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

Trace Evidence Symposium
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Disclaimer

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Purpose

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

Instrumentation

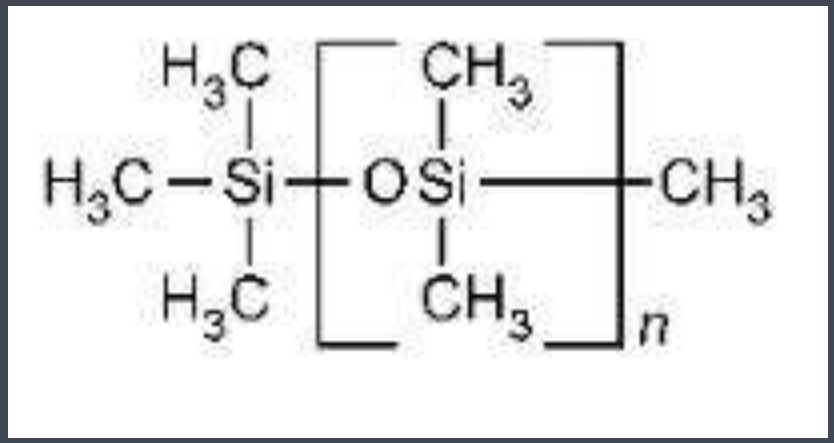
- Direct sampling
- Disposable media
- Rapid analyis over large mass range

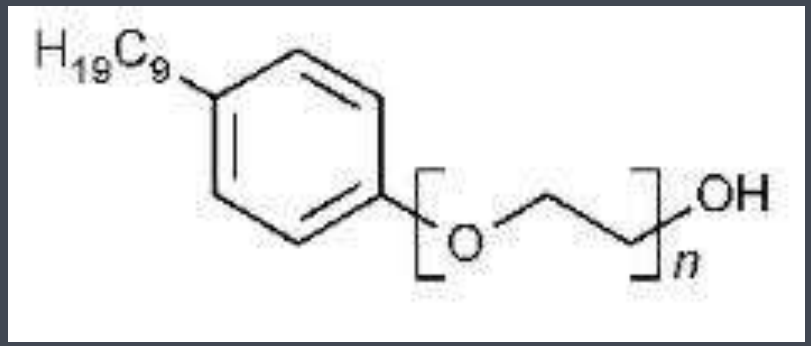
Compounds of Interest

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

QA/QC compound: Reserpine: 609.281 amu

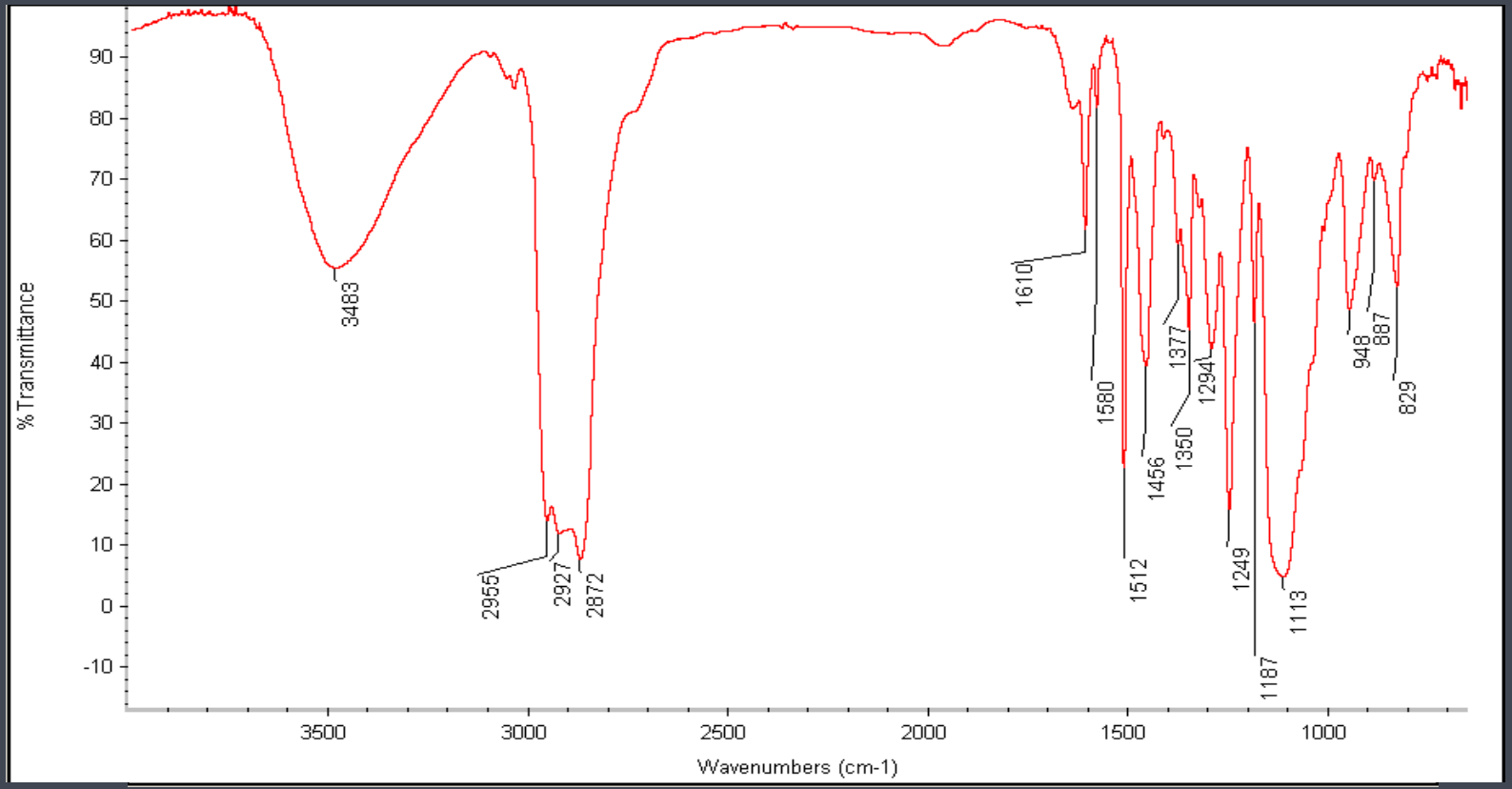
- PDMS: 200-600 amu





- 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

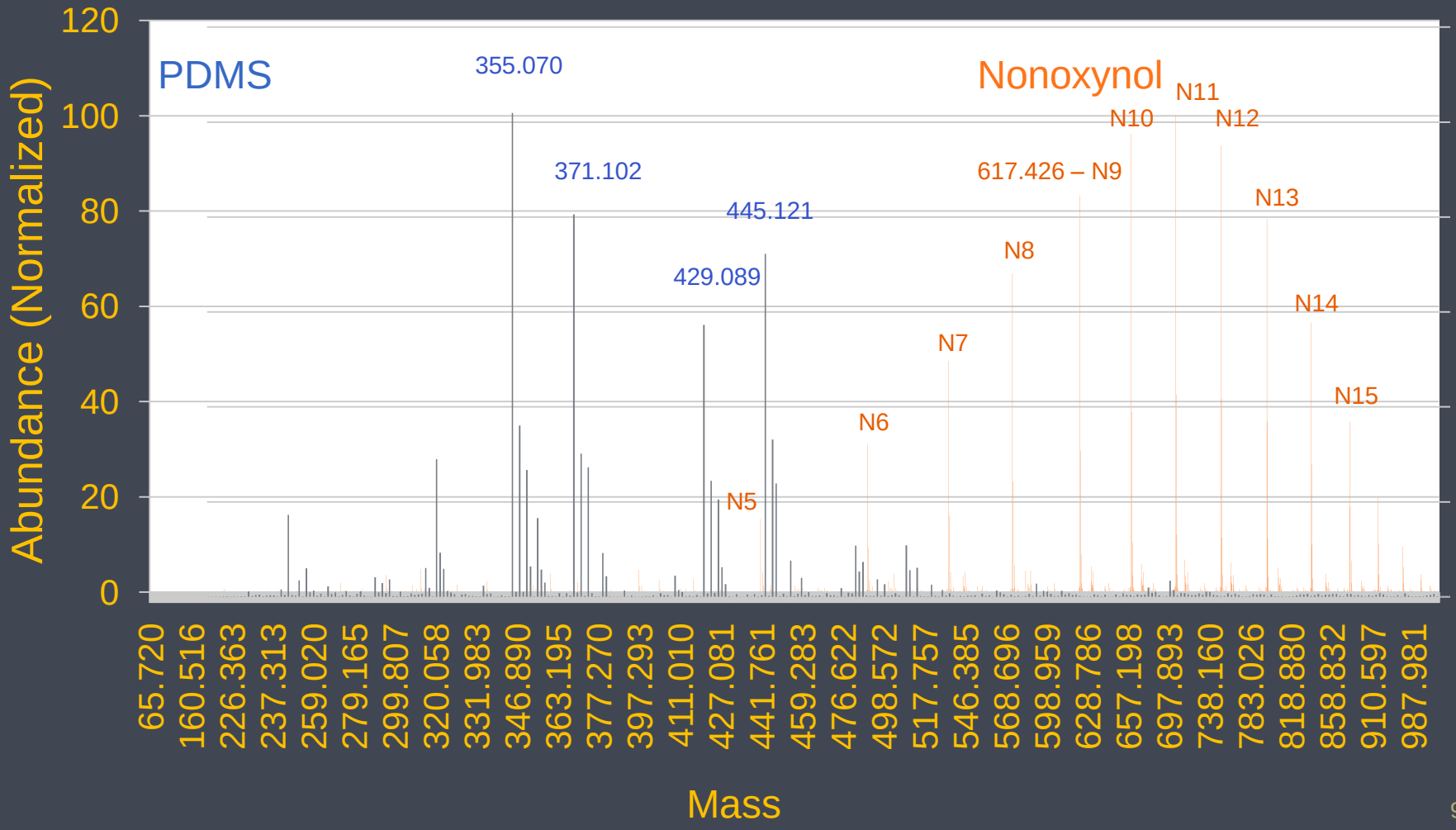
Orifice Temperature

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

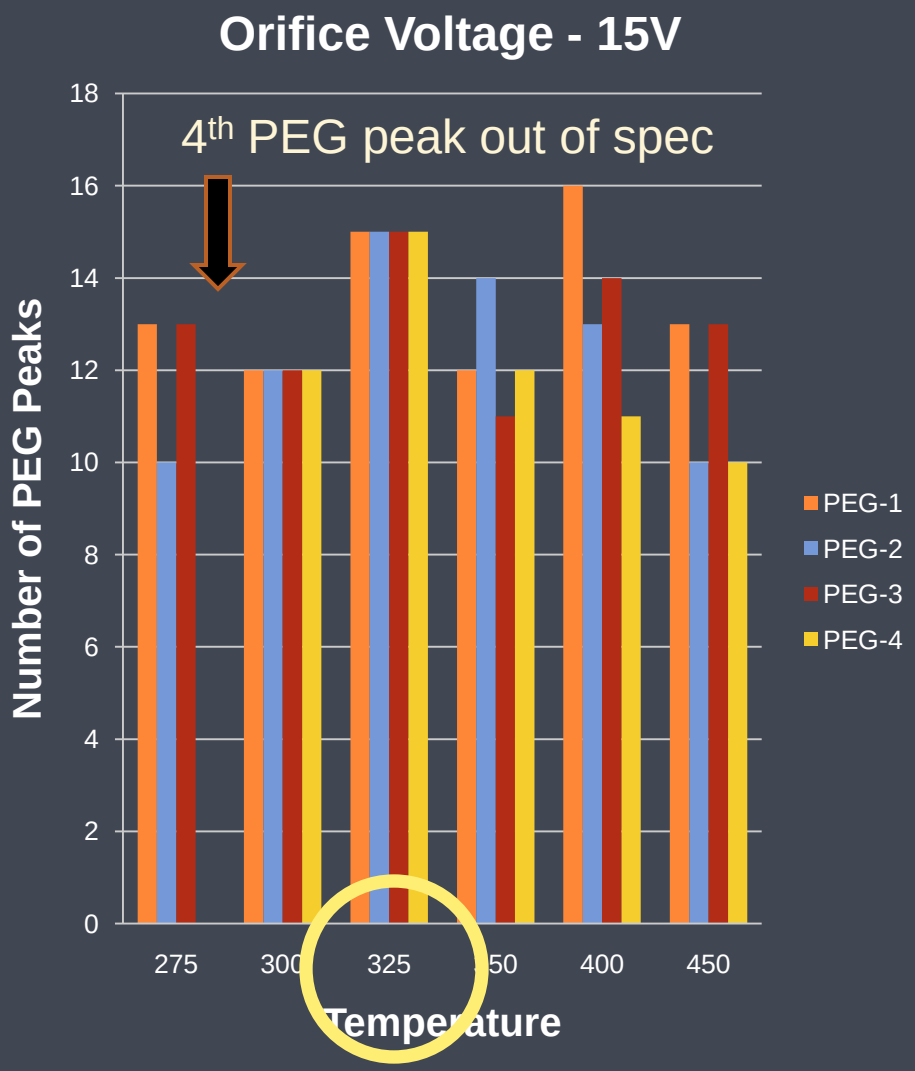
Detector Voltage: Controls overall signal

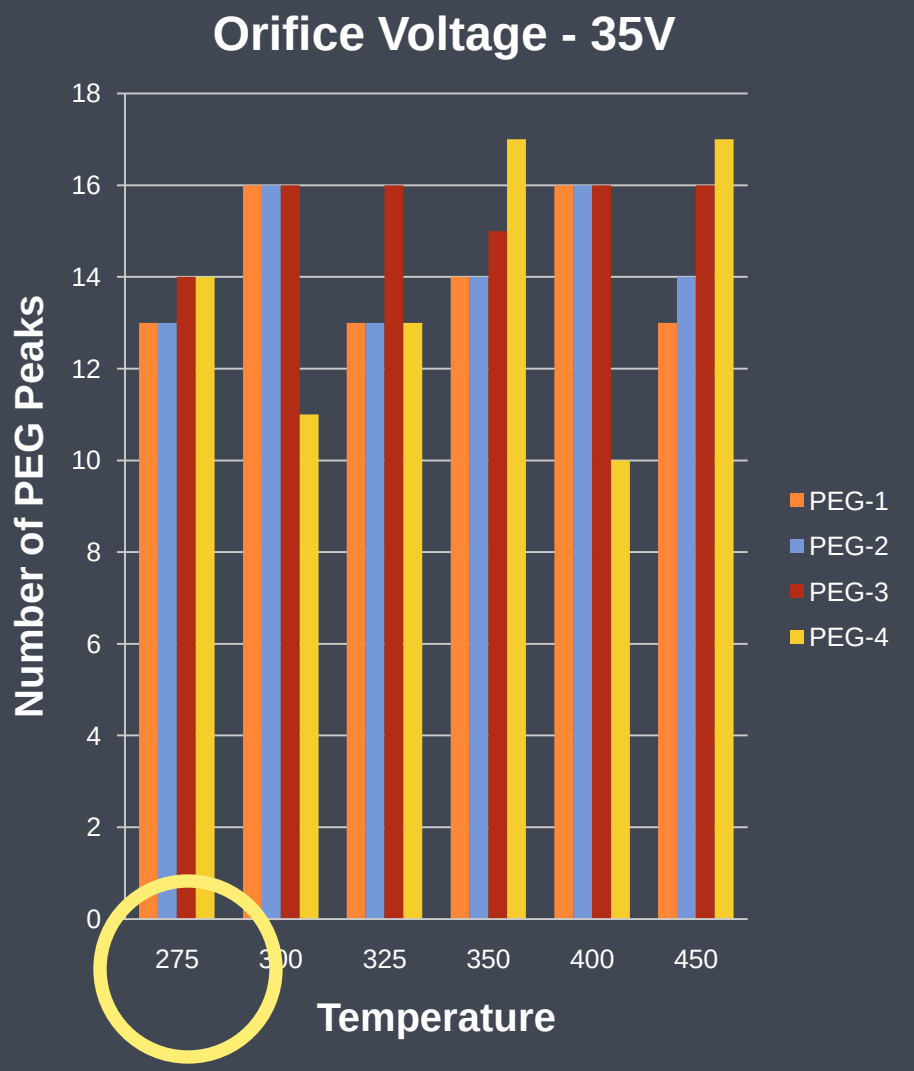
- Low V = Low signal (decreased noise)
- High V = High signal (increased noise)
- Values: 2200 and 2400 V

Mass Spectral Data

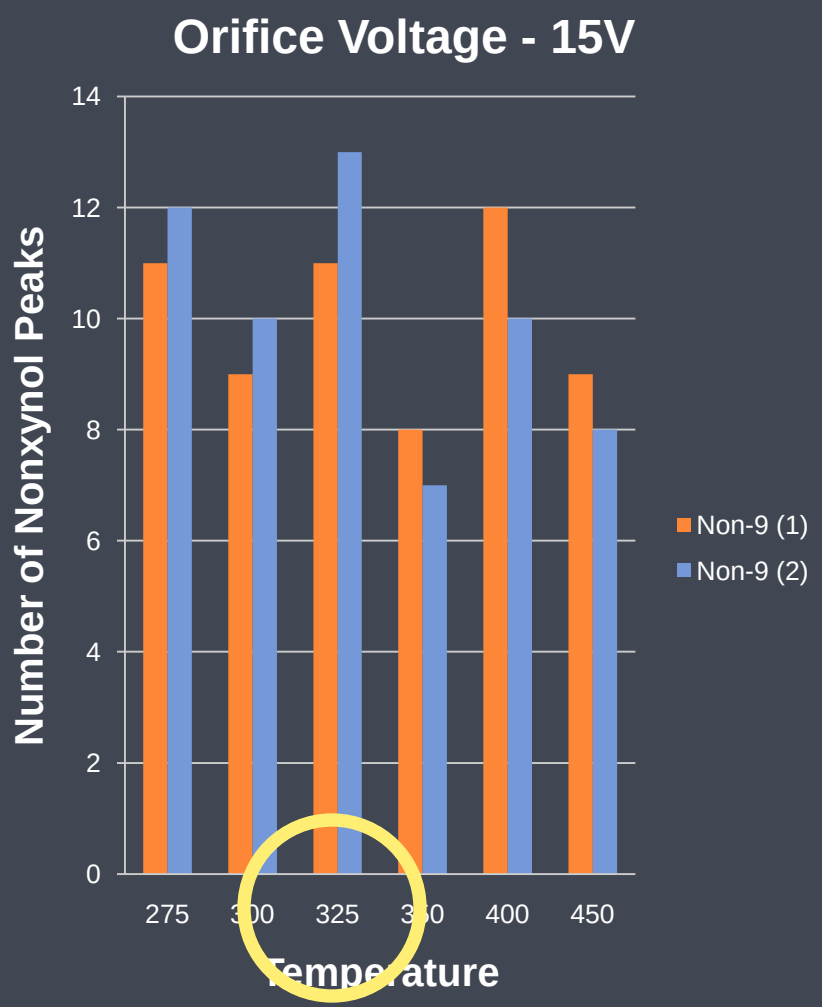


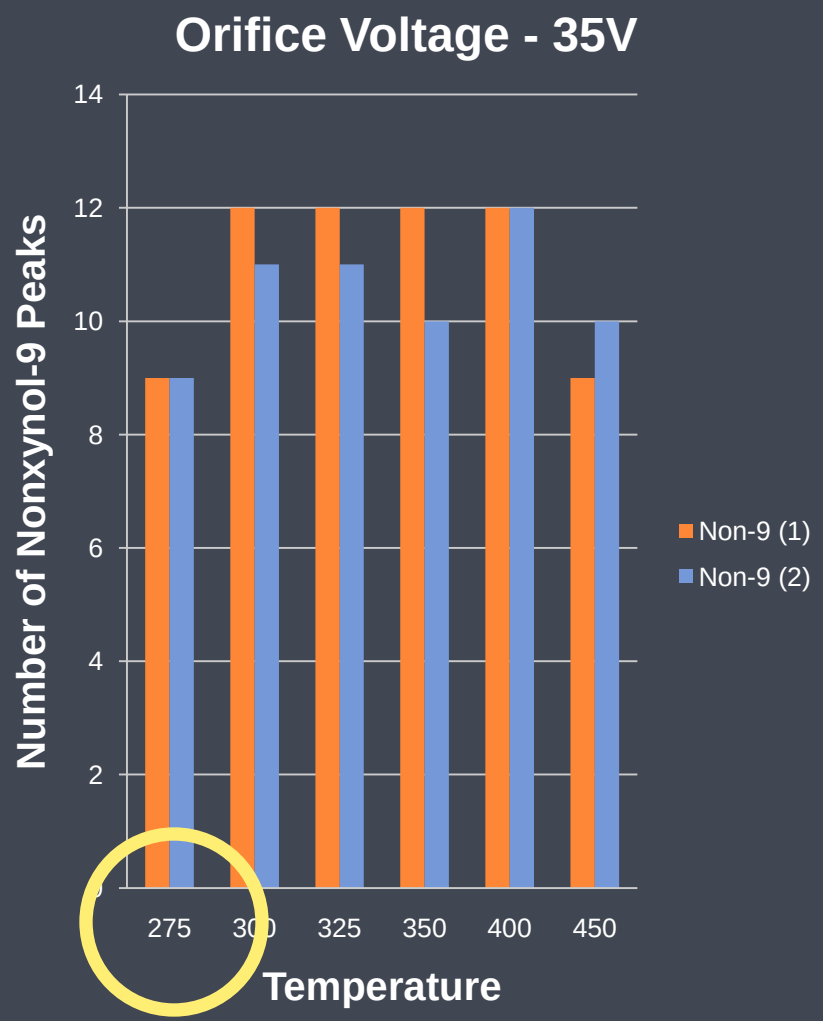
Choosing of optimal parameters - PEG





Choosing parameters Nonoxynol-9 (2200V)

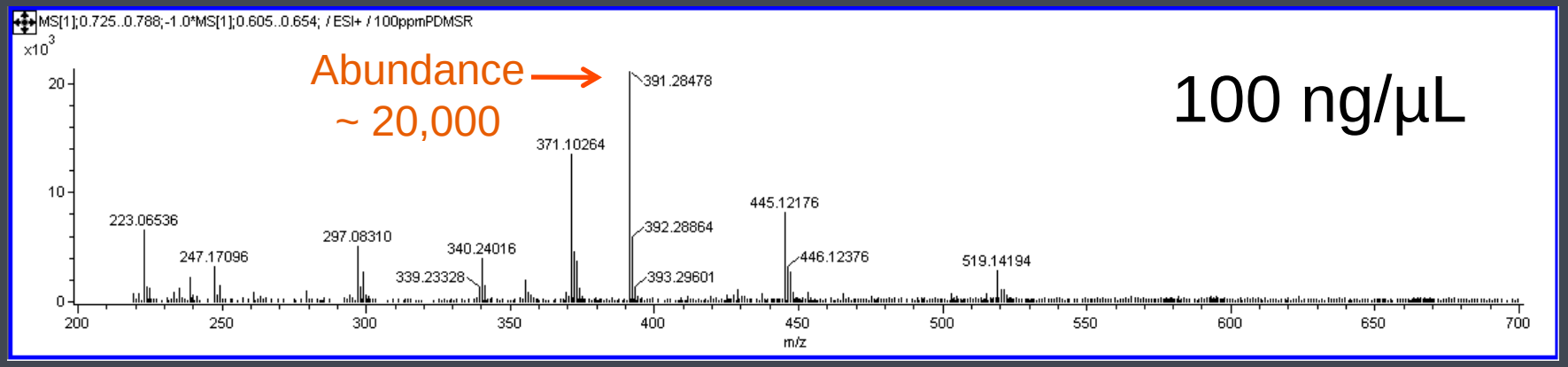


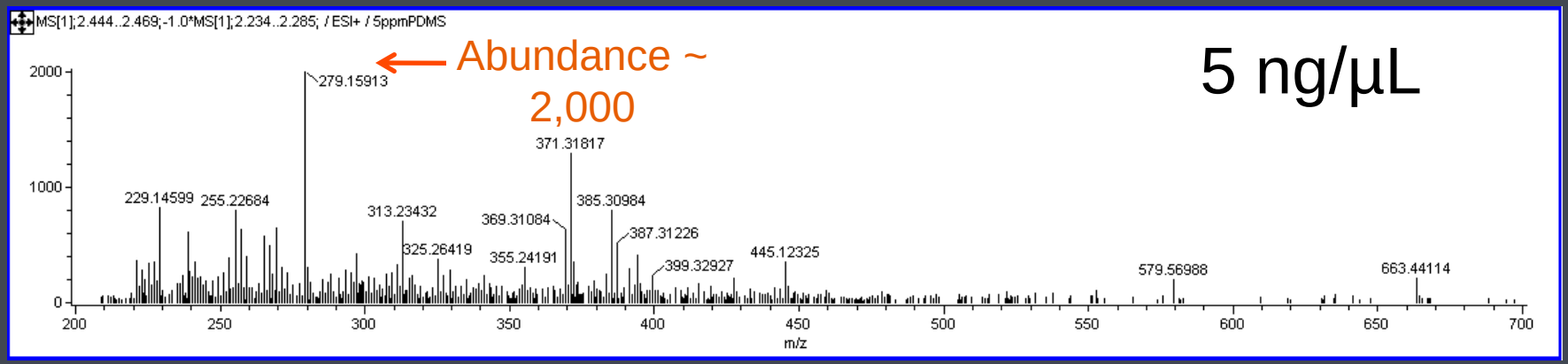


Limit of Detection

- 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



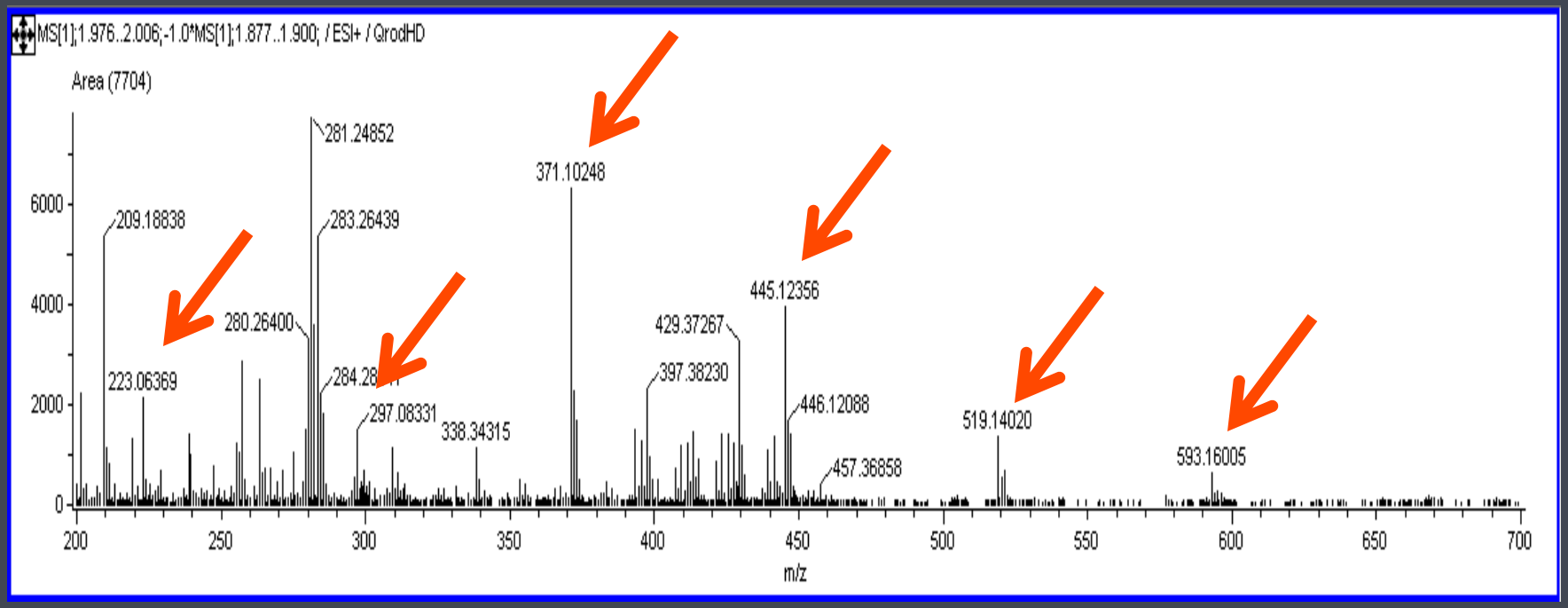


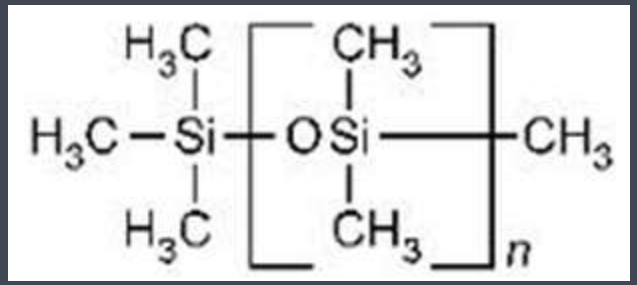
Sample:	PDMS by FTIR	PDMS by DART	N9 by FTIR	N9 by DART
LOD:	~50 ng	~30 ng	~500 ng	~ 10 ng

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

- Special Thanks
  - NIJ
  - Amy Michaud
  - Derek Dorrien, USACIL

QUESTIONS??