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# Delft Solids Solutions

**Buro Blauw B.V.**  
**Mr Raoul van Onzenoort**  
**Nude 54**  
**6702 DN Wageningen**

Our reference 11-DSS-0271

Your reference Maltodextrine

Subject Particle size distribution

Date 21 July 2011

26 JULI 2011



Dear Mr Van Onzenoort

Please find enclosed the evaluation of the particle size distribution of your maltodextrine samples: Niro 1 voor cycloon, Niro 1 na cycloon, Niro 1 schouw, Niro 2 voor cycloon, Niro 2 na cycloon. The particle size distribution has been assessed using the laser diffraction technique. Below are a description of the technique, the results and a discussion of the results.

## ***Principles of Laser Diffraction***

Laser diffraction is an instrumental method for the characterization of particle size distributions (PSD) of dry powders, suspensions and emulsions. The technique is also known as (Near) Forward Light Scattering, Low Angle Laser Light Scattering or Fraunhofer Diffraction.

The particles are dispersed in liquid or air and circulated through a laser beam. The particles interact with the monochromatic laser light by scattering (diffraction, reflection, refraction and absorption). The scattered, mainly diffracted, light forms a pattern of concentric rings of alternating high and low intensities and is focused on the detectors by a (Fourier) lens.

The diffraction pattern is characteristic for particle size: small particles scatter under wide angles, large particles under small angles (the shape is not taken into account, the particles are presumed to be spherical). The way of scattering is independent of particle place and velocity within the laser beam.

An optical model is used for mathematical deconvolution of the diffraction pattern into a particle size distribution. Typically, two different models are used for deconvolution: the Fraunhofer and the Lorentz-Mie model, which both have specific advantages and limitations.

The Fraunhofer model does not take into account the optical characteristics of the material. It uses a standard relative refractive index (RI) which is the RI of the sample divided by the



RI of the medium and is mostly used when the optical characteristics of the sample are unknown or inhomogeneous (mixtures, coated materials, pores etc.). The true-ness of this model when applied to particles below 50  $\mu\text{m}$  can be doubtful when the RI of the material varies much from the used standard RI, although the precision (repeatability) is good.

The Lorentz-Mie model adequately takes into account all types of light interactions. As a consequence, this theory requires full knowledge of the optical properties of the particles and dispersion medium. The optical properties are expressed in a complex refractive index (ratio of RI of the particle to that of the medium), in which a real part represents the refractive properties of the particle and medium and an imaginary part the light absorption properties of the particle. These data cannot be measured easily and must be retrieved from handbooks and tables (the values of the RI are dependent on the wavelength of the used light; the given values cannot always be used as such). When the RI is known, the true-ness of the Lorentz-Mie model is best. The advantage of the Lorentz-Mie model is a greatly improved true-ness for particles below 10  $\mu\text{m}$ .

The principal result of the LD technique is a volume based PSD for a collective of spheres having defined optical properties, for which the calculated scattering behaviour gives an optimum match with the measured pattern.

### ***Equipment and methodology***

The particle size distribution via laser diffraction has been measured with a Malvern Mastersizer 2000 in a configuration from 0.02 up to 2000  $\mu\text{m}$  with Lorentz-Mie as optical model, in accordance with ISO 13320-1:2009. Three different units for dispersing the sample are available: the Scirocco 2000 sample dispersion unit with a venturi tube for dispersing in air, the Sample Presentation Unit with ultrasonic's and with a volume of 1L for dispersing in water and the Small Volume Sample Dispersion Unit with a volume of 100 ml for dispersing in organic dispersants.

The scirocco 2000 sample dispersion unit has been used for dispersion in air. The maltodextrine samples are measured at an optimum venturi pressure of 2 bar.

Each of the samples has been measured as an independent duplicate.

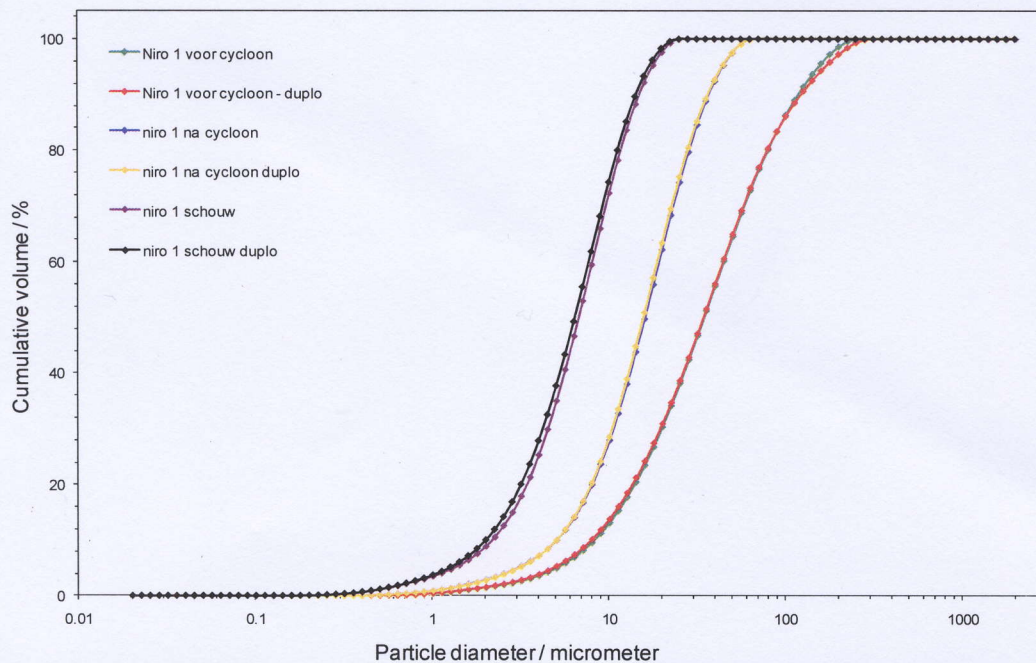
The optical model used has a Particle Refractive Index  $[n]$ /Absorption Index 1.52/0.1

### ***Results and discussion***

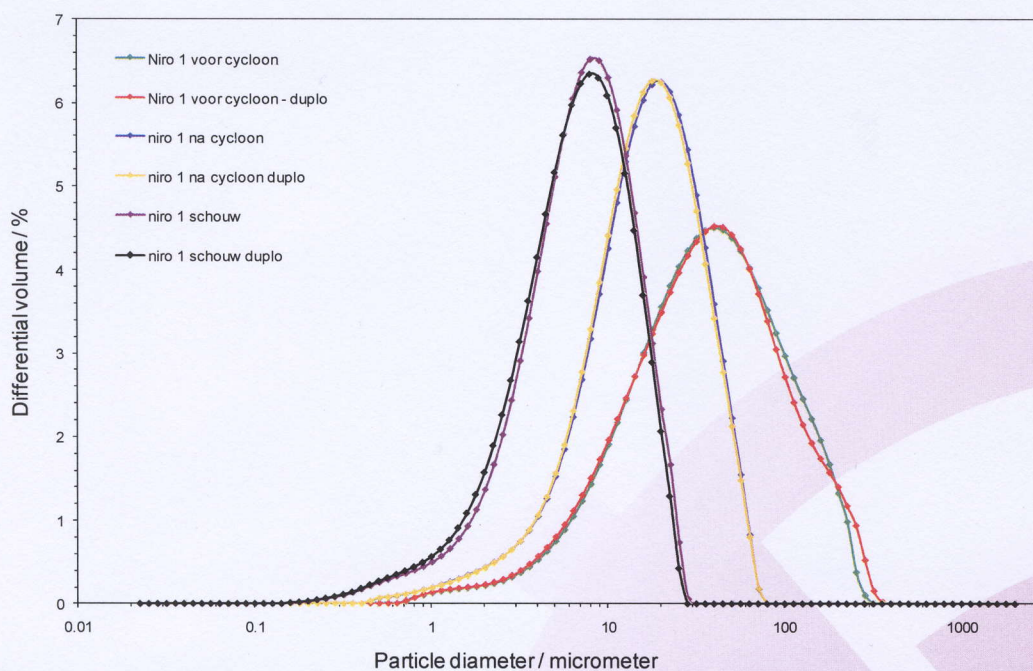
The samples collected on the filters are highly packed and contain agglomerates. The optimum venturi pressure is investigated for optimal de-agglomeration without breaking the primary particles. The measurements done with a venturi pressure of 0 and 1 bar weren't be able to fully de-agglomerate the samples, 3 and 4 bar venturi pressure seems to break the primary particles.

The data is summarized in figures 1a up to 2b and in table 1.



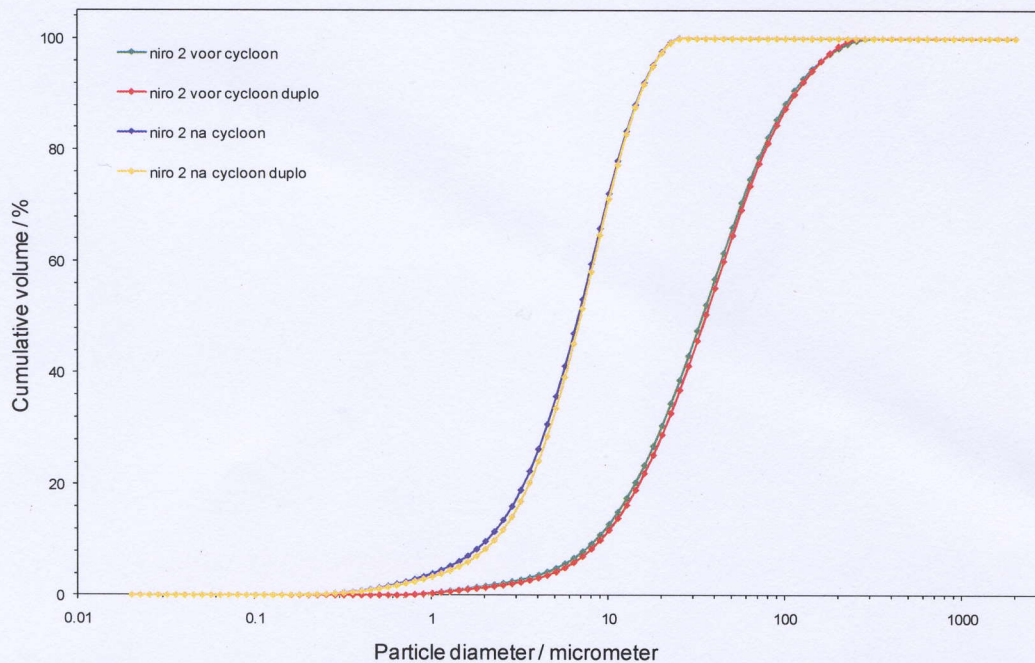


**Figure 1a.** Cumulative particle size distribution of the Niro 1 maltodextrine samples dispersed in air at 2 bar; (—) voor cycloon first measurement, (—) voor cycloon independent duplicate, (—) na cycloon first measurement, (—) na cycloon independent duplicate, (—) schouw first measurement, (—) schouw independent duplicate.

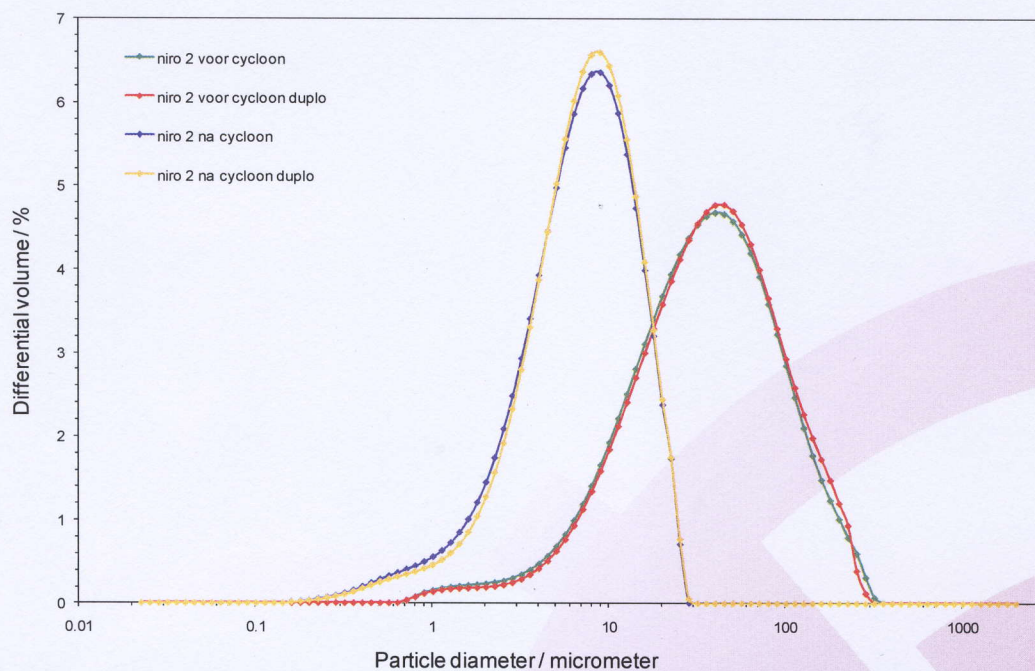


**Figure 1b.** Differential particle size distribution of the Niro 1 maltodextrine samples dispersed in air at 2 bar; (—) voor cycloon first measurement, (—) voor cycloon independent duplicate, (—) na cycloon first measurement, (—) na cycloon independent duplicate, (—) schouw first measurement, (—) schouw independent duplicate.





**Figure 2a.** Cumulative particle size distribution of the Niro 2 maltodextrine samples dispersed in air at 2 bar; (—) voor cycloon first measurement, (—) voor cycloon independent duplicate, (—) na cycloon first measurement, (—) na cycloon independent duplicate.



**Figure 2b.** Differential particle size distribution of the Niro 2 maltodextrine samples dispersed in air at 2 bar; (—) voor cycloon first measurement, (—) voor cycloon independent duplicate, (—) na cycloon first measurement, (—) na cycloon independent duplicate.



The table below quantitatively summarizes the particle size data.

**Table 1.** Summary of the particle size distribution data.

Sample Name	d (0.1) $\mu\text{m}$	d (0.5) $\mu\text{m}$	d (0.9) $\mu\text{m}$	D [4, 3] $\mu\text{m}$	Span	Mode 1 $\mu\text{m}$
Niro 1 voor cycloon	8.2	34.5	118.1	50.3	3.18	37.9
Niro 1 voor cycloon - duplo	7.9	34.2	122.2	51.7	3.34	39.4
<b>Average-N1 VC</b>	<b>8.1</b>	<b>34.4</b>	<b>120.1</b>	<b>51.0</b>	<b>3.26</b>	<b>38.6</b>
Niro 1 na cycloon	5.0	16.0	36.9	18.8	2.00	18.3
Niro 1 na cycloon duplo	5.0	15.6	36.4	18.5	2.01	17.6
<b>Average-N1 NC</b>	<b>5.0</b>	<b>15.8</b>	<b>36.7</b>	<b>18.6</b>	<b>2.00</b>	<b>17.9</b>
Niro 1 schouw	2.2	6.7	14.9	7.7	1.89	7.9
Niro 1 schouw duplo	2.0	6.4	14.3	7.4	1.92	7.7
<b>Average-N1 Schouw</b>	<b>2.1</b>	<b>6.6</b>	<b>14.6</b>	<b>7.6</b>	<b>1.91</b>	<b>7.8</b>
Niro 2 voor cycloon	8.4	33.7	108.9	48.6	2.98	38.7
Niro 2 voor cycloon duplo	9.0	35.2	112.8	49.8	2.95	40.2
<b>Average-N2 VC</b>	<b>8.7</b>	<b>34.5</b>	<b>110.9</b>	<b>49.2</b>	<b>2.96</b>	<b>39.4</b>
Niro 2 na cycloon	2.0	6.7	15.0	7.7	1.93	8.1
Niro 2 na cycloon duplo	2.3	6.9	15.1	7.9	1.86	8.1
<b>Average-N2 NC</b>	<b>2.1</b>	<b>6.8</b>	<b>15.0</b>	<b>7.8</b>	<b>1.89</b>	<b>8.1</b>

The definitions in the table are as follows:

d(0.1): Distribution Percentile, 10% of the particle volume is smaller than the table value.

d(0.5): Distribution Percentile, median particle size.

d(0.9): Distribution Percentile, 90% of the particle volume is smaller than the table value.

Span: Distribution width, calculated as  $d(0.9) - d(0.1) / d(0.5)$

D [4,3]: Volume weighted mean diameter  $(\sum n_i D_i^4) / (\sum n_i D_i^3)$ .

Mode: Modal size, particle diameter present with the highest frequency.

The above figures evidence the high repeatability of the laser diffraction technique and sampling, the particle size distribution curves nearly fully overlap. The sample “Niro 1 voor cycloon” has a mode at 39  $\mu\text{m}$ , the mode shift to 18  $\mu\text{m}$  for sample “Niro 1 na cycloon”, the sample “Niro 1 schouw” has the smallest mode at 8  $\mu\text{m}$ . The sample “Niro 2 voor cycloon” has a similar mode at 39  $\mu\text{m}$  like “Niro 1 voor cycloon”, and shift to 8  $\mu\text{m}$  for sample “Niro 2 na cycloon”.

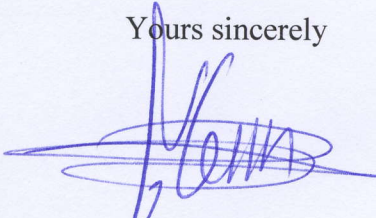


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We trust to have given you a complete and clear overview of the particle size measurements using the laser diffraction methodology and we hope that the results are in line with your expectations.

Should you have any questions, please do not hesitate to contact us.

Yours sincerely



G. Alfons