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# Laser diffraction particle size analysis of additives used in the animal feed sector

This datasheet describes the above measurement protocol according to the method requested by TECALIMAN. It takes as its main reference a comprehensive study carried out at the end of the 1990s.

## 1. Principle

A laser particle size analyser works by diffracting a coherent light source in contact with a stream of particles into a thin cell. Particle diameter is given by the diameter of its projected area rotated in every possible direction.
When a spherical particle is illuminated by a parallel beam of monochromatic, coherent light (Laser), a light diffraction pattern is superimposed on the geometric image. This pattern is bigger than the image, with small particles diffracting light through a larger angle than do large particles. The particle size range is divided into size intervals that each generate a specific diffraction pattern according to average size; the intensity varies according to the number of particles present. Particle size distributions are established by analysing diffraction patterns (Nathier-Dufour *et al.*, 1993).
This measurement has to be made on a product in suspension in liquid or air. This datasheet only studies the case of air dispersion. The principle of liquid dispersion was abandoned due to the necessity of developing a method that could be adapted to work with the entire range of products used in the animal feed sector. Where liquid dispersion is concerned, it would have been necessary to develop a method that would not act as a solvent for any product in the mix.

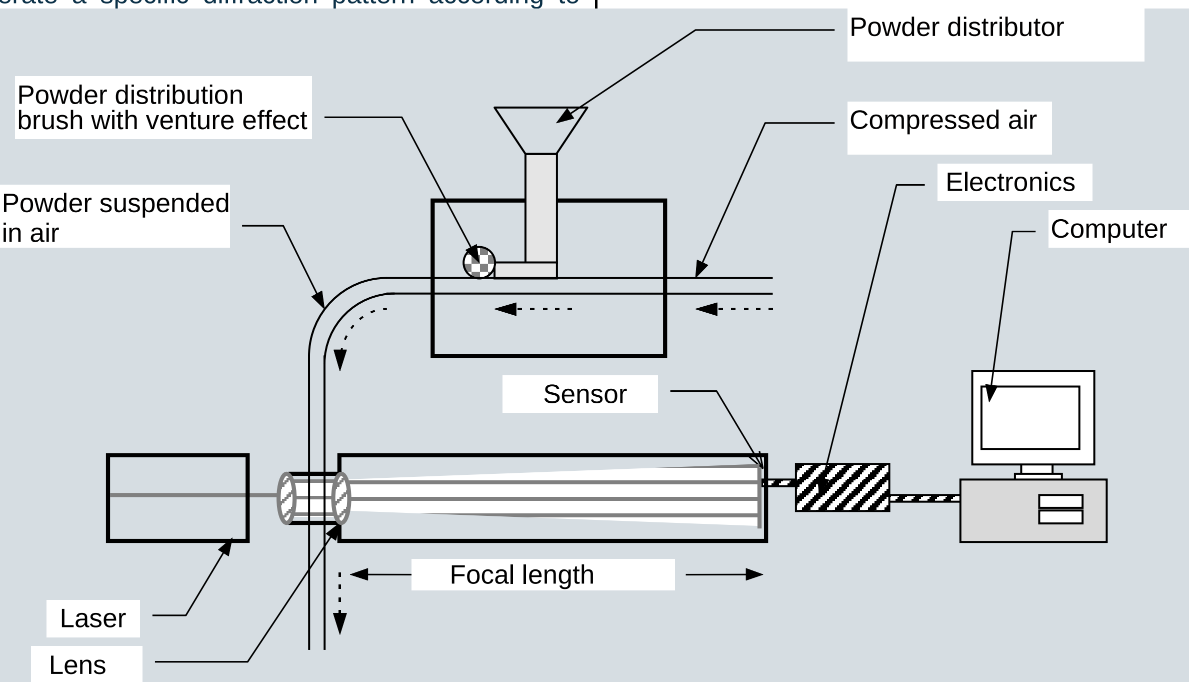


**Figure 1: Diagram of a laser particle size analyser with the dispersion of dry products in air**

## 2. Equipment and apparatus

The laser particle size analyser used was the MALVERN brand Mastersizer IP (Figure 1). The module used to disperse the products in air was the "Dry Powder Feeder MS65" machine.

## 3. Operating procedure

The particle size analyser takes the measurement via a lens with a focal length of 1000 mm. The air is pressurised to 1 bar.

Each measurement is taken according to the following protocol:

The product is placed in the MS65 distribution cell.

The system is flushed with an air flow.

The laser beam is lined up on the centre of the sensor.

A blank measurement is taken of the air flow.

Start of product throughput

The product's flow rate is adjusted to ensure an adequate beam focus.

The measurement is taken during 4,500 cycles.

The air flow and product throughput is switched off.

The computer makes the calculation.

The results are displayed on the screen.

The spectrum is checked in order to decide whether to retain this measurement. If the measurement is retained, the results are printed out and saved. The measurement will be rejected in the following cases:

Error in the analysis procedure

Presence of clusters in the top part of the spectrum: in this case, the measurement will be retaken by blocking the screening of certain central sensors.

Residual value of over 2%: the residual value represents the error between the estimated particle size distribution based on the applied dispersion model (in our case, a separate model) and the actual measurement taken. In other words, a residual value below 2% indicates that the model is correct in 98% of cases.

## 4. Expression of the results

There are several ways in which to express particle size distribution. Measuring the particle population of a powder gives either a frequency table, or, in accordance with each diameter size category, the percentage of detection for this particle category. Each category is characterised by an intermediate diameter that lies between the category's upper and lower bounds. Based on the expression of the empirical moment of the order q, given by:

$$m_q = \frac{\Sigma n_i \, (d_i)^q}{\Sigma n_i}$$

the following diameters are defined according to convention (Standard NF X 11-630):

$D_{4.3} = m_4 / m_3$ : mean weight or volume diameter

$D_{3.2} = m_3 / m_2$ : mean surface area diameter

$D_{2.1} = m_2 / m_1$ : mean straight line diameter

$D_1$ : mean number diameter

The advantage of studying the full range of mean diameters lies in the differences in expression of certain fractions of the spectrum depending on the diameter. The size of the fine-grained particle fraction is easier to express using the median diameter for numbers rather than weight, as this represents a large number of particles but a low weight.

There is another diameter that expresses particle size distribution in a more conventional manner. This is the median diameter $D_{50}$ below and above which can be found 50% of the population. The laser diffraction method uses the median volume diameter: diameter of a sphere with the same volume.

The closer the particle size distribution is to a Gaussian curve, the closer the median diameter will be to the median volume diameter $D_{4.3}$.

A comprehensive study carried out by TECALIMAN found that the median diameter gave the most accurate results as, in contrast to the other diameters, it is less affected by mathematical interpretations based on the measurement of the diffracted light beam.

A particle size spectrum can only be illustrated by a mean or median diameter. This requires a factor for the distribution around this diameter.

For the median diameter, when using the Malvern laser particle size analyser, the distribution factor is referred to as a Span, and is calculated as follows:

$$Span = \frac{D_{90} - D_{10}}{D_{50}}$$

$D_{90}$: diameter below which lies 90% of the population

$D_{10}$: diameter below which lies 10 % of the population

The MALVERN results tables include:

The name of the product and the general measurement conditions

The table of percentages for 32 particle size categories

The various diameters and the Span

The spectrum

## 5. Intrinsic properties

Based on 5 measurements taken on 5 very different products (from 10 to 550 µm) selected as being representative of the additive domain conventionally used in the animal feed sector at a four-day interval, the performance of this method was tested by determining the median volume diameter.

This method can be considered as being:

Sensitive: It differentiates at least four groups among the 5 products (Table 1). While it does not differentiate between products 3 and 5 with respective diameters of 13.1 and 14.3 µm, it does identify the latter as being different from product 2 with a diameter of 9.2 µm.

Accurate: Over the whole of the tested domain, the variation that cannot be explained directly by the tested factors (product, repetition, days) referred to as the residual error is 2.8%. This suggests that at least 2 basic measurements are needed to obtain reliable data.

Reproducible: There is no significant variation in the overall mean according to day.

| Products | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Median diameter | 315.9 | 9.2 | 14.3 | 554.7 | 13.1 |
| Group | *b* | *d* | *c* | *a* | *c* |
| Average SPAN | 2.2 | 24.6 | 1.9 | 1.3 | 60.2 |
| Group | *c* | *b* | *c* | *c* | *a* |

**Table 1: Test results for the method's intrinsic properties**

## 6. Data used to interpret the results

The Spans for products 2 and 5 are very high (Table 1). While they have small median diameters, they have far wider distribution spreads than the other three products.

Their distributions show a dichotomous population (Figure 2 and Figure 3). When using laser diffraction particle size analysis, this type of curve may have three different origins:

The effective presence of two populations of differing size

The formation of agglomerations of small particles

The presence of a single, rod-shaped particle population. The direction of observation of a rod-shaped particle (Figure 5), particularly one that is in motion, alters its size assessment when its surface area is projected, as it may appear as a sphere with a large or small diameter.

Examining the particles under the microscope, or using image analysis to assess particle size makes it possible to choose between these assumptions.

For instance, for product 2, the image analysis shows that the particles are rod-shaped.

This is not the case for product 5, which shows the formation of agglomerates rather than the presence of two particle populations.

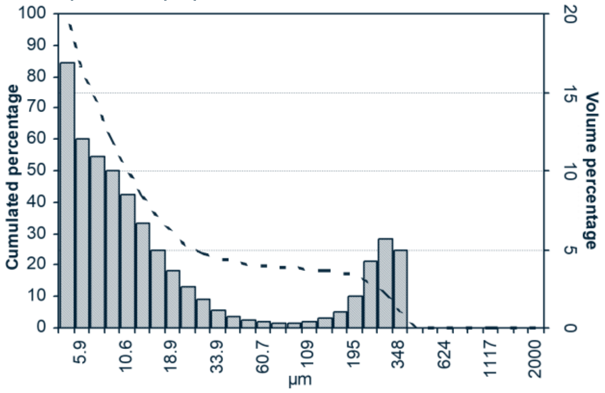


**Figure 2: Particle size spectrum of product 2**

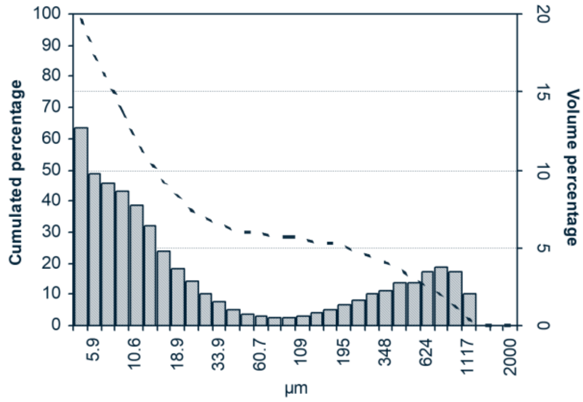


**Figure 3: Particle size spectrum of product 5**

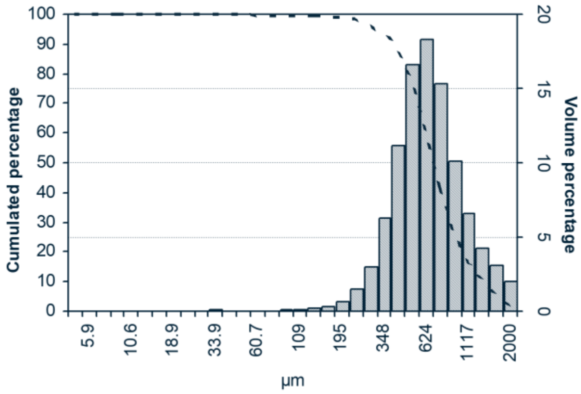


**Figure 4: Particle size spectrum of product 4**

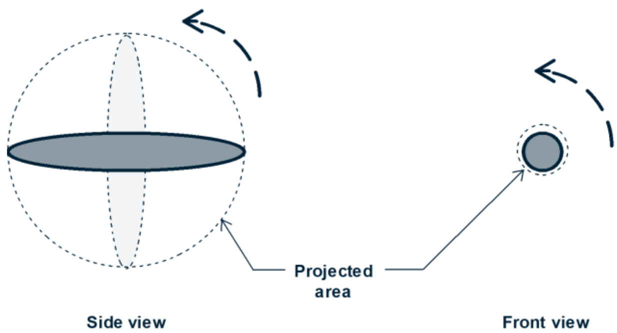


**Figure 5: Diagram of the variation in the projected area of elongated particles**

Conversely, as illustrated by product 4 (Figure 4), a product can be a mix of two substances of different sizes without revealing a dichotomous particle size distribution in the laser diffraction analysis. The particle size spectrum plots the particles' volume distribution according to size. When there is a large difference in size between the two particle populations, large particles take up a far greater volume than small particles. This volume percentage distribution thus masks the presence of the smaller particles.

## 7. Report on sifting results

This is a difficult comparison to make. It is likely that the median diameter given by this method will be greater than that obtained by sifting. The error between the two methods is compounded by the fact that the shape of the particles (especially large particles) varies from the sphere and, where raw materials are concerned, that the particles have varying densities, as demonstrated by Nathier-Dufour *et al*. (1993).

The main advantage in this laser method is its speed and the fact that it can be used with the finest-grained products.

## 8. Range of animal feed additives

The statistical results for the description of this measurement when applied to a range of 30 products chosen for their representativeness (Table 2) revealed:

A fairly fine-grained product population: 220 µm

A heterogeneous population (Figure 6). Most products (19 out of 30, i.e. 63.3%) lie in the 0-200 µm range, which explains the difference between mean and median values.

| | Median diameter $D_{50}$ µm |
|---|---|
| Mean | 224.2 |
| Standard deviation | 244.4 |
| Minimum | 7.4 |
| Maximum | 889.1 |
| Min./max. difference | 881.8 |
| Median | 122.8 |

**Table 1: Statistical reporting on the range of 30 representative products**

Most oligo-elements (6 out of 7) are found among products with a particle size of less than 200 µm. Out of the 11 products with a particle size greater than 200 µm, six are coccidiostatics and three are growth factors.

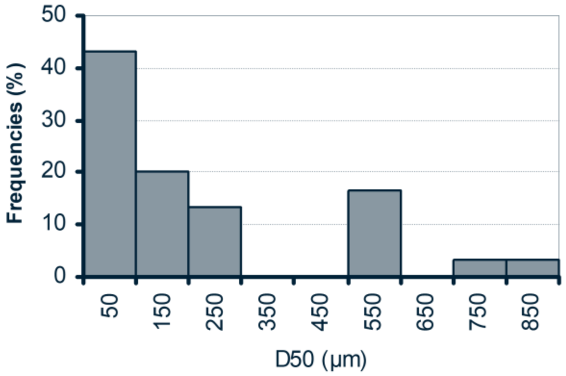


**Figure 6: Frequency bar chart**

The entire group of products sold without further processing is found within the group of products with particle size below 200 µm. The preparations generally have a particle size greater than 200 µm. These make up the vast majority: 7 out of 11.

They have a very wide range of between 7 and nearly 900 µm.

## 9. Conclusions

Overall, any assessment of this method should take account of its pros:

The method is sensitive, repeatable and accurate based on the median diameter.

The measurements are relatively simple and quick to take.

Particle size can be measured over a wide range using the same machine and the same protocol.

The particle size of most additives can be measured easily in air.

Reliable measurements can be taken at best cost.

and also its cons:

Clearly, the particle size data provided by this method only provides a partial image of the physical reality of the tested powders.

This is an expensive machine, which means it cannot be used at an industrial site, but has to be deployed at a central laboratory or research lab.

## 10. Bibliography


**Nathier-Dufour, N., Bougeard, L., Devaux, M.F., Bertrand, D., Le Deschault de Monredon, F., 1993.** Comparison of sieving and laser diffraction for the particle size measurements of raw materials used in foodstuff. Powder Tech., 76, 191-200

**Rapport Sypram** Phase 2a, 1998.