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Fine bubble technology — Elimination method for sample characterization —

Part 2: Fine bubble elimination techniques

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Contents

Foreword

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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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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 281 *Fine bubble technology*.

A list of all parts in the ISO 20480 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

Fine bubble technology has recently seen growth in its application to markets such as cleaning, water treatment, agriculture and aquaculture and biomedical fields. Now methods are needed to evaluate the efficacy and efficiency of generation systems. The characteristics of most importance are the number concentration and size indices of the fine bubbles produced by such systems.

Furthermore, fine bubble dispersions in water can contain other solid and liquid particles. Contaminants such as these particles make accurate characterization of fine bubbles extremely difficult. Therefore, distinction of fine bubbles from contaminants is an urgent issue. This is also why the term index is used as it can never be 100 % established that a particle is a bubble and not a contaminant.

Currently, there are several measurement methods widely used to evaluate the number concentration index and the size index of particles. However, there are few methods to distinguish bubbles in fine bubble dispersions from other particles.

If the bubbles can be totally eliminated (with no residual bubbles) by mechanisms such as dissolution or flotation, this issue can be solved. If a method that eliminates fine bubbles in a specific size range is used, it is potentially possible to distinguish fine bubbles from other solid and liquid particles. The concentration of material after potential elimination can be compared in particle concentration index to the liquid used prior to generation. If elimination has been successful, they should be the same, and by this comparison, elimination techniques can be developed and optimized.

This document is intended to specify the most effective techniques for eliminating fine bubbles from fine bubble dispersions in water and their optimization.

Fine bubble technology — Elimination method for sample characterization —

Part 2: Fine bubble elimination techniques

1 Scope

This document specifies the elimination techniques for removing fine bubbles from fine bubble dispersion in water and how to optimize the elimination procedures to obtain better efficiency.

This document is applicable to fine uncoated bubbles (without shells).

It does not apply to fine coated bubbles (with shells).

NOTE Fine bubbles dispersed in liquid are classified into “fine bubble with shells” and “fine bubble without shells”. Fine bubble with shells means the fine bubble whose surface/interface is covered almost completely by an object or a collection of objects.

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 20480-1, *Fine bubble technology — General principles for usage and measurement of fine bubbles — Part 1: Terminology*

ISO 24261-1:2020, *Fine bubble technology — Elimination method for sample characterization — Part 1: Evaluation procedure*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20480-1 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 http://www.electropedia.org/

3.1
fine bubble dispersion
FBD
liquid which contains fine bubbles

[SOURCE: ISO 20298-1:2018, 3.1]

3.2
frequency
number of cycles per second

Note 1 to entry: Expressed in Hertz (Hz).

[SOURCE: ISO 5577:2017, 3.1.1]

3.3
ultrasonic wave
any acoustic wave having a *frequency* (3.2) higher than the audible range of the human ear, generally taken as higher than 20 kHz

4 Requirements

4.1 Sample

Fine bubble dispersions in water that are going to be used to test fine bubble elimination techniques shall be generated using cleaned fine bubble generating systems with clean water and clean gas such as air, nitrogen or oxygen.

The purity level of water and gas depends on the sample. The size and concentration indices of fine bubbles in the sample should be evaluated because fine bubble elimination is one of the evaluating processes for specified samples. Therefore, the purity level cannot be generally determined as a measurement condition for evaluations of the elimination efficiency.

The fine bubble dispersion shall not contain stabilizing agents such as surfactants.

If the measurement accuracy of size and concentration indices is very important (for example, in the case of application to the accreditation of fine bubble business), a water purity level of ISO Grade 1 (see ISO 3696) is recommended for the water used for generating FBD.

4.2 Measuring instruments

When the measuring instruments are selected for evaluating the potential for fine bubble elimination, the following requirements for the concentration and the size range shall be considered. These requirements depend on the characteristics of sample to be evaluated.

1) The total number concentration and total volume concentration of the entire sample including fine bubbles and contaminants (solid and liquid particles) shall be measured. Water diluent can be used for diluting the FBD when the concentration is too high for the measurement technique in question.

2) The size range of the entire sample including fine bubbles, contaminants and aggregates of contaminants shall be measured. Different measuring instruments may be used to confirm the larger aggregates.

NOTE For example, the particle tracking analysis method can be used for evaluation of number concentration, and the laser diffraction method can be used for the evaluation of volume concentration. ISO/TR 23015 can be referred to for details of measurement techniques which can be used for FBD in water.

5 Environment

The air cleanliness should be considered for the measurement to prevent the introduction of impurities. Ambient temperature and atmospheric pressure should be stable to maintain the stability of ultrafine bubbles.

Air cleanliness, ambient temperature and atmospheric pressure depend on the local environment and can vary. However, as they are important settings and can influence the elimination process they should be recorded prior to any elimination occurring.

If the measurement accuracy of size and concentration indices is very important, (for example, in the case of application to the accreditation of fine bubble business), the air cleanliness of ISO Class 7 (see ISO 14644-1) is recommended as the environment for generation and measurement of FBD in water.

6 Elimination technique

6.1 General

Fine bubbles can be eliminated using the following elimination techniques. The elimination technique should be selected based on the appropriate elimination efficiency which is determined by tuning and optimizing the conditions and parameters for the corresponding technique.

6.2 Ultrasonication method

6.2.1 General

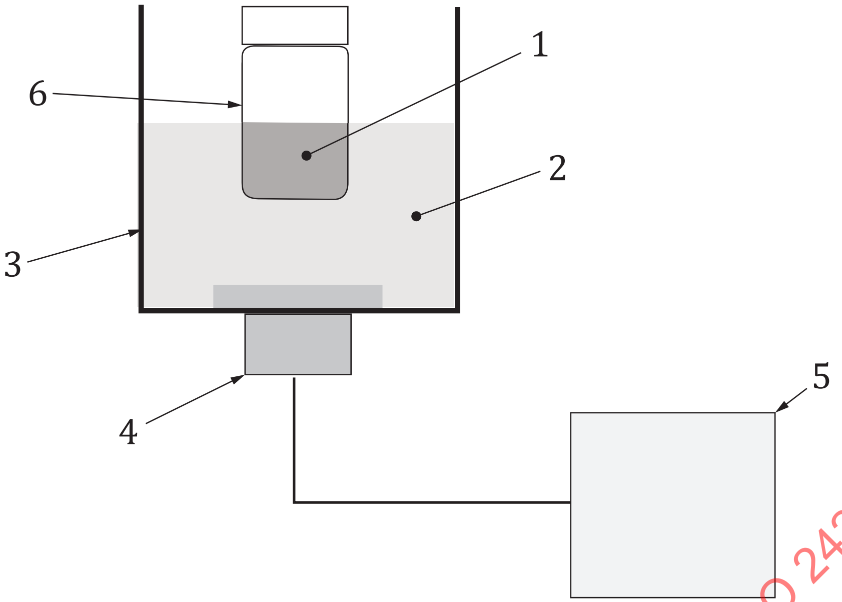
The ultrasonication method can eliminate fine bubbles in fine bubble dispersion in water by irradiating ultrasonic wave with suitable frequency (see Annex A for an example), while irradiation of ultrasonic wave with unsuitable frequency can generate fine bubbles. Therefore, setting of the frequency is very important.

Other variables that can also change the effectiveness of elimination include driving power and irradiation time.

6.2.2 System configuration

The typical system to eliminate fine bubbles by ultrasonication method has an oscillator, transducer and tank as shown in Figure 1. A container that has fine bubble dispersion in water is put into a tank filled with water and irradiated by ultrasonic waves. It is recommended to irradiate ultrasonic waves whose frequency is higher than 430 kHz.

The oscillator shall not be immersed in the fine bubble dispersion in water because direct contact with the oscillator can generate contaminants. For example, an ultrasonic probe or ultrasonic homogenizer shall not be used.



Key

1 fine bubble dispersion in water
2 water
3 tank
4 transducer
5 oscillator
6 container

Figure 1 — Scheme for the ultrasonication system configuration

6.2.3 Key experimental variables for the ultrasound elimination technique

There are several key experimental variables that needed to be considered when using the ultrasound elimination method. These should be specified in the method. If these parameters are not kept consistent then results are likely to be inconsistent (between users, laboratories, samples, etc). They are stated in the following list:

1) Specification of ultrasonic device (make, model, power);

2) Container (type of vessel, volume);

3) The volume of fine bubble dispersion in water in container;

4) The height of water surface in tank;

5) The position of the vessel in the tank (The distance between transducer and the bottom of container and location of the container in relation to the sides of the vessel).

The container shall have a flat bottom and be made of a rigid material such as glass. The thickness of the container bottom should be thick enough that it does not influence the ultrasonication environment. The container shall not be filled with fine bubble dispersion in water. The height of water surface in the tank should be almost as high as the height of water surface in the container. If the distance between transducer and the bottom of container is too small, ultrasonic waves will not be irradiated normally. On the other hand, if it is too far, ultrasonic power will be attenuated.

6.2.4 Conditions and parameters to be tuned or optimized

It can be possibly to set the following experimental parameters using the ultrasonic device under test. Not all devices will necessarily provide the user with the ability to change all of the parameters. For example, a simple ultrasonic bath might only allow the irradiation time to be adjusted.

1) Frequency

2) Driving power

3) Irradiation time

The variables which can be adjusted by the device shall be optimized to maximize elimination efficiency according to the procedure outlined in 6.2.5.

6.2.5 Procedure for parameter optimization

The key experimental parameters described in 6.2.4 shall be tuned or optimized in accordance with the following procedure:

1) measure the size and number concentration of fine bubble dispersion in water;

2) set a certain set of conditions on the device such as frequency, driving power and time;

3) put an appropriate volume of fine bubble dispersion in water into a container;

4) put the cap on the container;

5) put water in the tank;

6) fix the container directly above the transducer;

7) homogenize the processed sample in the container;

8) evaluate the elimination efficiency (in accordance with ISO 24261-1:2020, 6.3);

9) set several kinds of frequency, driving power and irradiation time and repeat steps 2) to 8);

10) evaluate the elimination efficiencies corresponding to the respective conditions and parameters;

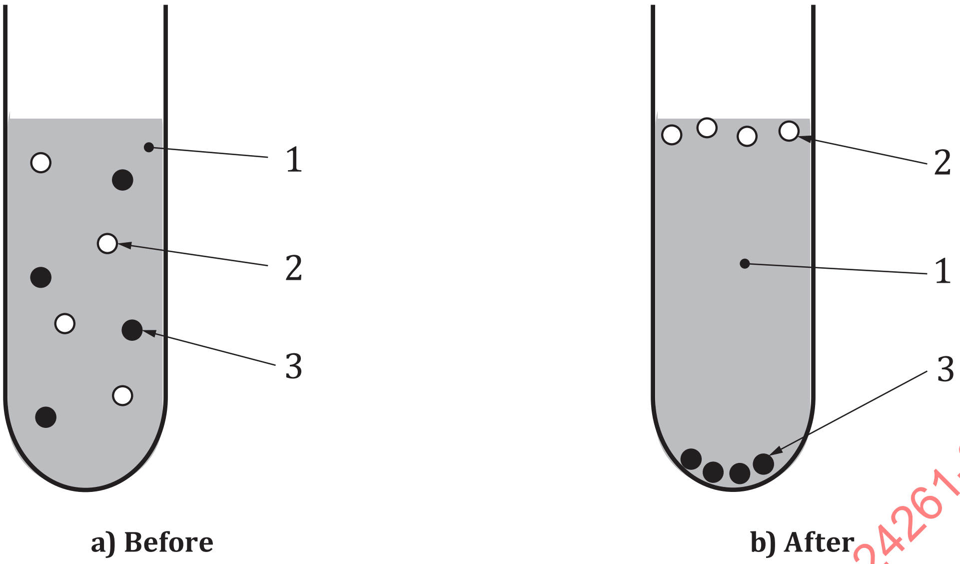
11) determine the most appropriate conditions for the ultrasonication method based on the data generated in step 10, these being the conditions at which the maximum elimination is achieved.

6.3 Ultracentrifugation method

6.3.1 General

This elimination method uses an ultracentrifuge. Fine bubbles and particles in the FBD are separated according to their density. Particles heavier than water move towards the outer circumference of motion and fine bubbles and particles lighter than water move towards the inner circumference of motion as shown in Figure 2. The fine bubbles gathered on the surface of a sample tube can disappear (see Annex B for an example).

If the fine bubble dispersion includes liquid or solid particles as contaminants, they are also separated by this method. The liquid or solid particles with a higher density than water can aggregate on the bottom of sample tube. Aggregated particles shall be dispersed one by one again to evaluate the number of particles accurately (in accordance with ISO 24261-1:2020, 6.3) after the centrifuge procedure. The liquid or solid particles with a lower density than water float to the surface of sample. Floating particles shall be homogenized to evaluate them as well.



Key

1 water

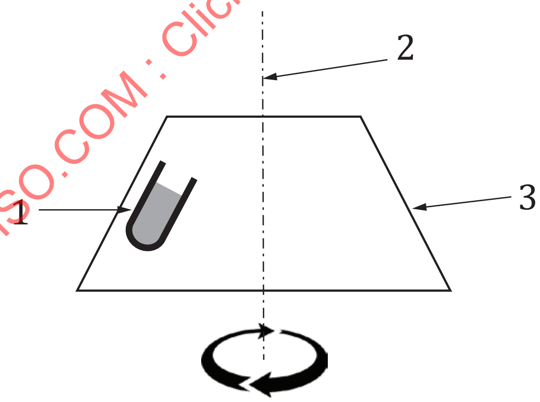
2 fine bubbles or particles lighter than water

3 particles heavier than water

Figure 2 — Ultracentrifugation of fine bubble dispersion in a sample tube

6.3.2 System configuration

As shown in Figure 3, the ultracentrifuge has a rotor where sample tubes are set, with a cooling system. It is recommended to generate acceleration as high as 1 000 000 g.



Key

1 sample tube

2 rotating shaft

3 rotor

Figure 3 — Scheme for the system configuration of ultracentrifugation

6.3.3 Specifications, conditions and parameters to be selected and predetermined

There are several key experimental variables that needed to be considered when using the ultracentrifugation elimination method. These should be specified in the method. If these parameters are not kept consistent then results are likely to be inconsistent (between users, laboratories, samples etc). They are stated in the following list:

1) Specification of ultracentrifuge (make, model);

2) Sample volume in each tube;

3) Temperature of the sample.

6.3.4 Conditions and parameters to be tuned or optimized

The following list outlines the key experimental parameters that will be available to the user for adjusting and optimization. Their magnitude will need to be set according to the procedure outlined in 6.3.5 to optimize elimination conditions:

1) Number of rotations or centrifugal acceleration

2) Ultracentrifugation time

The size distribution of fine bubbles and the viscosity of water affect the performance of elimination efficiency. The number of rotations or centrifugal acceleration and ultracentrifugation time shall be optimized following this subclause to maximize elimination efficiency. The sample volume in each tube can be defined by the instruction of the instrument. The temperature can be set to keep constant.

6.3.5 Procedure of parameter optimization

The key experimental parameters described in 6.3.4 shall be tuned or optimized in accordance with the following procedure:

1) measure the number concentration of fine bubble dispersion;

2) set a certain number of rotations or a certain centrifugal acceleration;

3) set a certain ultracentrifugation time and a temperature;

4) put the same appropriate volume of fine bubble dispersion into each tube;

5) put tubes in a rotor so they are directly across from each other to balance a centrifuge;

6) homogenize the processed sample in a tube;

7) measure the number concentration of the processed sample;

8) evaluate the elimination efficiency (in accordance with ISO 24261-1:2020, 6.3);

9) set several kinds of ultracentrifugation time and repeat from 4) to 8);

10) plot time and reduction rate and evaluate the effective ultracentrifugation time at this condition;

11) set several kinds of number of rotations or several kinds of centrifugal acceleration, repeat from 3) to 10); and evaluate the elimination efficiencies corresponding to the respective conditions and parameters;

12) determine the most appropriate conditions for the ultrasonication method based on the data generated in step 10), the conditions being those at which the maximum elimination is achieved.

6.4 Freeze and thaw

This method consists of two steps: freezing and thawing. By freezing until the temperature of the sample is below −15 °C, fine bubbles will be eliminated (see Annex C for an example). After thawing, the samples return to the liquid state. A typical home/laboratory freezer is adequate for this purpose.

For a freezing experiment to take place, a sample of water from the water source, but which has not been used for bubble generation, shall also be taken. The fine bubble dispersion should be generated in the usual way (documenting time taken to generate and left to settle before any characterization).

Samples should be placed in a sample container which should be washed with the control water to remove any dust prior to the placement of ultrafine bubble dispersion in it.

Samples should be frozen for at least 24 h and preferably at least 48 h before thawing.

Thawing should be done at room temperature, and the sample should be completely defrosted before it is remeasured. Devices such as hot plates and microwaves should not be used to speed up thawing.

The samples should then be measured in the usual way. The sample before and after freezing can be compared. If elimination has been successful, the concentration index data of the thawed fine bubble dispersion should have a similar concentration index to the unprocessed water.

6.5 Boiling and cooling

To date, boiling by holding the sample at approximately 100 °C for 1 h has not been found to eliminate bubbles.

Annex A
(informative)

Example of results for the ultrasonication method

Tables A.1 and A.2 present an example of elimination results using the ultrasonication method. Ultrafine bubble dispersion is prepared using the swirling flow method with pure water and filtered air. Number concentration is measured by the PTA method.

Table A.1 — Example of the conditions

	Conditions
Frequency (kHz)	1 600
Driving power (W)	100
Irradiation time (min)	15

Table A.2 — Example of the measurement results and the reduction rate

Sample	Number reduction rate[a] r_0 %	Number concentration index before elimination process $q_{0,b}$ particles/ml	Number concentration index after elimination process $q_{0,a}$ particles/ml
Typical ultrafine bubble dispersion	90,0	$5,40 \times 10^8$	$5,39 \times 10^7$

[a] $r_0 = (q_{0,b} - q_{0,a}) / q_{0,b} \times 100$

Annex B
(informative)

Example of results for the ultracentrifugation method

Tables B.1 and B.2 provide an example of an elimination result using ultracentrifugation methods. Ultrafine bubble dispersion is prepared using the swirling flow method with pure water and filtered air. Number concentration is measured by the PTA method.

Table B.1 — Example of the conditions

	Conditions
Sample volume in each tube (ml)	1
Temperature (°C)	25
Relative centrifugal force (g)	1 050 000
Ultracentrifugation time (min)	15

Table B.2 — Example of the measurement results and the reduction rate

Sample	**Number reduction rate**[a] r_0 %	**Number concentration index before elimination process** $q_{0,b}$ particles/ml	**Number concentration index after elimination process** $q_{0,a}$ particles/ml
Typical ultrafine bubble dispersion	65,9	$3,69 \times 10^8$	$1,26 \times 10^8$
[a] $r_0 = (q_{0,b} - q_{0,a}) / q_{0,b} \times 100$			