A 8 d - Particle Size Distribution by Laser (Malvern)

GEA Niro Method No. A 8 d

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1. Definition

A suspension of powder in isopropanol is measured with a low angle laser beam, and the particle size distribution is calculated.

2. Scope

This is a fast method for measuring particle size distribution of powders.

3. Principle

The method can be used on all powders containing less than 10% fat.

4. Apparatus

1. Malvern Instrument, Mastersizer Basic, equipped with software version B.0 or similar equipment.

2. Malvern QS Small Volume Sample Dispersion Unit.

3. Malvern in/out measuring cell, beam length 2.0 mm.

4. Dispenser 0-50 ml with container.

5. Filling knife.

6. Waste container.

5. Reagents

1. Isopropanol, IPA (technical quality).

6. Procedure

1. Look at the particle size in a microscope and choose a lens capable of measuring the largest particles, see Remarks 7.1.

2. Prepare the instrument for measuring in wet mode using IPA as the liquid, as described in the user manual.
The stirrer regulator should be set at 2000 rpm on the Malvern unit.

3. Measure the background for IPA.

4. Quickly add a sufficient amount of milk powder and measure as soon as the powder is dispersed and not later than 20 seconds after addition of the powder. For detailed instructions about measuring, see the Malvern user manual.

5. Rinse twice with IPA.

All measurements are made in duplicate

7.   Result

The following calculations are done automatically:

1.   The volume median diameter D(v,0.5) is the diameter where 50% of the distribution is above and 50% is below.

2.   Two determinations of mean particle size should not differ by more than 5% relative. The shape of the curves in the two determinations should be the same.

3.   D(v,0.9), 90% of the volume distribution is below this value.

4.   D(v,0.1), 10% of the volume distribution is below this value.

5.   The span is the width of the distribution based on the 10%, 50% and 90% quantile.

$$Span = \frac{D[v,0.9] - D[v,0.1]}{D[v,0.5]}$$

8.   Reproducibility

N/A

9.   Remarks

1.   The lens should be chosen according to the actual particle sizes. All lenses are capable of measuring a specific area of microns; therefore, ensure that the lens measures 100% of the largest particle. The software estimates the particles below the lens capacity, but cannot account for larger particles. The most precise results are obtained when the lens cuts off just above the largest particles because the estimate of the particles below is more precise.

2.   The obscuration is the amount of sample added to the system or more correctly the light intensity absorbed by the sample. The optimal obscuration is 0.1-0.3. The results are unreliable outside this interval.

3.   The powder particles should be insoluble in the dispersing agent (IPA).

4.   Fat filled products, containing more than 10% fat, and milk powder containing sugar cannot be measured by this method.

5.   D(4.3) is the equivalent volume mean diameter or the De Broncker mean diameter.

6.   D(3.2) is the equivalent surface area mean diameter or the Sauter mean diameter.

7.   Non-agglomerated milk powders will give the same mean diameter measured in wet or dry mode; whereas agglomerated powders will give considerably finer mean particle sizes when measured in dry mode due to wear of the particles.

8.   It is possible to use ultrasound in-line. The use of ultrasound affects the particles. Agglomerates and primary particles are separated and it is possible to measure the primary particle size.

10.   Literature

- GEA Niro Research Laboratory

   o   The Malvern Mastersizer Basic user manual.
       QS Small Volume Sample Dispersion Unit user manual.