Stability Operations

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The measurement of product stability (shelf life testing) is a requirement of sound product development. The controlled measurements integral to such programs establish useful product life, minimum packaging requirements and proper storage and shipping conditions. This supports and complements the R&D, quality control and manufacturing efforts required to bring a product to market.

Potential marketing expenditures coupled with costly and sometimes limited resources make product development a major expense. Stability guidelines for product, raw materials and packaging need to be established early in the development cycle. If the best product on the market will only last a very short time on the shelf, how can it be a part of a marketing strategy to win customers, build a brand and be associated with a company identity?

How then does a company proceed to develop a product in a cost-conscious manner? The process starts while the concept of the product is still within the R&D group. Through stability operations (a planned series of chemical and sensory testing) data are developed that document the facts needed to formulate, package, ship and store the embryonic product or current product line.

A properly designed program establishes shelf-life guidelines for raw materials, i.e., ingredients such as flavors, sweeteners and fats, that can degrade and lessen the quality of the product. It will establish the formulation with the longest shelf life and the easiest-to-preserve product integrity, when produced in a quality manner and packaged in the most functional but least expensive fashion. In addition, product development could enter the next step as a functional food or nutraceutical, which would require a shelf-life study for the active ingredient stability properties according to established regulatory guidelines.

The basis for a stability program is a chamber or chambers for conducting experiments at controlled temperatures

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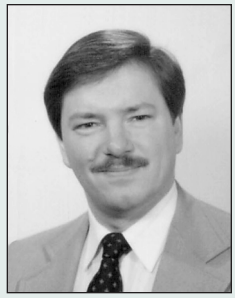

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and humidity conditions that stress the product and packaging. Stability studies also provide control samples for quality monitoring during the shelf-life determination of the product. After analysis of these samples, decisions are made as to what should be added or enhanced to increase shelf life. Finally, stability studies document when the product ends its useful life, a concept not always fully considered until consumers do the job by declining or returning purchases. The cost of raw materials, production and packaging can all be lost if a product is marginal, improperly stored in the warehouse or damaged during shipping. The results of this can be customer complaints, returns or lost market share due to product quality issues. These concerns are properly addressed through a functional stability operations area.

STABILITY OPERATIONS

The development of stability operations begins with a controlled environment in which to base product and packaging development. It can be very basic or highly sophisticated, depending on the needs of the company. Once established, this space and the technical personnel responsible for it deliver valuable information to every part of the company.

Chamber Types

There are a variety of chamber types available to a product development team. The choices are based on budget, need and the desired level of scientific sophistication. A variety of configurations will be explored, as all of these variations can be employed by a company, regardless of size, to determine product shelf life.

The most basic form of chamber is one that controls temperature and humidity in a limited space through external heat and chemical solutions of concentrated salts. By reviewing the literature, e.g., the *Handbook of Chemistry and Physics*, a series of saturated salt solutions are prepared and placed in sealed glass chambers to produce the desired level of humidity. These chemicals are available through scientific supply houses. Chambers can be as small as tabletop desiccators. For larger specimens, shelving units are sized to testing needs. Heat is controlled by an oven that will contain the sealed chamber, or in a room with controlled temperature. These examples would be the simplest in design and the lowest in cost.

The next level would be the reach-in style chamber, about the size of the basic home refrigerator. Chambers of this type are available from a variety of sources such as custom equipment manufacturers and scientific supply houses. The usual composition is a stainless steel interior, with full or mesh stainless steel shelving and a metal or glass door. These systems establish a controlled environment inside the chamber volume and have a monitoring chart for temperature and/or humidity. Study materials or products placed on the shelves are withdrawn at set intervals for analytical work, quality inspection or sensory evaluations during the stability study of the product.

The final level will depend on the volume of work and capital expenditure levels of the company. Large, walk-in style rooms can be constructed to accommodate very large numbers of samples, bulk raw materials or numbers of studies. The rooms can be built in place or constructed by prefabricated panels. Shelving is mesh-type stainless steel to allow air and humidity to freely circulate throughout the chamber and contact all the materials placed inside.

Regardless of the size of the enclosure, it is necessary that product study environments be calibrated and maintained to ensure that misleading data are not being obtained. This means adequate temperature and humidity sensors, maintenance and a chamber profile for the set points.

Chamber Validation, Calibration and Maintenance

A chamber calibrated to a set of conditions is required to establish the stability of a product, recommend proper and minimal packaging, and determine useful shelf life. A set maintenance program will ensure that the chamber is consistently supplying valid information for product development.

In the most simple cases, calibrated thermometers placed on shelves determine the temperature profile. Thermocouples and other electronic devices can also be purchased for this critical measurement. The measurement of humidity requires more complex devices. Purchase these through chamber vendors or instrument supply houses. Be certain that these measuring devices are calibrated routinely so data is reliable. Chamber temperature and humidity are set for a constant range that the chamber can maintain by purchase specification. These set points are monitored by a dedicated chart recorder so that a permanent record is available.

Temperature and humidity measurement devices are calibrated instruments designed to measure the range of the chamber's set points. The chamber is profiled at each shelf and along each shelf to demonstrate that the same conditions are present throughout the chamber regardless of storage position. This is accomplished by a 24-hour period of measurements when empty and another 24-hour test simulated as loaded at the chamber's desired set points of temperature and humidity. A predetermined cycle of testing checks on chamber readings independent of this validation will assure that data continues to be reliable. Product development can then proceed based on the results of samples taken from the chamber. In addition, maintain a routine of daily chamber checks. Implement these with written standard operating procedures for the maintenance and calibration of the chambers, set down for quality and future regulatory needs.

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Chamber Conditions

Given that a company is committed to a stability program, how are chamber conditions established for product development?

There are several internationally recognized organizations that have established a set of conditions for products. The World Health Organization (WHO) and the International Conference on Harmonization (ICH) are established, worldwide, for testing pharmaceuticals. These conditions serve as a guideline for products and chamber standards.

However, unless the company has plans to enter the pharmaceutical or health care products area, these conditions may be beyond the needs of a normal confectionery product stability study.

The evaluation of product should be centered on the production stresses, warehouse conditions and storage conditions likely for a product to be in contact with during its wait for customer purchase. In addition, there is value in stress-testing products to determine how adverse conditions will affect them or to reduce the time for shelf-life determination.

Manufacturing can contribute stress to a product. Reviewing the manufacturing

Stress conditions are determined by process, markets or warehouse conditions.

process to measure how high or low the product temperature goes during production cycles allows chamber temperature candidates to be established. Repeated temperature stress, cycling or spiking can start degradation. These conditions are certainly candidates for chambers in a stability operation.

Once produced, how is the product shipped? Does it experience high temperatures, humidity or cycle through a variety of conditions?

How do customers, pool points and warehouses store the product? Is it with nonfood items that may contain an odor the product is likely to absorb? Finally, how long can a department wait for a product to fail so that formulation adjustments can be instituted or packaging changed?

Working with the quality assurance department and customers, record product shipping temperatures, warehouse conditions and vendor or customer storage conditions. The quality team can measure product exposure to environments with temperature and humidity recorders or even through spot temperature measurement strips that record desired ranges. Products are on the market that will change in color due to a sensing strip sensitivity to temperatures.

A viable program includes the most acceptable temperature for the long-term storage of the product. Stress conditions of heat, of humidity and of heat and humidity are also candidates. If low temperatures can affect your product's shelf life, this needs to be explored. Do so at this temperature or by cycling through these conditions. All conditions that affect a product should be examined in planning for a stability operations area. This is critical for chocolate-based or compound-coated products. These products are the most susceptible to temperature and humidity conditions and to cycling of storage conditions. The final choice is based on the team need for data, budget and sophistication of the stability operation.

Most product development will be based on a storage shelf in an air-conditioned environment. By setting one chamber to 70°F ± 2°F and relative humidity of 45 percent ± 5 percent, one standard condition is met. This is also the standard test condition for paper and paperboard products set by the Technical Association of the Paper and Pulp Industry (TAPPI), so that packaging can be evaluated together with product.

Stress conditions can also be determined by experimentation or by selecting from the WHO or ICH guidelines. The best selections will come from a set of actual measurements of product storage conditions. However, there is a need to stress a product during development.

Accelerated Stress Conditions

Every company wants a product to last as long as possible. Given proper storage, that product may be considered to be consumer acceptable for several years in an ideal environment. However, this length of time would make product development a little slow for most marketing departments. For this reason, a program to rapidly age the product is needed. Hence the need for an accelerated condition. Under this condition, heat and humidity stress the product so changes can be observed faster than in real-time stability. Testing can begin with 10°F increments above the 70°F temperature for ideal storage to the practical limit of your product's potential storage condition.

For storage below expected room temperatures but above freezing, 45°F will

match food storage warehouse conditions; 32°F and -10°F for shipping conditions can also be considered for chamber conditions.

Consider the conditions recommended by WHO and ICH because these are valuable to your product line should you want to market internationally and preserve a health claim. Outside of the U.S., many current health claims bring a product into the realm of a pharmaceutical product. A company must demonstrate stability under recognized testing conditions in addition to gaining regulatory approval to use the claim printed on the label.

Once teams have made product temperature and humidity selections, set up the chambers to monitor product quality and raw material specifications through laboratory analysis and sensory panels. By plotting analytical results, there should be a condition that will equate to an accelerated measurement of time compared to the ideal storage condition. Using chambers set at the ICH guidelines of 25°C/60% rh, the 40°C/75% rh condition is already established as a four to one relationship in terms of time in the lower temperature chamber. Statistical analysis of data can support other relationships and be used for calculated shelf life based on laboratory results.

The accelerated data will then allow for decisions on product formulation and packaging. This will be much quicker than waiting for active deterioration of actual product in real time. Refinements to the factors and useable shelf life of the product will occur as real-time stability data becomes available to product development teams.

Chamber Study Types

Once stability operations has established chamber conditions, what types of studies are desirable for product development benefit?

For existing products, the study most necessary is maximum shelf life date, i.e., the conditions of storage that will extend the product to the maximum length of consumer acceptance without danger of losing the customer to a bad experience with the product. Another type is the development of lower cost products or line extensions. Stability studies make certain that the extensions or new supplier materials are compatible and products remain at the same level of consumer satisfaction as previous formulations.

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One commonly overlooked study is the evaluation of the packaging used on the product. Does it maintain the product for the longest possible time? Can a less costly package do the same? Can the packaging be changed to another type to reduce cost and still protect the product? For example, will a twist wrap do or must it be sealed for a gum product? Can chocolate wrapped in a foil and then a paper sleeve achieve the same shelf life as one in a foil fin-sealed package? Will hard candy survive a high humidity condition in a paper tube or a plastic tub? Company R&D, marketing and purchasing departments can give many more conditions and possibilities than are feasible to list here for consideration.

Product refinements in terms of new manufacturing to lower costs or new ingredients to enhance the value of the product are first evaluated in the laboratory. Once shelf life is determined by testing, a decision to use them in a product will assure the same shelf life, taste and consumer acceptance after prolonged storage or vendor storage shelf time.

One last area of concern, again involving product packaging, is its protection of the product not only from the environment but

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things in the environment. Contact with other foods and nonfood items can cause problems for products with high fat contents. Most notable is chocolate, which can absorb the odor of an environment. Laboratory migration studies determine the susceptibility of the product and the packaging level required to prevent it.

The stability laboratory also provides samples for sensory evaluation, raw material interaction, processing stress and shipping stress. The ability to control the environment of a product allows for increased understanding of potential problems within the product before they crop up in costly, real-life situations.

CONCLUSION

With a functional stability operation, product development can go forward with reduced costs and long-term product quality supported by a properly set storage condition. Each facet of the company can benefit from this scientific investment.

The manufacturing process is studied for variability in manufacturing techniques, temperature changes, process holds or raw material changes. Each change or substitution considered must not impact the brand or consumer opinions.

New ingredients or substitutions are studied for their impact on the product. Do both ingredients provide the same shelf life and sensory results after prolonged storage or temperature excursions? There can be no guess work on the answer to this very real concern to food scientists.

Packaging costs are controlled through performance evaluation of different films or paper structures. The needs for paper or plastic, overwrap or complexity of films or pouch construction are then objectively evaluated. Will a fin seal package matter to increased shelf life? Can foil packaging add to freshness? Expensive questions like these are answered before machinery or packaging films are ordered.

Can this product be marketed with a health claim in other countries? With properly designed stability operations, acceptance of shelf-life data to substantiate the claim associated with the active ingredient is easily verified.

A properly prepared stability operation with calibrated chambers and planned product development reduce costs. New products will then move from the laboratory to the market place with greater confidence once shelf life is scientifically determined through stability operations. Additionally, the testing done establishes how the environment affects the product.

The level of sophistication of the stability function can increase from budget year to budget year as the reliability of reach-ins and walk-ins is very high. By increasing the amount of controlled space and range of temperatures available to food scientists or a product development team, new products reach customers with known, scientifically demonstrated shelf life and minimal concerns about ingredients, process and packaging.

This known product shelf life is then used to balance production, materials, sales and purchasing of raw materials. Consumers benefit from continued freshness of the product on the shelf. In turn, the company benefits from reduced manufacturing costs and reduced product loss. In this manner, a functional stability operation benefits the operation of any confectionery producer. □

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