Shelf Life Monitoring of Raw Material with E-Nose – Application with Aroma and Flavor

Results obtained at Alpha MOS Laboratory, Toulouse, France

Application Note

Stability testing provides important data related to the effect caused by a variety of environmental factors such as temperature, humidity, and light conditions over a define time period on product formulations or ingredients.

The ability to perform product evaluation by automated stability testing methods permits the defining of ingredients and selection based on the stability of the ingredient. Stability testing ultimately aids in establishing recommended storage conditions and times. Stability tests also define the most stable aroma or flavoring and aid in the selection of ingredients that when integrated into a formulation will provide the best product based on market expectations.

Objective – Make Decisions Fast with Confidence

The following is an overview of an experiment conducted to define the use of the Alpha MOS System as a method to monitor change of the flavor or aroma for food industry or pharmaceutical industry when placed under temperature stress over a defined time to simulate product aging. The data generated illustrate the easy to operate method design and the simple to understand data presentation, which clearly defines the evolution of the product over time. The study clearly demonstrates the ability of the Alpha MOS system to

- Monitor the evolution over time of two aroma /flavors
- A comparison under sunlight stress conditions and temperature conditions used to accelerate the stress providing data for faster decision making.

Experimental Plan and System Conditions

Samples

The two flavors (A and B) with the same fruit flavor obtained from different suppliers have been preselected to be integrated in a formula. For both flavors A and B, a sample is kept unchanged (stored in room temperature conditions). This sample will be used as a reference sample.
Flavor A and B were stored in the same type of packaging in the same location at 60℃ over 27 days. Every 3 days, samples were taken and analyzed. At the conclusion of the test, the sample data were compared and the degree of change and rate of change determined by comparison to the reference measured on Day 0.
The flavors were also compared to each other to determine which flavor compound when used in the formulation exhibits the better stability and would be better suited to be used in bulk production of the formulation.

Analytical Conditions

Fox 4000 – Dry TOC grade air is used as the carrier gas

Flow rate (ml/mn)	150	Injection volume (µl)	1500
Quantity of Sample in vial (g)	0.5g	Injection speed (µl/sec)	1500
Vial type (ml)	10	Acquisition time (sec)	120
Headspace generation temperature (℃)	50	Time between 2 analysis (min) (When Optimized)	< 5
Headspace generation time (min)	10	Samples per Hour	12 - 15

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System Used for the Study:
Fox 4000 –with HS100 Autosampler

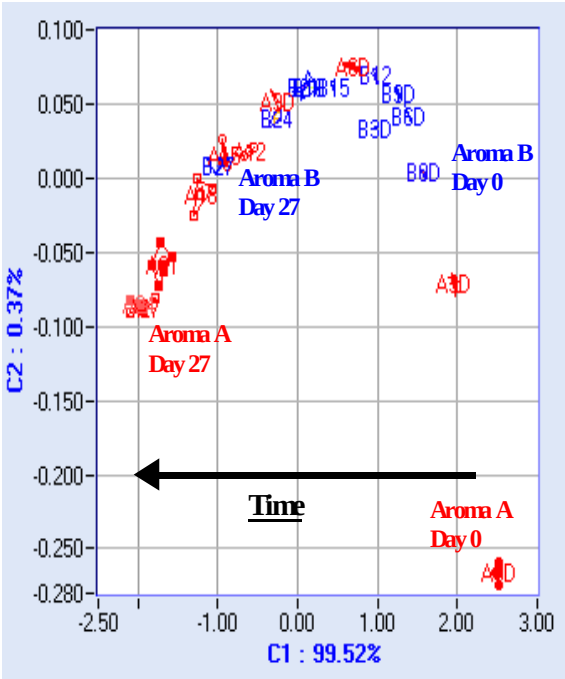
As shown, The Fox 3000/4000 with the HS100 OdorScanner Autosampler. The autosampler is configured with 2*32 Position trays designed to accommodate either 10-ml sealed vials or 20-ml vials. An Integrated Heating module conditions the samples according to a time and temperature program. Conditioning temperature ranges from 30℃ to 150℃. The sample maybe agitated while heated to insure repeatable sampling conditions.

The Fox 3000 is a continuous flow injection system configured with 6, 12 or 18 Gas Sensors. Sample Headspace conditioned in the autosampler, is injected into the flow of the Fox system. The sensors react to the volatile pattern present in the sample. This measurement is recorded an compared to other samples, standards or prediction calibrations for a quantitative prediction.

Evolution of Flavor Quality under Variable Temperature Conditions

Following the data collection several data treatments are available to define the changes in the product quality. To compare the samples comparatively a PCA Calculation is performed.

- The Day 0 sample is considered the flavor reference and is located on the right part of the graph. Day zero samples for both aroma/flavor samples have been plotted.
- While the flavor are from the same fruit, A and B are not exactly at the same location in the graph due to the fact that these flavors have come from different suppliers and are made up of different chemicals and under different manufacturing process.
- The data shows that the samples are immediately affected by the heat stress. The samples are dissociated from the reference: a quality change has been detected.
- A trend that follows the length of time of the stress from the right side to the left side of the graph can be seen. The data also indicates a larger effect on Flavor A to the conditions of the stress.



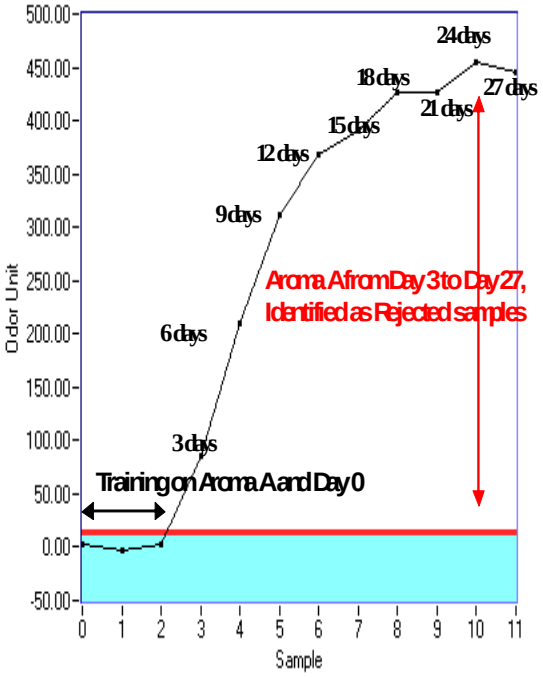
PCA Graph

Watch the Change, Over Time - Formula A Compared to Day 0

The progression of the change when compared to the Time 0 sample can be projected and the rate of change visualized as a function of time. The “no stress” sample is selected as the reference sample. This sample is used to define a control area within which the sample would be considered as statistically the same as the reference (Shown in green in the graph below).

By following the sample evolution over the time the samples are stressed it is possible to compare the sample to the reference. At any point when the stressed sample is plotted outside the acceptable range the samples can be considered as different.

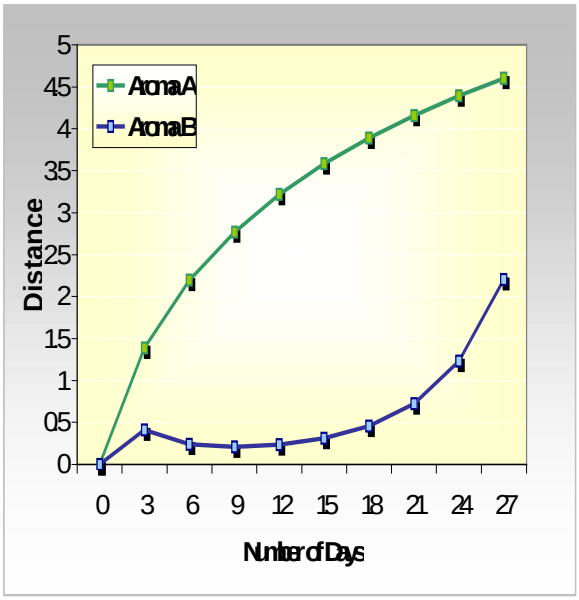
The SQC process (Statistical Quality Control) will rapidly assess the shelf life for the flavor and detect when the sample change is over the acceptability range. The result shows that as soon as the sample is stressed, it is statistically different from the reference sample.



SQC Graph

Which is Best? Flavor A or Flavor B

For both Flavor A & B, the distance or degree of change between the flavor at different days and the reference was calculated. By comparison the rate of change can be reported as on the graph.



The resulting plot provides the ability to define the sample evolution over time and to compare the performance of the flavors when stored under the same stress conditions.

The data clearly defines that flavor B is more stable under the temperature and time conditions. While some change is evident, the flavor and flavor profile are stable through day 21 after which a larger degree of change is evident. As a method to determine formulation suitability it would be recommended that Flavor Compound B be integrated in the formulation rather than the Flavor Compound A.

Conclusion – A clear winner

In general, the goal the Stability Labs in most companies is to identify the ingredients, which perform best and provide the stability needed to insure good shelf life. The tools available to these groups require time consuming and expensive manual tests or difficult to perform analytical procedures, requiring lengthy sample preparation which at best, in either case the data provided is ambiguous or partial in its assessment. As a result, formulation and stability suffer and substandard product could result.

The results obtained using the Fox 3000/4000 clearly define the products that demonstrate the best performance attributes. Representing the data in PCA and SQC formats provides an objective measurement regarding the performance of the formulations in either a global fashion or in comparison to each other.

The results obtained are simple to perform and can be fully validated based on repeatability and precision. The speed of the analysis makes it possible to test a larger variety of candidates under a broader range of experimental conditions. Get the formulation right the first time and have the data to make the right choices.

In addition to stress and rate of change, the data can be used to define:

- The breaking points in quality due to aging.
- Detect spoilage due to unsuitable storage conditions.
- Rapid aging stability - compare various formulations in term of stability using accelerated aging.
- Formulation stability in different packaging (for example glass vs. PET, modified or aseptic packaging)

The analysis shown can also be performed on samples subjected to other stress conditions such as; light and UV effects, humidity, storage and packaging and modified atmosphere conditions.

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