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Fine bubble technology — Measurement technique matrix for the characterization of fine bubbles

Technologie des fines bulles — Matrice de méthodes de mesure pour la caractérisation des fines bulles

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Foreword

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This document was prepared by Technical Committee ISO/TC 281, *Fine bubble technology*.

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

Fine bubble technology has numerous applications across industries such as cleaning, transport, maintenance, agriculture, aquaculture, food and drink, cosmetics as well as biomedical. The detection, characterization and quantification of properties of fine bubble mixtures are central to the development of this horizontal general purpose technology.

A number of techniques used for particle detection and characterization may be applicable to the characterization of fine bubble mixtures in liquids. Some techniques may have a number of special sample handling, sample preparation or equipment settings to yield quantifiable and reliable results.

This document lists a number of techniques and discusses their applicability for the characterization of fine bubble mixtures as well as their limitations. Fine bubbles are able to exist in opaque liquids or liquids of high viscosity. Some fine bubble samples are turbid due to a large number of bubbles. All fine bubble samples are dynamic in nature and their properties change with time. For this reason, the acquisition time of each technique is of great relevance. Most fine bubble samples contain particles as well as fine bubbles. Distinguishing particles and bubbles and then additionally characterizing them by size and number or vice-versa may not be possible with all particle characterization equipment.

Fine bubble technology — Measurement technique matrix for the characterization of fine bubbles

1 Scope

This document focuses on listing most commonly used preparation and characterization techniques for fine bubbles and their interpretation. The merits and limitations of each of the techniques are outlined.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Abbreviated terms

CCD	Charge coupled device
DLS	Dynamic light scattering
EZ	Electrical sensing zone method
LD	Laser diffraction methods
PSD	Particle size distribution
PTA	Particle tracking analysis method
RMM	Resonance mass measurement
SPOS	Single particle light interaction methods
SMLS	Static multiple light scattering
USS	Ultrasonic attenuation spectroscopy
ZP	Methods for Zeta-potential determination

5 Fine bubble characterization

5.1 General

A number of general particle counting and sizing techniques are available commercially. Some of them are applicable for the characterization of fine bubble dispersions and ultrafine bubble dispersions. Such dispersions may be in liquid of any kind. Some liquids may not be transparent (e.g. printer ink) or stable (e.g. flammable fuel). This document refers to a selection of commercially available techniques and evaluates their applicability and their limitations.

The parameters of interest are as follows.

— Fine bubble size – This usually refers to the equivalent hydrodynamic diameter but could be different depending on the techniques.

— Fine bubble size distribution – For the purpose of this document, this is the number-size (or equivalent) distribution.

— Number concentration – The total number (or equivalent) of bubbles per unit volume.

— Measurement time – The time to complete data acquisition.

5.2 Comparison of size and concentration indices from different sources

Consideration should be given when different techniques are being compared, that each technique measures a different physical property of the sample. In deriving size and/or concentration indices from different techniques, it should be anticipated that results will demonstrate differences in value but they will likely show trend and/or correlate.

Care should be taken when comparing size and concentration indices. Even if the same technique is used, the method from example two laser diffraction machines will need to be checked to verify parameters such as measurement time, analysis models and pump rate. Table 1 provides a quick reference for the typical size and concentration indices of different techniques in the measurement of bubbles.

Table 1 — Quick-use-matrix

Techniques		International Standard	Bubble measurands			
			Size	Size distribution	Number concentration (bubbles per ml)	Measurement time
Dynamic light scattering	DLS	ISO 22412	5 nm - 10 µm	Intensity-based	> 10^9	Typical 5 min
Methods for Zeta-potential determination	ZP	ISO 13099-2				< 5 min
Particle tracking analysis method[b]	PTA	ISO 19430	50 nm – 1 000 nm	Number-based	10^7 - 10^9	~5 min
Laser diffraction methods	LD	ISO 13320	100 nm – 3 mm	Volume-based	0,000 1 %	ms - 10 s
Resonance mass measurement[a]	RMM	Not available	120 nm – 1 000 nm	Number-based	10^7 - 10^9 micro-sensor[a]: 9×10^6 nano-sensor[a]: 2×10^8	0,2 nl/s ~15 min
Electrical sensing zone method	EZ	ISO 13319	50 nm- 8 000 nm	Number-based	1×10^8	10 min
Ultrasonic attenuation spectroscopy	USS	ISO 20998-1	100 nm – 1 mm	Volume-based	> 1×10^6 For ultra-fine > 1×10^8	5 min

[a] Technique may require a special procedure or detector for obtaining appropriate results.

[b] Nanoparticle tracking analysis (NTA) is often used to describe PTA. NTA is a subset of PTA since PTA covers larger range of particle sizes than nanoscale.

Table 1 *(continued)*

Techniques		International Standard	Bubble measurands			
			Size	Size distribution	Number concentration (bubbles per ml)	Measurement time
Single particle light interaction methods[a]	SPOS	ISO 21501-2 ISO 21501-3	0,1 µm - 100 µm	Number-based	$< 10^8$	30 s - 5 min
Static image analysis methods	—	ISO 13322-1	0,5 µm - > 1 000 µm	Number-based	$> 10^8$	Typical 15 min
Dynamic image analysis methods[a]	—	ISO 13322-2	0,5 µm - > 1 000 µm	Number-based	$> 10^8$	> 5 min
Static multiple light scattering	SMLS	Under development	10 nm - 100 µm	No	10^8 - 10^{12}	~10 s

[a] Technique may require a special procedure or detector for obtaining appropriate results.

[b] Nanoparticle tracking analysis (NTA) is often used to describe PTA. NTA is a subset of PTA since PTA covers larger range of particle sizes than nanoscale.

6 Characterization techniques

This clause deals with individual techniques and how they are applied to characterizing fine bubble samples. Most samples are assumed to be in a transparent liquid, but some reference to opaque samples may be found for techniques that allow their treatment.

6.1 Dynamic light scattering

6.1.1 General

Dynamic light scattering (DLS) is also known as photon correlation spectroscopy (PCS). It measures the hydrodynamic particle size by measuring the Brownian motion of the fine bubbles in a sample.

The technique uses a laser which is passed through a sample and the light scattering measured on a detector. The detector(s) could be at a variety of angles. The intensity of light is measured over a rapid timescale. The change in scattering intensity over time as particles diffuse in and out of the measurement zone is related to their size, small particles diffuse rapidly and large particles slowly. The translational diffusion coefficient is measured which can be turned into the hydrodynamic diameter by the Stokes Einstein equation [see ISO 22412:2017, Formula (A.5)].

This intensity change is expressed as a correlation function which examines the signal change over time. From this information on the size and polydispersity of the sample can be obtained. A size distribution is derived by applying an appropriate algorithm to the correlation function. It is an intensity-based technique.

6.1.2 Reference standard

ISO 22412, *Particle size analysis — Dynamic light scattering (DLS)*

NOTE Originally, there were two International Standards relating to this technique ISO 13321[1)] and ISO 22412 now merged into one single document, ISO 22412.

6.1.3 Size

The lowest size in DLS will depend on the system sensitivity, and for most systems will be sufficient for sizing fine bubbles. The upper size range will depend on when the bubbles no longer behaving as

1) Withdrawn.

objects undergoing Brownian motion. In the case of particles this is normally sedimentation, but for bubbles, rising is more likely.

6.1.4 Size distribution

Dynamic light scattering measures an intensity-based distribution. This leads to sensitivity on the large particle end of the distribution as they scatter light with more intensity. In size area of interest, there is a 10^6 dependence of scattering intensity with size. This may lead to issues obtaining accurate data if large particles or contaminants are present. For a clean sample, the technique is able to provide a distribution for fine bubbles in the size area of interest.

6.1.5 Concentration

Most dynamic light scattering instruments are set up to auto adjust for concentrations ranging from low to high. Number concentration of bubbles required for appropriate measurements is generally higher than that given for particles. A concentration of (or over) 10^9 bubbles/ml is normal.

6.1.6 Measurement time

As for PTA (see 6.3), theoretical derivation of Stokes-Einstein equation [see ISO 22412:2017, Formula (A.5)] assumes no change in particle population, their size or number on the time scale of the measurement. This may not be true for dynamically changing fine bubble dispersion where all these parameters may change. The measurement time is therefore very important. An appropriate technique needs to be selected in order to ensure appropriate sampling and data acquisition.

6.2 Methods for Zeta potential determination (electrophoretic mobility)

6.2.1 General

Zeta potential is the electrical potential at the slipping plane (hydrodynamic plane of shear). It is a measure of the electrical charge on a particle and hence the stability of a system (Zeta potentials < -30 mV and > 30 mV denote a stable system. The Zeta potential is derived from the electrophoretic mobility (the movement of a particle under an applied electric field). The technique of laser Doppler electrophoresis is used.

6.2.2 Reference standard

ISO 13099-2, *Methods for zeta potential determination — Part 2: Optical methods*

6.2.3 Charge

A mean Zeta potential is normally reported which is an indicator to the stability of the system.

6.2.4 Zeta distribution

A distribution is recorded, but the mean is normally used rather than the distribution.

6.2.5 Concentration

Dilution is complex as dilution can change the particle charge, so media with exactly the same pH, ionic strength, and stabilizer concentration should be used.

6.2.6 Measurement time

Less than 5 min.

