Method Validation for the Analysis of Condom and Sexual Lubricants using Direct Analysis Mass Spectrometry

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Jeffrey Dake – Forensic Chemist
US Army Criminal Investigation Laboratory
Jeffrey.dake@us.army.mil
Disclaimer

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Purpose

- Develop a technique for rapid, easy detection of two common lubricants
  - Polydimethyl siloxane (PDMS)
  - Nonoxynol-9 (N9)
Instrumentation

- Direct sampling
- Disposable media
- Rapid analysis over large mass range
Compounds of Interest

Tuning compound: Polyethylene glycol (PEG): 600 amu avg.

QA/QC compound: Reserpine: 609.281 amu

- PDMS: 200-600 amu
- Nonoxynol polymer: 350-900 amu
- N9: 617.426
FTIR Data

PDMS

NONOXYNOL
Experimental Process

1. Optimize instrumental parameters
2. Establish method of analysis
3. Determine LOD
4. Compare to case samples
5. Implement in analysis scheme
Parameter Matrix

**Orifice Voltage**
- Affects fragmentation:
  - Low E = Molecular ion
  - High E = Fragments
- Values: 15, 35 and 65 V

**Orifice Temperature**
- Affects types of compounds sampled:
  - Low T = Low Mass
  - High T = High Mass
- Values: 275, 300, 325, 350, 400 and 450 °C

**Detector Voltage**: Controls overall signal
- Low V = Low signal (decreased noise)
- High V = High signal (increased noise)
- Values: 2200 and 2400 V
Mass Spectral Data

Abundance (Normalized)

Mass

Nonoxynol

PDMS

355.070

371.102

429.089

445.121

N5

N6

N7

N8

N9

N10

N11

N12

N13

N14

N15
Choosing of optimal parameters - PEG

Orifice Voltage - 15V

Orifice Voltage - 35V

4th PEG peak out of spec
Choosing parameters
Nonoxynol-9 (2200V)
Limit of Detection

- Serial dilutions of PDMS and N9
- Tested each dilution in triplicate
  - 5 mmu tolerance
- 4 Examiners
- Also tested on FTIR
- Dipping vs. Syringe
Limit of Detection

Sample: PDMS by FTIR   PDMS by DART   N9 by FTIR   N9 by DART

LOD: ~50 ng   ~30 ng   ~500 ng   ~10 ng
Casework Shadowing

- 86 samples analyzed:
  - Case samples
  - Lubricant standards
  - Simulated case samples

- Two examiners performed work:
  - Blind testing
  - Analysis by current procedures: FTIR
  - Compare to results on AccuTOF-DART
Typical Sample
Comparison

PDMS

- 80% agreement
  - 28/86 Positive on Both
  - 41/86 Negative on Both

- ~20% disagreement
  - 10/86 Positive by DART ≠ FTIR
  - 7/86 Positive by FTIR ≠ DART

N9

- 100% agreement
  - 7/86 Positive on Both
  - 79/86 Negative on Both
Results

- **AccuTOF-DART effective for N9**
  - Eliminates additional extractions
  - Improves efficiency and accuracy

- **Inconsistencies with PDMS**
  - Strong agreement (>80%)
  - Possible interference from other compounds
  - Sample introduction at LOD
Continued Work

- Improve detection of PDMS
  - Increased sample amounts
  - Alternate parameters to decrease interference
- Analysis of lotions/additives
  - Numerous compounds characterized
  - Differentiation of lotion products
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QUESTIONS??