteristics

- Calculate x(Q) and Q(x) based on Q0
- Calculate x(Q) and Q(x) based on Q3

<table>
<thead>
<tr>
<th>Q1</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Q2</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td></td>
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</tbody>
</table>
Analysis of size and shape of broken, granulated, and coated fertilizers.
Different Fertilizer Product Types, Different Production Methods, as well as Different Sizes and Shapes
Sample Quantity in Mass or Volume in Relation to the Particle Size

ISO 13322-1

Tables 1 to 3 list, as a function of the geometric standard deviation $\sigma_g$ of the test powder, the number of particles to be measured, $N$, the median particle size of a cumulative mass distribution, $x_{30.3}$, the Sauter Diameter, $x_{1.2}$, and the average mass diameter, $x_{3.0}$, for the precision, respectively. $\delta = 0.05, 0.1$ and $0.2$. Here, the confidence coefficient probability, $p$, is equal to 0.95.

Table 1 - Number of particles to be measured $N$, $\delta = 0.05, p = 0.95$

<table>
<thead>
<tr>
<th>$\delta$</th>
<th>$\sigma_g$</th>
<th>$x_{30.3}$</th>
<th>$x_{1.2}$</th>
<th>$x_{3.0}$</th>
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<tbody>
<tr>
<td>0.05</td>
<td>1.10</td>
<td>585</td>
<td>389</td>
<td>131</td>
</tr>
<tr>
<td>0.15</td>
<td>1.15</td>
<td>1460</td>
<td>934</td>
<td>294</td>
</tr>
<tr>
<td>0.20</td>
<td>1.20</td>
<td>2939</td>
<td>1808</td>
<td>528</td>
</tr>
<tr>
<td>0.25</td>
<td>1.25</td>
<td>5223</td>
<td>3103</td>
<td>843</td>
</tr>
<tr>
<td>0.30</td>
<td>1.30</td>
<td>8526</td>
<td>4920</td>
<td>1247</td>
</tr>
<tr>
<td>0.35</td>
<td>1.35</td>
<td>13059</td>
<td>7355</td>
<td>1750</td>
</tr>
<tr>
<td>0.40</td>
<td>1.40</td>
<td>19026</td>
<td>10504</td>
<td>2363</td>
</tr>
<tr>
<td>0.45</td>
<td>1.45</td>
<td>26617</td>
<td>14457</td>
<td>3096</td>
</tr>
<tr>
<td>0.50</td>
<td>1.50</td>
<td>36007</td>
<td>19295</td>
<td>3956</td>
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<tr>
<td>0.55</td>
<td>1.55</td>
<td>47358</td>
<td>25093</td>
<td>4952</td>
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<tr>
<td>0.60</td>
<td>1.60</td>
<td>60811</td>
<td>31919</td>
<td>6092</td>
</tr>
</tbody>
</table>

A Sufficient Sample Quantity is Based on the Number of Particles
Separation of fine and coarse particles

Separation happens during
- Transport processes (container, train and truck)
- Feeding processes (funnels, vibration feeders, belts)
- and Storage (bulk pile, silo)
Separation of fine and coarse particles

Separation happens because of different sizes

Particles having different sizes separate

Particles with similar sizes keep homogeneity in mix
Separation of fine and coarse particles

Segregation (separation by size) happens during
- Filling processes (silo)
- Feeding processes (bulk pile)
- Accumulation of fines in the middle of the pile
How to attach small „Dust“ Particles to the larger Granules to get a „better“ Product?

Fertilizer producer coats the granules with petrochemical oil and amine to bind the dust to the granules for better handling to avoid accumulation of dust.
Only complete Fertilizing provides good Nutrition

Plant availability

Optimum soil condition

Anything else is wasted money and pollution of our environment

Chalk / Calcium

2nd fertilizing

1st fertilizing

nitrogen

potash

phosphoric acid
A German Fertilizer Producer is mining and producing KCl, MgSO₄ and KSO₄

Before 2000 => Sieve analysis 0.05 – 2.5 mm
After 2000 => CAMSIZER analysis
Applications – Fertilizer

Shaft Mining of Potash KCl
Handling of Potash KCl
Bulk Blending Companies and Farmers want the Same Size of the Components
Fertilizer in the Silo
Urea Fertilizer (also for AdBlue / BlueTec)

SCHEME I

\[
\begin{align*}
\text{OC(NH}_2\text{)}_2 & \rightarrow \text{H}_2\text{N-C-NH}_2 \quad \text{(Decomposition)} \\
\text{NH}_4^+ \quad \text{O} & \text{C} \quad \text{N} \\
\end{align*}
\]
Applications – Fertilizer

Prilling Wheel

Prilling = Multiple Core Encapsulation

Seconds of flying time to 1/3 congealed
(detailed conditions in text)

- Rot. - 30 m/s
- Rot. - 3 m/s
- Pnoz. - 50 m/s
- Shower - 10 m/s

Size (my)

0 1000 2000 3000

2-Fluid Nozzles
~150 m/s, narrow angle

Rotary, + - vanes etc.
~150 m/s, flat spray

Pressure Nozzles
~50 m/s, ~60° angle

Rotary, small Holes
~30 m/s, flat spray

Shower Head
~5 m/s, narrow angle

SPAN = 2.0
d99 = 3.5 x

SPAN = 1.6
 d99 = 2.8 x

SPAN = 1.2
 d99 = 2.2 x

SPAN = 1.0
 d99 = 2.0 x

SPAN = 0.8
 d99 = 1.8 x

SPAN = 0.5
 d99 = 1.5 x

PRILLING FERTILIZER & IN-ORGANICS

PRILLING FOOD & PHARMA

Avg. particle size my

0 5 10 20 50 100 200 500 1000 2000 5000 10000

d50

mm

0.01 0.02 0.05 0.1 0.2 0.5 1.0 2.0 5.0 10.0

Solutions in Particle Sizing

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Fluidized Bed Granulation

Applications – Fertilizer

Top-Spray

Bottom-Spray

Tangential-Spray
Urea Agglomerates
Previous
Roundness Measurement System
CAMSIZER Test Fertilizer

excellent repeatability
Sample Taking Fertilizer

with spoon from bag
Extreme Segregation because of Bimodal Size
Sample Splitting

not Representative

Representative
Sample Splitting

Representative
Sample Splitting

$\frac{1}{6}$, $\frac{1}{8}$, $\frac{1}{10}$
Sample Splitting Fertilizer Mix

Riffle Splitter

![Graph showing sample splitting with Riffle Splitter](image-url)
Mixture of Fertilizers

*Blue* = bimodal sample
*Green* = monomodal Poly S
*Red* = 3-modal Mixture
3-Size Mixture
Improved Product Fertilizer

Blue = monomodal Poly S
Green = “1.5”-modal Urea + Phosphate
Red = “1.5”- modal Mixture of 3 products
Slow Release Fertilizer Particulates

- Urea Core
- Sulfur Layer
- Polymer Coating

Urea Nutrient Core

Outer Polymer Coating

Inner Sulfur Coating

Urea Core + Sulfur layer + Polymer Coating
Coating Layer Thickness

Urea Pellets are a solid round form of nitrogen fertilizer. The individual pellets are almost spherical with diameters usually between 1000 µm and 3000 µm, sometimes 5000 µm.

Coatings can be measured with an accuracy of 2 µm layer coating thickness.
Problems during Fluid Bed Processing

- Outer External Polymer Coating
- Inner Sulfur Coating
- Urea Nutrient Core

Particles stick together during the coating and drying.
Type and amount of fertilizer is dependent on the soil content, the water quality, the type of plant, and the developing process of the specific plant.

30% – 60% of nutrition application can be solved with depot fertilizer.
Slow Release Fertilizer Particulates

- NPK + Traces Core
- Polymer Resin Layer
- “Glue” Layer

NPK+ Core
+ Polymer Resin Layer

Outer “Glue“ Coating

Inner Polymer Resin Coating

NPK Nutrient Core

NPK+ Core
+ Glue Coating Layer
Particle Shape

- **Breadth-/Length-ratio**
  \[ X_{Fe\,max} \quad X_{C\,min} \]

- **Roundness**
  \[ A \quad P \]

- **Symmetry**
  \[ r_1 \quad r_2 \quad C \]

- **Convexity**
  \[ A_{\text{convex}} \quad A_{\text{real}} \]
Optical Process Control
analysis for size and shape

Measurement Results

- x-values in mm
  - Nr=0,  d=2.148,
  - xFe_min=2.148,  xFe=2.203,  xFe_max=2.241,
  - xMa_min=2.134,  xMa=2.199,  xMa_max=2.241,
  - xc_min=2.148,  xc=2.200,  xc_max=2.241,
  - Symm=0.9827,  b/l=0.9480,
  - SPHT=0.9818,
  - Convex=0.9997

- x-values in mm
  - Nr=0,  d=1.966,
  - xFe_min=1.970,  xFe=2.546,  xFe_max=3.063,
  - xMa_min=1.889,  xMa=2.419,  xMa_max=3.047,
  - xc_min=1.966,  xc=2.419,  xc_max=3.060,
  - Symm=0.9289,  b/l=0.6361,
  - SPHT=0.9066,
  - Convex=0.9995

OK
Urea with Granular Potash

Amounts of urea beads and pressed/crushed potash granules in a mixture
Urea with Granular Potash

CAMSIZER can find the mixing ratio of components of Urea Beads and Granular Potash.

Measurement Results

Q₃ (round particles) = 32.8%
Traditional Measurement Pellets

- Sieve analysis → 1.0 – 3.0 mm
- Vibrating plate and weighting
  (amount of broken particles)
CAMSIZER – Advantages
Measuring Broken Beads

good product

broken particle

20% broken
17.4% broken

Symmetry $\sim \min \left(\frac{r_1}{r_2}\right)$
Spreading Fertilizer Particulates
Larger pellets can be thrown further than smaller pellets.

The exact size is important to get controlled spreading conditions.

**Spreading Fertilizer Particulates**

- **Graph 1:** Comparison of fertilizer spread (N/ha) under controlled conditions for different tracks and tubule heights (mm).
  - **Track 1:**
    - 15 m: 94 N/ha
    - 24 m: 56 N/ha
  - **Track 2:**
    - 15 m: 78 N/ha
    - 24 m: 59 N/ha

- **Graph 2:** Comparison of fertilizer spread under different conditions for different tracks and tubule heights (mm).
  - **Track 1:**
    - 6 N/ha
    - 64 N/ha
    - 61 N/ha
  - **Track 2:**
    - 6 N/ha
    - 78 N/ha
    - 59 N/ha
Samples 1-8, following samples show identical results (split samples?)
1=7, 3=5, 2=8, 4=6
Calcium Phosphate

Samples 1-4 b/l = aspec ratio, 2 groups “1+4” and “2+3”
Applications – Fertilizer

Calcium Phosphate

Repeatability: 5 consecutive measurements
Sieve Correlation Calcium Phosphate

CAMSIZER sieve correlation using Elementary Fitting
one single sieve class and entire sample
## Competing Measuring Methods

### MEASURING DEVICES

<table>
<thead>
<tr>
<th>Method</th>
<th>Particle Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Analysis (dry)</td>
<td>20µm to 30mm</td>
</tr>
<tr>
<td>CAMSIZER</td>
<td>30µm to 30mm</td>
</tr>
<tr>
<td>Light Diffraction</td>
<td>1nm to 3mm</td>
</tr>
</tbody>
</table>

- **Sieve Analysis (dry)**: Suitable for particles ranging from 20µm to 30mm.
- **CAMSIZER**: Can measure particles from 30µm to 30mm.
- **Light Diffraction**: Suitable for particles as small as 1nm up to 3mm.
Comparison of Methods: Sieving

Advantages

- robust and industrial-suited
- easy handling
- references available from user

Disadvantages

- high amount of time and work
- low resolution,
  small number of investigatable classes
- limited sample amount
- no shape analysis possible

Worn out sieves

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Sieving problems

1. Move

2. Sliding friction

3. Static friction

Round particles are captured without rerelease
Fitting of CAMSIZER result to Sieving

**fitted result**

CAMSIZER-measurement \(x\) (red) to sieving \(\ast\) (blue)
Digitale Imaging ↔ Sieving
Digital Imaging ⇔ Sieving Cubes / Angular Particles
Fitting of CAMSIZER result to Sieving

**fitted result**

CAMSIZER-measurement \( x \) (red) to sieving * (blue)
Digital Imaging ↔ Sieving

New elementary fitting with single (narrow) sieve class and entire distribution

Elementary - Fitting
Digital Imaging ⇔ Sieving

Q₃ – Fitting

Elementary - Fitting
12 Years CAMSIZER

550 installed Instruments worldwide:
Nearly on all continents
For many applications/industries:
**Ammonium phosphate, Bulk**
blending companies, **Crop**
protection, **Dicalcium phosphate, Explosives**
**Fertilizer**.......
Thank you for your attention!