

First edition
2022-01

Corrected version
2022-03

Nanotechnologies — Evaluation of the mean size of nano-objects in liquid dispersions by static multiple light scattering (SMLS)

Nanotechnologies — Évaluation de la taille moyenne des nano-objets dans les dispersions liquides par diffusion statique multiple de la lumière (DSML)



Reference number
ISO/TS 21357:2022(E)

© ISO 2022

This document is a preview generated by EUS



COPYRIGHT PROTECTED DOCUMENT

© ISO 2022

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
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
Introduction.....	v
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
4 Symbols and abbreviated terms.....	2
5 Principles.....	3
5.1 Relevant theory.....	3
5.2 Key measurands.....	5
5.3 Method applicability and limitations.....	6
5.3.1 General.....	6
5.3.2 Sample concentration.....	6
5.3.3 Mean equivalent particle diameter.....	7
5.3.4 Sample homogeneity and stability.....	7
5.4 Method characteristics.....	7
6 Apparatus.....	8
7 Measurement procedure.....	9
7.1 Instrument preparation.....	9
7.2 Sample handling.....	9
7.3 System settings.....	10
7.3.1 General.....	10
7.3.2 Procedure to verify sample homogeneity.....	10
7.3.3 Volume fraction.....	10
7.3.4 Refractive index.....	10
8 Performance qualification.....	11
9 Data record.....	11
10 Measurement uncertainty.....	11
Annex A (informative) I_{BS} and I_T versus I^* and I.....	13
Annex B (informative) I_{BS} and I_T as a function of D for titanium dioxide and melamine resin particles.....	14
Annex C (informative) Instrument qualification.....	16
Annex D (informative) Comparative analysis of Latex suspensions at various concentrations.....	17
Annex E (informative) Analysis of titanium dioxide suspensions at different concentrations.....	18
Annex F (informative) Results of an interlaboratory comparison study.....	20
Bibliography.....	23

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 229, *Nanotechnologies*.

This corrected version of ISO/TS 21357:2022 incorporates the following correction:

- the IEC logo has been removed from the cover page.

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

Dispersions of nanoparticles in liquids are widely used in industry. Nanoparticles dispersed in liquids interact via a variety of weak and strong forces, which can lead to aggregation or agglomeration of objects (primary particles, agglomerates, aggregates, etc.). As a result, the dispersion state and the apparent mean particle size and size distribution can differ from those determined during product manufacturing, storage, and processing, particularly when using measurements requiring sample dilution or extensive preparation. Sample preparation can result in breaking or formation of aggregates or agglomerates and in some cases can also affect morphology of primary particles. Industrial stakeholders require analytical methods that are applicable to dispersions in their native state for reasons of product development, quality control and regulatory compliance.

While many methods exist for characterization of nanoparticle properties, in particular their size and size distribution, these methods typically require a specific and frequently complex sample preparation (e.g. dilution, stirring, shearing or pumping) and, therefore, do not yield characteristics specific to as-received dispersions. In addition, some experiments do not require measurement of a full particle size distribution with the mean particle size being the main measurand. Using the mean particle size measurement, it is possible to monitor other dispersion parameters of the system such as the state of agglomeration, aggregation or dissolution.

Static multiple light scattering (SMLS) based methods do not require sample preparation allowing, within limitations outlined in this document, direct measurement of the mean equivalent particle diameter in the native (as-received) state of dispersion. In addition, and beyond the scope of this document, SMLS is capable in some cases of monitoring in real time the temporal evolution of mean equivalent particle diameter due to agglomeration or aggregation processes.

This document describes a standardized method for evaluating the mean equivalent particle diameter in various sample types (including as-received samples) having a wide range of concentrations using the SMLS based method.

Nanotechnologies — Evaluation of the mean size of nano-objects in liquid dispersions by static multiple light scattering (SMLS)

1 Scope

This document provides guidance and requirements for the determination of the mean (spherical) equivalent diameter of nano-objects (i.e. particles, droplets or bubbles) dispersed in liquids using the static multiple light scattering (SMLS) technique. The technique is applicable to a wide range of materials and does not require dilution of concentrated samples.

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/TS 80004-1, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-4, *Nanotechnologies — Vocabulary — Part 4: Nanostructured materials*

ISO/TS 80004-6, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1, ISO/TS 80004-2, ISO/TS 80004-4, ISO/TS 80004-6 and the following apply.

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

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

3.1

static multiple light scattering SMLS

technique in which transmitted or backscattered light intensity is measured after multiple successive scattering events of incident light in a random scattering medium

3.2

transport mean free path

average distance that a photon travels before its direction vector in its initial direction of motion is reduced to $1/e$ of its initial magnitude by elastic scattering alone

[SOURCE: ISO 18115-1:2013, 4.299, modified — "an energetic particle" has been changed to "a photon"; "momentum" has been changed to "direction vector"; "initial value" has been changed to "initial magnitude"; notes to entry have been deleted.]