
# An Introduction to Gas Chromatography – Vacuum Ultraviolet Spectroscopy

Tom Steen

Technical Sales Representative,
Northeast US and Canada, VUV Analytics

# Today's Speaker

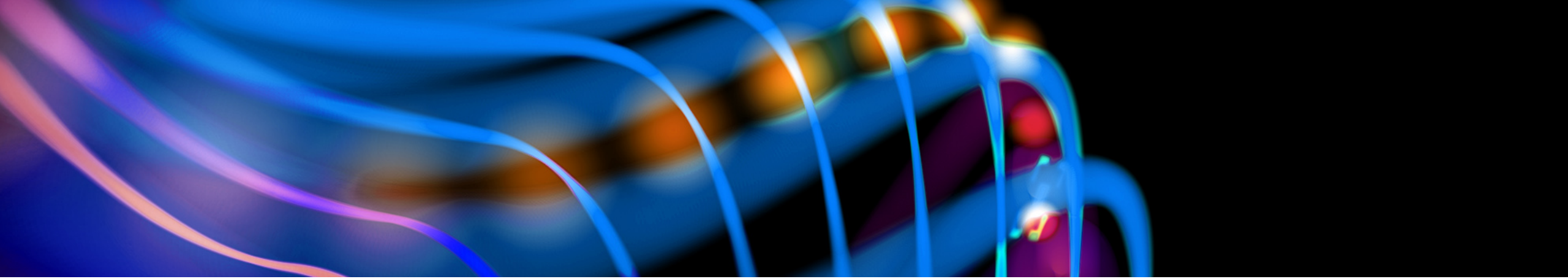
Tom Steen

Northeast & Canada Technical Sales

VUV ANALYTICS

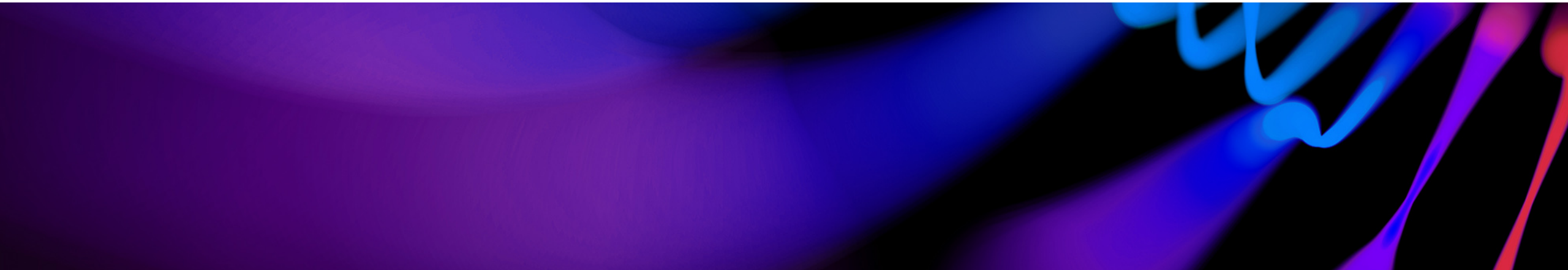
# Presentation Overview

- Introduction to VUV Analytics, Inc.
- What is Vacuum Ultraviolet Spectroscopy?
  - Theory of Operation
  - VGA Family of Detectors
  - Detector Operation
  - Data and Spectral Deconvolution
- Example Applications
- Summary



# VUV Analytics, Inc.

About Us



# Who is VUV Analytics?

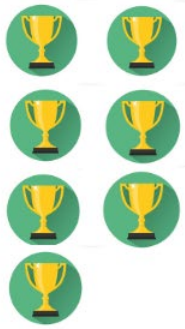
VUV ANALYTICS

## FAST FACTS

**Since it's founding in 2009...**

### HEADQUARTERS IN CEDAR PARK, TX

Just outside of Austin, TX, VUV Analytics HQ is home to the global R&D, manufacturing and operations.



### 7 AWARDS & 20+ PATENTS

VUV Analytics has won over 7 Awards for the VGA Detector from multiple industries - process, oil & gas, R&D and cannabis. VUV holds over 20+ global patents on our technology and software. :

### GLOBAL NETWORK

Even though VUV Analytics is based in the United States, their global partner network of distributors and 2 OEM partners gives them a worldwide presence, that is still growing.

### DIVERSIFIED GLOBAL CUSTOMERS

The VUV customer base is deployed globally across multiple industries including:

**Oil & Gas**
**Pharma**
**Forensics**
**Cannabis**
**Petrochemical**
**CRO**

### PUBLICATIONS & METHODS

VUV Analytics has 48+ published scientific papers on GC-VUV technology.
VUV Analytics has 2 approved ASTM methods and 1 working number.

ASTM INTERNATIONAL

**LEARN MORE AT VUVANALYTICS.COM.**

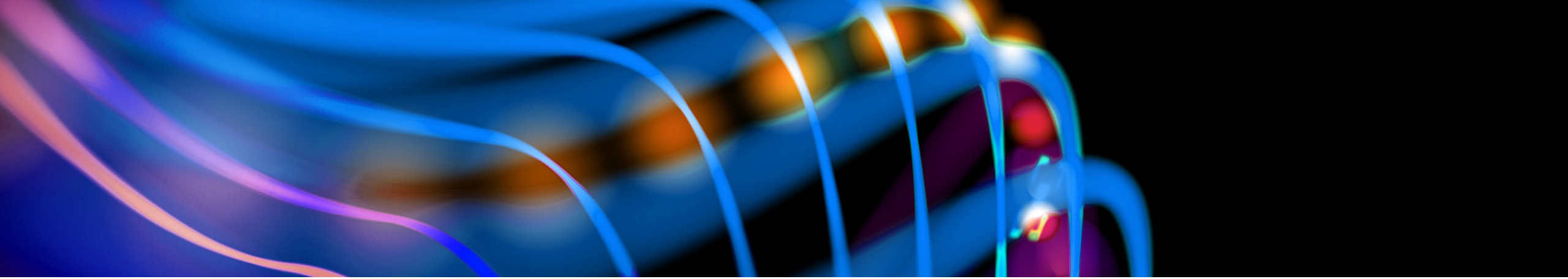
# Who do we work with?

## Key Customers

TOTAL
DOW
CDC
Centers for Disease Control and Prevention
lyondellbasell
ExxonMobil
LG Hausys
AIR LIQUIDE
o2si smart solutions
An LGC Standards Company
ALBERTA INNOVATES
R.J. REYNOLDS TOBACCO
PHILLIPS 66
ارامكو السعودية
Saudi Aramco
intertek
Altria
bp
GM
AMYRIS
CITGO
Monroe Energy
FUELING THE CLIMB
Chevron
sasol
FDA
NREL
NATIONAL RENEWABLE ENERGY LABORATORY
Bristol Myers Squibb™
SAMSUNG
MARATHON
VALERO

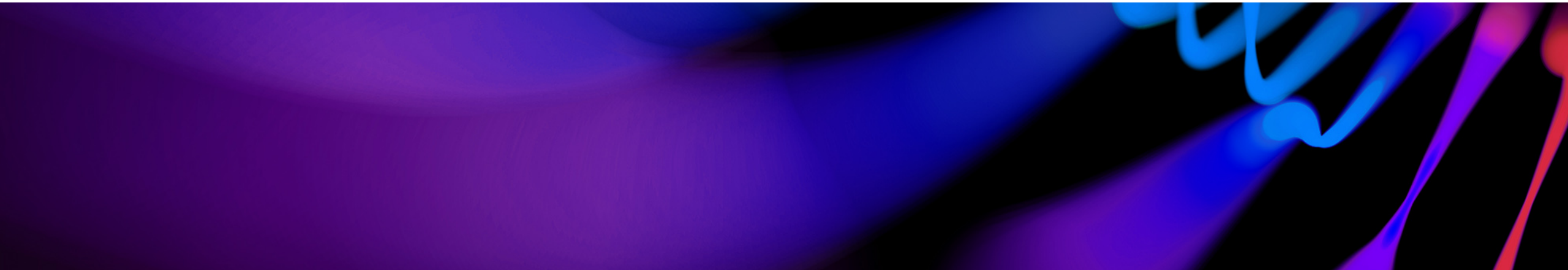
## Industry Awards

- 2014 Best New Analytical Instrument – *Gulf Coast Conference*
- TASIA 2014 Top 5 – *Analytical Scientist*
- 2015 Award Winner – *R&D 100*
- Innovative Product of the Year – *ISA 2016*
- TASIA 2016 Top 10 – *Analytical Scientist*
- 2018 Best New Analytical Technology – *Terpenes and Testing World*



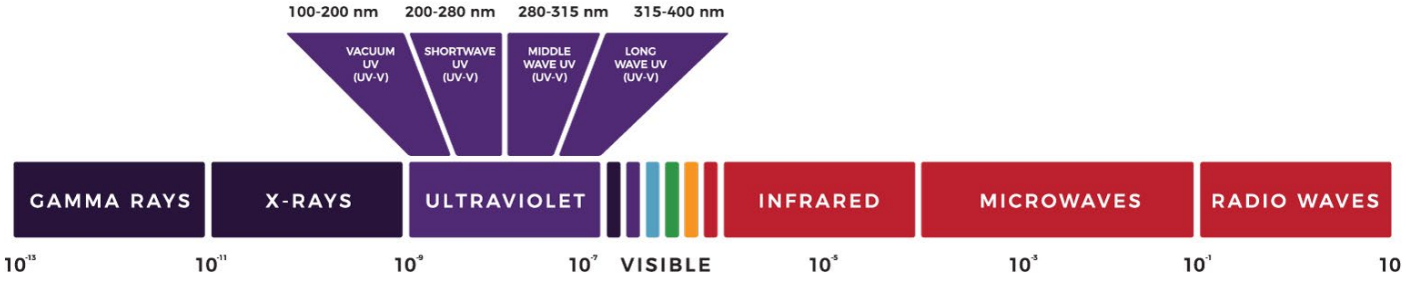
# What is Vacuum Ultraviolet Spectroscopy?

Theory, Operation, Benefits



# What is Vacuum Ultraviolet (VUV) Spectroscopy?

It is all about the light

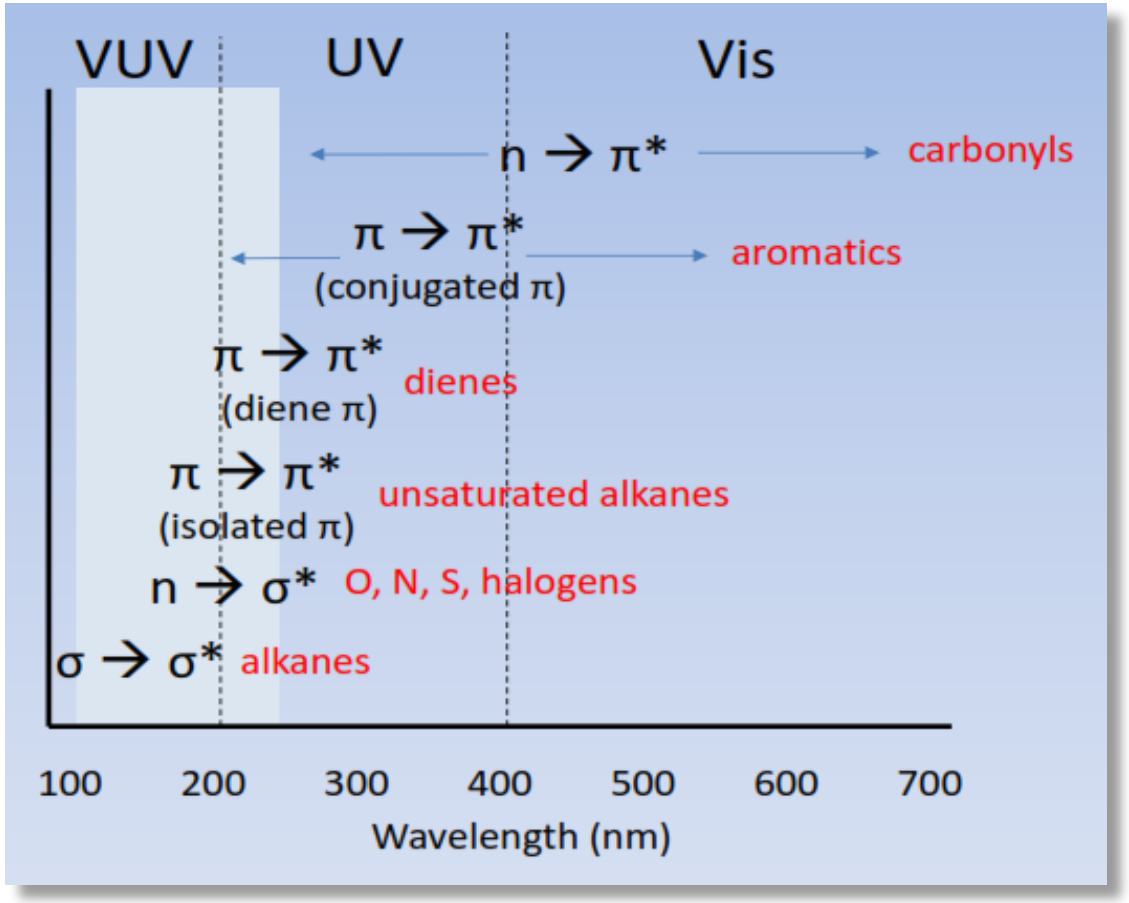



- Works in a part of the electromagnetic spectrum that has previously been difficult to commercialize
- Characterized by very short wavelength (125 – 240 nm), high energy absorbance
- Nearly every compound absorbs in this region (except He, Ar, H)
- Compounds that absorb in this region have unique spectral fingerprints

# What is Vacuum Ultraviolet (VUV) Spectroscopy?

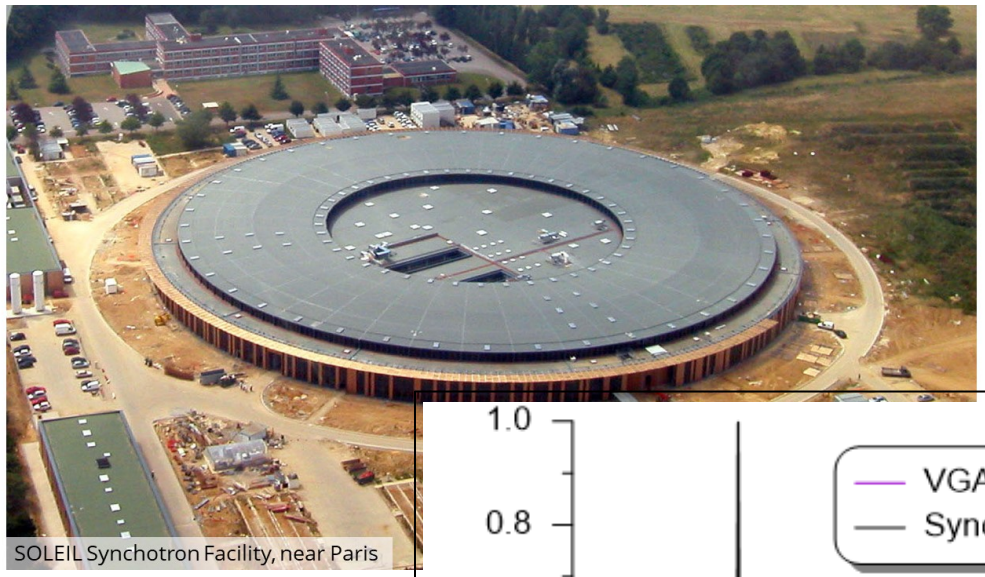
## Theory of Operation

- High energy, short wavelength **VUV** light induces electronic transitions in most chemical bonds chemical bonds including σ→ σ* and π→ π*.
- These excitations provide for unique spectral fingerprints.
- Unique spectral fingerprints allows for unambiguous compound identification – even positional isomers

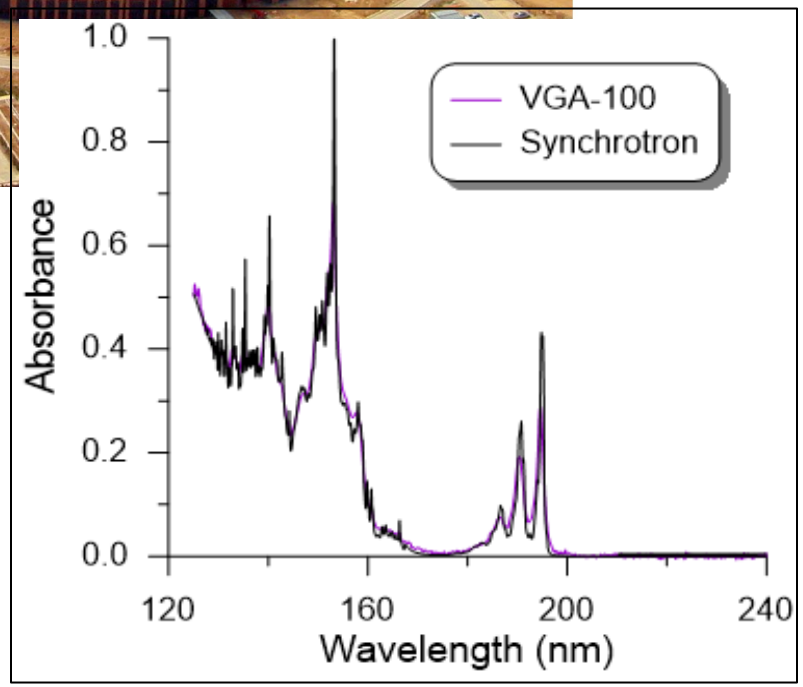


# What is Vacuum Ultraviolet (VUV) Spectroscopy?

## It's like having your own particle accelerator



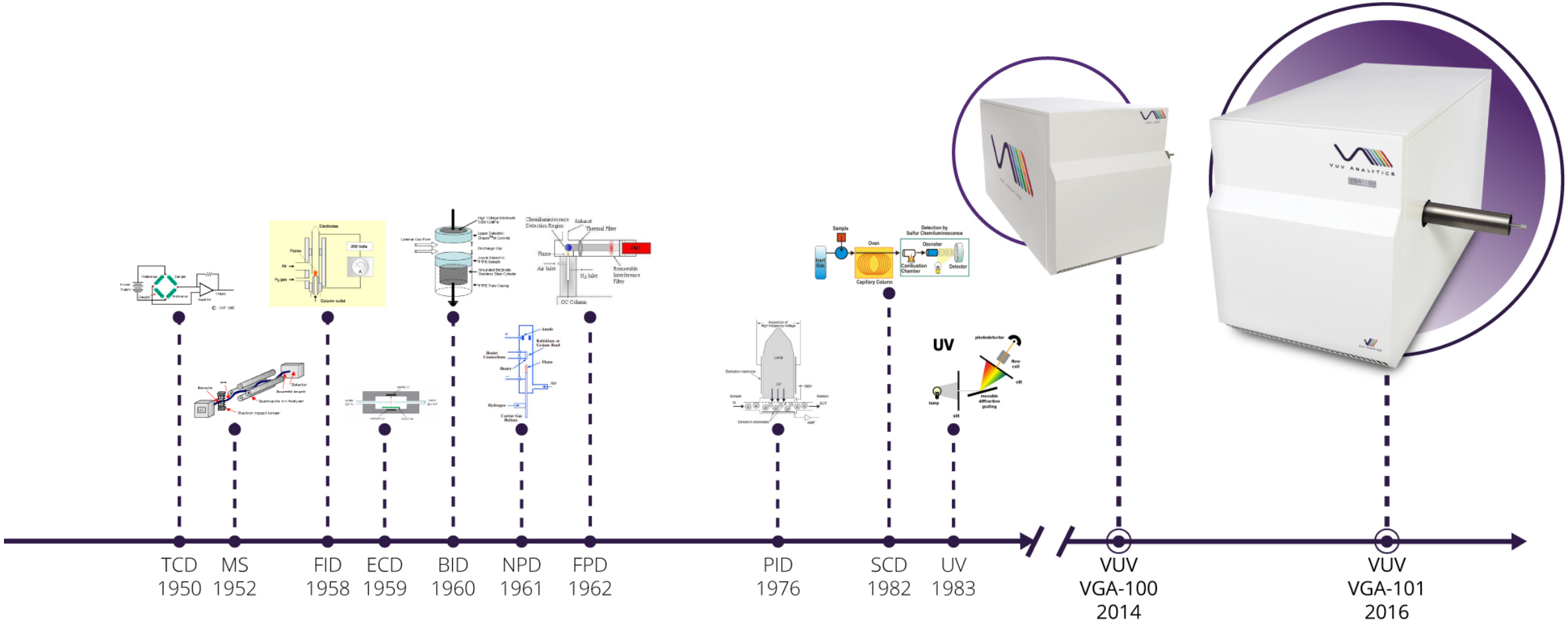
SOLEIL Synchotron Facility, near Paris




- Previously required the use of a Synchrotron (particle accelerator)
- VUV Analytics pioneered the first benchtop VUV Detectors – The VGA-100 and VGA-101

# Benchtop VUV Spectroscopy

The 1st GC Innovation in ~30 Years!



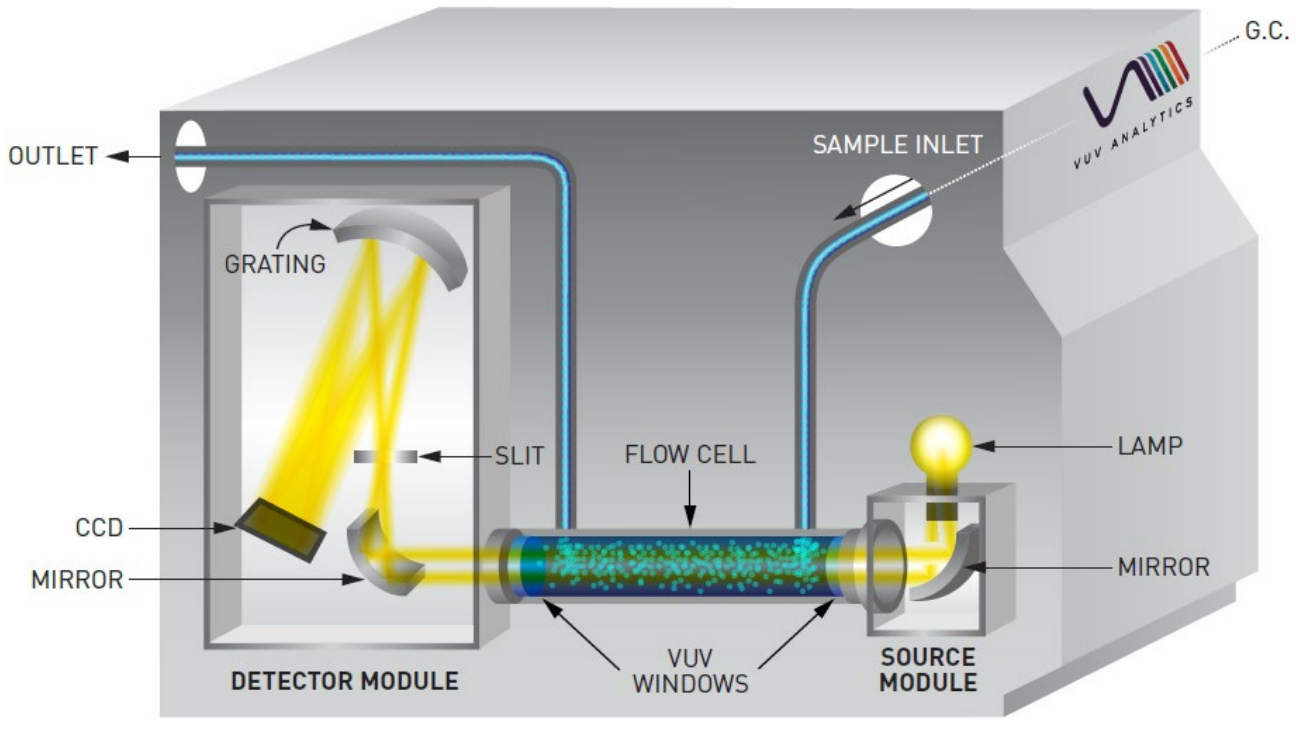
# What is Vacuum Ultraviolet (VUV) Spectroscopy?

## VGA Family of Detectors

- **Data confidence through spectral identification**
  - Unique Spectra=Unambiguous compound identification
  - Easily deconvolve co-eluting analytes
  - Clear and easy isomer differentiation
- **Excellent sensitivity**
  - Low picogram
- **Excellent temporal resolution**
  - Up to 75Hz Sampling
- **Predictable linear response**
  - 1st principle detection reduces calibration burdens
- **Reliable & Easy to use**
  - No vacuums pumps
  - No need for baseline resolved peaks

| | VGA-100 | VGA-101 |
|---|---|---|
| Max Temp | 300°C | 430°C |
| λ Range | 125 - 240 nm | 125 - 430 nm |
| Acq Rate | 75 Hz | 75 Hz |

# How does a VGA detector Work?



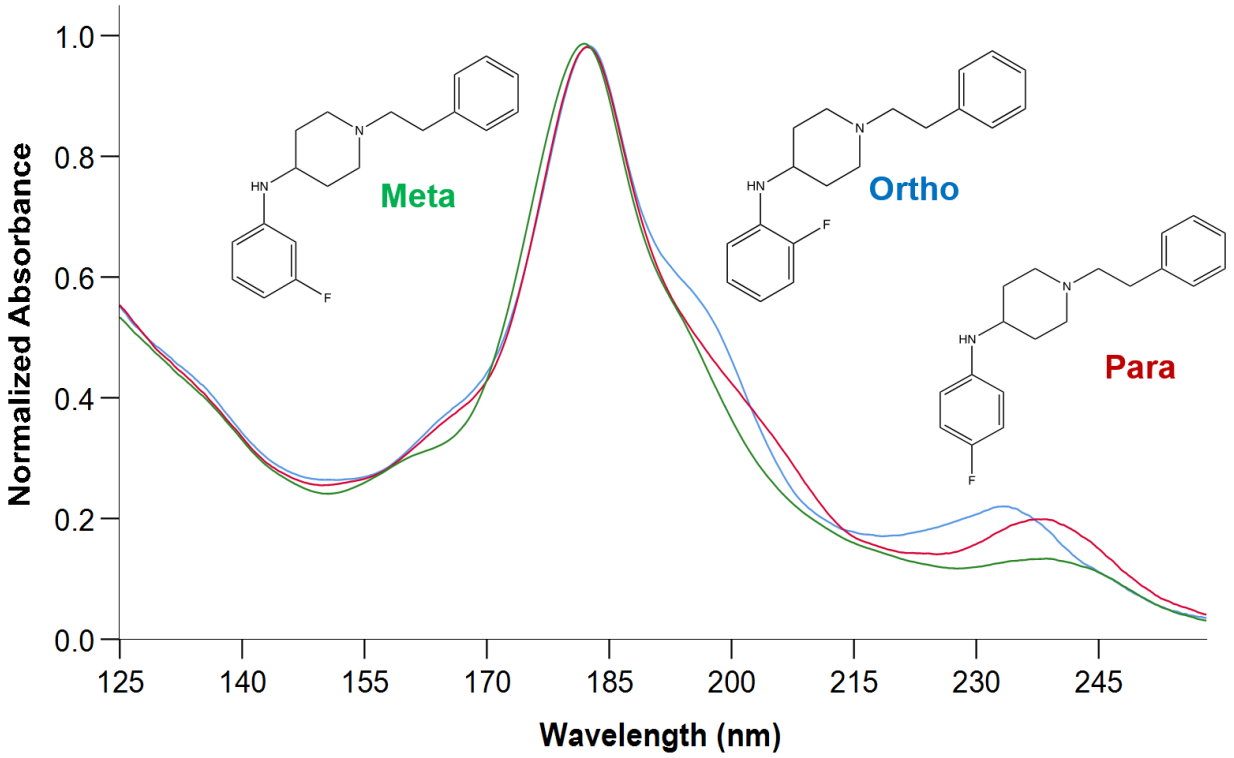

- GC analytes are passed to the VGA detector and excited by deuterium lamp
- Electronic transitions create unique spectral fingerprints
- Data is acquired in 3D in VUVision™ Software for analysis
- Data is both Qualitative and Quantitative

# Vacuum Ultraviolet Spectroscopy

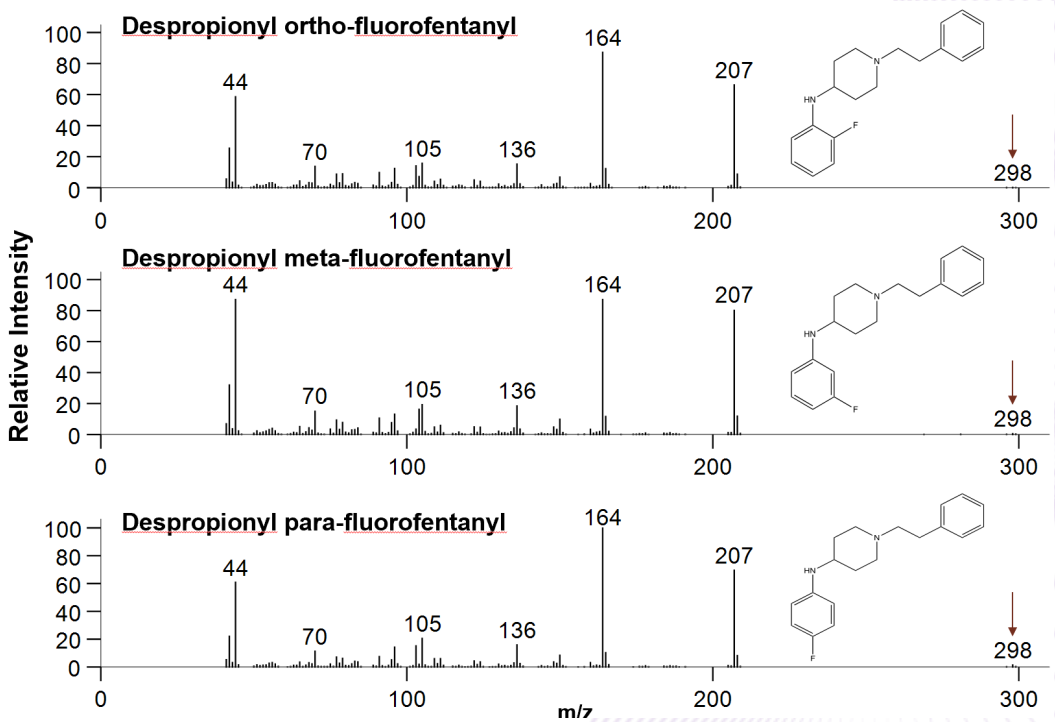
## Data in Three Dimensions

**Isomeric compounds that are difficult to differentiate with MS can be identified using VUV spectroscopy**

VUV absorbance identification
of Despropionyl Fluorofentanyl Isomers

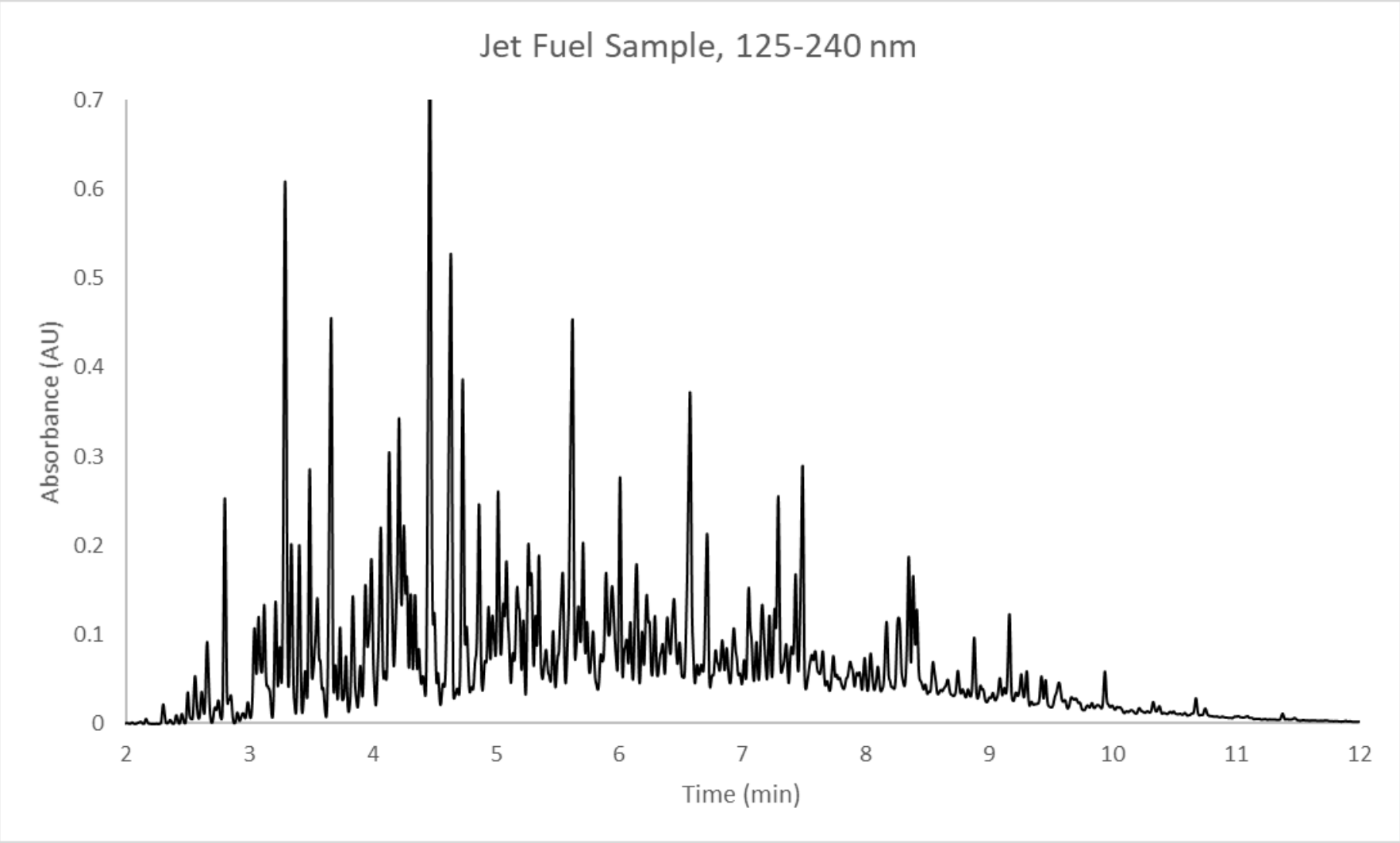



Mass Spectrometry Analysis
of Despropionyl Fluorofentanyl Isomers



# Vacuum Ultraviolet Spectroscopy

## Complex Analyses Simplified



# Vacuum Ultraviolet Spectroscopy

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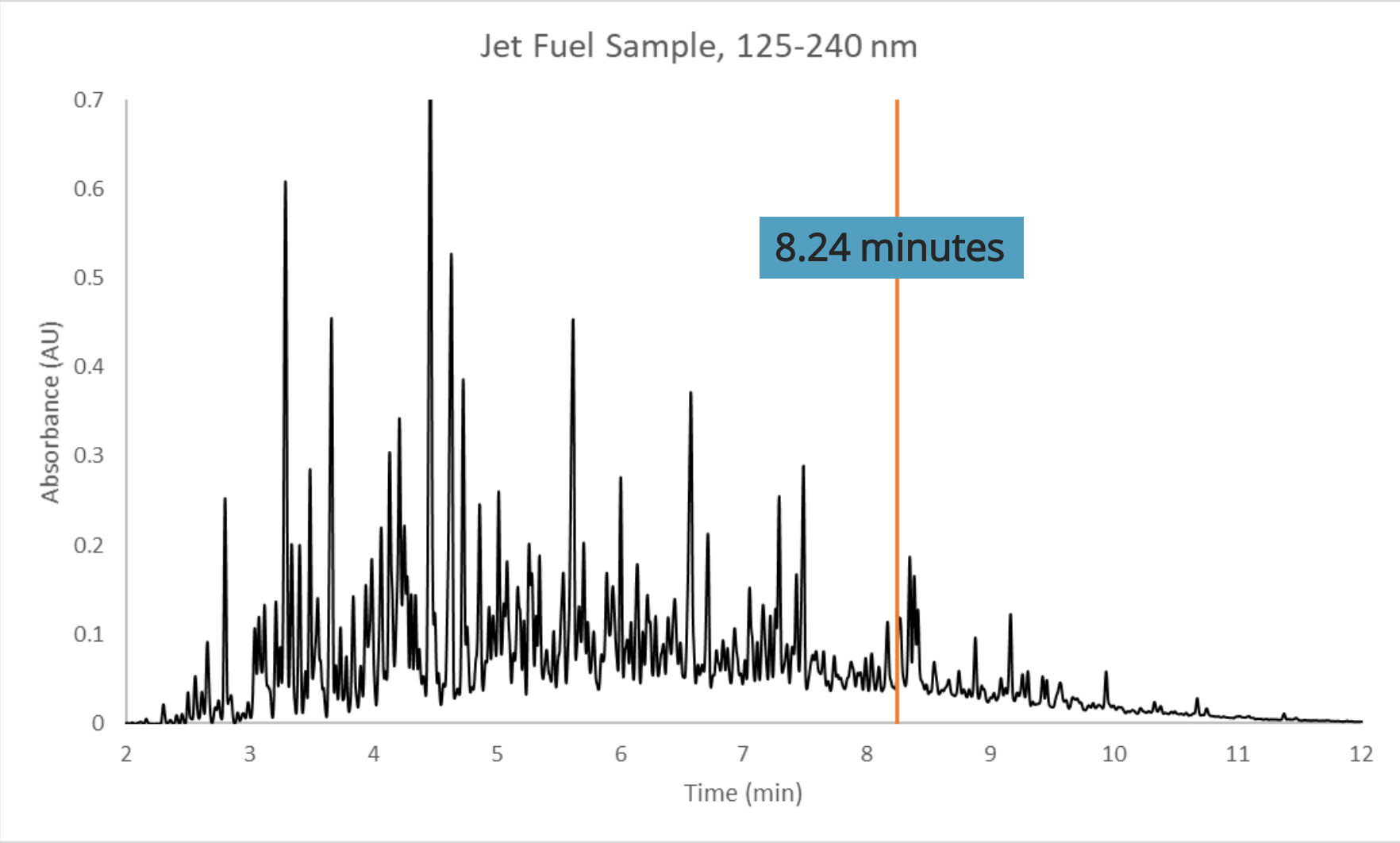
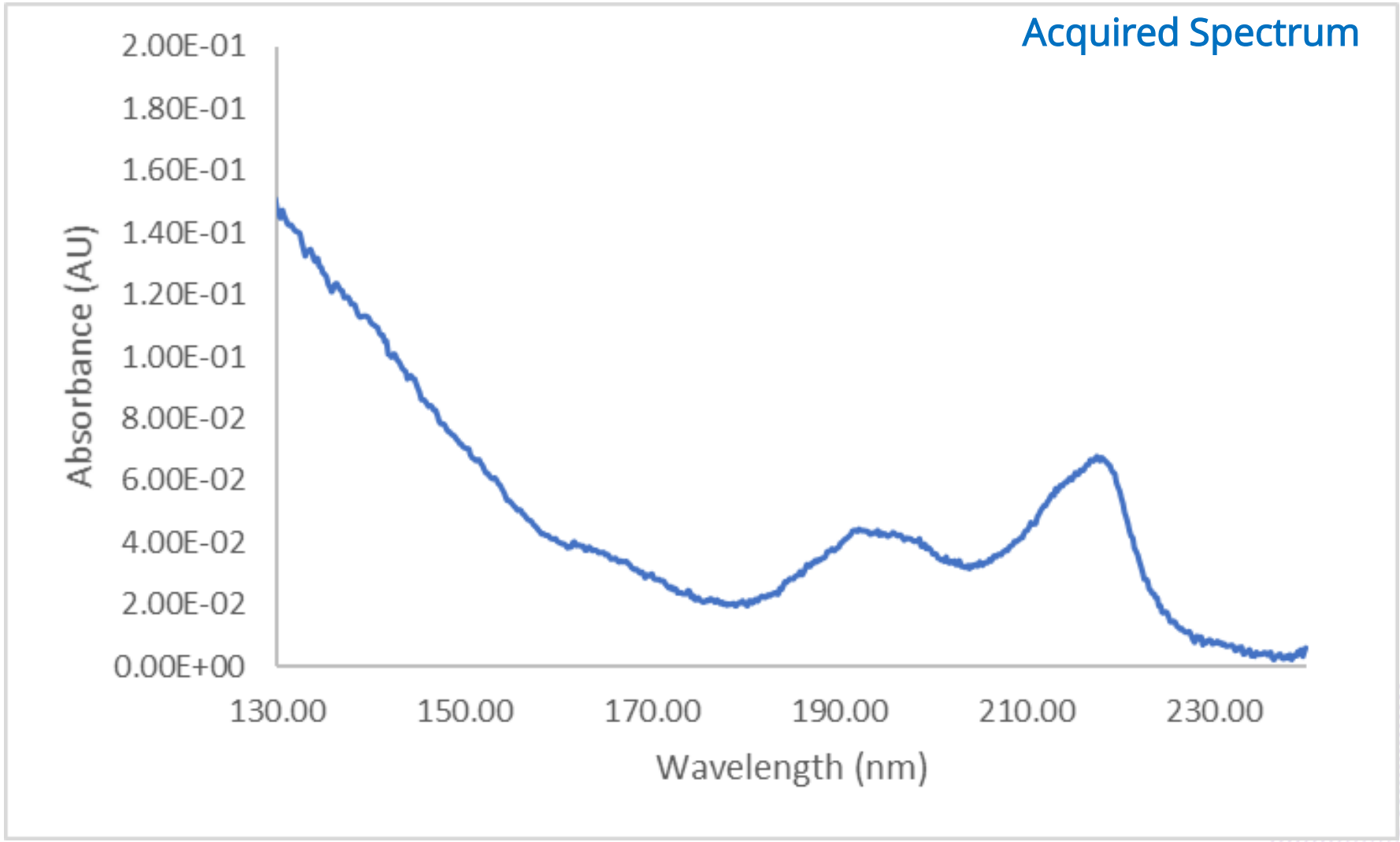


# Vacuum Ultraviolet Spectroscopy

## Spectral Deconvolution



# Vacuum Ultraviolet Spectroscopy

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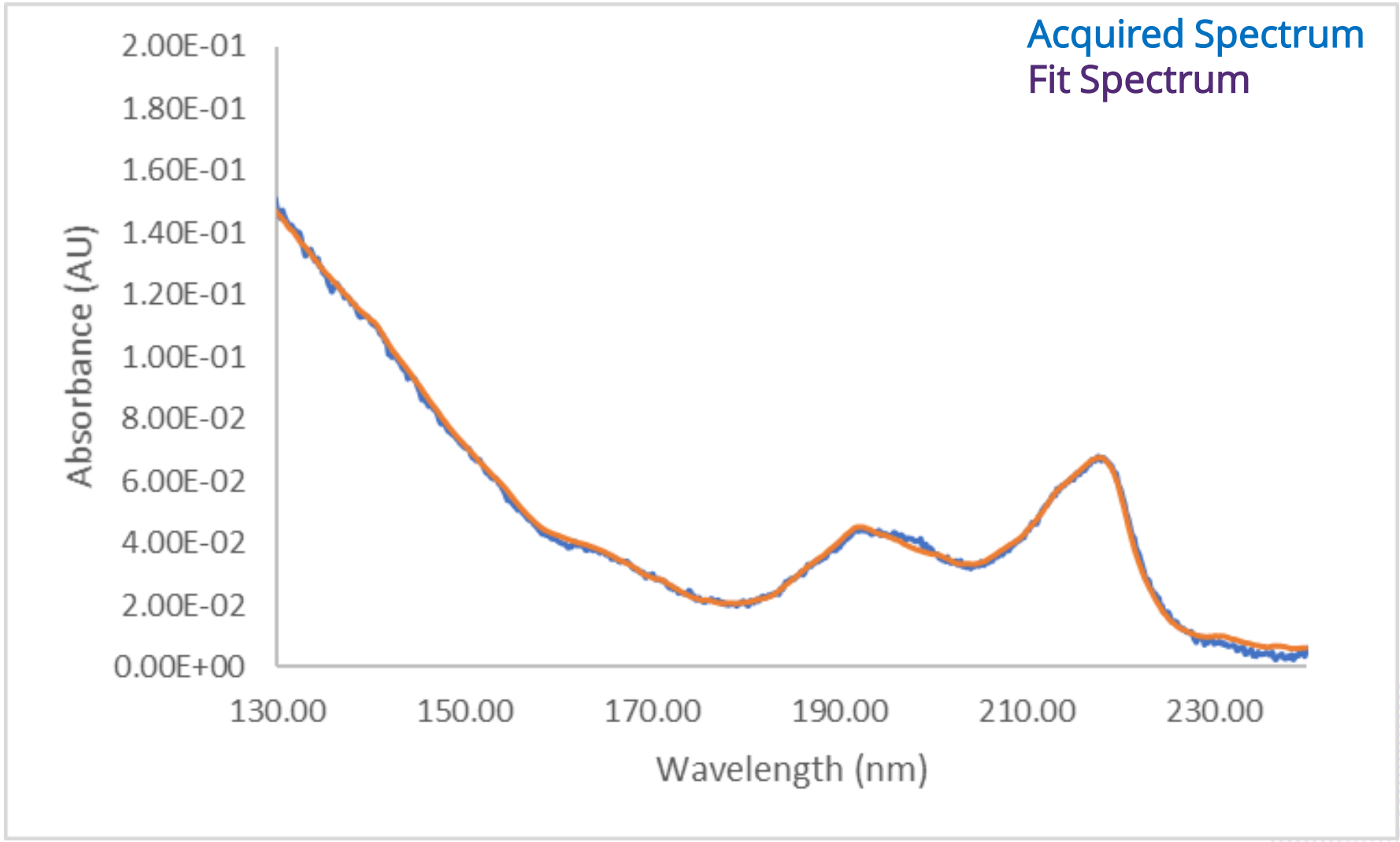
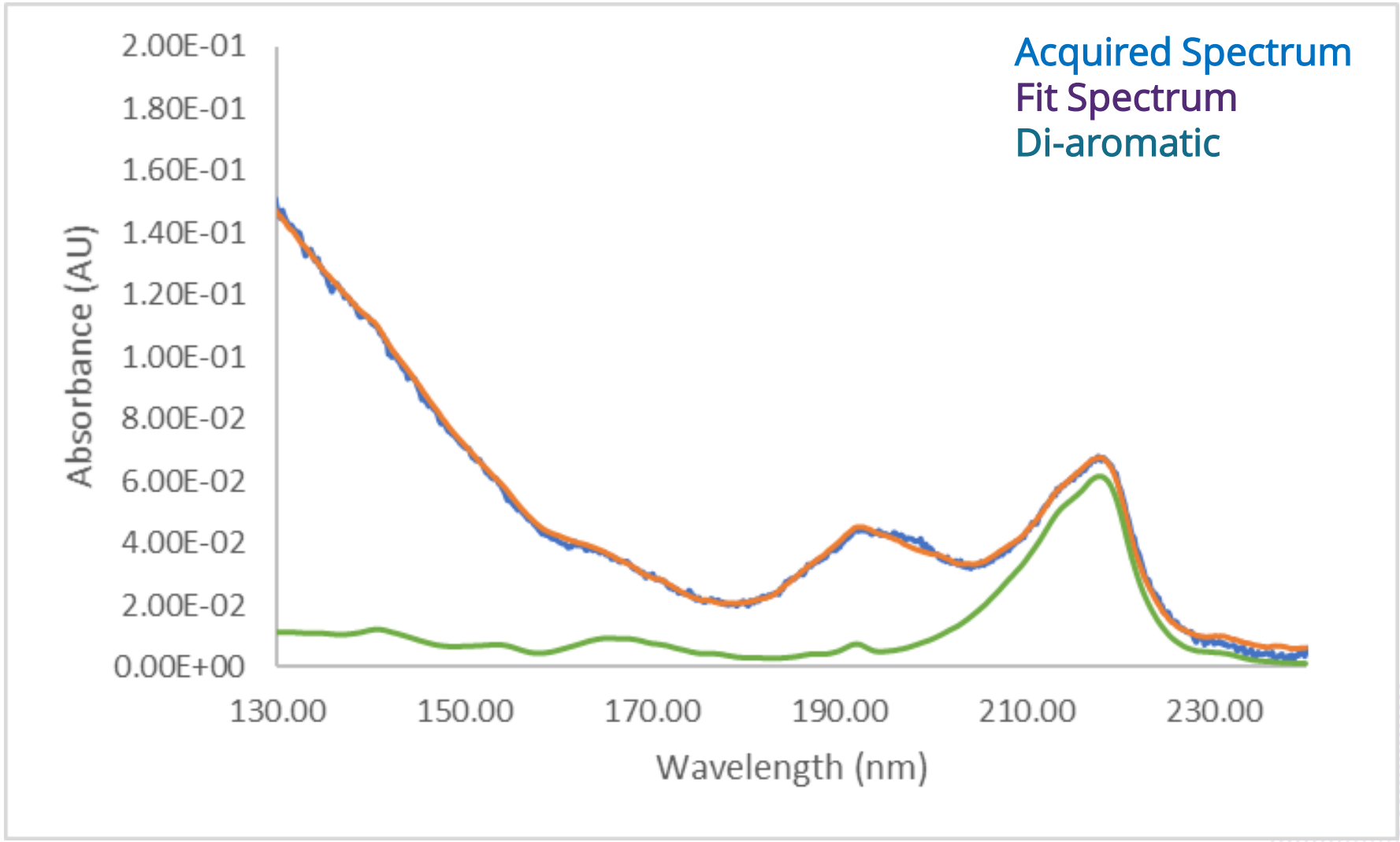


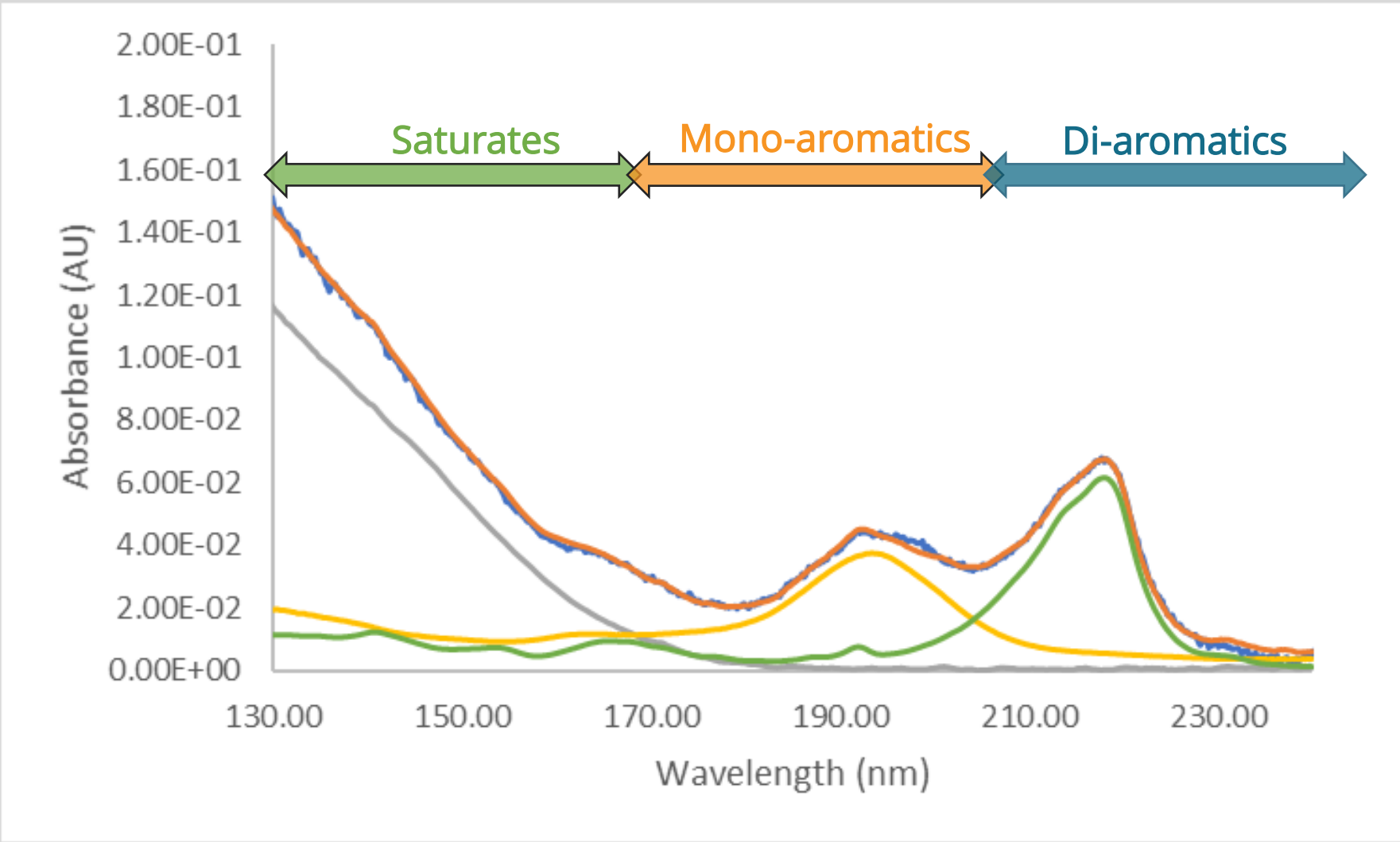
# Vacuum Ultraviolet Spectroscopy

## Spectral Deconvolution



# Vacuum Ultraviolet Spectroscopy

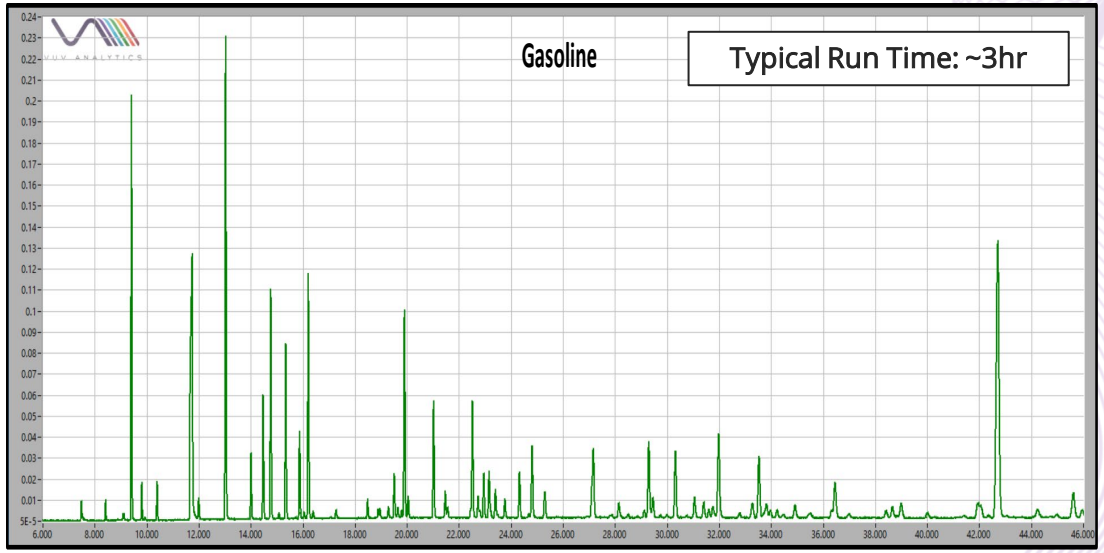
## Spectral Deconvolution



# Why use VUV Spectroscopy?

## Traditional Approaches

- Goal of Chromatography (“separation science”) is to fully separate each compound “peak” in time
- Traditional detectors only offer two dimensions – retention time and amplitude
- Compound identification relies wholly on retention time
- Cannot distinguish between isomers and other coelutions




## The Reality…

- Some samples can be complex and do not fully separate
- In order to get baseline resolution long run times are typical required
- Scientist/human intervention is often required
- Identification errors can and will occur
- Results are reviewed manually

## The Impact…

- Lost Productivity and efficiency (Cost?)
- Inconsistent results from location to location (Impact?)
- Highly dependent on operator skill

# Why VUV Spectroscopy?

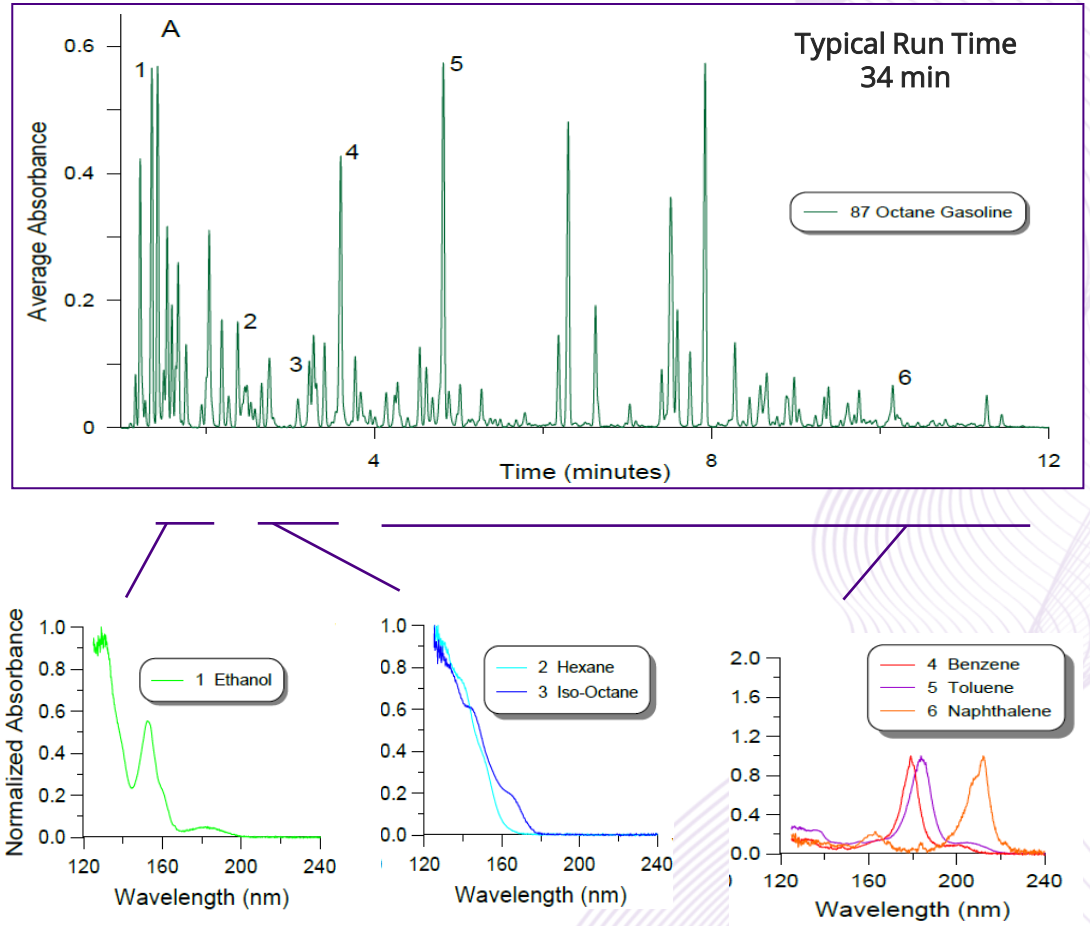
## Why VUV Spectroscopy?

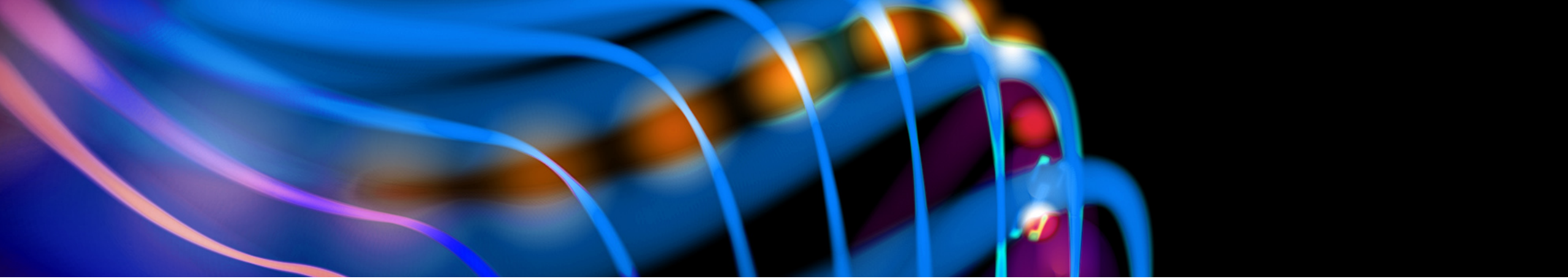
- “Everything” absorbs strongly in VUV (except He, H, Ar)
- Every peak has unique compound-specific absorption response
- Compound classes show like absorption responses, e.g., aromatics
- 3-dimensional (retention time, wavelength, amplitude) and persistent
- Differentiates isomers (highly prevalent among hydrocarbons)

## VUV Spectroscopy Enables …

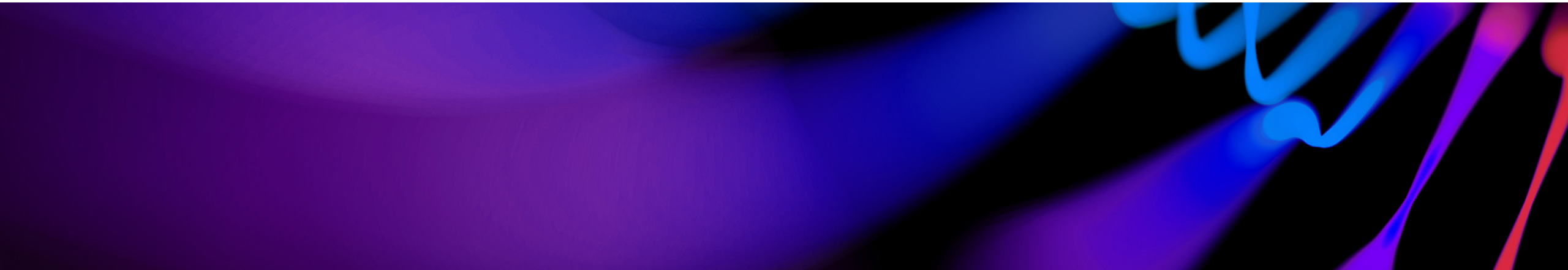
- Shorter run times [3x – 20x]
- Full automation
- Consistently correct results
- Rich and persistent 3D data sets

What would be the impact of generating consistently correct results, in an automated fashion, faster have on your operation?

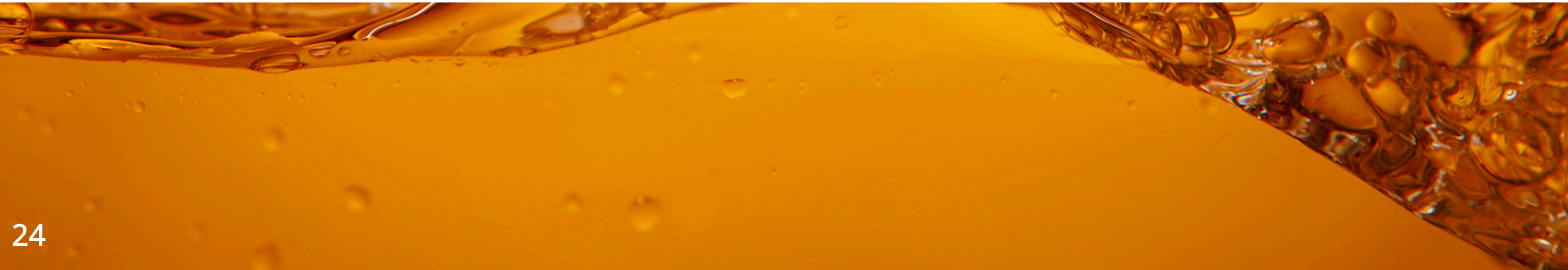




# Application Examples



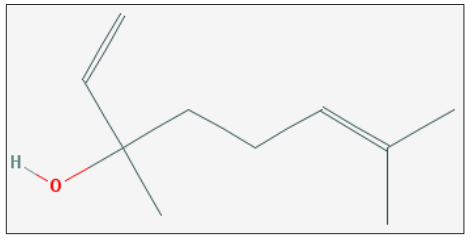
# Terpenes in Cannabis Analysis by GC-VUV



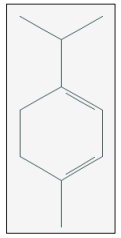
# Cannabis

## Terpenes

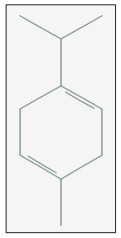
### Monoterpenes and Monoterpenoids



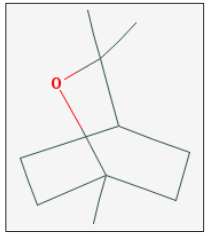

Linalool

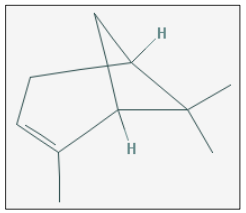


α-Terpinene

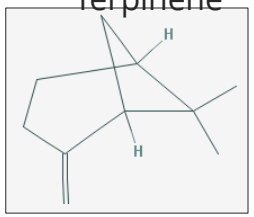


γ-Terpinene



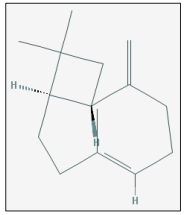




α-Pinene

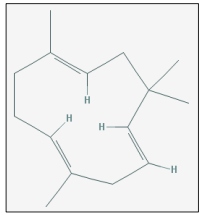



β-Pinene

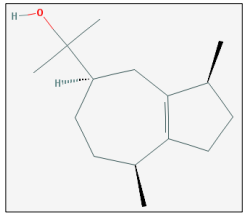
### Sesquiterpenes and Sesquiterpenoids



β-Caryophyllene



α-Humulene

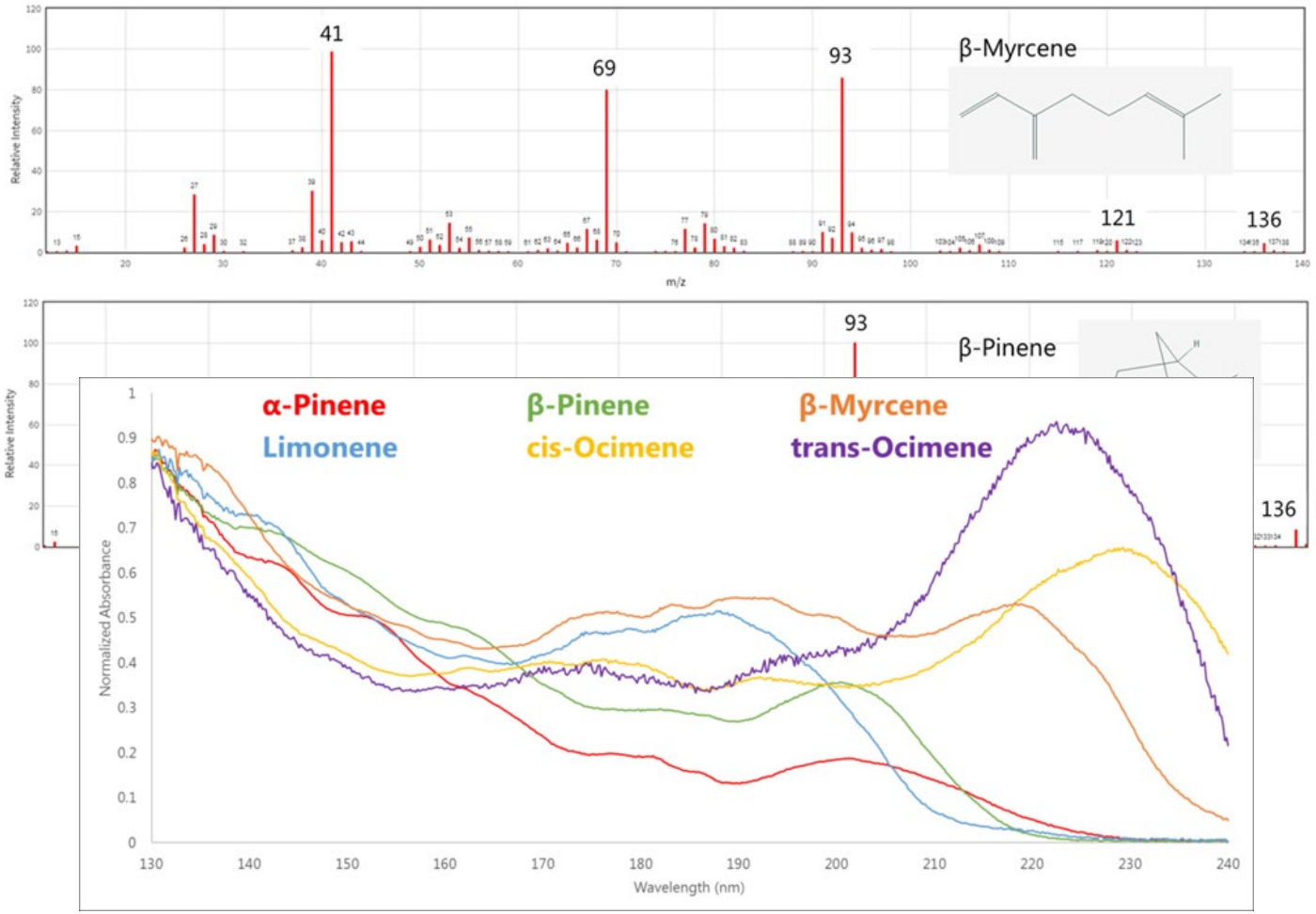



(-)-Guaiol

- Terpene isomers with very different chemical properties can have the same chemical formula.

# Cannabis

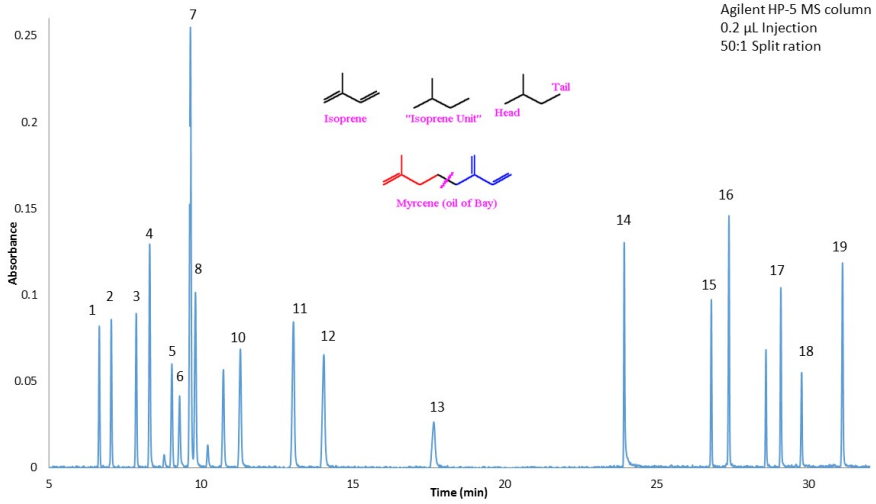
## Terpenes




- Terpene isomers with very different chemical properties can have the same chemical formula.
- Many terpenes have closely related structures that are difficult to differentiate with mass spectrometry.
- The spectral range of GC-VUV addresses these challenges since every terpene isomer has a unique spectral fingerprint.

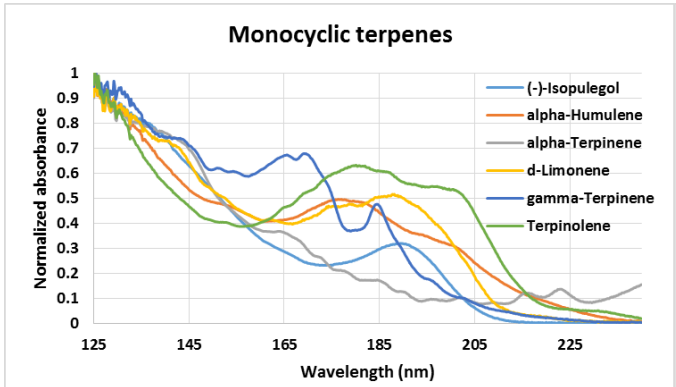
# Cannabis

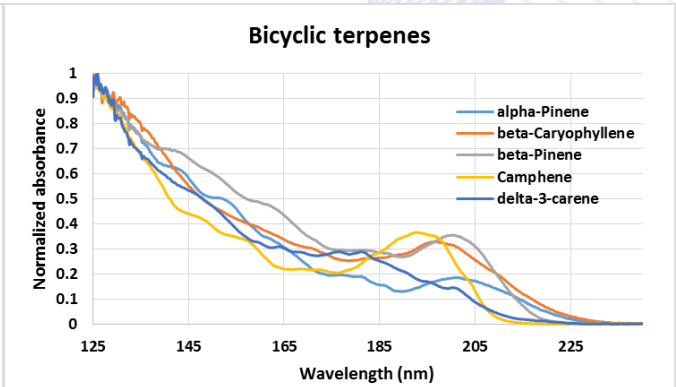
## Terpenes

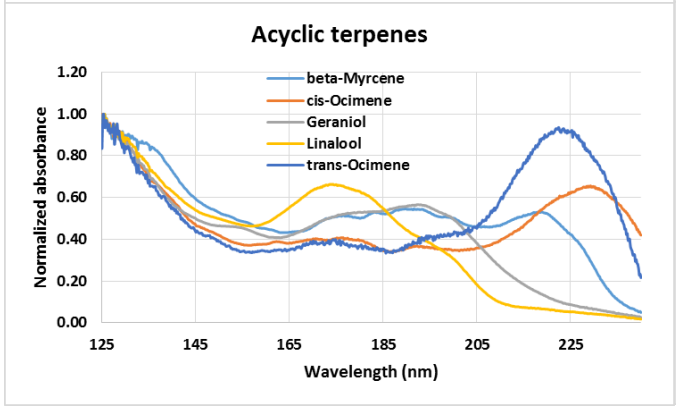


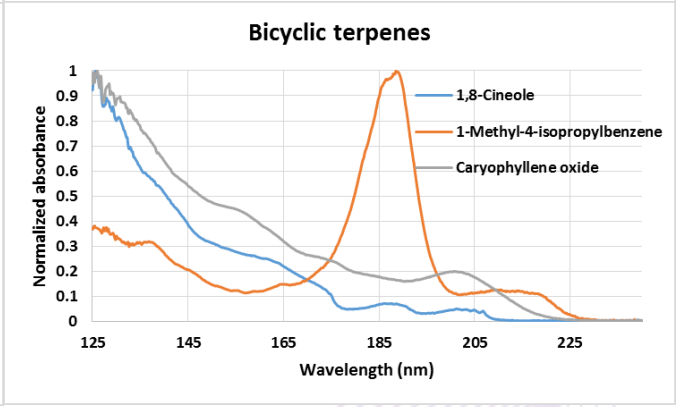

Agilent HP-5 MS column
0.2 µL Injection
50:1 Split ration

| | |
|---|---|
| 1 | Alpha-Pinene |
| 2 | Camphene |
| 3 | (-)-beta-Pinene |
| 4 | Beta-myrcene |
| 5 | Delta-3-careen |
| 6 | Alpha-Terpinene |
| 7 | p-Isopropyltoluene |
| 8 | d-Limonene |
| 9 | Ocimene |
| 10 | Gamma-terpinene |
| 11 | Terpinolene |
| 12 | Linalool |
| 13 | (-)-Isopulegol |
| 14 | Geraniol |
| 15 | Beta-caryophyllene |
| 16 | Alpha-humulene |
| 17 | Nerolidol |
| 18 | (-)-Guaiol |
| 19 | (-)-alpha-Bisabolol |



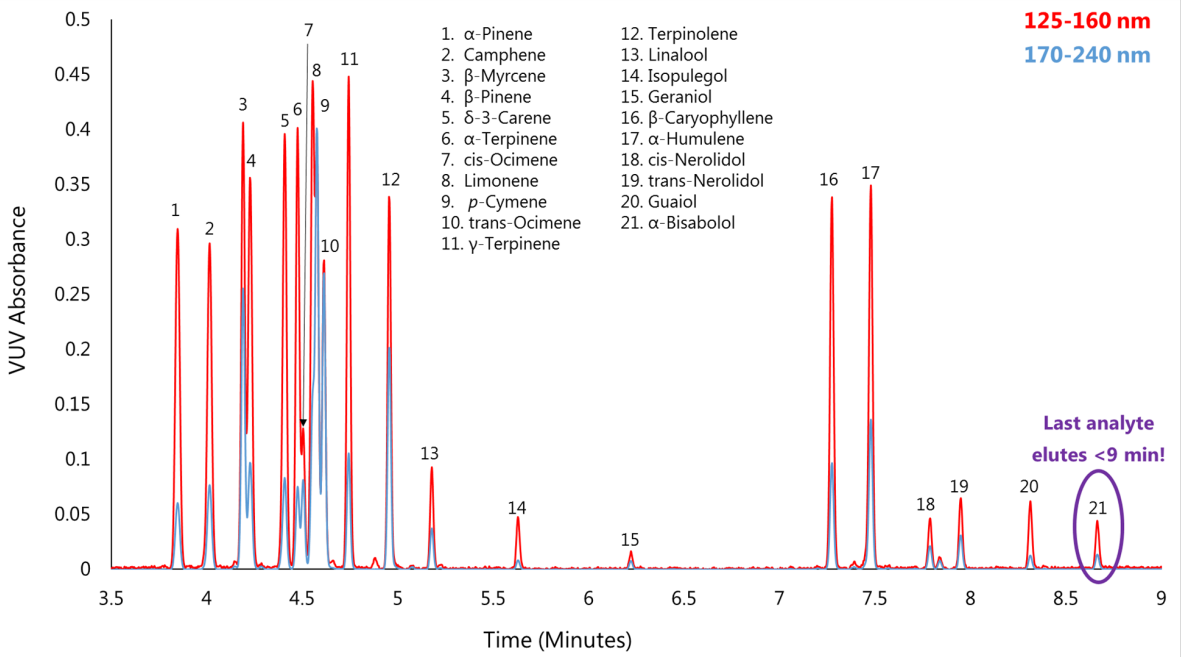




- Terpenes have distinct VUV spectra that provide both qualitative and quantitative information, including structural isomers
- Co-elution of flavor components is easily deconvolved in post-processing
  - Allows GC runtimes to be intentionally shortened

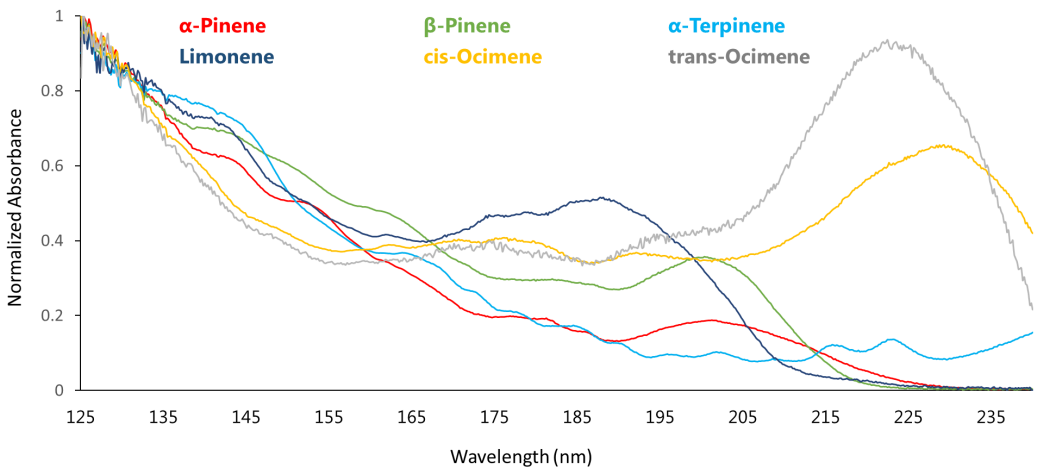
# Cannabis Summary

## Terpenes

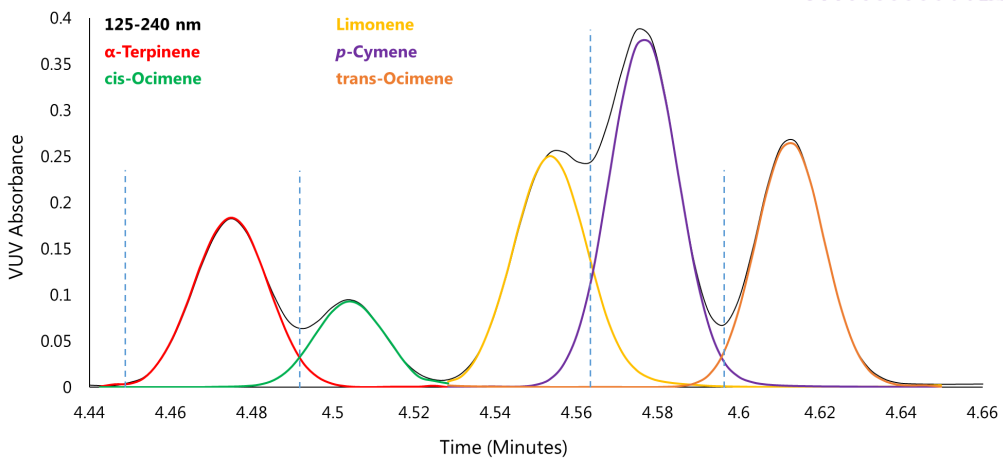
### Fast GC-VUV Chromatogram of Terpenes




### VUV Spectral Identification of Monoterpene Isomers

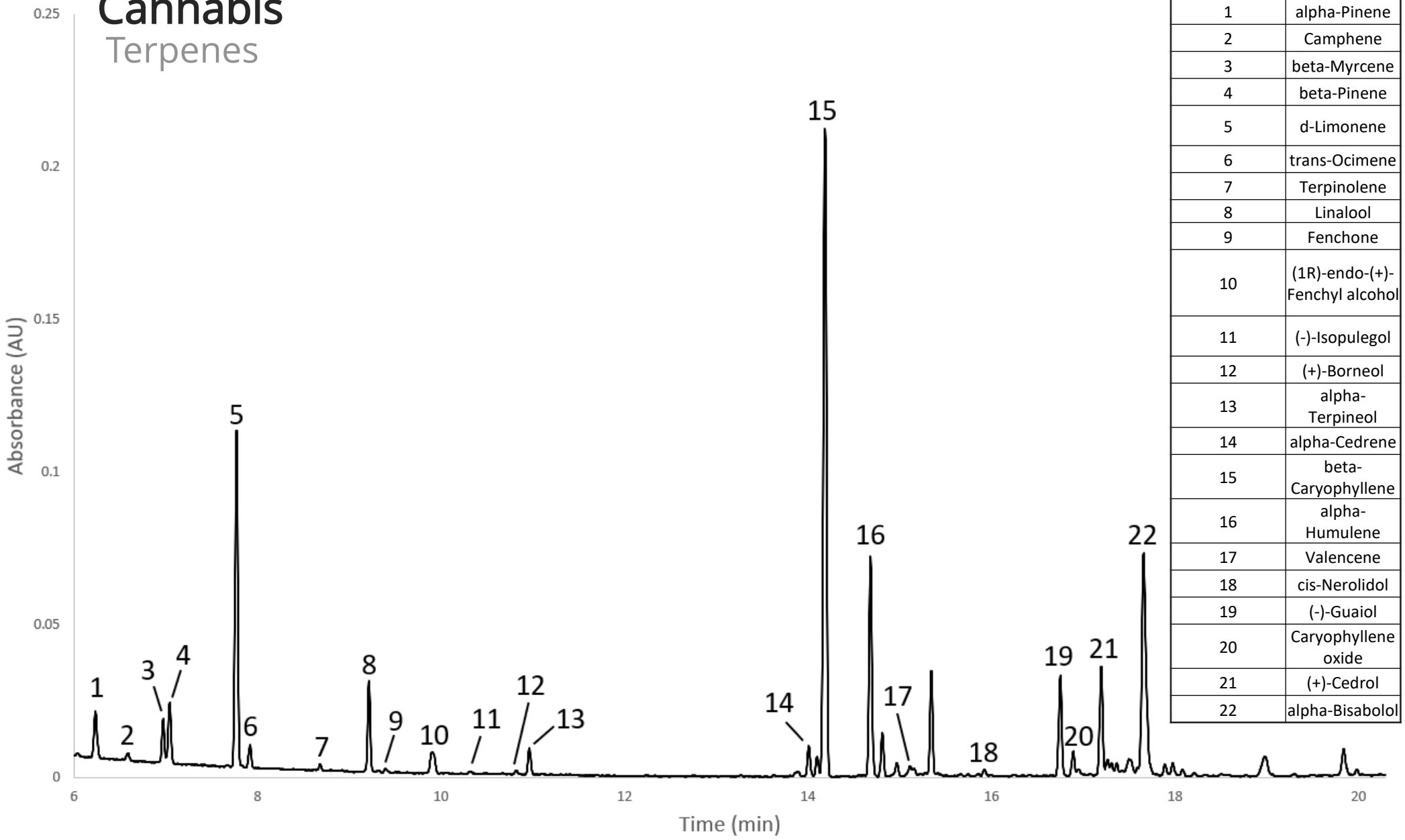



### VUV Deconvolution of Co-Eluting Isomers




- Terpenes have VUV spectra that are distinct
  - Includes structural isomers and co-eluting analytes
- VUV spectral identification of terpene isomers allows the chromatographic compression of GC runtimes
  - Can shorten GC runtimes by 2 – 3X or more
- Natural and forced co-elutions are deconvolved by VUV software
  - Eliminates inaccuracy inherent to dropping vertical integration lines to quantitate

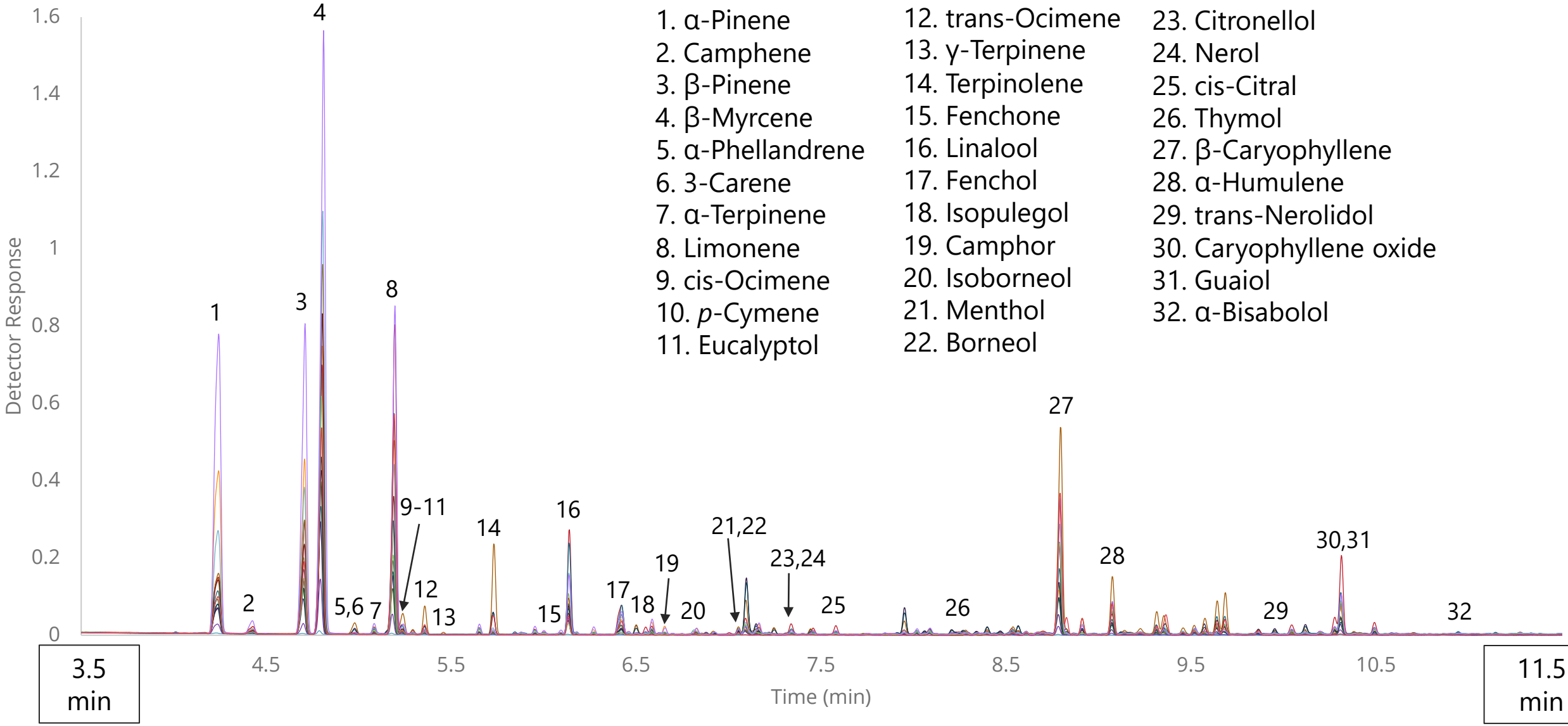
# Cannabis
## Terpenes




| | |
|---|---|
| 1 | alpha-Pinene |
| 2 | Camphene |
| 3 | beta-Myrcene |
| 4 | beta-Pinene |
| 5 | d-Limonene |
| 6 | trans-Ocimene |
| 7 | Terpinolene |
| 8 | Linalool |
| 9 | Fenchone |
| 10 | (1R)-endo-(+)-Fenchyl alcohol |
| 11 | (-)-Isopulegol |
| 12 | (+)-Borneol |
| 13 | alpha-Terpineol |
| 14 | alpha-Cedrene |
| 15 | beta-Caryophyllene |
| 16 | alpha-Humulene |
| 17 | Valencene |
| 18 | cis-Nerolidol |
| 19 | (-)-Guaiol |
| 20 | Caryophyllene oxide |
| 21 | (+)-Cedrol |
| 22 | alpha-Bisabolol |

# Cannabis

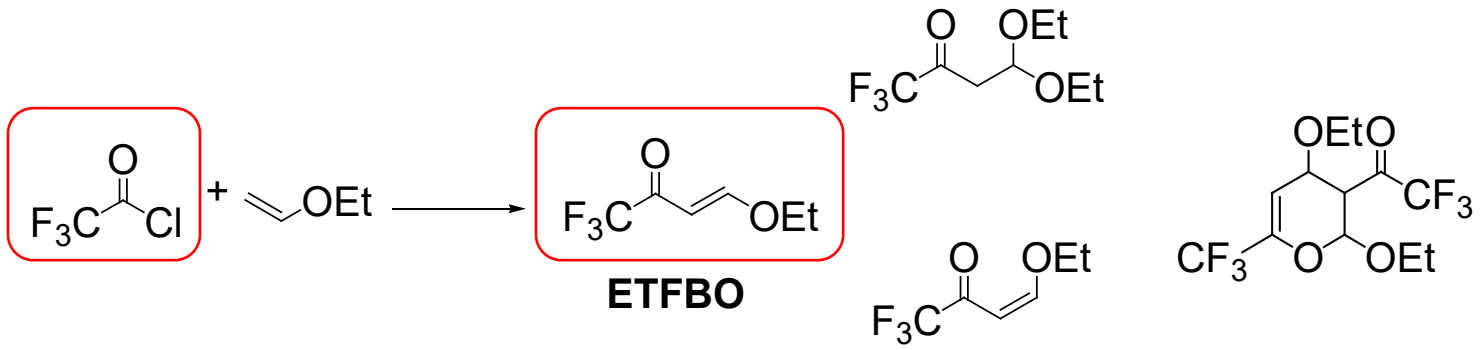
## Overlay of 17 Cannabis Samples (1:100 Dilution in EtOH)



# Analysis of Pharmaceutical Products by GC-VUV

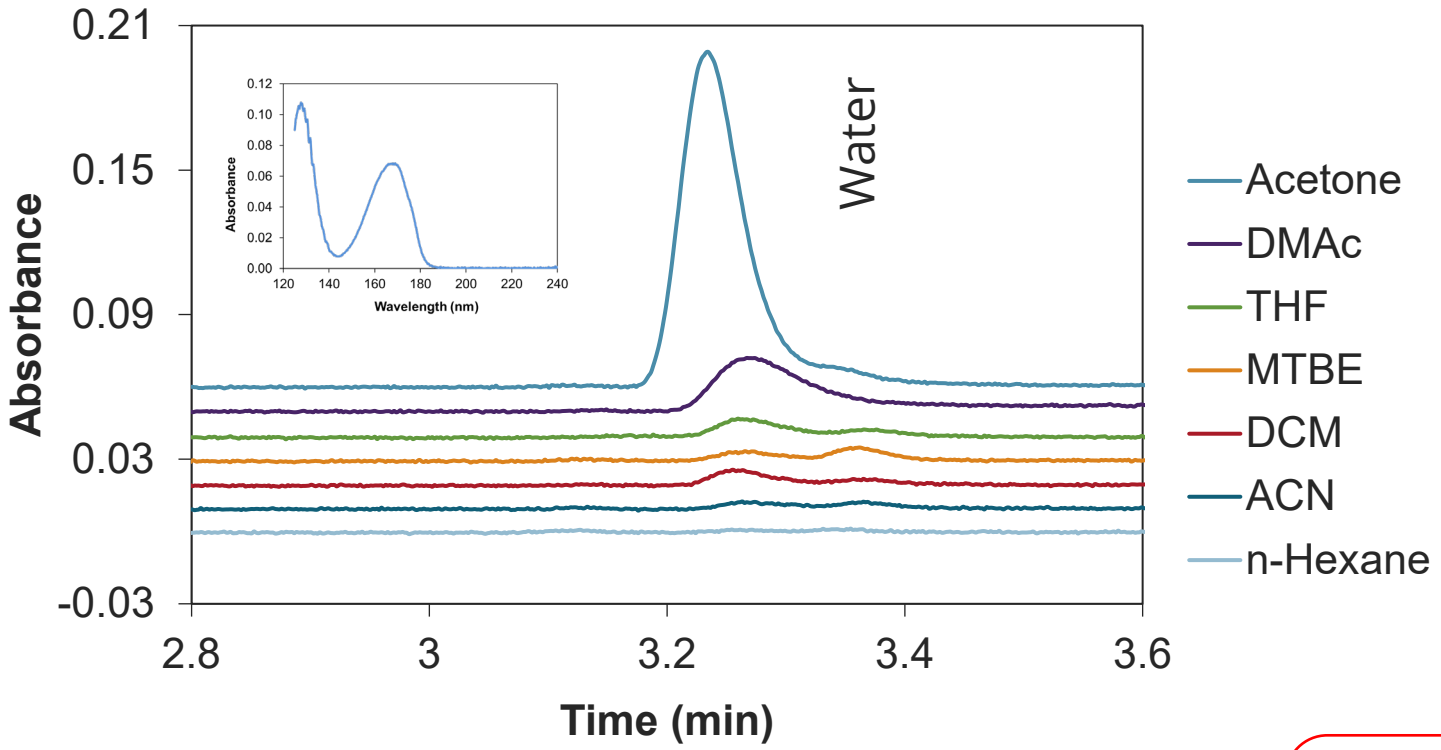
# ETFBO and Related Impurities

Synthesis of ETFBO: 4-ethoxy-1,1,1-trifluoro-3-buten-2-one




- ETFBO is unstable. Large number of unknown impurities/degs
- Trifluoroacetyl chloride does not have C-H bond, and cannot be detected by Flame Ionization Detector (FID)
- Thermal Conductivity Detector (TCD) has to be used, which has much lower sensitivity than FID

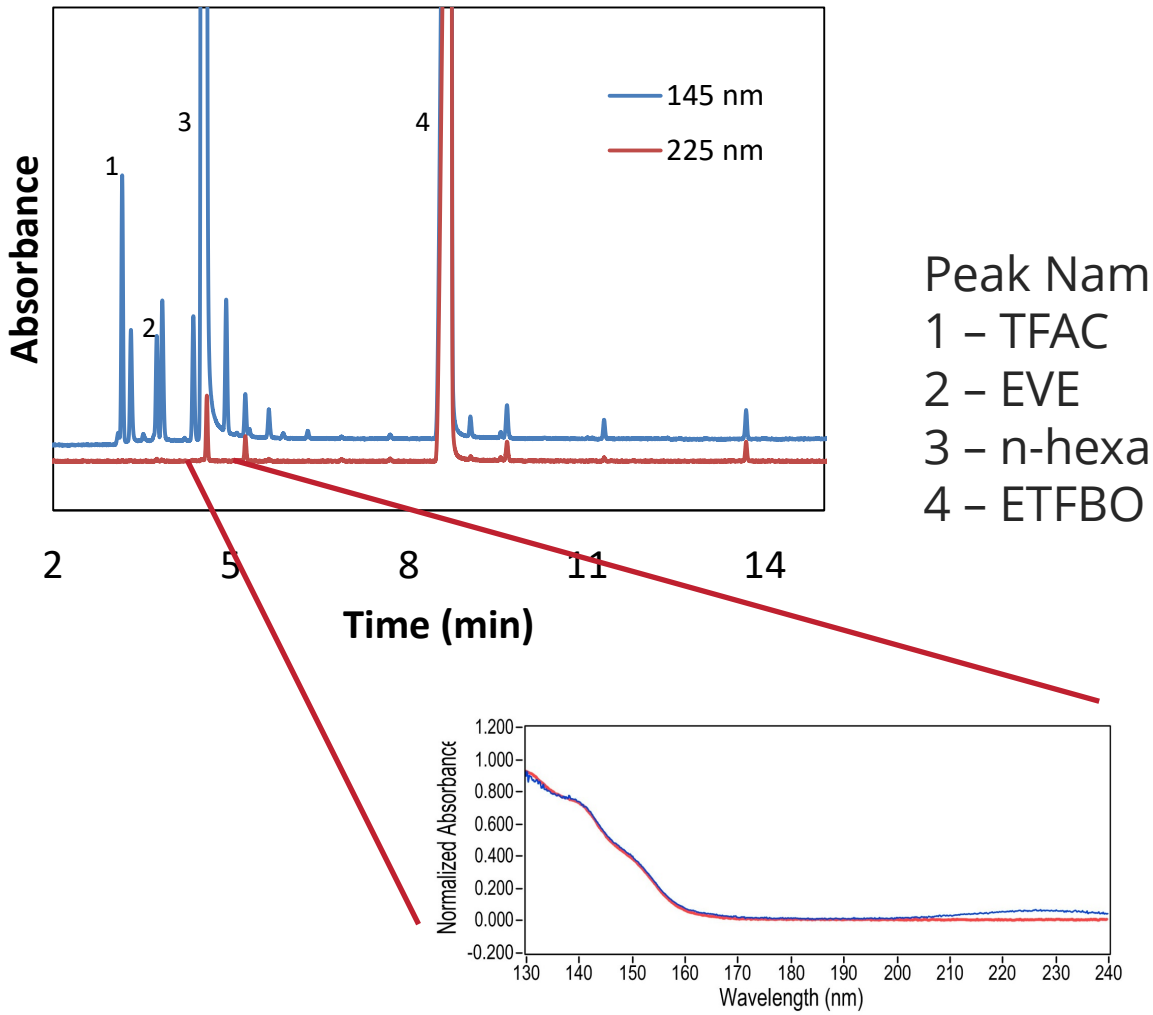
# Water Content in Organic Solvents




| Solvent Name | Acetone | DMAc | THF | DCM | MTBE | ACN | n-Hexane |
|---|---|---|---|---|---|---|---|
| Water contents (ppm) | 506 | 102 | 32 | 24 | 16 | 11 | 5 |

Hexane is used as diluent as it has the lowest water content

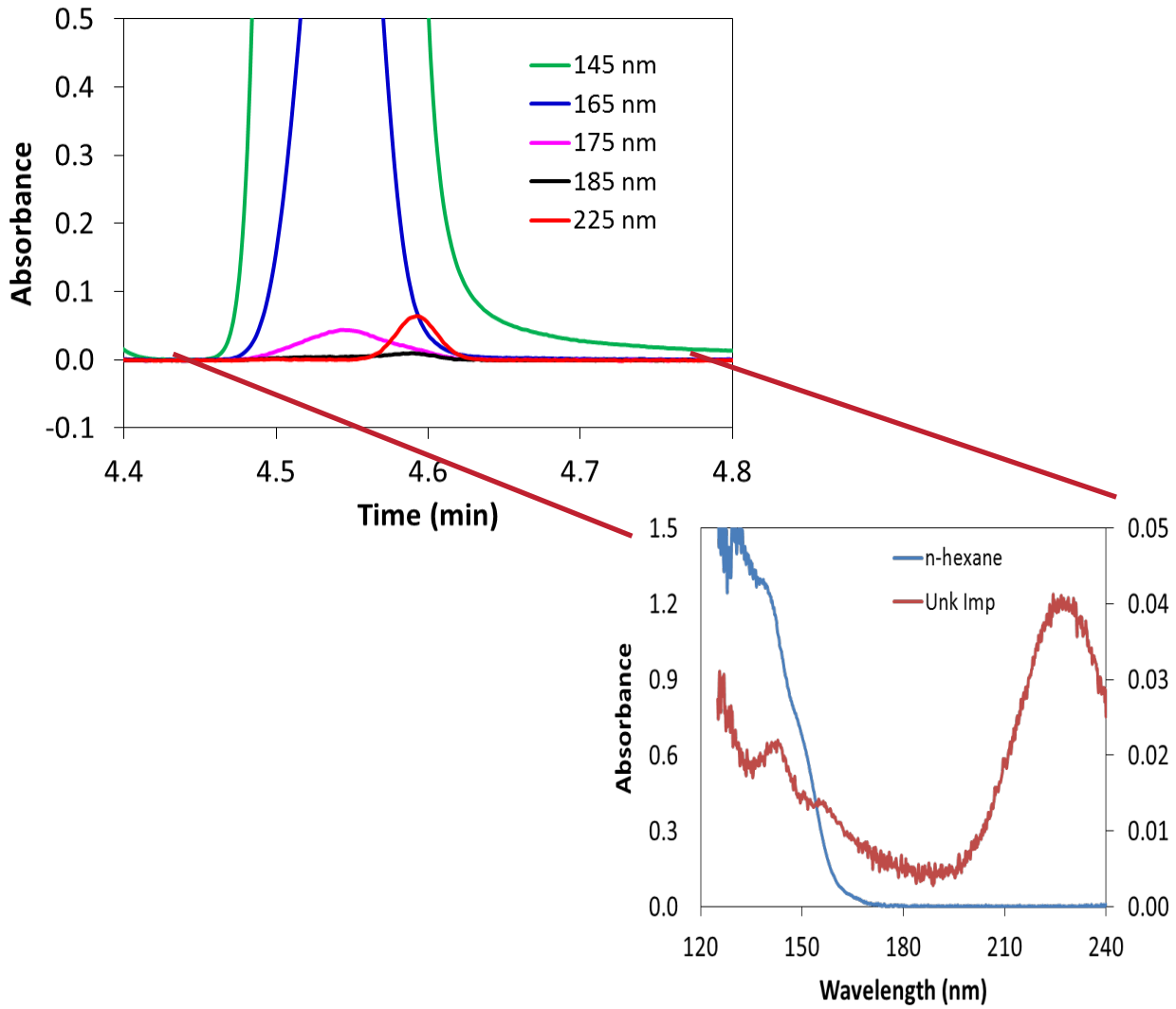
# Pharmaceuticals




Peak Name:
1 – TFAC
2 – EVE
3 – n-hexane
4 – ETFBO

- Several useful applications have been demonstrated in the pharmaceutical industry
- Applications include selective detection of compounds and peak purity assessment
- GC-VUV can detect virtually all volatile organic compounds, making it a powerful tool for routine analysis.

# Pharmaceuticals

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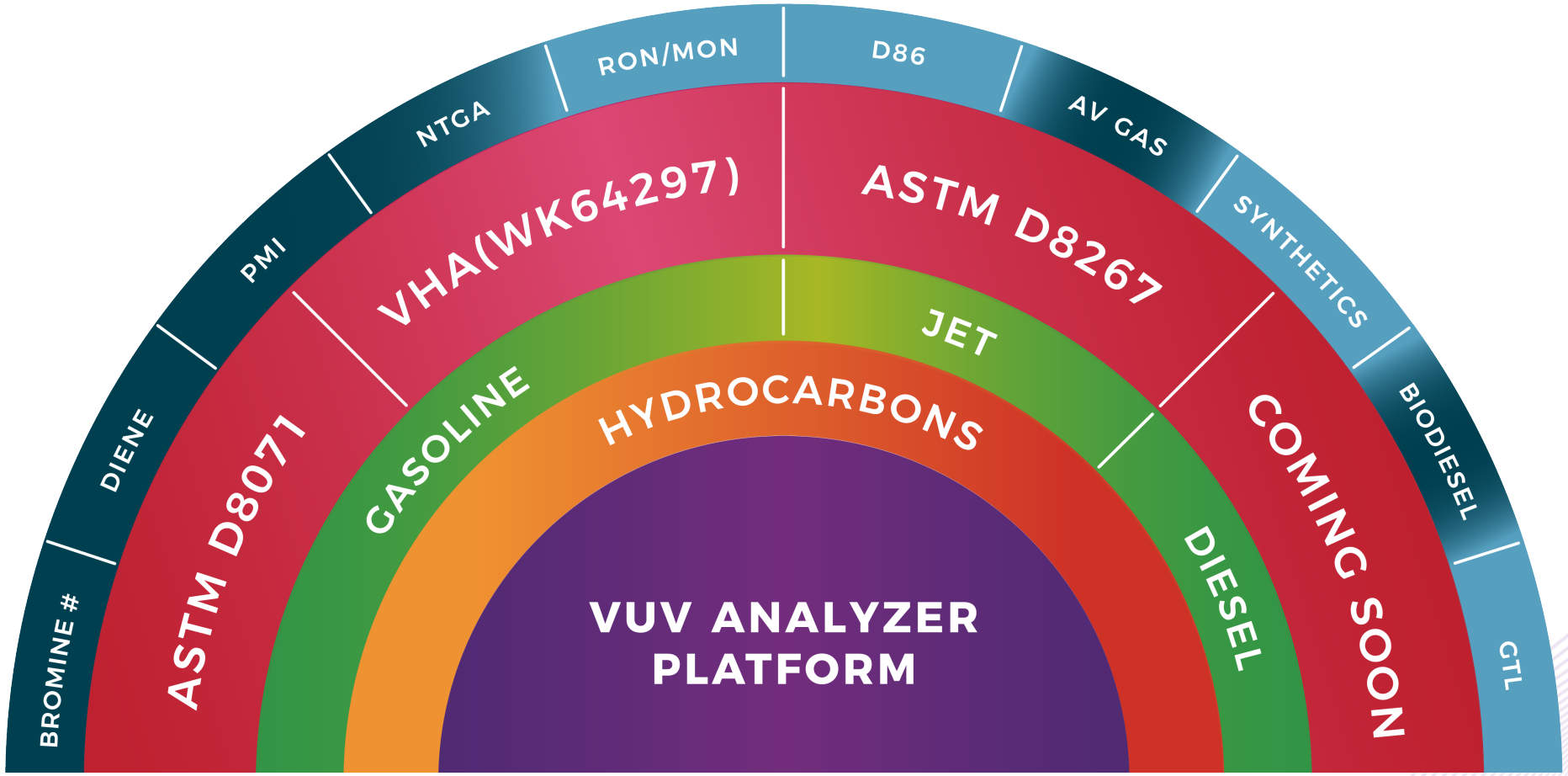
# VUV Analyzer™ Platform for Fuels

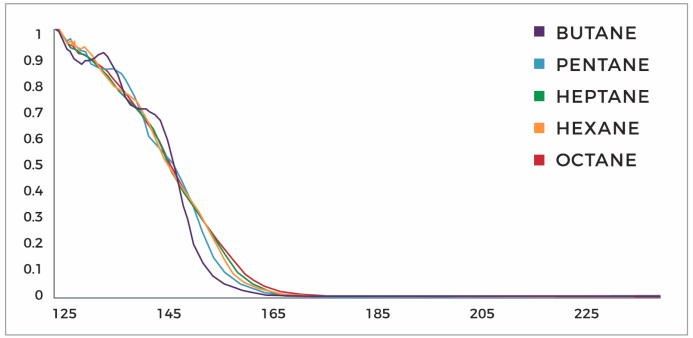
# VUV Analyzer™ Platform for Fuels



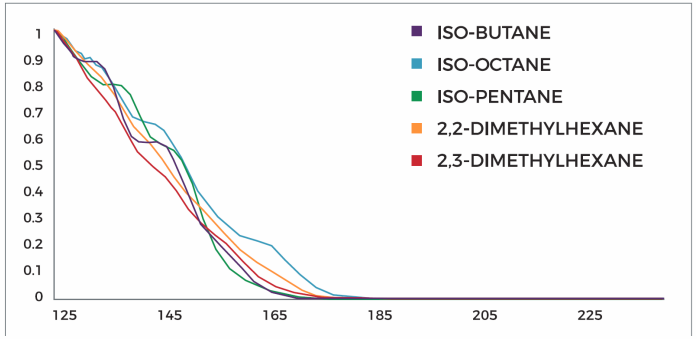
# Fuels Analysis

## Gasoline, Jet Fuel, Diesel

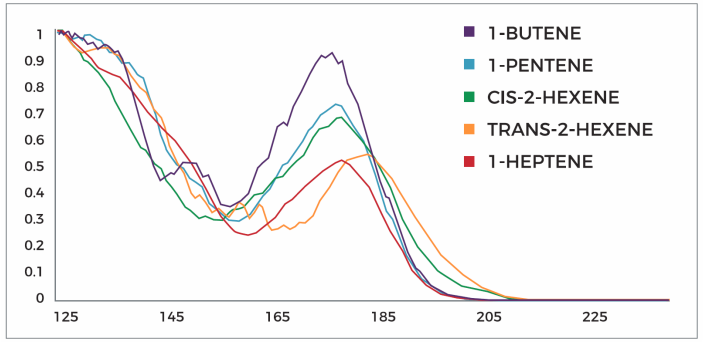
PARAFFINS



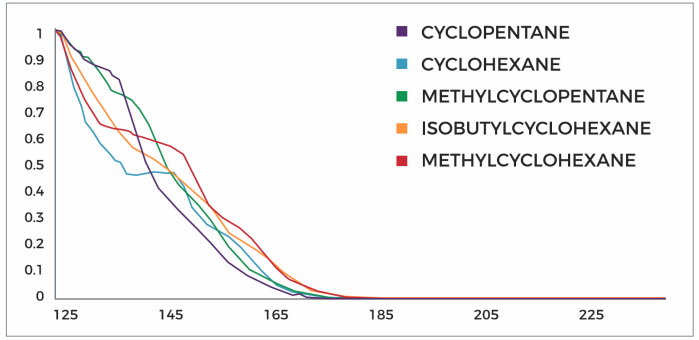

ISOPARAFFINS



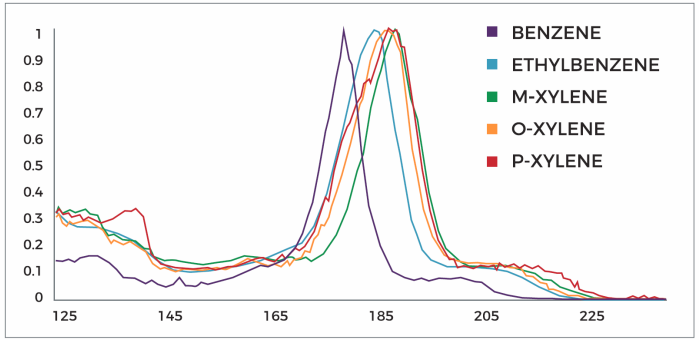

OLEFINS




NAPHTHENES




AROMATICS




- Analysis of complex fuel mixtures are simplified by leveraging the VUV spectral information collected with GC-VUV
- The VUV spectra of hydrocarbons exhibit class-based similarities.
- This enables straightforward compound class identification and quantitation, even for samples containing hundreds of compounds.

# CASE STUDY

A major U.S. refinery suspected that their 2N+A yield was low resulting in lower market prices for heavy naphtha

## Closed Revenue Leak ($8 - $16 million lost)

# Summary

- VUV Analytics has produced the world's first benchtop vacuum ultraviolet detector

- The power of GC-VUV technology arises from the spectral range in which it operates, 125 – 240 nm
  - Allows for exact spectral matching
  - Isomer identification
  - High sensitivity for gas phase analytes

- GC-VUV technology has found success in both routine and R&D laboratories in many global markets

# QUESTIONS???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