

Technology report

The Electronic Nose

The use of e-nose technology to bridge the gap between analytical and sensory science

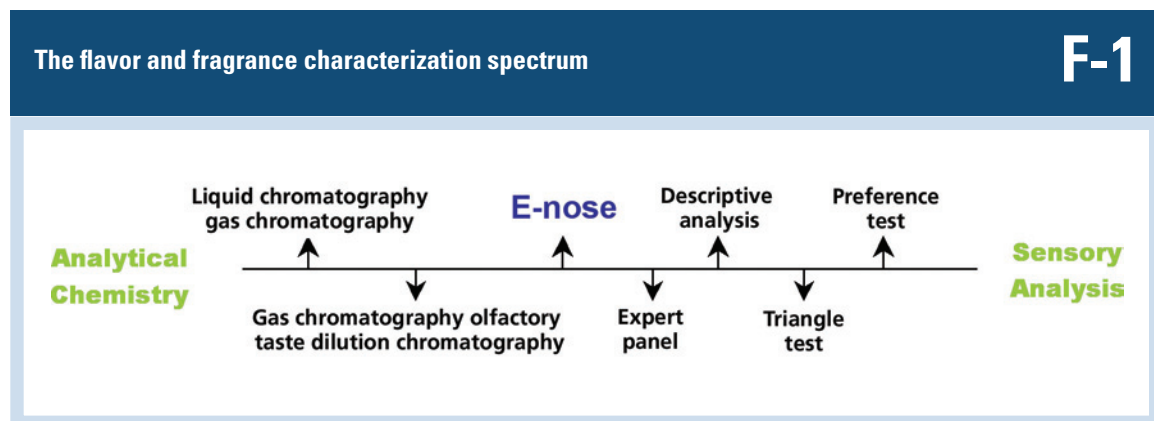
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Electronic, or artificial nose technology (e-nose), has received a considerable amount of scrutiny since its inception over two decades ago. From the first publications by Persaud and Dodd in 1982 (*Nature*), to today's proposed development of an electronic mouth, debate continues as to its suitable home within the flavor and fragrance industry. Much of the scrutiny of this tool originates with the vastly different disciplines of analytical chemistry and sensory analysis.

As shown in F-1, analytical and sensory sciences are on opposite ends of a flavor and fragrance characterization spectrum. Where the analytical approach prides itself on providing a bias free quantifiable value to flavor characterization, sensory science relies on quality-based subjective results obtained from the most advanced receptors and neural network — our nose and brain. The evaluation techniques utilized by these two disciplines, such as gas chromatography (GC), gas chromatography olfactory (GCO), expert panels, descriptive analysis, triangle tests and preference tests, span the length of the spectrum from quantity to quality-based. The e-nose can be found in the middle of this spectrum as

a compliment to the tools on either side of it.

In order to properly evaluate the e-nose's potential contributions to the flavor and fragrance industry, we must first understand its makeup and intended use. E-nose instrumentation is generally composed of two primary parts: sensory inputs and the predictive processing algorithm into which they are fed. The sensory inputs mimic the receptor cells in our olfactory system. The predictive processing algorithm should be representative of the logic processing found in the brain. Similar to our own organoleptic development, the e-nose system must be "taught" to recognize volatile organic compounds (smells) through experience and/or training. Once properly trained, the e-nose system has the capacity to predict the identity of vapors found in its training set. The techniques an e-nose utilizes to process the signal response data can be as





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simple as a statistical component analysis (SCA) or as sophisticated as a family of powerful data processing algorithms known as artificial neural networks.

E-nose technology is not a replacement for human sensory panels and/or standard analytical techniques. Rather, its intended use is as a complementary tool bridging the gaps between these disciplines. For example, sensory panels are a great method for comparing flavors between two products, as in a paired comparison test, or to identify an odd sample using a triangle test. Humans can identify a difference and then express what that difference is. However, the human nose and tongue can become fatigued very quickly. This desensitization effect greatly reduces the reliability and predictability of the sensory panel results. This process is noticeable for a whole host of flavors and sensations such as alcohol, mint and bitter flavors. With e-nose, an infinite number of products can be tested without fatigue, allowing for precise results every time. It provides verification for the sensory panel's results using an alternative method.

Standard analytical techniques have an important role in assisting identification of the discreet components present in a matrix when attempting to match a target. However, selecting the appropriate analytical technique is a challenge and can make the rigorous process of flavor and aroma characterization difficult to accomplish analytically. This characterization usually requires the use of multiple techniques to deliver a complete picture of the target flavor profile. The benefit that the e-nose can offer to the developer over analytical tools alone is the ability to evaluate the synergy that exists among complex vapor mixtures rather than just their components. With this ability, the e-nose serves as a guide offering directional infor-

mation on flavor or aroma development.

Due to these unique capabilities, the e-nose is already being tested and utilized in the food industry in ways that may be applicable in the flavor industry. The US Food and Drug Administration has applied it in grading the freshness of fish by detecting the level of amines present in a sample. Instruments have been used to successfully discriminate cheeses at different stages of maturity and verify the authenticity of Parmesan and other regional cheeses. The e-nose has been proposed to the coffee industry as a tool to precisely control roasting and blending and

detect such problems as insect damage or mold growth. Japanese electronics company Sharp is reportedly evaluating its use in microwave ovens to trigger automatic shut-off when chemicals associated with overcooked foods are present.

In the flavor industry, the opportunities for process and quality improvement through integration of the e-nose are numerous. Its potential role in flavor development and sensory evaluation was discussed earlier. As a QC tool, the e-nose could be calibrated to recognize acceptable and unacceptable raw materials, intermediates and finished products, verify the origin of materials like vanilla beans and peppercorns, and verify the shelf life of products.

The e-nose will certainly not be a replacement for human sensory analysis or traditional analytical techniques, nor is it designed to be. Whether used as a flavor development, sensory support or QC tool, when applied appropriately it provides a unique opportunity to support subjective testing methods with objective results. Only time will tell if this technological revolution will penetrate the flavor and fragrance industry and carve itself a niche in the characterization spectrum.

Case Study: Maillard Reaction Monitoring Using E-Nose Technology

The following research involves the utilization of a simple e-nose system for Maillard reaction monitoring. The measurements

and results obtained from an e-nose system engineered and constructed by researchers at Northern Illinois University's department of chemistry and biochemistry in conjunction with FONAs technology solutions group are outlined. This project, started in 2003, resulted in a small-batch Maillard reaction monitoring system in late 2004. The current cooperative partnership established between FONAs and Northern Illinois University has proliferated to more sophisticated proprietary e-nose and e-tongue developmental projects.

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Non-enzymatic browning reactions, such as the Maillard reaction, have been well characterized both qualitatively and quantitatively. These simple amino acid/reducing sugar reactions can generate a tremendous number of chemical components, adding to the sophistication and complexity of their aroma and flavor profiles. Major factors influencing the reaction and its subsequent aroma profile include time, temperature, composition and activity of reducing sugar/amino acid

pair, and chemical properties of the solution. Properly controlling these variables aids in the development of a chemical reaction meeting certain taste and aroma criteria. Subtle variation in natural raw materials and batch sizes represent just some of the challenges faced by the reaction-flavors industry. Therefore, establishing and probing for a target aroma and flavor profile minimizes batch-to-batch variability while simultaneously enhancing process control.

The Maillard reaction used for this experiment was a simple L-leucine and xylose reaction. The target aroma and flavor profile was defined using both sensory and analytical techniques. The analytical methods examined both liquid and headspace composition while sensory evaluation was accomplished using a small descriptive panel. The data obtained from both studies had multiple applications. First, it was used to establish the chemical composition of the desired aroma and taste profile. Next, the identified components gave directional information on engineering of an appropriate microsensor array. Finally, the concentration information was normalized and used during the e-nose training phase.

Key flavor components indicative of L-leucine and xylose Maillard reactions are: isovaleraldehyde, acetic acid, chicory furanone, furfural and furfuryl alcohol. Isovaleraldehyde concentration was determined from headspace data while the remaining components and their concentrations were determined from liquid phase analysis. Concentration profiles over reaction time are shown in the T-1 and were obtained by sampling the reaction mixtures at the specified time interval.

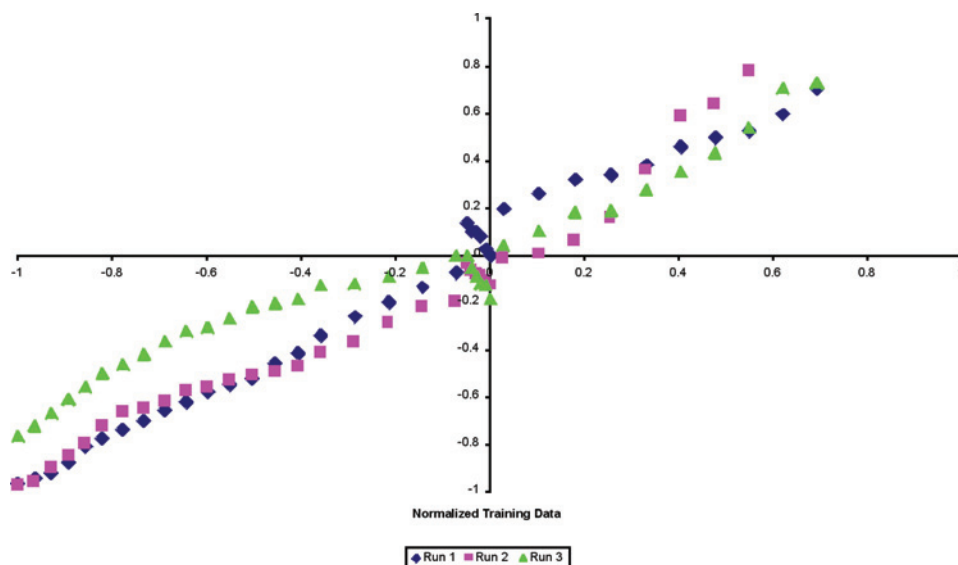
The reaction monitoring system designed for this study consisted of five surface acoustic wave (SAW) microsensors. The microsensor array was connected to a pressurized reaction vessel using a vapor-sampling loop in conjunction with an in-line vapor stream dryer. A PC outfitted with data acquisition software was responsible for handling the signal processing and housed the predictive algorithm, a feed-forward neural network. The e-nose system was connected to the headspace valve of a reaction vessel and trained using the same experimental parameters present in the

Concentration profiles of key Maillard reaction components

T-1

Component	Reaction time (min)							
	10	20	30	40	50	60	70	80
isovaleraldehyde	1143.0*	1912.0	2432.0	2836.0	990.0	920.1	619.4	550.8
acetic acid	-	-	3.4	9.2	37.2	37.6	88.7	131.5
chicory furanone	-	97.5	150.9	273.7	240.7	197.6	186.7	156.6
furfural	4.4	469.7	1250.2	1974.8	2704.8	2715.5	2875.1	3069.8
furfuryl alcohol	-	2.3	13.4	33.5	197.6	240.7	333.7	443.1

*concentrations reported in ppm (< 10 percent RSD)



initial characterization process. The system was also trained using component concentration values correlated to sensor array response data at the prescribed sampling time intervals.

Both analytical and sensory evaluations resulted in selection of a desired aroma and taste profile at 50 min of reaction time. The output of the artificial neural network (ANN) was related in terms of a reaction completion index scaled between -1 and 1 where a value of 0 represented the ideal aroma and taste profile (50 min of reaction time). A value of -1 represents the lower boundary (under reacted) and value of 1 represents the upper boundary of training (over reacted). For this experiment, the predicted data set was compared to the normalized training data set values. Graphical representation of this data can be seen in F-2. Each point represents the predicted progression of the reaction independent of time. Values close to the origin correspond to reaction completeness.

The findings shown herein reflect the robustness of e-nose as a reaction monitoring system. This process was validated using the initial analytical and sensory results to verify aroma and taste profiles of subsequent reaction mixtures. While this technique is not quantitative, it does present a proven semi-quantitative level of assurance surrounding the progression of the reaction. The Maillard reaction outlined here is extremely complex and suggests the need for a higher order or multi-component analysis. This type of analysis is where the e-nose technology finds a considerable amount of utility. It has the capability to characterize the complex headspace mixture in real-time. This study shows that e-nose systems can be trained for general qualitative and semi-quantitative applications.

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