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Cosmetics — Guidelines on the stability testing of cosmetic products

Cosmétiques — Lignes directrices relatives aux essais de stabilité des produits cosmétiques

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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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This document was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

Introduction

Stability studies are aimed at assessing the ability of a product to maintain the desired physical, chemical and microbiological properties, as well as functionality and sensorial properties when stored and used under appropriate conditions by the consumer. More simply, the objective of a stability study is to determine the shelf life of a product and to evaluate whether a product in the package is stable when subjected to the market conditions in which it is sold and used. The "market conditions" encompass distribution (transportation), warehouse storage and conditions during use.

Thus, the stability study may be seen as a prerequisite for ensuring product quality. Stability tests on cosmetic products are required for

— obtaining a guidance on the formulation of the product, and the appropriate packaging material,

— optimizing the formulation and manufacturing process,

— determining conditions of transportation, storage, display and manner of use,

— estimating and confirming shelf life, and

— ensuring customer safety.

This document identifies readily available references to assess the stability of cosmetic products on the market. Its purpose is to provide a resource for the selection of the appropriate stability tests. Although these guidelines provide a helpful starting point to evaluate new products and technologies, adapting the testing to reflect differences between product types and formulations may still be necessary.

Cosmetics — Guidelines on the stability testing of cosmetic products

1 Scope

This document gives guidelines for the stability testing of cosmetic products. It reviews readily available bibliographic references that provide a resource for the assessment of the stability of cosmetic products. This review of the available guidelines that assess the stability of cosmetic products can serve as a technical/scientific framework to identify the most suitable methods for the assessment of the stability of cosmetic products.

This document does not aim to specify the conditions, parameters or criteria of stability testing.

Considering the wide variety of cosmetic products, storage and use conditions, it is not possible to define a single way to assess product stability. Therefore, it is up to the manufacturer to specify and justify the stability protocol to cover test methods, specifications and conditions at which products will be tested.

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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
accelerated stability evaluation
study designed to speed up naturally occurring destabilization processes due to intrinsic or extrinsic factors and which predicts the behaviour over the long term

Note 1 to entry: Typically, physico-chemical, mechanical or thermal procedures are employed.

3.2
real time stability evaluation
study that monitors the state of a product to determine the time course of any alteration to it under reasonably expected conditions of storage and use

Note 1 to entry: Often called “long term test” or “standard stability test”.

3.3
stability
ability of a cosmetic product to resist change or variation of its initial properties over time under stated or reasonably foreseeable conditions of storage and use

Note 1 to entry: See Reference [1].

3.4
stability criteria
deviations from initial properties or behaviour at production state, which are acceptable

Note 1 to entry: See Reference [1].

3.5
stability metrics
properties/parameters of the state or behaviour of a cosmetic product which should be monitored according to demanded, specific product qualities

Note 1 to entry: See Reference [1].

3.6
shelf life
recommended time period that a cosmetic product can be kept after its production, during which the defined quality of the product remains acceptable under expected conditions of distribution, storage, display and usage

Note 1 to entry: See Reference [1].

4 Basic principles of cosmetic stability

Design, formulation and the manufacturing process of cosmetic products have to fulfil general and specific demands and requirements of distribution pathways, and especially of customers. Specification, functionality and aesthetics have to be preserved, i.e. have to be stable, over the entire life cycle of a product.

Processed cosmetic products are complex matrixes which undergo spontaneous alterations to reach the free energy minimum in accordance with the second thermodynamic law.[2] These so-called intrinsic causes may be of physical, physico-chemical or chemical origin.[3] Many reactions and processes may lead, under the condition of the "market", to a deviation from the initial, original product properties at the date of manufacturing. Departure may be caused by thermodynamically driven internal or externally driven effects,[1] microbiological impact[4][5] or interactions with packaging[6] and may finally lead to a loss of specified product functionality or/and aesthetic attributes. This impacts usability, shelf life and marketability. Destabilization processes of the original product may also be provoked, enhanced or magnified due to extrinsic (external) factors. For example, state changes may be triggered by thermal energy loss or gain, light and UV irradiation, mechanical energy input (such as vibration or pressure), oxygen uptake, humidity, interaction by or with container/closure system (packaging), and proliferation of microorganisms.[3]

Cosmetics are of different natures and some consist of several phases, dispersed ones and a continuous one, and may be classified as suspensions or emulsions. Suspensions are solid particles dispersed in a liquid phase. Emulsions are composed of two liquid phases, typically oil-in-water (o/w) or water-in-oil (w/o). Cosmetic dispersions are usually very complex and may contain several phases.

A stability test aims at providing information on the state/behaviour of the cosmetic products in the container/enclosure under the different conditions to which they may be subjected from their manufacturing date until the end of their recommended period of use.[7][8][9] The state of a cosmetic product and its stability depend upon numerous interrelated physical, physico-chemical and chemical parameters as well as interaction with environment, and its nature is therefore very complex. In general, they may be roughly classified as mechanical-driven, thermal- or diffusion-driven, interaction-force- driven or externally provoked processes.[1][7][10][11] There is no universal method or technique to quantify all stability aspects due to the complexity of different pathways of destabilization. Therefore, it is always necessary to specify precise stability metrics and acceptance criteria.

After determination of the stability metric(s), it is necessary to select appropriate stability test methods to monitor the alteration of the product over time. It is recommended to select methods that do not require sample preparation (e.g. dilution) and quantify kinetics of the product destabilization based on defined metric(s) in a direct way.[1] Real time measurements can be made by traditional visual

observations, sensory techniques or use of different measuring techniques which are advantageous in being quantitative, objective, traceable, reproducible and retrievable.[1] Scanning and spatially resolving techniques are appropriate in particular to detect phase separation and help to discriminate between phase separation and phase changes.[1][7][10][11]

In the case of very stable products, analytical techniques having high resolution/sensitivity should be used and procedures can be required in order to accelerate the alteration to shorten the detection time to meet the predefined stability criteria.[1][11] Typical acceleration approaches are mechanical ones (e.g. high gravity by centrifugation) or elevated storage temperatures for a given time. Preservation efficacy testing (challenge-test) aims to test microbiological attributes under accelerated conditions.[3][5] However, because of the interrelated physical, physico-chemical and chemical properties of cosmetic emulsions or suspensions, adequate acceleration methods may be chosen and verified in the context of a specific product.[1][11]

Beside direct methods, correlative test methods are utilized focusing on determination of a single parameter of the state of a product that is known to correlate with product stability.[1] Typical parameters measured and compared with pre-defined acceptable values are, for example, mean particle/droplet size or zeta-potential. These quantities reflect the state at the time of the measurement and do not yield kinetic information. Such test procedures may be used for quality assessment of a specified product but, due to the complexity of the state of cosmetic products, currently no theoretical basis exists to predict the time course of any product alteration based on a single parameter obtained at any single time point. In addition, most of these measuring techniques demand sample preparation and often product dilution.[1]

Due to the cosmetic product formulation's complexity overall, caution should be employed in stability assessment and predicting its shelf life, and the properties of the constituents should be considered and understood, as well as the product's behaviour and the factors which influence it.[11]

5 Aspects to be addressed during stability testing

5.1 General

The key aspects to consider when assessing the stability of cosmetics products are listed in 5.2 to 5.6. Guidance is provided based on the review of relevant sources cited in the text.

5.2 Stage/scale of tested batches

When working on a brand new product (new formula, new manufacturing process, new packaging) and therefore where no significant body of knowledge is available, it might be appropriate to carry multiple independent stability studies, usually referred as "preliminary tests". It is up to the manufacturer to decide on the number of batches, the scale of their manufacture that are subject to stability testing and at what stage of the project development cycle this takes place.[7][12][9][13]

The stability of the final product (final formula, final manufacturing process, final packaging) may be demonstrated before its commercialization. Accelerated stability evaluation may be conducted prior to commercialization to predict product shelf life. Following commercialization, the shelf life confirmation is obtained by long term stability testing on representative formulations.

Minor variations between products subjected to stability testing versus final commercial product can be acceptable if they are deemed as not having an impact on product characteristics and integrity, but need to be documented and justified.[9]

Common acceptable practices include but are not limited to

— usage of formulation samples from the development stage for preliminary tests,

— usage of pilot batches instead of production scale, for some specific non-scale up products and where the manufacturing process is considered equivalent,[13][14] and

— usage of unlabelled packaging, or packaging of different shape, when packaging size, closure system and material remain unchanged, or when decorative material remains of same nature but with different design (e.g. same ink versus different print).

Additionally, matrixing and bracketing[14] are acceptable practices but should be documented and justified to ensure that the stability study protocol will cover the entire product portfolio and provide enough information (e.g. multiple shade, packaging size).[13][14][15]

5.3 Test procedures and conditions

5.3.1 Principle

The general objective of a stability test is to determine whether a given product, in the container in which it is being marketed, has an adequate shelf life, under the conditions of the market in which it is being sold.[16]

Stability testing may be of sufficient duration to cover storage, shipment and subsequent use, and to guarantee safety and quality.

Manufacturers may, for each formula type, select the pertinent test criteria according to their experience and evaluate these test criteria at one or more temperatures.

Because of the wide variety of cosmetic products and their inherent complexity, standard stability tests cannot always be prescribed.[7] Both real time and accelerated test procedures are used to provide the desired information. Most cosmetics, due to their short development cycles, require accelerated test protocols to help predict stability parameters in a shorter period of time. To achieve these end points, some samples are kept under test conditions designed to accelerate changes that may occur and some are stored at normal conditions. Test conditions refer to the various manufacturing or storage conditions (e.g. batch) or combination of conditions (e.g. container closures) to be studied.[10] The recommended shelf life may be estimated by accelerated stability tests and can be confirmed by real time (long term stability) tests.[13]

While shelf life is determined by the stability of the product, the overall stability profile is made up of several components, such as

— the inherent chemical and physical stability of the product, and

— the possible interactions between the product (contents) and its primary packaging.

In order to generate the desired information recommended, standard/long term and accelerated test parameters are outlined in 5.3.2 to 5.3.6.

5.3.2 Temperature and humidity

NOTE See References [10] and [16].

Accelerated and long term conditions can be used. The long term condition is conducted at regular storage conditions (e.g. controlled room temperature), while the accelerated testing is conducted under stress conditions (e.g. elevated temperature) which aims to increase the rate of potential degradation.[3][7][8][9][10][12][13][16]

Throughout this document it is assumed that all temperature and humidity storage conditions might experience a variation, for example ±3 °C and ±5 % RH.

Accelerated test conditions may vary and should be established based on correlations to real time storage conditions for the specific region or market. References to commonly used accelerated test conditions for testing cosmetic products are provided in the Bibliography. The temperature used and the duration will depend on the product type.[3][7][10][13][16][17][18][19]

Samples stored at elevated temperatures represent a more constant degree of acceleration and render stability predictions as being more accurate. Cosmetic stability guidelines list various storage conditions and durations for accelerated stability testing:[3][16][17][19]

— (30 ± 2) °C;

— (37 ± 2) °C;

— (40 ± 2) °C;

— (45 ± 2) °C;

— (50 ± 2) °C.

Durations range from one week to three months. Relative humidity may be ambient[16] or controlled, such as 37 °C to 40 °C/75 % to 80 % RH.[9][17]

Alternative temperature and humidity conditions may be used, including an intermediate condition of (30 ± 2) °C and 65 % RH.[9][14][20] Test conditions and durations may be adjusted where justified to cover the product's distribution and storage conditions.[9][18]

Instability can be caused by either chemical reactions or physical processes and often a combination of both. These alterations proceed at a faster rate at higher temperatures but the degree of acceleration is variable as it depends on the specific rate constants, which are often unknown. Care should be taken in the interpretation of results when using temperatures far removed from ambient, as the observed changes may never occur at normal in market temperatures. Use of moderate elevated temperature, e.g. 37 °C to 40 °C, is a more realistic condition for predicting in market stability.[10][17]

Tests at low or elevated relative humidity are normally tests of the package and not of the product. They serve either to show the effect of storage at varying humidity on the container or as a measure of the barrier properties of the container. Products may be adversely affected by atmospheric humidity but if this happens in the product in its sale package it indicates that the package provides inadequate protection from the atmosphere.

Tests at elevated humidity are less likely to accelerate changes at normal storage conditions compared to tests at elevated temperatures and ambient humidity. If absorption of water vapour presents a risk to the packaged product's properties, then testing at elevated humidity may accelerate changes. If the risk is loss of water or other volatile constituents (such as in permeable packaging), then elevated humidity may actually retard changes, and testing at low humidity may be more appropriate.

It may be appropriate to consider low temperature storage during stability testing.

— Refrigeration at 5 °C (2 °C to 8 °C) / ambient humidity: This condition may be used to store samples to be used as reference samples.[3]

— Freezer at −5 °C to −10 °C: This condition may be used to determine the effects of extreme low temperature, for example during transportation.[3]

5.3.3 Cycling of temperature and/or humidity

Tests in which the temperature and/or humidity are changed at regular intervals, and which subject the package to variations other than static stresses, are sometimes more severe tests than continuous storage at one condition. These tests provide evidence of emulsion stability, tendency to crystallization, deposition or clouding, and whether the reaction is reversible. This data are also applicable to determining how robust a product is to extreme fluctuations in temperature during distribution and storage.[3][10][16][19]

Freeze/thaw tests are applicable to

— liquid products as a measure of the potential to develop crystallization or cloud formation, and

— emulsions or creams as an indicator of emulsions stability.