

Electronic Nose for Space Program Applications

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Introduction

An Electronic Nose (E-nose) consists of an array of non-specific vapor sensors. In general, a sensor array is designed such that each individual sensor responds to a broad range of chemicals, albeit with a unique sensitivity relative to the other sensors. Chemical identification is achieved by comparing sensor response pattern of unknown vapor to previously established patterns of known vapors. In recent years, the E-nose has been applied to increasingly complex and complicated chemical systems. The E-nose is ideally suited for the identification of specific chemicals, such as the contents of storage tanks and numerous instruments have been commercialized to meet this market (e.g., Sawtek, Cyranose). Other applications pertain to less precisely defined chemical systems. For example, the chemical composition of vapors from spices or other food products is not necessarily completely known, yet a properly trained E-nose can distinguish between specific types of spices. The E-nose can be trained to provide a level of quality for flavors and food products. There are many types of E-nose instruments^[e.g.1] with various types of sensors. Sensor types that have been used in arrays include metal oxide semiconductor (MOS), conducting polymer, surface acoustic wave (SAW), composite polymer (CP), electrochemical, and others. Sensor types have different analytic performance. Some sensors are more sensitive than others to specific vapors, while others are less prone to drift due to changes in ambient conditions (e.g., temperature, RH, pressure). NASA at the Kennedy Space Center (KSC) is currently assessing the ability of several commercially available and developing E-nose technologies to meet specific requirements for applications in the space program. Three critical applications have been identified.

The ability to monitor air contaminants in a closed environment, such as the Shuttle, the International Space Station (ISS), and future human missions to Mars or the moon is important to assure the health and safety of astronauts and integrity of equipment. Post-mission analyses of grab air samples from the Shuttle have confirmed the occasional presence of on-board volatile organic contaminants^[2]. The spacecraft maximum allowable concentration (SMAC)^[3,4] was established as a guideline to maintain the air quality in spacecraft. To assure compliance to SMAC, air monitoring must be performed. Specific compound identification is required. A light, low power, miniature instrument capable of reliably identifying abnormalities and contaminants at trace levels in specific operating environments would fulfill these needs. Continuous air monitoring could provide notification of adverse events such as of spills or leaks.

Continuous air monitoring could also provide notification of an impending fire. Fire in a closed environment such as spacecraft can be disastrous. Electrical fire would be a major concern. Frequently one of the earliest indicators for a pending electrical fire is the overheating of wires. As the wires heat up, chemical vapors would be emitted from the insulation. The emission would be a complex and possibly indeterminate mixture of residues left over from the fabrication process (e.g., solvent residues, additives, low molecular weight oligomers) and of thermal degradation products of the insulation itself. For the Shuttle and other space program applications, wire insulation is typically Teflon®, Kapton® or silicone-based materials. These insulation materials are quite inert and pure, so that the vapor concentration from

thermal offgassing would be low. A gas monitor could detect these vapors, but would require high sensitivity. With sufficient sensitivity, an E-nose could distinguish between electrical and other types of fires because of the unique composition of the vapors from heated wires relative to other combustion sources. An E-nose could provide an early warning even before actual combustion. A chemical monitoring system should be capable of alarming faster than conventional smoke detectors since vapor emissions occur prior to actual combustion or even smoke generation.

A third application currently being pursued is for monitoring the hypergolic propellant contaminants in the airlock of the Shuttle and the ISS. The airlock is the interface between the crew quarters and space, and is depressurized prior to astronaut exiting the spacecraft and re-pressurized for entry back into the crew quarters. During Extravehicular Activity (space walks) astronauts could be exposed to hypergolic propellants⁽⁵⁾, namely hydrazine (Hz) or monomethyl hydrazine (MMH) as the fuel, and nitrogen tetroxide (N₂O₄) as the oxidizer. Residues can be present on the astronaut's suit or other equipment. The hydrazine family vapors are toxic and are suspected human carcinogens. The current Threshold Limit Value (TLV) set by the American Conference of Governmental Industrial Hygienists (ACGIH) is 10 ppb. Prior to reentry to the crew quarters through the airlock, it is important to verify that no residual vapor is present. Moreover, vapor monitoring at this level must be done at the operating pressures of the airlock, which range from ca. 150 to 750 torr.

Approach

A literature and market search for available E-noses was performed to identify instruments suitable for the space program applications. A number of miniature commercial instruments were available in a moderate price range. In addition, several instruments that were not yet commercially available but were in an advanced stage of development were considered. A short series of tests were conducted to determine whether or not a specific instrument could meet the required sensitivities for a specific application. Those instruments that performed favorably for a specific application were tested further.

It is recognized that the vapor levels required by SMAC and the other applications are low and that the deployment conditions can be quite challenging. This paper does not address the ability of an instrument to respond to or identify vapors, but rather the ability of an instrument to respond to vapors at the levels and conditions required for specific applications within the space program. Presented in this paper is a summary of the preliminary results for an on-going program to identify viable E-noses for deployment in NASA's Space Program.

Experimental

Vapor Generation—Calibrated Standards

Test vapors were generated using permeation tubes and ovens (Kintek Model 360). The permeation tubes were maintained at a constant temperature and were purged continuously with dry air at 100 cc/min flow rate. The resulting vapor was blended with 1.9L/min of dry air to generate 2.0 L/min of the vapor. (Note: For safety considerations, nitrogen was the purge gas through the HZ and MMH permeation tubes.) Flows were verified prior to tests using flow meters (SKC Accuflow). In the standby mode, the purge gas through the permeation tubes was not mixed with the 1.9L/min air stream, but rather was vented through a separate pneumatic line. This provided a source of non-contaminated air. Activation of a solenoid valve internal to the permeation oven would allow the purged vapor stream to mix with the clean air, thus generating the test vapors. Table I summarizes the various vapors generated in this manner.

Table I: Test vapors and base permeation oven concentrations

Vapor	Concentration (ppm)	Verification Method	SMAC (7-day) (ppm)
HZ	0.07, 0.20, 1.25, 17.7, 35.0	Coulometric Titration	0.04
MMH	0.15, 1.06, 5.32, 16.0	Coulometric Titration	0.002
Ammonia	9	PD weight	10
Nitrogen Dioxide	12, 46	ASTM Method 1607-9	
Toluene	59	PD weight and GC ¹	16
Acetone	115	PD weight and GC ¹	22
Methylethyl Ketone	48	PD weight and GC ¹	10
Xylene	40	PD weight and GC ¹	50
Isopropylalcohol	55	PD weight and GC ¹	60

¹Independent GC analyses were performed to confirm the vapor concentration

Vapor concentrations from the permeation ovens were verified monthly. For the organic solvents, the vapor concentrations were determined by the weight loss of the permeation tube during the operational period. This concentration was independently verified by using a Gas Chromatograph (Photovac Model 10S) calibrated with certified vapor standards. HZ and MMH vapor concentrations were verified monthly using an impinger filled with 0.1M H₂SO₄ to scrub a known volume of vapor. The HZ or MMH concentration in the resulting solution was determined by coulometric titration using a method described previously^[6]. Nitrogen Dioxide vapors were verified using ASTM method 1607-9.

Vapors from the permeation ovens either were used directly or were precisely blended with air from a temperature (T), humidity (H) and flow rate (F) controller (Miller-Nelson Model HCS-40). Dilution factors up to 25 were conveniently obtained. Three ranges of RH values were typically used in testing—low (<10%), medium (25 to 35%), and high (60 to 75%).

Earlier work has shown that stainless steel and other materials are incompatible with HZ or MMH vapors, especially at low concentrations^[7]. The use of stainless steel tubing and fittings was kept to a minimum. All pneumatic lines were either Teflon or Bev-a-Line IV ® tubing, which were shown to have minimal effect on these vapors, even in the low-ppb range.

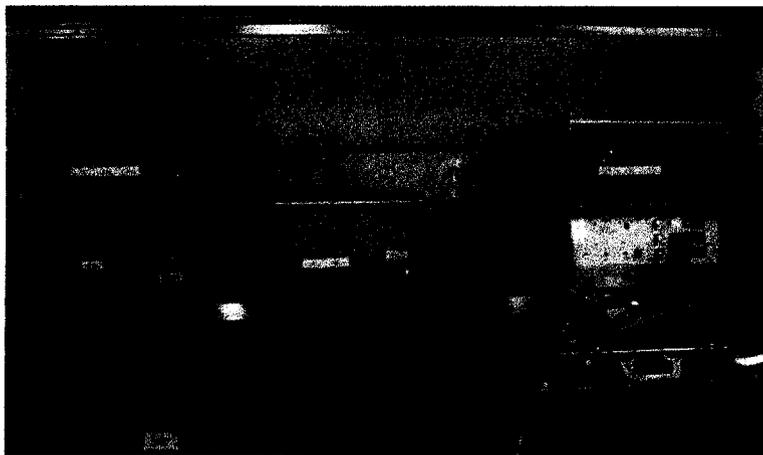


Figure 1 Basic Test Setup

Low Pressure Test

E-nose performance was measured at sub-ambient pressures, ranging from 150 to 750 torr. To do this, the E-nose was mounted in an evacuated bell jar. The base of the bell jar had two vacuum-tight electrical feedthroughs. One provided line voltage to power the E-nose, the second was an RS232 to allow computer control and logging of results for the E-nose. An ion gauge (Granville-Phillips Convectron) monitored the internal pressure of the bell jar. A vacuum pump evacuate the bell jar. A sample line was connected to the vapor source. Metering valves were installed on both the vacuum line and the sample line and were adjusted so as to balance the inlet and outlet vapor flow. The internal pressure of the bell jar could be controlled to within ± 2 torr with this setup.

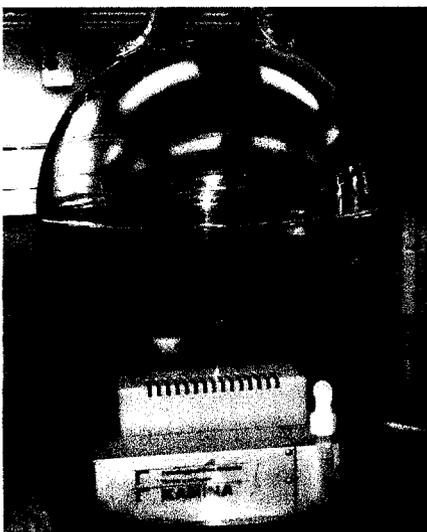


Figure 2: Vacuum Chamber Test Setup

Pre-Combustion Vapor Generation

Samples of wires types certified for use on Shuttle and ISS were obtained. These were coated with various insulating materials (e.g., Teflon®, Kapton®, and silicone based materials). Commercial grade wire with polyvinylchloride (PVC) insulation was also used in the test. A 15 to 20 cm length of wire was connected to the leads of a high current transformer. During testing, the wire heater and E-nose(s) were placed inside a metal enclosure. To mimic an imminent electrical fire, the wire was heated electrically by the passage of current. The current was measured inductively using a current clamp. The current was increased slowly to control the temperature of the wire and to prevent the generation of visual smoke. As the wire heated up, the thermally generated vapors were monitored by the E-nose. Further heating was stopped once a response was observed on the E-nose.

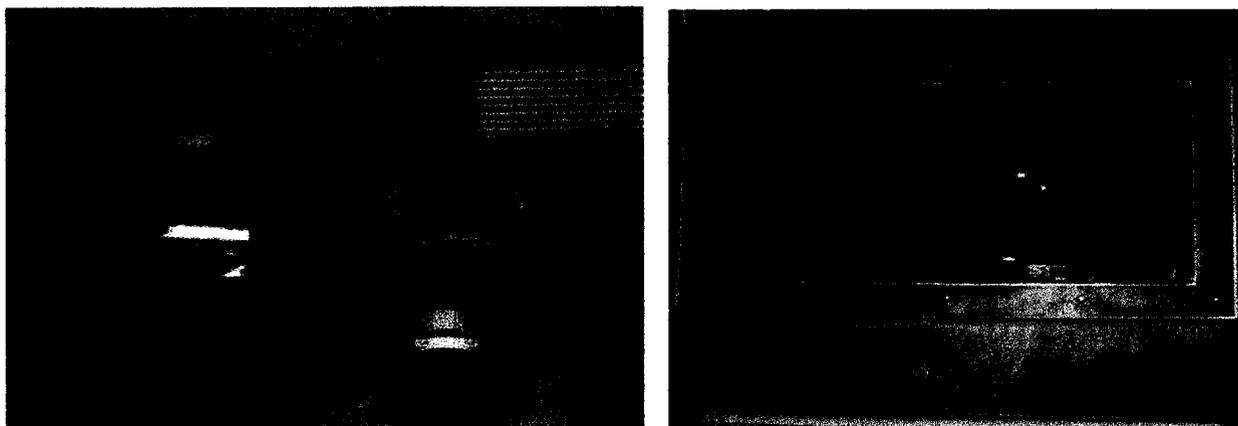


Figure 3: Experimental apparatus for the thermal generation of vapors from various electric wire types. The wires are heated via a power transformer. This process is typically carried out in an enclosure so as to allow access of the vapors to an E-nose. Various E-nose technologies were used in this study and were mounted within the chamber during the heating process.

E-nose Instrumentation

The various E-noses that were included in this study are provided in Table II. Either commercially available E-noses or preproduction models were used. Instruments had to be light-weight and portable. Battery operation was not a critical parameter. The extent of evaluation was based first upon the ability to detect relevant vapors at the appropriate concentrations. Not all instruments have yet to be tested for each of the applications identified in the introduction. Instruments with sufficient sensitivity to vapors of interest were subjected to more rigorous testing. This testing encompassed an assessment of the analytic performance of the instrument, an evaluation of the ability of a “trained” instrument to identify test vapors using the vendor supplied operating system, and the in-house development of identification algorithms to assess the information content of the raw data:

Table II: E-nose instrumentation used in this study

Instrument	Manufacturer	Array
KAMINA ¹	Karlsruhe Research Center	38 MOS
i-Pen2	Air Sense	7 MOS
SamDetect	Daimler-Benz Aerospace	5 MOS
Cyranose	Cyrano Sciences	32 CP
JPL ²	N/A	32 CP
Early Fire Detection System	Marconi Applied Technologies	4 SAW
VaporLab	SawTek	4 SAW

¹A pre-production model of the KAMINA was used in this study

²The JPL E-nose instrument is not commercially available. It was developed by NASA/Jet Propulsion Laboratory (JPL).

Organic Vapors Test

Sensitivity Screening: The first series of tests were a quick screening to determine whether each instrument had sufficient sensitivity to detect selected vapors at relevant concentrations. For general breathing zone monitoring, the relevant concentration was the SMAC level, as listed in Table I. The standard test conditions were at 22°C and approximately 30% RH at ambient pressure. Sensor signals

were allowed to stabilize in clean air. The instruments were then exposed to vapors at or below the SMAC level. Typically, three or four exposures were made. Several instruments were found to be able to detect the organic vapors the SMAC level (e.g., SAM Detect, KAMINA, and i-PEN2).

Performance and Results: Instruments that showed a reasonable response to vapors at the SMAC level were subjected to more extensive vapor exposures at various concentrations and relative humidity. Data sets for training and validations were collected for both the KAMINA and the i-Pen2 E-noses for the other organic vapors. Data collection is on-going for the Sam Detect. For the i-Pen2, the data set consisted of approximately 25 individual measurements. Concentration ranges varied between ca. 50% to 200% of the SMAC value. Results for the i-Pen2 are shown in Figure 4. Plotted is a time profile of the sensor response as induced by the indicated vapor. A radial plot is also shown to illustrate that unique pattern is obtained for the vapor. For the KAMINA, over 40 individual exposures were made on the organic vapors. As discussed in "Sample Ratio" section of this paper, the lower the number of sensors for the i-Pen2 relative to the KAMINA (e.g., 7 vs. 38) requires a smaller data set. Time constraints restricted the amount of data that could be collected and the size of both the KAMINA and i-Pen2 data sets was smaller than ideally desired. Nevertheless, as discussed in "Vapor Identification" section of this paper, the data allowed significant conclusions regarding E-nose use in the space program.

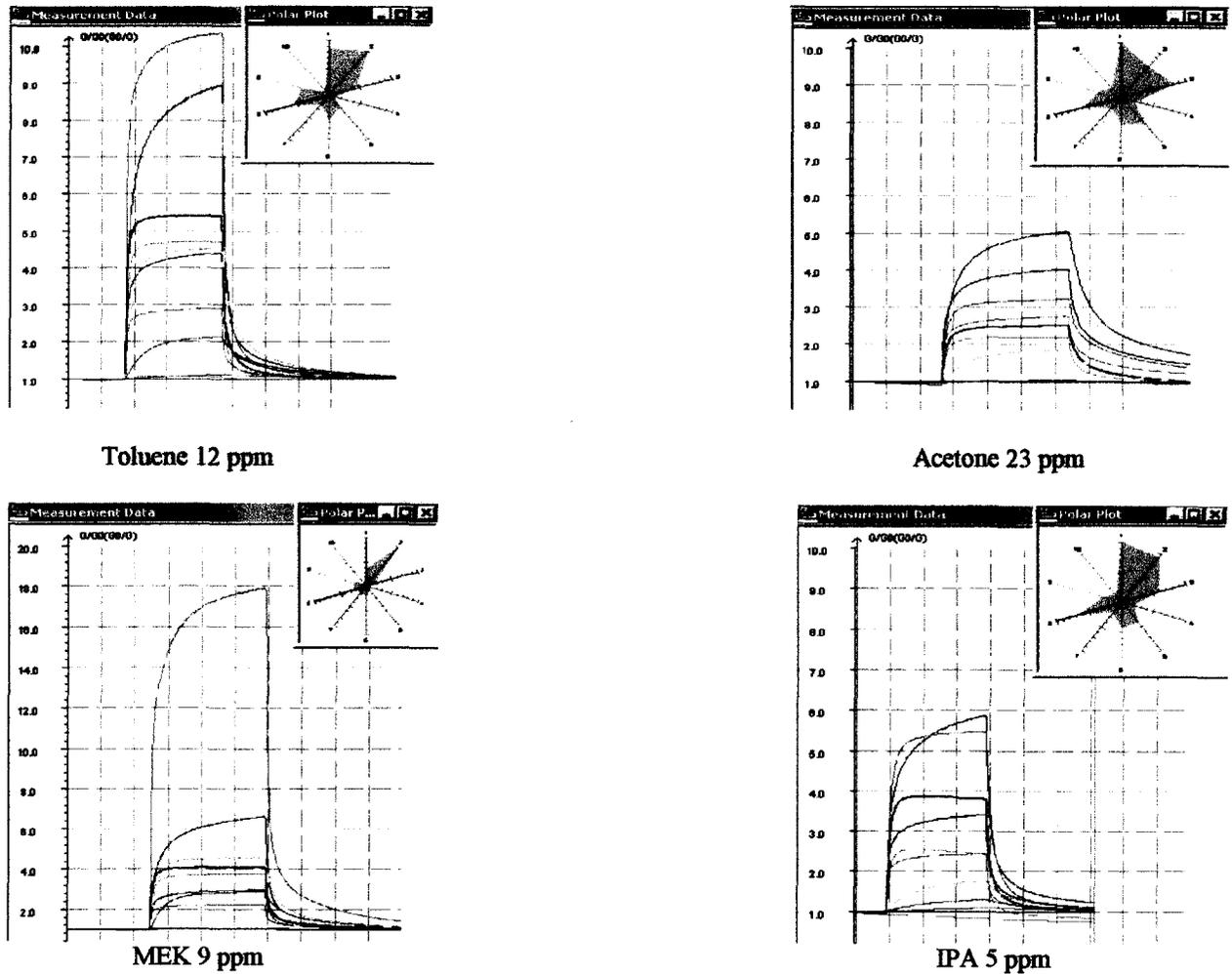


Figure 4: Individual sensor response observed on the i-Pen2 to the indicated vapor

Hypergolic Fuels Tests

Sensitivity Screening: In the case of HZ and MMH, the ACGIH TLV standard of 10 ppb was selected. KSC has adopted that ACGIH standard for its operations. Several of the technologies showed reasonable sensitivity to ppm levels of HZ or MMH. Of all the instruments tested, only the KAMINA was able to respond to 10 ppb levels of HZ and MMH with a signal to noise ratio greater than 3.

Performance and Results: The response of the KAMINA to 10 ppb HZ and 10 ppb MMH is displayed in Figure 5. A data set made up of over 40 individual HZ and MMH exposures were made at concentrations ranging from 10 to 150 ppb and relative humidity in the low, medium and high range. Since the KAMINA sensitivity met the minimal concentration requirement for the airlock application, pressure dependence was also included in the data set. Exposures were performed for pressures at 150, 300, 450, and 720 torr. For this application it is important to verify that an instrument is sufficiently robust physically to withstand vacuum operation.

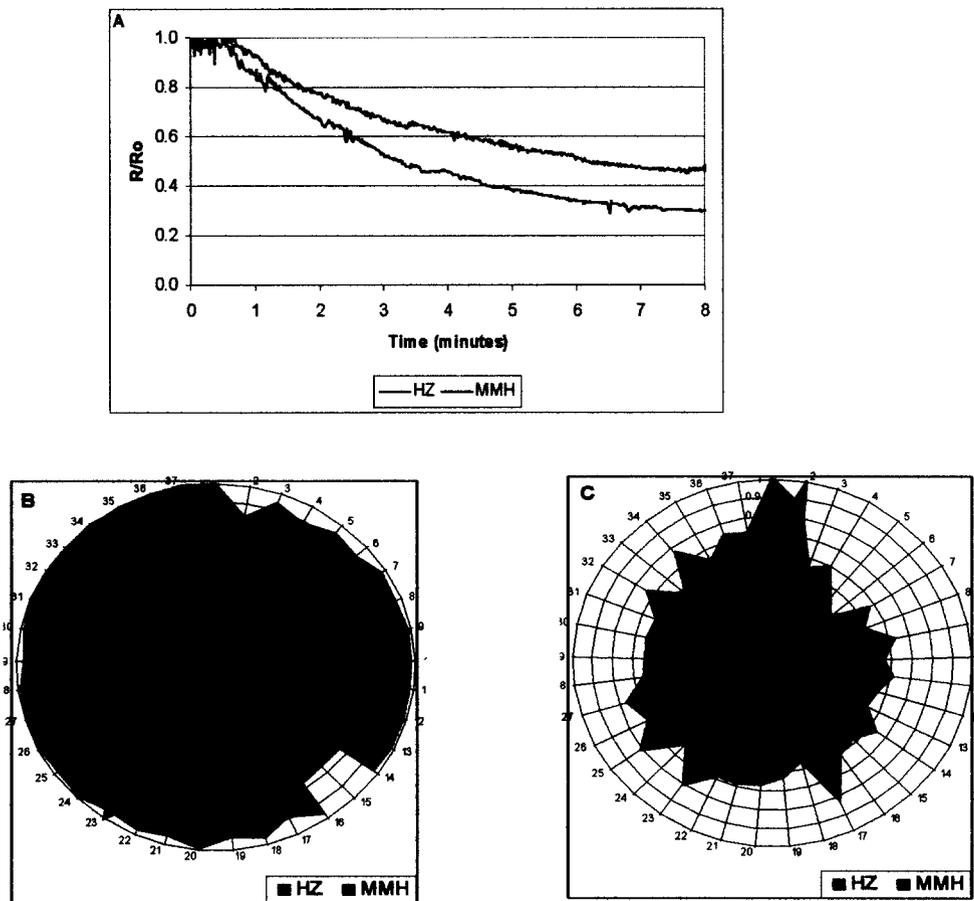


Figure 5: Response of the KAMINA sensor array to HZ and MMH. (A) The real-time response of the Kamina sensor array to 10 ppb of HZ and MMH. The array was exposed with clean air for approximately 30 sec followed by exposure to the HZ or MMH vapor. The average of the 38 sensors are plotted as R/R_o where R is the sensor response at any point in time and R_o is the response of the sensor in clean air. (B) Radial plot illustrating individual sensor response to the vapor. Data is shown at 30s. (C) Same as B, except exposure time is 8 minutes.

Pre-combustion fire test

Sensitivity Screening: A section of electrical wire was mounted on the burning apparatus (Figure 3) and the voltage was gradually increased to heat the wire. Wires with PVC, Teflon, and Kapton types of insulation were tested. PVC is a common insulation, but is not used extensively on the Shuttle or ISS. It readily emits offgassing products when subjected to heat excursions. This allowed confirmation that the E-nose is positioned properly within the test enclosure. Most of the E-noses that were tested did respond to the thermal products of PVC. This included the KAMINA, Sam Detect, and the Cyranose. Other instruments were not tested. Vapors from Teflon or Kapton wires were at much lower concentration. Only the KAMINA and Sam Detect were able to detect vapors from these wires.

Performance and Results: Figure 6 shows the response pattern of the KAMINA to the off-gas vapors of the polyvinyl chloride, Teflon, and Kapton electrical wire insulation materials. Clearly the off-gas from each wire types induces its unique response pattern on the KAMINA Sensor array. Several other array designs exhibited an equally impressive response as illustrated in Figure 6 for the SAM Detect. Modeling of the SAM Detect data resulted in well-separated two-dimensional projections indicating that thermal degradation of the various wire types can be identified via chemical monitoring.

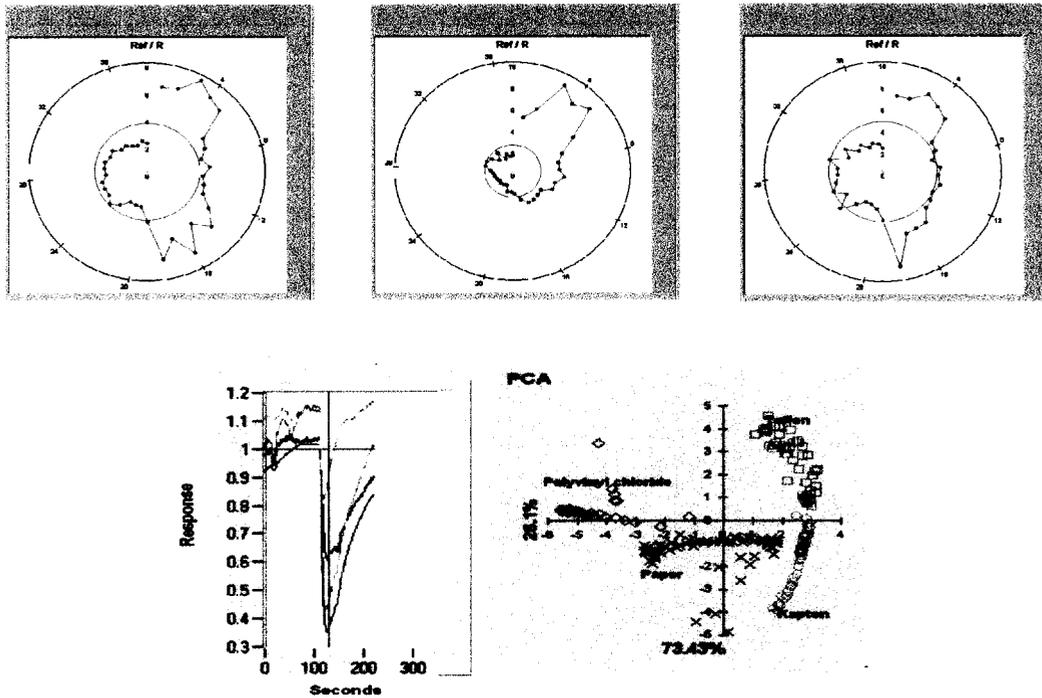


Figure 6: (Top) Radial plots from KAMINA showing individual sensor response to the thermal out-gassing products of three wire types (left to right: PVC, Teflon, Kapton). (Bottom) Time plot of individual sensors in the Sam Detect showing response to a PVC wire. PCA analysis of data from various wire types showing individual wires type congregated in well-defined 2-dimensional space.

Vapor Identification

The process of pattern recognition or classification involves using multiple *examples* (single exposure to a vapor), each of which consists of one or more *features* (defined below) to create a model of the *classes* (vapor types). This model is then used to classify a new example as belonging to one of the existing classes, or to an “unknown” class.

An important measure of quality for a classifier is the estimate of the future success rate of classification given the current model. While both the KAMINA and the i-Pen E-noses provide a PC-based graphical user interface program to control the collection of vapors and to display the results of classification, neither program calculates this estimate. Thus programs were written in Matlab® to calculate the classification success rate, and to explore ways of improving the classification using different features and different classifiers.

Feature Extraction

In pattern classification, a “feature” is any direct or derived measurement of the system that helps differentiate between classes. There are many different possible features which can be extracted from E-nose data, but the final value and maximum initial slope (see Figure 7) have been shown to be the some of the best at discriminating between classes^[8]. However, the value of the maximum initial slope is very sensitive to noise, and some E-nose sensors can be quite noisy, especially at low vapor concentration. While the final value is robust and simple to calculate, most sensors require a long time to stabilize (one to 20 minutes depending on the application and the sensor response time). Instead, the sensor values at a specific time were used for our application, which was considerably shorter than the time needed for the sensor to reach steady-state. It was determined that the sensor values at 30 seconds were nearly as useful for discrimination as the final value, as discussed below.

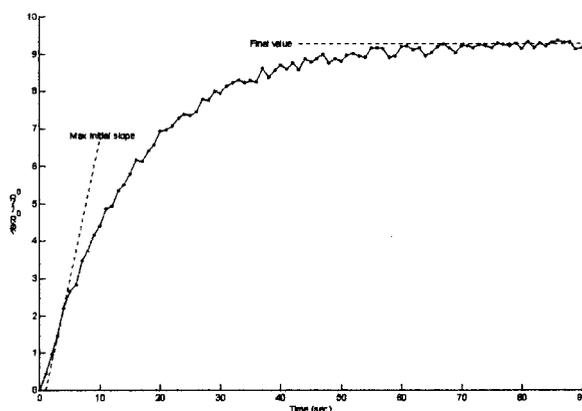


Figure 7 – Typical time response of a single E-nose sensor

Sample Ratio

One of the biggest problems with using an E-nose is the large number of features available compared to the number of examples, due to the use of multiple sensors. For example, the KAMINA E-nose has 38 sensors, and so if two features are extracted per sensor (such as max initial slope and final value), there

are a total of 76 features per example. The term “sample ratio” (SR) is defined here as the number of examples per class divided by the number of features. A commonly held rule of thumb in pattern recognition is that the SR should be at least five to ten^[9]. However, it can often take several minutes to collect a single E-nose example, so SR’s less than one are quite common in E-nose research. Small SR’s make all aspects of the pattern classification process more difficult^[9,10,11]. The average SR for the I-Pen data was 0.89, that of the KAMINA organic data was 0.38, and that of the KAMINA fuel data was 1.02.

Classification

There are many different possible ways to classify an unknown example given a set of known examples. Statistical classifiers include the nearest Euclidean mean classifier, the Linear classifier, the nearest Mahalanobis distance classifier, and the Quadratic classifier^[13,14], all of which have the advantage of being very fast. However, the Euclidean classifier ignores the shapes of the distributions of the classes, the Linear classifier assumes the class shapes are identical, and the Mahalanobis and Quadratic classifiers require relatively large amounts of data (SR > 1.2) in order to model the classes at all. The Kth nearest neighbor classifier is also very popular^[13,14], but it tends to overfit the data for small values of K, and does not perform well for small SR’s in general^[14]. Iterative methods, including the relaxation, Widrow-Huff, and Ho-Kashyap linear classifiers^[13], as well as neural networks and support vector machines, are generally very slow and have many user-selectable parameters which can significantly affect the classifier’s performance. Because the Linear classifier averages the class shapes, it effectively increases the SR (at the potential expense of some accuracy), and so it was used for this application.

Error Estimation

In order to determine how well the classes can be discriminated, some estimate of predicting the classifier’s future performance must be made^[15]. While the ideal method would use part of the data for building the model and the other part for testing the model (called the Holdout estimator), this is not possible when the SR is very small. Holdout requires a large amount of data to build an accurate model, and more data to accurately estimate how well it performs. On the other hand, if all the data is used to build the model, and is also used to estimate the success rate (known as Resubstitution), the estimate will be too optimistic. This problem is usually solved by using techniques such as N-Fold Cross Validation, which sets aside part of the data, builds a model with the remaining data, and uses the first part to estimate the performance. N different portions are set aside, and the N estimates are then averaged. If N equals the number of examples, the result is the Leave-One-Out estimator, which is often called simply “cross validation”.

Leave-One-Out becomes increasingly pessimistic (that is, underestimates the classification success rate) as the SR gets smaller, while Resubstitution becomes increasingly optimistic^[10]. However, the average of these two estimators has been shown to be an excellent estimate of future classification performance at small SR’s^[11]. This average will be called the “RU” estimator.

Results

Using the 30 second sensor values with the Linear classifier and the RU estimator, the i-Pen was able to achieve a 91% success rate in distinguishing between acetone, IPA, MEK, NO₂, ammonia, TCE, toluene, and xylene across a wide range of concentrations and relative humidities. The KAMINA was able to differentiate between acetone, IPA, MEK, toluene, and xylene with 86% success across a wide range of concentrations and relative humidities. This estimate increased to 98% when several redundant sensors were not used^[11,15], thus increasing the SR. The KAMINA was also able to separate HZ and MMH with 84% success, increasing to 93% when fewer sensors were used.

The following table shows the classification success rate as a function of the sample time (using all sensors). As can be seen, the shorter time is within a few percentage points of the longer time.

Dataset	30 second samples	90 second samples
i-Pen2/Organic	91%	88%
KAMINA/Organic	86%	89%
KAMINA/Fuels	84%	84%

Unfortunately, due to the extremely small number of examples available to create these estimates, all the success rates given so far may differ by up to ± 5 to 10 percentage points from the true population. In addition, the short-term nature of these tests ignores sensor drift, which is a common and significant problem with E-nose sensors. Thus the next goal of this program is to gather enough data to improve the accuracy of these estimates, and study the effects of drift. More data may also allow the use of the Mahalanobis or Quadratic classifiers, which can differentiate between the odors even better than the Linear classifier.

SUMMARY

Numerous applications in the Space Program exist that could benefit from the unique, powerful analytical capability of the E-nose. Three specific examples had been identified (general air monitor for organic vapors in closed cabins, hypergolic fuel monitor at pressures ranging from 3 to 15 psi, and as an pre-combustion alarm). These represent specific needs within current NASA programs, and each presents a unique challenge for the E-nose.

The viability of the E-nose technology for the three identified applications was confirmed. Specific instruments have been identified which could readily respond to the vapors of interest at the requisite concentration levels. The MOS arrays appear to be the sensor system of choice due to lower limits of detection relative to the other arrays systems tested. Several instruments were identified that could detect the organic vapors at the SMAC levels (e.g., KAMINA, I-Pen2, and Sam Detect) and the pre-combustion vapors (e.g., KAMINA and Sam Detect). Only the KAMINA instrument was able to detect hypergolic fuels at 10 ppb. Instrument sensitivity is only one aspect of the E-nose. For identification, the E-nose must be trained and tested. Data sets were collected and modeled using vendor supplied algorithms and protocols. It is found the verification runs did not necessarily confirm the accuracy of the model. It was found that in many cases, the vendor software did not provide information on probability of identification, and this render it difficult to do a thorough assessment of any model. To alleviate this shortfall, in-house modeling was performed on the data sets. This not only provided an independent means of assessing the information content of the raw sensor data, but also allowed greater flexibility in developing models than that provided by vendors. It was demonstrated that short exposures can provide as high a accuracy as steady-state responses, a finding that will facilitate the collection of larger data sets. It was also predicted that the accuracy of identification could exceed 90% for the organic vapors and for the hypergolic vapors.

As the E-nose is successfully deployed, it is probable that additional applications will be identified. The early results of this program were presented in this paper. Although preliminary, significant findings have been demonstrated. Much more testing and development needs to be done before deployment for actual missions.

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Biography

Rebecca Young :

William Buttner received his B.S. degree in chemistry from Loyola University of Chicago in 1976 and his Ph.D. in chemistry from Michigan State University in 1982. After a two-year post-doctoral appointment at the University of Arizona, he joined Argonne National Laboratory. Between 1985 and 1990 he was a staff scientist in the Environmental Research Division. While at Argonne, Dr. Buttner was the Principal Investigator on numerous projects dealing with environmental monitoring of toxic chemicals. These programs included the development of chemical detection instrumentation and the design and characterization of miniaturized chemical sensors. In December of 1990, Dr. Buttner joined Transducer Research, Inc. where his duties include the development and characterization of new chemical sensors and instrumentation. Dr. Buttner has been the Project Manager and Principal Investigator for the DOE-Funded Program "Field-Usable Portable Analyzer, For Chlorinated Organic Compounds", the LMITCO funded program "In-Situ Sampling of Aqueous-Phase Contamination by Chlorinated Solvents", and the Waterways Experiment Station funded program "Chemical Sensors for the Scanning Cone Penetrometer Analysis System", among others. In 1993, Transducer Research, Inc. was purchased by TSI, Inc. In 1999, he joined Dynacs Engineering at the Kennedy Space Center and has been active in supporting the use of chemical sensors in NASA space program.

Bruce Linnell received his B.S. in Electrical Engineering from the University of Southern California in 1981, his M.S. from North Carolina State University in 1998, and his Ph.D. from NCSU in 2001. He is currently working at Dynacs Engineering at the Kennedy Space Center, providing pattern recognition analysis for various E-noses.

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