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## Guidelines for the characterization of dispersion stability

*Lignes directrices pour la caractérisation de la stabilité des dispersions*

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# Contents

	Page
Foreword .....	iv
Introduction .....	v
<b>1 Scope .....</b>	<b>1</b>
<b>2 Terms and definitions .....</b>	<b>1</b>
<b>3 Basics of stability .....</b>	<b>3</b>
3.1 Stability — Summary .....	3
3.2 Characteristic features with regard to dispersion stability .....	4
3.3 Alteration of the state of a dispersion .....	4
<b>4 Characterizing the change of the state of a dispersion .....</b>	<b>6</b>
4.1 General comments .....	6
4.2 Direct methods .....	7
4.3 Correlative methods .....	8
4.4 Procedures to accelerate the evaluation of long-term stability .....	8
<b>5 Prediction of the shelf life of a dispersion .....</b>	<b>10</b>
5.1 General comments .....	10
5.2 Comparative analysis .....	10
5.3 Predictive analysis .....	10
<b>Annex A (informative) A compilation of relevant international and national standards .....</b>	<b>12</b>
<b>Bibliography .....</b>	<b>14</b>

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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. [www.iso.org/directives](http://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. [www.iso.org/patents](http://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.

The committee responsible for this document is ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

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## Introduction

Stability with respect to changes in relevant product specifications and product performance is important in industry and for end users.

Various terminologies are used to reflect different phenomena as well as different user perspectives. In the literature and in practice, one frequently finds terms such as dispersion, suspension or emulsion stability, demixing or separation stability, sedimentation or creaming stability, physical stability, colloidal stability, and kinetic stability.

This Technical Report focuses on instability driven by thermodynamics and does not include phenomena that are due to, e.g., radiation, chemical or enzymatic reactions<sup>1)</sup> or are related to the growth/metabolism of biological organisms like bacteria. These phenomena are often described as photo, UV or irradiation stability, thermal or chemical stability of one or the other constituent, enzymatic or microbial stability, etc.

The Technical Report concerns general aspects of stability test methods, acceleration procedures and data evaluation. In addition, recommendations of instrument manufacturer, information from the scientific or user community as well as from regulatory bodies are intended to be taken into account.

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1) Chemical and physical properties are often interrelated.

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# Guidelines for the characterization of dispersion stability

## 1 Scope

This Technical Report addresses the stability characterization of liquid dispersions (suspensions, emulsions, foams and mixtures thereof) for applications, such as new product design, optimization of existing products, quality control during processing and during usage of the product. The stability of a dispersion in the sense of this Technical Report is defined in terms of the change in one or more physical properties over a given time period. Stability can be either monitored (determined) in real time or predicted on the basis of physical quantities related to stability. In the case of very stable dispersions, procedures that accelerate the changes under consideration or accelerated aging tests administered over a shorter time scale can be appropriate. Shelf life can be estimated based on the observed rate of the change in the physical property and the user-required specifications for the product. Guidelines are given for choosing relevant measurements that can be used for the ranking, identification and quantification of instability.

## 2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 2.1

#### **agglomeration**

assembly of particles in a dispersed system into loosely coherent structures that are held together by weak physical interactions

Note 1 to entry: Agglomeration is a reversible process.

Note 2 to entry: Synonymous with coagulation and flocculation.

[SOURCE: ISO 14887:2000,<sup>1</sup> 3.1, modified — text altered; IUPAC Gold Book,<sup>2</sup> modified]

### 2.2

#### **aggregation**

assembly of particles into rigidly joined structures

Note 1 to entry: Aggregation is an irreversible process.

Note 2 to entry: The forces holding an aggregate together are strong, for example covalent bonds or those resulting from sintering or complex physical entanglement.

Note 3 to entry: In common use, the terms aggregation and agglomeration are often applied interchangeably.

[SOURCE: ISO 14887:2000,<sup>1</sup> 3.2, modified — text has been altered; ISO 26824<sup>3</sup>]

### 2.3

#### **coalescence**

disappearance of the boundary between two particles (usually droplets or bubbles) in contact, or between one of these and a bulk phase followed by changes of shape leading to a reduction of the total surface area

Note 1 to entry: The flocculation of an emulsion, namely the formation of aggregates, may be followed by coalescence.

[SOURCE: IUPAC Gold Book<sup>2</sup>]

## 2.4

### **creaming**

rise (separation) of the dispersed phase in an emulsion due to the lower density of the dispersed phase (droplets) compared to the continuous phase

Note 1 to entry: Creaming velocity has a negative sign as particle movement is opposite to the acting force.

## 2.5

### **dispersion**

in general, microscopic multi-phase system in which discontinuities of any state (solid, liquid or gas: discontinuous phase) are dispersed in a continuous phase of a different composition or state

Note 1 to entry: If solid particles are dispersed in a liquid, the dispersion is referred to as a suspension. If the dispersion consists of two or more liquid phases, it is termed an emulsion. A suspoemulsion consists of both solid and liquid phases dispersed in a continuous liquid phase.

[SOURCE: Hackley *et al.*<sup>4</sup>; IUPAC Gold Book,<sup>2</sup> modified]

## 2.6

### **dispersion stability**

ability to resist change or variation in the initial properties (state) of a dispersion over time, in other words, the quality of a dispersion in being free from alterations over a given time scale

Note 1 to entry: In this context, for instance agglomeration or creaming represents a loss of dispersion stability.

[SOURCE: IUPAC Gold Book<sup>2</sup>]

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## 2.7

### **flocculation**

assembly of particles in a dispersed system into loosely coherent structures that are held together by weak physical interactions

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Note 1 to entry: The term flocculation is used frequently to denote agglomeration facilitated by the addition of a flocculating agent (e.g. a polyelectrolyte).

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Note 2 to entry: See 2.1.

## 2.8

### **flotation**

migration of a dispersed solid phase to the top of a liquid continuous phase, when the effective particle density is lower relative to the continuous phase density

Note 1 to entry: It may be facilitated by adhering gas bubbles, for example dissolved air flotation, or the application of lipophilic surfactants (e.g. in ore processing).

## 2.9

### **particle**

minute piece of matter with defined physical boundaries

Note 1 to entry: A physical boundary may also be described as an interface.

Note 2 to entry: A particle may move as a unit.

[SOURCE: ISO 14644-5:2004,<sup>5</sup> 3.1.7, modified — Note 1 is different and Note 2 has been added; ISO/TS 27687:2008,<sup>6</sup> modified — Notes 1 and 2 have been altered and Note 3 has been deleted.]

## 2.10

### **Ostwald ripening**

dissolution of small particles and the redeposition of the dissolved species on the surfaces of larger particles

Note 1 to entry: The process occurs because smaller particles have a higher surface energy, hence higher total Gibbs energy, than larger particles, giving rise to an apparent higher solubility.



[SOURCE: IUPAC Gold Book<sup>2</sup>]

### 2.11

#### **phase inversion**

phenomenon whereby the phases of a liquid-liquid dispersion (emulsion) interchange such that the dispersed phase spontaneously inverts to become the continuous phase and vice versa, under conditions determined by the system properties, volume ratio and energy input

[SOURCE: Yeo *et al.*<sup>7</sup>]

### 2.12

#### **phase separation**

process by which a macroscopically homogeneous suspension, emulsion or foam separates into two or more new phases

[SOURCE: Yeo *et al.*<sup>7</sup>]

### 2.13

#### **sedimentation**

settling (separation) of the dispersed phase due to the higher density of the dispersed particles compared to the continuous phase. The accumulation of the dispersed phase at the bottom of the container is evidence that sedimentation has taken place

Note 1 to entry: In the case of a dispersed liquid (emulsion), droplets can sediment if their density is higher than that of the continuous liquid phase (e.g. water in oil emulsion).

[SOURCE: IUPAC Gold Book<sup>2</sup>]

### 2.14

#### **shelf life**

recommended time period during which a product (dispersion) can be stored, throughout which the defined quality of a specified property of the product remains acceptable under expected (or specified) conditions of distribution, storage, display and usage

[SOURCE: Gyeszly<sup>8</sup>]

## 3 Basics of stability

### 3.1 Stability — Summary

Stability is the capacity of a dispersion to remain unchanged with respect to predefined stability criteria over a given time under stated or reasonably expected conditions of storage and use. It depends therefore on the application. For instance a cosmetic emulsion may be considered stable if no oil phase formation is observed during a period of three years. On the other hand, natural fruit juice can exhibit pulp settling without any reduction in quality. There is no universal method or technique to quantify all stability aspects due to the complexity of stability related phenomena. Therefore, it is always necessary to define:

- a) stability metrics: properties of the state or behaviour of a dispersion which should be monitored according to the demanded specific product qualities.
- b) stability criteria: deviations from the initial properties at production date, which are acceptable.

Shelf life is defined in terms of the alteration of stability metrics. In general, faster alteration leads to shorter shelf life.

In order to meet the predefined stability criteria of very stable products, analytical techniques having high resolution/sensitivity need to be used and procedures can be required in order to accelerate the alteration. However, because of the interrelated physical, physico-chemical and chemical properties of a liquid dispersion, adequate acceleration methods should be chosen and validated in the context of a specific product.

### 3.2 Characteristic features with regard to dispersion stability

Generally speaking, dispersions are thermodynamically unstable.<sup>[9]</sup> However, for a dispersed system, the rate of change in its state may be acceptably low and therefore it exhibits kinetic stability. Kinetic stability may be improved by electrostatic, steric or electrosteric stabilization, or particle coating, as well as by pickering or rheological additives to the continuous phase.

The state of dispersion stability depends upon numerous interrelated physical, physico-chemical and chemical parameters, and its nature is therefore complex. The parameters may be categorized as follows:

- a) volume or mass concentration of dispersed phase (e.g. spatial homogeneity, diluted or concentrated);
- b) state of the continuous phase (e.g. density, viscosity, surface tension, chemical potential, quality of solvent);
- c) state of the dispersed phase (e.g. size, shape and density distribution, as well as viscosity of droplets, deformability of particles, structure of particulate surface);
- d) interaction between particles/droplets (e.g. electrostatic and van der Waals force, steric and depletion force);
- e) interaction between dispersed and continuous phase (e.g. wettability, interfacial tension, surface and volume rheology, solubility, dissolvability, network formation).

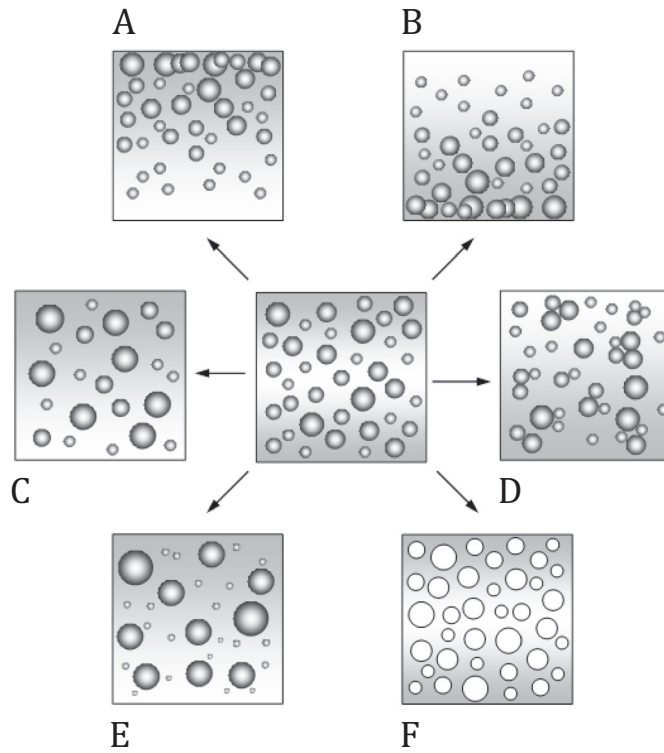
The volume concentration of the dispersed phase of a dispersion is one of the primary requirements of any product design and it should be homogenous within the entire product during the entire life span. In general, the higher the volume concentration, the higher the physical stability (e.g. less phase separation).

Formulators have to achieve product specifications and sufficient dispersion stability as demanded by the application or customer. This is accomplished by choosing the state of the dispersed phase (e.g. size distribution, shape, density match, restrictions to oversize, surface charge and coating) and the appropriate behaviour of the continuous phase. Traditionally, electrostatic stabilization has been principally used. Today, polymeric additives are commonly employed to tailor properties of the continuous phase of innovative products. Two essential aspects with regard to dispersion stability are particle-particle interactions and interactions between the dispersed and continuous phase. Tuning of particle interactions is an important tool to stabilize a dispersion. Electrostatic, steric and depletion stabilization or combinations of these are the most commonly used approaches. The theoretical foundation of these approaches is based on the classic DLVO (Derjaguin, Landau, Verwey, Overbeek) theory (see Overbeek<sup>[10]</sup>) and more recently, the extended DLVO theory.<sup>[11]</sup> In general, any specific interaction energy between two particles (e.g. double layer interaction, van der Waals attraction, steric interaction) is calculated as a function of the particle distance. The dependence on the distance is interaction specific. The different interaction energies are additive and the resulting energy-distance curve allows for stabilization evaluation.<sup>[11]</sup> It should be emphasized that products today (e.g. paints, nutritional suspoemulsions, cosmetic multiple emulsions) often consist of several dispersed phases, and that the continuous phase may contain many constituents.

This complex structure of dispersions implies that a single parameter is generally insufficient to characterize or predict the stability state of a dispersion.

### 3.3 Alteration of the state of a dispersion

[Figure 1](#) and [Figure 2](#) schematically display a selection of primary and secondary mechanisms, respectively, which, over time, change the state of the dispersed phase and/or homogeneity of the dispersion. They are indicators of loss of stability. Additionally, aged dispersions may undergo phase separation that is obvious by visual observation. Destabilization mechanisms are sequenced simply for the sake of clarity and cannot be distinguished in most practical cases.



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**Key**

- A creaming, flotation
- B sedimentation
- C coalescence
- D flocculation, agglomeration
- E Ostwald ripening
- F phase inversion

Modified from source. [12][13]

**Figure 1 — Primary destabilization phenomena of the state of liquid-solid or liquid-liquid dispersions**