Verified Hydrocarbon Analysis™ (VHA™): Automating Complex Hydrocarbon Analysis on the VUV Analyzer Platform (GC-VUV)

Alex Hodgson, Applications Manager

VUV Analytics, Inc

Overview

- Who is VUV Analytics?
- Detailed Hydrocarbon Analysis (DHA)
- Verified Hydrocarbon Analysis™ (VHA)
    - Fast
    - Easy to use
    - More accurate results
- Case Study
- VUV Analyzer™ Platform for Fuels
- Summary

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.

OIL & GAS CUSTOMERS

The VUV Analyzer(TM) for Fuels continues to grow its customer base on a daily basis:
9 out of the top 13 Oil & Gas Companies
7 out of the top 10 Fuel Refineries
10+ Major Chemical Companies

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.

Detailed Hydrocarbon Analysis

Detailed Hydrocarbon Analysis

Traditional Approaches

DHA Methods						
Method	D6729	D6730	D6733	Fast DHA	D7900	D5134
Sample Scope	Spark-ignition fuels Oxygenate blends				Straight naphthas Reformate Alkylate Crude oil (No Gasolines and FCC Naphtha)	
Column (m)	100m	100m + 5m	50m	40m	50m	
Conc. Range (m/m)	0.01 - 30 %	0.01 - 30 %	0.01 - 15 %	0.01 - 30 %	0.01 - 30 %	
Max Olefin Conc.	25%	25%	20%	20%	20%	
Max FBP	225 ˚C	225 ˚C	225 ˚C	225 ˚C	270 ˚C	
Analysis Time (min)	140	174	135 - 162	28	118	

Detailed Hydrocarbon Analysis

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Max FBP	225 ˚C	225 ˚C	225 ˚C	225 ˚C	270 ˚C	
Analysis Time (min)	140	174	135 - 162	28	118	

No separation of vital oxygenates and toluene

Detailed Hydrocarbon Analysis

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Max FBP	225 ˚C	225 ˚C	225 ˚C	225 ˚C	270 ˚C	
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No 1-methylnaphthalene / tridecane separation

Detailed Hydrocarbon Analysis

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Analysis Time (min)	140	174	135 - 162	28	118	

Requires other methods for benzene, toluene, and oxygenate analysis

Detailed Hydrocarbon Analysis

Traditional Approaches

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Max FBP	225 ˚C	225 ˚C	225 ˚C	225 ˚C	270 ˚C	
Analysis Time (min)	140	174	135 - 162	28	118	

Only major peaks are identified after toluene

Detailed Hydrocarbon Analysis

Traditional Approaches

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No separation of oxygenates

Detailed Hydrocarbon Analysis

Traditional Approaches

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Max Olefin Conc.	25%	25%	20%	20%	20%	
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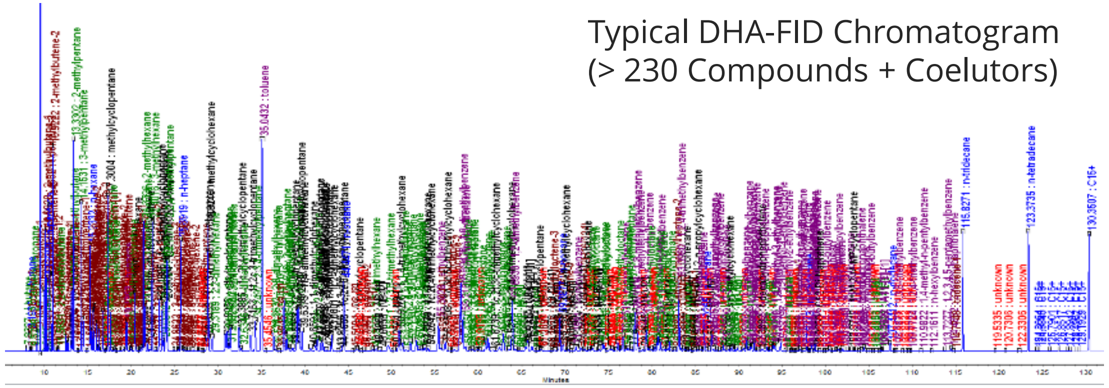
Detailed Hydrocarbon Analysis

Traditional Approaches

DHA Methods	
Method	D6730
Sample Scope	Spark-ignition fuels Oxygenate blends
Column (m)	100m + 5m
Conc. Range (m/m)	0.01 - 30 %
Max Olefin Conc.	25%
Max FBP	225 ˚C
Analysis Time (min)	174



Typical DHA-FID Chromatogram
(> 230 Compounds + Coelutors)

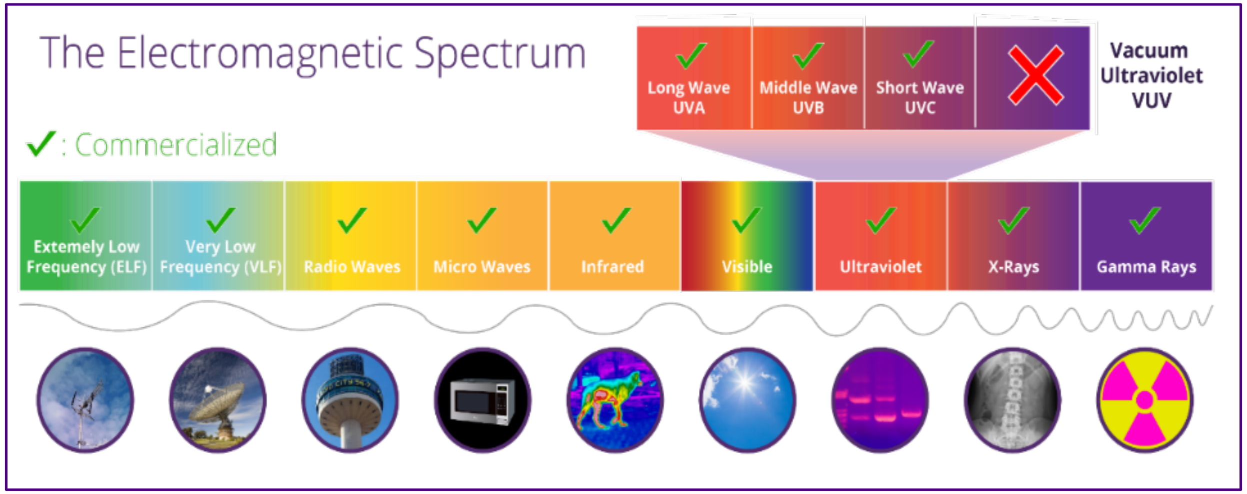
Source: Envantage

- **Uses Gas Chromatography (GC) with a Flame Ionization Detector (FID)**
    - GC provides high resolution separation of individual hydrocarbon components
    - FID (mass sensitive) detection is used to determine quantity of each component
- **Peak Identification is a combination of relative retention time databases and manual corrections**
- **Speciation of compounds in gasoline-range fuels, though not all compounds are identified by name.**
- **Potential areas of improvement**
    - Faster
    - Easier
    - More Accurate

Introduction to GC-VUV Spectroscopy

What is Vacuum Ultraviolet (VUV) Spectroscopy?

It's 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_2)
- Compounds that absorb in this region have unique spectral fingerprints

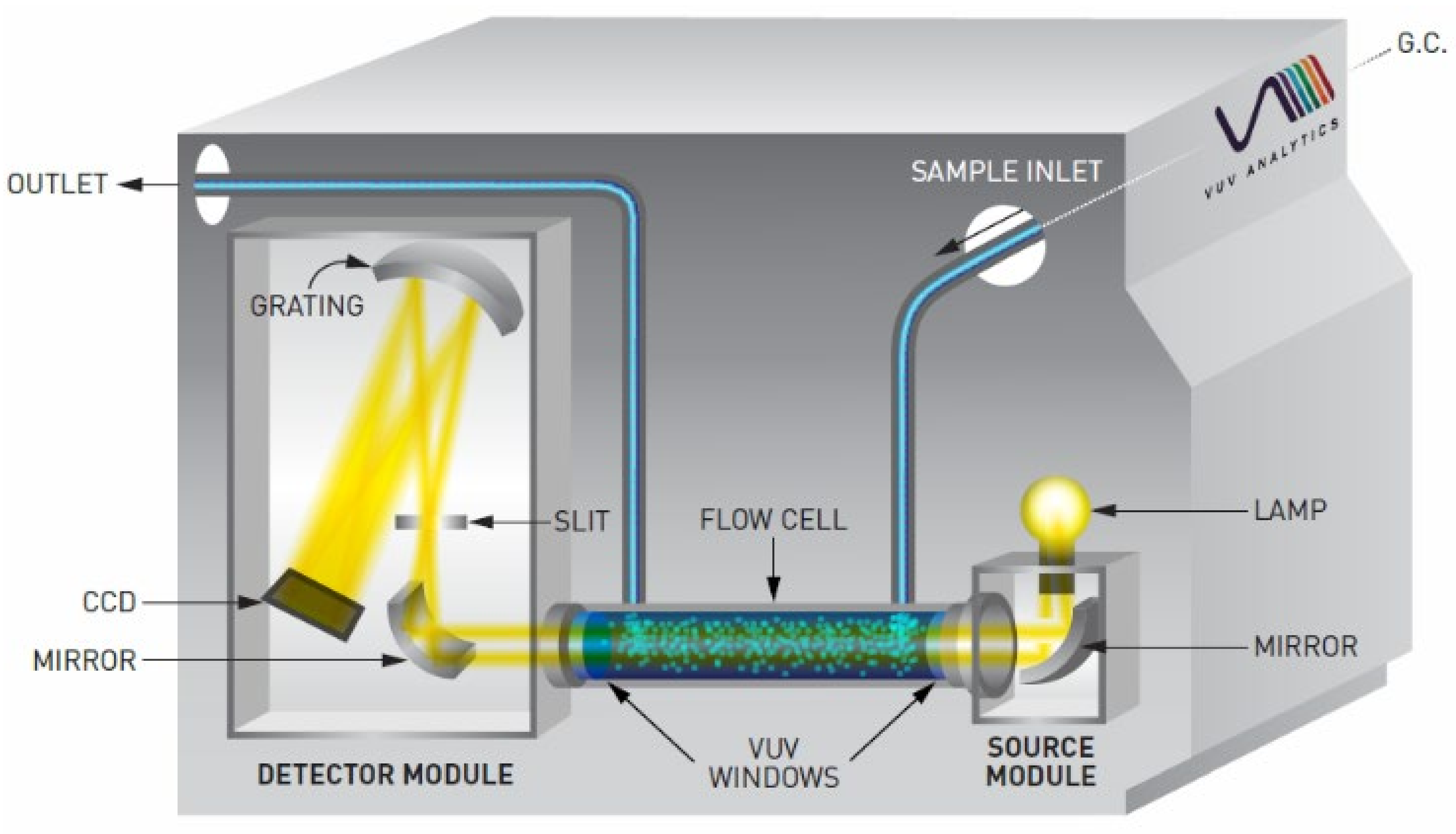
What is Vacuum Ultraviolet (VUV) Spectroscopy?

The 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

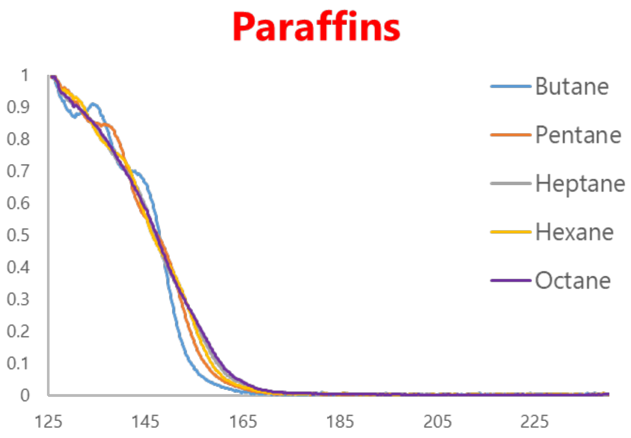
	VGA-100	VGA-101
Max Temp	300°C	430°C
λ Range	125 - 240 nm	125 - 430 nm
Acq Rate	75 Hz	75 Hz

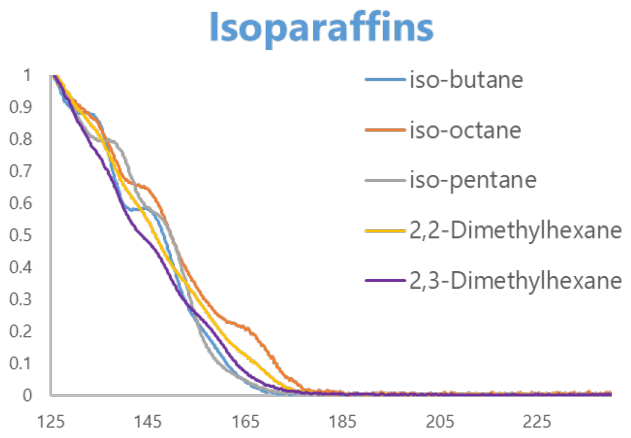
How does a VGA detector work?

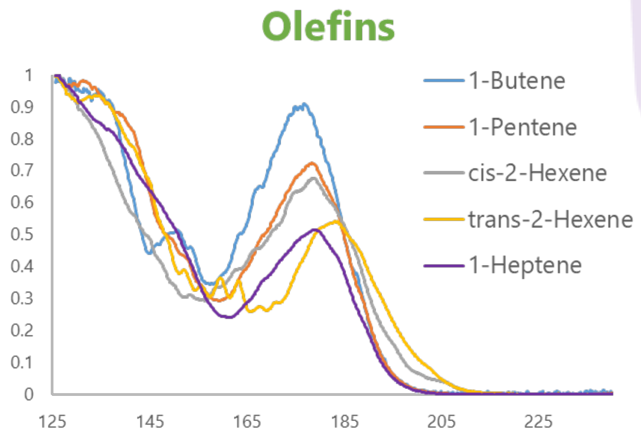


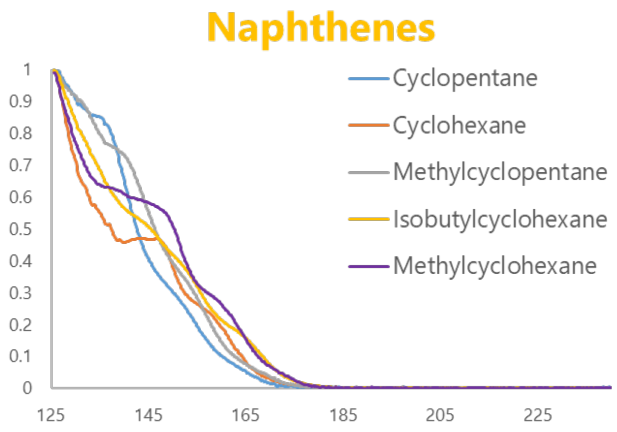
- GC analytes are passed to the VGA detector and excited by deuterium lamp
- Electronic transitions create unique spectral fingerprints
- Data is acquired in 3D (time, absorbance and wavelength) in VUVision™ Software for analysis
- Data is both qualitative and quantitative

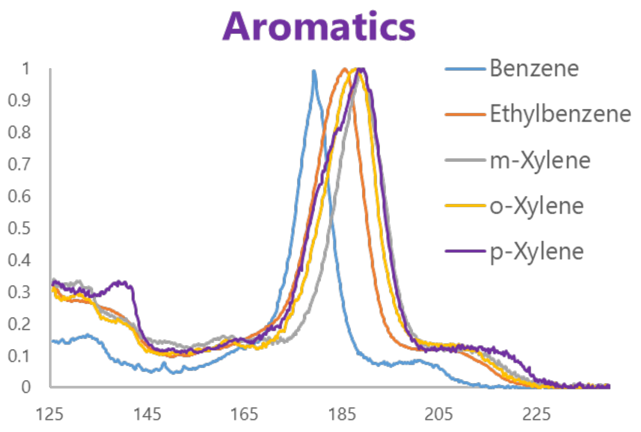
Spectra Demonstrate Class-Based Similarities



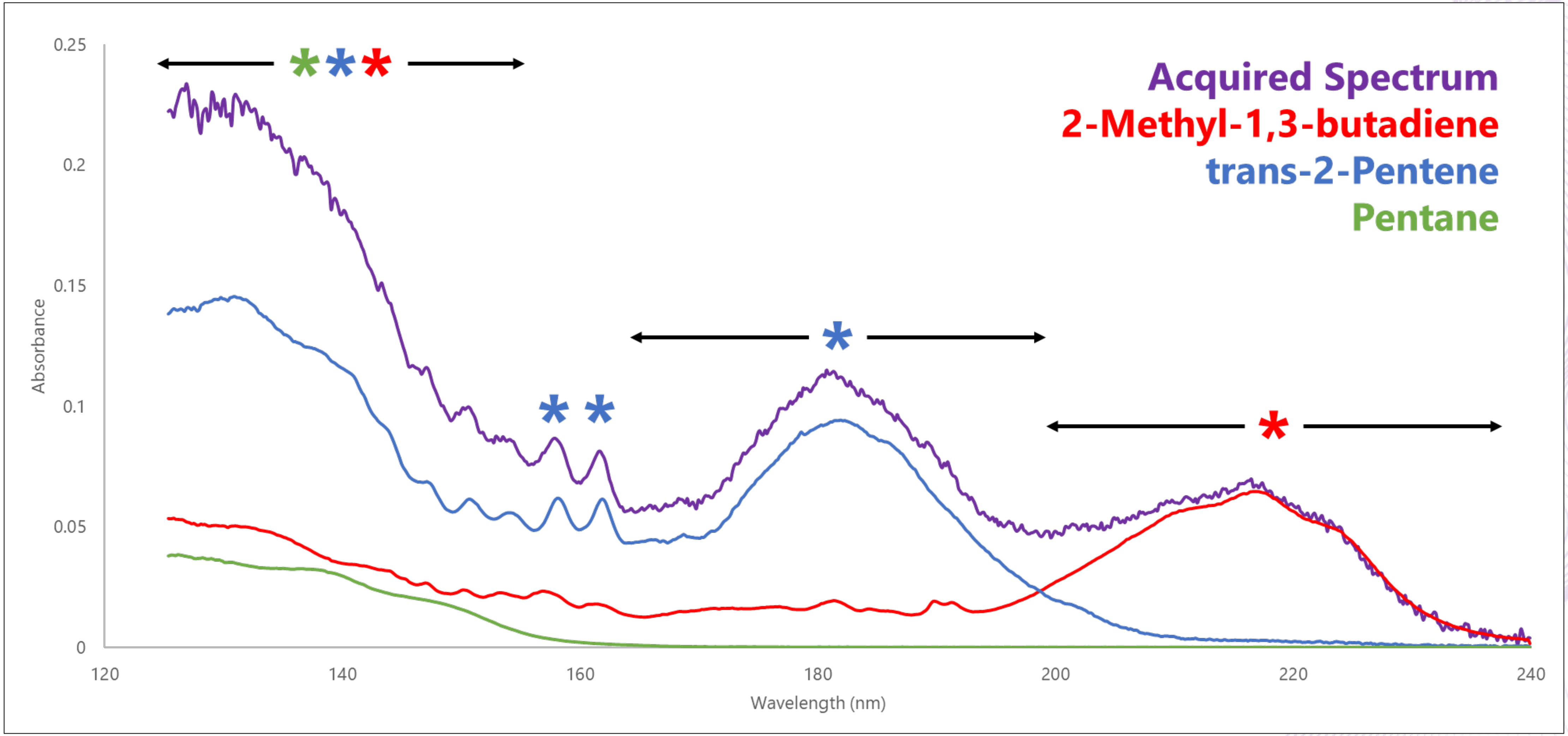






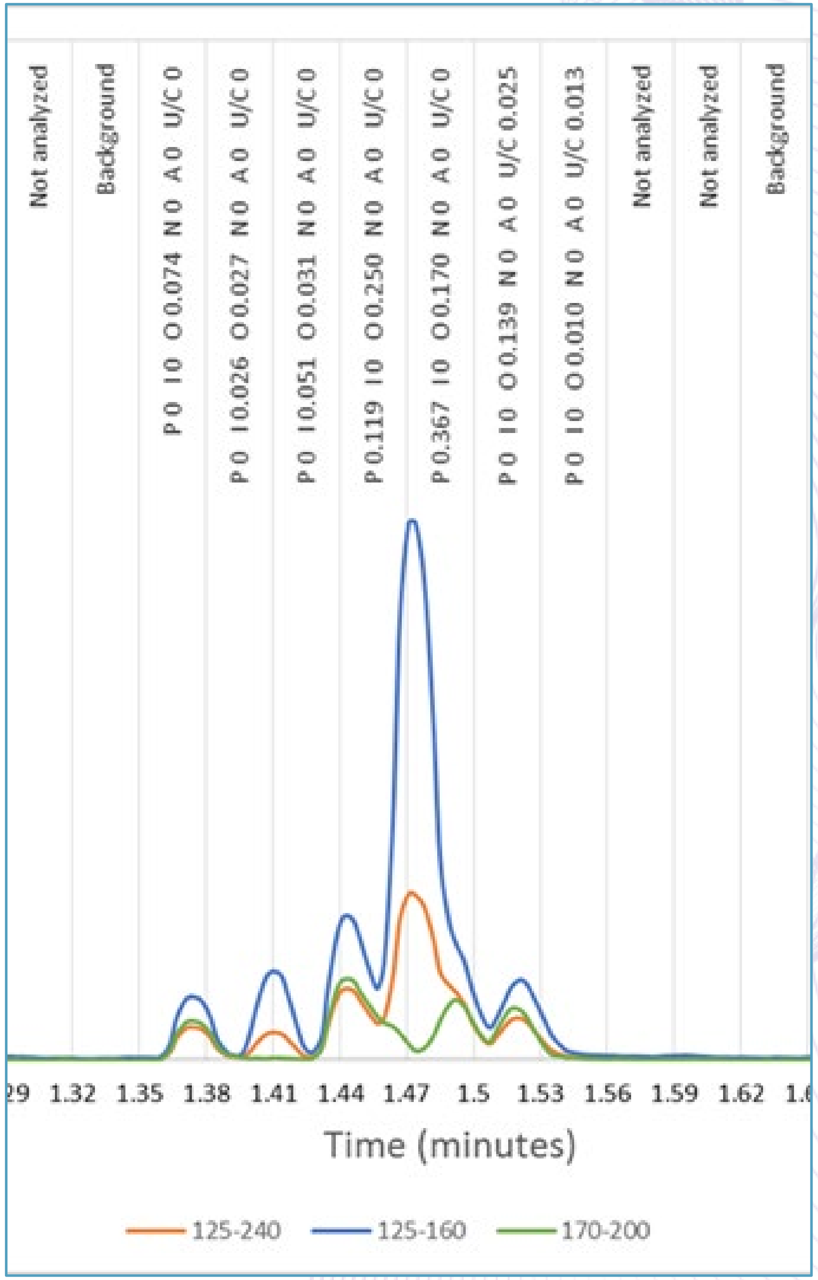


Spectral Deconvolution of Coeluting Hydrocarbons



Time Interval Deconvolution™ (TID)

- Quantitation method using VUV Analyze™ Software
- Chromatogram is divided into small time intervals (typically <0.05 min)
- For each time interval, compare measured spectrum against reference spectra in designated library, best analyte(s) fit determined
- Quickly determines total response per analyte
    - Converted to Mass % and Volume %



Verified Hydrocarbon Analysis™

Verified Hydrocarbon Analysis™

A Better Approach to DHA

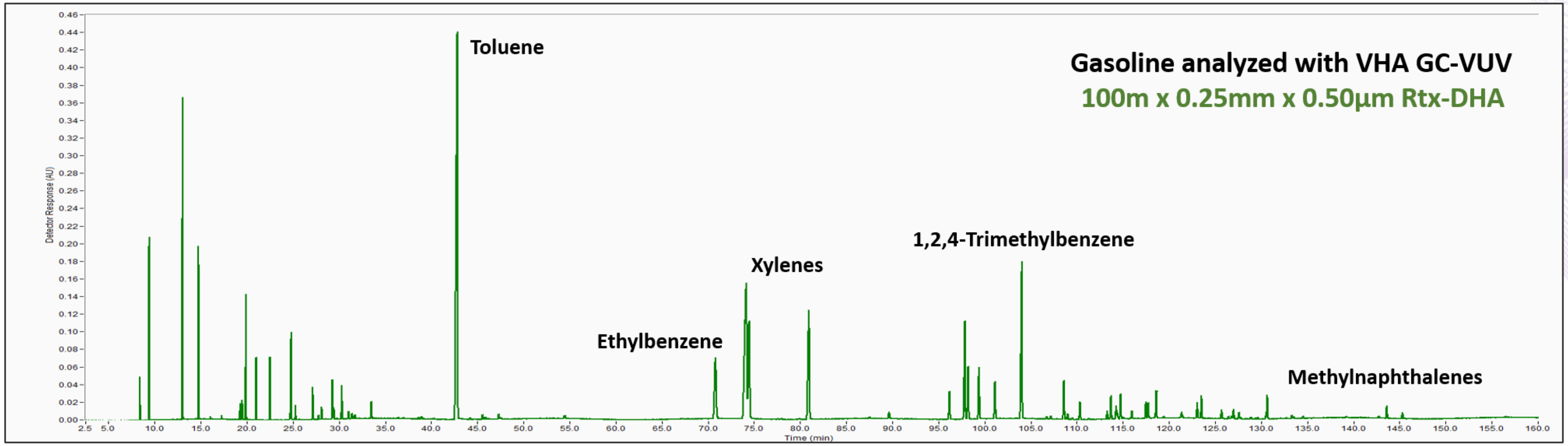
Typical DHA versus VHA

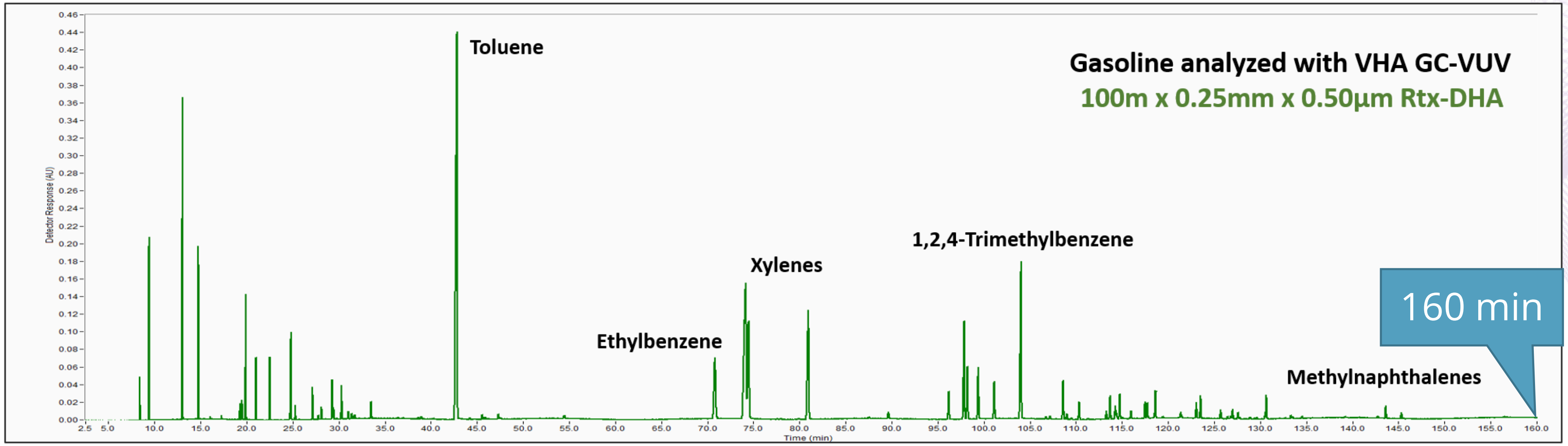
Method	ASTM D6730 (DHA) GC-FID	VHA VUV Analyzer™ Platform GC-VUV
Run Time (min.)	174	50
Column	Single 100m	Single 60m
Tuning Precolumn	Yes (5m)	no
Data Processing	Automated	Hands-off Analysis
Data Review	Manual	Automated
Verification	Retention Time	Spectral Validation

Fast

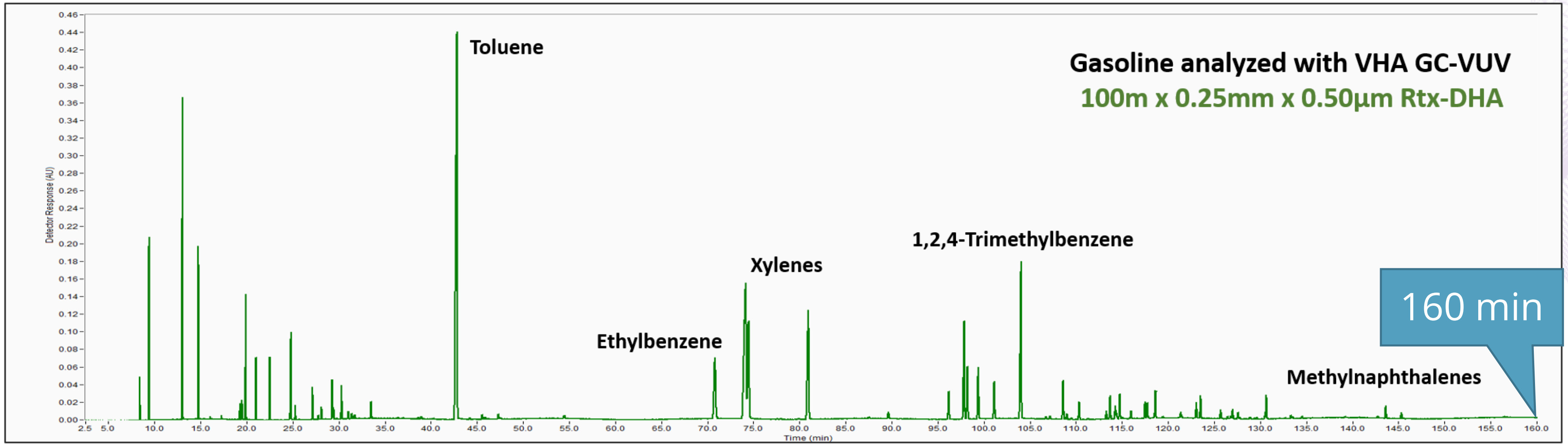
Easy

Accurate


Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
Toluene
Ethylbenzene
Xylenes
1,2,4-Trimethylbenzene
Methylnaphthalenes
Detector Response (AU)
Time (min)



Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
Toluene
Ethylbenzene
Xylenes
1,2,4-Trimethylbenzene
Methylnaphthalenes
160 min
Detector Response (AU)
Time (min)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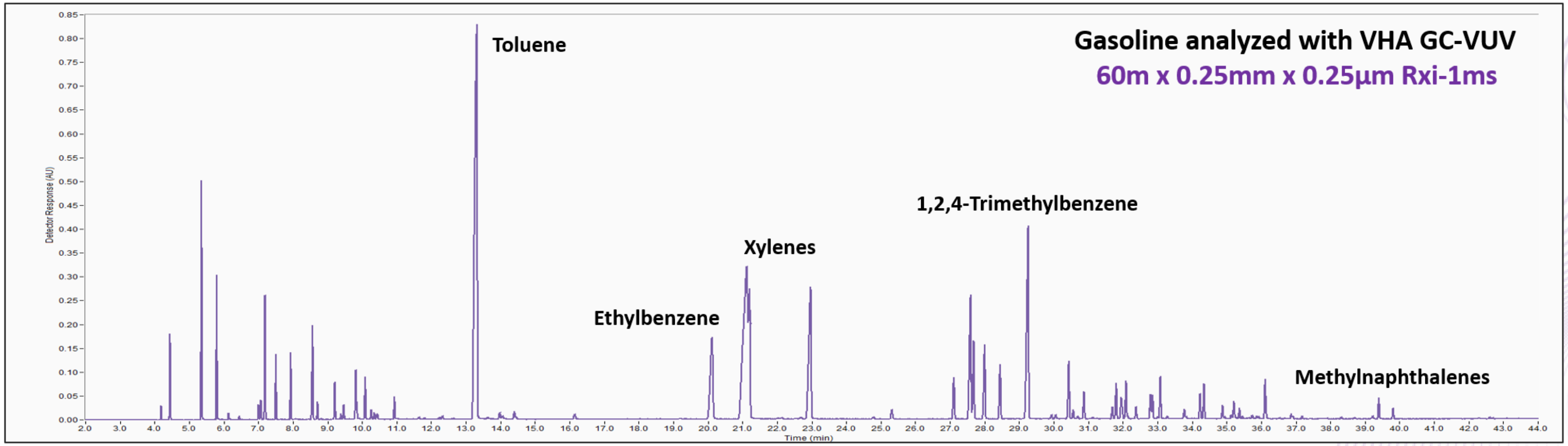
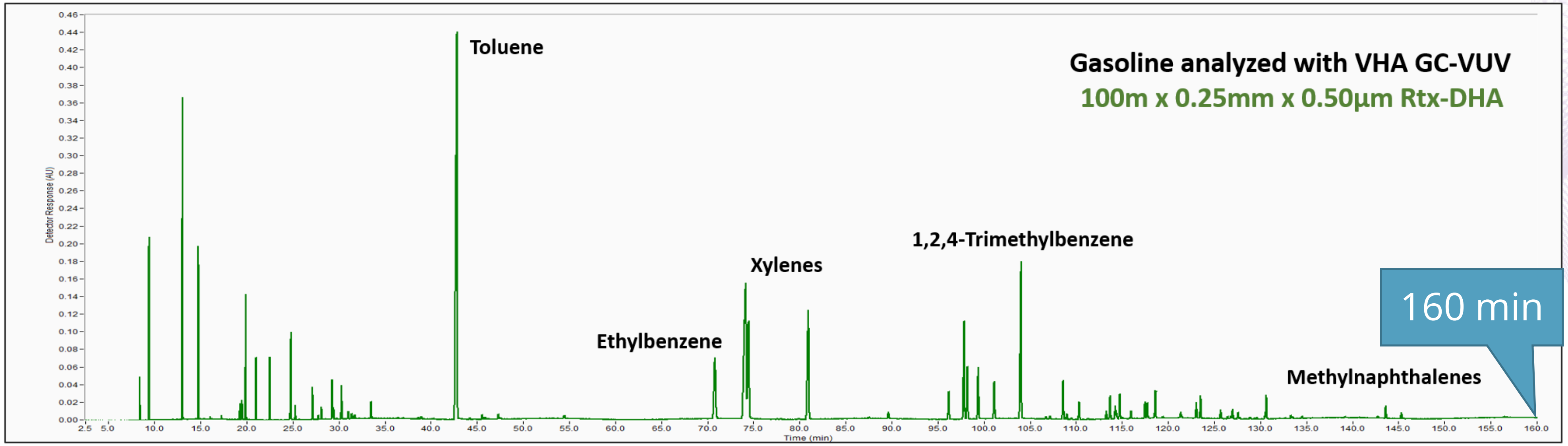
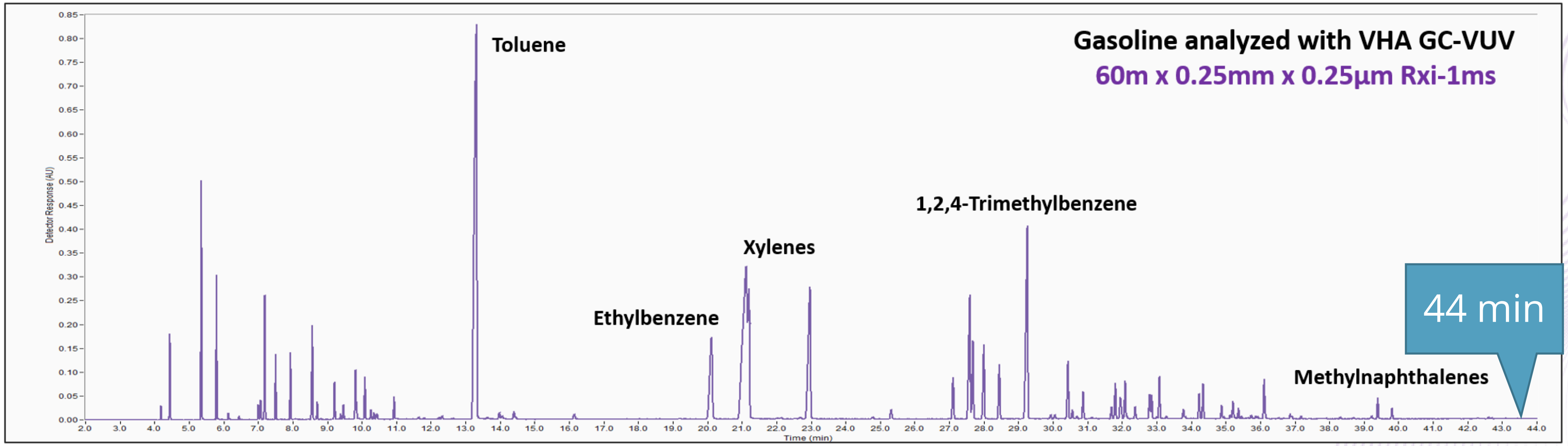
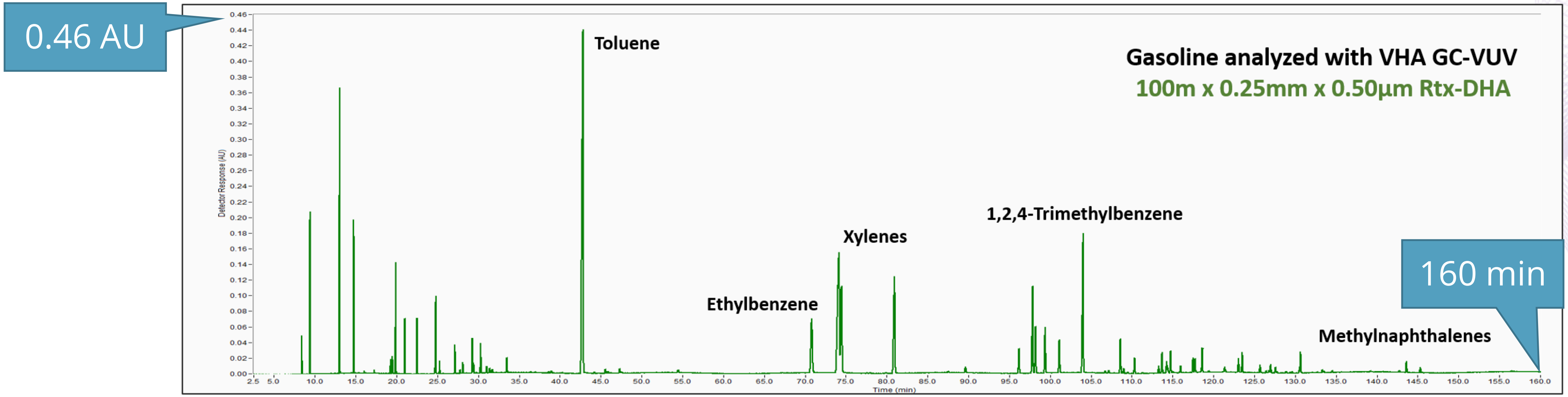
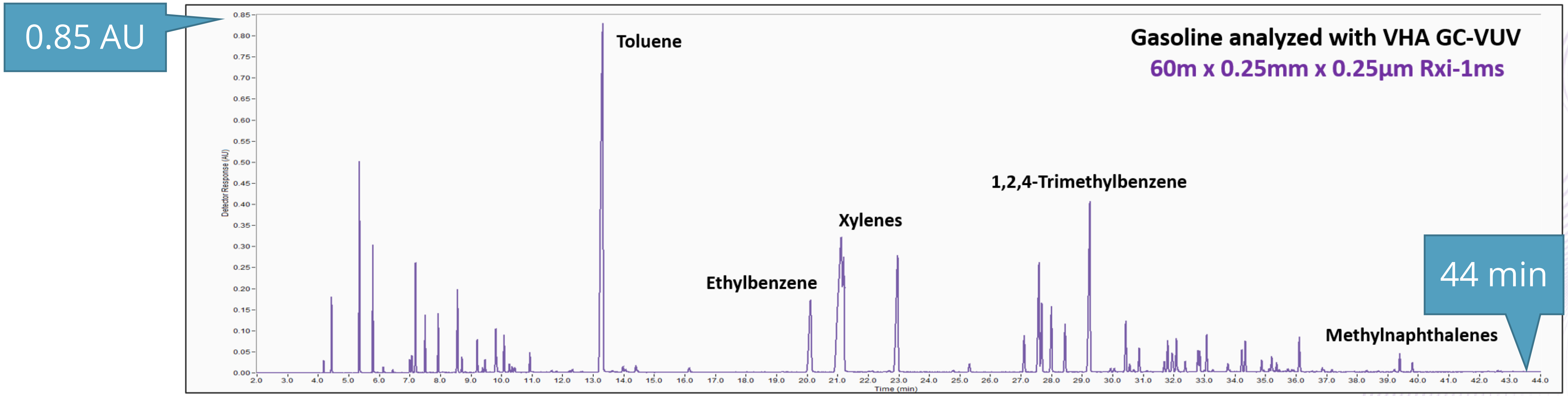
100m x 0.25mm x 0.50µm Rtx-DHA
1,2,4-Trimethylbenzene
Xylenes
Ethylbenzene
160 min
Methylnaphthalenes
Detector Response (AU)
Time (min)

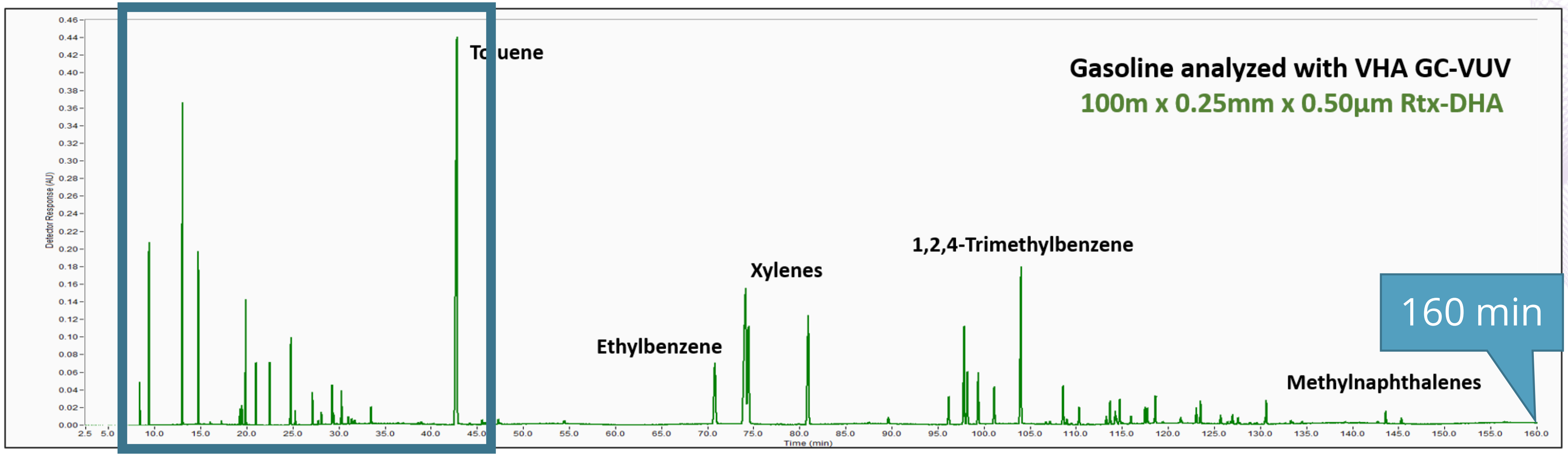

Toluene
Gasoline analyzed with VHA GC-VUV
60m x 0.25mm x 0.25µm Rxi-1ms
1,2,4-Trimethylbenzene
Xylenes
Ethylbenzene
Methylnaphthalenes
Detector Response (AU)
Time (min)


Toluene
Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
1,2,4-Trimethylbenzene
Xylenes
Ethylbenzene
160 min
Methylnaphthalenes
Detector Response (AU)
Time (min)

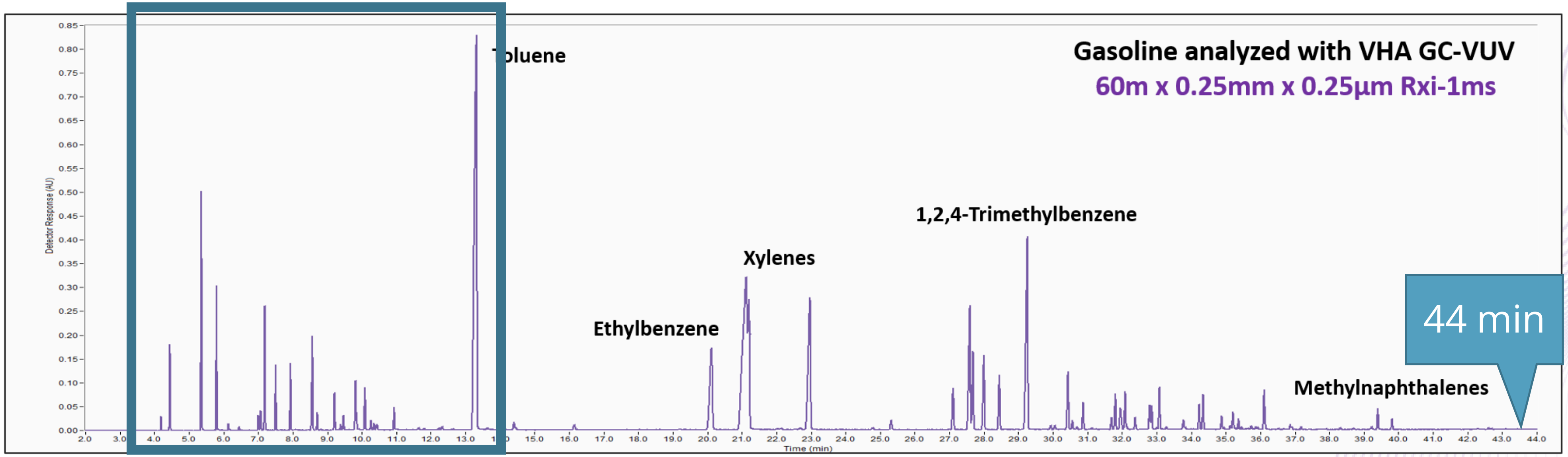

Toluene
Gasoline analyzed with VHA GC-VUV
60m x 0.25mm x 0.25µm Rxi-1ms
1,2,4-Trimethylbenzene
Xylenes
Ethylbenzene
44 min
Methylnaphthalenes
Detector Response (AU)
Time (min)


0.46 AU
Toluene
Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
1,2,4-Trimethylbenzene
Xylenes
Ethylbenzene
160 min
Methylnaphthalenes
Detector Response (AU)
Time (min)

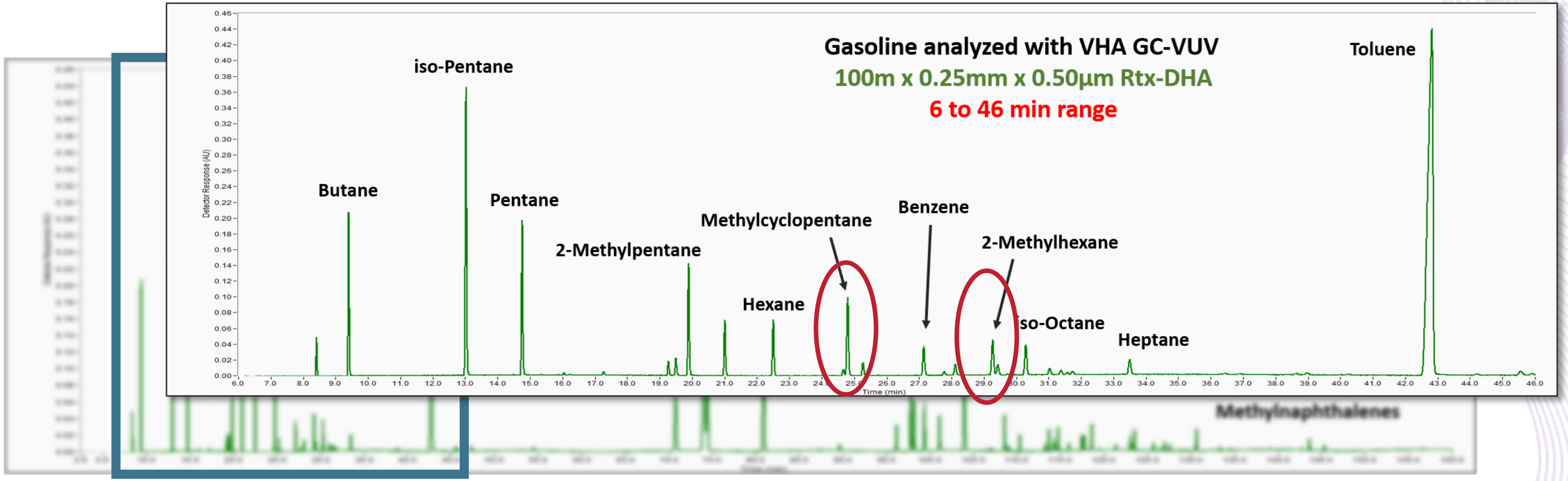

0.85 AU
Toluene
Gasoline analyzed with VHA GC-VUV
60m x 0.25mm x 0.25µm Rxi-1ms
1,2,4-Trimethylbenzene
Xylenes
Ethylbenzene
44 min
Methylnaphthalenes
Detector Response (AU)
Time (min)

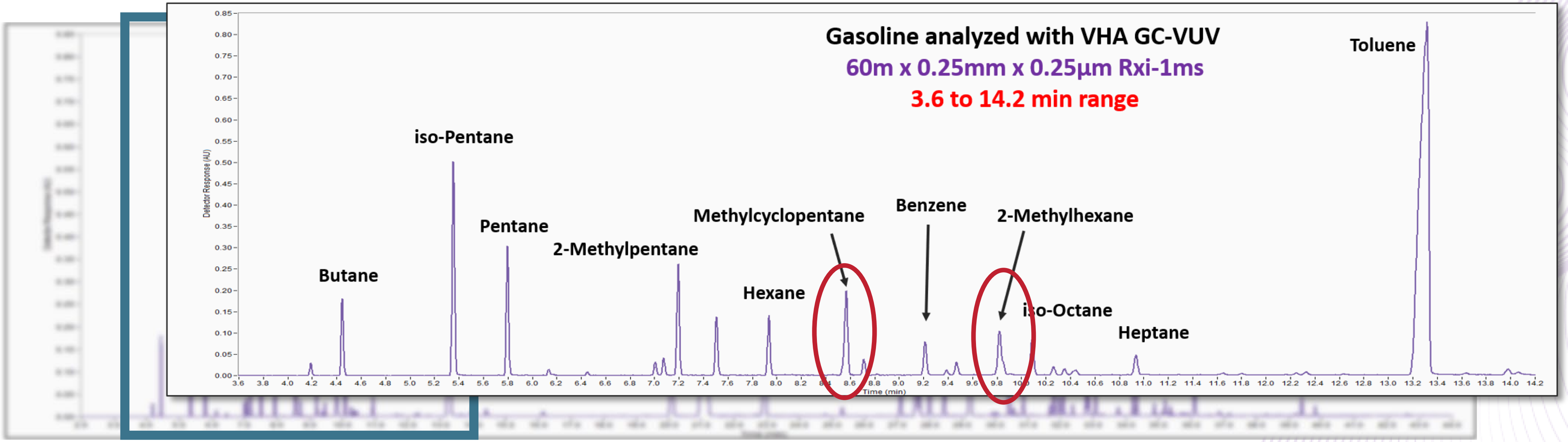


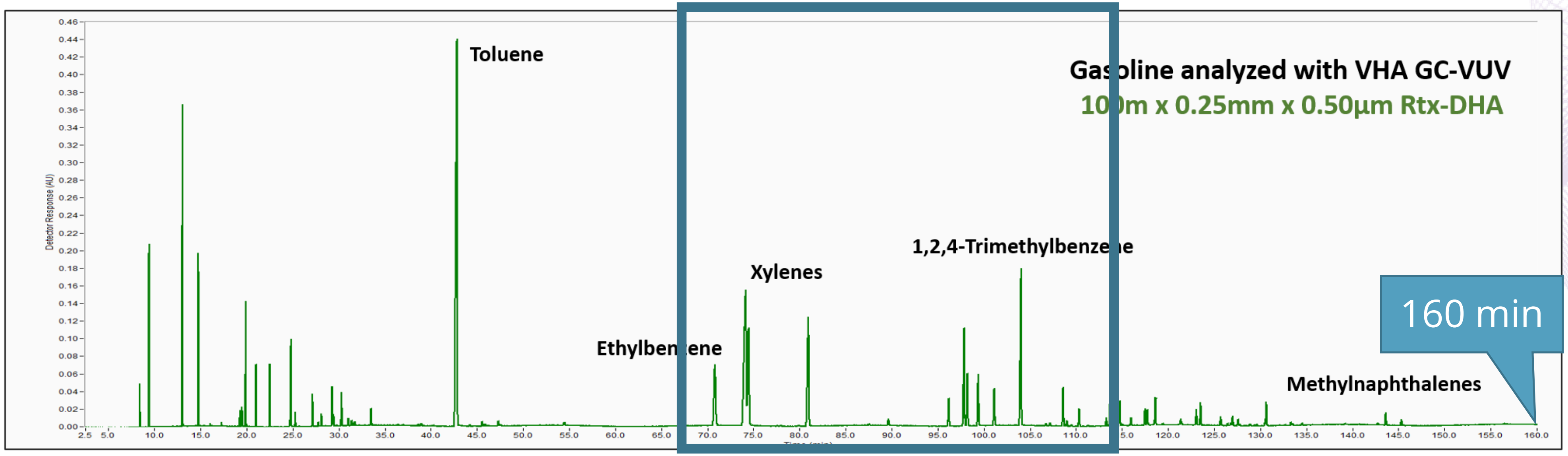
Toluene
Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
1,2,4-Trimethylbenzene
Xylenes
160 min
Ethylbenzene
Methylnaphthalenes
Detector Response (AU)
Time (min)

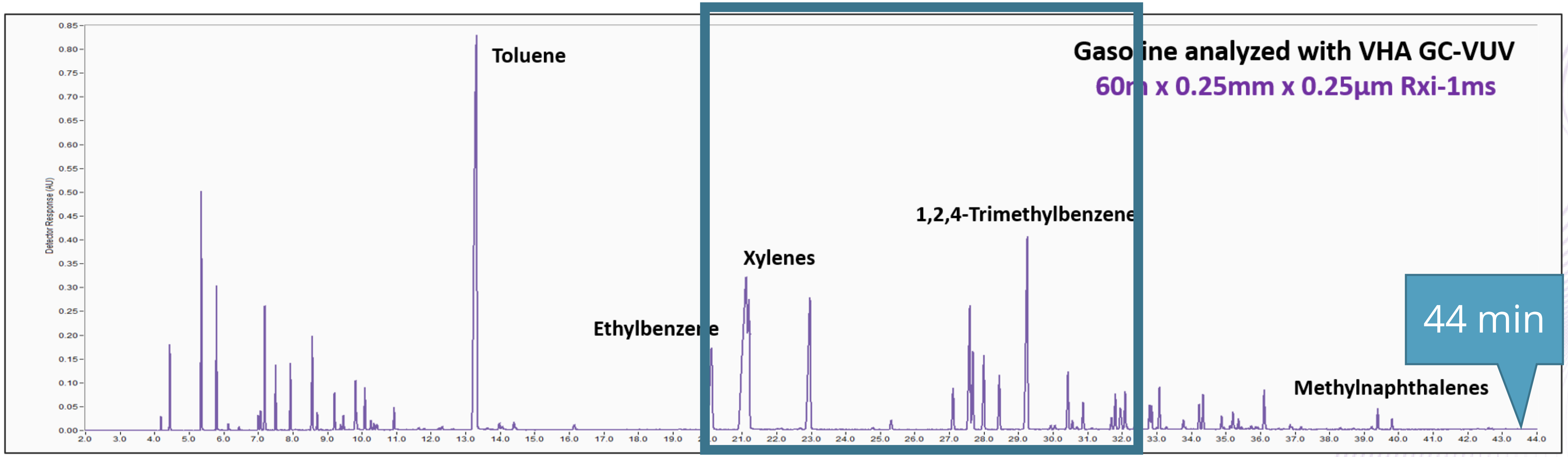


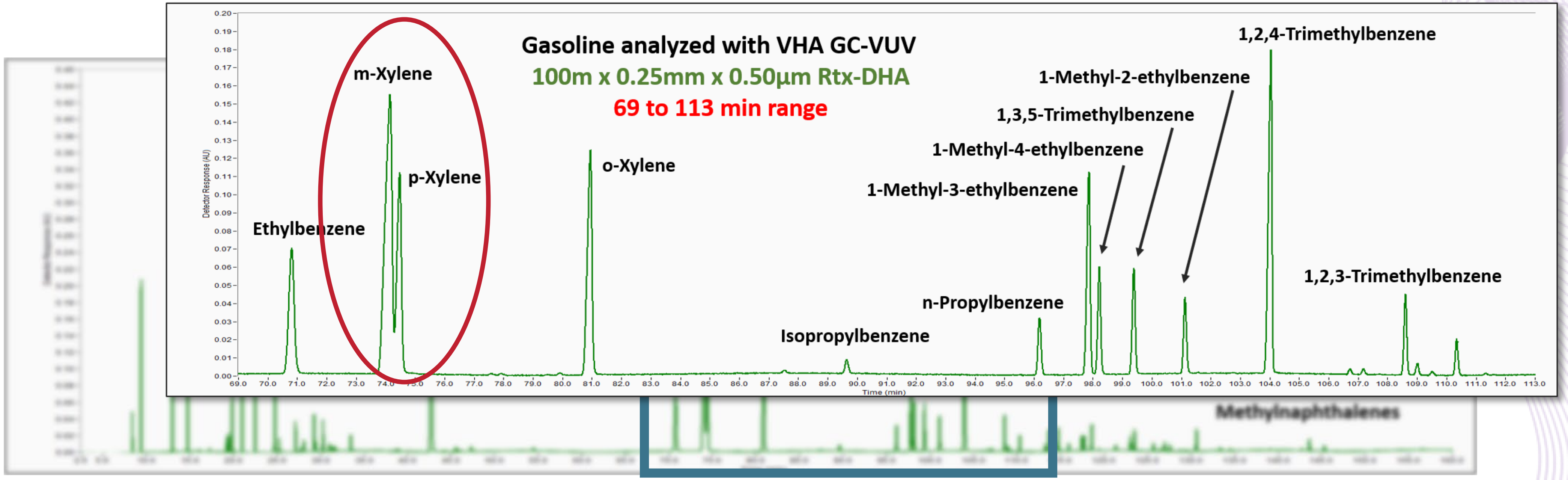
Toluene
Gasoline analyzed with VHA GC-VUV
60m x 0.25mm x 0.25µm Rxi-1ms
1,2,4-Trimethylbenzene
Xylenes
44 min
Ethylbenzene
Methylnaphthalenes
Detector Response (AU)
Time (min)

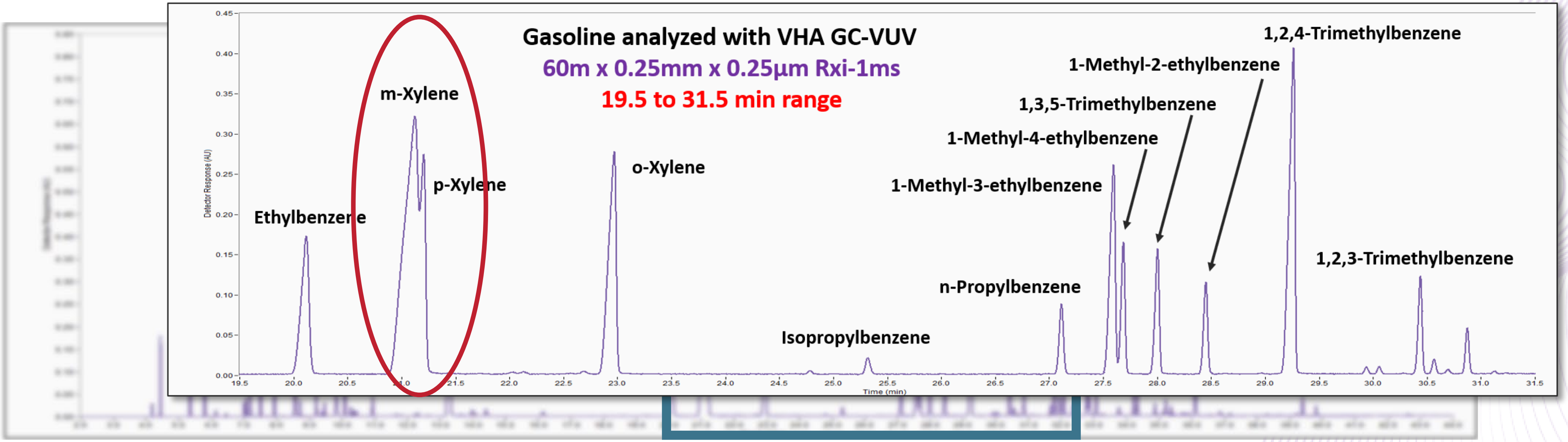

Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
6 to 46 min range
Butane
iso-Pentane
Pentane
2-Methylpentane
Hexane
Methylcyclopentane
Benzene
2-Methylhexane
iso-Octane
Heptane
Toluene
Methylnaphthalenes
Detector Response (AU)
Time (min)


Gasoline analyzed with VHA GC-VUV
60m x 0.25mm x 0.25µm Rxi-1ms
3.6 to 14.2 min range
Butane
iso-Pentane
Pentane
2-Methylpentane
Hexane
Methylcyclopentane
Benzene
2-Methylhexane
iso-Octane
Heptane
Toluene
Detector Response (AU)
Time (min)

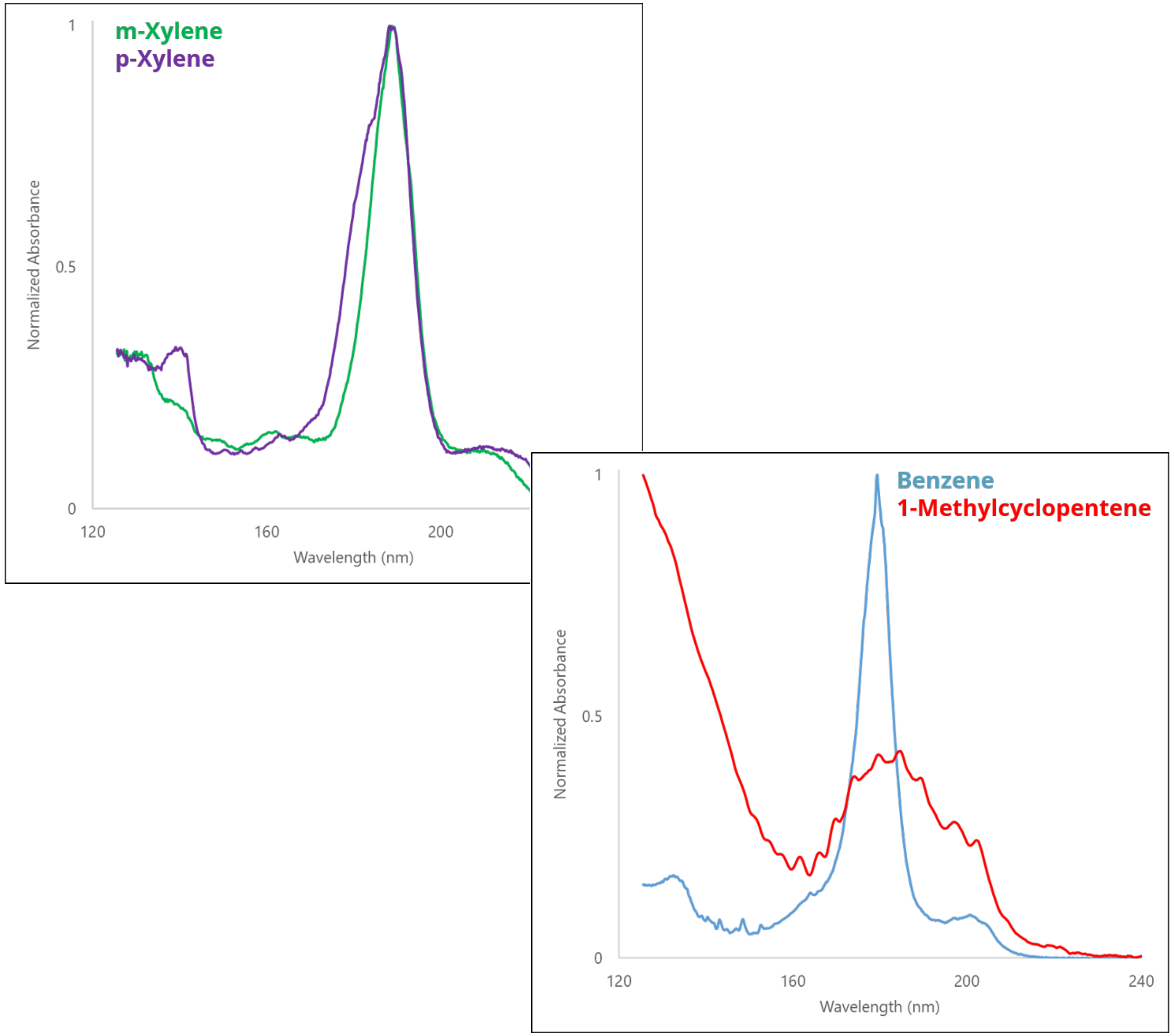

Toluene
Ethylben ene
Xylenes
1,2,4-Trimethylbenze e
Methylnaphthalenes
Gas line analyzed with VHA GC-VUV
10 m x 0.25mm x 0.50µm Rtx-DHA
160 min
Detector Response (AU)


Toluene
Ethylbenzer e
Xylenes
1,2,4-Trimethylbenzene
Methylnaphthalenes
Gaso ine analyzed with VHA GC-VUV
60r x 0.25mm x 0.25µm Rxi-1ms
44 min
Detector Response (AU)


Gasoline analyzed with VHA GC-VUV
100m x 0.25mm x 0.50µm Rtx-DHA
69 to 113 min range
Detector Response (AU)
Time (min)
m-Xylene
p-Xylene
Ethylbenzene
o-Xylene
Isopropylbenzene
n-Propylbenzene
1-Methyl-3-ethylbenzene
1-Methyl-4-ethylbenzene
1,3,5-Trimethylbenzene
1-Methyl-2-ethylbenzene
1,2,4-Trimethylbenzene
1,2,3-Trimethylbenzene
Methylnaphthalenes

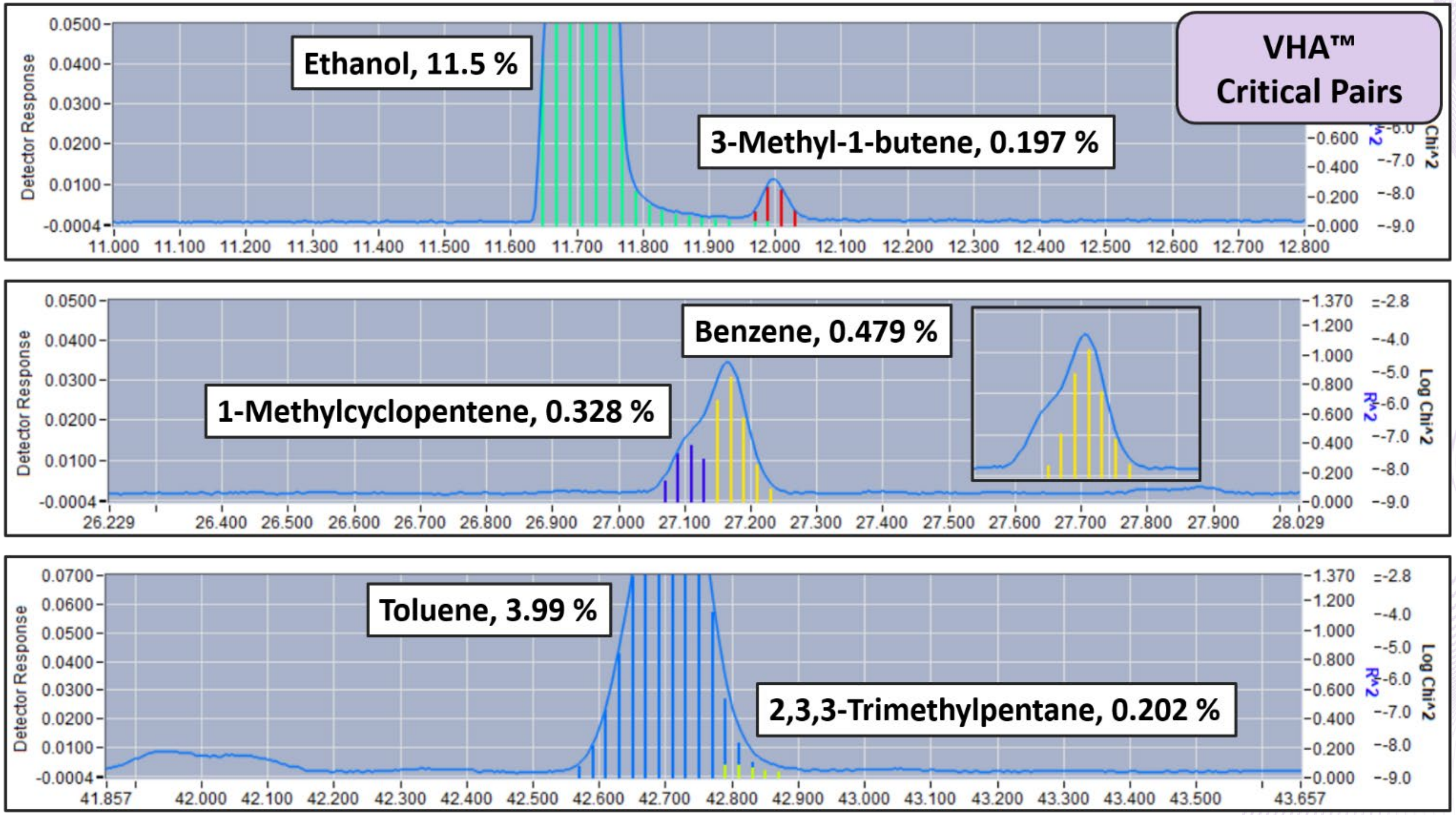

Gasoline analyzed with VHA GC-VUV
60m x 0.25mm x 0.25µm Rxi-1ms
19.5 to 31.5 min range
Detector Response (AU)
Time (min)
m-Xylene
p-Xylene
Ethylbenzene
o-Xylene
Isopropylbenzene
n-Propylbenzene
1-Methyl-3-ethylbenzene
1-Methyl-4-ethylbenzene
1,3,5-Trimethylbenzene
1-Methyl-2-ethylbenzene
1,2,4-Trimethylbenzene
1,2,3-Trimethylbenzene

VHA™ – Deconvolution of Critical Pairs

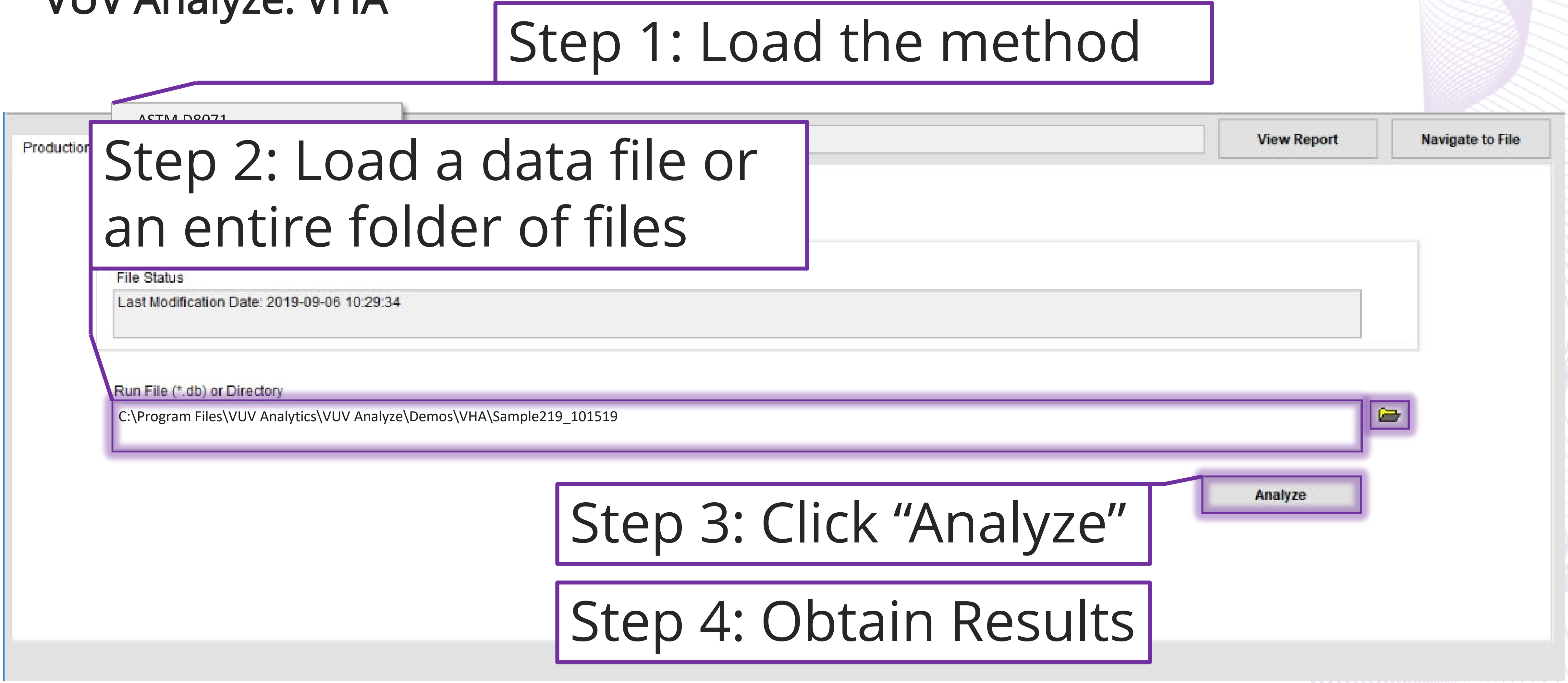


- ASTM D6730
  - “Cut column until you have identified critical separations”
- NOT required with VHA thanks to critical pairs identification
- Fast
  - VHA enables compressed chromatography
- Easy
  - Deconvolution software automatically identifies compounds
- Accurate
  - Human guesswork is never a factor

VHA™ – Deconvolution of Critical Pairs



VUV Analyze: VHA



VHA™ vs DHA: Naphtha Streams

Mass %

Category	Naphtha A			Naphtha B		
	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA
Paraffin	21.0	20.8	19.1	23.2	23.4	22.0
Isoparaffin	31.4	30.8	27.6	31.5	31.3	25.4
Olefin	0.223	0.0775	0.861	0.210	0.0692	0.746
Naphthene	**32.9**	**33.7**	**19.3**	**33.2**	[illegible]	**23.0**
Aromatic	14.5	14.6	15.2	[illegible]	[illegible]	13.3

Category	Naphtha A			Naphtha B		
	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA
Cyclopentane	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	0.106
MeCyclopent[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1.38
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	0.335	0.345
[illegible]	[illegible]	[illegible]	[illegible]	1.31	1.32	1.45
[illegible]	[illegible]	[illegible]	ND	0.047	0.074	ND
Me[illegible]	[illegible]	3.16	3.11	4.60	4.67	4.75
[illegible]	1.48	1.50	1.41	1.90	1.92	1.79
Ethylbenzene	1.51	1.52	1.89	0.700	0.702	0.948
m-Xylene	**2.84**	**2.99**	**1.61**	**2.56**	**2.64**	**1.33**
p-Xylene	**1.01**	**0.935**	**1.58**	**0.835**	**0.794**	**1.27**
o-Xylene	1.38	1.42	0.905	1.02	1.01	0.704

VHA Unidentified: <1.5%

DHA Unidentified: >15%

VHA™ vs DHA: European Oxygenates

Mass %

Compound	Gasoline A			Gasoline B		
	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA
Cyclopentane	0.359	0.334	0.359	0.348	0.350	0.358
Methyl tert-butyl ether	**1.56**	**1.43**	**ND***	**2.08**	**1.99**	**0.285***
Methylcyclopentane	1.76	1.77	1.66	1.82	1.83	1.67
Benzene	0.812	0.804	0.759	0.785	0.775	0.750
Cyclohexane	1.03	1.02	1.11	1.02	0.967	1.09
tert-Amyl methyl ether	**0.065**	**0.047**	**ND**	**0.089**	**0.071**	**0.062**
Isooctane	0.033	0.058	0.065	0.032	0.072	0.077
MeCyclohexane	2.94	2.95	3.10	2.90	2.92	3.02
Toluene	10.6	10.6	10.3	10.1	9.96	9.89
Ethylbenzene	2.47	2.50	2.45	2.42	2.41	2.39
m-Xylene	5.58	5.99	5.42	5.53	5.83	5.30
p-Xylene	2.70	2.44	2.42	2.60	2.33	2.43
o-Xylene	3.13	3.19	3.05	3.09	3.09	3.01

*Misidentified as cis-4-Methyl-2-pentene in DHA method and data review

VHA™ vs DHA: Aromatic Concentrates (aka PyGas)

Mass % Category	PyGas A			PyGas B			PyGas C		
	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA
Paraffin	3.58	3.62	3.75	3.06	3.23	3.40	0.345	0.479	0.507
Isoparaffin	4.12	4.55	4.60	3.61	3.84	4.16	1.26	1.54	2.02
Olefin	**15.0**	**15.6**	**16.0**	**23.3**	**24.1**	**21.0**	**23.6**	**24.8**	**18.0**
Naphthene	6.69	7.25	5.56	4.06	4.42	5.05	0.469	0.507	0.487
Aromatic	**70.6**	**69.0**	**66.2**	**66.0**	**64.4**	**60.9**	**74.4**	**72.6**	**60.3**

VHA™ vs DHA: Aromatic Concentrates (aka PyGas)

Mass % Compound	PyGas A			PyGas B			PyGas C		
	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA
Cyclopentane	0.846	0.852	0.853	0.533	0.525	0.571	0.071	0.081	0.086
MeCyclopentane	2.40	2.37	2.24	1.84	1.79	1.70	0.065	0.077	0.086
Benzene	55.6	54.0	51.9	50.9	49.4	47.2	41.6	40.1	37.3
Cyclohexane	0.872	1.04	1.27	0.627	0.770	0.937	ND	ND	0.025
Isooctane	0.040	0.053	0.053	ND	0.012	0.025	0.044	0.017	0.009
MeCyclohexane	0.228	0.249	0.231	0.160	0.165	0.178	ND	0.011	0.020
Toluene	9.21	8.77	8.54	7.83	7.40	6.99	5.36	5.10	4.70
Ethylbenzene	1.06	0.999	0.976	0.666	0.669	0.608	0.958	0.936	0.838
m-Xylene	0.243	0.231	0.223	0.196	0.204	0.188	0.206	0.200	0.169
p-Xylene	0.132	0.118	0.108	0.096	0.104	0.095	0.098	0.087	0.083
o-Xylene	0.160	0.160	0.165	0.207	0.214	0.203	0.103	0.095	0.104

VHA™ vs DHA: Aromatic Concentrates (aka PyGas)

Mass % Compound	PyGas A			PyGas B			PyGas C		
	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA	VHA 1	VHA 2	DHA
1-Pentene	0.656	0.709	0.609	0.924	0.976	0.986	1.11	1.16	1.07
n-Pentane	0.610	0.655	0.644	0.258	0.270	0.313	0.097	0.095	0.102
Isoprene	1.03	1.08	0.812	0.703	0.722	0.612	0.371	0.392	0.311
t-1,3-C5diene	0.910	0.954	0.608	1.24	1.27	0.892	0.624	0.619	0.413
Cyclopentadiene	0.370	0.414	0.485	0.432	0.490	0.532	0.750	0.814	1.35
c-1,3-C5diene	0.526	0.548	0.459	0.702	0.714	0.609	0.304	0.310	0.231
Cyclopentene	0.767	0.785	0.866	0.763	0.760	0.900	4.04	4.04	4.53
2-Methylpentane	1.05	1.04	0.946	0.961	0.939	0.929	0.174	0.190	0.181
3-Methylpentane	1.04	1.02	1.04	0.963	0.941	1.01	0.105	0.103	0.112
n-Hexane	2.49	2.44	2.41	2.33	2.26	2.33	0.030	0.040	0.065
EtCyPentane	0.580	0.569	0.570	0.371	0.370	0.365	ND	ND	0.009
Styrene	1.93	1.83	1.61	3.09	2.92	2.47	3.86	3.57	3.184
DCPD	**3.95**	**3.85**	**3.68**	**7.47**	**7.21**	**6.72**	**7.19**	**6.99**	**ND**
Indene	0.445	0.548	0.585	0.651	0.780	0.744	1.04	1.20	1.09

Verified Hydrocarbon Analysis™

Key Benefits

- Fast
  - 50 minutes from start to finish
- Easy to use
  - Data processing and review is completely automated
- Accurate
  - Spectral verification and retention time

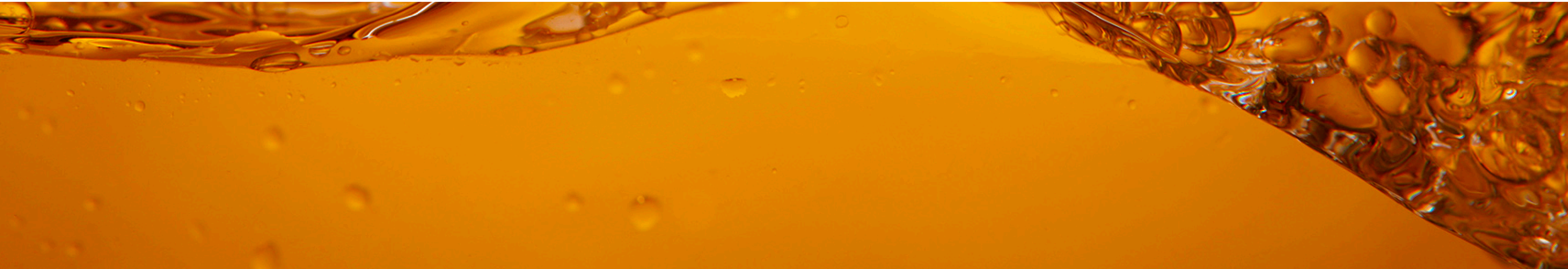
- **Application Scope**
  - Finished Gasoline
  - Oxygenates in gasoline
  - Naphtha
  - Reformate
  - Alkylate
  - PyGas
  - FCC gasoline
  - LPG
- **Greatest Applicability**
  - Refinery streams
  - VHA Templates
  - On-line application

CASE STUDY

A major U.S. refinery suspected that their N2+A yield was low resulting in lower market prices for heavy naphtha

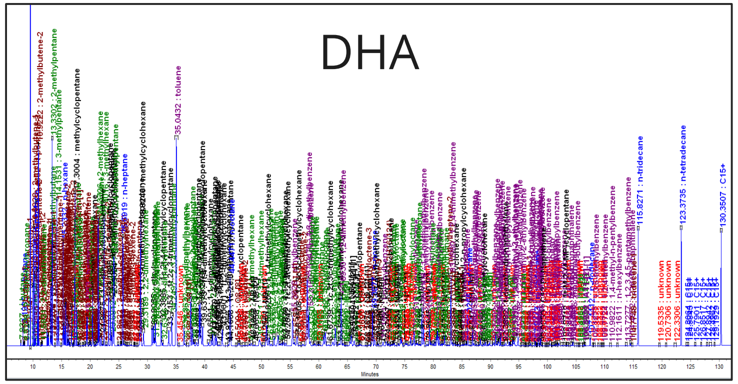
Closed Revenue Leak ($8 - $16 million lost)

The VUV Analyzer™ Platform



The VUV Analyzer™ Platform

One Platform; Many Possibilities



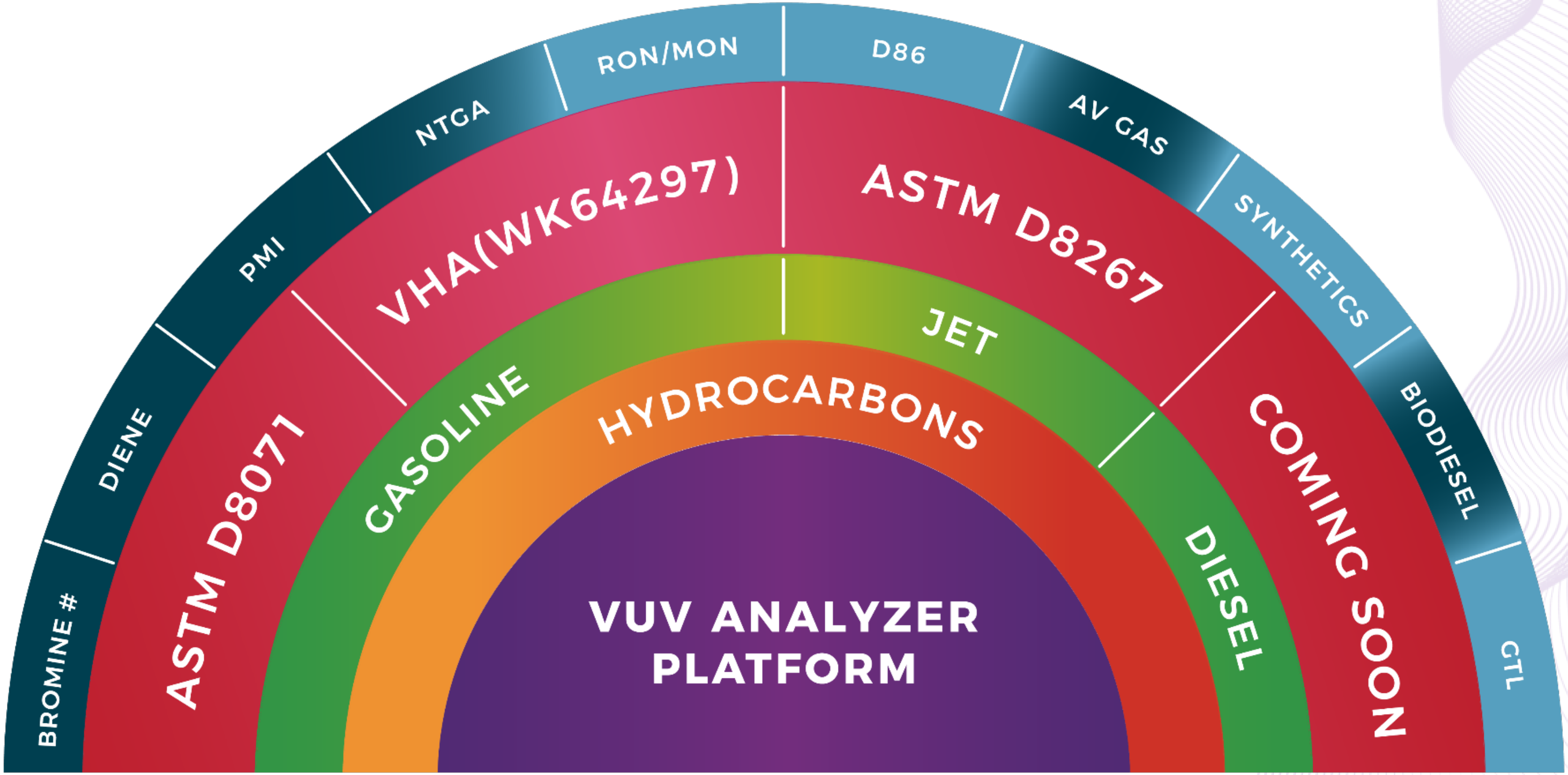
DHA

ASTM D5599
GC-OFID

ASTM D5769
GC-MS

ASTM D3606
GC-TCD

ASTM D1319
FIA


BROMINE #
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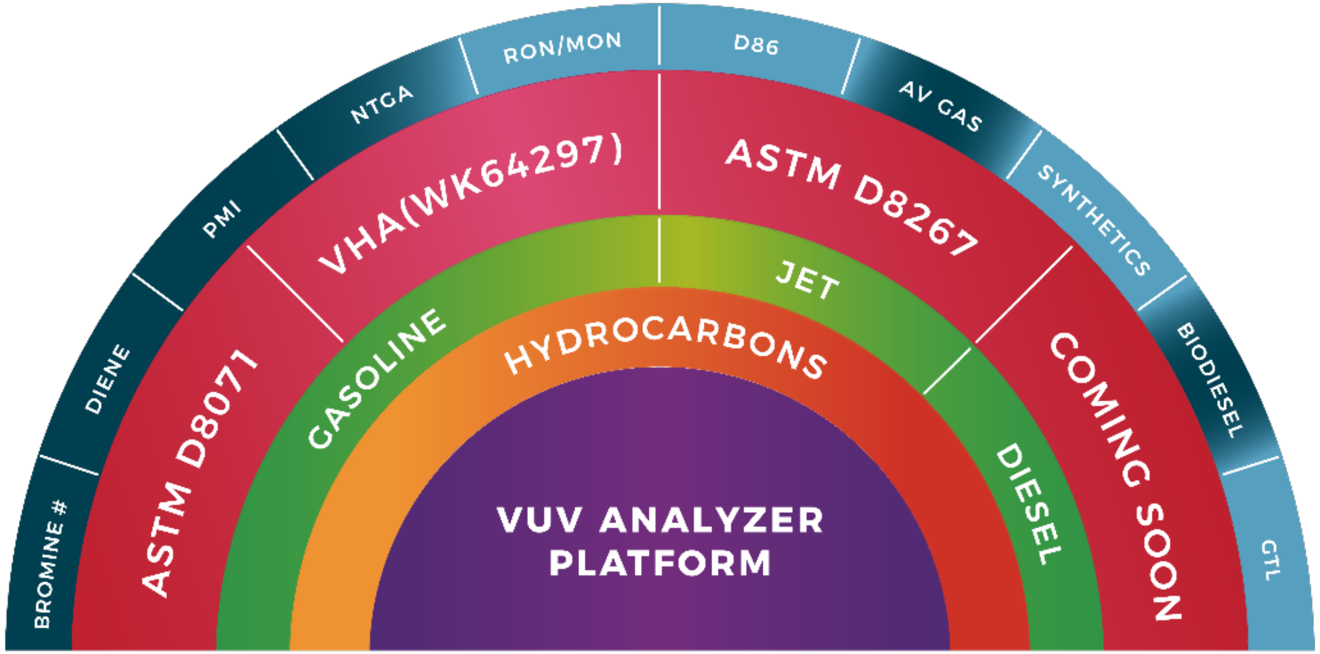
PMI
NTGA
RON/MON
D86
AV GAS
SYNTHETICS
BIODIESEL
GTL
ASTM D8071
VHA(WK64297)
ASTM D8267
COMING SOON
GASOLINE
JET
DIESEL
HYDROCARBONS
VUV ANALYZER PLATFORM

The VUV Analyzer™ Platform

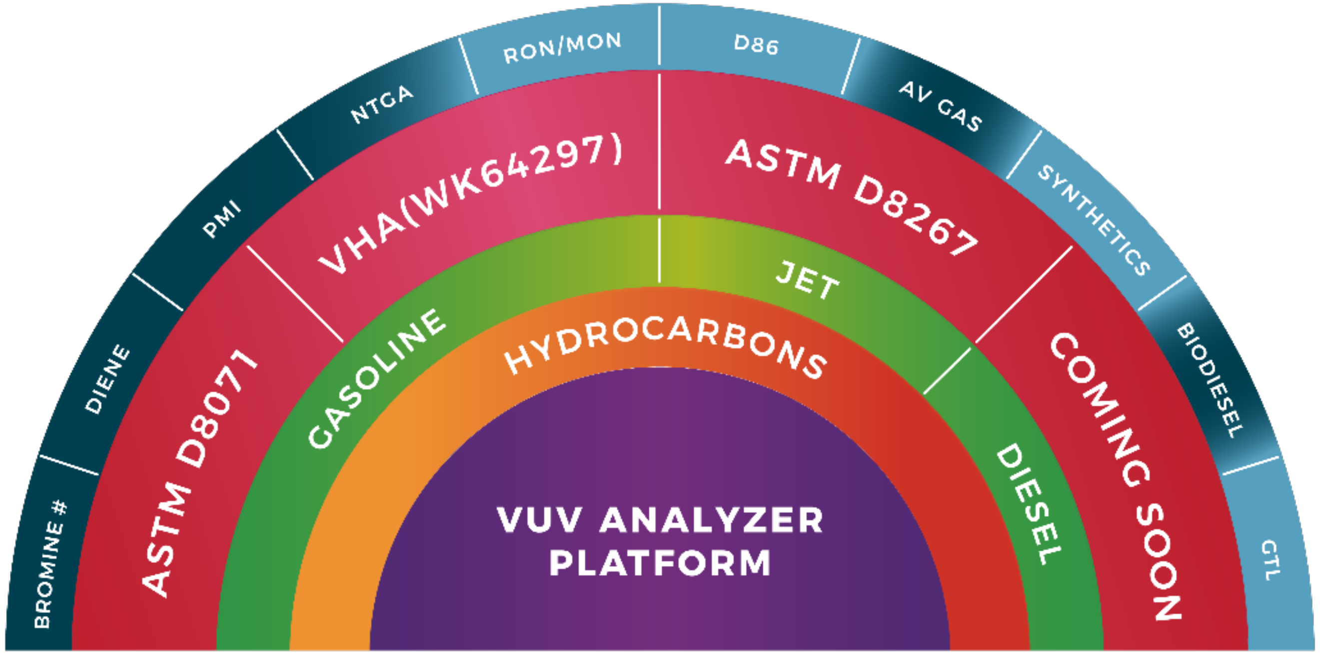
One Platform; Many Possibilities



Platform Advantages:

- One platform capable of running different methods
- No changes in hardware or setup
- Scalable from R&D to Production
- Fully automated applications
- Multi-method architecture
- Lower Total Cost of Analysis (CoA) compared to other techniques

Verified Hydrocarbon Analysis™ (VHA)



Summary

- Fast
  - VHA runtime is only 50 minutes
  - OVER 3X faster than traditional DHA
- Easy
  - Data processing and data review is fully automated with VUV Analyze™ Software
  - DHA requires skilled operator and manual data review
- Accurate
  - Data verification is performed using both retention time and spectral identification
  - DHA relies upon retention time alone

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