SLOVENSKI STANDARD
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Priprava vzorcev - Postopki za disperzijo prašnatih snovi v tekočinah

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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INTERNATIONAL STANDARD

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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.

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.

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.

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.

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 µm. 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

4 Symbols and abbreviated terms

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
$\overline{q}_{3,i}$	Density distribution of particle volume
x_i	Upper particle size of the i-th particle size interval (m)
µm	Micrometer
ζ	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 × 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 µm, 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 (m^2/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

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

where

$$M_{-1,3} = \sum_{i=1}^{n} \overline{q}_{3,i} \ln \frac{x_i}{x_{i-1}}$$ (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.

7 Examination of the dispersion

7.1 Evaluate for under- or over-grinding

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

