

IMA Metrology WG

Version 1

Date: April 2015

**Guidance for industrial minerals on the implementation of the European
Commission recommendation of 18 October 2011 on the definition of
nanomaterial (2011/696/EU)**

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Foreword

This document has been prepared by the Industrial Minerals Association, IMA-Europe (<http://www.ima-europe.eu>).

Introduction

This guidance describes in a standardised way the procedure to determine whether industrial minerals fall under the European Commission recommendation of 18 October 2011 on the definition of nanomaterial (2011/696/EU).

According to this definition, "nanomaterial" means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1nm-100 nm."

Evaluating bulk materials using this guidance will help to identify if the material needs to be considered as a nanomaterial. At this stage, the guidance is designed mainly for minerals not intentionally manufactured in the nano range. The result of the test will help to check compliance with the EC definition of nanomaterials and with some (national) laws.

This guidance describes techniques like dry sieving with a shaking table, laser diffraction and electron microscopy. These techniques can be used in combination for evaluation.

The results cannot be used for the evaluation of exposure to nanomaterials at the workplace.

1 Scope

This guidance specifies techniques which can be used for the evaluation of bulk materials considering the European Commission (EC) recommendation of 18 October 2011 on the definition of nanomaterial. It gives the ability to decide whether or not an industrial mineral falls under the scope of the EC definition.

The described techniques will help expressing the relative content of particles in bulk materials that, based on the number particle size distribution, should be considered as nanomaterials. At this stage, the guidance is designed mainly for minerals not intentionally manufactured in the nano range.

2 Normative references

ISO 13317-1: 2001 Determination of particle size distribution by gravitational liquid sedimentation methods — Part 1: General principles and guidelines.

ISO 13317-2: 2001 Determination of particle size distribution by gravitational liquid sedimentation methods — Part 2: Fixed pipette method

ISO 14887: 2000 Sample preparation – Dispersing procedures for powders in liquids

ISO 29301: 2010 Microbeam analysis – analytical transmission electron microscopy – methods for calibrating image magnification by using reference materials having periodic structures

ISO 9276-1 to -6: Representation of results of particle size analysis

ISO 13320: 2009 Particle Size analysis – laser diffraction methods

ISO/DIS 3310-1: 2014 Test Sieves Technical requirements and testing – Part 1: Test sieves of metal wire cloth

ISO 565: 1990 Test sieves; metal wire cloth, perforated metal plate and electroformed sheet; nominal sizes of openings

ISO 2591-1: 1988 Test sieving Methods using test sieves of woven wire cloth and perforated metal plate

3 Terms and definitions

In this industry guidance, the terms and definitions given in the EC recommendation of 18 October 2011 on the definition of nanomaterial apply.

“nanomaterial” means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1nm-100 nm.”

See annex B for a detailed description of the term ‘number size distribution’.

4 Techniques for evaluation

Annex A contains a list of techniques not to use.

4.1 Screening of coarse and medium sized products by sieving

In fine products, the potential occurring nanoparticles can be measured with modern laser diffraction devices (cf. chapter 4.2 and 5.2). Since even coarse and medium sized products can possibly contain nano sized particles in their fines, an easy and quick investigation by sieving with a sieving machine is recommended. The main purposes of this sieving is to:

- 1) Check if there is a relevant content of fine fraction which could possibly contain nanoparticles
- 2) Separate the fine fraction for further analysis by laser diffraction and by electron microscopy (SEM/TEM).

4.2 Technical requirements to determine the number size distribution by laser diffraction using blue light source

Characterise the particle size distribution as best as possible with currently available equipment. The best equipment should have the shortest wavelength available (currently known as blue source).

To cover the whole size distribution range, equipment is required which employs both short (around 400 nm) and long (around 700 nm) wavelengths.

To determine the number size distribution, including small particles in the nano range, the instrument should be equipped with a light source (e.g. blue laser or light emitting diode (LED)) with sufficient wavelength and the range of analysis of the equipment should extend to below 0.1 µm.

Care should be taken on the preparation of the sample and the dispersion conditions of the material which are described in the annexes C to K for each mineral.

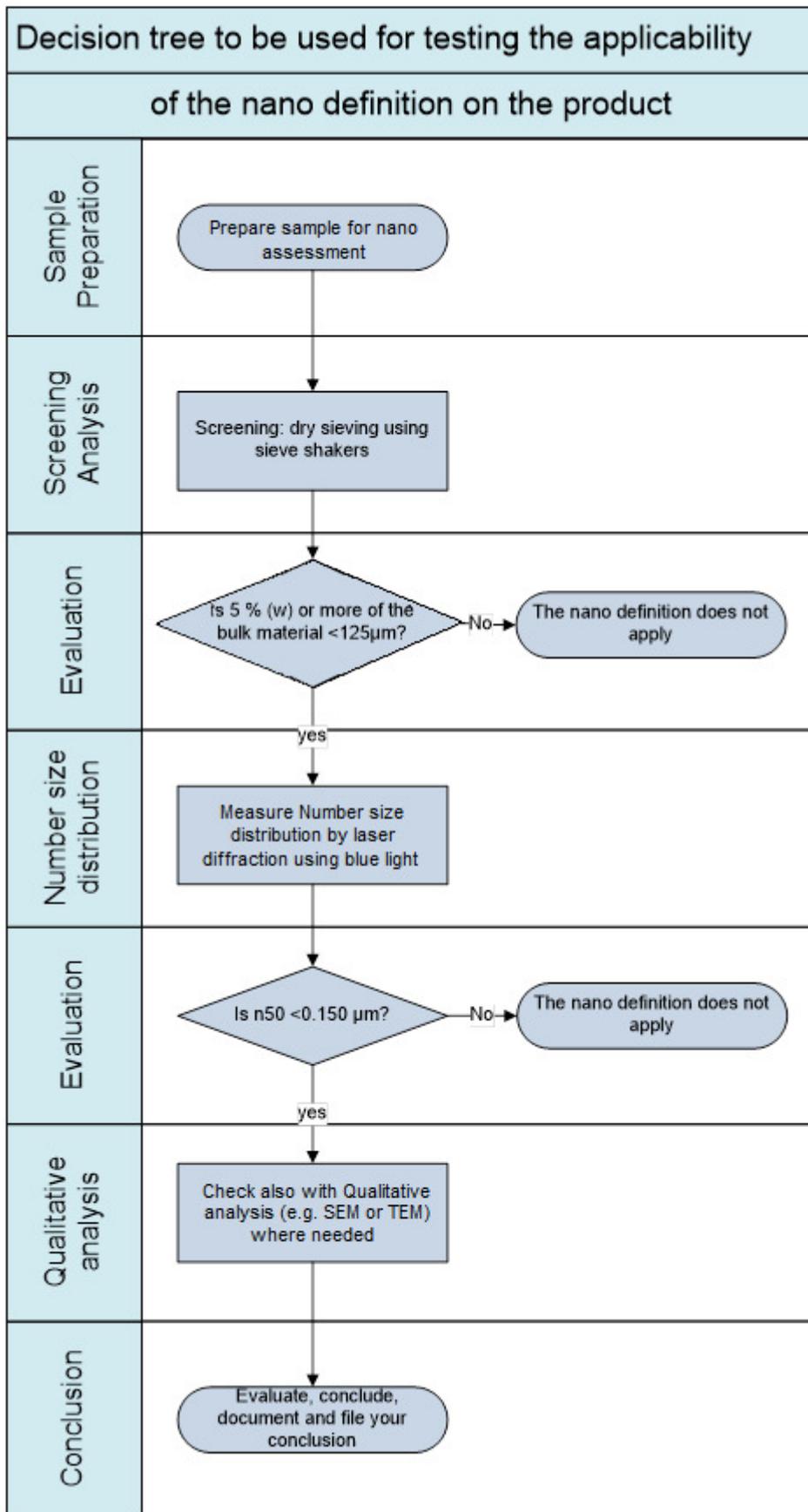
The particle size analysis by laser diffraction is well described in the standard ISO 13320:2009.

4.3 Electron Microscopy

Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) can be used to visualize the particle size, shape and agglomeration/aggregation status. Although electron microscopy is a very useful visualisation tool, it is not suitable for fully quantitative particle size measurement of particulate material with broad size distribution (above 3-4 orders of magnitude).

5 Method for the implementation of the EC recommended definition of nanomaterial on bulk materials

To determine if certain materials will need further investigation for the implementation of the EC recommended definition of nanomaterial, the first step consists of a screening of the material by sieving. Following internal investigations within IMA-Europe on a wide range of powders from the mineral industry, it has been concluded that if less than 5 mass % of the bulk material is below 125 µm, the material does not need further investigation. If this is not the case, the material will be measured by laser diffraction and complementary qualitative information from electron microscopy to evaluate if the definition applies to the material.



For micronised materials with a d_{97} below 125 μm , clause 5.1 is not applicable and clause 5.2 directly applies.

5.1 Screening by sieving: separation of the fine fraction

The used sieving procedure depends on the kind of product analysed and its grain size distribution. As lower limit for sieving, a nominal size of 0.125 mm is recommended since it is a common mesh size in the mineral industries to determine fines. If there is a relevant fine fraction for the nanomaterial, the passing fraction will be of sufficient quantity to be easily handled for further investigation by laser diffraction and SEM/TEM.

Coarse products (> 4mm)

In the case of some coarse products it may be necessary to split the very coarse part from the finer fraction (by hand sieving) before further sieving with a sieve machine. By doing so, the fine fraction will be enriched and a better statistical evaluation can be achieved.

Medium size particles (0.2 - 4mm)

For products with a particle size distribution from 0.2 to 4 mm, usually a one-step sieving with a dry sieve machine is sufficient.

Usually the material should be in a dry and flowable condition for analysis. The dry-sieve screening is performed by the use of a (vibratory) sieve shaker. If you dispose of a reliable standard operation procedure (SOP) to estimate and select the passing of a 0.125 mm sieve, you can use your SOP.

If this is not the case, a general procedure for dry-sieve screening using a (vibratory) sieve shaker is described below for guidance (cf. ISO 2591-1):

Selection of test sieves

Select a set of test sieves with mesh size between 0.125 mm and the maximum grain size of the material. The number of sieves used in the test should be sufficient to split the sample in a way that the permitted maximum volume after sieving is not exceeded. The mesh sizes should follow ISO 565. Use a dry sieve pan (without an outlet) to collect the fines (< 0.125 mm) for further investigation. A dry sieving head should also be used to avoid sample loss on the top sieve.

Determination of the initial weights of the bulk material

The acceptable initial weight of the bulk material depends on the range of grain sizes and the bulk density of the material. If this is not known from previous examinations, the initial weight of charge has to be estimated in a pre-test. The advised restrictions of ISO 2591-1 are listed in the table below.

Nominal aperture size	Bulk volume of material		Nominal aperture size	Bulk volume of material	
	Approximate volume of charge	Maximum volume of residue		Approximate volume of charge	Maximum volume of residue
[mm]	[cm ³]	[cm ³]	[mm]	[cm ³]	[cm ³]
22.4	1600	800	1.400	160	80
16.0	1000	500	1.000	140	70
11.2	800	400	0.710	120	120
8.0	500	250	0.500	100	100
5.6	400	200	0.355	80	80
4.0	350	175	0.250	70	70
2.8	240	120	0.180	60	60
2.0	200	100	0.125	50	50

Table 1: Guide to quantify the material for test sieving on a 200 mm diameter round sieve. The masses of materials can be determined by multiplying the specified volume values by the apparent bulk density, in grams per cubic centimetre, of the material to be sieved (ISO 2591-1:1988).

Sieving time

The sieving time has to be estimated by a pre-test and is defined by the sieving endpoint. The dry sieving endpoint for non-friable materials is reached when the passing through the sieve(s) in 1 minute is less than 1% by weight.

To shorten the sieving time, sieving aids can be placed in every sieve with mesh sizes larger than 125µm. During sieving, the balls impact on the sieved and accelerate the removal of the substance being sieved. The following can be used as sieving aid:

- Agate balls: 10 mm Ø: 10 balls per sieve, or
- Rubber balls: 20 mm Ø: 5 balls per sieve

Amplitude of vibratory sieving

The adjustable amplitudes of vibratory sieve machines usually range from 0 up to 3 mm. To achieve the target amplitude of 3 mm (full amplitude), at least 3 sieves in addition to the collecting vessel and a sieve cover must be fastened to the sieve shaker. If fewer sieves are used, it may not be possible to achieve the target amplitude of 3 mm (full amplitude). In this case, the regulation of the sieve shaker can no longer attain the optimal operating point; the sieve shaker will vibrate at a lower amplitude and frequency. The target amplitude must in this case be lowered accordingly. Once the machine has warmed up, the target value can under some circumstances be increased accordingly.

Typical values of sieving parameters estimated by the above mentioned guidance are listed in Table 2.

Product type	Coarse	Medium	Fine
PSD of main fractions	4 to > 100 mm	0.2 to 4 mm	< 0.2 mm
Set of Sieves mesh size in mm	4 / 5.6 / 8 / 16 / 22.4 31.5 / 45 / 63 / 90 / 125	0.125 / 0.18 / 0.25 0.355 / 0.5 / 0.71 / 1 1.4 / 2 / 2.8 / 4	use laser diffraction
Initial weight of bulk material	> 200 g	50 to 200 g	
Sieving time	3 to 20 min	5 to 30 min	
Amplitude	2.5 to 3	1 to 2.5	

Table 2: Typical ranges of the above mentioned sieving parameters. The division in coarse, medium and fine follows the classification of T. Allen, 1993

Due to electrostatic and cohesive properties, especially in bulk materials containing a high portion of fines, a wet method can be used for removal of the fines. Afterwards, the fines (< 0.125 m) of the material have to be collected in a vessel during sieving with a wet-sieving machine or by hand sieving.

Fine particles (<0.2 mm)

If the product has a maximum grain size below 0.2 mm, it can directly be measured by laser diffraction.

Evaluation of sieving

If more than 5% by weight of the bulk material is below 125 µm, the separated fine material needs further investigation by laser diffraction (and SEM/TEM). Keep in mind that the result of laser diffraction must be related to the whole bulk material.

5.2 Measuring the number size distribution by laser diffraction

The particle size distribution as measured by laser diffraction can be calculated based on several models, most often as a volume/mass but also as a number distribution. The EC recommended definition is expressed in number size distribution which is not commonly used in the mineral industry but can be recalculated out of the volume/mass particle size distribution obtained from laser diffraction assuming that a particle is spherical.

The procedure to follow for measuring the number size distribution by laser diffraction using blue LED is described below:

- For sample preparation, optimum dispersion conditions should be taken into account and details are provided in the annexes (C. to K.) for each individual mineral.
- Measure the particle size distribution using the **ISO 13320:2009**.
- Use the Mie theory:
Perform measurements at different particle concentrations to determine the optimal concentration (obscuration) range in order to obtain an acceptable signal-to-noise and to avoid multiple scattering. This concentration must be of maximal 5%. Smaller the particles are, lower the mass concentration (obscuration) must be. In any case the weighted residual must be below 1.5 %
Information on the Refractive index can be found in ISO 13320:2009 Annex D
- Determine the number size distribution of the material using the software;
- Check if the n_{50} is $< 0.150\mu\text{m}$. If yes, your material needs further investigation. Go to clause 5.3 for qualitative analysis.
- If not, then the material is not considered as falling under the EC recommended definition.

NOTE: Although the definition indicates 100 nm, we strongly recommend a cut-off point of around 150 nm as a safety margin for error.

NOTE: At this stage, no indication is given on the following parameters of the equipment, but care should be taken that they can influence the results: obscuration level/light intensity, model used for distribution (general or single model), shape of the particles, weighted residual/goodness of fit, pump speed.

5.3 Qualitative analysis by Electron Microscopy (SEM or TEM)

Samples come in all types, shapes and sizes. Therefore, techniques have to be found to cope with all variations and deviations from the perfect sample, a naturally conducting regular solid providing high emission of electrons.

There are many ways in which a sample can be prepared for electron microscopy. The exact choice of sample preparation would be determined by the type of mineral.

The results of the electron microscopy can be used to qualitatively validate the laser diffraction results.

Annexes

A- Evaluation techniques identified as not applicable for the quantitative determination of nanomaterials in industrial minerals

Techniques	Comments
STEM - Scanning Transmission Electron Microscopy	Limit of detection is <0.1 nm resolution Low concentration at ppm level and is therefore not representative for bulk material
TEM - Transmission Electron Microscopy	Sample preparation (HVac) The technique is not applicable for minerals with a broad size distribution, and not widely available in the mineral industry
AFM - Atomic Force Microscopy	The technique might be applicable for intentionally manufactured nanomaterials and not for broad particle size distributions like in the mineral industry
STM - Scanning Tunneling Electron Microscopy	The technique might be applicable for intentionally manufactured nanomaterials and not for broad particle size distributions like in the mineral industry
FFF - Field Flow Fractionation	The technique is restricted to sizes from 1 nm to 1 µm for the Flow FFF, and from 50 nm to 1 µm for Sed FFF and not relevant for broad particle size distributions like in the mineral industry
HDC - Hydrodynamic Chromatography	The technique is restricted to sizes from 5 nm to 1200 nm and not relevant for broad particle size distributions like in the mineral industry
SEC - Size Exclusion Chromatography	The technique is widely used for polymer characterisation and is not relevant for minerals
CFF - Cross-Flow Filtration	The technique is restricted to sizes from 7 nm to 2 µm and not relevant for broad particle size distributions like in the mineral industry
UC - Ultracentrifugation	This technique is more qualitative than quantitative and does not measure number size distribution.
CFUF - Cross Flow Ultrafiltration	The technique is restricted to sizes from 1 nm to 1 µm and not relevant for broad particle size distributions like in the mineral industry
SPMS - Single Particle Mass Spectrometry	The technique is restricted to sizes from 3 nm to 1 µm and not relevant for broad particle size distributions like in the mineral industry
SAXS - Small-Angle X-ray Scattering	The technique is restricted to sizes from 5-25 nm and not relevant for broad particle size distributions like in the mineral industry
UCPC - Ultrafine Condensation Particle Counter	The technique is restricted to sizes from 2-10 nm and not relevant for the definition as it is used for measuring exposure at the workplace

XRD	This technique is used to determine the crystal structure and is more qualitative than quantitative and cannot be used for number size distribution. Nevertheless, XRD is a standard technique to check whether nanoforms have other diffraction patterns as compared to the bulk material.
SMPS - Single Particle Mass Spectrometry	The technique is restricted to sizes from 3 nm to 1 µm and not relevant for the definition as it is used for measuring exposure at the workplace
DLS – Dynamic Light Scattering	The technique is not relevant for polydispersed broad particle size distributions like in the mineral industry
DMA - Differential Mobility Analyzer	The technique is not relevant for the definition as it is used for measuring exposure at the workplace
FIFFF - Flow Field-Flow Fractionation	The technique is restricted to sizes from 1 nm to 1 µm, is mostly used for environmental and biological samples and is not relevant for minerals

B - Detailed description of number particle size distribution

The easiest way to understand a number distribution is to consider measuring particles using a microscope. The observer assigns a size value to each particle inspected. This approach builds a number distribution—each particle has equal weighting once the final distribution is calculated. As an example, consider the nine particles shown left in Figure 1. Three particles are 1 μm , three are 2 μm , and three are 3 μm in size (diameter). Building a number distribution for these particles will generate the result shown on the right part of Figure 1, where each particle size accounts for one third of the total. If this same result were converted to a volume distribution, the result would appear as shown in Figure 2 where 75 % of the total volume comes from the 3 μm particles, and less than 3 % comes from the 1 μm particles.

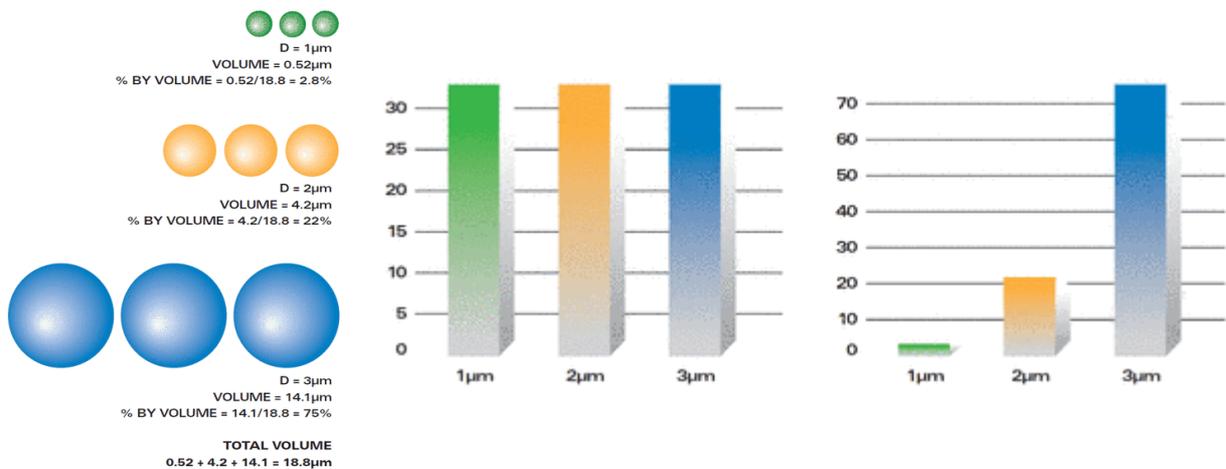


Figure 1: Particles of 1, 2, and 3 micron (left) with their Number- and volume distributions (right)

When presented as a volume distribution it becomes more obvious that the majority of the total particle mass or volume comes from the 3 μm particles. Nothing changes between the left and right graph except for the basis of the distribution calculation.

Another way to visualize the difference between number and volume distributions is supplied by courtesy of the City of San Diego Environmental Laboratory. In this case beans are used as the particle system. Figure 2 shows a population where there are 13 beans in each of three size classes, equal on a number basis. The same figure shows these beans placed in volumetric cylinders where it becomes apparent that the larger beans represent a much larger total volume than the smaller ones.

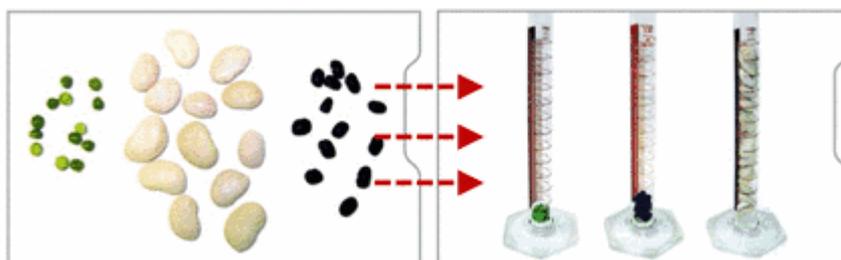


Figure 2: (Click to enlarge) Equivalent number of beans

Figure 3 shows a population of beans where it may not be intuitively obvious, but there is an equal volume of each size, despite the wide range of numbers present. It becomes apparent that when the beans are placed in volumetric cylinders that each volumes are equal.

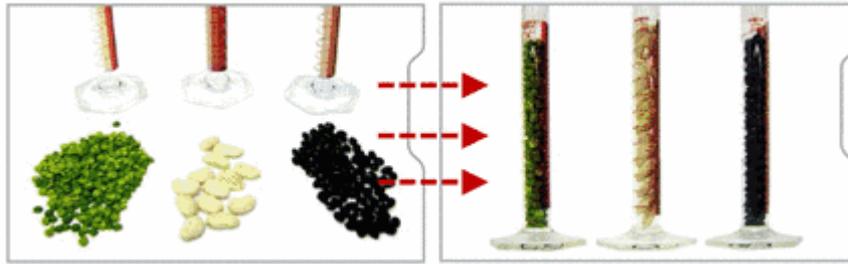


Figure 3: Equivalent volume of beans

Transforming Results

Results from number based systems, such as microscopes or image analyzers construct their beginning result as a number distribution. Results from laser diffraction construct their beginning result as a volume distribution. The software for many of these systems includes the ability to transform the results from number to volume or vice versa. It is perfectly acceptable to transform image analysis results from a number to volume basis. In fact the pharmaceutical industry has concluded that it prefers results be reported on a volume basis for most applications (ref. 1). On the other hand, converting a volume result from laser diffraction to a number basis can lead to undefined errors and is only suggested when comparing to results generated by microscopy. Figure 4 below shows an example where a laser diffraction result is transformed from volume to both a number and a surface area based distribution. Notice the large change in median from 11.58 μm to 0.30 μm when converted from volume to number.

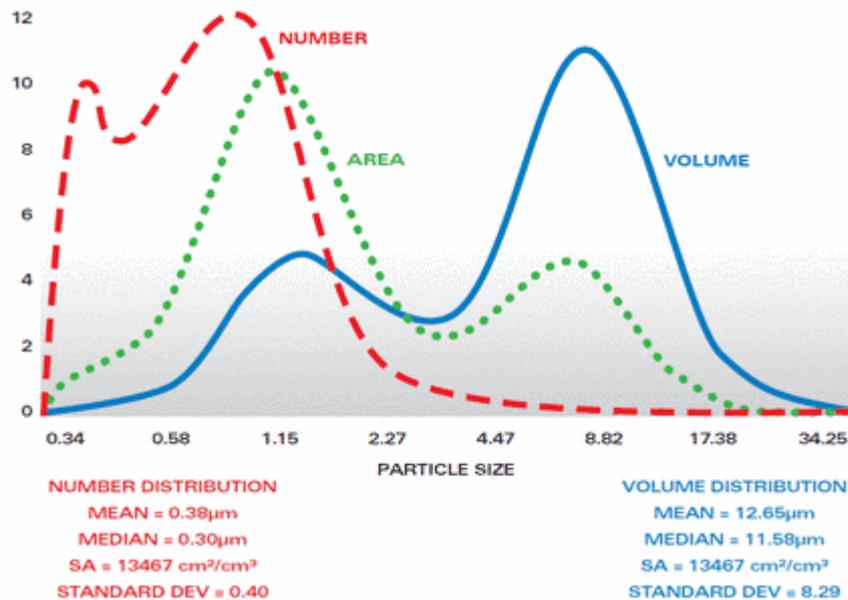


Figure 4: A volume distribution converted to area and number will include conversion errors

Source for Annex B: <http://www.horiba.com/it/scientific/products/particle-characterization/education/general-information/data-interpretation/number-vs-volume-distributions/>

C - Dispersion conditions for quartz and cristobalite products

C.1 Scope

This instruction applies to quartz and cristobalite products to prepare the sample for laser diffraction.

C.2 Equipment

Standard laboratory glassware and the following supplies:

- 1) Glass beaker
- 2) Demineralised (deionised) water
- 3) Analytical balance with an accuracy of 0.001 grams
- 4) Ultrasonic bath or shaker.
- 5) Dispersant or deflocculant, when required

C.3 Preparation of the sample

The following steps should be executed to achieve optimum dispersion conditions of a sample:

- 1) Take a sample of the material of approx. 5.0 g.
- 2) Determine the weight M of the sample with a precision of 0.001 g.
- 3) Disperse the sample in 50 ml of sedimentation liquid in a 100 ml pre-weighed, dry and clean beaker. The weight of the beaker should also be determined with a precision of 0.001 g.
- 4) Treat the sample in an ultrasonic bath or a shaker until completely deagglomerated.
- 5) If necessary, add a suitable dispersant or deflocculant to keep the particles from flocculating or coagulating.
- 6) When using a dispersant or deflocculant this should be clearly identified in your report on the results.

C.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP).

Mie parameters:

	Real part (n_p)	Imaginary part (k_p)
Quartz	1.54	0.1
Cristobalite	1.487	0.1

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

D - Dispersion conditions for Kaolins and Kaolinitic Clays

D.1 Scope

This instruction applies to most kaolins and kaolinitic clays and may be adapted for other coarse to fine-grained materials for which water is generally a good medium for separation by sedimentation.

Water is a suitable separation medium provided the following conditions are met:

- The particles in the sample must be completely de-agglomerated
- The particles in the sample should not dissolve, swell or disintegrate
- The particles in the sample should not react

The method is therefore not particularly suitable to bentonite type minerals or kaolins / kaolinitic clays that contain significant bentonite or other swelling minerals.

D.2 Equipment

This method requires standard laboratory equipment and some additional items as described below.

Beakers (preferably glass) or similar containers to hold ~ 3 litres by volume

Water bath with temperature control (+/- 1°C) to accommodate the 3 litre glass beakers (a bath that holds 3-4 beakers gives a good compromise between taking up too much bench space and adversely affecting sample throughput).

Demineralised (deionised) water

Analytical balance with an accuracy of 0.001 g

Ultrasonic bath or shaker

Dispersant – e.g. sodium hexametaphosphate (Calgon), sodium polyacrylate etc., when required

Dilute (~10 %) Sodium carbonate solution for pH adjustment

Dilute (~10 %) Sulphuric acid for pH adjustment

D.3 Preparation of the sample

Weigh out on a clean and dry dish or weighing boat approximately 40 g of the sample material to be analysed with a precision of 0.001 g.

Add approximately 300 ml of deionised water (ideally - that has been pre-heated to 25 °C in a beaker in the water bath) to a 600 ml clean glass beaker and then add dispersant if desired at an appropriate dosage for the type / particle size range of the mineral to be sedimented. The optimum dose of dispersant will depend on the particle size distribution (surface area) of the material under test. The finer (higher surface area) materials will require a higher dosage of dispersant. Typically, for kaolins, a dispersant dose of between 0.1 wt.% and 0.3wt.% on the equivalent dry-weight should suffice. E.g. 6g (mls) of 1 wt.% dispersant on 40 g dry-weight of kaolin would be equivalent to 0.15wt % active dispersant.

Carefully add the pre-weighed sample material into the water/dispersant and gently mix until fully wetted-out. Ensure that the entire sample is transferred to the mixture by either brushing or washing out with deionised water. Full dispersion of the sample may then be achieved by using an ultrasonic probe or placing the beaker of slurry into an ultrasonic bath for a specified time. The pH of the slurry should also be checked and adjusted as necessary by the addition of a few drops of sodium carbonate solution (to raise pH) or dilute sulphuric acid (to lower pH). The optimum pH will depend on the dispersant type used, e.g. for sodium hexametaphosphate (Calgon) a pH of around 7 is optimum. If a polyacrylate dispersant is used then a higher pH around 9 – 9.5 is preferred.

D.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction or capable to identify nano particles, using your standard operation procedure (SOP).

Mie parameters:

	Real part (n_p)	Imaginary part (k_p)
Kaolin / Calcined Kaolin	1.52	0.1

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

E - Dispersion conditions for other Minerals which may be treated in a similar way to Kaolins / Kaolinitic Clays

E.1 Scope

The general methodology developed for kaolins and kaolinitic clays may also be used for the following minerals: Andalusite, Mica, Vermiculite and Talc.

E.2 Equipment

Standard laboratory glassware and the following supplies:

1. Glass beaker
2. Demineralised (deionised) water
3. Beakers (preferably glass) or similar containers to hold ~ 3 litres by volume
4. Analytical balance with an accuracy of 0.001 grams
5. 1 mm screen
6. Oven with an automatic temperature control set to ~ 100° C
7. Magnetic stirrer
8. Ultrasonic bath
9. Dispersant or deflocculant: Triton X100, Calgon (Sodium metaphosphate) or Sodium polyacrylate (e.g. 0.1% Daxad 30), when required

E.3 Preparation of the sample

E.3.1 Andalusite

Andalusite is a blocky, coarse alumina-silicate mineral but some products may contain fine particles. Generally, Andalusite products are very coarse (> 1 mm) and the procedure described below should be followed.

1. Take approximately 100 g of dry product and accurately weigh to 2 decimal places (weight A). Water-wash the 100 g of product through a 1 mm screen and collect the < 1 mm fraction (the portion passing through the screen) and the > 1 mm fraction (the portion remaining on the screen) separately.
2. Dry, to constant weight, the >1 mm fraction in an oven at ~ 100° C (weight B).
3. Calculate the weight percentage of > 1 mm fraction (weight% C).
Weight % of >1 mm fraction C = weight B/weight A x 100%
4. If the weight % of > 1 mm is greater than 99.00 then no further action is required. (If weight % >1 mm is > 99.00 then wt.% < 1 mm will be <1 wt.%).
5. If the weight % of > 1 mm is less than 99.00 then screen sufficient product to carry out a sedimentation analysis as detailed for kaolins and kaolinitic Clays.

E.3.2 Mica

Mica is a platy mineral with similar particle shape to kaolins but generally a coarser particle size. For very coarse mica products an initial pre-screening should be carried out to determine whether the full sedimentation procedure is required (see C.3.1 Andalusite).

E.3.3 Vermiculite

Vermiculite, in its unexpanded form, is another platy mineral similar to a mica and kaolin but even coarser particle size. Because of the coarse nature of Vermiculite, especially when expanded, it should be given the same pre-screening process as for coarse micas and Andalusite (see C.3.1 Andalusite).

E.3.4 Talc

Talc is a platy mineral, it exhibits some degree of hydro-phobicity which means that a suitable surfactant should be added to the talc / water suspension in order to thoroughly wet-out the mineral particles and give a fully dispersed and deflocculated suspension.

Add 2 ml of Triton X100 to 500 mg of Calgon (Sodium metaphosphate) or Sodium polyacrylate in a 2000 ml beaker, complete to 2000 ml with demineralised water and mix it during 45 min using a magnetic stirrer.

- a) Take a sample of the material to be analysed of approx. 5 g
- b) Determine the weight M of the sample to be analysed with a precision of 0.001 g.
- c) Disperse the sample in 80 ml of dispersant in a 175 ml dry and clean beaker.

NOTE: To avoid the presence of foam, introduce the demineralised water and the dispersant in several steps in order to obtain a “pastry” and then a “liquid mixture”.

- d) Place the sample in an ultrasonic bath during 4 minutes.

E.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP).

Mie parameters:

	Real part (n_p)	Imaginary part (k_p)
Andalusite	1.648	0.000
Mica	1.52	0.1
Vermiculite	1.54	0.01
Talc	1.589	0.1

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

F - Dispersion conditions for Diatomaceous Earth (DE)

F.1 Scope

This instruction applies to diatomaceous earth (DE) products to prepare the sample for laser diffraction. The DE are classified in three categories of product: natural, calcined and flux-calcined.

F.2 Equipment

1. Particle size analyser (blue laser) according to ISO ... (laser diffraction)
2. Demineralised (deionised) water
3. Ultrasonic bath or shaker

F.3 Preparation of the sample

Particle size distribution of the sample is determined by laser diffraction, in liquid (demineralised water), with a pre-sonication during 20s

F.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP) and the following Mie model parameters:

	Real part (n_p)	Imaginary part (k_p)
Diatomaceous Earth	1.54	0.01

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

G- Dispersion conditions for feldspar products

G.1 Scope

This instruction applies to coarse to fine-grained material samples (maximum 100 µm), for which water is generally a good medium for separation by sedimentation.

Water is suitable as a separation medium provided the following conditions are met:

- the particles in the sample must be completely de-agglomerated
- the particles in the sample should not dissolve, swell or disintegrate
- the particles in the sample should not react
- e.g. clays or cement should not be analysed in water

G.2 Equipment

Standard laboratory glassware and the following supplies:

- 6) Jar and glass beaker
- 7) Demineralised (deionised) water
- 8) Sodium hexametaphosphate (NaPO_3)₆
- 9) Analytical balance with an accuracy of 0.001 grams
- 10) Ultrasonic bath or shaker.
- 11) Desiccator
- 12) Oven with an automatic temperature control set to 103 ± 5 °C

G.3 Preparation of the sample

Weigh in a sample jar approximately 5 g of the sample material to be analysed with a precision of 0.001 gram.

Prepare a small glass beaker of approximately 50 ml capacity by heating in the oven at 103 ± 5 °C for 1 hour. Let the glass beaker cool down for 15 minutes in a desiccator and weigh it to within 0.001 gram accuracy.

Add approximately 20 ml demineralised water to the sample jar to prepare a slurry. **Add 2.5 ml of sodium hexametaphosphate 50g/l (0.05%)**. Place the sample jar in an ultrasonic bath for about 5 minutes, or for 1 hour in a shaker.

G.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP).

Mie parameters:

	Real part (n_p)	Imaginary part (k_p)
Feldspar	1.4900	0.0000

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

H - Dispersion conditions for Calcium carbonate

H.1 Scope

This instruction applies to fine-grained calcium carbonate powder products to prepare the sample for laser diffraction.

H.2 Equipment

4. Demineralised (deionised) water
5. Ultrasonic bath or shaker
6. Analytical balance with an accuracy of 0.001g
7. Sodium pyrophosphate (Na₄O₇P₂, purity \geq 95%, CAS-No. 7722-88-5)
8. Photo-Flo (Kodak CAT 146 4510), when required (for coated material)
9. Erlenmeyer plastic flask 200 ml
10. High-speed blender (e.g., Polytron PT 3100)

H.3 Preparation of the sample

The following steps should be executed to achieve optimum dispersion conditions of a **fine-grained uncoated calcium carbonate powder** sample:

1. Water down 8 g powder with 100 ml sodium pyrophosphate (Na₄O₇P₂, purity \geq 95%, CAS-No. 7722-88-5) solution 0.1% in demineralised water (m/v)
2. Disperse sample by means of a high-speed blender (e.g., Polytron PT 3100) at 10'000 rpm for about 3 min in a plastic Erlenmeyer flask 200 ml
3. Place the sample for 10 min in an ultrasonic bath (approx. 90 W)

Whenever possible, it is recommended to avoid coated products and use the uncoated equivalent instead.

However, for a **fine-grained coated calcium carbonate powder sample**, the following steps should be executed to achieve optimum dispersion conditions:

1. Remove the surface treatment thermally in a muffle furnace at 430°C for 1 hour prior to analysis. The samples having their surface coating thermally removed should then be analyzed following the procedure for uncoated samples described above.

H.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles; using your standard operation procedure (SOP).

Mie parameters:

	Real part (n_p)	Imaginary part (k_p)
Calcite	1.49-1.74	No value
Aragonite	1.53-1.69	No value
Carbonate / Dolomite	1.596	0.1

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

I - Dispersion conditions for Lime

I.1 Scope

This instruction applies to lime products to prepare the sample for laser diffraction.

I.2 Equipment

11. 250 ml cylinder
12. Analytical balance with an accuracy of 0.001g
13. Ultrasonic bath or shaker
14. 20 ml isopropanol (the used isopropanol has to be absolutely waterfree).

I.3 Preparation of the sample

The following steps should be executed to achieve optimum dispersion conditions of a sample:

1. Weigh the sample into a 250 ml cylinder and add 20 ml isopropanol. The isopropanol used has to be absolutely waterfree.
2. Place the cylinder into an ultrasonic bath for 5 min to disperse the material, made up to 250 ml with isopropanol and covered.

I.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP).

Mie parameters:

	Real part (n_p)	Imaginary part (k_p)
Quicklime	1.838	No value
Slaked lime	1.555	No value

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

J - Dispersion conditions for Bentonite (option A)

J.1 Scope

This instruction applies to bentonite products to prepare the sample for laser diffraction.

J.2 Equipment

1. De-ionised Water
2. Overhead stirrer capable of running at 2000 rpm or greater
3. Glass Beaker 1litre
4. Balance able to measure to 2 decimal places

J.3 Preparation of the sample

The following steps should be executed to achieve optimum dispersion conditions of a sample:

1. Weigh 8 g of bentonite, add to 400 ml deionised water.
2. Allow bentonite to hydrate for 24 hours
3. Stir the bentonite slurry for 5 min to disperse the material at a peripheral speed of approximately 10 m/sec.

J.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP).

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.

K - Dispersion conditions for Bentonite (option B)

K.1 Scope

This instruction applies to bentonite products to prepare the sample for laser diffraction.

K.2 Equipment

1. De ionised Water
2. IKA Ultra-Turrax or similar homogenizer
3. 800 ml Glass Beaker
4. Balance able to measure to 2 decimal places

K.3 Preparation of the sample

The following steps should be executed to achieve optimum dispersion conditions of a sample:

4. Weigh 2% of bentonite to deionised water e.g. add 8 g of bentonite in 400 ml water.
5. Allow bentonite to hydrate for 24 hours
6. Place the Homogenizer into the bentonite slurry for 10 min to disperse the material, at 13500 rpm.

K.4 Particle Size Distribution

Use this prepared sample to measure the Particle Size Distribution with Laser diffraction capable to identify nano particles, using your standard operation procedure (SOP).

When using a different strategy of sample preparation, this should be clearly identified in your report on the results.