STABILITY ASPECTS OF COSMETICS

STABILITY: It is defined as time lapse during which the drug or cosmetics products retain the same property and characteristics that it possessed at the time of manufacture.

SHELF LIFE: It is time required to reduce the concentration of the reactant to 90% of its initial concentration.

FACTORS THAT INFLUENCE STABILITY

These are related to external factors to which the product is exposed, such as:

a) **Time**: The time near of the expiry date of the product can lead to alterations in the organoleptic, physical-chemical, microbiological and toxicological characteristics.

b) **Temperature**: High temperatures accelerate physical-chemical reactions, generating alterations in component activity, viscosity, appearance, color and odor of the product.

Low temperatures accelerate possible physical reactions such as turbidity, precipitation and crystallization. The speed of many reactions increases about two or three times with every 10◦ rise in temperature.

Problems created by high or very low temperatures can also derive from non-conformity during the manufacturing process, storage or transport of the product.

c) **Light and Oxygen**: Ultraviolet light along with the oxygen leads to the formation of free radicals and sets in motion oxidation-reduction reactions. Products that are sensitive to light action must be conditioned away from light, in opaque or dark flasks and must also have antioxidant substances added to the formulation for the purpose of retarding the oxidation processes.

UV rays with wavelength below 320nm are responsible for therapeutic as well as noxious effects caused due to sunlight.

Titanium dioxide is used as a white pigment for cosmetics but undesirably induces a certain degree of decomposition of sebum on the skin on exposure to UV radiation in sunlight.

Colourants such as lakes, inorganic dyes and synthetic dyes should be stored in well closed, light resistant containers at a temperature below.

d) **Humidity**: This factor affects mainly the solid cosmetic forms, such as powder, bar soap, eye shadow, bath salts.

Before initiating the stability studies it is recommended that the sample be centrifuged at 3000 rpm for 30 minutes. The product must remain stable and any sign of instability shows the need for reformulation. If approved in this test, the product can then be submitted to the stability tests.

PRELIMINARY STABILITY TEST

This test is also known as the Screening Test, or Short Term. The study of preliminary stability consists of making the test in the initial phase of product development using different laboratory formulations and with a reduced duration. It uses extreme temperature conditions with the objective of accelerating possible reactions among the components and the appearance of signs that must be observed and analyzed according the specific characteristics of each type of product.

PROCEDURE

It is recommended that samples for the evaluation of stability be placed in neutral, transparent glass flasks with a lid that assures good closing, avoiding gas or vapor losses to the environment.
It is important not to fill the total volume of the package, allowing a head space of approx one third of the capacity of the flask for possible gaseous exchanges.
The duration of the study is generally fifteen days and helps in the screening of the formulations.
The formulations under test are submitted to stress conditions aimed at accelerating the appearance of signs of possible instability. Generally the samples are submitted to heating in ovens, cooling in refrigerators and to alternated cooling and heating cycles.
The values generally adopted for elevated temperatures can be:
Oven: Temp = 37 ± 2° C
Oven: Temp = 40 ± 2° C
Oven: Temp = 45 ± 2° C
Oven: Temp = 50 ± 2° C
The values generally adopted for low temperatures can be:
Refrigerator: Temp = 5 ± 2° C
Freezer: Temp = -5 ± 2° C or Temp = -10 ± 2° C.
The values generally adopted for the cycles are:

Cycles of 24 hours at 40 ± 2° C, and 24 hours at 4 ± 2° C - during four weeks.

Cycles of 24 hours at 45 ± 2° C and 24 hours at –5 ± 2° C – during 12 days (6 cycles).

Cycles of 24 hours at 50 ± 2° C and 24 hours at – 5 ± 2° C – during 12 days (6 cycles).
In this type of study, the samples are stored under different temperature conditions for regularly alternated time intervals.
Preliminary study is for evaluation to be made at the very beginning and then during all the days in which samples are submitted to the study conditions.
The evaluation generally is of:
Organoleptic characteristics: appearance, color, odor and flavor
Physical-chemical characteristics: pH value, viscosity, density.
A reference sample must also be taken, also denominated as the standard sample, which generally can be kept in the refrigerator or at room temperature, protected from light.

ACCELERATED STABILITY

Also known as normal or exploratory stability, has the object of providing data to forecast the stability of the product, its useful life span, and the compatibility of the formulation with the containing material. It is a predictive study that can be used to estimate the expiry date of the product.
PROCEDURE
Samples for the evaluation of stability should be placed in neutral, transparent glass flasks with a lid that assures good closing, avoiding gas or vapor losses to the environment. The quantity of product must be sufficient for the necessary evaluation.
The incorporation of air in the product must be avoided during placement in the test recipient.
It is important not to fill the total volume of the package, allowing a head space of approximately one third of the capacity of the flask for possible gaseous exchanges.
The final containing material may be used parallel to the neutral glass thus anticipating the appraisal of compatibility between the formulation and the package material.

The duration is generally of 90 days and the test formulations are submitted to conditions less extreme than in the Preliminary Stability Test. In some cases, the duration of this test can be extended for 6 months or even one year, depending on the type of product. The samples can be submitted to heating in ovens, cooling in refrigerators, exposure to light radiation and to the environment.
The values generally adopted for elevated temperatures can be:
Oven: Temp = 37 ± 2° C
Oven: Temp = 40 ± 2° C
Oven: Temp = 45 ± 2° C
Oven: Temp = 50 ± 2° C
The values generally adopted for low temperatures can be:
Refrigerator: Temp = 5 ± 2° C

Freezer: T = -5 ± 2° C, or T = -10 ± 2° C

Exposure to light radiation:
This may alter the color and the odor of the product and lead to the degradation of formulation ingredients. In conducting the study, the light source can be sunlight captured through glass panels specially designed for the purpose or lamps that have an emission spectrum similar to that of the sun, such as xenon lamps. Ultraviolet light sources are also used.
The samples must also be submitted to Accelerated Stability Test accommodated in the containing material.
The products must be stored under more than one temperature condition, so that behavior can be evaluated for the many environments to which they may be submitted.
However what is most usual in the accelerated study is for evaluation to be made at the very beginning, after 24 hours and then on the 7th, 15th, 30th, 60th and 90th days. If the study is to be extended then a monthly evaluation is recommended until the end of the study.
The parameters to be evaluated must be defined by the formulator and depend on the characteristics of the product that is being studied and on the ingredients being used in the formulation. Generally they are:
organoleptic parameters: appearance, color, odor and flavor, whenever applicable;
physical-chemical parameters: pH value, viscosity, density, among others;
microbiological parameters: microbial count and challenge test of the preserving system made before and/or after the accelerated study period.
A reference sample must also be taken, also known as a standard sample, which generally can be kept in the refrigerator or at room temperature, protected from light. In a complementary manner, samples from the market of products with a known acceptability or of other similar products deemed to be satisfactory in relation to the parameters being evaluated, may be used as standards.

SHELF TEST or Long-term Stability Test

The aim of the Shelf Test is to validate the stability limits of the product and to test the expiry date estimated using the accelerated stability test.
This study is carried out over a period equivalent to the time of expiry estimated during the stability studies previously mentioned. It is used to evaluate the behavior of the product under normal storage conditions.
The frequency of the analyses must be determined according to the product, the number of the batches produced and the estimated expiry date, and if the intention is to extend the expiry period then the follow up process can be continued.

PROCEDURE

In the shelf stability study, the representative samples of the product are stored at room temperature. The number of samples must be sufficient to allow for the carrying out of all the tests foreseen in the study. These samples are analyzed periodically up until the expiry date. The same tests suggested in the previously mentioned procedures must be done and others may be defined by the formulator according to the characteristics of the formulation.