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Metallic powders — Determination of particle size distribution by gravitational sedimentation in a liquid and attenuation measurement

*Poudres métalliques — Détermination de la distribution granulométrique
par sédimentation par gravité dans un liquide et mesure de l'atténuation*



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Foreword

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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.

International Standard ISO 10076 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*.

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

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Introduction

The settling behaviour under gravity of a given mass of particles dispersed in an initially static liquid is the basis of widely used sedimentation techniques for particle-size determination. The particle size is determined from the settling velocity by the use of Stokes' equation. The particle diameter so determined, the Stokes diameter, is the diameter of a sphere having the same density and the same free-fall velocity as the particle in a fluid of a given density and viscosity. The particle concentration must be low so that interaction between particles is negligible, and the Reynolds number must be low so that laminar flow conditions prevail.

Monitoring of the concentration of particles at a known depth below the surface of an initially homogeneous suspension enables the particle-size distribution to be calculated as a function of the measured surface or mass.

In this International Standard, two attenuation methods for the determination of concentration are considered:

- absorption of a beam of light;
- absorption of a beam of X-rays.

Although they are indirect, these sedimentation-attenuation methods are frequently employed in powder metallurgy. They give reproducible results as long as precise conditions of preparation of the suspension and of measurement are followed.

Metallic powders — Determination of particle size distribution by gravitational sedimentation in a liquid and attenuation measurement

1 Scope

This International Standard specifies methods for determining the particle size distribution of metallic powders by measuring the attenuation of an electromagnetic beam projected through a suspension of particles settling in a liquid under gravity.

The methods are suitable only where Stokes' equation is applicable, i.e. laminar flow corresponding to a Reynolds number less than 0,25, and for particles whose settling rate is not affected by Brownian motion. They are therefore suitable for all metallic powders, including powders for hardmetals, containing particles in the size range 1 μm to 100 μm . They should not, however, be used for

- a) powders containing particles whose shape is far from equiaxial, i.e. flakes or fibres, except by special agreement;
- b) mixtures of powders;
- c) powders containing lubricant or binder;
- d) powders which cannot be dispersed in a liquid.

These considerations set an upper and a lower limit on the size of particles to be tested by a sedimentation method (see 5.1).

If the largest particle present in the sample exceeds this limit, the viscosity of the liquid needs to be increased to meet this requirement.

Stokes' law may be assumed to be valid for an initial concentration of the powder in the liquid of up to 0,5 % (V/V). In some cases, higher concentrations of up to 1 % (V/V) still give correct results, but validation tests are necessary for each material.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3252:1982, *Powder metallurgy — Vocabulary*.

ISO 3954:1977, *Powders for powder metallurgical purposes — Sampling*.

3 Definitions

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

3.1 Stokes diameter: Diameter of a sphere having the same density and the same free-fall velocity as a powder particle in a fluid of a given density and viscosity.

3.2 effective density: Ratio of the mass of the powder to the volume as measured by pycnometry.

3.3 sedimentation height: Vertical distance between the upper surface of the suspension and the level of measurement of concentration.

3.4 cumulative undersize by mass: Mass of all the particles whose Stokes diameter is less than a given value. This is expressed as a percentage of the total mass of the particles.

3.5 mass fraction: Mass of all the particles whose Stokes diameter lies between two given values. This

mass fraction is expressed as a percentage of the total mass of the particles.

3.6 blank intensity: Intensity of the emergent beam with clear liquid in the sedimentation cell.

3.7 suspension intensity: Intensity of the emergent beam during sedimentation.

4 Symbols

Table 1 — Meanings of the symbols used in the text

Symbol	Meaning	Unit	Observations
<i>Sedimentation system</i>			
g	Acceleration due to gravity	m/s ²	$g = 9,81 \text{ m/s}^2$
L	Absorption length	m	Length of the light or X-ray beam path through the suspension
h	Sedimentation height	m	
c_0	Initial concentration	kg/m ³	
c	Concentration	kg/m ³	
ρ_l	Density of the liquid	kg/m ³	
ρ_e	Effective density of the powder	kg/m ³	
η	Viscosity of the liquid	$\frac{\text{N} \cdot \text{s}}{\text{m}^2}$	
<i>Measurement</i>			
I_0	Blank intensity		
I	Suspension intensity		
D	Optical density		$D = \log_{10} \left(\frac{I_0}{I} \right)$
K_m	Extinction coefficient		
A_w	Mass projected area of the particles in random orientation	m ² /kg	
S_w	Mass-specific surface area of the powder	m ² /kg	
μ_a	Mass absorption coefficient of the atoms present in the powder		
t	Sedimentation time	s	
d_{st}	Stokes diameter	m	
ΔM	Mass of particles between two given diameters	kg	
q	Mass fraction	%	$q = \frac{\Delta M}{\sum_{d=0}^{d=d_m} \Delta M} \times 100$
Q	Cumulative undersize by mass	%	$Q = \frac{\sum_{d=0}^{d=d_{st}} \Delta M}{\sum_{d=0}^{d=d_m} \Delta M} \times 100$
d_m	Stokes diameter of the largest particle in the suspension	m	