NIST Program to Support T&E of Trace Explosives Detection

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Trace detection is a primary strategy for thwarting terrorism activities in the United States and abroad. The development of effective reference materials and methods for this purpose relies on fundamental knowledge regarding the size, mass, morphologies and chemical distributions in residues from explosives handling, personal exposure and sampling. The authors are working with the National Institute of Standards and Technology (NIST) Office of Law Enforcement Standards (OLES) and the Department of Homeland Security (DHS) Transportation Security Laboratory to strengthen the chemical metrology system that supports the widespread operational deployment of explosive trace detectors (ETDs).

In this effort, available resources of microanalytical and trace detection instrumentation are being applied to investigate and build fundamental understanding in: (1) sampling of fingerprints and other residues containing trace explosives; (2) inkjet printing of explosive reference and calibration materials; and (3) dynamics of particle release, capture and vapor processes in portal-based ETDs. The following will highlight some NIST activities in these areas.

■ *Sampling of residues*—A methodology was developed to evaluate particle collection efficiency by swipe sampling of trace residues. Collection efficiencies were evaluated for fluorescent micrometer-sized polystyrene latex spheres with respect to particle size and mode of deposition, collection swipe, surface type and swiping force. The particles were placed on polytetrafluoroethylene (PTFE) or muslin surfaces using either a dry deposition technique, or by embedding the particles in an artificial sebum (skin oil) expected to be more representative of real residues. Test surfaces containing particles were prepared under controlled conditions and swiped with a slip/peel tester that allowed for the evaluation of frictional forces. Collection efficiencies were determined by optical imaging and particle counting, with some data shown in *Figure 1*.

Primary conclusions include: (1) major differences in collection efficiency exist for the two different sampling materials; (2) large particles are collected more efficiently than small particles; (3) many particles are detached but not collected; (4) the sebaceous particles are more efficiently collected off surfaces than the dry particles; and (5) applying greater downward force during swiping does not significantly improve collection efficiencies. These facts imply that collection efficiency may be best optimized by improving the design of col-

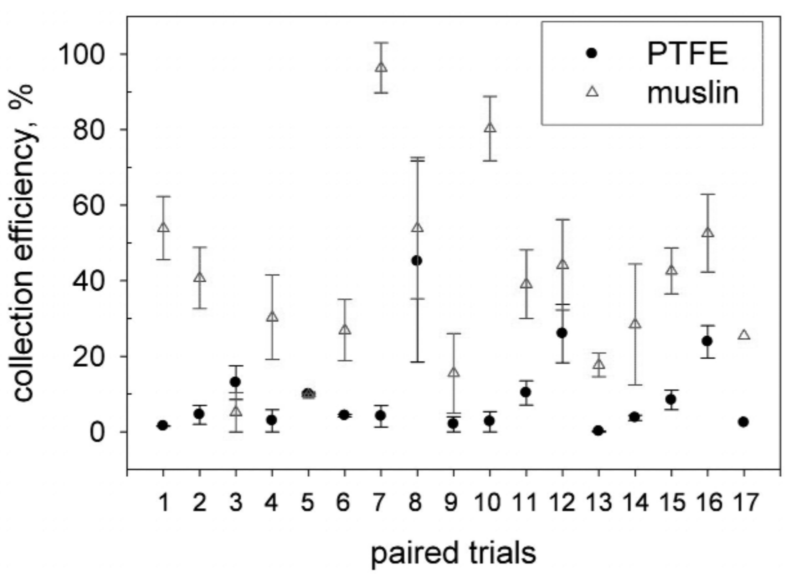

Figure 1. Paired comparison of sphere collection efficiencies for polytetrafluoroethylene (PTFE) versus muslin swipes across 17 different collection conditions (not specified here); (bars represent the standard uncertainties of the means)

lection surfaces in order to maximize the inherent adhesion to the particles, rather than by applying larger forces to detach the particles. These results have impacted sampling and screening protocols used at airports and other points of entry, thereby significantly improving explosives detection.

■ *Inkjet printing of testing and evaluation materials*—Many tens of thousands of ETDs are currently deployed, and the performance of each must be monitored frequently to verify operational readiness. Because vast numbers of reference materials are needed for this purpose, inkjet technology is being adapted as a means to print trace explosives on appropriate media. As exemplified in inkjet printing, piezoelectric nozzles offer precise and accurate control over the microdeposition of materials. Several piezoelectric systems for production of standard materials have been developed at NIST. The following section briefly describes two systems useful for the testing and calibration of trace explosive detectors.

The NIST vapor calibrator uses precise microdroplet injection and evaporation to introduce trace compounds into a calibrated airstream, where vapor signatures may be produced at fg/L to ng/L levels. *Figure 2* shows photoionization detection (PID) data from three pure fluid vapors: ethyl-2-hexanol, which has been linked with canine olfaction of plasticized explosives; isobutanol, a solvent used for preparing trace explosive standards; and methyl salicylate, a chemical warfare agent simulant. By injecting diluted solutions of explosives, concentrations have been generated that are appropriate for the calibration of ETDs, the training of canines, as well as the testing of absorptive materials designed to trap trace substances from air flows. An example is described in the next section.

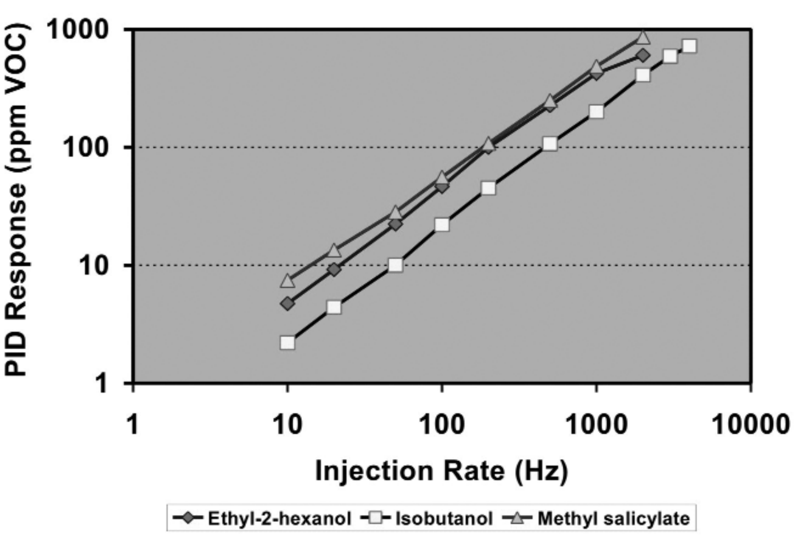

Figure 2. Responses of a photoionization detector (PID) to trace vapors of three volatile organic compounds (VOCs) injected individually at various rates in the NIST vapor calibrator

The Jetlab[1] is a drop-on-demand printing system with an XYZ-stage where substrates may be manipulated precisely under a piezoelectric nozzle. Methods have been developed to deposit trace explosives and other substances in patterns onto a variety of substrates, including sampling swipes, computer diskettes and luggage handles. The printing of bitmapped fingerprints has been demonstrated *(see Figure 3, next page)*, which may be used to simulate residues found on objects at crime scenes for training purposes. The JetLab has been used to prepare reference materials containing trace amounts of composition C4, and have distributed these materials in an ongoing pilot study to intercompare ETDs deployed in varied locations. Results from intercomparisons have been extremely valuable for evaluating and improving the effectiveness of test methods and standards in the field.

NIST is developing "smart" reference swipes that provide feedback to the user. ETDs commonly use thermal desorption to liberate explosive vapors from sampled residues, so ETD reliability is dependent on desorption temperature and the co-location of the heat and deposit on the swipe material. Microdot arrays of thermochromic inks have been printed on these swipes, formulated to change color irreversibly at a specific temperature. After use in an ETD, a user with a low-power microscope may determine the location, temperature achieved and symmetry of the heated area of the swipe. This feedback provides a valuable assessment of sampling effectiveness for protocols involving swipe sampling and ETD measurements.

■ *Testing materials used in portal-based ETDs*—Many ETD technologies use preconcentration to increase detection sensitivity of the analyte. In some portal-based ETDs, particles dislodged by air jets are collected quickly on large filters, which are heated to release vapors

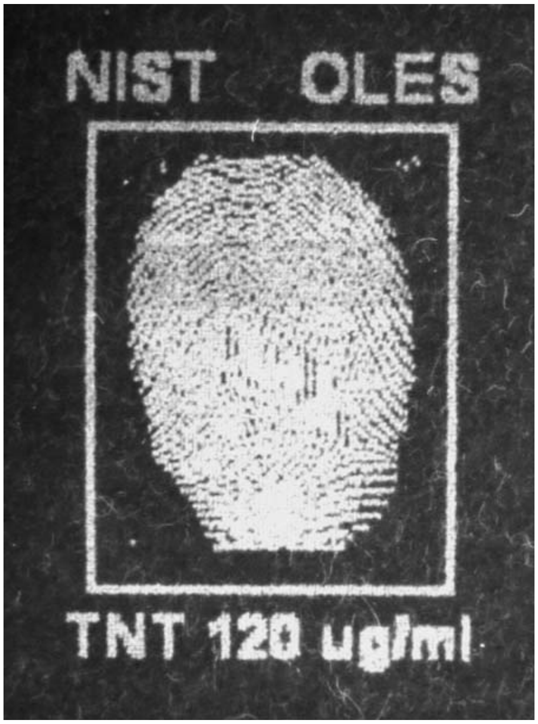

Figure 3. A bitmapped fingerprint, printed using microdroplets containing trinitrotoluene (TNT), artificial sebum and a fluorescent dye

that are subsequently adsorbed on small filters. These small filters, in turn, are heated to release the concentrated vapors for analysis. In an analogous process, some handheld vapor detectors sample a low flow of ambient air for prolonged periods to concentrate the analyte on sorptive materials and traps before analysis. In both cases, the sensitivity of detection depends on the vapor collection efficiency of the sorptive material utilized. Stainless steel filters are frequently used for the purpose of preconcentration, and the authors noted that the surface of this material develops oxide coatings (heat tint) after repeated thermal cycling.

To test the consequences of this surface alteration, stainless steel filter replicates were treated to produce oxide films on the fibers. These filters were installed on a temperature-controlled vapor collection platform and exposed to known trace amounts of cyclotrimethylenetrinitramine (RDX) using the vapor calibrator described previously. ETD measurements of these filters indicated that heat-generated iron/chromium oxide films do not significantly affect RDX vapor collection, while silanated surfaces reduce sorption and wall effects. Absorption data such as these are enabling improvements in designs of next-generation ETDs.

In summary, the goal of the NIST trace explosive metrology program is to develop measurements and standards that support the reliability of trace detection and enable the timely development of effective countermeasures to high-priority explosive threats. NIST has focused on developing fundamental measurements of particles, residues and vapors needed to improve sampling, test and evaluation materials, operational methods and technologies deployed for explosive detection. An experimental approach has been designed to determine particle collection efficiency during swipe sampling, and the authors have obtained results of high impact to agencies responsible for public safety.

Procedures also have been developed to deposit, by piezoelectric inkjet nozzles, known quantities of substances in patterns on a variety of media, useful for the production of large numbers of "smart" reference materials, and in the fabrication of training materials for first responders, security screeners and crime investigators. Another piezoelectric system, a vapor calibrator, has been developed that can deliver concentrations of trace explosives in air across at least six orders of magnitude. This has been applied to the calibration of explosive vapor sniffers and to challenge and evaluate the performance of next-generation materials in ETDs. ❑

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Endnote

[1]Certain commercial equipment, instruments or materials are identified in this article to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.