

# Application of Odor Sensors to Ore Sorting and Mill Feed Control

## Final Report

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## ABSTRACT

Abstract – Control of the feed provided to mineral processing facilities is a continuing challenge. Much effort is currently being devoted to overcoming these problems. These projects are usually described under the general headings of Mine-to-Mill Integration or Mine-Mill Optimization.

It should be possible to combine the knowledge of ore type, mineralogy, and other characteristics (located in the mine modelling system), with the advanced capabilities of state-of-the-art mill control systems, to achieve an improved level of control in mineral processing that will allow optimization of the mill processes on an almost real-time basis. This is not happening because mill feed it is often treated as a uniform material, when in reality it varies in composition and characteristics.

An investigation was conducted to assess the suitability of odor sensors for maintaining traceability in ore production and processing. Commercially available sensors are now used in food processing, environmental monitoring, and other applications and can detect the presence of very small amounts (0.1 - 500 ppm) of some molecules. An assortment of such molecules could be used to “tag” blocks of ore as they are mined, according to their respective characteristics. Then, as the ore came into the mill, an array of “electronic noses” could be used to assess its characteristics in real time.

It was found that the Cyranose 320™, a commercially available odor sensor, can easily distinguish among samples of rock marked with almond, cinnamon, citronella, lemon, and orange oils. Further, the sensor could detect mixtures of rocks marked with various combinations of these oils. Treatment of mixtures of galena and silica with odorant compounds showed no detrimental effects on flotation response in laboratory tests.

Additional work is recommended to determine how this concept can be extended to the marking of large volumes of materials.

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## EXECUTIVE SUMMARY

It should be possible to combine the knowledge of ore type, mineralogy, and other characteristics (located in the mine modelling system), with the advanced capabilities of state-of-the-art mill control systems, to achieve an improved level of control in mineral processing that will allow optimization of the mill processes on an almost real-time basis. This is not happening because mill feed it is often treated as a uniform material, when in reality it varies in composition and characteristics.

An investigation was conducted to assess the suitability of odor sensors for maintaining traceability in ore production and processing. It was found that the Cyranose 320™, a commercially available odor sensor, can easily distinguish among samples of rock marked with almond, cinnamon, citronella, lemon, and orange oils. Further, the sensor could differentiate among mixtures of rocks marked with various combinations of these oils.

In laboratory tests, it was found that treatment with odorant compounds had the following effects on flotation response in a simple quartz/galena mixture: no frother addition was required – the citronella and cinnamon oils created a very strong froth; slightly higher lead recoveries were obtained on mixtures that had been treated with the oils; and lower rougher concentrate grades were obtained on mixtures that had been treated with the oils.

Additional work is recommended to determine how this concept can be extended to the marking of large volumes of materials. After suitable sensors and odorants are identified in lab tests, an exhaustive field testing program will be required. Nonetheless, odor sensing systems show promise for use in tagging various types of ore, to allow tracking of that ore through the mining and milling process. This could improve the integration of mine and mill operations, allowing the knowledge held in the mining and geological computer models to be transferred to the mill control system. Ore response to many mineral processing operations could be improved by having a more detailed knowledge of the nature of the feed.

## INTRODUCTION

A brief survey of publications since 2000, under the keywords Mine-to-Mill Integration or Mine-Mill Optimization, found 15 articles. Seven of these articles discussed the control of fragmentation, and how it can be optimized between blasting (in the mine) and comminution (in the mill) [Dare-Bryan, et al., 2001, Grundstrom, et al., 2001, Kanchibotla, 2003, Marton and Crookes, 2001, Morrell and Munro, 2000, Scott, et al., 2000, and Wooller, 2001]. Two articles discussed how the mine design could minimize dilution and waste production [Anon., 2001a, and Klein, et al., 2003]. Five articles discussed the relationship between ore mineralogy and overall mill performance, and addressed, at least in part, how the integrated 'mine-mill system' might be optimized [Lotter, 2002, Paley and Kojovic, 2001, Pease, et al., 2001, Taylor and Altman, 2001, and Williams and Holtzhausen, 2001]. The final article was general in nature [Herbst, 2000].

This brief literature survey indicated that the primary effort to date in mine-to-mill integration has been optimizing the relationship between blast fragmentation and comminution. The concept is referenced in the literature as early as 1991 [Smith and Hautala], and is addressed in some detail beginning in 1995 [McKee, et al.] In some locations, this part of the mine-to-mill integration effort has been successful. At the same time, there is the potential to accomplish much more.

The relationship between ore composition or mineralogy and downstream processing is well known in the mining industry. In fact, the very definition of a mineral deposit as an orebody depends on its susceptibility to processing in an economical manner. It should be possible to combine the knowledge of ore type, mineralogy, and other characteristics (located in the mine modelling system), with the advanced capabilities of state-of-the-art mill control systems, to achieve an improved level of control in mineral processing, allowing optimization of the mill processes on an almost real-time basis. This concept was described as early as 1993 [Robertson and Sehic], and is often discussed by mine and mill operators. Unfortunately, it is difficult to accomplish, for two reasons: First, mining processes, including drilling, blasting, loading, and transport, may allow for segregation of mined ore by composition, mineralogy, or other characteristics important in subsequent processing; second, mill facilities may not allow segregated storage of ore, at least not to the extent that might be used in an optimized operation.

Both these limitations could be overcome to some extent by a method of attaching tracers or tags to individual blocks or volumes of ore. If those tracers could be detected or sensed, the information about each ore block, gained during exploration and mining, could be used as input data to the mill control system. This would allow the real-time optimization of ore processing.

### 1. TRACERS AND TAGS – CURRENT TECHNOLOGY

A brief discussion of tracer technology is useful at this point. Tracers or tags are widely used in the manufacture and distribution of discrete products. The most familiar example is the well-known bar code, which is found on virtually every consumer product [Footlik, 1994, and Scanlon, 2003]. Of course, bar codes must be attached to individual objects, and those objects must be transported or presented in such a way that the bar code is accessible to the sensing device, a scanner. Clearly, bar codes cannot be used for large volumes of ore, where particle size and shape vary widely, and particle position changes at random as the ore is transported.

Radio frequency (RF) tags are small plastic chips, each with an embedded RF transmitter. These tags can be sensed from a distance, and do not require direct, line-of-sight contact with the sensor [Pidgeon, 2003, and Anon., 2001b]. In some cases, they can be sensed through a covering of solid or particulate material. Thus for tagging ore, they overcome some of the limitations noted for bar codes. However, there is no reasonable way that RF tags can be used with volumes of ore, when those volumes comprise large numbers of discrete particles, because tags would have to be attached to a large number of the particles within each volume.

Radioactive tracers have been used to determine the behavior of particles in a natural or synthetic process [Cheong, 1993, and Walsh and Rao, 1986]. Typically, the radioactive tracers are selected to have properties similar to those of the particles that must be followed through the process in question, and mixed with non-radioactive particles for feed to the process. After processing, the tracer particles are located with radiation detector, allowing documentation of their individual responses to the process. For example, Walsh and Rao [1986] used irradiated gold particles of various sizes to determine how particle size was related to recovery in gravity concentration devices. The size distribution of the irradiated particles was matched closely to that of the gold being processed. Note that the tracers are used to determine the response of particles with certain characteristics to a given process, and not to mark a given particle or group of particles. Again, the limitations of this technique for tagging large volumes of ore are clear.

Where a large number of particles must be tagged, surface coatings are used. An example is the study of particle diffusion through membranes. Particles of a given size are tagged with an analyte that is sensitive to a given method (usually a form of optical spectroscopy), and then coated with glass to protect the analyte from the liquid in which the particles are transported [Mulvaney, et al., 2003]. This method allows the simultaneous coating of large numbers of particles, but is designed for small particles of relatively uniform size. Furthermore, it requires optical line-of-sight for particle sensing. Thus it would be difficult and expensive in applications large volumes of ore.

## 2. TAGS FOR LARGE VOLUMES OF ORE

Reflection on the preceding discussion of tracer and tag technology allows compilation of a 'specification list' for tagging large volumes of ore:

1. It must be possible to tag all or almost all the particles in a given volume of ore, regardless of particle size. This may be all the ore in the bucket of a loader or shovel, in a haul truck, or on a conveyor belt.
2. Tagging must be relatively inexpensive, and uncomplicated.
3. Ore particles must remain tagged at least until they enter the milling process. For example, we might want to scan the ore on the belt feeding a SAG mill or crusher, and calculate the distribution of ore types in the mill feed.
4. Detection of the tags must be possible from a distance, and, ideally, through a covering of ore particles.
5. Detectors must function in typical mine/mill environments, being resistant to vibration, dust, shock, temperature variation, etc.
6. Tagging must not cause problems in the mill operations; for example, tags must not alter the surface chemistry of ore particles in a way that will interfere with froth flotation.
7. The tags must be inexpensive and environmentally benign.

A review of available sensor technology indicated that chemical odor sensors may be suitable for this application.

### 3. ODOR SENSOR TECHNOLOGY

Odor sensors are modeled after the human olfactory system. Human smell is quite sensitive: allyl mercaptans are typically detected at a concentration of 0.05 ppb [Maruniak, et al., 1983]. It is also very discriminatory: skilled perfume chemists can recognize and distinguish 8,000 to 10,000 substances on the basis of odor quality [Axel, 1995].

Odor sensors fall in the general category of chemosensors, which are devices that are capable of converting a chemical quantity into an electrical signal. In general, chemosensors function with a change in mass, or in the electrical, thermal, optical, or properties of a sensing material. The development of chemical odor sensors began in the early 1980s [Vanneste and Geise, 2003], and has progressed rapidly. Commercially available sensors are described briefly in Table 1 [Nanto and Stetter, 2003, and Vanneste and Geise, 2003]. Of the sensors listed in Table 1, the MOSFET type is the most widely used.

The sensors listed in Table 1 vary widely in size and price. Some are handheld units, with an approximate cost of US\$5,000 each. Others must be operated with a laptop or desktop computer, and may cost as much as US\$100,000. Much of the variation in cost is related to the pattern recognition software that is used by the sensor.

Odor sensors are used with several data-processing or pattern recognition techniques, including multi-layer perception, principal component analysis, fuzzy learning vector quantization, cluster analysis, Kohonen network, linear regression, feature weighting, least squares, discriminant function analysis, and fuzzy reasoning. Use of one or more of these techniques makes it possible for sensors to function with a kind of low-level intelligence, with which patterns and trends can be detected and indicated. These techniques are described in detail in the technical literature. Odor sensors are used in numerous applications. Pearce, et al. [ibid.] identify the categories shown in Table 2.

In addition to the sensors listed in Table 1, several sensors based on changes in optical properties have been demonstrated in laboratory settings. There are also spectrographic (mass or infrared) systems available commercially, for on-line or at-line analysis of gases. These systems are smaller and more rugged than comparable laboratory units. For detailed information on sensor technologies, the reader is referred to Pearce, et al. [2003].

### 4. ODOR SENSORS FOR MINING APPLICATIONS

To allow the tagging and tracking of ore types from mining through milling, several odorants would be selected. Each type of ore would be tagged with a different odorant at an appropriate and convenient place in the mining process. Tagging would likely be done by spraying the ore with odorant compounds. This could be done when the ore is being loaded from the blast pile, or as it is hauled past a certain location. The spray tagging system could be interfaced with the mine modelling system, so tagging could be automated. The number of marker compounds used in a mine would depend on the number of ore types present and the number of variables to be characterized.

During transport to the mill or stockpile, the ore would retain the odors of its markers. Mixtures of ore types will be detected as mixtures of odors. An array of “electronic noses” could be used to assess the characteristics of that ore. The nose array would communicate with the mill control system, providing periodic updates on the nature of the ore coming into the mill. Nose arrays could be installed at more than one location in the mill, depending on need and application.

The marker compounds must have several key properties:

1. They must disperse widely over the ore when sprayed;
2. They must be non-toxic to mine and mill personnel;
3. They must be detectable above background odors in the mine and mill;
4. They must remain on the ore for a certain period of time (determined by the flow of ore from the mine through the mill);
5. They must be detectable through a certain thickness of ore (perhaps the thickness on a conveyor belt);
6. Their presence must not interfere with downstream operations, like flotation and thickening; and
7. They must be inexpensive.

Further research and experience will certainly add to and modify the above list.

Table 1. Summary of Commercially Available Odor Sensors

Technology	Summary Description	Commercial Supplier	Typical Applications
Chemoresistors	CP (conducting polymer)	Alpha M.O.S., Bloodhound Sensors, Cyano Sciences Inc., Marconi Applied Technologies, Osmetech	H <sub>2</sub> S, NH <sub>3</sub> , other organic gases.
	MOS (metal-oxide semiconductor)	Alpha M.O.S., Applied Sensor, Element, Forschungszentrum Karlsruhe, Lennartz Electronic, Marconi Applied Technologies, WMA Airsense Analysetechnik	H <sub>2</sub> , CH <sub>4</sub> , CO, H <sub>2</sub> S, NH <sub>3</sub> , CFCs, smoke, exhaust, cooking gases, alcohol vapors, water vapor, other combustible gases
Potentiometric	MOSFET (metal-oxide semiconductor field-effect transistor)	Applied Sensor	H <sub>2</sub> , NO <sub>2</sub> , NO <sub>x</sub> , CO, H <sub>2</sub> S, NH <sub>3</sub> , CH <sub>3</sub> SH, O <sub>2</sub> , Cl <sub>2</sub> , SO <sub>2</sub> , alcohols and other hydrocarbons
Gravimetric	QCM (quartz-crystal microbalance)	Applied Sensor, Daimler Chrysler Aerospace, HKR Sensorsysteme, Lennartz Electronic, Marconi Applied Technologies, Quartz Technology	Toluene, acetaldehyde, ammonia
	SAW (surface acoustic wave)	Alpha M.O.S., Daimler Chrysler Aerospace, Electronic Sensor Technology, Forschungszentrum Karlsruhe, Microsensor System	NO <sub>2</sub> , H <sub>2</sub> , H <sub>2</sub> S, SO <sub>2</sub> , CH <sub>3</sub> , C <sub>6</sub> H <sub>6</sub> , C <sub>2</sub> H <sub>5</sub> OH

Table 2. Applications of Odor Sensors

Application Category	Application Details	Examples
Gas analysis	Personal safety units, for one or two gases - O <sub>2</sub> , CO, CH <sub>4</sub> , organic volatiles (handheld or palmtop)	Inspecting containers, detecting leaks, identifying chemicals, verifying shelf life, monitoring waste and emissions.
	Electronic nose units for analysis of complex gas mixtures	In food processing: monitoring status, consistency, and spoilage; detecting fuel contamination
Olfaction for mobile robots	Trail following	Cleaning surfaces, exploration
(Mimicbehavior of small organisms; conceptual)	Plume tracking	Exploration, law enforcement
Environmental monitoring	Monitoring of water quality and contamination, landfills, contaminated soils, air quality (industrial emissions, indoor air quality, vehicle air quality, etc.)	Livestock odor classification for improvement of animal health and production, mold detection in air conditioning system
Medical diagnostics and health monitoring	Monitoring of breath, blood, ulcers, cultured bacteria, urine, and general environment	Detection of halitosis, sick building syndrome, and infections; sampling breath alcohol, rapidly discriminating among micro-organisms
Recognition of natural products	Detection of odors associated with processing, ripening, spoilage; discrimination among types of a product, sorting by type,	Toasting wood barrels, tofu freshness, cheese ripening, coffee roasting, tomato ripening, grain quality and classification, soft-rot in potatoes, fish or meat freshness or rancidity
Process monitoring	Bioprocessing	In bacterial cultures: estimation of metabolites in cultures, estimation of cell growth, visualization of cell stress; detection of infection
	Food processing	Aroma quality of cured ham, identification of sugar beet spoilage, off-flavor identification in grape juice and milk
Automotive/aerospace (mostly conceptual)	Monitoring of engine exhaust, cabin air quality, engine compartment, fluid and gas leaks	Space shuttle flight STS-95
Explosives detection	Detection of explosives vapors - DNT, TNT, RDX, etc., using optical excitation	Land mine detection - one instrument near commercialization
Cosmetics and fragrances	Application currently limited; mainly to improve sensitivity and selectivity of human workers	Detection of off-odor Yves St.Laurent perfume, discrimination among citrus juices with similar chemical composition but different odors

## PROJECT OBJECTIVES

The University of Utah has completed a project to investigate the properties of odor sensors and odorants, with the preceding application concept in mind. The objectives of the project were:

1. Locate commercially-available odor sensors and determine their characteristics;
2. Locate detectable, sprayable compounds with properties that appear suitable for use in marking ores;
3. Purchase selected sensors and marker compounds;
4. Perform lab tests on marker/sensor combinations, to determine sensitivity vs. time functions;
5. Perform lab tests on marked rock samples, to determine sensor response and ability to discriminate odor combinations; and
6. Perform bench-scale flotation tests on rock and ore pre-treated with selected marker compounds.

## METHODS

### 1. SENSOR SELECTION

A search of the technical literature and the internet indicated that several odor sensors are commercially available, as shown above in Table 1. Two sensors from Table 1 were identified for possible use in the project, the Cyranose 320™, manufactured by Cyrano Sciences, Inc., and the MOSES II, manufactured by Lennartz Electronic GmbH.

The Cyranose 320™, shown in Figure 1, was selected for further investigation for the following reasons:

- It has been demonstrated in commercial applications;
- It is portable and field programmable; and
- It is easily interfaced to a personal computer.



Figure 1. The Cyranose 320™

Important characteristics of the Cyranose 320™ are summarized in Table 3, on the next page.

Table 3. Important Characteristics of the Cyranose 320™ Odor Sensor

Weight	< 32 Ounces (0.91 kg)
Sensors	32-channel polymer composite sensor array
Display	320 × 200 graphic with LED backlight
Response Time	10 seconds
Field Calibration	User selectable
Keypads	5 buttons: Scroll Up, Scroll Down, Select, Cancel, Run
Communication	RS-232 link, up to 57,600 bps
Sampling Pump	50 – 180 cm <sup>3</sup> /min
Algorithms	KNN, Kmeans, PCA, CDA

The MOSES II, shown in Figure 2, was selected for further investigation for the following reasons:

- It has is modular, allowing configuration to suit the user;
- Various modules available, again to meet user needs;
- It is constructed with a ‘slot assembly,’ similar to that found in a standard personal computer, allowing easy changes in configuration; and
- It can learn to recognize various ‘odor suites.’



Figure 2. The MOSES II Odor Sensor

According to the manufacturer’s website, there are three modules available for the MOSES II sensor: the input module, the quartz microbalance (QMB) sensor module, and the MOS sensor module. The input module includes a pump, a mass-flow controller, temperature and humidity sensors, a three-way gas input multiplexer, and an on-board microcontroller. The QMB sensor module includes eight QMB sensors coated with different polymers, eight high-precision circuits to measure frequency changes, and a thermostat to maintain the chamber temperature stable. The MOS sensor module includes eight semiconductor sensors based on tin dioxide, each of which can operate at a temperature of 250 to 400 °C, programmed for each sensor individually.

## 2. SENSOR PURCHASE

Repeated efforts to communicate with the manufacturer of the MOSES II sensor were unsuccessful. The Cyranose 320™ was therefore selected for purchase, at a price of \$8,015.

### 3. SELECTION OF MARKER COMPOUNDS

The Cyranose 320™ can detect and discriminate among many compounds, including volatile gases, vapors of aromatic oils. *The Practical Guide to the Cyranose 320™* (Anon., 2001c), is a user's guide provided with the instrument. As examples, it describes the following applications of the sensor:

- Distinguishing between fresh and old basil;
- Distinguishing among three liquid fragrances;
- Distinguishing among phenyl ether, acetone, and phenol;
- Detecting and distinguishing among contaminants in water: 1,000 ppm propionic acid and 1,000 ppm high molecular weight acid; and
- Distinguishing among different types of pet food.

Thus it was clear that marker compounds could be selected from a wide range of chemicals. For convenience and safety in the planned laboratory tests, small amounts of aromatic oils were purchased from a chemical supply house. Aromatic oils were chosen because they are relatively inexpensive, they require no special storage and handling procedures, they clean up easily, and their odors are generally inoffensive to humans. The oils purchased included pine, orange, citronella, lemon, and almond. Later in the project, after it was determined that pine oil was unsuitable for use with the Cyranose 320™, cinnamon was purchased for use in subsequent tests.

### 4. SENSOR OPERATION AND INTERPRETATION

As noted above, the Cyranose 320™ is based on a 32-channel array of composite polymer sensors. Each sensor adsorbs a given odor to a greater or lesser degree, causing a change in its resistance. The changes in resistance ( $\Delta R/R$ ) of all 32 sensors comprise the 'smellprint' of a given odor. The software provided with the Cyranose 320™ uses four pattern-recognition algorithms to analyze these smellprints.

Smellprints are based on the instrument's response to samples readings of the same odor. These samples are called the training set. Before a sample is drawn, air is pumped through the sample chamber for purging. The sample is then drawn into the sample chamber, and the change in resistance of each sensor is recorded. The data are then transferred to a computer and analyzed using the Cyranose 320™ software. Each smellprint can be thought of as a point (with associated variance) in 32-dimensional space. A typical smellprint is shown in Figure 3. The black/gray bars summarize a training set of 10 samples of citronella oil, and the red bars show the response to a single sample of orange oil. The differences are clear.

The pattern-recognition software uses various algorithms to render this data into more understandable forms. One algorithm calculates the Euclidean distances for the K-means for each sample. A mean-distance diagram for five of the marker compounds is shown in Figure 4, on the next page. Note that this diagram could easily be used to distinguish cinnamon from the other compounds, but that the differences between orange and lemon, or almond and citronella, are less clear.

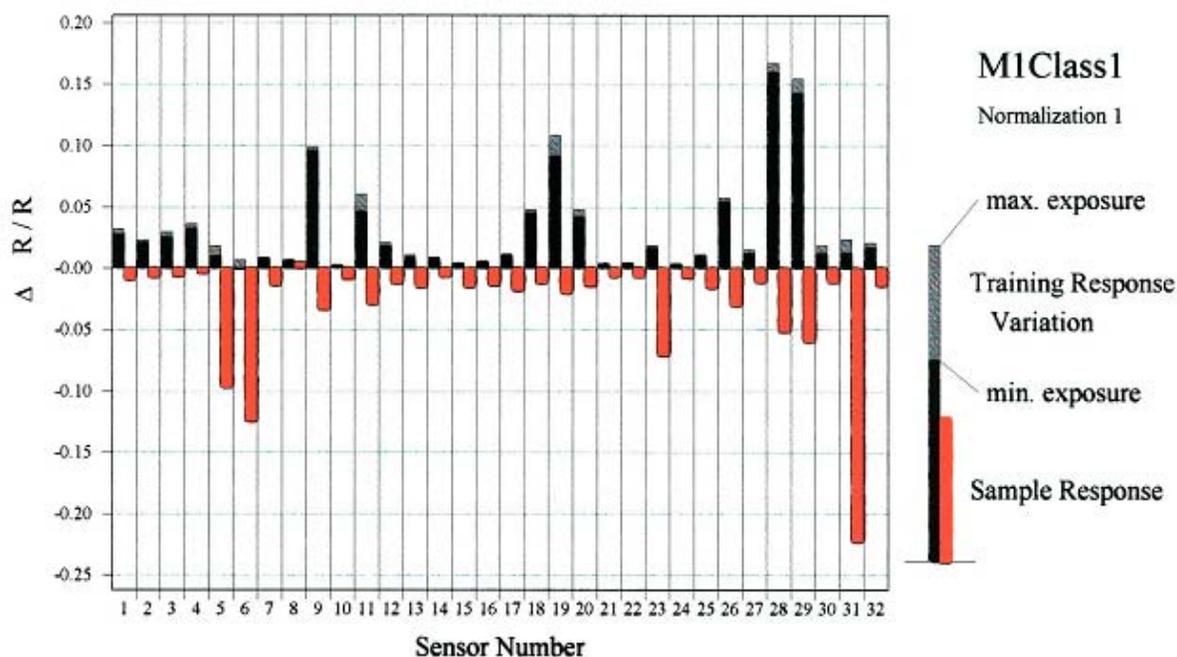


Figure 3. Typical Cyranose 320™ Smellprint

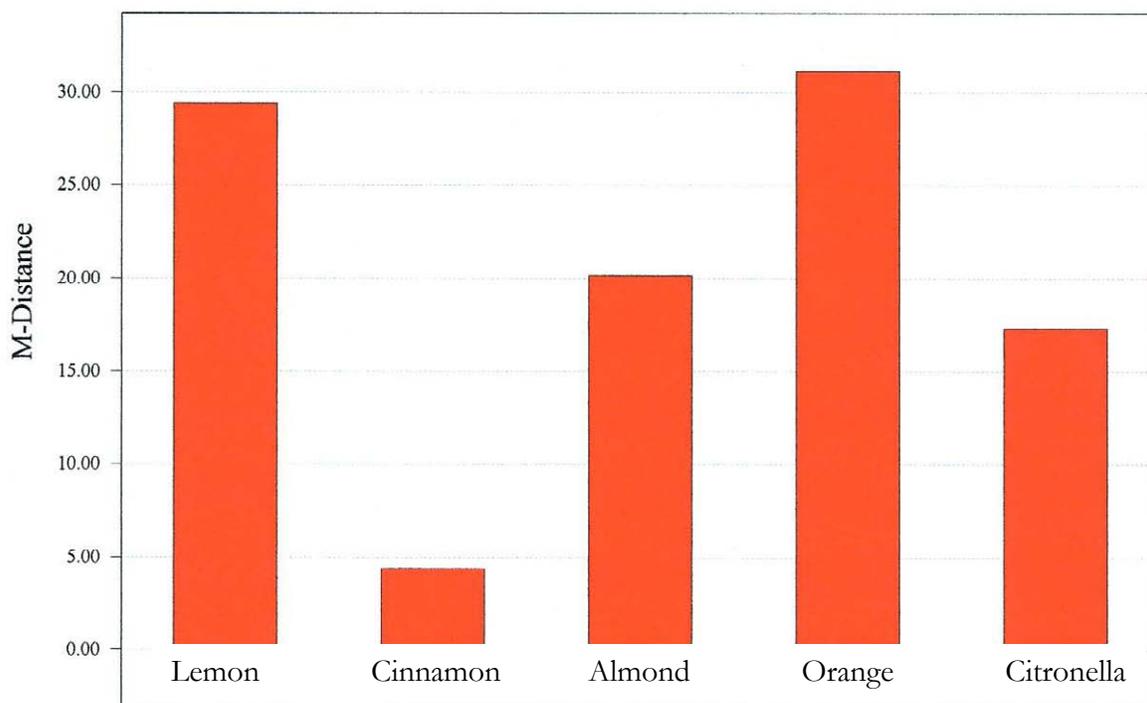


Figure 4. Mean-Distance Diagram for Five Marker Compounds Applied to Rock Samples

A second algorithm projects the 32-dimensional location of each point onto a 3-dimensional plot called a PCA plot. Figure 5, again on the next page, is a PCA plot for the same five marker compounds described in Figure 4. Finally, the 3-dimensional PCA plot can be projected onto a 2-dimensional canonical plot, as shown in Figure 6.

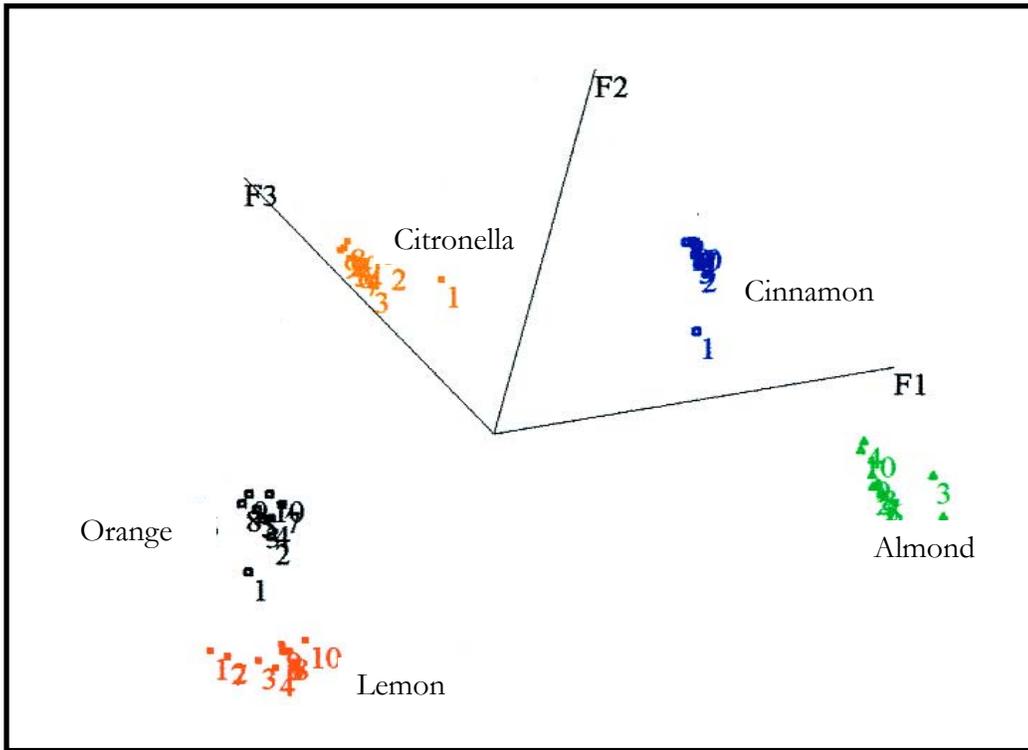


Figure 5. PCA Plot for Five Marker Compounds Applied to Rock Samples

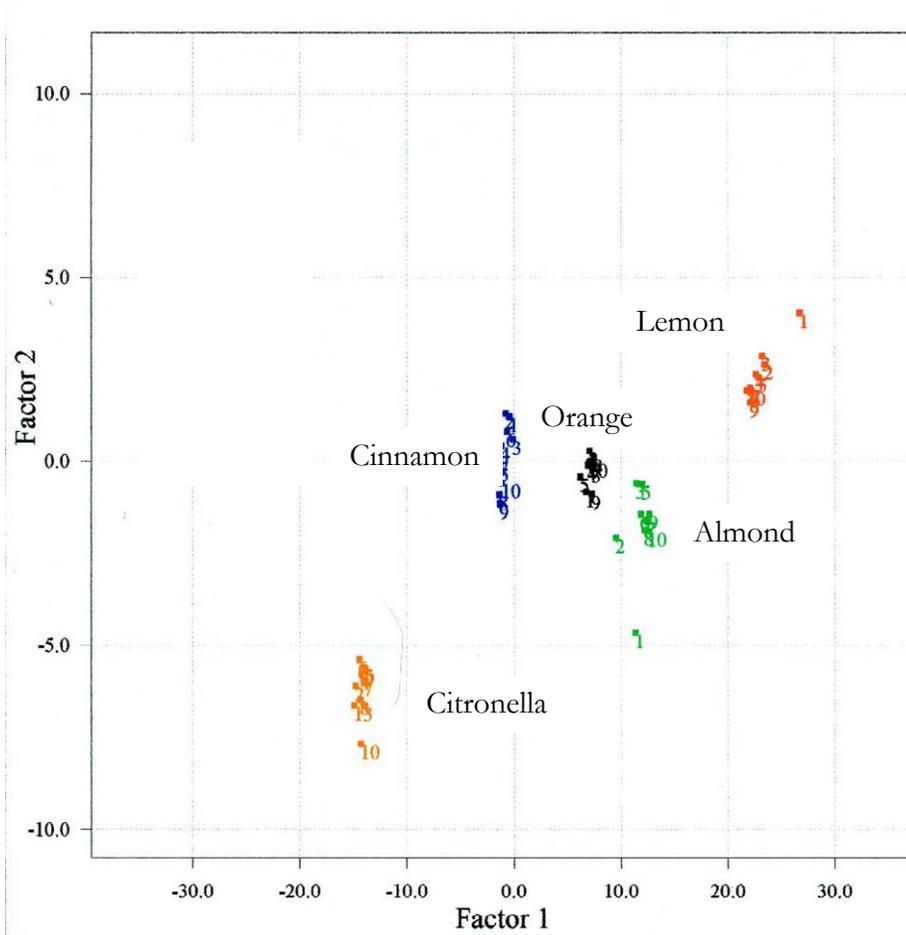


Figure 6. Canonical Distance Plot for Five Marker Compounds

Note that the PCA and the canonical plot show all ten of the measurements in the training set for each sample. Also note that in some of the samples, there are points that are not tightly grouped in the cluster, for example, Point 1 in the magenta cluster. In some cases, the performance of the instrument in discriminating among various odors can be improved by deleting outlying measurements from the training set.

From Figures 3-6, it is clear that the various outputs of the Cyranose 320™ can be used to discriminate among aromatic oils. The instrument can store 5 groups of 10 training sets each.

There is also a function called ‘Identify’ in the sensor software. In the Identify function, a single sample of an unknown is compared to the smellprints of all the training sets in a given group or method. If the smellprint of the unknown corresponds closely to that of one of the training sets, the instrument will display the name of that training set, along with a level of confidence for the identification. The level of confidence is given by a number of stars, from one to five. The quantitative meaning of these stars is not specified by the manufacturer

When the Cyranose 320™ is connected to a computer, the accompanying software allows modification of almost all aspects of the instrument’s operation. Purging and sampling times can be changed, parameters of the pattern recognition algorithms can be altered, etc.

## **LABORATORY TESTS**

Laboratory tests were conducted to determine the following:

1. Longevity of marker compounds on rock samples;
2. Experimental methods for assessing instrument response to various samples.
3. Instrument response to marker compounds;
4. Instrument response to marked rock samples (single marker compounds);
5. Instrument response to mixtures of marked rock samples (various marker compounds); and
6. Effect of marker compounds on the flotation response of rock samples.

None of the tests were exhaustive. Rather, they were designed as scoping tests, to determine whether there were any significant difficulties in the concept of using odor sensor for ore control.

### **1. MARKER COMPOUND LONGEVITY**

If odor sensors are to be used for ore control, it will be necessary for the marker compounds to be detectable for a certain period of time, as determined by the mining and milling cycles at a given site. However, compounds that can be detected by an odor sensor are by nature volatile, Thus each is expected to have a finite longevity when applied to ore or rock.

Further, it will be important to determine effective methods for applying marker compounds to large volumes of rock. Application by spraying is one method that might work well. To control costs, it may be desirable to dilute the marker compounds before they are applied.

This series of tests was designed to determine what longevities might be expected with different combinations of marker compound, diluted marker compound, and rock type, and what methods are suitable for applying marker compounds to rocks. The tests were designed for scoping, and were not intended to be exhaustive. Initially, it was intended to conduct these tests by applying marker compounds to rocks in different combinations, and then to record the

response of the Cyranose 320™ to the marked rocks. However, there was an unexpected delay in the delivery of the Cyranose 320™, and the test method was modified to maintain the project schedule, with longevities of marker compounds on rocks being determined by weight.

*Dilution of Marker Compounds:* The marker compounds were all volatile oils. Two diluents were tested: a 5% solution of dishwashing liquid in water and full-strength rubbing alcohol. Mixtures of mixture of oil and diluent (1:1) were blended at high speed for 5 minutes. The detergent solution did not mix well with the oils, and large amounts of foam were generated in blending. Alcohol worked well as a diluent.

Future work will require a careful study of the costs of marker compounds, and how those costs might be influenced by dilution. It will also be necessary to assess the effects of dilution compounds and methods on cost and subsequent process response.

*Longevity Tests:* The following variables were tested:

1. Marker compound – almond, citronella, orange, and pine oil;
2. Rock type – beryl, forsterite, serpentine, and wollastonite;
3. Application methods – spray and dip; and
4. Application strength – 50 and 100%.

The general test procedure was as follows:

1. Small rock samples were selected, and their weights were recorded. Rock samples were not washed before testing, and some rock types had much more dust on their surfaces than others.
2. Marker compounds were applied to the rock samples, by dipping or spraying.
  - 2.1. Dipping procedure:
    - Rock samples were held with tongs and dipped directly in marker compounds (full strength or diluted).
    - Samples were held above the marker compound container until dripping of excess ceased.
    - Samples were placed in tared weighing boats.
  - 2.2. Spraying procedure:
    - Selected marker compounds (full strength or diluted) were placed in standard plastic spray bottles.
    - Rock samples were placed on paper towels, spaced to avoid overspray among samples.
    - Samples were sprayed.
    - Samples were placed in tared weighing boats.
3. Weights of the marked rock samples were recorded at times of 1, 3, 5, 10, 15, 20, 30, 60, 90, 120, 150, 180, 240, 300, 360, and 420 minutes, or until there was no measurable change in weight.

These tests produced a large body of data, which is available on request. The following conclusions were drawn:

1. Longevity of diluted marker compounds was less than that of full strength compounds.
2. The amount of marker compound initially placed on a rock sample has a great influence on the longevity of the marker compound on that sample.

3. Dipping places a much larger amount of marker compound on samples than spraying. Retention of diluted marker compounds decreased to about 50% after about 50 minutes, and did not markedly decrease after 420 minutes; similarly, retention of full-strength marker compounds remained at or above 90% after 420 minutes.
4. The longevity of marker compounds applied by spraying is short – almost always less than 60 minutes. Thus spraying is not a satisfactory method of applying marker compounds.
5. Larger amounts of marker compound were adsorbed on the surfaces of rock compounds that were coated with rock dust. This had a greater effect on the amount of marker adsorbed than did the type of rock.

## 2. METHOD DEVELOPMENT

Accurate determination of instrument response to various samples required a consistent and carefully-developed procedure. All the samples were liquids or solids coated with liquids. It was thus necessary to contain the samples in a manner that allowed consistent sampling of a uniform headspace above the sample. Similarly, it was necessary to use a consistent procedure to draw odor-bearing air from above the samples into the instrument. Finally, it was important to devise a procedure that minimized or eliminated cross-contamination in the sample aspiration path and the sensor chamber.

Preliminary tests showed lack of repeatability when samples were taken by placing the instrument's sampling port directly over an open container. Various methods for use in sample containment during aspiration were tested. Thus a system using 50-ml, dropper-top bottles in conjunction with disposable aspirating needles was selected. Figure 7 shows one of the dropper-top bottles. Figure 8 shows an assortment of sampling needles. The two needles on the right were provided with the instrument, and are intended to be cleaned, dried, and re-used. The needle on the left is disposable; its cost is about one-tenth that of either of the re-usable needles. Figure 9, on the next page, shows the equipment configuration for sampling.



Figure 7. Dropper-top Sample Bottle



Figure 8. Sample Aspiration Needles

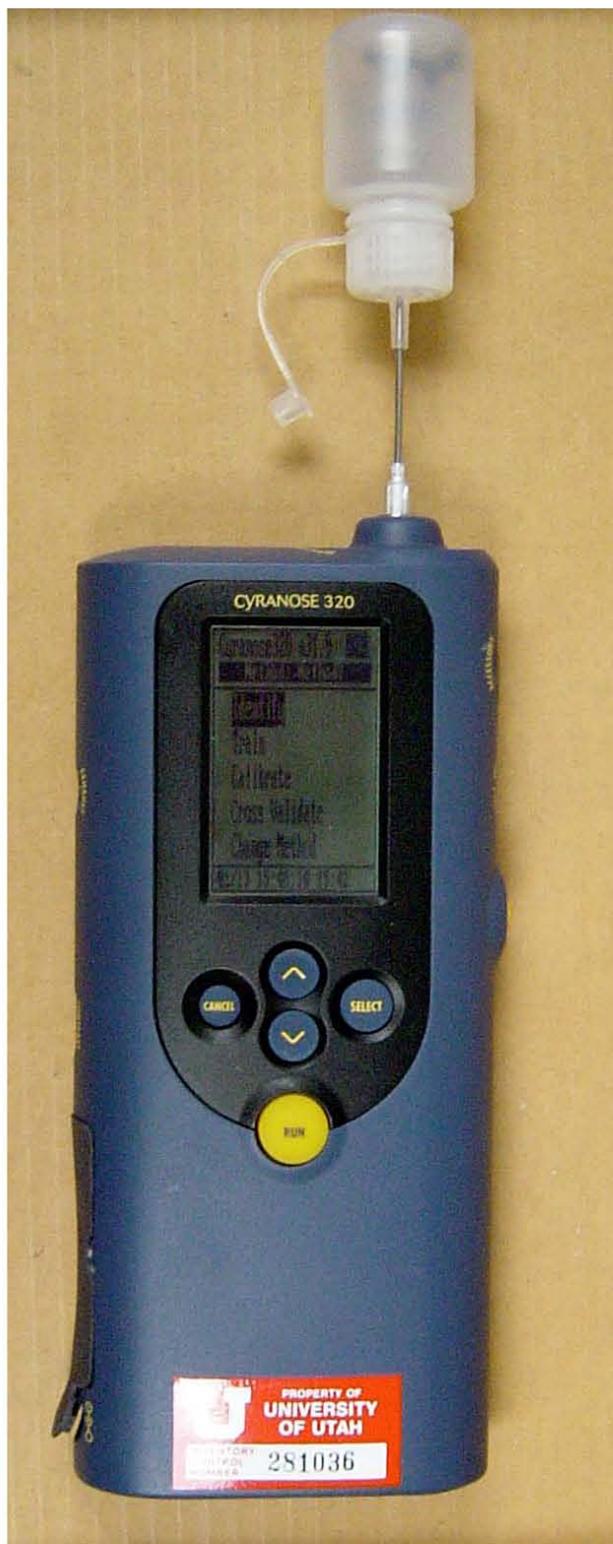


Figure 9. Equipment Configuration for Sampling

Additional effort was required to arrive at correct settings for the sampling parameters. It was found that accurate sampling of the aromatic oils selected as marker compounds required purge times between samples that were longer than the default settings. It was also found that the odor of pine oil was so strong that it ‘overpowered’ the instrument, making discrimination among other compounds impossible. Apparently the compounds associated with the odor of pine oil

had a long residence time on the sensor array, effectively deadening its response to weaker odors. Thus after the initial tests, pine oil was not used.

### 3. INSTRUMENT RESPONSE TO MARKER COMPOUNDS

Using the methods developed, small amounts of each marker compound were placed in dropper-top bottles. Training sets were developed for each compound by drawing 10 odor samples of each. Figures 10 and 11 show smellprints for pine and orange oil, respectively. The sample in both cases is almond oil.

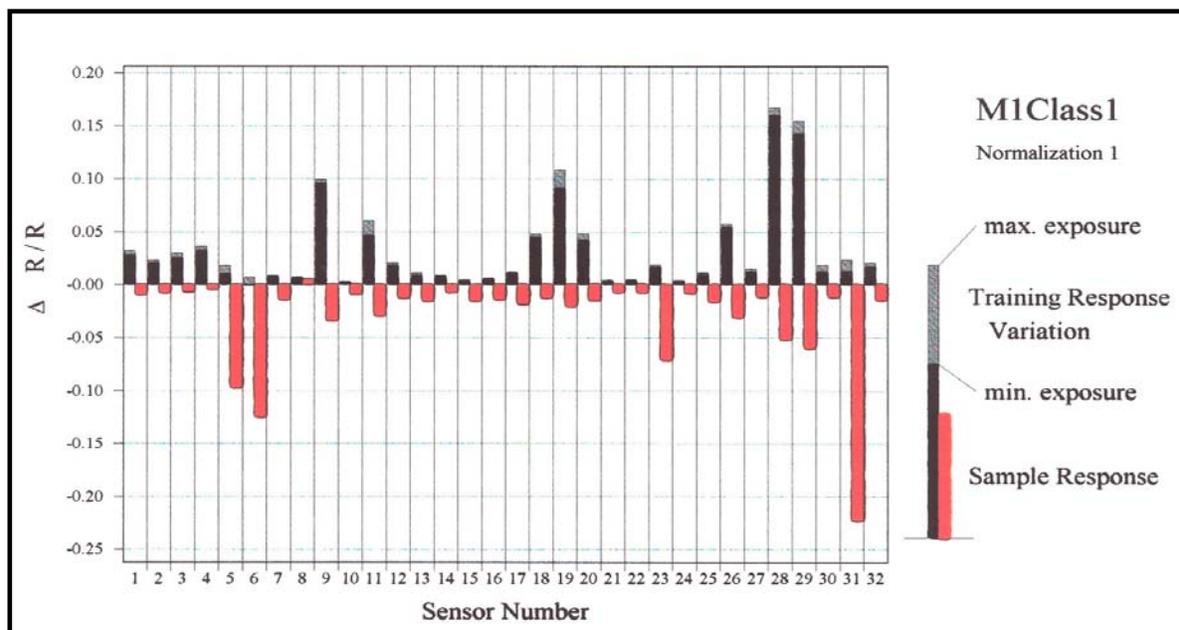


Figure 10. Smellprint for Pine Oil

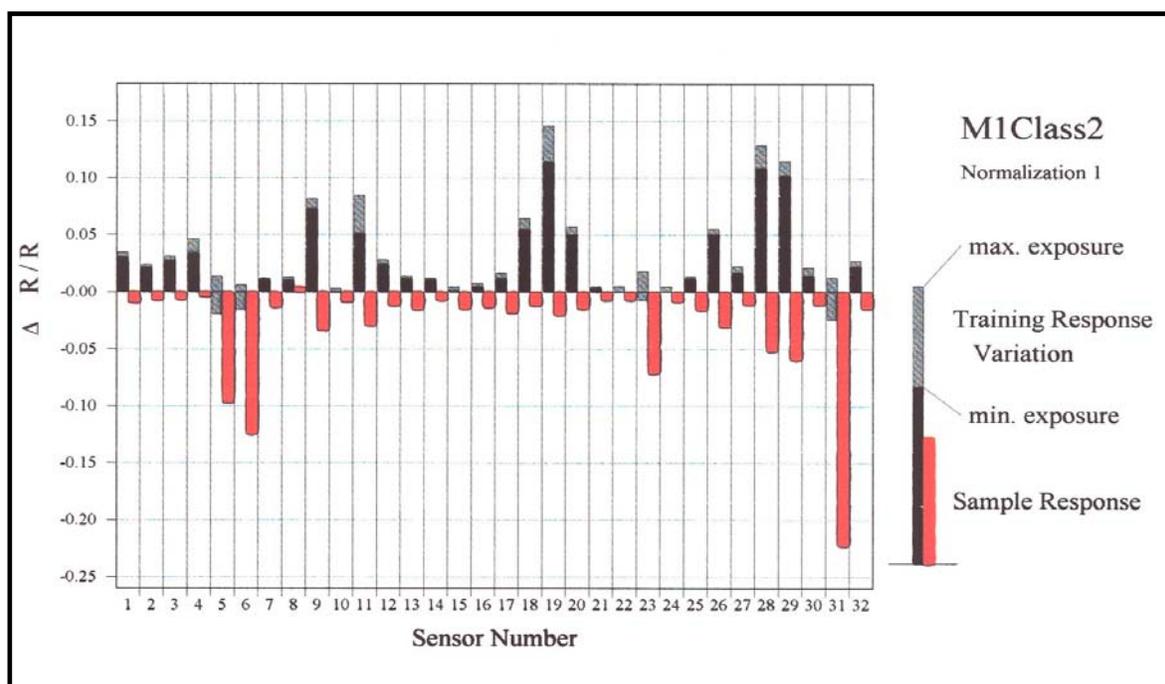


Figure 11. Smellprint for Orange Oil

Note that there is not much observable difference in the smellprints. However, the PCA Plot and the Canonical Plot (Figures 12 and 13) show how the software allows discrimination among pine oil (red training set), orange oil (blue training set), and almond oil (magenta sample).

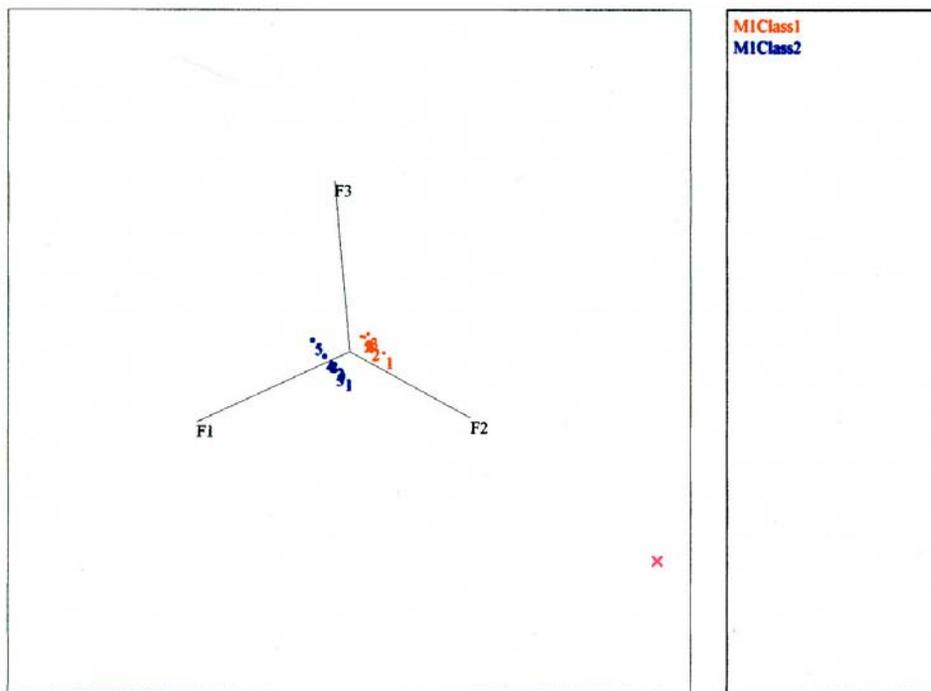


Figure 12. PCA Plot for Pine and Orange Oil Training Sets and Almond Oil Sample

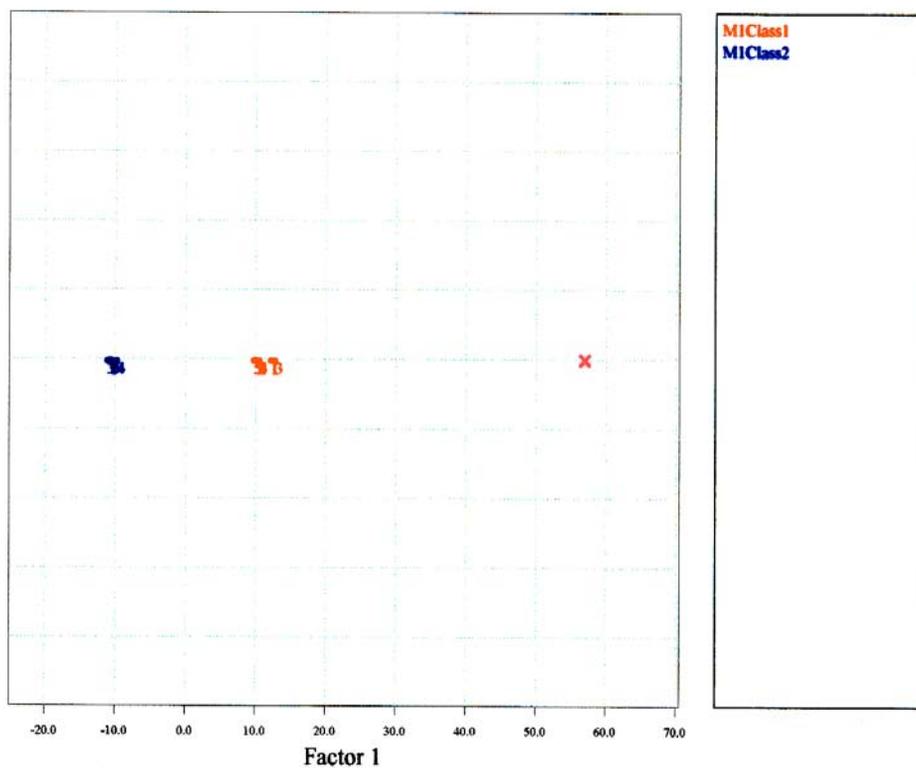


Figure 13. Canonical Plot for Pine and Orange Oil Training Sets and Almond Oil Sample

The PCA and Canonical plots show clear differences among all the marker compounds. Using the built-in ‘Identify’ algorithm, the instrument could readily distinguish among the five marker compounds, (almond, citronella, lemon, orange, and pine oils), tested in this portion of the project. The performance of the ‘Identify’ algorithm consistently identified marker compounds with a 5-star confidence.

It was thus concluded that the output of the Cyranose 320™, when correctly analyzed, could be used to distinguish among the odors of almond, citronella, lemon, orange, and pine oils. The ‘Identify’ algorithm reliably identified all of these compounds.

#### 4. INSTRUMENT RESPONSE TO MARKED ROCK SAMPLES (SINGLE MARKER COMPOUNDS)

Sample rocks for these tests were selected from a bag of aquarium gravel, with particle size of approximately 1 centimeter. This gravel appeared to be mostly sandstone and limestone. Sample rocks for testing were chosen to be close to the same size. Using the methods developed, each rock was dipped in the selected marker compound. Six rocks were marked with each compound, and placed in dropper-top bottles. Training sets were developed for each compound by drawing 10 odor samples of each. It was at this point that the use of pine oil was discontinued, and cinnamon oil was used instead.

Figure 14 shows a typical smellprint, for citronella on rock samples. Note that the training response variation (the gray portion of the bar) for each of the sensor elements is quite small.

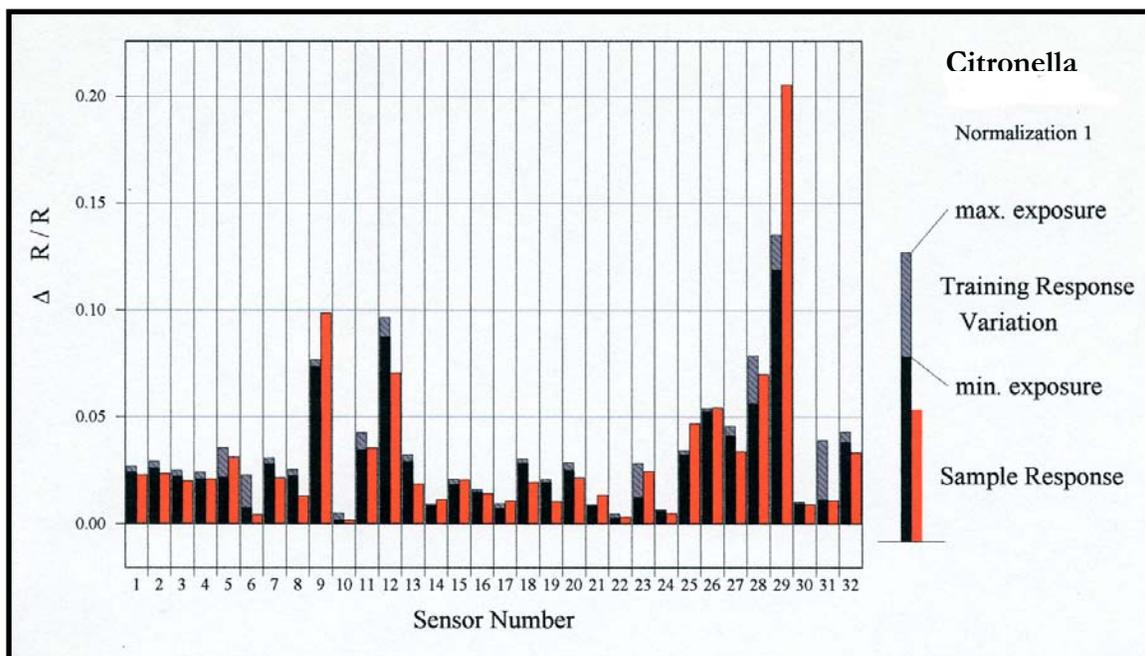


Figure 14. Smellprint for Rock Samples Marked with Citronella

The pattern analyses of the smellprints of marked rocks are presented above (Figures 4-6), as examples of how the Cyranose 320™ software operates. The Mean Distance Diagram (Figure 4) shows the difference in distances for cinnamon, citronella, and almond oil; however, the distances for lemon and orange oil are close to the same, showing that lemon and orange oil cannot reliably be distinguished using the Mean Distance. The PCA and Canonical plots (Figures 5 and 6, respectively) show clear differences among all the marker compounds.

It was thus concluded that the output of the Cyranose 320™ could be used to distinguish among the odors of almond, cinnamon, citronella, lemon, and orange oils. Again in this series of tests, the ‘Identify’ algorithm worked reliably, identifying the marker compounds on the rock samples.

#### 5. INSTRUMENT RESPONSE TO MIXTURES OF MARKED ROCK SAMPLES (VARIOUS MARKER COMPOUNDS)

The same gravel was used in this test series as in the previous set. Groups of eight rocks were dipped in one of the marker compounds and placed in a dipper-top bottle. Training sets were developed for each compound by drawing 10 odor samples of each.

Figures 14 and 15 show the pattern analyses for one of these tests, which used mixtures of rocks marked with cinnamon and citronella oils as shown in Table 3.

Table 4. Sample Makeup for Cinnamon/Citronella Marked Rock Test

Sample Number	Canonical Plot Color	Number of Rocks	Marked with Cinnamon	Marked with Citronella
1	Red	8	8	0
2	Magenta	8	6	2
3	Blue	8	4	4
4	Green	8	2	6
5	Black	8	1	7
6	Orange	8	0	8

The Mean Distance plot (Figure 15) shows the difference in sensor response to the mixtures of marked rocks. Remember that the Mean Distance can be thought of as the distance to the origin in the 32-dimensional plot of the training sets, so that Mean Distance does not necessarily correspond to the fraction of a given marker compound on the marked rocks. This is clear from the Canonical Plot, shown on the next page in Figure 16.

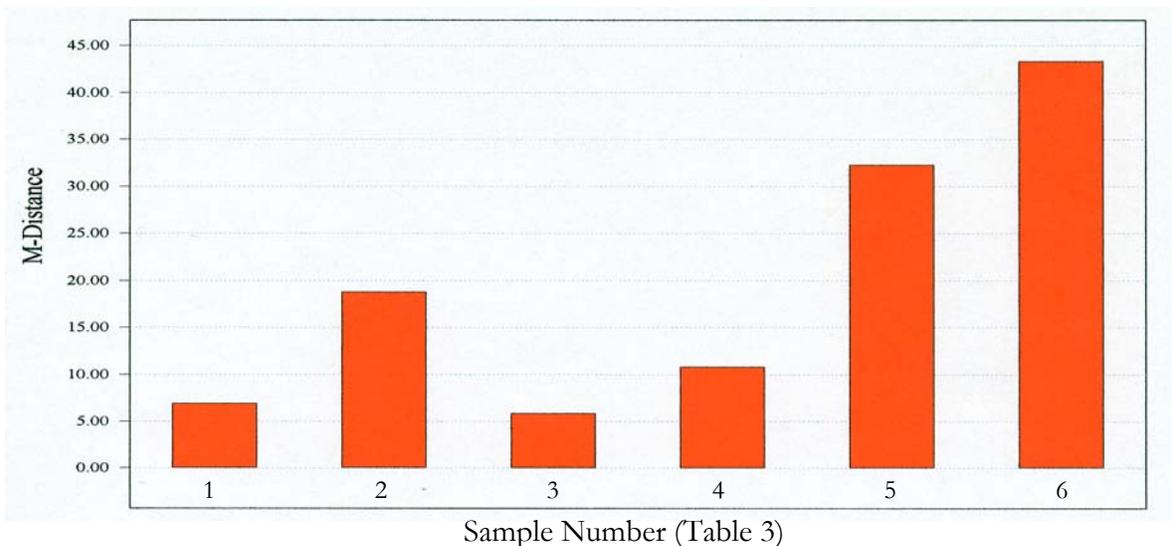


Figure 15. Mean Distance Plot for Mixtures of Marked Rocks

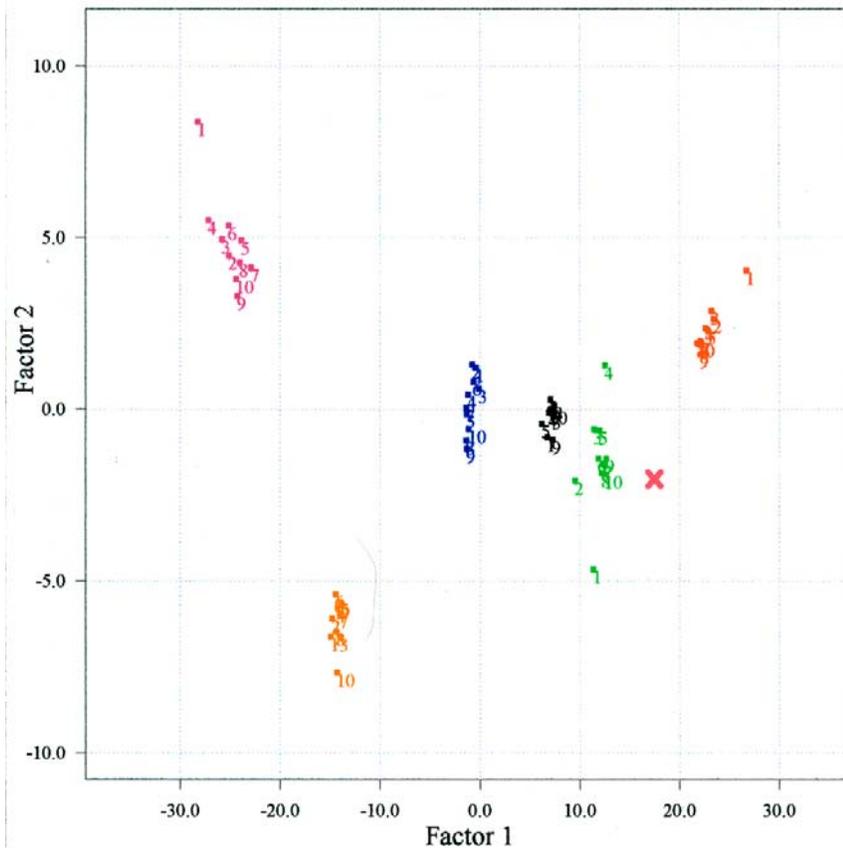


Figure 16. Canonical Plot for Mixtures of Marked Rocks

The 'Identify' algorithm did not reliably distinguish among mixtures of marked rocks. The performance of the 'Identify' algorithm could probably be improved by further refinement of the sampling procedure.

It was thus concluded that the output of the Cyranose 320™ could be used to distinguish among the mixtures of rocks marked with odors of almond, cinnamon, citronella, lemon, and orange oils. The 'Identify' algorithm did not function reliably for this application.

## 6. EFFECT OF MARKER COMPOUNDS ON THE FLOTATION RESPONSE OF ROCK SAMPLES

It is well known that froth flotation is based on surface chemistry effects on the mineral compounds involved, and that aromatic compounds can be used to alter mineral surfaces. Further, aromatic oils can also be used to modify the surface tension of water, so that bubbles and froths form more readily. Thus it was thought possible that the treatment of rocks with aromatic oils, for detection by an odor sensor, might affect the response of those rocks to flotation. This would obviously be a serious drawback to using such odorants as tracers in mining operations.

*Sample Preparation:* To assess the effects of the presence of marker compounds on flotation response, materials were prepared for standard, laboratory flotation tests. Samples of mineralogically pure quartz and galena were ordered from a science supply house. Approximately 2,500 grams of quartz and 250 grams of galena were crushed by hand to minus

1.5 cm. The crushed minerals were separated into three lots each, and prepared as shown in Table 5 below.

Table 5. Sample Lots Prepared for Flotation Testing

Lot	Weight, grams	Treatment
1-Galena	60	None
2-Galena	60	Dipped in Citronella Oil
3-Galena	60	Dipped in Cinnamon Oil
1-Quartz	250	None
2-Quartz	250	Dipped in Citronella Oil
3-Quartz	250	Dipped in Cinnamon Oil

The samples were delivered to Dawson Metallurgical Laboratories in Salt Lake City for flotation testing. Each sample was lightly pulverized to approximately minus 10 mesh in a ring-and-puck pulverizer with the ring removed. Pilot grinding tests on 200 grams of quartz were conducted in our rod mill to determine the time required to produce a 106-micron,  $P_{80}$  product size.

The back-calculated head assays for the laboratory flotation tests on mixtures containing 10% by weight galena varied from 7.58 to 8.50% Pb. The mean back-calculated head assay for the nine (9) tests was 8.03% Pb.

*Flotation Tests:* Twenty grams of galena were mixed with 200 grams of quartz and the mixture was ground in a rod mill, at 42% solids, to approximately 80% passing 106 microns. The three mixtures described in Table 5 were used in triplicate tests for a total of nine tests.

The ground sample was placed in a 500-gram, Agitair, laboratory flotation machine and adjusted to approximately 18% solids. Cytec 3418A aerophine lead collector was added to the pulp at a dosage of 5 grams per metric ton of solids and the pulp was conditioned for 5 minutes. The air was subsequently introduced into the flotation machine and a rougher froth product was collected for four 4 minutes. MIBC frother was added to the tests on the control sample that contained no oil.

*Flotation Test Results:* Results of triplicate tests on each mixture are summarized below and illustrated in Figure 17, on the following page.

Table 6. Results of Flotation Tests on Samples Marked with Odorants

<b>P-2843: Mike Nelson PbS Flotation Test Summary</b>													
Dup. Test #	<b>Standard Quartz + Standard Galena</b>				<b>Cinamon Quartz + Cinamon Galena</b>				<b>Citronella Quartz + Citronella Galena</b>				
	Tails Assay %Pb	Con Assay %Pb	Calc. Head %Pb	% Pb Recov.	Tails Assay %Pb	Con Assay %Pb	Calc. Head %Pb	% Pb Recov.	Tails Assay %Pb	Con Assay %Pb	Calc. Head %Pb	% Pb Recov.	
1	0.279	80.3	8.26	97.0	0.147	75.3	7.99	98.4	0.427	69.8	7.81	95.1	
2	0.471	82.6	7.80	94.5	0.163	77.6	7.81	98.1	0.387	71.9	8.50	96.0	
3	0.534	83.6	7.58	93.5	0.178	79.1	8.30	98.1	0.421	79.6	8.19	95.4	
avg.	0.428	82.2	7.88	95.0	0.163	77.3	8.03	98.2	0.412	73.8	8.17	95.5	

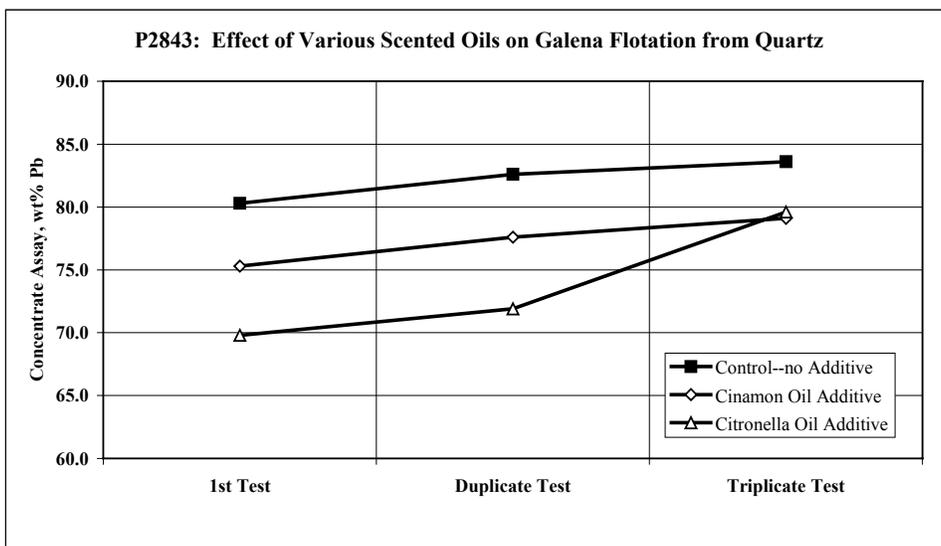
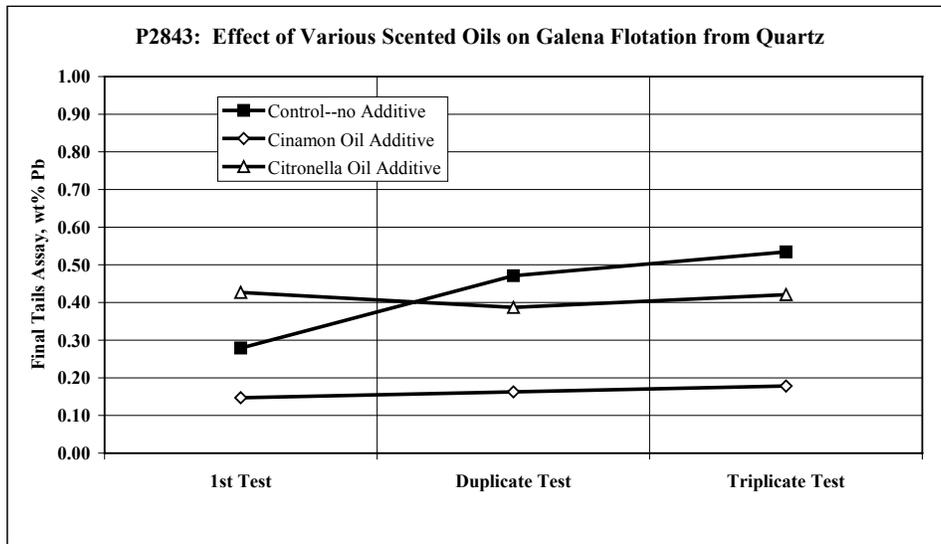
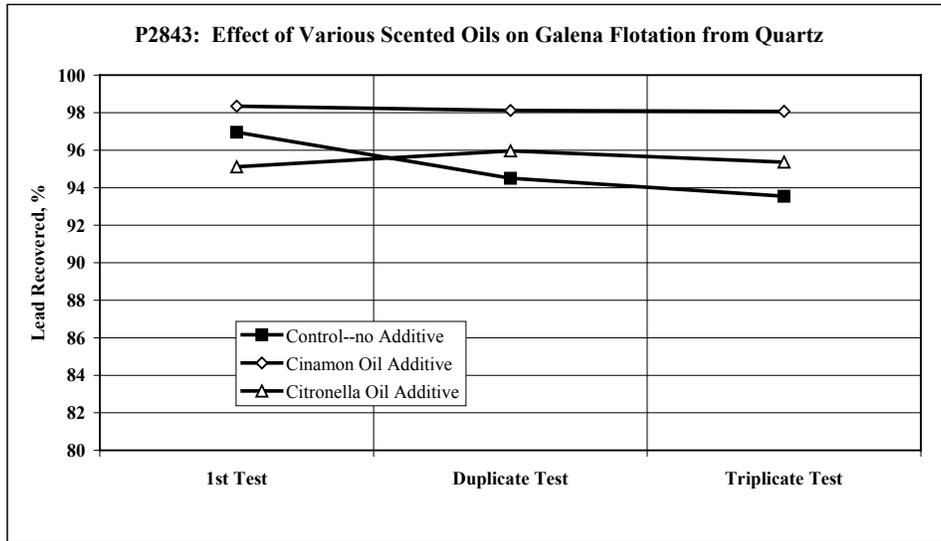


Figure 17. Results of Flotation Tests on Mineral Samples Marked with Odorants

Slightly higher lead recoveries and lower rougher concentrate grades were obtained on mixtures that contained oil, compared with the tests on mixtures with no oil. This may be due to the fact that a more voluminous froth was observed during flotation of the mixtures that contained oil compared to tests on mixtures with no oil. Frother was not required in tests on the oiled samples.

It should be noted that these preliminary tests were performed as rougher tests. The increased amounts of quartz that floated from the oiled samples may be rejected if the rougher concentrates were subjected to cleaning. Additional testing would be required to evaluate this.

## CONCLUSIONS

Investigation of odor sensing systems for mining and milling applications is just beginning, and many questions remain to be answered. After suitable sensors and odorants are identified in lab tests, an exhaustive field testing program will be required. Nonetheless, odor sensing systems show promise for use in tagging various types of ore, to allow tracking of that ore through the mining and milling process. This could improve the integration of mine and mill operations, allowing the knowledge held in the mining and geological computer models to be transferred to the mill control system. Ore response to many mineral processing operations could be improved by having a more detailed knowledge of the nature of the feed.

The present study was a preliminary examination of the potential for the use of odor sensors in distinguishing among mixtures of rock marked with various odorant compounds. The following specific conclusions were reached:

1. The longevity of marker compounds on rock samples (volatile oils) depends on oil dilution, application method, and rock type.
2. In application, markers will have to be carefully tested for use.
3. Application with an atomizing sprayer is not a good method for applying volatile oils to rock samples.
4. Dipping is a good method for applying volatile oils to rock samples. However, it will likely be unsuitable for large-scale application.
5. Further work is required to develop a good method for large-scale application.
6. The Cyranose 320™ can easily distinguish among pine, orange, almond, lemon, citronella, and cinnamon oils. The different samples could be distinguished with a PCA Plot, a Canonical Plot, or using the instrument's 'Identify' algorithm.
7. Because of its strong odor, pine oil is difficult to use with the Cyranose 320™, requiring long purge times after sampling. It is not recommended for future work.
8. The Cyranose 320™ has a good response to fresh cinnamon oil. However, cinnamon oil forms a crust when exposed to air, which decreases the response of the Cyranose 320™.
9. The Cyranose 320™ can also distinguish among rocks that have been marked with various oils and then mixed in various proportions. This identification must be done by examination of a PCA Plot or a Canonical Plot. In the 'Identify' mode, the Cyranose 320™ cannot identify the composition of mixed rock samples.
10. Further work is required to determine proper instrument configuration for identification of mixed rock samples.
11. Treatment with odorant compounds (citronella and cinnamon oils) had the following effects on flotation response in a simple quartz/galena mixture:
  - a. No frother addition was required – the citronella and cinnamon oils created a very strong froth.

- b. Slightly higher lead recoveries were obtained on mixtures that had been treated with the oils.
  - c. Lower rougher concentrate grades were obtained on mixtures that had been treated with the oils.
12. The effects of odorant treatment on flotation response are unlikely to have a significant detrimental effect on the processing of ores marked with odorant compounds.

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