
**Fine bubble technology —
Characterization of fine bubbles —**

**Part 1:
Evaluation of size and concentration
indices by laser diffraction method**

Technologie des fines bulles — Caractérisation des fines bulles —

*Partie 1: Évaluation des indices de concentration et de taille par
diffraction laser*



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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

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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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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 24218 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 seen growth in its application to markets such as cleaning, water treatment, agriculture, aquaculture and biomedical fields. Now the methods to evaluate the characteristics of fine bubbles such as the size and concentration indices become necessary to clarify the performances of fine bubble generating systems used for various applications.

The shape of the size distribution of fine bubble dispersions (FBD) can be bimodal or multimodal, and this distribution can extend broadly from ultrafine bubble (UFB) range to microbubble (MB) range.

To evaluate the relationship between the characteristics of fine bubbles and their effects, it should be considered that the respective modes of multimodal size distribution can have their independent contributions to the total performance of fine bubbles.

The laser diffraction method can evaluate multimodal size distributions from the range of UFB ($<1\text{ }\mu\text{m}$) to that of MB (on the micron scale).

Due to the nature of number-based size distribution, any examination of a sample on a number basis which mixes populations of ultrafine and micro bubbles is unduly weighted to the fraction of smaller size bubbles (ultrafine bubbles). The important fraction of larger size bubbles (micro bubbles) can therefore be lost. At the viewing of size distribution data, the confirmation of its dimension (number basis or volume basis) is necessary. Moreover, the suitability of approach about the dimension of size distribution should be taken into consideration in terms of what it can do to the size distribution and what is the most appropriate approach for the application under evaluation.

This document is intended to specify the evaluation of fine bubbles size and concentration indices by combined use of number-based size analysis and volume-based size analysis by laser diffraction method. Both bimodal and multimodal samples are appropriate. It is applicable to fine bubbles with and without shell over a size range which includes UFB and MB dispersions and any combination thereof.

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Fine bubble technology — Characterization of fine bubbles —

Part 1: Evaluation of size and concentration indices by laser diffraction method

1 Scope

This document specifies the evaluation of fine bubbles size and concentration indices applied to the combined use of number-based size analysis and volume-based size analysis by the laser diffraction method. The methodology described is appropriate to both bimodal and multimodal samples over a broad size range (from tens of nanometers to tens of micrometers) and applies to ultrafine bubble and microbubble dispersions (MBD) and mixtures thereof.

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 20298-1:2018, *Fine bubble technology — Sampling and sample preparation for measurement — Part 1: Ultrafine bubble dispersion in water*

ISO 21910-1, *Fine bubble technology — Characterization of microbubbles — Part 1: Off-line evaluation of size index*

ISO 13320, *Particle size analysis — Laser diffraction methods*

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 <https://www.electropedia.org/>

3.1

fine bubble dispersion

FBD

liquid which contains fine bubbles

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

3.2
ultrafine bubble dispersion
UFBD

liquid which contains ultrafine bubbles

[SOURCE: ISO 20298-1:2018, 3.3]

3.3
microbubble dispersion
MBD

liquid which contains microbubbles

[SOURCE: ISO 20298-1:2018, 3.4]

4 Test requirements

4.1 Sample

The fine bubble dispersion (FBD) to be evaluated shall be generated by the use of a clean fine bubble generating system using pure water and pure gas such as air, nitrogen or oxygen.

4.2 Measuring instrument

A measuring instrument based on the laser diffraction method shall be used to evaluate the size and concentration indices of the FBD in water. The laser diffraction method can evaluate broad multimodal size distributions from the range of UFB to that of MB. It can be applicable for the combined use of the number-based size analysis and the volume-based size analysis for a sample whose size distribution is bimodal or multimodal in this size range.

The size distribution obtained from measurement is generally expressed showing the relationship between the size and the normalized volume concentration (%). The size distribution between the size and the normalized number concentration (%) can be obtained easily using the software attached with the instrument. This number-based size distribution can also be calculated using a spreadsheet software such as Excel.

Note Some instruments can obtain the size distribution between the size and the volume concentration ($\mu\text{l/ml}$) and the size distribution between the size and the number concentration (number/ml) as shown in [Annex A](#). Similar data processing using the measured data from general instruments to obtain the size distribution of size and concentration can be realized using the spreadsheet software but it is not so easy for general users, without the support of the manufacturer.

Laser diffraction methods shall conform to ISO 13320.

4.3 Environment

The classification of air cleanliness should be applied for the measurement to prevent the contamination from impurities. Ambient temperature and atmospheric pressure should be stable to prevent the change of fine bubbles characteristics.

5 Number concentration and volume concentration

5.1 Difference of impression between number-based and volume-based size distributions

The measurement result of a number-based size analysis can be very different from that of a volume-based size analysis when the same sample is analysed. The impression caused by different measurement results can also be very different. It can introduce misjudgement in the evaluation of fine bubbles effect if only the number-based size analysis is used.

The shape of fine bubbles is almost spherical which leads to the following discussion.

In the case of fine bubble size ratio 1:10, number ratio 1:1 is equivalent to volume ration 1:1 000 as shown in [Table 1](#), and volume ratio 1:1 is equivalent to number ration 1 000:1 as shown in [Table 2](#). [Table 1](#) and [Table 2](#) show the importance to accurately recognize the difference of dimension between number and volume for the evaluation of the particle or the bubble amount.

Therefore, the combined use of number-based size analysis and volume-based size analysis by laser diffraction method is necessary for this characterization.

Table 1 — Number ratio of fine bubbles 1:1 in the case of size ratio 1:10

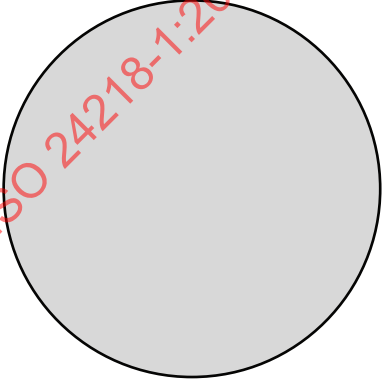
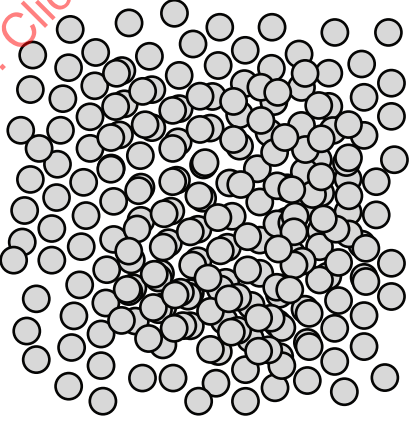
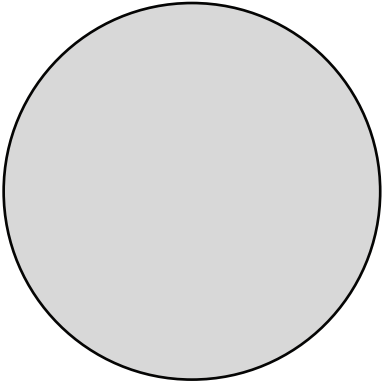
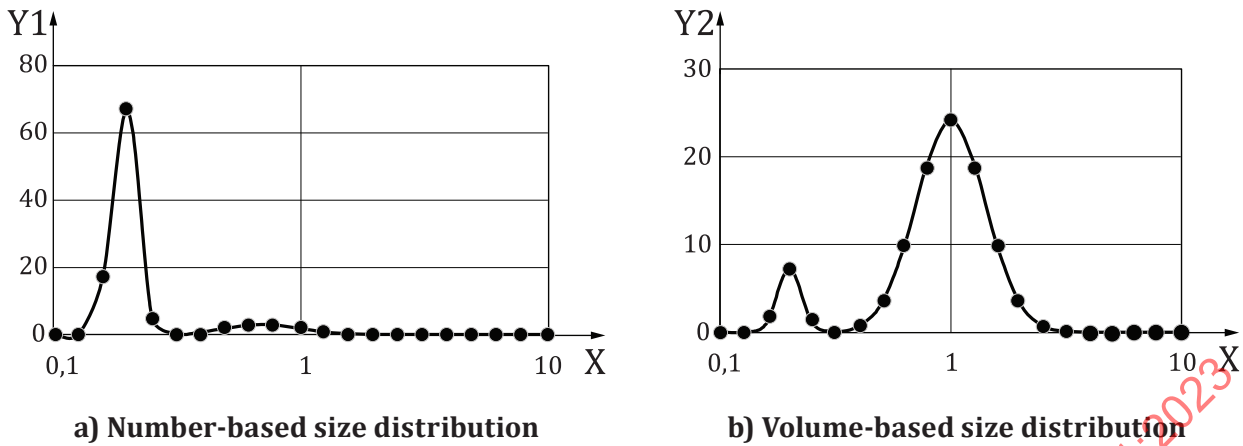
Bubble size ratio	1	10
Image		
Number ratio	1	1
Volume ratio	1	1 000

Table 2 — Volume ratio of fine bubbles 1:1 in the case of size ratio 1:10

Bubble size ratio	1	10
Image		
Number ratio	1 000	1
Volume ratio	1	1

[Figure 1](#) shows the two types of size distribution graphs of bimodal which are equivalent, but the impressions are very different as dimensions such as number and volume are different.



Key

- X particle size or bubble size (μm)
Y1 ΔQ_0 normalized number concentration (%)
Y2 ΔQ_3 normalized volume concentration (%)

Figure 1 — Example of number-based and volume-based size distribution

As shown in Figure 1, the amount of smaller fine bubbles can be emphasized in the number-based size distribution while the amount of larger fine bubbles can be emphasized in the volume-based size distribution. It can be difficult to evaluate the amount of larger fine bubbles in the number-based size distribution but it can also be difficult to evaluate the amount of smaller fine bubbles in the volume-based size distribution.

Subclause 5.1 shows the importance and the necessity of the combined use of number-based size analysis and volume-based size analysis for the sample whose size distribution is bimodal or multimodal in the broad size range.

Laser diffraction method can evaluate multimodal size distribution extended broadly from range of UFB to that of MB.

5.2 Conversion between number dimension amount and volume dimension amount

Most of the measuring instruments based on the laser diffraction method have the function to obtain both, the number dimension amount and the volume dimension amount as concentration of bubbles or particles. As the shape of fine bubbles is almost spherical, the normalized number concentration and the normalized volume concentration can be effectively mutually convertible as shown in Formulae (1) to (4).

$$y_{0,i} = \frac{\Delta Q_{3,i}}{\frac{\pi \cdot x_i^3}{6}} \quad (1)$$

$$\Delta Q_{0,i} = \frac{y_{0,i}}{\sum_{j=1}^n y_{0,j}} \times 100 \quad (2)$$

$$y_{3,i} = \Delta Q_{0,i} \times \frac{\pi \cdot x_i^3}{6} \quad (3)$$

$$\Delta Q_{3,i} = \frac{y_{3,i}}{\sum_{j=1}^n y_{3,j}} \times 100 \quad (4)$$

where

- $\Delta Q_{0,i}$ is the normalized number concentration (%) in the i th size range;
- $\Delta Q_{3,i}$ is the normalized volume concentration (%) in the i th size range;
- x_i is the mean diameter representing the i th size range.

5.3 Evaluation of fine bubble effect

FBDs can contain bubbles and contaminants whose size range extends from UFB to MB, and their size distribution shape can be bimodal or multimodal.

To evaluate the relationship between the characteristic of fine bubbles and their effects, the following possibilities about cause of effect should be considered.

- 1) Effect of smaller bubbles caused by number.
- 2) Effect of larger bubbles caused by volume which means a large amount of gas.
- 3) Synergistic effect caused by number of smaller bubbles and volume of larger bubbles.

In other cases, it should be considered that larger bubbles such as microbubbles may suppress the effect of smaller bubbles such as UFB.

So, to discuss the relationship between the characteristic of fine bubbles and their effect, the combined use of number-based size analysis and volume-based size analysis is very effective.

The characteristic of FBD can change depending on the operating conditions, the environment, the elapsed time, etc. This change can occur within a broad size range. Ultrafine bubble dispersion (UFBD) can be stable but FBD extended to broad size range containing larger microbubbles can be unstable. To discuss the relationship between the characteristic of fine bubbles and its effect, the combined use of number-based size analysis and volume-based size analysis by laser diffraction method becomes very important as the basis of this discussion. This combined use can cover the broad size range from UFBs to microbubbles.

6 Evaluation

6.1 Sampling and sample preparation

For the sampling and sample preparation of FBD in water whose upper limit of size range to be analysed is smaller than 10 μm , ISO 20298-1 shall apply.

In the case upper limit of size range to be analysed is extended to a larger size more than 10 μm , FBD in water shall be transferred into a measuring instrument in accordance with ISO 21910-1.

6.2 Procedure of the combined use of number-based size analysis and volume-based size analysis by laser diffraction method

The combined use of number-based size analysis and volume-based size analysis by laser diffraction method should be operated in accordance with the following procedure.

- 1) Select the sampling and sample preparation of the FBD sample to be analysed.
- 2) Transfer the sample into the measuring instrument.

If a stirring device, a pump and an ultrasonicator are used as the compartments included in the measuring instruments, these effects to characteristics of fine bubbles shall be minimized through the trial changing several conditions before the actual measurements.

Optimum blank water as background shall be used.

- 3) Detect any scattered light intensity distribution pattern, which are raw data of the laser diffraction method.
- 4) Calculate the volume-based particle size distribution data from the scattered light intensity distribution pattern. Generally, the laser diffraction method first obtains the volume-based size distribution data.
- 5) Convert the volume-based size distribution data to the number-based size distribution data.
- 6) Evaluate the respective distribution mode of volume-based size distribution and confirm the size and the concentration for the respective distribution mode.

If the volume of bubbles or contaminants larger than 100 μm is more than 10 % of the total fine bubbles volume to be measured, this influence to the measurement accuracy cannot be ignored. In this case, sampling and sample preparation should be reconsidered. Larger bubbles can be eliminated over time. In some cases, it can be necessary to consider the maintenance of the fine bubble generating system to reduce contaminants.

- 7) Evaluate the respective distribution mode of the number-based size distribution and confirm the size and the concentration for the respective distribution mode.

7 Test report

The information about sampling, sample preparation and transferring should refer to ISO 20298-1 and ISO 21910-1, and the information about measurement and measuring instruments should refer to ISO 13320.

The test report shall include the following information.

- a) a reference to this document, i.e. ISO 24218-1:2023;
- b) the type and number of the fine bubble generating system or device;
- c) the purity of water and gas used for the fine bubble generation;
- d) the instrument type and number of the laser diffraction method;
- e) the software version;
- f) the volume-based and number-based size distribution;
- g) the names of operator and laboratory;
- h) the date and time of the test.

The following information should also be reported regarding the respective distribution modes of both number-based size distribution and volume-based size distribution.

- a) the mean diameter of the distribution mode;
- b) the modal diameter of the distribution mode;
- c) the size range of the distribution mode (minimum diameter and maximum diameter);
- d) the concentration or normalized concentration.

Annex A (informative)

Examples of combined use of number-based size analysis and volume-based size analysis by laser diffraction method

A.1 General

While the particle size distribution obtained by the particle size analyser based on the laser diffraction method is the relationship between the particle size and the normalized particle concentration, some analysers can obtain the particle size distribution of the particle size and number or volume concentration.

In this annex, measurement results of the number-based size distribution and the volume-based size distribution by the laser diffraction method are shown as the relationship between particle size (bubble size) and concentration. As a reference, the basic concept to express the particle size distribution as the relationship between particle size and concentration is defined in [A.2](#). At the time of publication, detail of this expression process depends on the respective manufacturers.

The quantitative laser diffraction method^[1] which obtains the particle size distribution as relationship between size and concentration can be used for the assessment of protein particles.

A.2 Concept of procedure to obtain the number or volume concentration

The scattered light intensity distribution pattern is proportional to the number or volume concentration. Therefore, the particle amount calculated from the scattered light intensity distribution pattern is proportional to the number or volume concentration. When no information exists about the relationship between the calculated particle amount and the concentration, normalized concentration should be used. However, this information is obtained using a certificated reference material (CRM) such as Polystyrene latex (PSL) particles. The influence caused by the difference of refractive indices between the CRM and fine bubbles can be modified numerically based on the Mie scattering theory. In the case of fine bubbles with shell, the influence of the refractive index of the shell can be generally ignored.

In this case, the particle size distribution can be obtained as the relationship between the particle size and the concentration.

[Figure A.1](#) shows the example of the scattered light intensity distribution pattern. In this case, the particle size distribution shapes of sample A and B are identical, but the concentration of sample B is twice the concentration of sample A. Therefore, the light intensity of sample B is twice the intensity of sample A.

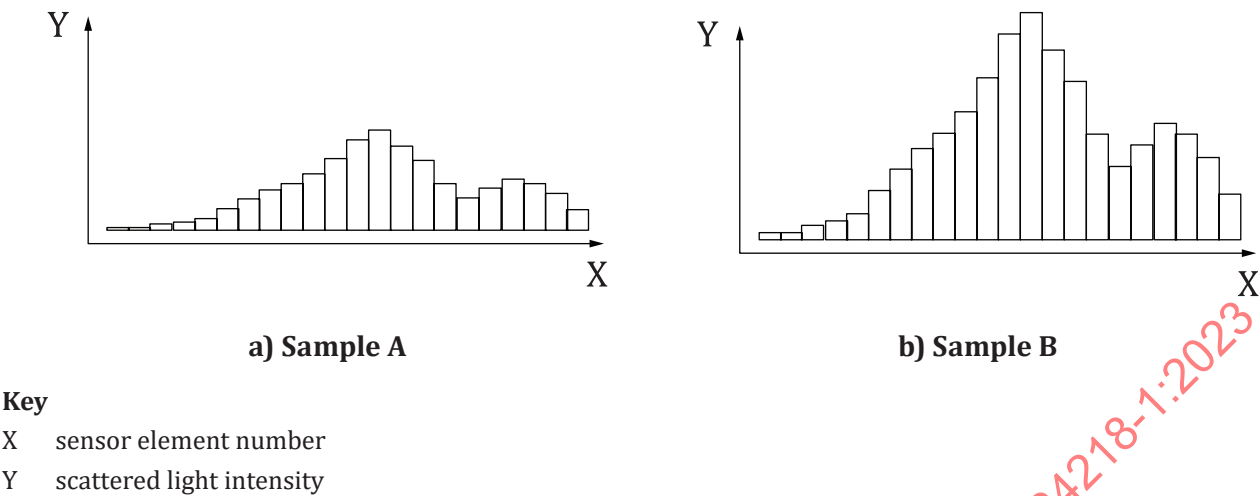


Figure A.1 — Scattered light intensity distribution pattern

Figure A.2 shows the particle size distribution calculated from scattered light intensity patterns shown in Figure A.1. The particle amount is proportional to the scattered light intensity and to the concentration. When no information of coefficient exists about the relationship between the calculated particle amount and concentration, the unit of the vertical axis of figure about the particle concentration is unknown. Therefore, the particle size distribution as the relationship between the particle size and normalized concentration expressed as percentage (%) can be shown. The information about the relationship between the calculated particle amount and concentration enables to express the particle amount as volume concentration ($\mu\text{l/ml}$) or number concentration (100 millions/ml).

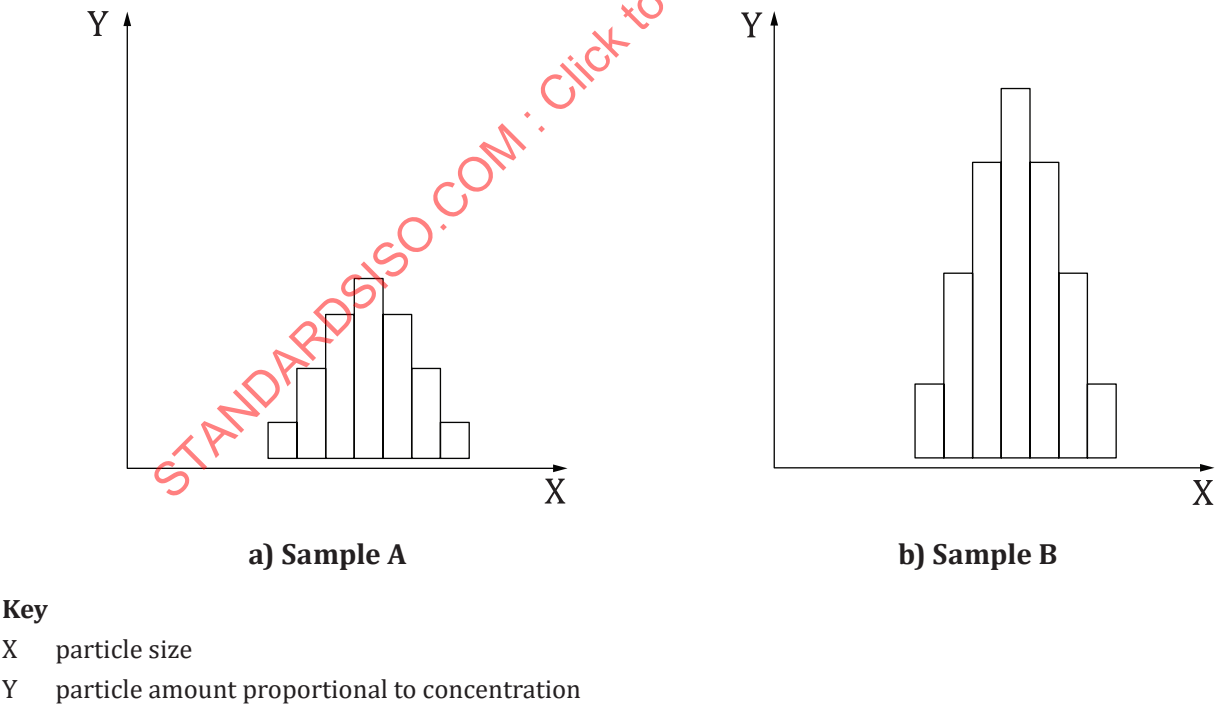


Figure A.2 — Particle size distribution proportional to concentration