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Fine bubble technology — Characterization of microbubbles —

Part 1: Off-line evaluation of size index

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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.

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).

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

A list of all parts in the ISO 21910 series can be found on the ISO website.

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

Recent development in the fine bubble technology expands its market, such as cleaning, water treatment, agriculture and aquaculture as well as biomedical. Above all, the application of microbubble technology accelerates the market penetration.

Many measurement technologies have been historically developed to assess the characteristics of microbubbles and are now used in various application fields. However, the dynamic nature of microbubbles makes it hard for the users to report their measurement results with confidence. The low stability of microbubbles that includes shrinking, deformation, coalescence and dissolution of individual microbubble can require a specific sampling procedure and short measurement time.

This document is intended to specify an evaluation method for size index of microbubbles in water to be used in a measurement laboratory. The application of the document to measurement system will yield comparable results over an application field, as far as the specified types of measuring instruments are equipped and the specified sampling procedures are met. Since the comparability relevance depends on the sampling procedures and the measurement environments, each measurement can require relevant descriptions. The specifications of the measuring instruments are described in other standards or the individual operation instruction manuals.

Fine bubble technology — Characterization of microbubbles —

Part 1: Off-line evaluation of size index

1 Scope

This document specifies the evaluation method for the size index of microbubbles in microbubble dispersion. It is only applicable to microbubbles with or without shell in water within the range from 1 µm to 100 µm. It describes the sampling methods from the point generating or dispersing microbubbles in the retention container to the detecting point of the measuring instruments.

2 Normative references

There are no normative references in this document.

3 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
measurement time
period for a sequence of measuring process, whereas the size index and/or the number concentration index of the microbubbles can be assumed stable all through the period and reproducible over the periods with similar measurement condition

Note 1 to entry: Measurement time is described by the starting time and the ending time, or by either of them and the duration.

3.2
water diluent
homogeneous water which is used for dilution without causing any deleterious effects and whose number concentration of ultrafine bubbles is known

Note 1 to entry: Water diluent is used to decrease the number concentration of ultrafine bubbles in a dispersion without changing their total number, state of aggregation with particles, size or surface chemistry.

Note 2 to entry: Water diluent is called blank water when its number concentration of ultrafine bubbles is known to be zero and when it is used for the evaluation of ultrafine bubbles.

[SOURCE: ISO 20298-1:2018, 3.2]

3.3
retention time
period from the point generating or dispersing microbubbles in the retention container to the detecting point of the measuring instruments

3.4
method repeatability
closeness of agreement between multiple measurement results of a given property in different aliquots of a sample, executed by the same operator in the same instrument under identical conditions within a short period of time

Note 1 to entry: The variability includes those uncertainties due to operator sub sampling technique, any changes in the sampled material together instrument variations.

[SOURCE: ISO 13320:2020, 3.22]

4 Requirements

4.1 Requirements on the sample

Microbubbles should be present in pure water or can be in tap water if the numbers of contaminant particles are much lower than those of microbubbles. The microbubbles should contain air, nitrogen and oxygen. In cases where they are surrounded with a coating, e.g. a lipid, other gas can be applied.

The size range of microbubbles that can be measured depends on the specification of the measuring instrument to be used as is the number or volume concentration range of microbubbles. The reliability of the results shall be confirmed for each measuring instrument.

Microbubbles which diameter is equal or larger than 10 µm should be measured promptly (see 7.6.1). Microbubbles less than 10 µm and microbubbles with shell may not need to be measured quickly. This classification is referred to in ISO 20480-2.

4.2 Requirements on the sample transfer and measurement system

An appropriate sample transfer and measurement system which connects a microbubble generating system and a measuring instrument enable to characterize unstable microbubble dispersion in water as a size index. Because of the low bubble stability of microbubbles without shell and the decrease in dispersibility of microbubbles with shell, it is recommended to have the measuring instrument in the vicinity of either the generating system or the dispersing system.

The size index of microbubbles should be determined just after the sample transfer. Signal acquisition time of measuring instrument should be set at a minimum interval which is necessary for the detection of sufficient signals from the microbubbles to determine its size index with good reproducibility. Before loading the sample into a measuring instrument, clean the inside of a generating system by rinsing it several times with water diluent to remove contaminants.

Attention should be paid to avoid microbubbles to adhere to the inside of the tube which is used for loading to the measuring instrument.

Before each measurement, it should be confirmed if microbubbles are dispersed homogeneously or saturated in water at a trial run.

Microbubbles with shell are inherently stable; however, they may settle down to the bottom of the container. The procedure to measure microbubbles without shell can be applicable to measure microbubbles with shell when they are dispersed uniformly during the measurement.

Storage or transportation of microbubbles without shell in microbubble dispersion is almost impossible due to the low bubble stability.

5 Measuring instruments

Measuring instruments based on the following measurement techniques should be used to determine the size index of microbubbles.

— Dynamic image analysis methods (see ISO 13322-2).

— Laser diffraction methods (see ISO 13320).

— Light extinction liquid-borne particle counter (see ISO 21501-3).

The reference of the method used shall be reported.

Refer to Annex E for an example of comparison among three measurement techniques.

6 Environment

The classification of air cleanliness should be applied for the measurement to prevent the contamination of impurities.

Ambient temperature and atmospheric pressure should be stable to maintain the stability of microbubble size.

7 Sample transfer and measurement system

7.1 General

The dynamic changes of microbubbles without shell may make it difficult to measure the microbubble size. To obtain reproducible results with off-line measurement, the appropriate way to load microbubble dispersion is the key technology to measure them promptly before they disappear.

For this purpose, the sample transfer and measurement system as shown in Figure 1 shall be used.

Essential information to define a sample transfer and measurement system is given in 7.2 to 7.7.

7.2 System structure

Microbubbles to be measured shall be generated or discharged in a retention container. The container may work as a buffer to circulate microbubbles dispersion. The samples shall be sucked into a flow cell using loading tube and loading pump set at the back of a flow cell.

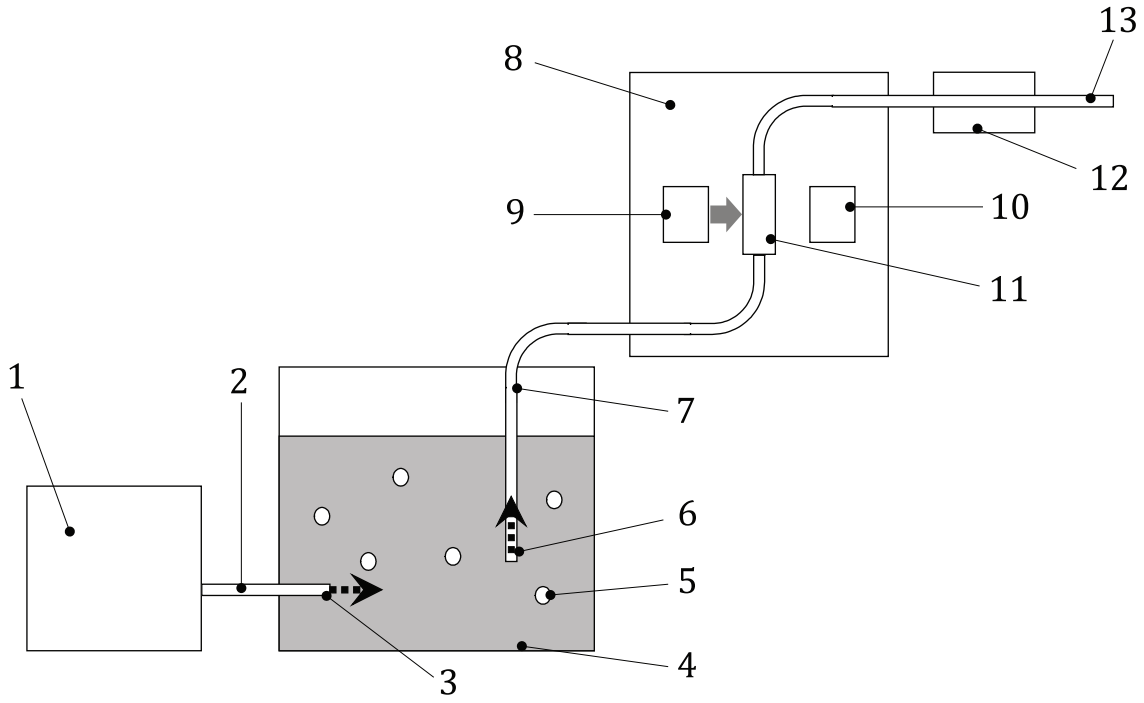
7.3 Arrangement of components

7.3.1 Position of the inlet tube mouth

The position of the inlet tube mouth and the direction of it should be optimized for the spatial stability at a trial run, or microbubbles should be measured at several positions to be averaged in consideration to the velocity distribution in the retention container. If the position of the inlet tube mouth is set close to the container wall and the surface of microbubbles dispersion, results may show low concentration. If the position of the inlet tube mouth is set in front of outlet from a generating system, results may show high concentration.

7.3.2 Position of the measuring instrument

The position of the measuring instrument should be close to a retention container to minimize the tube length.



Key

1	generating system	8	measuring instrument
2	outlet tube for generating system	9	light source
3	outlet tube mouth for generating system	10	detector
4	retention container	11	flow cell
5	microbubble	12	loading pump
6	inlet tube mouth for measuring instrument	13	drainage tube
7	loading tube		

Figure 1 — Sample transfer and measurement system (side view)

7.4 Retention container

7.4.1 General

A retention container should be defined depending on the purpose of the measurement, e.g.to evaluate the specification for a generating system under the recommended condition or to understand the dispersed situation for each actual usage.

7.4.2 Configuration requirements for retention container

The retention container size should be defined after testing different sized containers at a trial run. In a large container, microbubbles may disperse quickly leading to a low concentration.

The retention container structure should not be cylindrical shape in which microbubbles may rotate inhomogeneously. It also should not be shallow shape in which microbubbles may float quickly to the surface.

7.5 Loading tube

7.5.1 Loading tube inner diameter

Inner diameter shall be equal or larger than at least 2 mm. Small inner diameter can affect microbubbles characteristics due to interaction of the loading tube inner surface (see B.2).

7.5.2 Loading tube length

Loading tube length should be short and may be defined by the length from the inlet tube mouth for the measuring instrument to the detecting point of the measuring instrument. Long tube length may cause microbubbles extinction due to interaction of the tube inner surface. It may be defined by the retention time, flow rate and tube inner diameter. It is recommended to consider the volume inside a measuring instrument, that is the hidden tube and the flow cell volume up to the detecting point (see B.2).

7.5.3 Curvature of the loading tube

Curvature of the loading tube should be larger than 100 mm. Small curvature may cause an inner vortex and affect microbubbles characteristics.

7.5.4 Surface roughness

As surface roughness of loading tube, the arithmetical mean deviation of the assessed profile, *Ra*[1], is available. Surface roughness should be less than 0,4 µm. High surface roughness may cause microbubbles adhesion and extinction due to interaction of the loading tube inner surface.

7.5.5 Loading tube materials

Loading tube materials shall have hydrophobic characteristic and shall not be eluted into microbubble dispersion. They shall not be soft materials like natural rubber to prevent vibration. If microbubbles are negatively charged as microbubbles without shell, the tube materials shall not be positively charged materials like polyamide to prevent adhesion. They should be negatively charged materials like PTFE (polytetrafluoroethylene) and PFA (perfluoroalkoxy alkane). (See B.3).

7.5.6 Suppression of loading tube sway

Sway or vibration of the loading tube may damage a measuring instrument and may interfere with accurate measurements.

To minimize the sway or vibration of the loading tube, the tube should be fixed at appropriate position.

7.6 Loading pump

7.6.1 General

The samples shall be sucked from the back of the detecting point using a loading pump. The loading pump should be adjustable of the flow rate. In addition, it should be sucked stably and be drained under the atmospheric pressure. The pulsation created by a loading pump should be minimized to prevent from producing large uncertainties in the results. The loading pump can include devices to minimize pulsation like a dumper, an air chamber and longer drainage tube.

7.6.2 Flow rate (Flow velocity)

Flow rate should be slow to avoid deformation and coalescence due to rapid fluid flow. Reynolds number calculated from flow rate shall be less than 2 300 at which the transition from laminar to turbulent flow occurs. It should preferably be less than 1 150.