Developing a Combined Approach to Green Gunshot Residue Analysis in the Forensic Laboratory

Using Scanning Electron Microscopy with Energy-Dispersive X-Ray Spectroscopy and Comprehensive Two-Dimensional Gas Chromatography

Barbara Grace Saunders, Katelynn Perrault Uptmor

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The Case of the Invisible Trace

Scotland Yard receives a call.

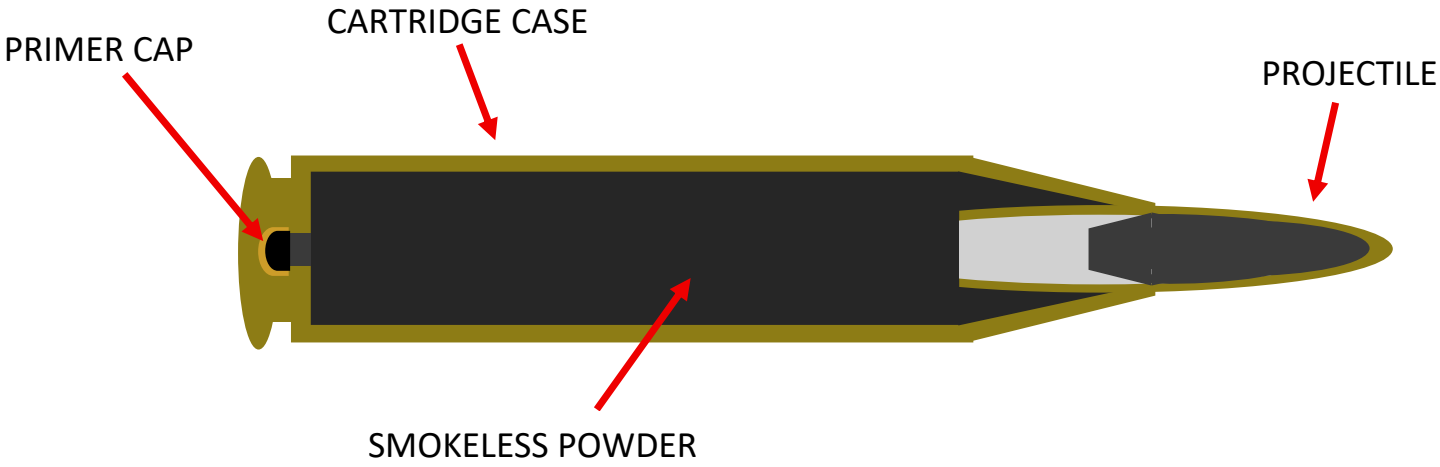
Professor Plum found prostrate on the ground.

Three suspects: Miss Scarlet, Colonel Mustard, and Mrs. Peacock all plead innocent.

Each suspect had a motive and an unsupported alibi.

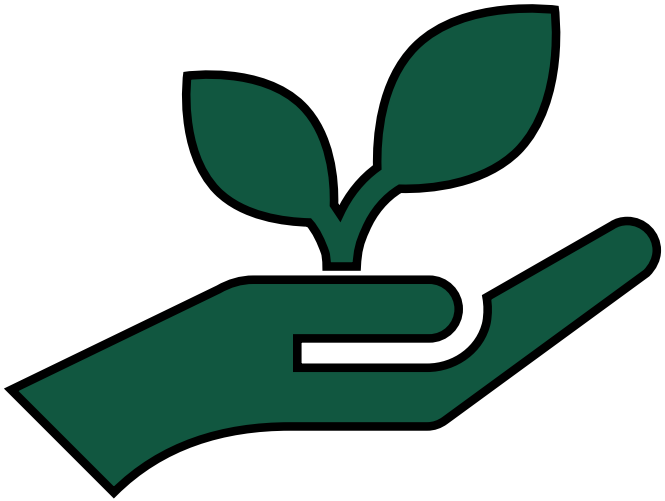
Suspects booked and brought in for questioning.

What is gunshot residue?



Gunshot residue (GSR) is particulate matter that is expelled from a firearm during a discharge event in the form of a “plume” of vaporized material.

Will the trace remain invisible?



- OSAC standards
  - Detection of heavy metals (lead, barium, antimony)
- “Green” ammunition
  - Heavy metal-free
  - Contains environmentally ubiquitous materials
- Samples of “green” GSR don’t adhere to analytical expectations!

Section 1

PRIOR WORK

Sample Collection

Developed SEM-EDS Profile for GSR Characterization

1. Morphological Characterization via SEM

- Particle appears in contrast to the background.
- Particle is between 0.5 and ~100 μm in diameter.
- Particle has spheroidal or irregular geometry.

2. Elemental Characterization via EDS

- Particle contains some assortment of three or more common metallic elements.
- EDS spectrum shows background noise.
- Particle is compositionally complex.

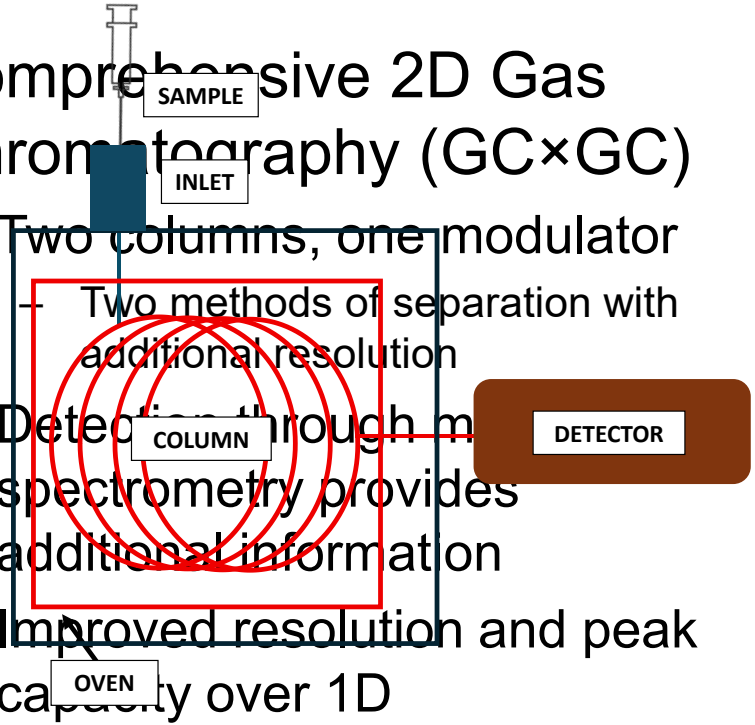
Section 2

GC×GC METHOD DEVELOPMENT

Why GC×GC?

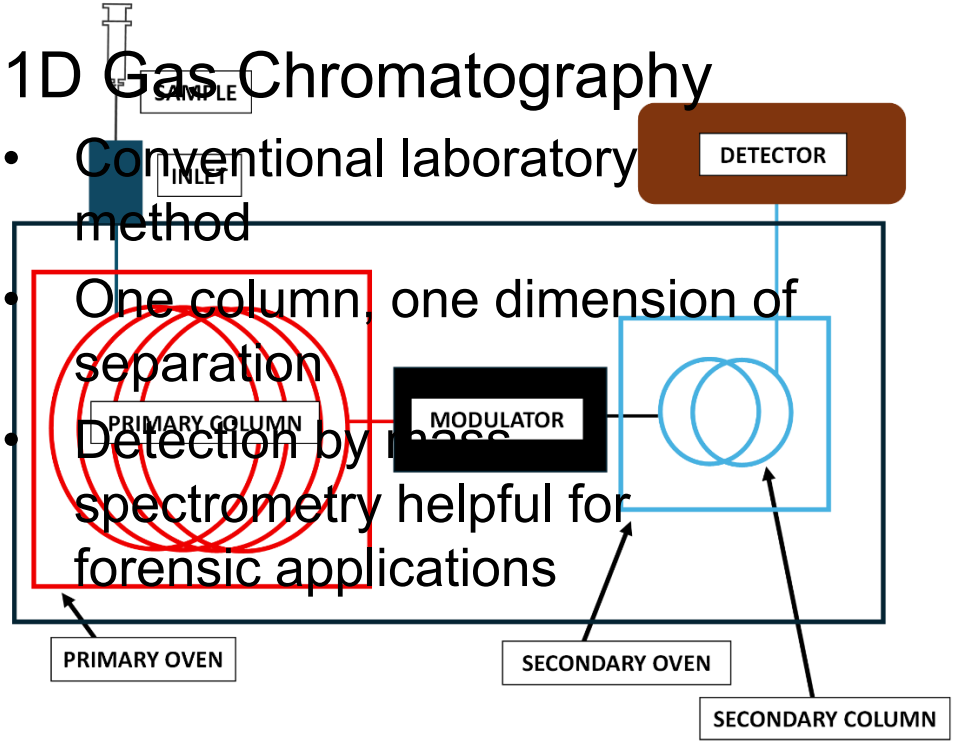
Comprehensive 2D Gas Chromatography (GC×GC)

- Two columns, one modulator
  - Two methods of separation with additional resolution
- Detection through mass spectrometry provides additional information
- Improved resolution and peak capacity over 1D

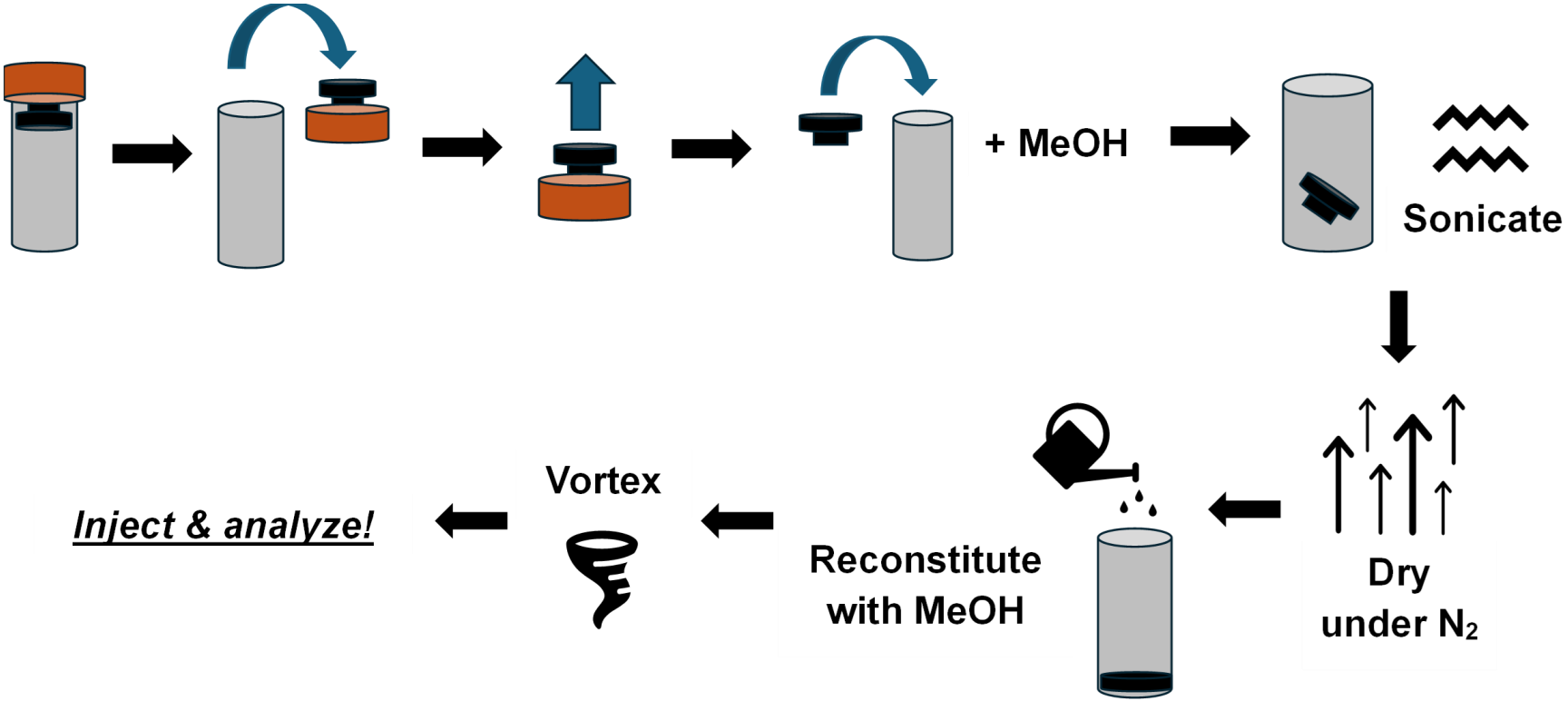


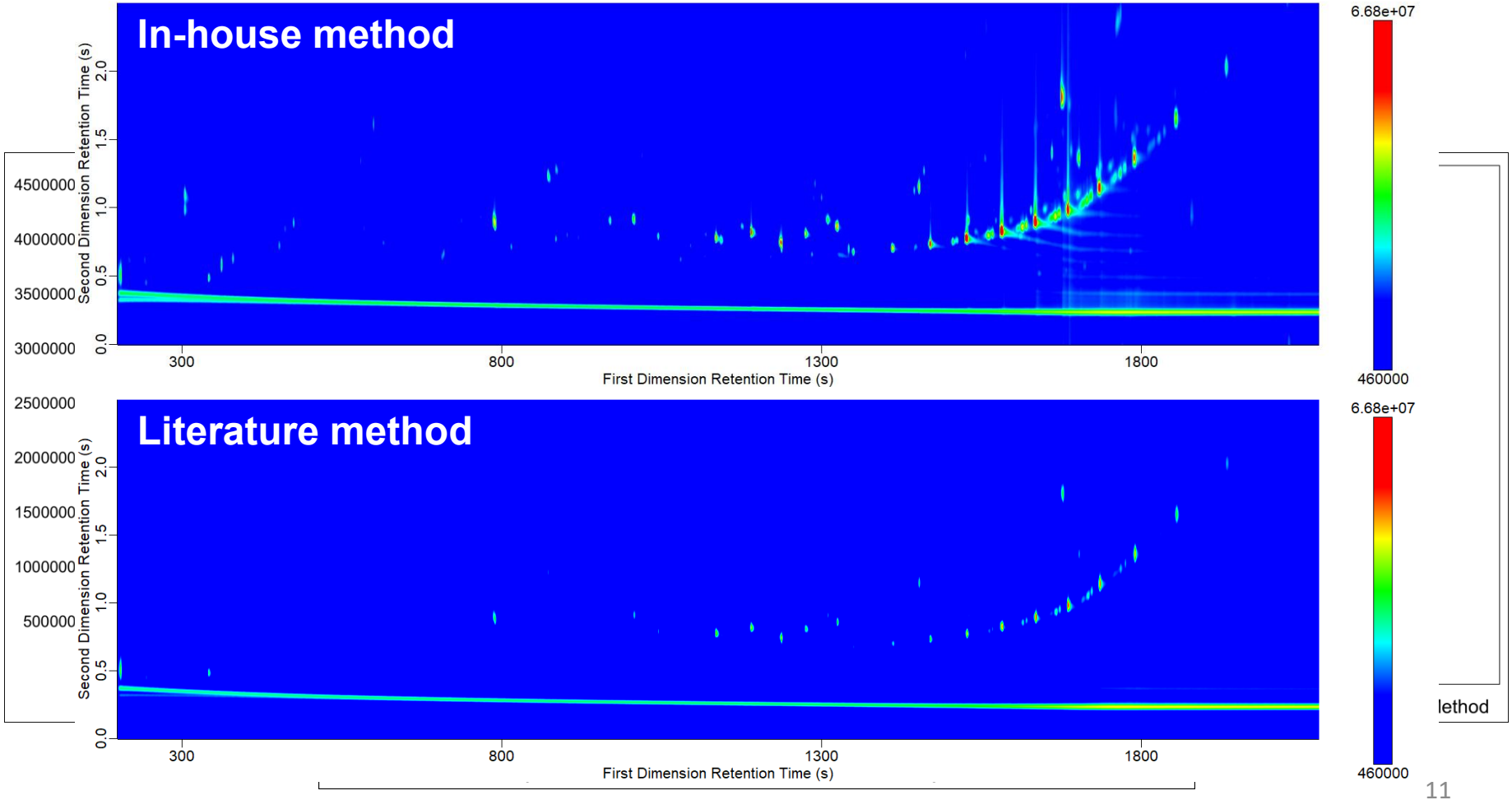
1D Gas Chromatography

- Conventional laboratory method
- One column, one dimension of separation
- Detection by mass spectrometry helpful for forensic applications



Sample Extraction




In-house method
Second Dimension Retention Time (s)
4500000
4000000
3500000
3000000
0.0
0.5
1.0
1.5
2.0
300
800
1300
1800
First Dimension Retention Time (s)
6.68e+07
460000
Literature method
2500000
2000000
1500000
1000000
500000
0.0
0.5
1.0
1.5
2.0
300
800
1300
1800
First Dimension Retention Time (s)
6.68e+07
460000
lethod

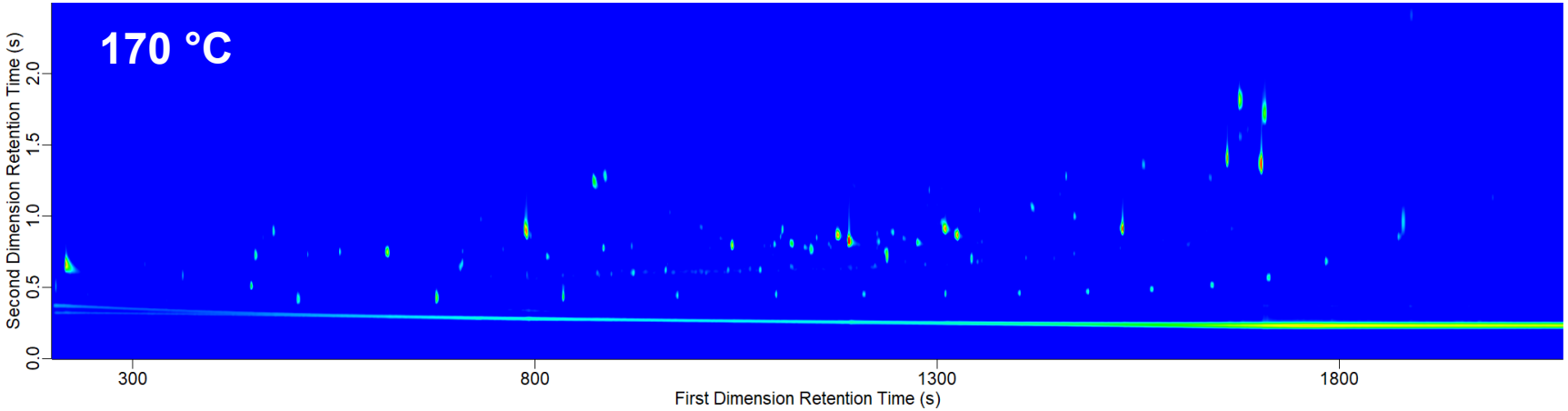
GC×GC Method Optimization

Prior work:

- Oven temperature program
- Choice of H_2 as carrier gas

What's left:

- Inlet temperature
- Modulation period


170 °C
Second Dimension Retention Time (s)
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1.5
2.0
300
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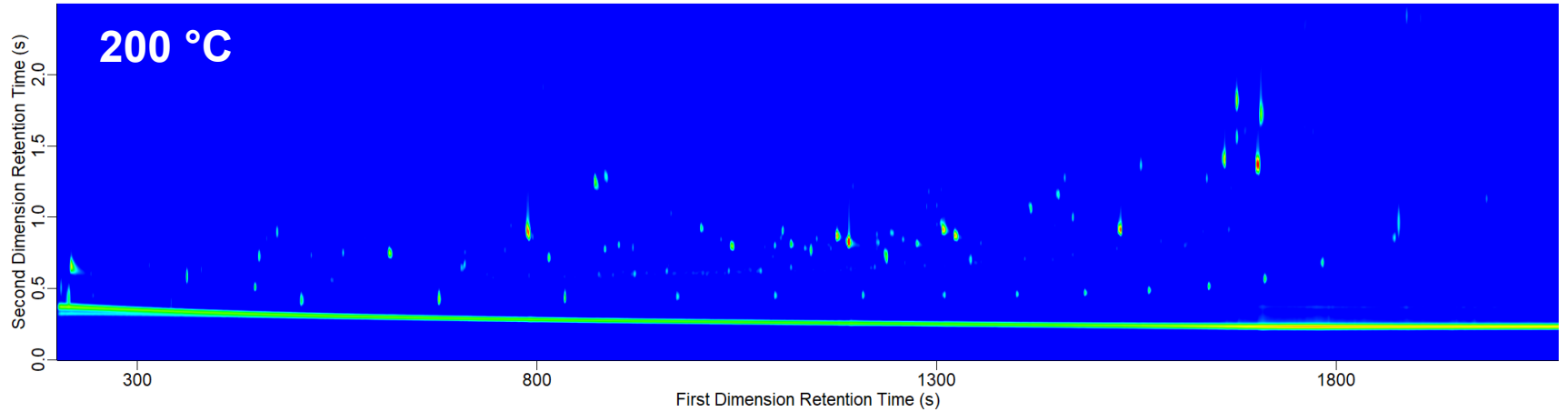
First Dimension Retention Time (s)
4.2069e+07
576430


200 °C
Second Dimension Retention Time (s)
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First Dimension Retention Time (s)
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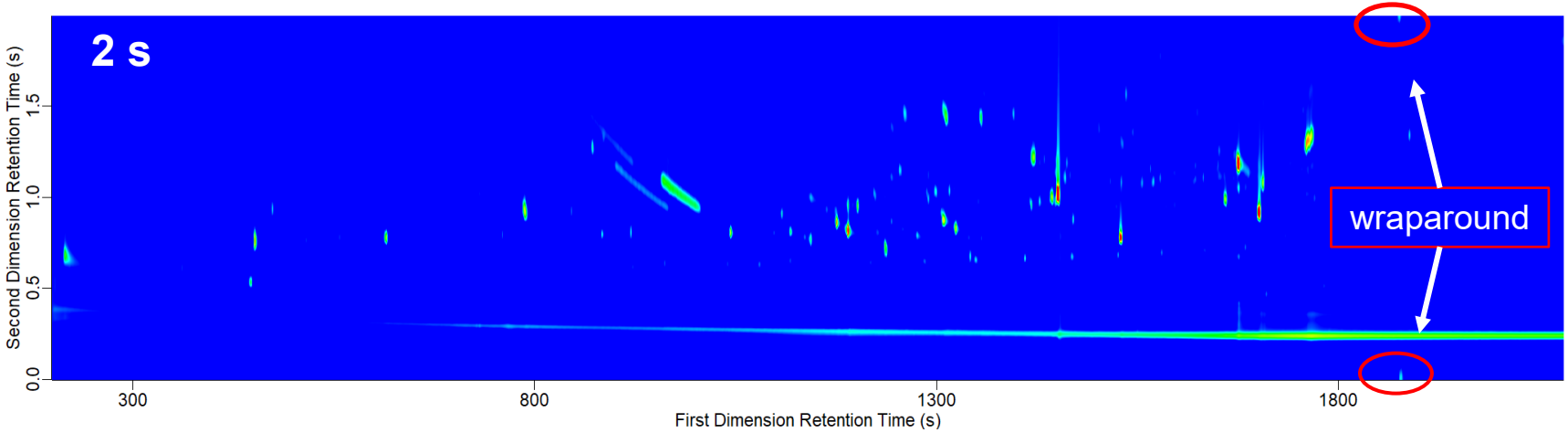
2 s
wraparound
Second Dimension Retention Time (s)
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800
1300
1800
First Dimension Retention Time (s)
4.45043e+07
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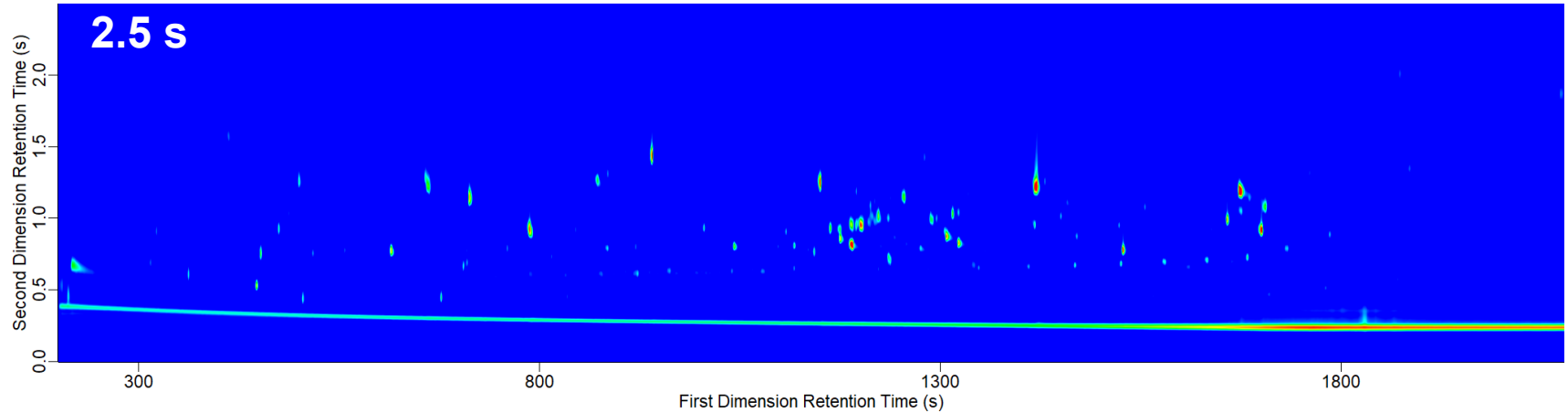

2.5 s
Second Dimension Retention Time (s)
0.0
0.5
1.0
1.5
2.0
300
800
1300
1800
First Dimension Retention Time (s)
2.05054e+07
610804

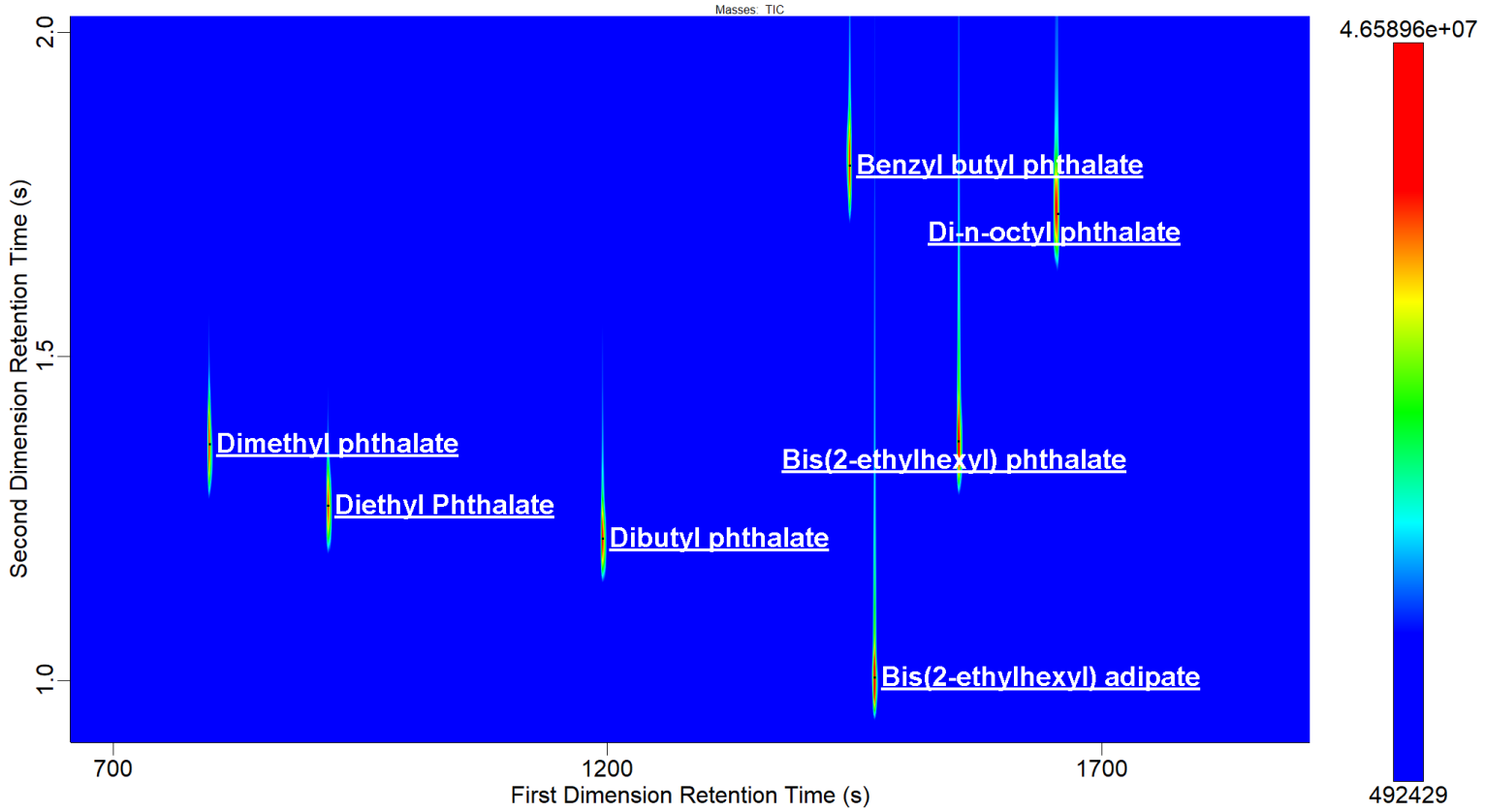
Optimized GC×GC Program

Columns (Agilent)	1D: HP-5Q (30 m × 0.25 mm i.d. × 0.25 µm) 2D: DB-17MS (1 m × 0.25 mm i.d. × 0.25 µm)
Carrier Gas	Hydrogen at 1.25 mL/min
Injection Type	Liquid, Split
Inlet Temperature	200 °C
Modulation Period	2.5 s (0.75 s hot pulse, 0.5 s cold pulse)
Oven Temperature Program	50 °C for 1 min to 280 °C at 8.3 °C/min, hold for 6 min
Secondary Oven Offset	+5 °C
Modulator Temperature Offset	+15 °C
Transfer Line Temperature	250 °C
Ion Source Temperature (Mass Spectrometer)	300 °C

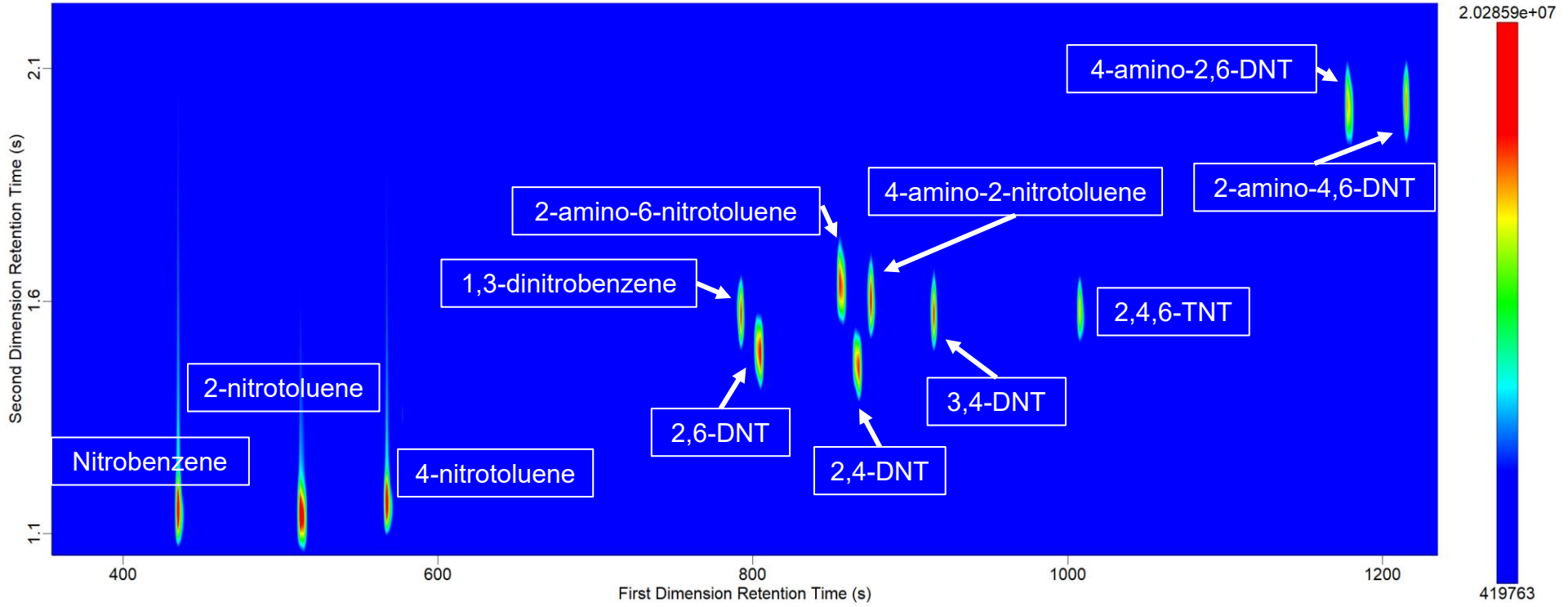
Standards Analysis

- **Phthalate standard**
  - 7-component, EPA 506 Phthalate Mix
- **Nitroaromatic mix**
  - 12-component, AccuStandard
- **Nitroglycerin**
  - Single-component, AccuStandard
- **Gun surveillance standard**
  - 9-component, AccuStandard

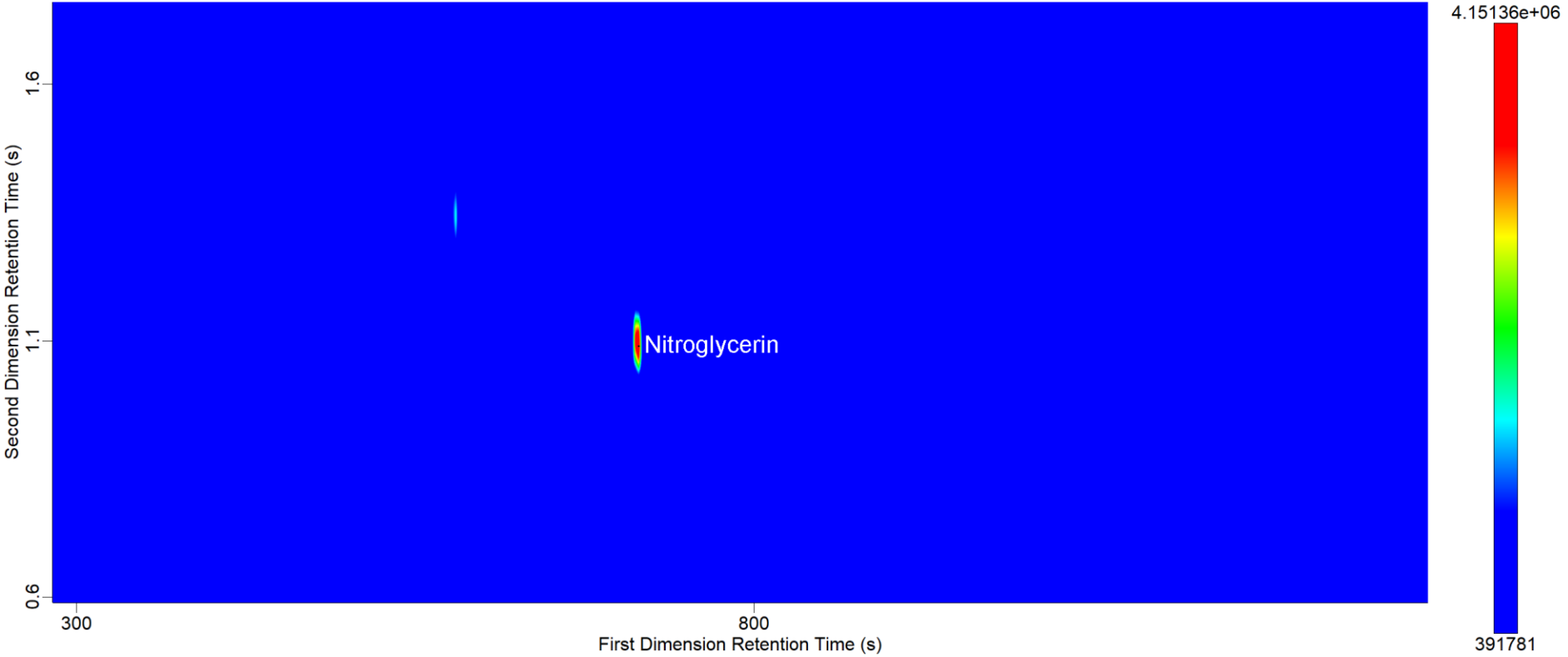
Phthalate Standard



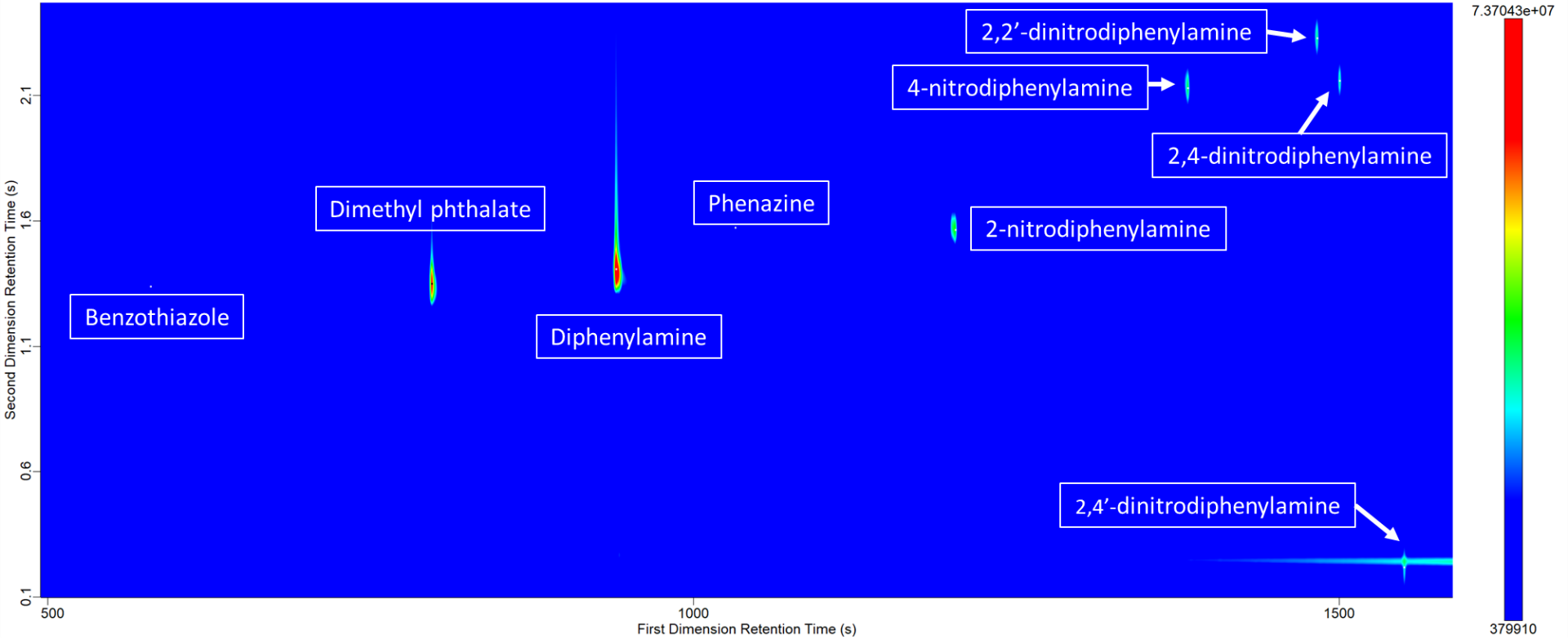
Nitroaromatic Mix



Nitroglycerin



Gun Surveillance Standard



Conclusion

So far:

- ✓ Developed strategy for SEM-EDS analysis for green IGSR
- ✓ Optimized extraction method for OGSR analysis
- ✓ Optimized GC×GC OGSR analysis conditions
- ✓ Verification of relevant analyte visibility and identity in chromatograms using standards

Next steps:

- Further development
- Strengthen credibility under Rule 702
  - Sorbent-based extraction methods
  - Retention indices

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LECO
EMPOWERING RESULTS

Seplab Instagram

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