INKJET METROLOGY AND STANDARDS FOR ION MOBILITY SPECTROMETRY

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Types of Standards

- **Standard materials**
  - chemical, physical, optical, etc
    - certified reference materials (SRM)
    - testbed (RM, IM, TM)

- **Standard data**
  - chemical, physical, optical, etc
    - certified reference data (SRD)
    - interactive (RD, MTD)

- **Calibration services**

- **Documentary standards**
  - performance or design
  - voluntary or regulatory
Trace Contraband Detection Using Ion Mobility Spectrometry

Vapor-based detection

Swipe-based residue detection

Portal-based particle detection

Ion Mobility Spectrometer

Explosives Detection
TNT Standard ALM00632

ISIMS-2006 O‘ahu
Metrological Issues

- Massive deployment of IMS detectors
- Diversity of applied technologies
- Metrics of reliability & comparison of performance
- Consensus for technical improvements
- Prioritization of R&D
- Liability
“Inkjet” Technology for Trace Contraband Metrology

Advantages

• Dispenses small quantities (picoliter-sized droplets, femtograms of analytes)
• High precision delivery
• Dynamic range (6+ orders of magnitude)
• Digital programmability
• Fieldability
Microdroplet Formation

Sheath

Piezo-ceramic

Fluid reservoir → Glass nozzle

Cavity

Voltage (+) applied

Droplet Volume & Velocity Trends

Pulse width (microseconds)

Droplet Velocity (m/s)

N-butyl carbitol at 2000 Hz

Droplet volume (m³)

Pulse amplitude (volts)

N-butyl carbitol at 2000 Hz

Movie

MicroFab Technologies, Inc.

ISIMS-2006 O‘ahu
Piezoelectric Microdispensing

Technical Factors

Fluid factors
• Thermodynamics (viscosity, specific gravity, surface tension, vapor saturation pressure, heat capacity, boiling point), solute concentration, particles

Nozzle factors
• Piezoelectric waveform, cavity & orifice dimensions (droplet size, resonance limits: drop-on-demand), wettability (jet direction), fluid back pressure

Post-injection factors
• Ambient media & flow rate, target surface characteristics (temperature, conformation, roughness)
Outline

• Microdispensing systems

- Arrays
- Particles
- Spheres
- Vapors

• IMS measurements
Quant-Jet
(MicroFab Technologies JetLab III)

- 2-D arrays & patterns
- IMs and TMs
- Verification
  - Optical
  - Microanalysis
  - IMS
  - Classical methods

C4 + fluorescein on PTFE
IMS Intercomparison Reference Material
Thermal Desorption
Analyte Position Factor on Sample Trap

- Aliquots of RDX solution placed on traps within virtual grid
- IMS measurements performed and replicated
- IMS response dependent on grid position
- Asymmetries indicated
Thermochromic Inks

- Formulated for drop-on-demand inkjet printing
- Color change at specified temperature
  - Desorption temperature profile
  - Misuse of printed standard materials
Thermochromic Indicator of Desorption Isotherm

PTFE swipe in Barringer IonScan 400B

Before desorption

After desorption, 200 ºC for 10 seconds
Polymer Microsphere Formation using Emulsion/Solvent Extraction Printing

“Sphere-Jet”

Pharmaceutical delivery technology applied to generation of IMS particle standards

• Non-toxic
• Biodegradable
• Monodisperse
Polylactic-glycolic Acid (PLGA) – TNT Microspheres
PLGA Microspheres containing Butylated Hydroxytoluene (BHT)
Particle-Jet

SEM Images of SEMTEX Particles

~5 μm

1800x

500x
Vapor-Jet

- Trace vapors of explosives, CWA simulants, odor signatures
- Calibration of vapor detectors
- Sets performance targets of next generation vapor detection technologies

movie
Vapor-Jet Performance

Microdroplet diameter = 58.4 micrometers
Air flow = 10 L/m
Photoionization Measurements
Isobutanol

PID response (ppm)

Time Sequence (mm:ss.s)
PID Results for 3 Fluids

- Ethyl-2-hexanol
- Isobutanol
- Methyl salicylate
Thermodynamics of Trace Solute Evaporation

Maximum Evaporative Mass Flux

\[ \Gamma = \frac{(m \cdot p_s)}{(2 \cdot \pi \cdot m \cdot k_B \cdot T)^{1/2}} \]

Mass Injection Rate

\[ \Lambda = C \cdot \nu \cdot I \]

Table II. Coefficients for the Reduced Clausius-Clapeyron Equation (Eq. 2), and Calculated Evaporation and Injection Rates.

<table>
<thead>
<tr>
<th>Compound (^{10})</th>
<th>(\alpha (K^{-1}))</th>
<th>(\beta)</th>
<th>(\Gamma_{130°C} \times \text{Area}^*) (ng/s)</th>
<th>(\Lambda_{\text{max}}) (ng/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RDX</td>
<td>-6473</td>
<td>16.50</td>
<td>2.5</td>
<td>1.0 (^{†})</td>
</tr>
<tr>
<td>PETN</td>
<td>-7243</td>
<td>19.56</td>
<td>42</td>
<td>4.2 (^{‡})</td>
</tr>
<tr>
<td>TNT</td>
<td>-5481</td>
<td>16.37</td>
<td>550</td>
<td>4.2 (^{‡})</td>
</tr>
</tbody>
</table>

*K Droplet impingement area = 2700 \(\mu m^2\)
IMS Calibration Curves

GE Security VaporTracer 2
Single Vapor Mode w/ Preconcentrator
RDX, TNT, PETN
Summary

- Quant-Jet ... Printed Swipe Standards (IMs, TMs)
- Vapor-Jet ... Vapor Standards (Calibrations)
- Sphere-Jet & Particle-Jet... Portal Standards (TMs)