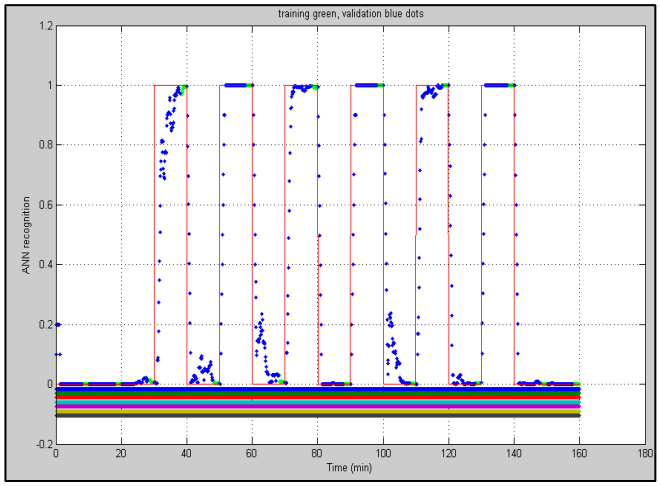
Detection of Hazardous Chemicals Using Temperature-Programmed Sensing in Solid-State Gas Microsensor Arrays

The threat of chemical weapons or toxic industrial chemicals (TICs) used in terrorist acts against civilian populations has increased the need for sensitive, reliable chemical detector technology. Reliable sensors have the potential to save human lives by quickly alerting people of imminent dangers from deliberate or accidental releases of harmful compounds. However, the challenges of recognizing low concentrations of harmful TICs and chemical warfare simulants (CWAs) among common benign "smells" with chemical sensors are formidable. They must be highly sensitive, accurate, robust, small, and low-cost. These requirements also place high demands on the data processing and recognition routines used for interpreting data. NIST work in this area is supported by the Departments of Defense (DoD) and Homeland Security Advanced Research Projects Agency (HSARPA).

D.C. Meier, J.K. Evju, K.D. Benkstein, C.B. Montgomery, S. Semancik, and Z. Boger (Div. 836)

The primary goal of this microsensor project is to develop reliable, low-cost measurement technology and signal processing algorithms for rapid detection of toxic industrial chemicals (TICs), chemical warfare agents (CWAs), and chemical warfare simulants (CWSs) for military and homeland security needs. Not only must the sensors be highly sensitive to provide early detection of toxic compounds at sub-lethal concentrations, but the devices must also have low "false positive" recognition rates, even in complicated air backgrounds. In addition, sensors need to be robust, small, and low-cost, so that widespread deployment is feasible. We have demonstrated that sensing film conductance data collected from chemical microsensor arrays and analyzed with artificial neural networks (ANN) can be used to identify a number of hazardous analytes over a wide range of concentrations and in the presence of a variety of interfering species. A unique aspect of our microsensors is that they can be operated in a temperature-programmed sensing (TPS) mode, which greatly increases the density of data and allows sensitive, reliable detection of analytes in complex backgrounds. The measurements were performed using four-element sensor arrays featuring SnO_2 and TiO_2 sensing films. The sensors were operated in the TPS mode, wherein each sensor sampled 29 temperatures in less than 7 seconds. Five TIC analytes (acrylonitrile, hydrogen cyanide, arsine, methyl isocyanate, and parathion) and one CWS (dimethyl methylphosphonate) were measured at their IDLH (immediate danger to life and health) and their PEL (permissible exposure limit) concentrations in air at 50% relative humidity and 0 °C, 25 °C, and 40 °C, and at 90% relative humidity and 25 °C. Furthermore, each analyte was measured at IDLH in background air including 0.1%, 1.0%, and 2.0% relative saturation of a common potential interference, such as window cleaner vapors, diesel fuel vapors, or bleach vapors. After over 500 systematic presentations of analyte in over 20 different detection conditions (such as the hydrogen cyanide detection data shown in the graph.), ANN models trained on the data yielded a success rate of 97.4% for correct analyte identification. Most of the identifications occurred within one minute of analyte presentation.

The axes on the graph are ANN Recognition vs Time, and show ANN-processed sensing data of hydrogen cyanide detection at 0 °C and 50% relative humidity. Analyte presence is indicated by the red trace (0=off, 1=on). Blue points denote the sensor signal's derived probability of analyte presence, as assigned by the trained ANN. The best sensor performance is found where the red trace and blue detection probabilities coincide.



The NIST team has shown that their temperature-controlled microscale detection system can, using ANN models, rapidly and reliably identify relevant concentrations of hazards under a variety of challenging detection conditions.

We have honed our microsensors' capabilities by implementing improved TPS modes and data processing schemes. We have demonstrated recognition of TICs and CWSs in a variety of atmospheric conditions with several interfering vapors at TIC/CWS concentrations that would be useful for triggering alarms to save lives. Through a continuing research program supported by HSARPA, further experiments will be performed that will expand the analyte suite to include additional interferences and toxic chemicals, including some chemical warfare agents.

The continuing NIST research is aimed at improving the performance of sensing films, expanding the variety of sensing films, developing new detection electronics, and examining long-term sensor stability.

Further experiments will be conducted to enhance sensor signals and lower detection limits. In continuing research sponsored by the DoD, sensitivity toward new analytes and lower concentrations will be evaluated.

	Analyte Presentations	*Correct Recognition*	*False Readings*
No Analyte	**585**	**574**	**14**
Acrylonitrile	**84**	**84**	**0**
Arsine	**84**	**80**	**4**
Hydrogen cyanide	**90**	**90**	**0**
Methyl isocyanate	**87**	**79**	**8**
Dimethyl methyl-phosphonate	**101**	**100**	**1**
Parathion	**87**	**85**	**2**

Analyte Presentations (neat, interference, environmental, PEL, and IDLH
Each presentation represents a 10-minute exposure to a background condition or listed analyte
False Readings (negative or positive)