
**Particle size analysis — Laser
diffraction methods**

Analyse granulométrique — Méthodes par diffraction laser



This document is a preview generated by EMS



COPYRIGHT PROTECTED DOCUMENT

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms, definitions and symbols	1
3.1 Terms and definitions.....	1
3.2 Symbols.....	6
4 Principle	8
4.1 General.....	8
4.2 Theory.....	8
4.3 Typical instrument and optical arrangement.....	9
4.4 Measurement zone.....	11
4.5 Application and sample presentation.....	11
4.6 Off-line measurements.....	12
4.7 In-line measurements.....	12
4.8 Online measurements.....	12
4.9 At-line measurements.....	13
4.10 Scattering and detectors.....	13
5 Operational requirements and procedures	13
5.1 Instrument location.....	13
5.2 Dispersion gases.....	13
5.3 Dispersion liquids.....	14
5.4 Sample inspection, preparation, dispersion and concentration.....	14
5.4.1 Sample inspection.....	14
5.4.2 Preparation.....	14
5.4.3 Dispersion.....	14
5.4.4 Concentration.....	15
5.5 Measurement.....	15
5.5.1 Setting up instrument and blank measurement.....	15
5.5.2 Sample preparation.....	16
5.5.3 Data collection of the scattering pattern.....	16
5.5.4 Selection of an appropriate optical model.....	16
5.5.5 Conversion of scattering pattern into PSD.....	16
5.5.6 Robustness.....	17
5.6 Resolution and sensitivity.....	17
5.6.1 General.....	17
5.6.2 Resolution.....	17
5.6.3 Sensitivity and result variability.....	17
6 Accuracy repeatability and instrument qualification	18
6.1 General.....	18
6.2 Accuracy.....	19
6.2.1 Introduction.....	19
6.2.2 Accuracy test.....	19
6.3 Instrument repeatability.....	19
6.3.1 Introduction.....	19
6.3.2 Repeatability test.....	19
6.4 Method repeatability.....	20
6.4.1 Introduction.....	20
6.4.2 Method repeatability test.....	20
6.5 Accuracy under intermediate precision conditions.....	20
6.5.1 General.....	20
6.5.2 Intermediate precision conditions (general test).....	21

7	Reporting of results	21
7.1	General	21
7.2	Sample	21
7.3	Dispersion	22
7.4	Laser diffraction measurement	22
7.5	Analyst identification:	22
Annex A	(informative) Theoretical background of laser diffraction	24
Annex B	(informative) Advice on dispersion liquids	41
Annex C	(informative) Dispersion methods — Recommendations	42
Annex D	(informative) Instrument preparation — Recommendations	44
Annex E	(informative) Error sources and diagnosis	46
Annex F	(informative) Refractive index — Recommendations	49
Annex G	(informative) Laser diffraction robustness and ruggedness	51
Annex H	(normative) Certified reference materials, reference materials and comparison parameters	54
Bibliography		57

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 13320:2009), which has been technically revised. The main changes compared to the previous edition are as follows:

- a) protocols for evaluation of accuracy and qualification of instrument were newly developed;
- b) new [Annex H](#) (normative) for usage of reference material has been added;
- c) new descriptions for wider applications, such as off-line, online, in-line and at-line have been added;
- d) some informative parts have been moved to new annexes;
- e) minor revisions and updates have been made throughout the document.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The laser diffraction technique has evolved such that it is now a dominant method for determination of particle size distributions (PSDs). The success of the technique is based on the fact that it can be applied to a wide variety of particulate systems. The technique is fast and can be automated, and a variety of commercial instruments is available. Nevertheless, the proper use of the instrument and the interpretation of the results require the necessary caution.

Since ISO 13320-1:1999 was first published, the understanding of light scattering by different materials and the design of instruments have advanced considerably. This is especially marked in the ability to measure very fine particles. Therefore, it was replaced with the first edition of ISO 13320 in 2009, and since then the method has been developed for a wider application. Additionally, demands raised recently not only on establishment of accuracy of measurements but also on necessity of evaluation of the accuracy and of qualification of instrument by users. Therefore, this document incorporates the most recent advances in understanding.

Particle size analysis — Laser diffraction methods

1 Scope

This document provides guidance on instrument qualification and size distribution measurement of particles in many two-phase systems (e.g. powders, sprays, aerosols, suspensions, emulsions and gas bubbles in liquids) through the analysis of their light-scattering properties. It does not address the specific requirements of particle size measurement of specific materials.

This document is applicable to particle sizes ranging from approximately 0,1 μm to 3 mm. With special instrumentation and conditions, the applicable size range can be extended above 3 mm and below 0,1 μm .

For spherical and non-spherical particles, a size distribution is reported, where the predicted scattering pattern for the volumetric sum of spherical particles matches the measured scattering pattern. This is because the technique assumes a spherical particle shape in its optical model. For non-spherical particles the resulting particle size distribution is different from that obtained by methods based on other physical principles (e.g. sedimentation, sieving).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

3 Terms, definitions and symbols

3.1 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1.1

absorption

reduction of intensity of a light beam not due to scattering

3.1.2

accuracy

closeness of agreement between a test result or measurement result and the true value

Note 1 to entry: In practice, the accepted reference value is substituted for the true value.

Note 2 to entry: The term “accuracy”, when applied to a set of test or measurement results, involves a combination of random components and a common systematic error or bias component.

Note 3 to entry: Accuracy refers to a combination of trueness and precision.