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**Sample preparation — Dispersing  
procedures for powders in liquids**

*Préparation de l'échantillon — Procédures pour la dispersion des poudres  
dans les liquides*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14887 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

Annexes A and B of this International Standard are for information only.

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## Introduction

The evaluation of particle size distribution is of crucial importance for research projects, product development, process control, quality control, and other technical activities where particle size effects are important. Paints, inks, filled plastics, ore processing, pharmaceuticals, agricultural and cosmetic products depend on accurate particle size analysis for their commercial production.

A typical powder is composed of clumps of "primary" particles that are held together by weak or strong forces. The size of clumps remaining after the powder has been wetted into a liquid depends in part on how much energy has been expended in breaking up these clumps. Since a clump responds to most particle sizing methods as a large particle would, the presence of clumps in incompletely dispersed samples skews the reported particle size distribution to larger sizes than if all the clumps were broken up. A particle size analysis is useful only if the sample is prepared so that the particles are in a well-defined degree of dispersion, preferably one in which most clumps are fully deagglomerated and in which the particles do not reagglomerate or adhere to the walls of the sample container during the time required for analysis.

While "complete" dispersion to primary particles is often desired, it is important to remember that in many cases the most useful information is obtained when the sample is not fully dispersed. For example, if a customer blends the powder into a liquid using a low-shear process that does not break moderately strong bonds in the clumps, the quality control tests for powder intended for that customer should use similarly low shear during sample preparation and analysis.

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Because of the impurities present, the equipment available for breaking up clumps, the methods used for particle size analysis, and the dispersing agents available for testing may vary from one site to another, the procedure developed at one site by applying the guidelines in this International Standard may differ from (but be as valid and as useful as) that developed at another site for the same powder.

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A list of references for further study, including standards for evaluation of some of these more complex systems, is given in the bibliography.

Annex A discusses some of the complications that arise

- when the powder has a surface treatment or soluble components;
- when the liquid contains ionic or polymeric solutes;
- when the dispersing agent contains minor ingredients.

Annex B covers the classification of commercial dispersing agents in the various dispersing agent categories.

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# Sample preparation — Dispersing procedures for powders in liquids

## 1 Scope

This International Standard was developed to help particle size analysts make good dispersions from powder/liquid combinations with which they are not experienced. It provides procedures for

- wetting a powder into a liquid;
- deagglomerating the wetted clumps;
- determining if solution composition can be adjusted to prevent reagglomeration;
- selecting dispersing agents to prevent reagglomeration;
- evaluating the stability of the dispersion against reagglomeration.

This International Standard is applicable to particles ranging in size from approximately 0,05 to 100  $\mu\text{m}$ . It provides a series of questions on the nature of the powder and liquid involved. The answers are used with charts that guide the user to generic dispersing agents that are likely to be suitable for dispersing the powder in the liquid.

This International Standard applies only to the preparation of simple, dilute dispersions (less than 1 % by volume solids) for particle size analysis. It does not deal with the formulation of complex and commercial mixtures highly loaded with solids, such as paints, inks, pharmaceuticals, herbicides and composite plastics.

## 2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 8213:1986, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps.*

## 3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

### 3.1

#### **agglomerate**

assemblage of particles which are loosely coherent

SEE **floc** (3.5)

**3.2  
aggregate**

assemblage of particles rigidly joined together

NOTE Because of the confusion which exists in the use of the above terms they are used sparingly throughout the text.

**3.3  
clump**

assemblage of particles which are either rigidly joined or loosely coherent

**3.4  
critical micelle concentration  
CMC**

concentration of dispersing agent above which micelles will form

**3.5  
floc**

assemblage of particles which are very loosely coherent

SEE **agglomerate** (3.1)

**3.6  
primary particles**

units that are to be measured in the particle size analysis, in general harder to break than clumps

**3.7  
Tyndall effect**

light scattered perpendicular to a beam of light passing through a liquid that contains particles

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**4 Symbols and abbreviated terms**

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For the purposes of this International Standard, the following symbols and abbreviations apply.

$S_V$	Volume-specific surface area ( $m^2/kg$ )
CMC	Critical micelle concentration ( $mol/m^3$ )
IS	Ionic strength ( $mol/m^3$ )
$M_{-1,3}$	Complete $-1$ -th moment of the density distribution of particle volume
PEO	Polyethoxy = $(-CH_2-CH_2-O-)_n$
PPO	Polyisopropoxy = $(-CH_2-CH(CH_3)-O-)_n$
$pH_{iso}$	pH at which the zeta potential is zero for an amphoteric surface (which is positively charged at lower pH and negatively charged at higher pH)
$pK_a$	pH at which half the hydrogen ions from acid groups are ionized
$pK_b$	pH at which half the hydroxide ions from base groups are ionized
$\bar{q}_{3,i}$	Density distribution of particle volume
$x_i$	Upper particle size of the $i$ -th particle size interval (m)
$\mu m$	Micrometer
$\zeta$	Zeta potential [V]
®	Registered trade name.



## 5 Examination of the dry powder

### 5.1 Sampling

Sampling shall comply with the requirements specified in ISO 8213, unless a method specified in a national standard or mutually agreed upon by the analyst and client takes precedence. Sample preparation shall always be done consistently so that repeated preparations based on replicate samples of a batch of powder (which was carefully mixed before being sampled or subdivided into samples) give closely comparable results.

### 5.2 Clump size range and particle size range

Sprinkle the dry powder on a microscope slide and examine it using an optical microscope at  $\times 200$  magnification or other suitable magnification. Put a cover glass over the powder on the microscope slide and tap the cover glass lightly with a spatula (take care to avoid breaking the cover glass) to see how easy it is to crush the clumps. Note the approximate size range of the clumps that are not broken up by such crushing. If the majority of the particles are smaller than  $1\ \mu\text{m}$ , use a transmission or scanning electron microscope to observe and characterize the particles.

### 5.3 Shape and surface roughness; their variation with size

Note whether the surfaces of the fundamental particles are spherical or crystalline, smooth or rough, porous or nonporous. Determine whether all the sizes of particle have the same morphology. If the particles are very rough or porous, obtain an experimental measure of the volume-specific surface area ( $\text{m}^2/\text{kg}$ ). If this value is large compared to the area computed for spheres with the powder's particle size distribution then an unusually large amount of dispersing agent (compared to a similar size distribution of spherical nonporous particles) may be required to stabilize the dispersion.

NOTE The volume-specific surface area of spheres may be calculated from

$$S_V = 6M_{-1,3} \quad (\text{equation 35 in ISO 9276-2})$$

where

$$M_{-1,3} = \sum_{i=1}^n \bar{q}_{3,i} \ln \frac{x_i}{x_{i-1}} \quad (\text{equation 31 in ISO 9276-2})$$

## 6 Selection of a liquid and trial dispersion

### 6.1 Selection of a liquid

The analyst shall list the liquids that are commonly used for dispersing the solids for the selected method of particle size analysis and shall strike from the list any that fail to satisfy the following criteria.

- If the method is sedimentation, the liquid shall have a specific gravity that differs sufficiently from that of the powder to permit the use of this method.
- If the method is light scattering, the liquid shall have a refractive index (at the analytical wavelengths) that differs sufficiently from that of the powder to permit the use of this method.
- The liquid shall have negligible reactivity with the powder.
- The liquid shall not swell or shrink the particles by more than 5 % in diameter.
- The liquid shall provide a solubility of less than 5 g of powder per 1 kg of liquid.

NOTE This is to minimize Ostwald ripening that could cause the particle size distribution to change during the measurement time.

— The liquid shall have a change in the solubility (for the powder) with temperature of less than 0,1 mg/l per kelvin, or the temperature shall be controlled throughout the preparation and analysis to keep the solubility from changing by more than 0,5 mg/l.

NOTE If the particle size analysis method requires 10 mg of powder dispersed in 1 litre of liquid, a temperature rise of 5 K (from an ultrasonic probe or particle-analysis instrument warmth) would cause the dissolution of 1 mg or 10 % of the powder.

## 6.2 Preparation of a test paste of the powder

Put two drops (or 0,1 g) of the liquid on an etch-roughened glass plate ("frosted" glass). Blend in a roughly equal amount of powder by sprinkling powder on the liquid surface and rubbing it into the liquid using a circular motion of a 10 mm wide spatula, applying a moderate amount of pressure (sufficient to read 1 kg on the scale of a balance). The objective is to wet all the powder surfaces and to break up all clumps of powder into primary particles. The high concentration of solids provides crowded conditions that favour collision between clumps and breakup into primary particles. These crowded conditions will also favour flocculation unless the particles repel one another.

## 6.3 Preparation of a dilute dispersion of the powder

Make a dilute dispersion (4 % by mass) from the concentrated paste by adding a few drops at a time of the liquid and blending in with the spatula until 50 drops (about 2,5 g) of liquid have been added. This quantity should be sufficient for examination with a microscope. If a larger quantity is required for other types of test, the analyst shall follow the instructions given in 7.2.

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## 7 Examination of the dispersion

### 7.1 Evaluate for under- or over-grinding ISO 14887:2000 <https://standards.iteh.ai/catalog/standards/sist/58bcd662-a3cf-4a8d-91f0-164a8be797f4/iso-14887-2000>

Examine the dilute dispersion using an optical microscope (for particles larger than 1 µm in diameter) or an electron microscope (for particles smaller than 1 µm in diameter). Use × 200 magnification with the optical microscope and view the particles by transmitted light.

Note whether the clumps originally seen in the dry powder have completely broken up during the procedure for making the paste and diluting it. If not, the analyst shall make a new dispersion using ultrasonic treatment (see 9.2). The analyst shall evaluate this new dispersion and increase, as needed, the energy put in to breakup clumps until full dispersion is attained.

Note what fraction of primary particles have become broken during the procedure for making the paste and diluting it. If the fraction of particles broken is over 5 %, the analyst shall make a new dispersion by simply stirring the powder into the liquid. The analyst shall evaluate this new dispersion and increase the energy put in to breakup clumps as needed until full dispersion is attained with less than 5 % breakage of primary particles (see 9.2).

Record the conditions that avoid under- or over-grinding and use these to prepare dispersions for evaluation until the clump breakup process is optimized according to the procedures in 7.2.

## 7.2 Evaluation of stability

### 7.2.1 Introduction

If the suspending liquid has a viscosity below 10 mPa·s and the particles are well-dispersed, very small particles will appear to move randomly in the microscope's field of view. Particles in the 1 µm to 5 µm range are best for observing this effect. Note that, even if the powder consists mostly of larger-size particles, there are likely to be a few particles inside the 1 µm to 5 µm range that can indicate whether or not the dispersion is stable. If the particles are smaller than 1 µm some other form of evaluation shall be used, such as measuring the rheological stress-strain

cycle of a 10 % by volume solids dispersion from  $0,1 \text{ s}^{-1}$  to  $100 \text{ s}^{-1}$  to see if it exhibits hysteresis (indicative of structure formation and breakage) or not (in which case the dispersion is stable).

Observe the dilute dispersion using the optical microscope. Note what happens when two particles come close together. Rate the stability as good if the particles repel each other rather than coming into contact. Rate the stability as marginal if the particles collide and stay together briefly before separating again. Rate the stability as poor if the particles collide and remain in contact to form a permanent floc. If the stability is good, no added dispersing agent is required to form a stable dispersion. If the stability is marginal or poor then either solution conditions (such as pH) shall be changed or a dispersing agent shall be added to provide stability.

Other methods for evaluating dispersions are noted in annex A. If microscopy and the other techniques are not feasible then particle size analysis may be used to evaluate stability. If a series of analyses separated by several hours lie within the reproducibility of the instrument (determined using a dispersion that is known to be stable) then the sample dispersion may be considered to be stable.

### 7.2.2 Notes on optical microscopy

Optical microscopy is the simplest and most effective way of evaluating the degree of deagglomeration and the stability of dispersions containing particles that are above  $1 \mu\text{m}$  in size. Note that particles whose refractive index is close to that of the liquid will not provide enough contrast to be viewed with the optical microscope. At a solids concentration of a few percent, well-dispersed particles will appear to behave as separate entities. As the cover glass is moved sideways over the surface of the slide, note whether the particles in the dispersion move individually and not as a bonded group. Note whether particles that are below about  $5 \mu\text{m}$  in size may exhibit "Brownian motion", as particles move about erratically due to unbalanced collisions of the particle with molecules of the surrounding fluid.

Particles smaller than the limit of optical resolution (about  $0,3 \mu\text{m}$ ) appear as bright spots when they are illuminated from the side with a dark field behind them (ultramicroscopy). Although the width of the spot is indeterminate, the size of the particle responsible for the spot may be estimated by its Brownian motion: the more actively a spot moves, the smaller the particle creating the spot. The size of the smallest detectable particle using this technique depends on the scattering power of the particles. Particles of titanium dioxide or of a metal as small as about  $0,02 \mu\text{m}$  may be observed using this technique, but for oil droplets the limit of observation is about  $0,1 \mu\text{m}$ .

Dispersion stability is destroyed if the particles stick to the glass microscope slide. This is a particular problem for positively charged particles, since glass is normally negatively charged. Such adhesion can also invalidate the measurement process, especially for light-scattering methods where the amount of solid circulating for analysis may be so small that it is completely removed by adsorption on the walls of the sample circulation system. In such cases, one can chemically treat the glass (with a cationic adsorbate such as dodecyl trimethyl ammonium bromide) so that it becomes positively charged and thus prevents deposition of the particles being analysed.

### 7.2.3 Notes on electron microscopy

Evaluation by electron microscopy requires that the dispersion be spread out on a thin support film and dried. As the liquid evaporates and the liquid surface shrinks between two particles, surface tension can pull previously well-dispersed particles into contact to form a clump. This problem can be minimized if the analyst can use a liquid with a low surface tension. Dispersion stability shall be judged as good if the particles are well spread out on the grid and bad if they are found mostly in clumps.

## 7.3 Evaluation of any flocs formed

If flocs have formed, put a cover glass over the dispersion on the microscope slide. Use a spatula to push the cover glass gently from the edge to slide it over the dispersion and apply shear force to the flocs. Note whether the flocs break up and how rapidly they reform. Flocs are reversible if they break up under shear and then reform similar flocs. Flocs are unstable if shear causes large, loose flocs to roll up into small, tight flocs. Flocs are strong if they do not break up with gentle sliding. In the last case, the addition of dispersing agent may not be effective unless a high shear force can be applied to break the floc in the presence of dispersing agent.

## 8 Identification of possible dispersing agents

### 8.1 Wetting of the solid particle by the liquid

The control of the wetting process allows the adhesion forces to be modified between the particles and the binding forces produced by liquids in the intermediate capillaries to be partially modified.

The general aim for particles size analysis is a spontaneous wetting as complete as possible. This can be searched by two ways:

- low-interfacial tension liquid/gaseous by wetting agents;
- low-interfacial tension solid/liquid by hydrophilizing agents.

In the case of insufficient wetting, a simultaneously mechanical treatment can be recommended (highly intensive ultrasonic treatment of the suspension, kneading of the system as a plastic mixture with a spatula).

### 8.2 General principles

Subclauses 8.2 to 8.4 explain the principles used in developing the decision charts in 8.5. Complete dispersion of a powder in a liquid occurs when the individual particles that made up the original clumps have become separated, move independently of each other, and remain separated from one another. This requires that there be no attractive force between the particles as they approach one another. If there is an attraction then the solid/dispersion will exhibit non-Newtonian flow and have a yield stress (i.e. the dispersion will be able to support a finite shear stress without any flow occurring.) Most of the indirect tests of dispersion rely on this effect. For example, a dispersion with a yield stress enables settling particles to form an open structure which does not collapse under the force of gravity. Such a dispersion will settle to form a higher sedimentation volume (lower sediment density) than a completely dispersed system would.

Highly anisotropic particles form a more or less rigid gel at very low concentrations of solids when there is a net attractive force between the particles.

### 8.3 Charge stabilization

#### 8.3.1 Introduction

Particles which bear a surface charge will repel each other if the electrostatic repulsion is larger than the polarizability attraction (also called the Hamaker or Van der Waals attraction). A surface charge corresponding to a zeta potential greater than 30 mV is generally sufficient to provide a stable dispersion. Charge stabilization is the best way to stabilize dispersions in which the liquid has a relative dielectric permittivity greater than 30 (methanol at room temperature has a relative dielectric permittivity of 33, water of about 80) and an ionic strength less than 0,1 mol/l (i.e. a low concentration of ions in solution).

#### 8.3.2 Surface ionization

The charge on the particle may arise from ionization of surface groups (influenced by the pH of the solution). For example, surface amine groups will adsorb a hydrogen ion from solution and become positively charged if the pH is below the  $pK_b$  for the powder. Surface carboxyl groups will lose a hydrogen ion and become negatively charged if the pH is above the  $pK_a$  for the powder. Amphoteric surface groups, such as the OH groups found on a metal oxide or hydroxide, will adsorb a hydrogen ion and become positive if the pH is below the  $pH_{i50}$  for that oxide and will lose a hydrogen ion and become negative if the pH is above the  $pH_{i50}$ . The dependence of hydrogen ion adsorption on pH is such that (when the ionic strength is below 0,1 mol/l) the zeta potential generally becomes large enough to stabilize a dispersion if the pH is two or more units away from  $pH_{i50}$ .