

Wednesday Morning Poster Sessions, November 5, 2003

Homeland Security Topical Conference Room Hall A-C - Session HS-WeP

Poster Session on Science & Technology for Homeland Security

HS-WeP1 Testing Air Jets as Trace Explosive Particle Removers, R.A. Fletcher, J.G. Gillen, E.S. Windsor, G.A. Klouda, National Institute of Standards and Technology

Detection of trace explosive particles is vital to airport security. One of the key steps in detecting explosive particles is the removal of the particles from the surface that they reside. This can be accomplished by swiping the surface or by energetic disruption such as an air jet that will provide enough force to overcome the particle attachment due to van der Waals forces. Our initial experiments are following the experiments of Phares et al. @footnote 1@ and Smedley et al. @footnote 2@ for polystyrene latex spheres on smooth surfaces. We are utilizing fluorescence tagged spheres of various diameters that we can image through an optical fluorescence microscope. Images of fluorescent particles are captured and recorded before an air jet interrogates the sample. Particle removal is seen in live time; the particles that are not removed from the surface are captured in a subsequent image. Image processing allows us to quickly determine the size distribution the particles that have been removed and the ones that remain. By using polydisperse spheres, we can detect the particle release threshold for a given jet condition in terms of particle diameter. A second study is under way to examine the efficiency of removal for actual high explosives from characteristic surfaces like clothing and luggage. A test substrate is mounted in a small vertically oriented laminar flow wind tunnel. The airflow in the tunnel is hepa filtered to remove all particles greater than 0.3 um diameter. An air jet is used to remove particles from the test surface. The size distribution of the removed particles is determined in real time using a particle detector mounted down stream that works on the basis of measuring particle impact energy. Results from both experiments will be presented. @FootnoteText@ @footnote 1@ Phares, D.J. et al. *J. Forensic Sci.* 2000:45;(4) 774-784. 2. Smedley, G.T. et al. *Exper. In Fluids* 1999:26, 324-334.

HS-WeP2 Temperature-Dependent micro-Raman Study of Some High Explosives, E.S. Etz, S.V. Roberson, G. Gillen, National Institute of Standards and Technology

In collaboration with the Transportation Security Agency, NIST has initiated a chemical metrology program that will support the nationwide operational deployment and utilization of trace explosives detection devices. The project critically examines the analytical methodology whereby collections of micrometer-sized high explosives (HE) particles undergo thermal vaporization at the front-end of an ion-mobility spectrometer (IMS), leading to sample detection and identification. We have started to examine this front-end process in detail with a focus on particle transport and HE thermalization-vaporization for the IMS detection system. Of interest here is the temperature behavior and the thermochemistry of the analyte HEs in the temperature regime leading to the melting point, and beyond to the vapor phase. To understand the fragmentation and decomposition of HEs, we are using Raman microspectroscopy to study the particle spectra of relevant HEs heated to their melting temperature, and beyond, on a microscope hot-stage. The spectra are examined for (i) shifts in the peak frequency of Raman bands, (ii) temperature-induced changes in the spectral halfwidth and asymmetry of bands, (iii) changes in the relative intensities of bands and (iv) the presence of decomposition products. The spectra are acquired with a commercial Raman microprobe utilizing 514.5, 532, and 785 nm excitation. Discussed are the results obtained for the explosives TNT (m.p. 80.7 °C), PETN (m.p. 141.3 °C), RDX (m.p. 204 °C), and the plastic binary composites Semtex and C-4. The spectra show considerable changes as the sample is ramped up to the higher temperatures. These changes take on various forms, including substantial peak shifts to lower frequencies, band broadening with lowering of symmetry, and pronounced relative intensity changes. These spectral manifestations are correlated with the structural and compositional changes of these organic samples.

HS-WeP4 Vapor Preconcentration during Explosive Detection: Study of Variables in a NIST Standard Test System, R.M. Verkouteren, National Institute of Standards and Technology

The reliable detection of ever-decreasing levels of explosives constantly challenges the public safety and analytical communities. The total absence

of false negative responses is required while minimizing the rate of false positive responses. While much progress has been achieved, further significant improvements in methods may be enabled by the systematic study of the total measurement process, especially near the limits of detection. Fundamental data from such studies are needed by system designers, instrument manufacturers, and public safety officials in improving the sensitivity and specificity of portal screening systems, while balancing the human dimensions that include ease of use, speed of analysis, detection risk tolerance, personal invasiveness, and profiling decisions. Described will be a NIST standard test system and preliminary flash desorption experiments, designed to investigate variables affecting the efficiency of one segment of explosive detection: the preconcentration stage. The standard test system was built to enable robust monitoring and control over static and dynamic temperature regimes and gas flow, and was designed to allow modifications in the configuration of components. We intend that this system be used to compare reliably the relative performances of materials, configurations, and methods, and to provide a benchmarking reference to enable measured improvements to technology. In the NIST system, a known quantity of sample is introduced onto a metal felt collector, which is then desorbed into a gas stream by resistive heating and monitored by IR thermography. The vapors released are then trapped on a second metal felt collector, the temperature of which is controlled by a thermoelectric cooler. The amount of trapped sample is determined by off-line quantitative methods. Factorial experimental design will be discussed as an effective tool to investigate a large number of variables in a limited number of experimental runs.

HS-WeP5 Chemical-Biological Nanosensors, N.L. Jarvis, Edgewood Chemical Biological Center; *A.W. Snow*, Naval Research Laboratory; *H. Wohltjen*, Microsensor Systems, Inc.; *R.R. Smardzewski*, Edgewood Chemical Biological Center

A new class of nanometer-scale, low power, solid-state devices is being investigated for the detection of hazardous vapors. These chemical vapor sensors are comprised of nanometer-sized gold particles encapsulated by monomolecular layers of alkanethiol surfactant deposited as thin films on interdigitated microelectrodes. These new, alkylthiol-stabilized, gold nanocluster materials are appropriately categorized as metal-insulator-metal ensembles (MIME). When chemical (agent, hazmat) vapors reversibly absorb into these thin MIME films, a large modulation of the electrical conductivity of the film is observed. The measured tunneling current between gold clusters is extremely sensitive to very small amounts of monolayer swelling or dielectric alteration caused by absorption of vapor molecules. For chemical agent simulants, a large dynamic range (5-logs) of sensitivities is observed and extends down to well below sub-ppm vapor concentrations. Tailored selectivities of the sensors are accomplished by incorporation of chemical functionalities at the terminal structure of the alkanethiol surfactant or substitution of the entire alkane structure. Current research efforts are focused on examining the molecular mechanism(s) of conduction and mapping the selectivity and sensitivity of sensor elements. Targeted applications include: low-cost, low-power CB agent sensors, filter residual life indicators and orthogonal detector applications.

HS-WeP6 A Portable GC with a Nanosensor Array Detector, A.W. Snow, Naval Research Laboratory; *H. Wohltjen*, Microsensor Systems, Inc.; *N.L. Jarvis*, U.S. Army Edgewood Chemical and Biological Center

Development of portable rapid-screening analytical methods/instrumentation for the detection of chemicals identified by the Chemical Weapons Convention (CWC) as associated with the production and use of agents is an activity of interest to homeland security as well as to CWC inspection capabilities. The instrument described in this presentation consists of a temperature programmable gas chromatograph combined with a novel detector array based on gold nanocluster chemiresistors. @footnote 1,2@ In this configuration peaks eluting from the GC are "fingerprinted" by the detector array using an on-board pattern recognition computer and an on-board library look-up. The thermoelectrically cooled array detector is very compact, works with any carrier gas, and uses minimal power. The array detector consists of four interdigital microelectrodes coated with different gold nanocluster materials. This device structure is called a "MIME" device (a mnemonic for Metal-Insulator-Metal Ensemble which reflects the electron transport through the organic monolayer shells encapsulating the metal cores of adjacent nanoclusters). Absorption of vapors into the insulating ligand shell surrounding the core produces a change in conductivity that is interpreted as a GC detector signal. The vapor sensitivity, selectivity and response time of each MIME device in the array are dependent on the composition,

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molecular structure and thickness of the ligand shell resulting in the array detector having a unique pattern for a particular analyte or interferent. Numerous candidate materials were evaluated for their effectiveness in the detection of organophosphorus compounds and other chemicals associated with the production of chemical weapons. @FootnoteText@ @footnote 1@ H. Wohltjen and A.W. Snow, "Colloidal Metal-Insulator-Metal Ensemble Chemiresistor Sensor", *Anal. Chem.* 1998, 70, 2856. @footnote 2@ A.W. Snow, H. Wohltjen, N.L. Jarvis, "MIME Chemical Vapor Microsensors", *NRL Review*, 2002, 45.

HS-WeP7 A Rapid Optical Assay for the Detection of Bioterrorism Agents Using Thin-Film Technology, *S.C. Francesconi*, Nova Research, Inc.; *A.M. Churilla*, Naval Research Laboratory

Rapid, field analysis of environmental samples for the presence of biological agents is important in both military and civilian law enforcement and public health applications. Development of assay formats, useful in these settings must be easy to operate and interpret. Additionally, the assay must be highly sensitive yet yield a low rate of false positive results. To this end, we have modified a commercially available, thin-film format to develop an assay capable of detecting multiple biological agents, simultaneously. The current assay is capable of simultaneously detecting ricin, botulinum toxin and anthrax, plus negative and positive controls, simultaneously in less than 30 minutes. The assay is performed on disposable silicon nitride wafers spotted with capture antibody specific to target agents. Samples, mixed with enzyme-coupled detector antibody are exposed to the capture antibody-spotted wafers. Exposure of the wafer to enzyme substrate results in a thin-film. Final detection of agents, therefore, is creation of a change in refractive properties of the wafer spots yielding a visible color change proportional to the amount of antigen originally present in the sample. The assays perform well using a number of sample matrixes including tap water, sea water or oily water. Further, the assay can be expanded to include additional target agent detection capability.

HS-WeP8 Detection of Nucleic Acid Hybridization by Infrared Absorption Spectroscopy, *K. Miyamoto*, *Y. Kimura*, *H. Ishii*, *M. Niwano*, Tohoku University, Japan

Biotechnology and medical diagnostics are currently in need of devices able to quickly and selectively detect biological molecules. DNA chip technology has gained considerable interest because of its importance to disease diagnostics, mutation detection, gene discovery, and so on. DNA chips facilitate the detection of specific DNA fragments (target DNA) by hybridization with a complementary strand (probe DNA). In conventional DNA chips, probe DNA that is immobilized on a solid substrate such as glass, are hybridized with fluorescently-labeled target probes, and the hybridization is measured with a laser scanner. However, if hybridization were in-situ monitored by spectroscopic tools, fluorescent label would be unnecessary. We propose an alternative method of monitoring hybridization of nucleic acids using infrared absorption spectroscopy in the multiple internal reflection geometry (MIR-IRAS). Since hydrogen bonding plays an important role in the hybridization of nucleic acids, we have investigated how hybridization induces spectral changes in the C=O and N-H stretching vibration regions of DNA. Results of ab-initio calculations demonstrate that changes in vibration frequency of the C=O stretching and N-H scissors modes are induced due to the base pairing of Guanine and Cytosine. We show the possibility of high-sensitive detection of DNA hybridization using MIR-IRAS.

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