GUIDELINES
ON STABILITY TESTING OF COSMETIC PRODUCTS

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I. GENERAL CONSIDERATIONS

1. INTRODUCTION

General

The purpose of stability testing cosmetic products is to ensure that a new or modified product meets the intended physical, chemical and microbiological quality standards as well as functionality and aesthetics when stored under appropriate conditions.

Because the development cycle of cosmetic products is relatively short, and also, in order that the testing activity does not become economically disproportionate in view of the multitude of product launches each year, each manufacturer must have at their disposal tests that are adapted to their activity. Each manufacturer should design their stability testing program such that it is reasonable and efficiently addresses the testing required.

This document aims to set out guidelines in order to predict and assure the stability of products in the market place. Its purpose is to aid manufacturers of cosmetic products in the selection and the refinement of the appropriate stability tests. Although this guideline can provide a helpful starting point, it is important that manufacturers carefully evaluate new products and technologies and, where appropriate, adapt their testing to reflect differences between product types and formulations.

However, all methods assuring the final stability of a cosmetic product against the categories cited in paragraph below, if it is at least equivalent to the recommendations of the following chapters, are considered as valid. Procedures must be put in place and documented within the manufacturer's internal system.

General Stability of a Cosmetic Product

Whether conducted in real time or under accelerated conditions, tests should be done in order to assure:
- Stability and physical integrity of cosmetic products under appropriate conditions of storage, transport and use,
- Chemical stability,
- Microbiological stability,
- The compatibility between the contents and the container.

Moreover, concerning methodology, the manufacturer should, for each formula type, select the pertinent criteria according to their experience and evaluate these at one or more temperatures. The evolution of the test parameters should then be judged by the corresponding expert and a decision made for each criterion on the basis of the company's internal procedures and experience. An evaluation, based on all of the criteria should then allow for the predicted or real stability of the product to be deduced.

Because of the wide variety of cosmetic products and their inherent complexity, "standard" stability tests cannot be prescribed. Manufacturers, who have an intimate knowledge of their products and packages, require the flexibility to modify testing protocols and to build a sound scientific basis for assessing product stability. Thus, specific tests may be developed in order to predict possible evolutions of the product, to address new/ unusual technologies, or to be adapted to products having extended shelf lives.

Accelerated Stability Tests

Accelerated tests, developed because of the relatively short development cycle for cosmetic products, enable the prediction of stability. A commonly accepted practice is to support the forecasts obtained from accelerated stability testing by carrying out periodic post-launch monitoring of retained samples stored at ambient temperatures. The resultant information can also be useful in further improving the product and in refining the methodology used for accelerated stability testing.

2. DESIGNING A COSMETIC STABILITY STUDY

A stability study should include the following considerations (each of which will be discussed in more detail later):
- Identify tests that will "accelerate and predict" the effects of normal conditions of storage and use.

Where relevant, consider stresses, including temperature, that will enable assessment of product integrity under anticipated product exposure conditions.
- Consider evaluation of critical aesthetic properties such as color, fragrance, texture, and flow, particularly after exposure to conditions designed to stress each specific property.
- Consider variation in process conditions.
- Consider the impact of packaging on the contained product, as well as any effects which the product might have on the packaging.

3. PREDICTING SHELF LIFE

There is very little generally-applicable published research to support specific accelerated methods for predicting cosmetic shelf life. Some of the reasons for this lack of information are:
• The variety and complexity of cosmetic formulas and packaging.
• The proprietary nature of many products and stability test methods.
• The variety of types of changes that need to be examined, including physical, chemical, microbial, functional or aesthetic changes.

"Accelerated" Conditions

Accelerated test conditions are internationally recognized as appropriately predicting product shelf life in many industries.

Appropriate conditions, for example with regard to temperature and/or duration, should be chosen according to the product category and the in-house know-how and should be based on sound scientific judgment. Data acquired using various techniques at different temperatures and durations can be used, possibly in conjunction with the use of mathematical models, to predict stability**.**

Tests are often performed at 37°C, 40°C or 45°C during 1, 2, 3… months but the temperature used and the duration will depend on the product type. For instance, for certain product categories other temperatures may prove to be more useful.
Based on the accelerated stability results and on its scientific experience, a company may then be able to predict the actual stability for given market conditions.

Scale-Up Stability Testing

In the early stages of product development, initial determinations of product stability can be made by testing samples from laboratory batches under appropriate conditions. As development progresses, it may be necessary to make more precise determinations of probable stability by testing samples from batches manufactured with equipment representative of that which will be used to manufacture the commercial product. Factors such as equipment type and configuration, process, batch size and product type need to be considered when developing a stability program.

Shade and Fragrance Variations

It is advisable that stability tests include a representative range of shades and/or fragrances which are judged to represent the stability extremes of the product. If any of these extremes yield results which are unsatisfactory or questionable, additional shades or fragrances should be tested.
In certain circumstances, It may be advisable to test all fragrance variations, because fragrances are more likely to interact with the product than are color shades. Also, some cosmetics come in so many shade variations that testing all shades would not be practical.

Packaging

Packaging can directly affect finished product stability because of interactions which can occur between the product, the package, and the external environment. Such interactions may include:
• Interactions between the product and the container (e.g. adsorption of product constituents into the container, corrosion, chemical reactions, migration);
• Barrier properties of the container (its effectiveness in protecting the contents from the adverse effects of atmospheric oxygen and/or water vapor, and in ensuring the retention of water and other volatile product constituents).

Stability testing should include packaging which is made of exactly the same material(s) and is as similar as possible in all other respects to the package in which the product will be marketed. If the product will be marketed in several different package types, it is advisable to study each package type. Where there is a range of package sizes, it is advisable to test the product in the smallest container. Appropriate controls (for example, product in glass containers) should be used. It may also be advisable to test the packaged product in various orientations (upright, inverted, on its side, etc.)

Parameter variability during product shelf life

When designing a stability test protocol, it is important to bear in mind that as products age, their properties may change. Because of this fact, stability testing may need to involve testing of properties beyond those which will be evaluated for initial release testing. The assessment of the following criteria is given as an example and is neither exhaustive nor a minimum requirement as the tests will depend on the product category and the packaging type:

- Color, odor and appearance,
- Changes in the container,
- pH,
- Viscosity,
- Weight changes
- Microbial tests demonstrating the ability of the products to prohibit microbial growth during normal use and other specific tests if necessary,
- Analytical data in relation to other parameters for specific product types

Confirmatory Monitoring

As a general rule, it is advisable to employ confirmatory ("real-time") monitoring to gain additional assurance that the accelerated testing is truly predictive of market stability.
Such monitoring should be carried out on the actual production product in the final package. Testing can be very simple (for example periodic visual monitoring of production retains stored at ambient temperature), or it can be more complex (conducting stability studies of production retains under controlled temperature conditions which parallel those used in the accelerated testing). Manufacturers may consider supplementing the information gained with such data as trade surveys and consumer feedback.

4. PREDICTING FUNCTIONALITY UNDER STRESS CONDITIONS

This section describes approaches to predicting how well cosmetics will resist common stresses such as temperature extremes and light. Typically, manufacturers determine whether to perform such specialized testing based on the vulnerabilities of the particular cosmetic product and its anticipated shipping, storage, display and use conditions.

Temperature Variations and Extremes

Temperature cycling and/or "freeze-thaw" tests can reveal some types of inadequacies more quickly than can storage at a constant temperature. Freeze-thaw testing should be considered for certain types of products.

Examples of problems which can be detected by freeze-thaw testing include suspension problems (a tendency to crystallize or cloud), instability of emulsions and creams, package design issues (such as wrinkling or loss of labels, cracking or distortion), corrosion of internal lacquers in aluminum tubes, etc

As products can be expected to encounter temperature and pressure extremes during transport and storage, stability testing at these extremes should be considered, for example :

- Low-temperature testing, as well as freeze/ thaw testing.
- High-temperature testing.

Mechanical and Physical Tests

Mechanical shock testing is often conducted in order to determine whether or not shipping movements may damage the cosmetic and/or its packaging. Vibration testing can help to determine for example whether de-mixing (separation) of powders or granular products is likely to occur.

Light Stability

Cosmetics whose packaging may allow the product to be exposed to light should undergo light stability testing. The lighting used in testing should simulate the intensity to which the cosmetic will likely be exposed.

II. DESIGNING A STABILITY STUDY: A PRACTICAL GUIDE

General

The following section outlines a logical approach to designing a stability study which highlights key considerations. In reality, however, it is most likely that companies will have already set up programs for each product category based on their own experience (which would nevertheless fit with these principles). This step by step reflection would therefore only be applied to totally novel products or by companies performing stability studies on a particular category of cosmetic products for the first time.

- Consider that throughout the design process the basic principle is to provide data that will assure that the product continues to fulfill its initial function and be of appropriate quality through the course of its intended usage.

- Consider the characteristics/specifications of the product and define clearly what will be considered as acceptable stability, as this may vary across the wide range of cosmetic products and will be dependent on each company's internal procedures and experience.

- Consider the points in the process where the appropriate level of confidence has been reached. The fast cycle time for cosmetic products means that while confirmatory studies are continued a well-designed study should use experience and scientific expertise to answer the key questions about product stability.

- Consider existing data on similar products with similar packaging.
  The study of past data may give indications as to what areas of product stability the new program should focus to increase the effectiveness and efficacy of the program.

Specific Considerations

- Determine the parameters to be investigated in the study. These should be relevant to the product type, its specific formulation and package and should take into account any specific legislative requirements. Not all parameters may need to be investigated at every sampling stage if this is reasonable based on experience and scientific principle.

- Determine the relevant conditions under which the product could be stored. These should reflect the normal expected exposure of the product during its life cycle. In addition to testing product in the selected packaging, controls may be considered.

  Throughout their life cycle products may be exposed to a number of different conditions during their storage, transport, retail and finally use by the consumer. A stability program should reflect the most likely conditions that will be experienced and attempt to replicate them. Possible areas to be considered may be temperature, light, humidity or physical effects.

- Determine the packaging in which the product should be stored in order to simulate actual marketed products as closely as possible.

- Determine what characteristics and potential interactions of the product and package should be inspected and/or tested. Consideration of the formulation and the package type, size and material should lead to identification of possible areas of interaction that may need to be investigated.

- Determine the type of batch to be tested (e.g. laboratory, pilot, or production) so that the product may be representative of commercial production.

- Determine the range of product shades or fragrances that need to be included in the study. Where there are a large number of shades or fragrance variants it may be appropriate to run stability on a subsection of the total product line.

- Determine the frequency of product inspection/testing. It is important to work out what the appropriate frequency of product sampling and testing is throughout the stability program. Adequate amounts of product should be placed to meet these requirements with significant over provision in case retesting is required.

- Determine the nature of any controls that should be included. The choice of products to place on stability to compare against, if required, should be based on the understanding of the stability of those controls and should be set up to investigate specific parameters.

- Ensure that plans are in place for the collation and storage of stability data and have a process for next steps based on this data.

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