IEEE P2520 Working Group Meeting #7 Minutes
11 November 2019 / 10:00 AM – 11:30 AM (EDT)
Zoom Teleconference (https://ncsu.zoom.us/j/945473904).

Approved: 6/22/2020

Members Present: Krishna Persaud, Troy Nagle, Susan Schiffman, Hua-Yao Li, Susana Palma, Peter Hesketh, Santigo Marco, Radislav Potyrailo , James Covington, Rachel Sunghee Lee (10 voting)

Members Absent: Luis Hoffman, Hugo Gamboa, Howard Choe, Omer Oralkan, Yogesh Gianchandani, Jan Mitrovics, Mike McGinley, Ehsan Danesh, John Saffell (3 voting)

Staff Absent: Vanessa Lalitte, IEEE-USA

1. **Call to Order – WG Chair, Schiffman**
   The Agenda was displayed to the attending WG members at 10:00 AM EDT. WG Chair Susan Schiffman called the meeting to order at 10:03 AM. She welcomed the participants to the 7th meeting of the Working Group to develop an IEEE Standard for Testing Machine Olfaction Devices and Systems. An announcement was made about recording the session for Minutes-preparation purposes. The file will be destroyed after the Minutes have been approved. She announced that Prof. James Covington will deliver (on Dec. 9) the second presentation in our new IEEE P2520 Seminars on Fundamentals of Odor Monitoring and Analysis.
2. **Identification of Participants & Declaration of Affiliation – WG Secretary, Nagle**
   At each meeting, each new member is asked to enter his/her name, affiliation, and email address into the Chat window.
3. **Approval of Agenda – Schiffman**
   The Agenda displayed at the opening of the meeting was adopted without objection.
4. **IEEE Patent Policy – Schiffman**
   The WG Chair briefly reviewed the IEEE-SA Patent and Copyright policies. This item is required for every WG meeting.
5. **Today's Discussion**
   a. What kind of standard should be our first?

      Susan started the discussion. The consensus from our last WG meeting was that we focus on a point-source for our first standard.

      The WG continued the discussion on sampling from the last WG meeting. The first topic was odor sampling using a Tedlar bag. We need a standard that defines the way to fill the bag and then to extract the odorous gas from the bag and deliver it to the sensor/sensor-array. If the bag contains the sample, we can put pressure on the bag to deliver the sample to the sensing chamber. But this makes controlling the pressure at the sensor measurement point very difficult. Another method is to pull the sample from the bag using a mass-flow controller to create a controlled-flow negative pressure at the sensors. This arrangement might be a one-off strategy for sensor calibration. Flow control over the sensors is important to maintain constant temperature during the measurement period. Some sensor technologies have active temperature control. Another technique is the pass the odor sample from a Tedlar bag over a SPME, and then use the SPME to inject the sample into a GC/MS. A third method is to collect the sample directly onto a SMPE and then inject directly into an e-nose sensor array.

      The size of a Tedlar bag varies between applications. One-, two-, and six-liter bags are examples. Flow rates from the bags can be tens of milliliters. During a human sniff, flow rates can approach 20 liters per minute. We might avoid this large difference in flow rates by defining instead the desirable concentration of odorants over the sensors during the measurement period. The volume of the chamber housing the sensor array can vary widely depending on the sensor technology being employed. Flow rates can be set to achieve specific temperature, pressure, and chemical concentration profiles. These profiles can be static or dynamic. The pressure can be negative or positive.

During calibration, a large volume of odorant can be used to drive the sensor chamber to a standard concentration, temperature, and pressure. A measurement device (PID, GC/MS, optical) on the exhaust could be used to validate the sensor readings. Our chosen application will control the range of concentrations being monitored. The concentration at the inlet is the parameter of importance.

Landfill applications employ Suma canisters. Negative pressure is used to fill the canisters in the field. Exponential dilution in used to measure the odor level. One inserts a small capillary into the canister and, under negative pressure, slowly bleeds out the sample. Another method inserts nitrogen to pressurize the Suma canister. The diluted sample is then delivered from the pressured canister to the sensor array. Tedlar bags are said to degrade a collected sample in 24 to 36 hours. On the other hand, Suma canisters hold a sample for 30 or more days with minimal degradation. This leads us to the conclusion that we need to define, for a specific application, the materials that can be in contact with the sample from the time of collection to the time at which the sample is presented to the e-nose sensor array. There are many different options for materials that can contact a sample during its delivery from the point source to the sensor array. Our standard could make materials suggestions for the specific application we choose.

To further focus our discussion on sampling, suggestions for two applications were made. First, the collection of an environmental sample from an interesting site for which an on-site monitor is not practical. The second could be medical/biomedical for which the sample comes from a human. The human sample could be a breath exemplar.

What are the ways a sample is altered as it passes from the source point to the sensor array?

1) Reaction in the wall/tube surfaces (materials and impurities)
2) Non-specific absorption on surfaces
3) Permeation though the wall/tube materials

E-noses are not good for quantitative measurements. So, if we focus on qualitative measurements, do we need to accurately preserve the point-source concentrations of odorants as they transition from the inlet to the sensor chamber? For environmental regulators, the number of odor units is important, as is the perception of offensiveness. Thus, the e-nose needs to mimic a human dynamic olfactometry response. Odor units are very difficult to specify in terms of a mixture of chemical compounds. A typical environmental has hundreds of compounds, with a dozen or more being dominant. Sulfur, ammonia, amines, and the like, present in various concentrations from site to site in a specific application. For example, H2S levels do not predict the offensiveness level some applications. So, for some of our applications, the reference/calibration instrument will be a human (or human panel).

Quantitative or qualitative? Can we decide which direction to take for our first standard? A general consensus was reached that our first focus should be qualitative.

Continuing the sampling methods discussion, Flex foil bags (PET/NY/AL/CPE) and Nalophan bags (PET) were examined. The foil bags are better than Tedlar because we lose fewer molecule of interest (less permeation). Thus, an odor sample is preserved for longer periods of time. But we still lose some molecules of interest due to non-specific adsorption to the metalized surface. Nalophan is a plastic that is often used for food packaging. It is quite inert, but small molecules like ammonia and H2S escape quickly. In our standard, we can explain the advantages and disadvantages for each of these odor sample transport approaches.

Continuing with glass vials: Glass vials are expensive but solve many of the problems with flexible bags. The only loss of molecules of interest is through adsorption on the glass walls. The glass vials are filled under negative pressure from a small pump. The volume of air to be captured is specified (by flow rate over a given time period). The vials may contain a sorbent (like Tenax, a SPME fiber, or cloth swatch) to trap the odorants of interest. The vials are then thermally desorbed to pulse the odorants into an e-nose or analytical instrument.

Capturing and transporting odor samples using bags, vials, and/or sorbent traps is a complicated process. All the variables would need to be defined and controlled in a one-shot calibration standard. Variables in the tubing, bags, vials, flow rates, etc., will make the standard difficult to write and to follow. The UK company Marks, Inc., markets these devices as standards for GC/MS systems that use their expensive thermal desorbers.

If an e-nose system meeting our standard takes odor samples in parallel with a panel of human subjects, and if that e-nose system always transports and alters the concentration profile of those samples in a standard prescribed way, then we should be able to train an e-nose through signal processing methods

to rate the odor sample and match the ratings of the human panel (for example on three 9-point scales for odor intensity, irritation, and unpleasantness).

One final question: How can we deal with humidity? One approach would be to specify that the e-nose meeting our standard should operate in specific humidity environments (say 50%). Humidity can interact with the molecules on interest in the odor sample. Water can form a shell around the molecule, and this changes its chemical species. For some sensor surfaces, there is a competition between water and the molecules of interest, and some of those sensor surfaces are hydrophilic and have more affinity to water that to the molecule of interest. For those sensor types, one can select a sorbent to preferentially capture the molecules of interest and reject water. Hydrophobic SPME fibers can improve the performance in such systems. But even though water is rejected, the concentration profile of the molecules of interest will still be altered.

Krishna will contact Ann-Claude Romain about joining our working Group. She has vast experience in environment odor sampling.

This discussion will continue at our next meeting.

b. Future Seminar Topics:

Chemical compounds of interest. Still no response from Samantha Henningsen, ALS Environmental, in California frequently who gives presentations about chemical compounds of interest to specific industries. John Saffell is contacting her.

c. Suggestions for soliciting new members:

This topic will be carried over to the next meeting.

d. Collaboration with other Standards Working Groups:

This topic will be carried over to the next meeting.

6. Approval of Minutes

At this point in the meeting, Troy indicated that a quorum was present. Voting members are those who have attended two of the last four meetings. Susan asked that the Minutes of our October 7 meeting be approved as distributed. Those Minutes were approved without objection.

7. Topics for Future Meetings - Schiffman

- **a.** Sensors and signal conversion: This item will continue on our future topics list. Members of our group will coordinate with ASTM and other groups.
- **b.** Signal processing best practices: This item will continue on our future topics list. Prof. Ricardo Gutierrez-Osuna has been invited to present a seminar on this topic.
- **c.** The enose market: This item will continue on our future topics list. Can we find an enose market expert to help us rate example use-case clusters.
- **d.** Best practices for enose testing: This item will continue on our future topics list.

8. New Business

There was no New Business.

9. Introduction of New Working Group Members

There were no new members.

10. Review of Action Items - Nagle

No outstanding action items were noted.

11. Future Meetings – Nagle

The next meeting was announced to take place at 10:45 AM EST on Monday, December 9.

12. Adjourn

With no other business being brought before the body, Susan thanked the WG members for their participation and adjourned the meeting at 11:12 AM EDT.

H. Troy Nagle
WG Secretary
11/30/2019