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Electronic Nose
Evaluation of Kamina prototype unit

Nathan Schattke
Research Associate
Illinois Institute of Technology
For Rebecca Young NASA/KSC

Abstract

The Kamina, Sam and Cyranose electronic nose systems were evaluated and partially trained. Much work was performed on the Kamina as it has the ability to respond to low (less than 10 ppb) concentrations of hydrazine compounds.

We were able to tell the difference between Hydrazine (Hz) and Monomethylhydrazine (MMH) in standard clean humid air. We were able to detect MMH in reduced pressure (1/3 atm) at about 250 ppb, however the training set was to far from the real situation to be useful now.

Various engineering and usability aspects of both the noses was noted, especially the software. One serious physical engineering flaw was remedied in the Kamina system. A gas flow manifold was created for the Sam system.

Different chips were evaluated for the Kamina system. It is still unclear if they can be exchanged without retraining the software.

The Sam Detect commercial unit was evaluated for solvent detection and evaluation. It was able to successfully identify some solvents.

The Cyranose was observed and evaluated for two days. It has the ability to detect gasses in the 100 parts per million level but not the 10 parts per billion level. It is very sensitive to humidity changes; there is software to partially handle this.

Introduction

The electronic nose idea is that several sensors when working together can give much more information than the individual sensors not networked. Multiply or exponentize instead of add. There are advantages and disadvantages with this much information. Such as, you have to calibrate much more upfront and much less in the future. You may also be able to calibrate on a general system then use that information to make all future calibrations for new sensors or components take much less time and money. Information from the electronic nose includes qualitative and quantitative elements and since it runs through a versatile analysis machine the direction of analysis can be performed several ways simultaneously. But in some packages the information can only be looked at one way thus allowing you to have only quantitative or qualitative but not both. As you can see the above explanation in not limited to chemical sensors, the idea would give even better service if other types of sensors were added to the matrix. The methods used for information analysis are perfectly applicable to the following sensors as well as all types of chemical sensors: pressure, temperature, positional, sonic, radar, sonar. The electronic nose doesn't really care what form its signal comes in. One interesting aspect of the Kamina nose system is the analog change between sensors. This causes the system to change as a whole, leaving the difference between the sensor elements as the indicating information. This may have positive effects on calibration and component replacement.

Electronic Nose Basics

If the above was a bit much for you, here is a primer on the electronic nose.
An electronic nose mimics the animal nose by doing three things: Sampling-Sniffing; Sensing-Smelling; Evaluating-Thinking. Sampling is very important; the signals from the sensors are different if gas flows over them for 15 seconds rather than 10 or if the gas flows past the sensors at 100 milliliters per minute or 200. In the animal nose we have physical control over how hard we sniff and how much goes through the mouth or nose. The brain takes that into account when it thinks about the signal. I have yet to see an electronic nose training set take advantage of sniffing deep or shallow. Many electronic nose systems spend a lot of money and build complicated equipment so that the gas with the smell in it comes to the sensors in easy to understand packets. For instance the operator may set up a series of jars with rubber lids, heat them to be warm, stick two needles in, push special clean gas in through one and take gas out

through the other. Other systems use less exacting methods such as: a fan to pull air over the sensor, diffusion, or a manifold and pressurized gas. This has disadvantages because the nose has to be trained to recognize all the different atmospheric conditions it could be exposed to (and you get many more with the open air than with jars). The sensors react differently to 10 ppb of Hydrazine at 70 F and 30 RH (relative humidity) or 70 F and 5 RH.

Sensing by the human nose is performed by more than a million different sensing cells, made up of over 1000 types of cells. These are spread throughout the complicated nasal passages. Signals from these sensors go to nerves. Sensing by the electronic nose as a general idea is that some change is produced on the sensor that causes or changes a signal to the computer. This can be a change in the capacitance of a catalytic surface, change in the resistance from a catalytic surface or sensitive plastic, change in the color of a catalyst at the end or on the side of a fiber optic cable, change in the frequency of a coated crystal, or increase of electrons from a fuel cell type sensor. All these types and more can be added to the system and tuned to detect the possible gasses of question. Note that this type of thing can work for liquids just as easy by using sensors that react in a liquid environment, thus giving you an electronic tongue.

Evaluating by the animal nose is both instinctive and learned. The action of pheromones on our behavior is an example of instinct. Most of what we know about the smells around us are learned. Evaluating by the electronic nose is very different. Each sensor will have a standard normal signal. Then the gas of interest will change some of the normal signals. These changes are compared to previously known samples and the results reported. One way to think about this is to imagine each sensor as an axis on a graph. If you had three sensors you would have a three-dimensional graph. As you trained the system with one chemical the three sensors would each react a certain amount. Then with another chemical they would each react in another way. So if you put a ball in the graph at the first chemical and another ball at the second they should be in different places. If they are not then the sensors chosen probably do not differentiate these two chemicals enough. This is overcome by having many sensors (10-100). Now with many sensors you have that many dimensions. As you can imagine visualizing and using 100 dimensional space is rather difficult. Practically the signals are normalized to the difference from the average or a fraction of initial signal then a plane is cut through all the dimensions that shows the best change between the areas of the different gasses in two dimensions.

Equipment and Results

The chemical applications laboratory at KSC is a large five-hood lab on the second floor of the O&C building. Two of the hoods have Kintek diffusion type gas generators. Samples of liquid are placed in special containers that have a part that will let the vapor diffuse through. These are in turn placed in an oven of very carefully controlled temperature. Inert gas is passed over the containers picking up a small amount of the vapor from the liquid. As long as there is some liquid in the container, the temperature and gas flow rate are constant the concentration of the exit vapor will remain the same. This "mother" gas can be further diluted with any gas or with a Miller-Nelson unit which produces air of known temperature, flow rate, and humidity. These

tools can accurately, quickly, and repeatedly deliver nine different types of vapors delivered to the noses.

The Kamina electronic nose is composed of a sensor chip, data acquisition and control, and computer control and analysis software.

The sensor chip is made with lines of conductor material interspaced with lines of semiconductor material (tin oxide, $SnO2$). These are in the X-axis. In the Y-axis are heat platens and silicone oxide (glass) covering. The heat (from underneath) is uneven so that the segments on one edge are hotter than the other edge. The glass covering is thicker on one edge than the other. This gives 38 segments each a little different from the next all of them showing the same surface to the gas stream. The chip is held up on four posts of ceramic material.

The data acquisition and control starts with the connection to the individual segments. It is performed by very fine (thinner than 0.001") gold (soft) wires attached to the chip then to the holder. These wires span through the air and are not insulated or kept separate except by bending. Although these wire should not be touched in the normal course of operation they are easily bumped which can cause shorting and a loss of signal. The other side of the gold wire is connected to the surface of a much larger chip holder with pins leading down and away from the sensing surface. This chip is plugged into a holder. This holder is plugged into another chip holder. This holder is soldered onto a small printed circuit board (PCB). This PCB has pins leading down to connect with a socket on the next PCB. This pin and socket connection was found to be a major cause of noise in the system from uneven pressure on the connections. In my opinion this connection system is too complicated and prone to failure. Training the nose requires that the signals be steady day to day. This bad connection was causing a different type of signal depending on how much the inner board was warping. Connected to the lower PCB are two data acquisition and control PCB's with electronics on them. The connection here has no faults. At the bottom of the module are two multipin sockets, one for data, and the other for power. The power module is a separate box that must be less than 18" away. The ribbon connectors are of a low cost and quality at times leading to a noisy signal.

The computer control and analysis software is supplied by Kamina. There are two ways to analyze the data, early transition detection (ETD) and resistance detection. ETD was used exclusively because it has an initial reference value that the signal is compared to. This allows the change in signal to be compared to the others no matter what level you start at. Some of the variables that can be modified in the Kamina program are:

Signal homogenization by logarithm- this allows vastly different signal changes to be treated as if they were not very different.

Signal Normalization by 1- Resistance 2- Resistance divided by Reference or 3- Reference divided by Resistance – this changes the difference from normal to be analyzed two different ways or just taking the resistance signal strait.

Median Normalization by divide by median- this gives you a stable baseline to reference all different measurements to.

Signal Filters by Time Series or Sensor Array- this option was not explored by this researcher.

Principal Component Analysis (PCA)- This is a very strong tool for this type of data analysis. It analysis the data to find the parts (components) of the data set that mostly (principally) cause the differences. The variables for this are number of principal components (up to 12 have been used) and weather or not to scale by standard deviation.

Linear Discrimination Analysis (LDA)- This is the final analysis tool used by this researcher. This analysis tool cuts the best plane through the dimensions and shows a two dimensional graph of the best fit to that.

SIMCA- this option was not used by this researcher. As explained by Susan Rose-Pehrsson this is similar to LDA in that it cuts a plane through the many dimensions but instead of being a strait plane it is a planer surface that can be bent. This could show differences better.

Neural Net- this option was not used by this researcher. I understand there is much difference between versions of this type of program. This could be a good or a bad one.

The best options for analyzing data for difference of chemicals HZ, air, and MMH was: No Logarithm, Reference divided by Resistance, divide by median, PCA w/ standard deviation and 12 components, LDA w/ 12 components. These settings and a good model on a stable sensor system were effective for determining the difference between MMH and Hz.

Lower Pressure tests

From May 31st to June 8th several tests were performed on the Kamina system at lower pressures in a bell Jar. Note that this was before the new chips were in and the board pull away problem was fixed. It is expected that if the experiment were to be performed today it would be more sensitive and accurate for detection of hydrazine compounds. Even with these limitations the group was able to successfully detect hydrazine compounds at 1/3 to ¼ atmosphere.

The equipment used was bell jar prepared by Paul Yocum of Dynacs. This chamber has as serial and 115 volt pass-throughs. The power supply and sensing head of the Kamina was placed inside. Another pass-through was a 1/4 " line with a ball valve. And the last was a ¾" pump down hose to a roughing pump (standard high quality lab roughing type with oil).

In practice the system was pumped down to 150 torr (0.20 atm), the Kamina responded. Then five minutes later (the standardization of the timing was Bill Buttners' idea) 4.0 liters of gas was introduced through the ¼" line. This raised the pressure to about 250 torr (0.32 atm). Then

five minutes later another 4.0 liters of gas was introduced. This raised the system to 350 torr (0.46 atm).

Tests were initially performed with air, the change from 760 to 150 torr was noticeable, and the change from 150 to 250 and 250 to 350 was insignificant. With MMH the system reacted clear and strong. The system concentration of 280 ppb MMH was the lowest tested. LDA plots were prepared and clear separation was established between 760 torr air, 150 torr air, 281 ppb MMH, 407 ppb MMH, 1286 ppb MMH, 1863 ppb MMH.

This experiment has been planed to change. The system will be modified to allow constant flow of gas to better simulate the changes expected.

Engineering the Kamina

The system produced by Kamina was a generalized system designed for burnt wire detection. It had a head with a small (1 ½" dia.) fan pulling air across the sensing area. The other side from the fan was a Teflon membrane, this had to be removed for hydrazine detection it can degrade the signal by as much as 2 ppm.

A new manifold was received June 19th made with glass as the main material in contact with the gas. A small amount of inside edge of a Viton gasket between the glass manifold and the chip holder surface was open to the gas, it is expected that this would not significantly effect the signal. The sensor chip holder surface is made of an epoxy coated metal surface. The volume of the glass manifold is relatively small at about 15 ml of volume over the chip. There is no disturbance in the manifold area above the chip, this could lead to an uneven and unpredictable flow over the sensing surface. The flow of gas should be disturbed in the sensing area for good mixing. The glass manifold was held in place with two half circles that were screwed down onto a ring. The half circles overlapped the glass manifold and pushed it down. At flow rates of 2 liters per minute this system can afford some leakage. If lower flow rates were preferred then a, low durometer closed cell silicon foam, would be preferable.

On July 13th the Kamina was repaired. The two PC boards that plugged into each other at the top were being pressed together unevenly. Bill Buttner, Steve Parks and Tim Hodge helped design a pin system to hold the two other corners in place. It worked; the random easily changing noise was eliminated.

The complete system for getting gasses onto the sensing surface and away and connecting to the electronics would have to be completely redesigned in order to travel to space and be usable. The present system is too fragile and prone to failure.

The software is good in that it measures the difference from the base on segments in order to determine the gas in question. Generating the rule set to check against could much easier, see the Cyranose software system. Currently effective models or rule sets are a shotgun approach of

trying all sorts of different analysis tools. Much of this could be generated and checked by the program.

A problem of 3.2 mega ohms exists for all four chips and both manifolds. The system responds to a gas signal by lowering resistance for the segments. The median of all the resistances is reported on a graph in the main part of the program. When this median reaches 3.2 Mohm it will stay at that level for a while or for a very long time, depending on how far down the final signal will go. This is probably an intermediate hardware translation problem centered around the 32768 or 2^{15} bit level transition. The data at this level is suspect.

The Sam Detect system was only used for a short time. Several areas of improvement should be explored. The filters over the sensors are sintered metal, this will catalytically react with some materials including hydrazine and cause it not to be detected. These should be removable. The system resets apparently at random. This should be either well automated during training or manually switchable. No manifold was available, a fine one was fabricated by the Dynacs group of Bill Buttner, Tim Hodge, Steve Parks, and Paul Yocum. The inside of this was Teflon.

Interchangeability

Four chips were evaluated with the Kamina system. The hardware was not working well enough to determine if they could be interchanged. From the data so far and visual observations this is not likely. The fabrication process leaves many differences between the different chips. The observed, after burn in, polar charts showed much different patterns. Further work needs to be performed. Future work should focus on reproducibility of sensors. This would assist the space exploration by allowing a signature of an unknown gas to be received and later analyzed on earth. Such a system could greatly help with medical diagnosis on long missions.

Conclusion

The ability to successfully detect hydrazine compounds in the 5 ppb range is a great success that cannot be understated. Tin Oxide sensors all alone cannot do the job. The current electrochemical sensors are the best available and yet are still not stable enough, being particularly susceptible to CO_2, a common element of human breath.

Use in the airlock of the ISS or shuttle is promising for Hz leak detection. This chip concept is very strong. The change in signal with time is easily handled and controlled. The sensitivity stays rather high. The range of chemicals available for detection is high but perhaps skewed towards the hydrocarbons. If this tool were to be used with medical diagnosis a predigestor or filament may be desirable. Use in the airlock of the ISS or shuttle is promising for Hz leak detection.

This concept of changing sensors a small amount across the array of an electronic nose that Kamina has introduced should be expanded to perform better. Of use to NASA would be a small

integrated nose system that has radio or IR communication to and from a computer, has the sampling system with: pump, flow path, calibration standards, sensors, filters, and data collection all on one platform. Even more useful but also much more complicated would be a heterogeneous sensor system of resistive and other types of sensors.