SepSolve Analytical

As easy as GC×GC

Aaron Parker

European Sales Manager

Who are SepSolve Analytical?

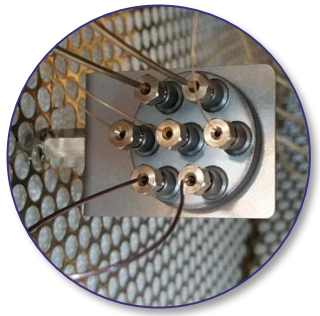
Experts in analytical chemistry

SCHAUENBURG Analytics

MARKES international

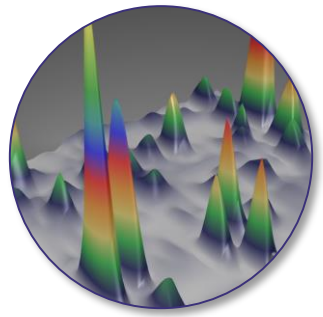
Sample preparation

SepSolve Analytical



Separation

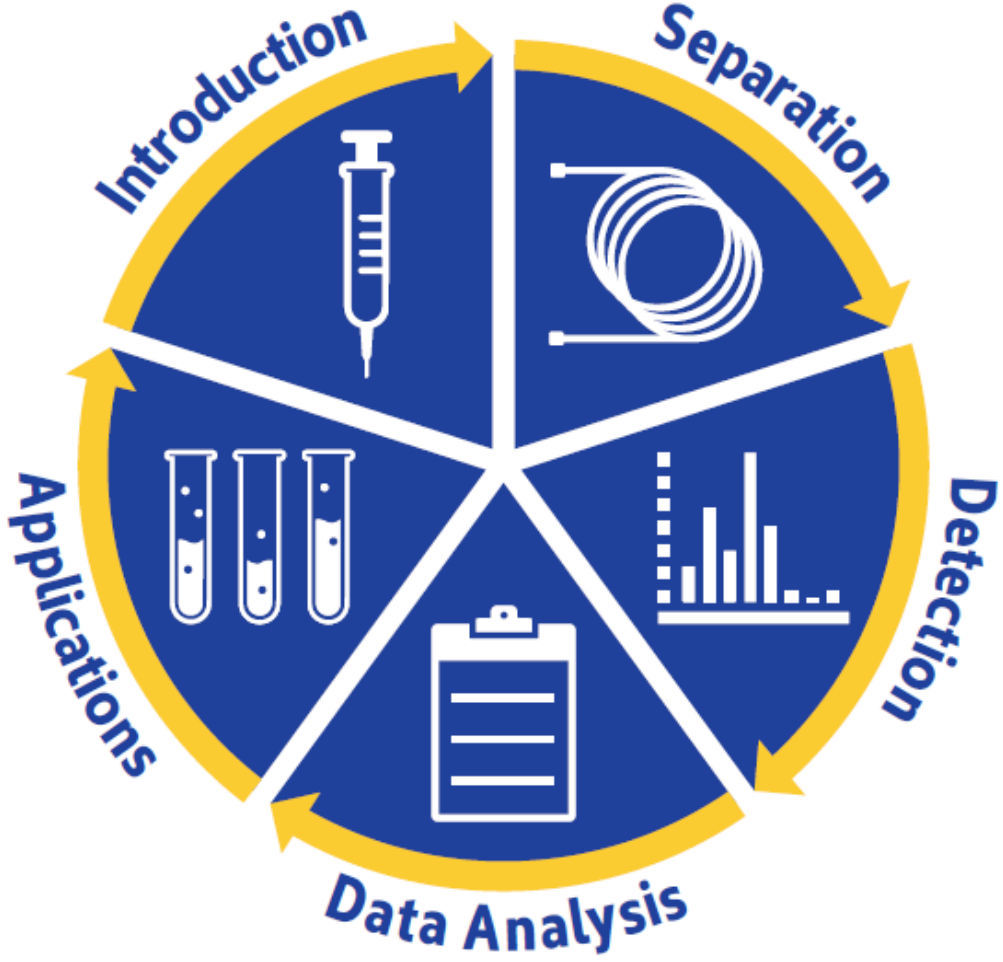
Identification



Data analysis

Who are SepSolve Analytical?

- We solve problems in separation science….
- …by delivering innovative GC-MS and GC×GC-MS solutions, including both hardware and software
- A single-vendor solution



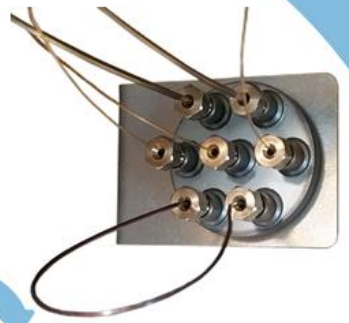
What can we offer?

Sample introduction

- Sample preparation robots
- Multi-mode inlets
- Full range of Markes' TD

Separation

- Heartcutting (GC-GC)
- GC×GC



Data analysis

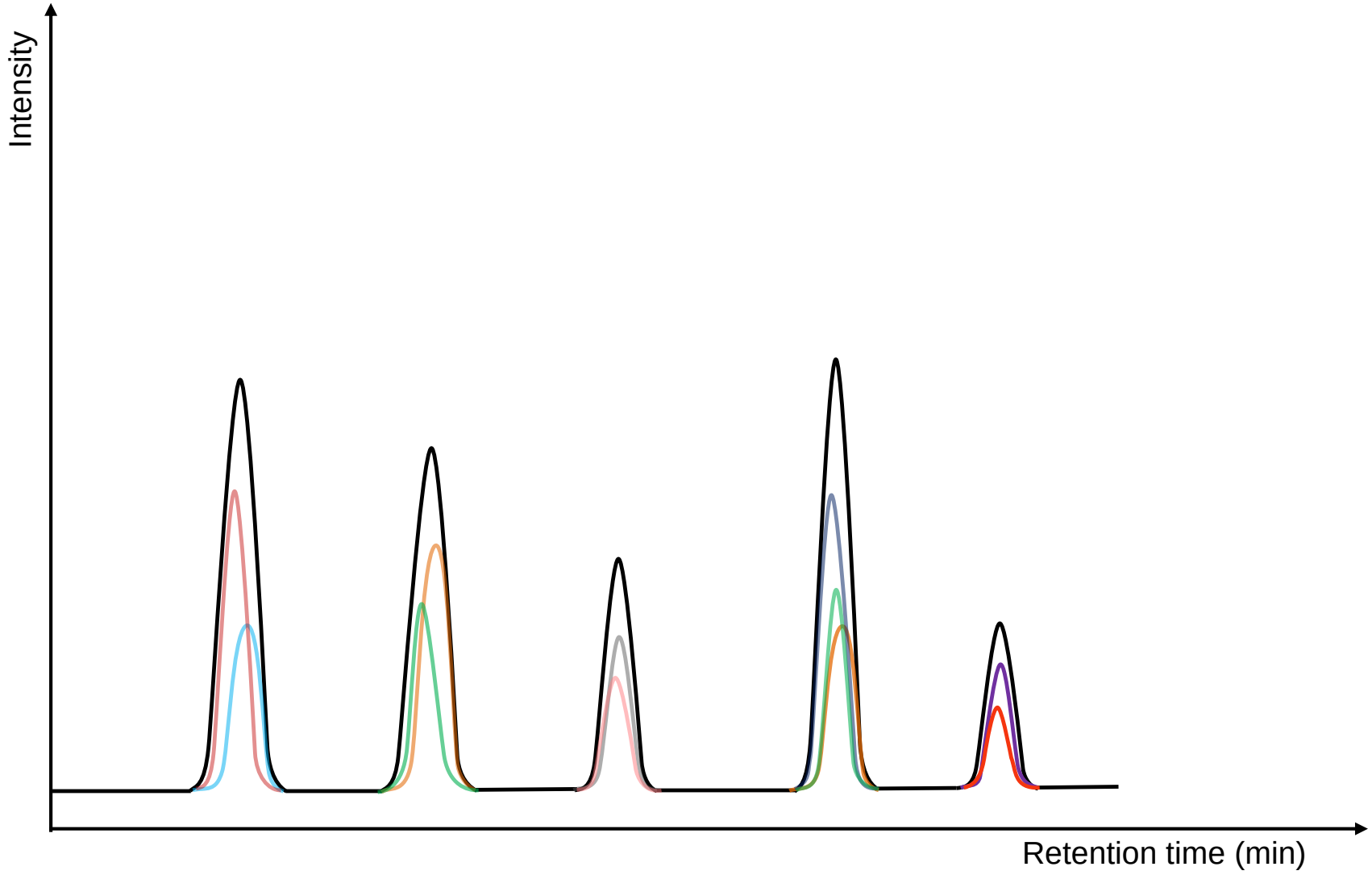
- TOF-DS for 1D GC-TOF
- ChromSpace for GC×GC

ChromSpace

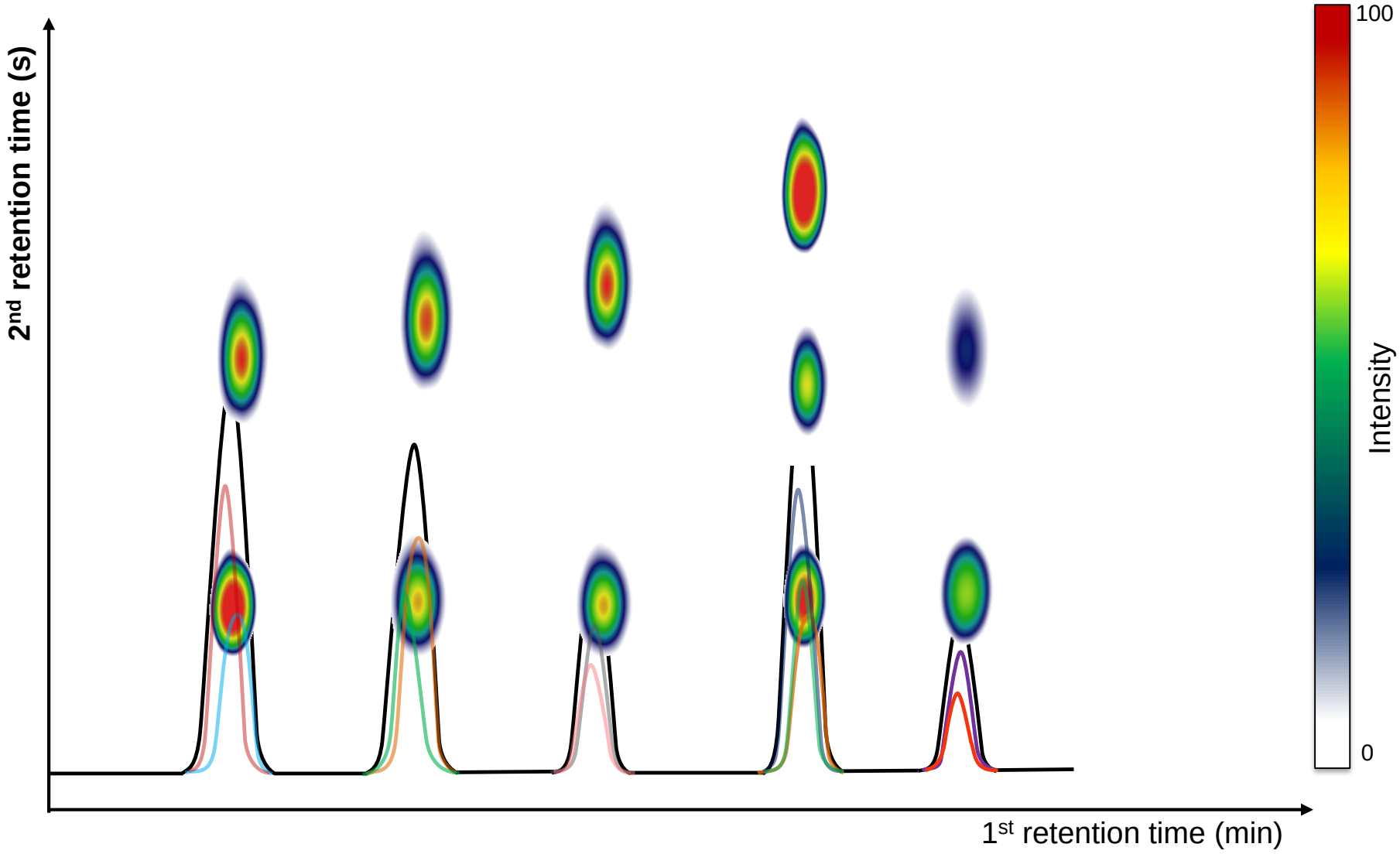
Detection

- TOF mass spectrometry
- FID, SCD, ECD…

Why use GC×GC?

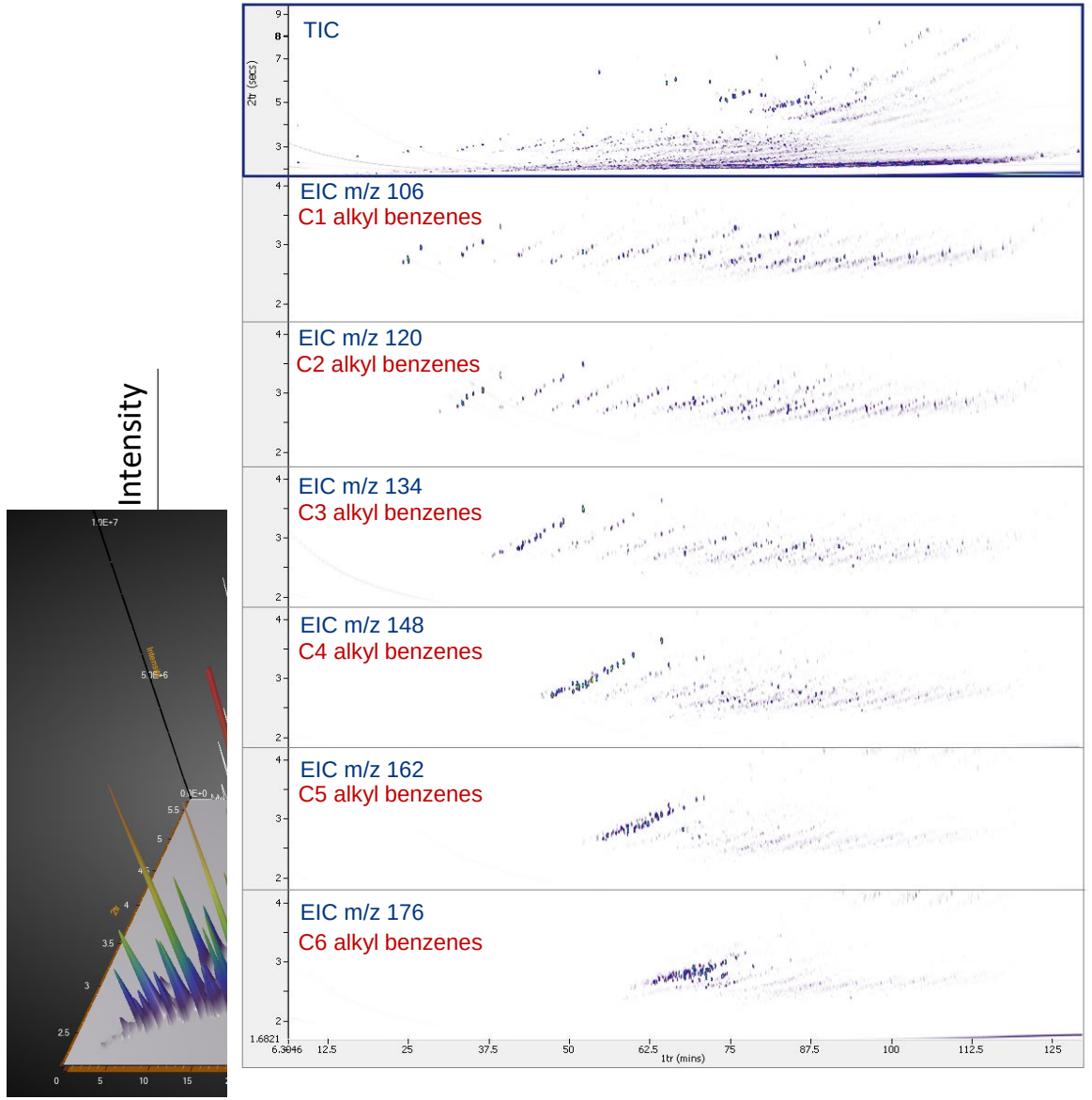


What is GC×GC?



Why use GC×GC?

- Increased separation capacity
- Enhanced sensitivity
- Structured chromatograms



Comprehensive Rules of 2D chromatography

Orthogonality rule

1. The sample is subjected to **two independent** separations.

Conservation rule

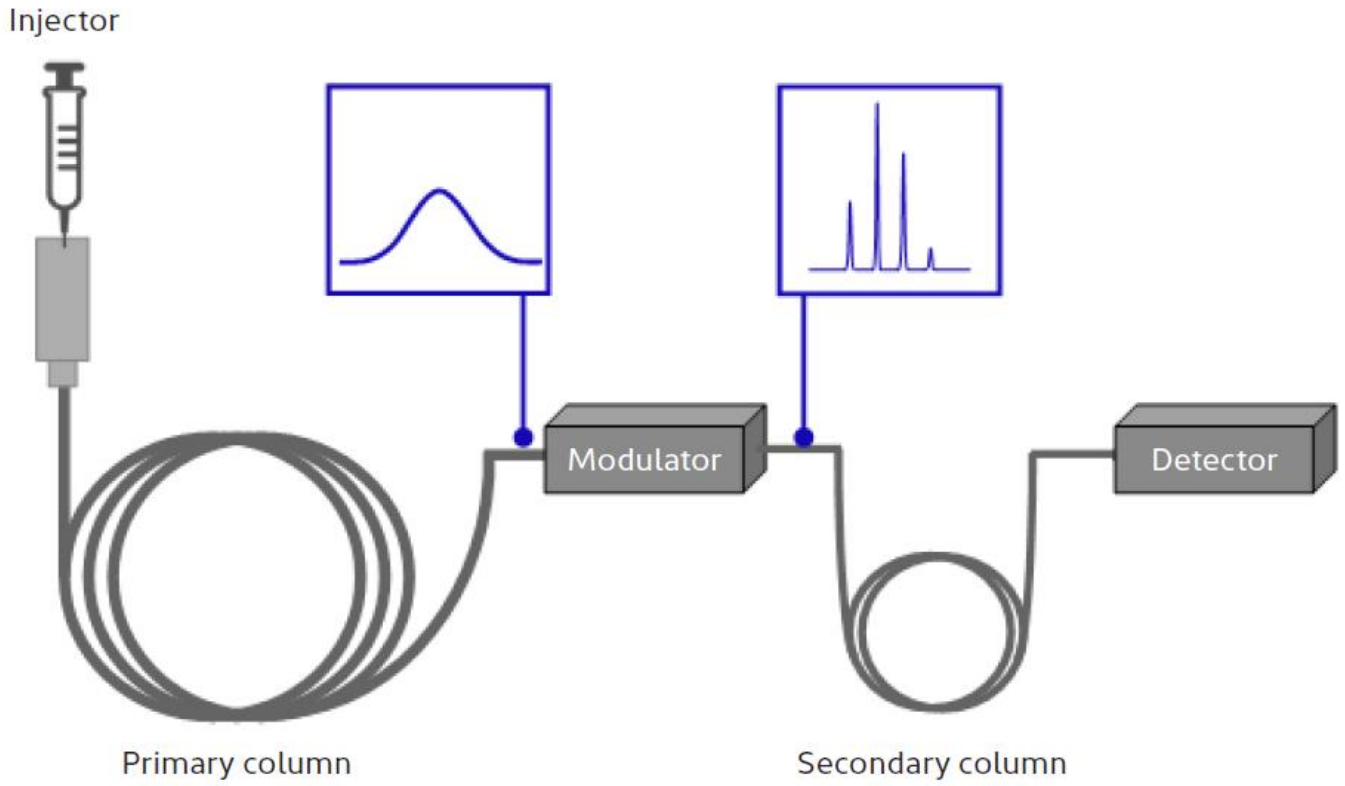
2. The separation from the first dimension is **preserved** throughout the process.

Speed rule

3. The second dimension separation must be significantly faster than the first dimension.

J. C. Giddings Anal. Chem. 1984, 56, 1258A-1270A.

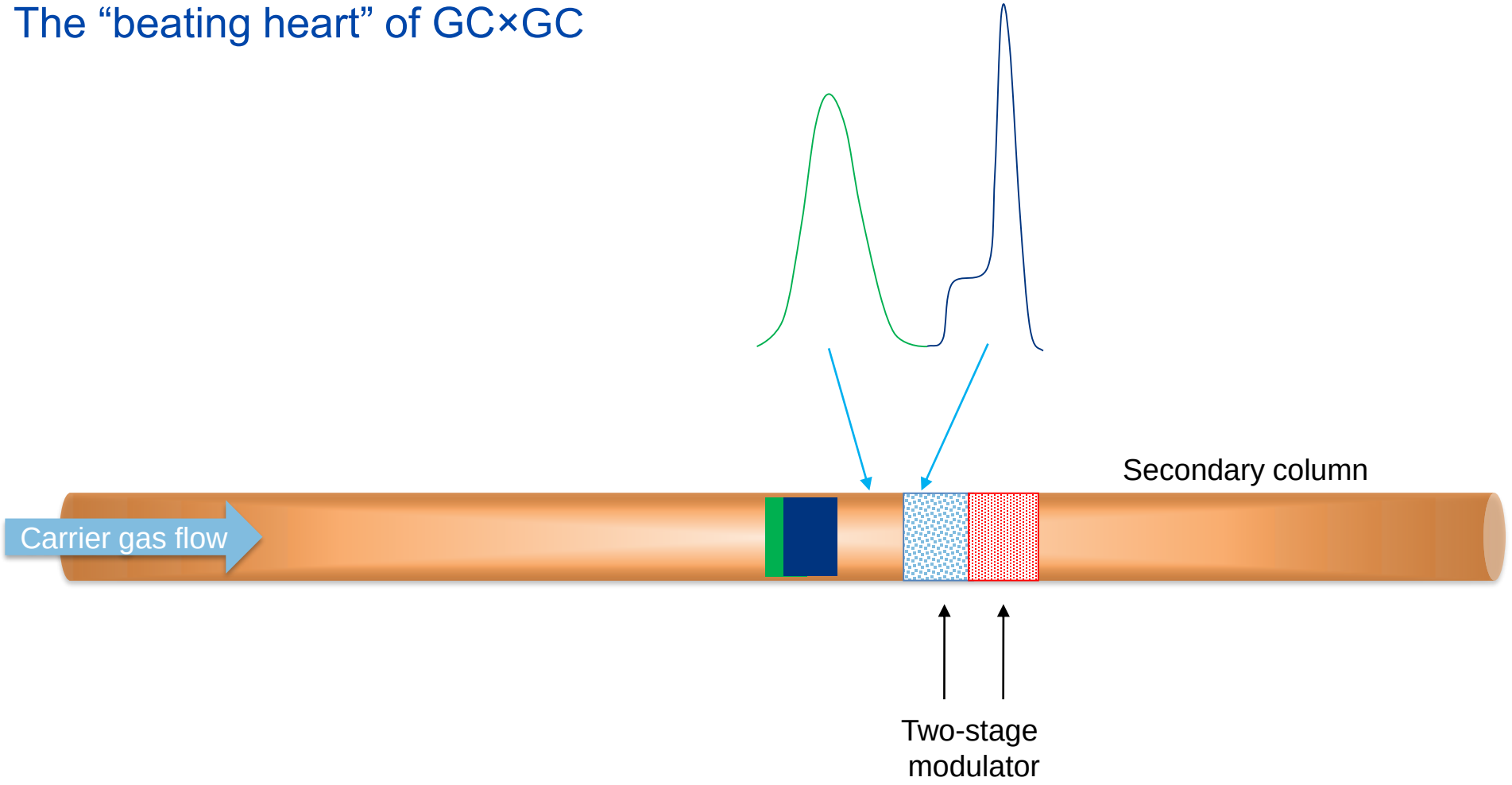
What is GC×GC?



- The modulator is the ‘beating heart’ of a GC×GC system

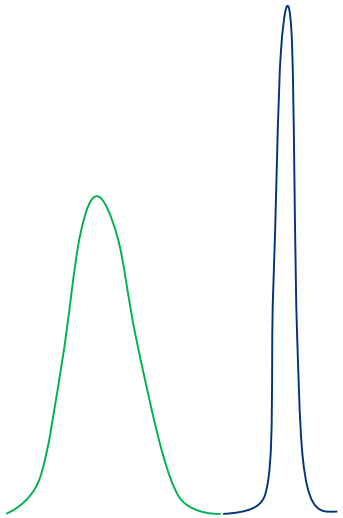
The modulator

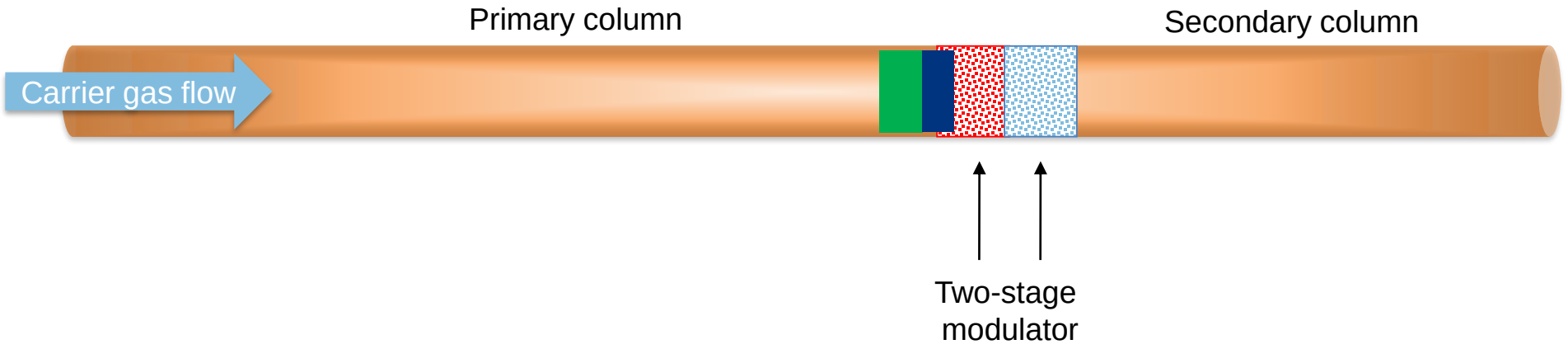
The “beating heart” of GC×GC



The modulator

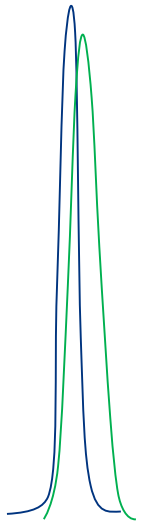
The “beating heart” of GC×GC

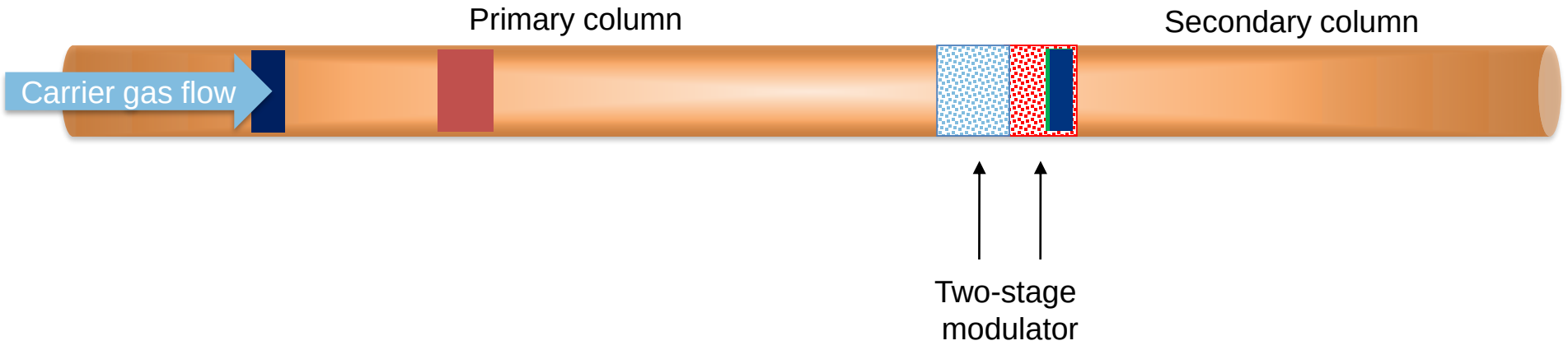




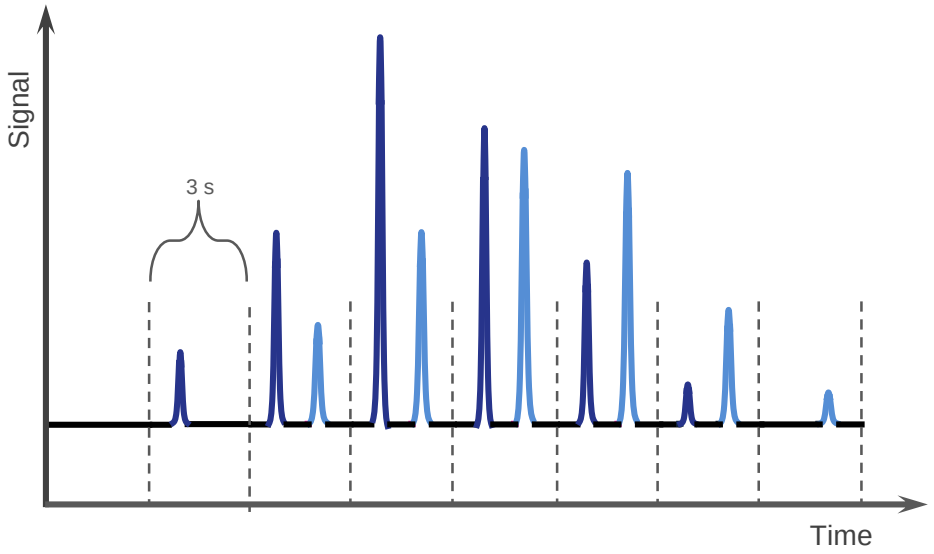
The modulator

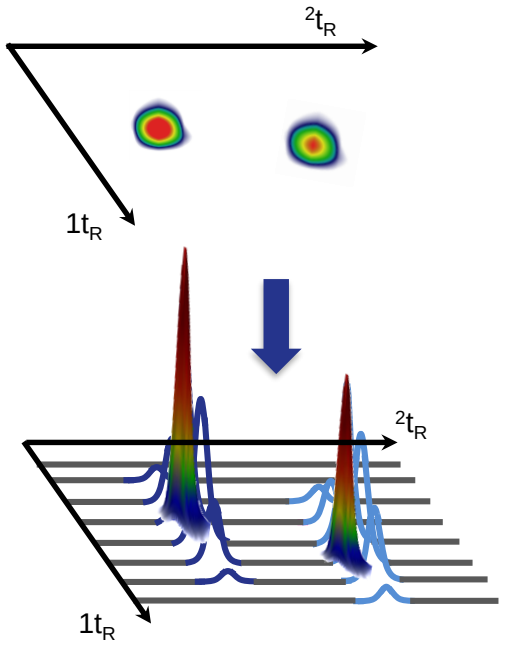
The “beating heart” of GC×GC





The role of GC×GC software





Colour plot

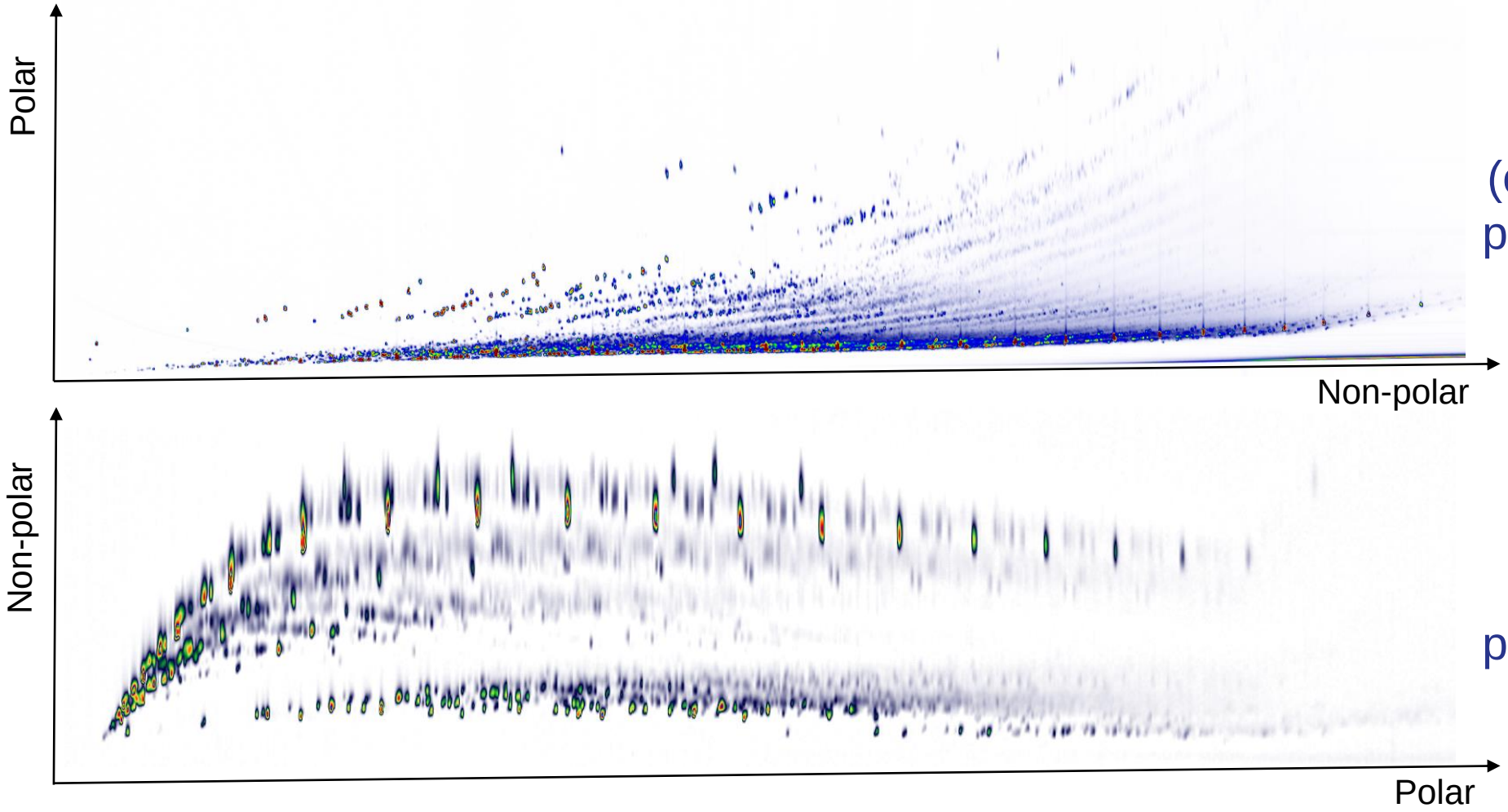
3D Surface chart

Stacked modulation slices

- GC×GC software must merge the sub-peaks for easy visualisation of the data

Choice of column set

Structured ordering (or “roof-tiling” effect)

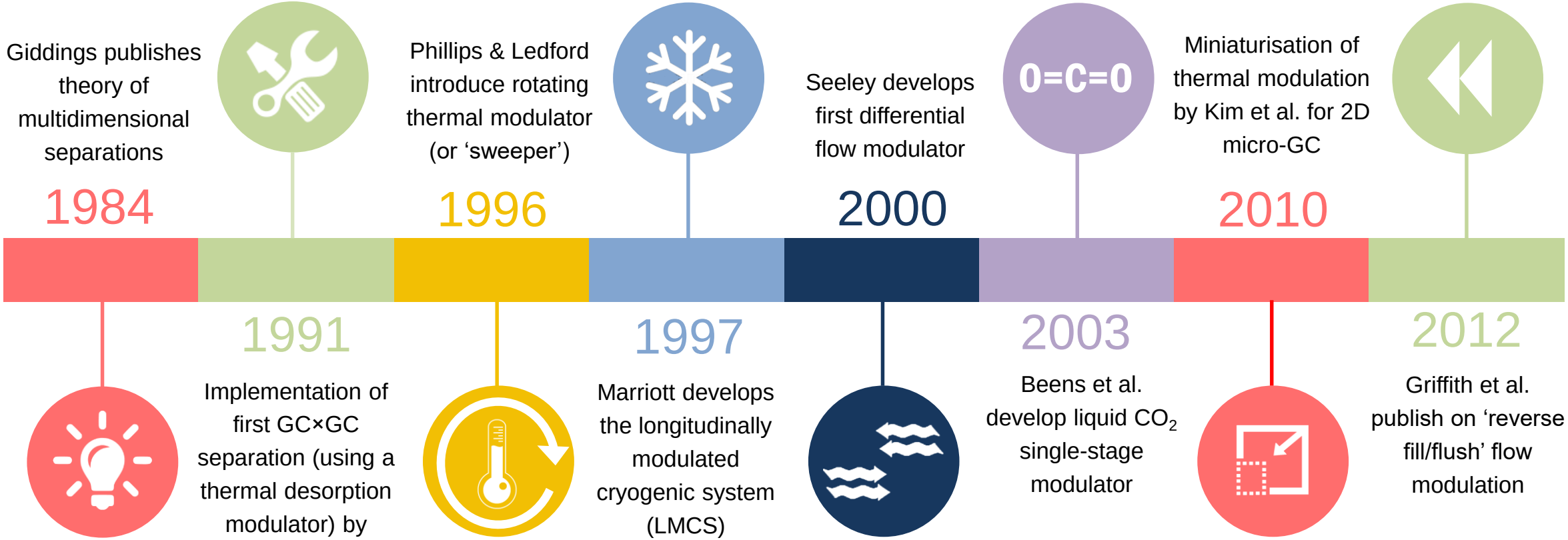


“Normal”
(or conventional)
phase separation

“Reverse”
(or inverse)
phase separation

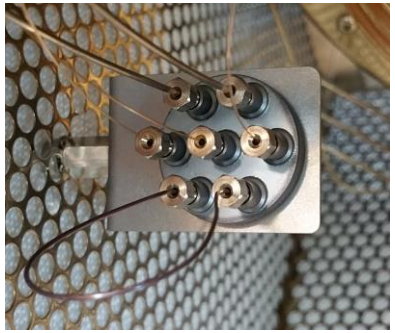
GC×GC modulation

20+ years of innovation



Types of modulator

- Commercial devices use:
  - Flow modulation
    e.g. INSIGHT modulator (SepSolve Analytical)



  - Thermal modulation
    e.g. Delay loop modulator (Zoex)

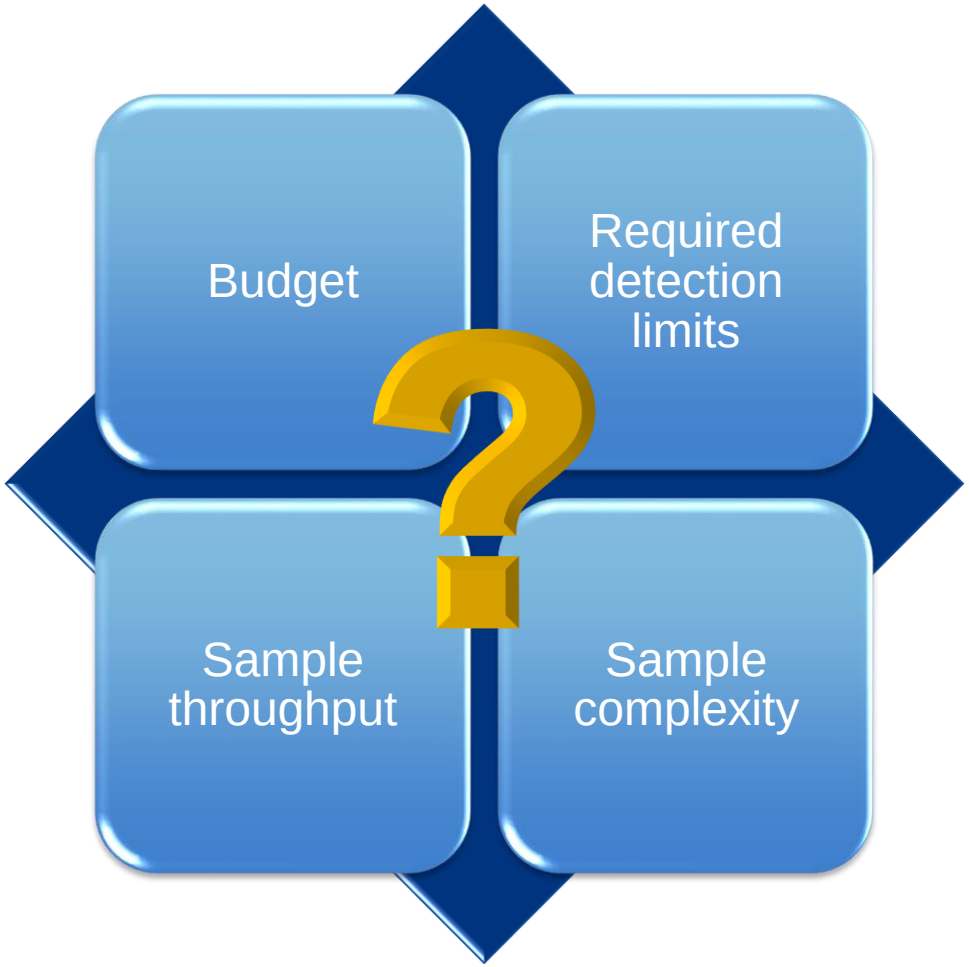
- Both have their own pros and cons – the choice will depend on the application

Modulator pros and cons

	Flow	Thermal
Compound range	No volatility restrictions (modulate from C_1)	Cannot modulate $\leq C_4$ (liquid cryogen needed for $<C_8$)
Cost of ownership	€	~~€€€~~
Repeatability	Peak area RSD routinely < 5%	Variable, peak area RSD 5-10%
Flexibility	Easily configured for heart-cutting, back-flushing and parallel detection	Limited
Sensitivity	Can be restricted if splitting the flow to multiple detectors	Suitable for ultra-trace analyses

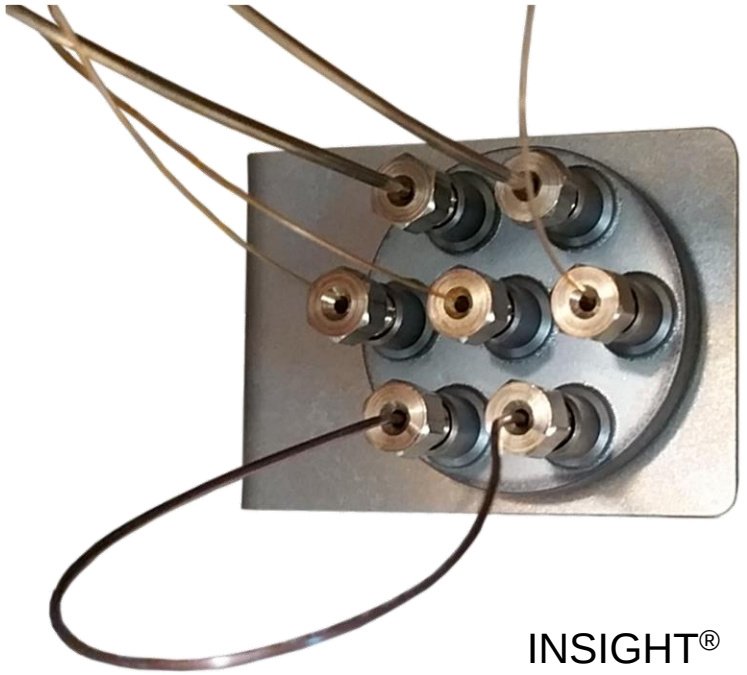
How do I choose a modulator?

Depends on a range of factors:



Benefits of flow modulation

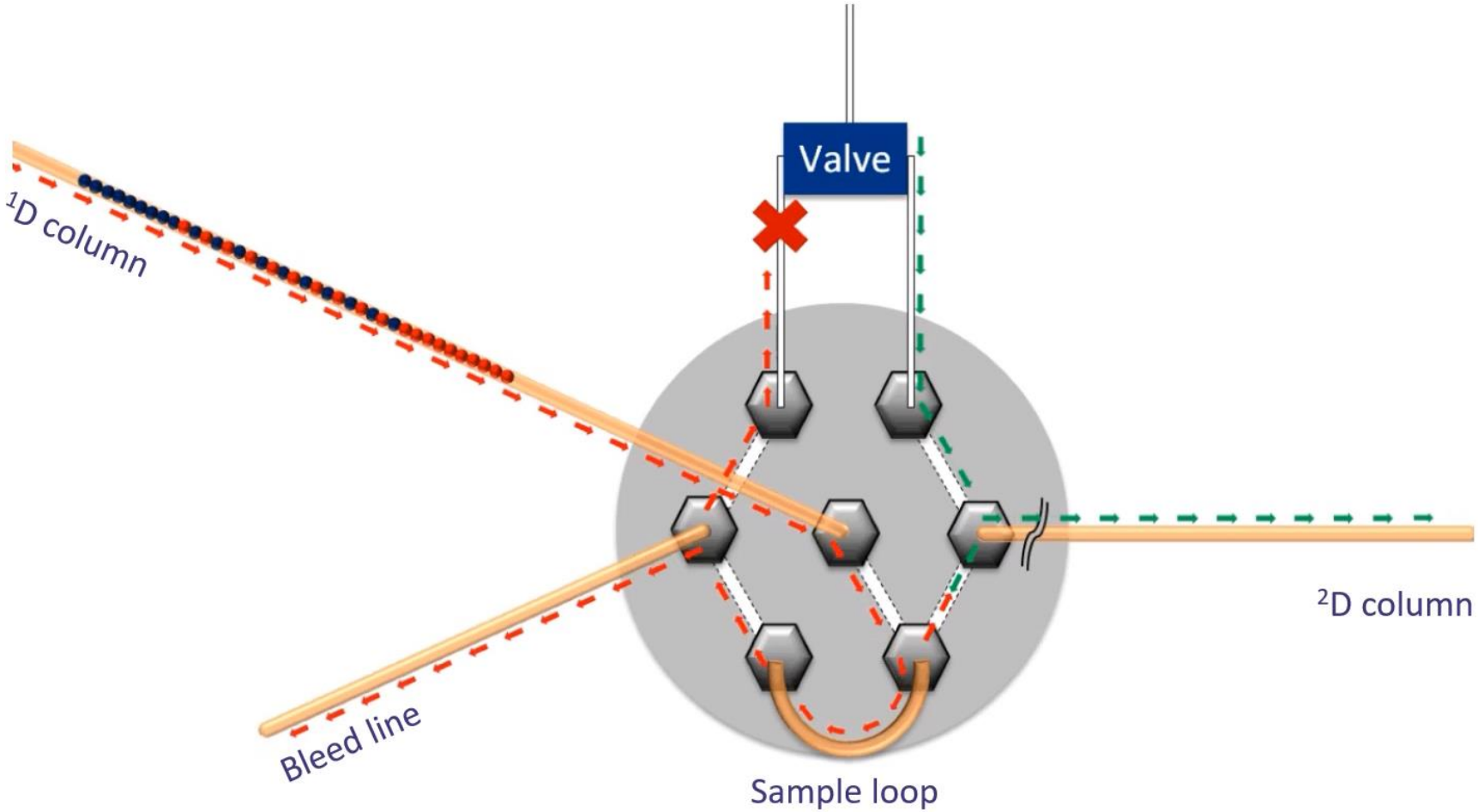
- Consumable-free operation
  - Low running costs
- Efficient modulation of volatiles
  - Extends application range
- Excellent repeatability
  - For routine analyses and large sample batches



INSIGHT®
(SepSolve Analytical)

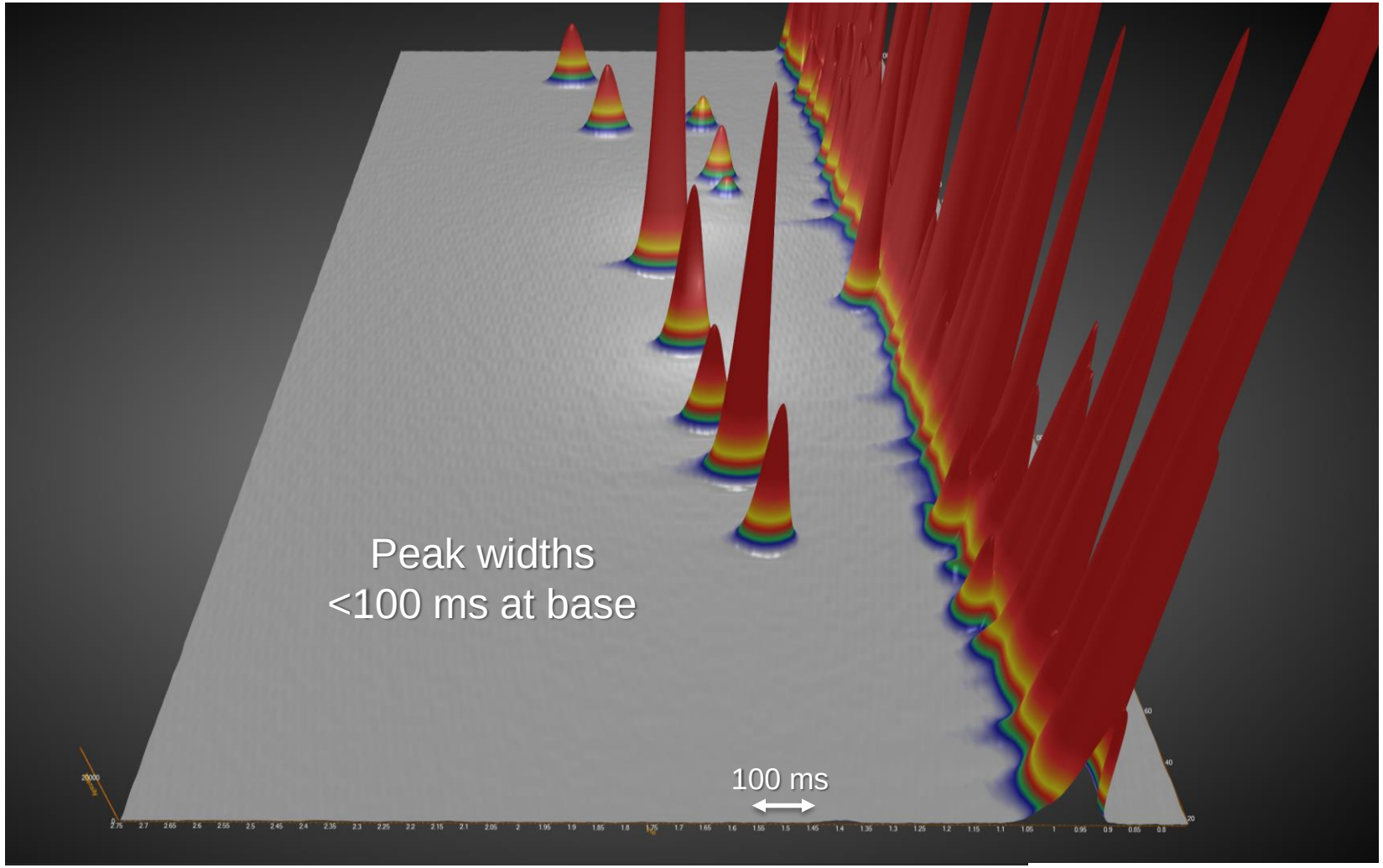
INSIGHT® modulator

How does it work?

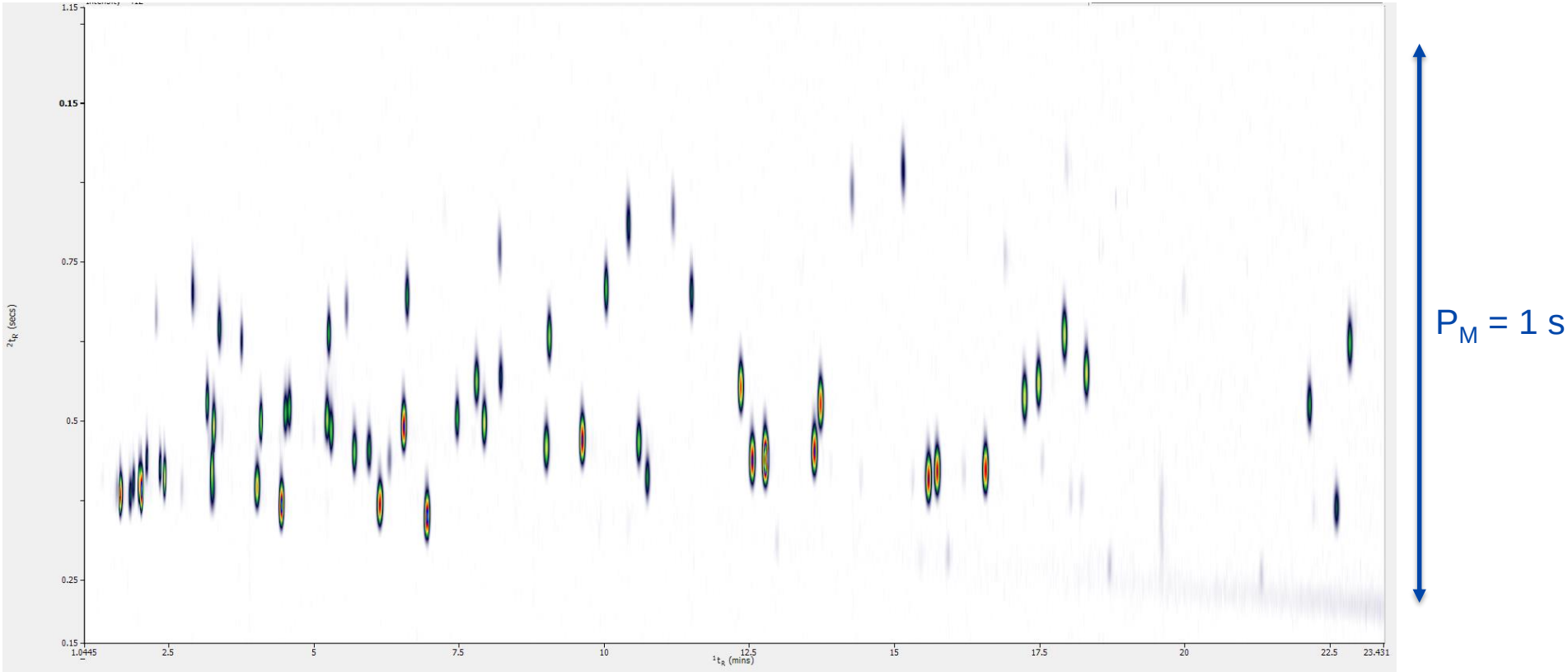


High peak capacity…

…and no tailing

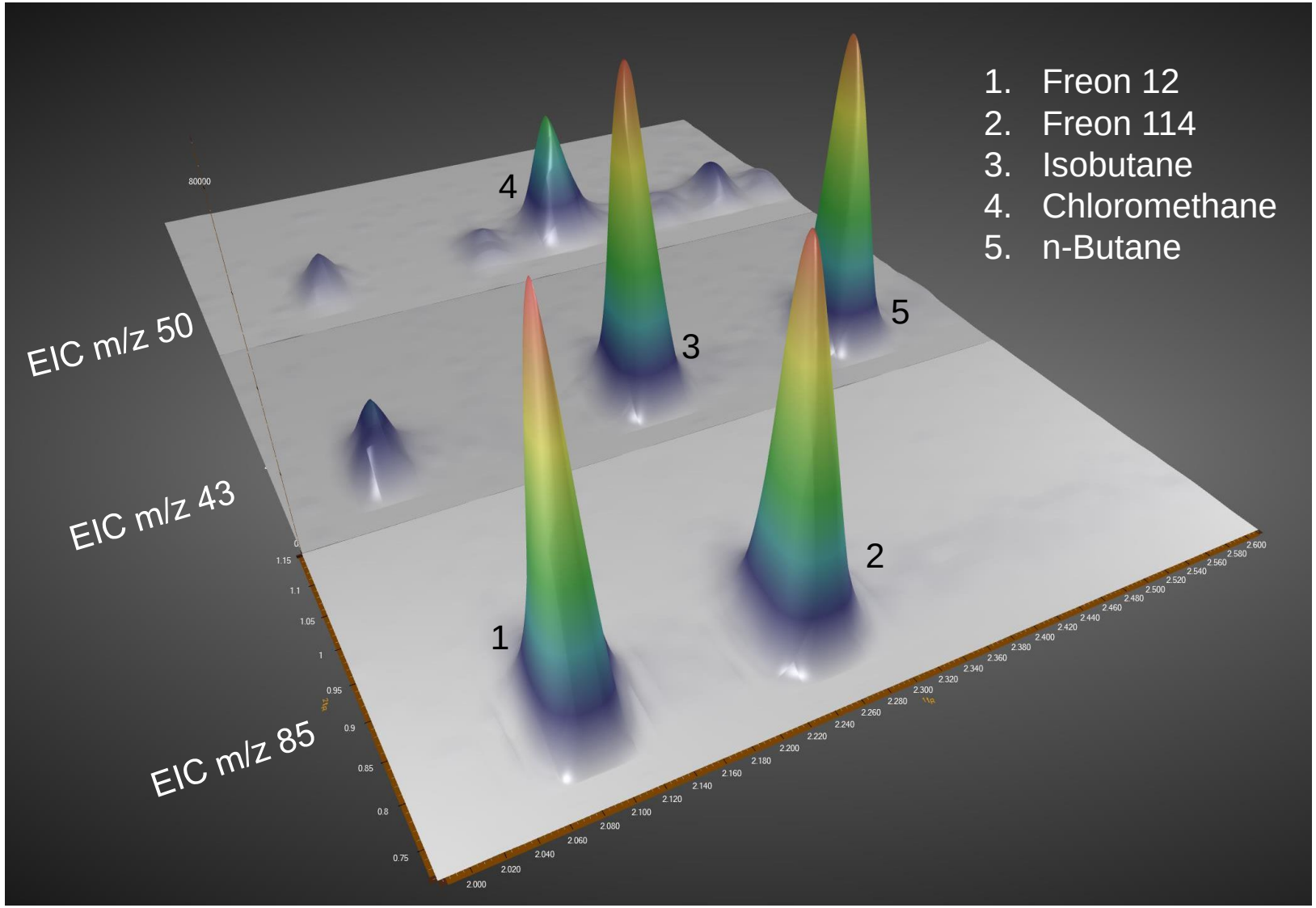


Efficient modulation of volatiles



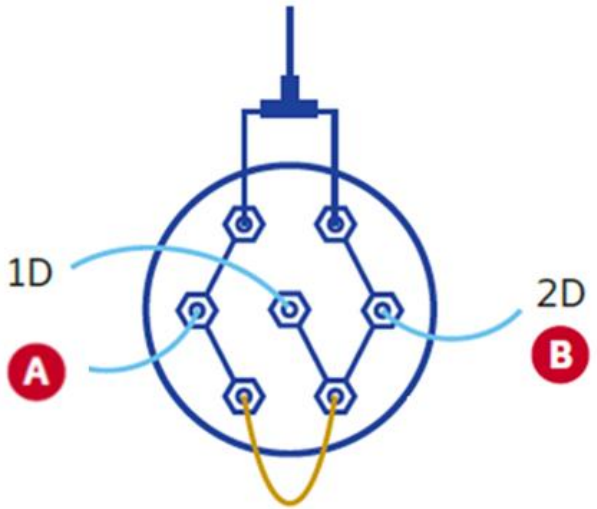
- Flow-modulated GC×GC using INSIGHT for analysis of a gas standard
- Excellent peak shape and peak widths (at base) of ~100-200 ms

Efficient modulation of volatiles



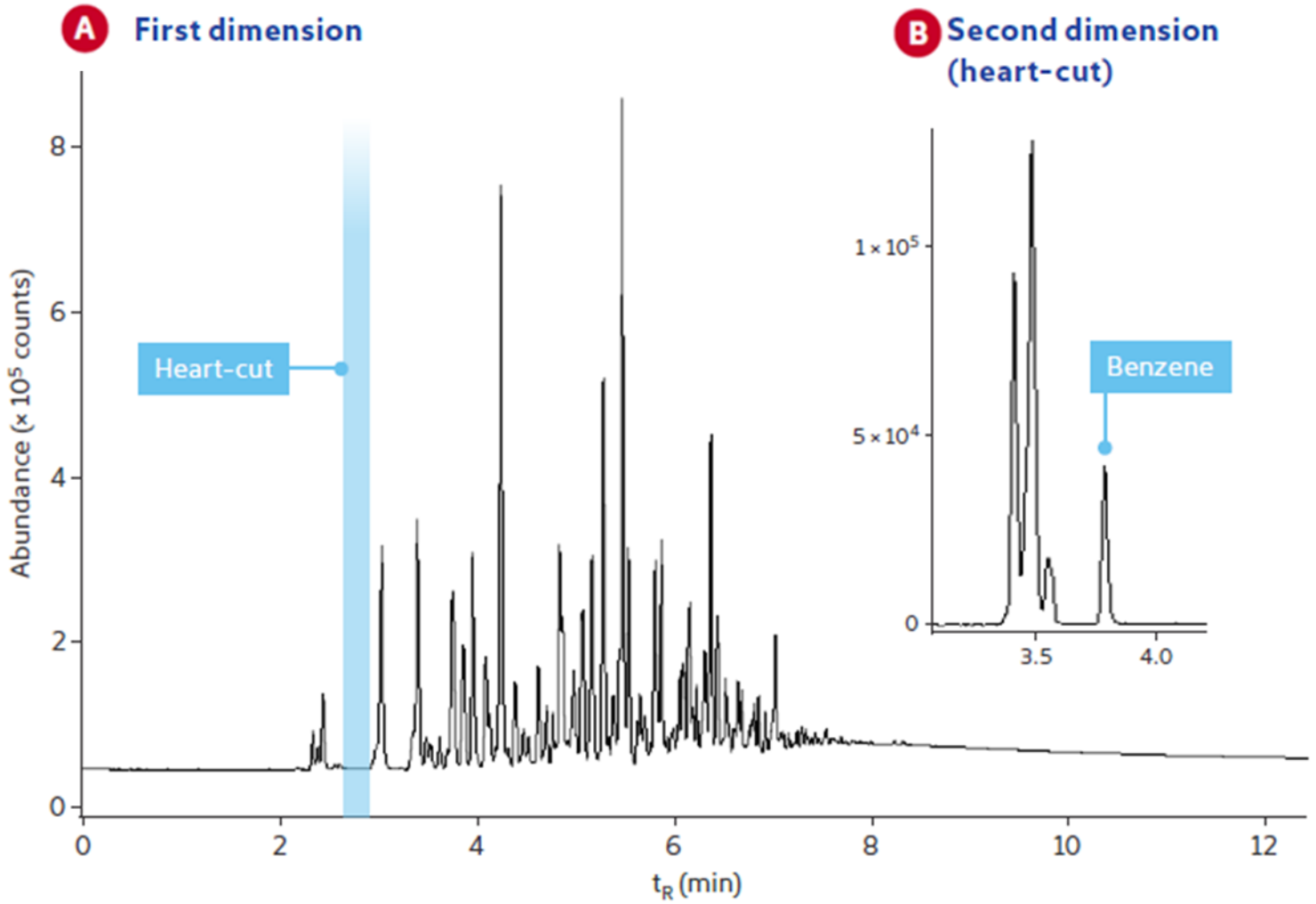
- Flow modulation by INSIGHT has no volatility restrictions
- Excellent peak shape for the 5 most volatile compounds

Heart-cutting capability

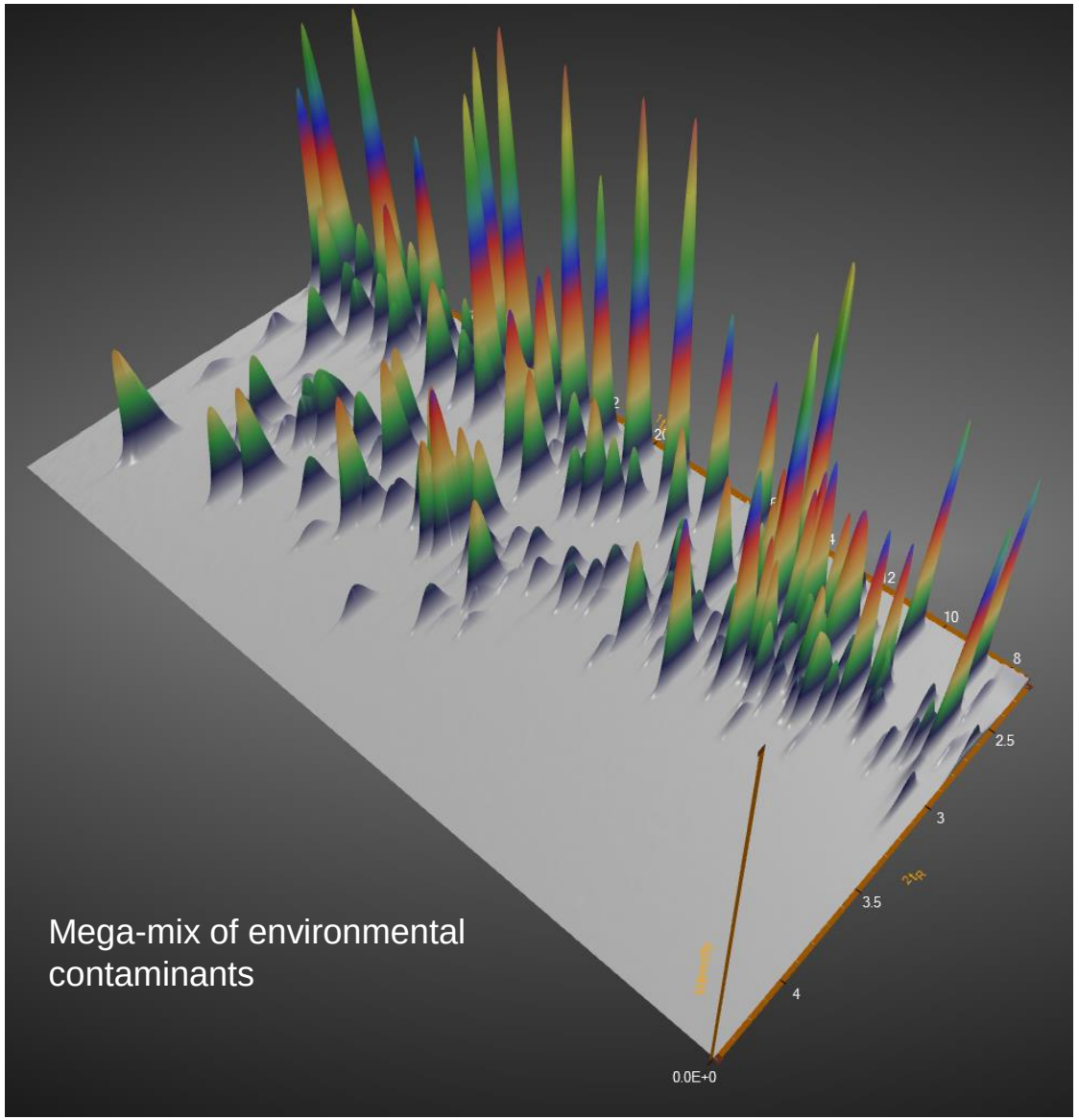


Configure two detectors:

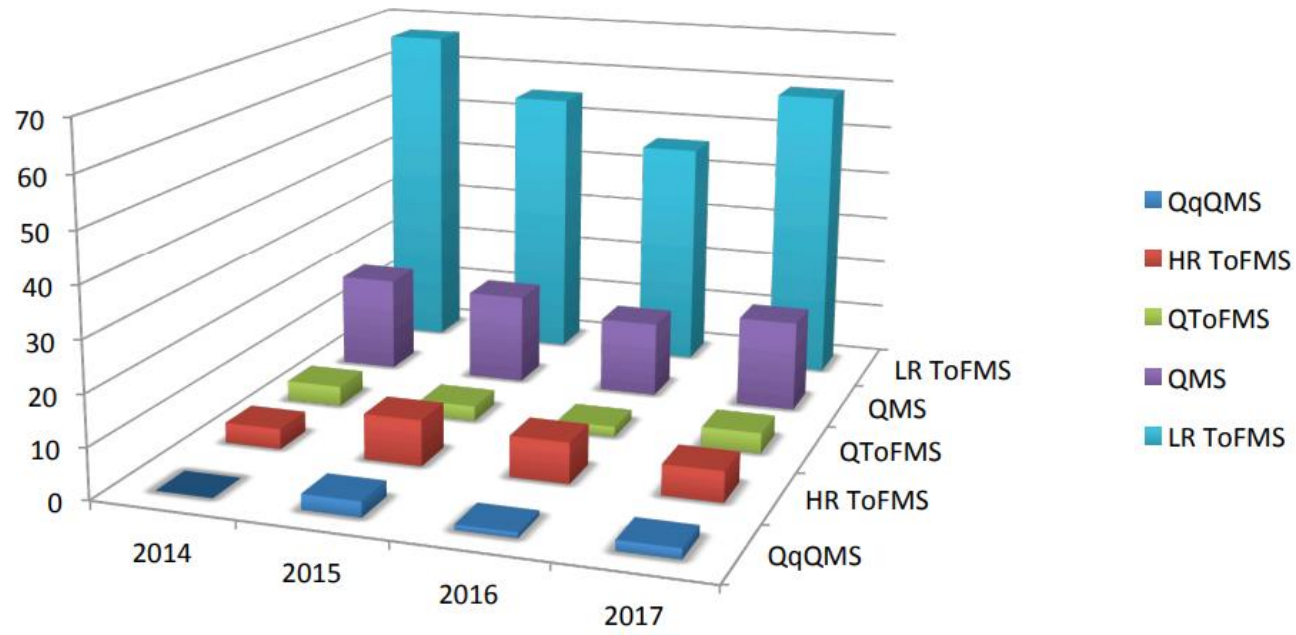
- Majority undergoes 1D separation and sent to detector “A”
- Heart-cut portion sent to second column and detector “B”



Retrofit to existing GC(-MS)



Choice of detector?



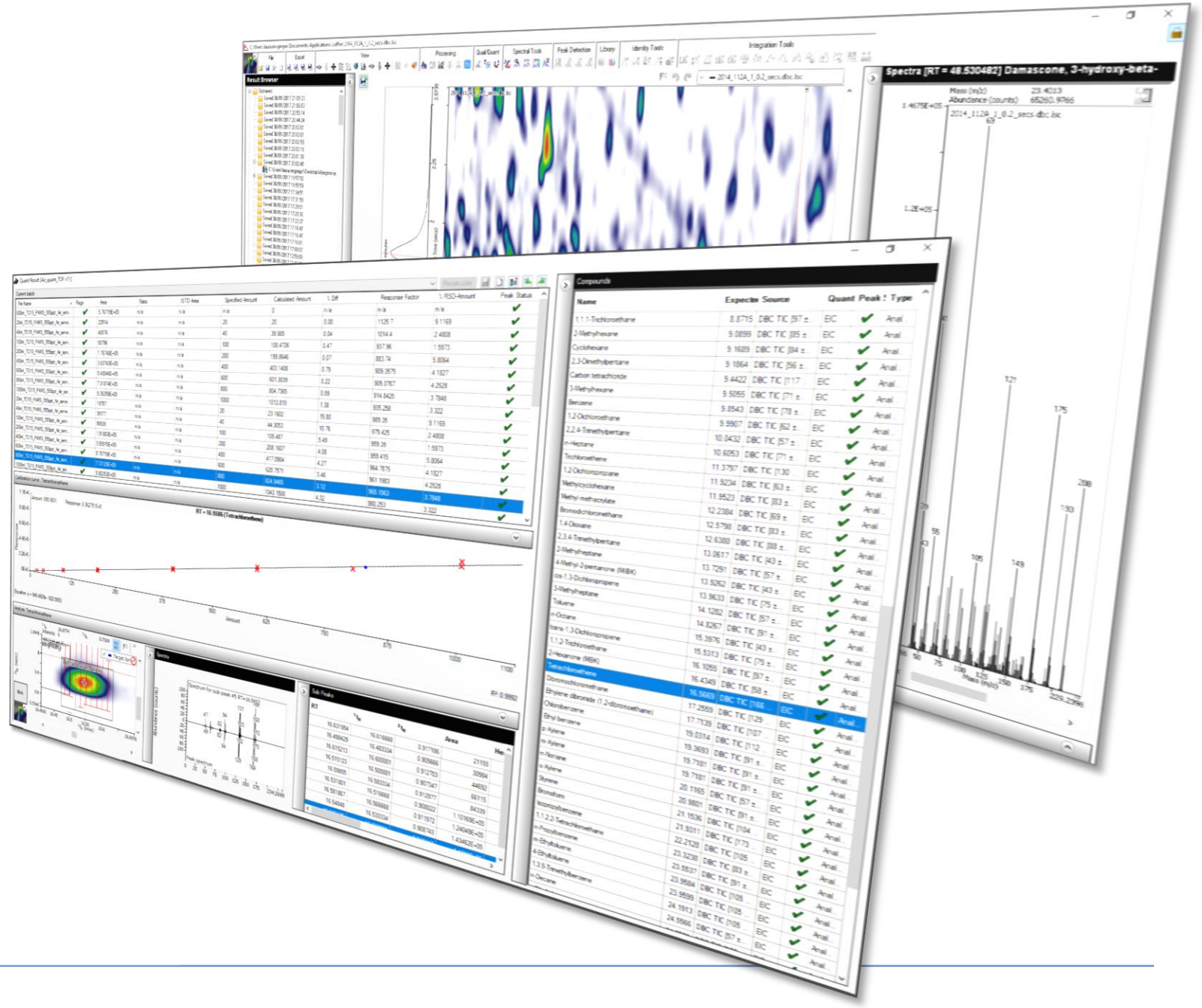
An overview of MS detectors used in published GCxGC studies

(P. Tranchida et al, TrAC Trends in Analytical Chemistry, 2018, 105, 360-366)

- SepSolve deliver flexible systems
  - Time-of-flight MS *(more about this shortly!)*
  - FID
  - And many other single-channel detectors e.g. ECD, SCD….
- Compatible with various detectors
- Parallel detection allows complementary datasets to be obtained simultaneously

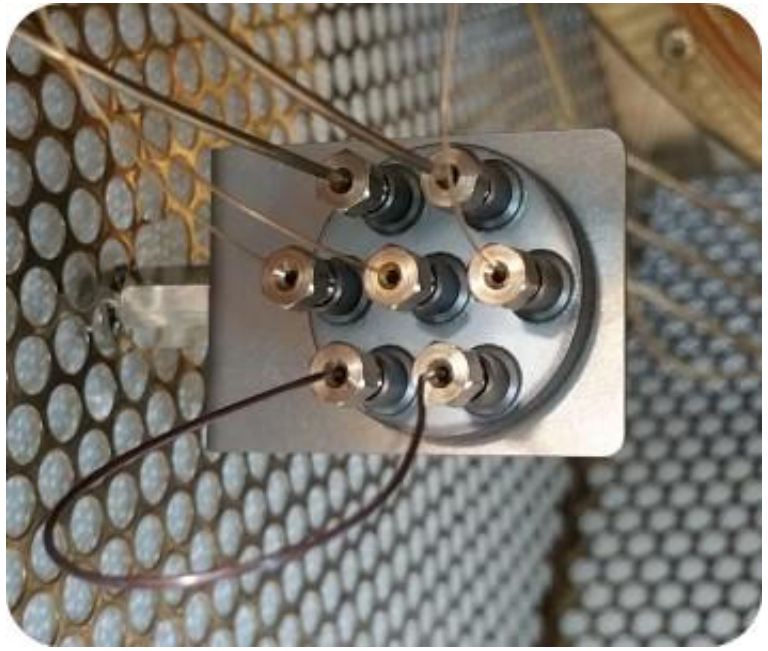
ChromSpace®

- Designed for chromatographers
- Compatible with third-party datafile formats
- Network licensing options now available
- Processing of 1D GC and GC×GC data



Summary

INSIGHT® provides:



- Reverse fill/flush dynamics for:
  - Improved peak shape and peak capacity
- Efficient modulation of both volatiles and semi-volatiles
- Excellent repeatability for large sample batches
  - Rigid retention times
  - Area %RSD routinely <5%
- Simple configuration of:
  - Parallel detection (e.g. FID/TOF MS)
  - Heart-cutting
  - Back flushing
  - Dual-channel configuration

Case studies

Cannabis terpenes

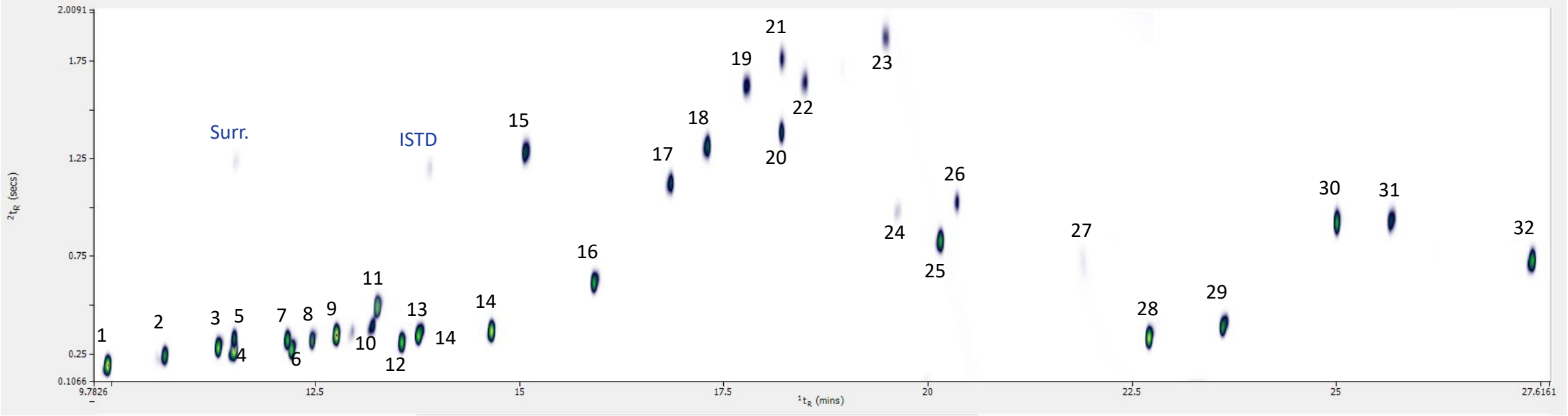
Total petroleum hydrocarbons (TPH)

Challenges

- Over 200 terpenes have been identified in cannabis
- Separation and quantitation of these diverse compounds can be challenging
- Conventional GC–FID or GC–MS results in co-elution of similar compounds or oxygenated derivatives
  - Abundance of important terpenes is over-estimated
  - Poor confidence in data quality

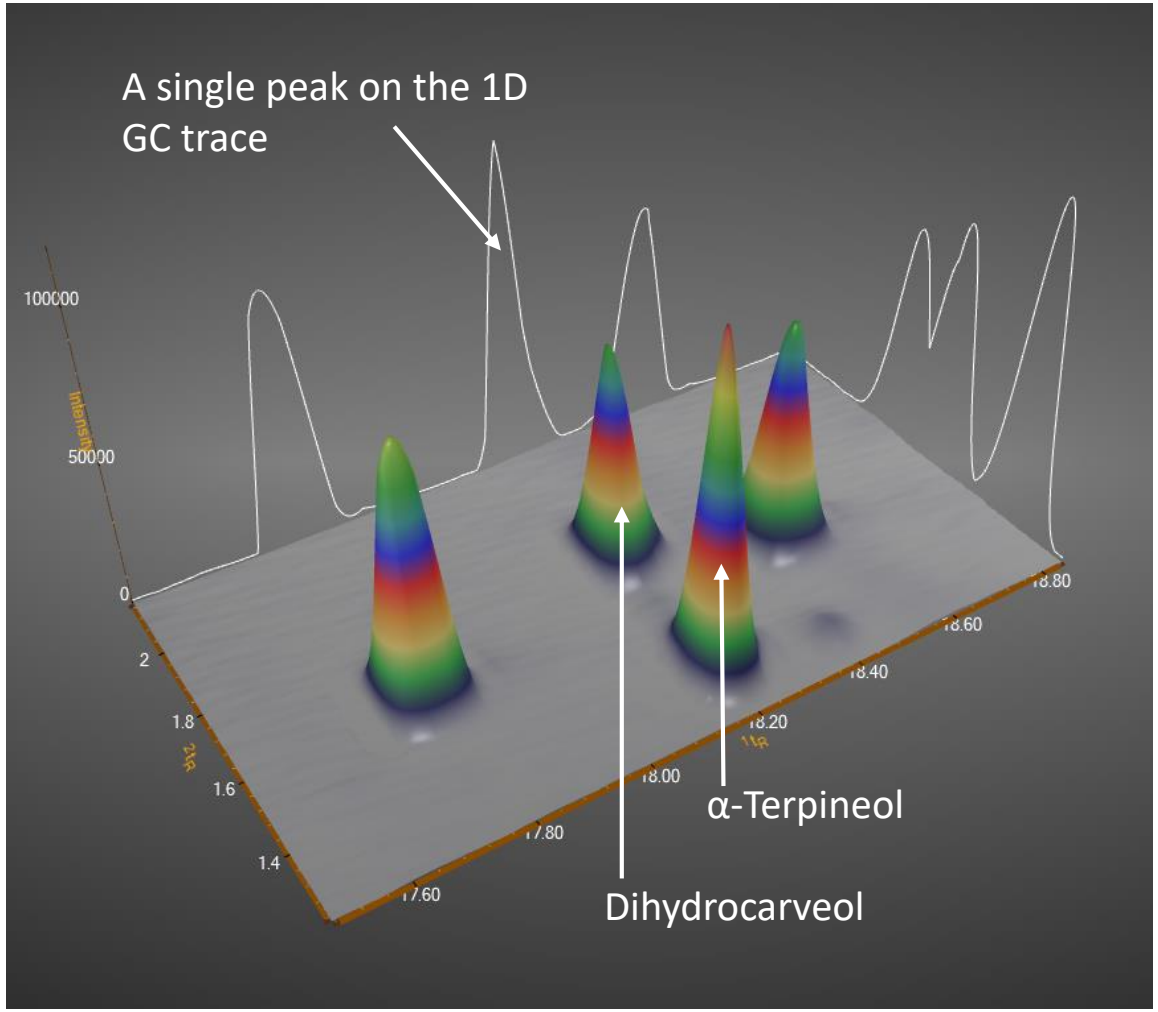
Cannabis terpenes…

…by GC×GC-FID



- Analysis of a standard containing 32 cannabis terpenes plus a surrogate and internal standard

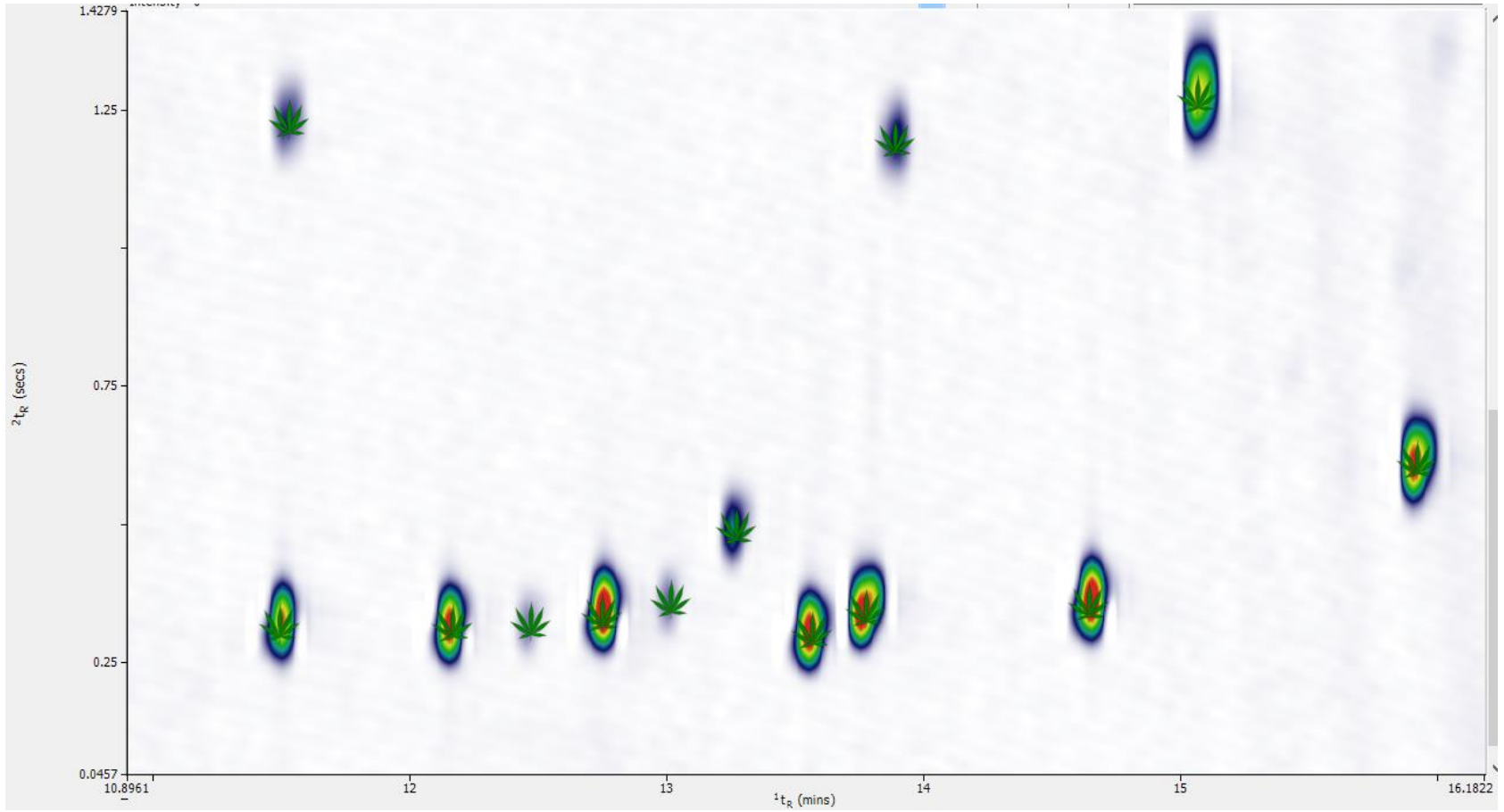
Enhanced separation of GC×GC



- Dihydrocarveol and α-terpineol would have perfectly co-eluted in a 1D GC separation
  - Causing one terpene to be overestimated and the other overlooked.
- Enhanced separation of GC×GC provides increased confidence in terpene profiling
- Without the need for expensive mass spectrometers or complicated deconvolution algorithms

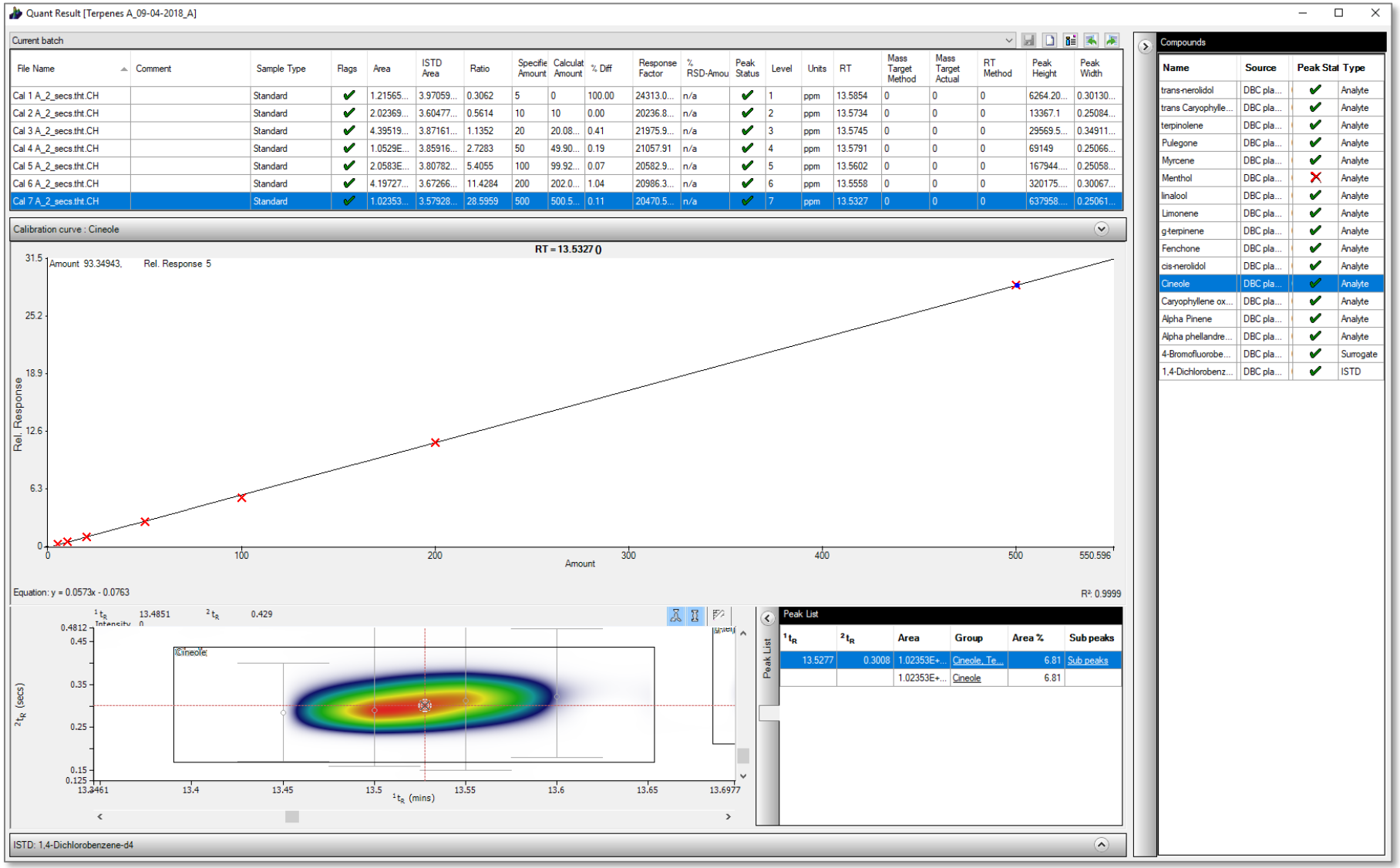
Streamlined software workflows

Just for fun…

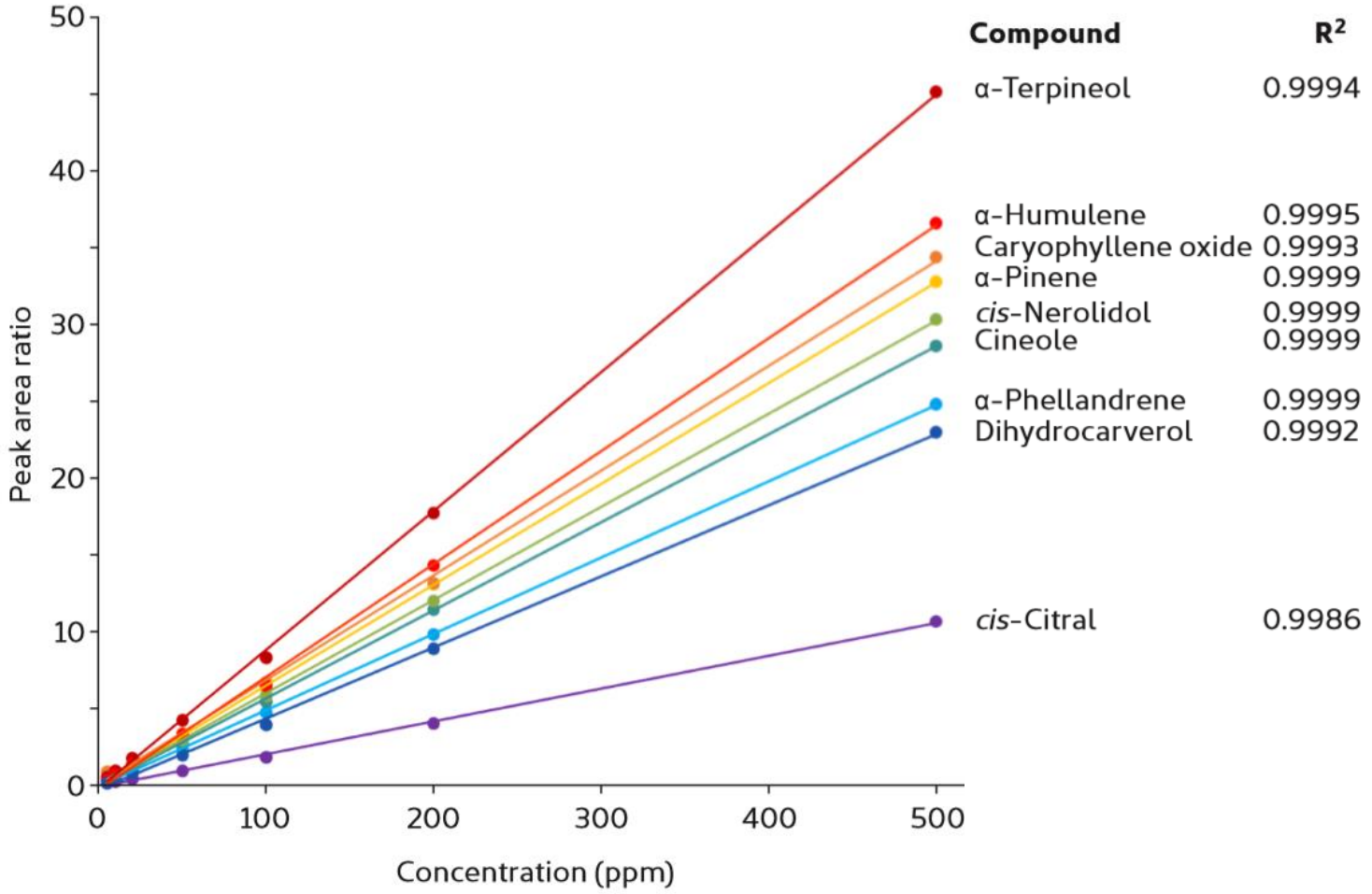


- Peak markers in ChromSpace can be set as <u>any</u> image

Full quantitative workflows



Linearity of GC×GC-FID



- Calibration curves prepared using standards of 5–500 ppm in dichloromethane
- All 32 terpenes displayed excellent linearity with $R^2 > 0.997$

Simple reporting of results

- Fast area percent results through the application of stencils

- Filtered group-type reports

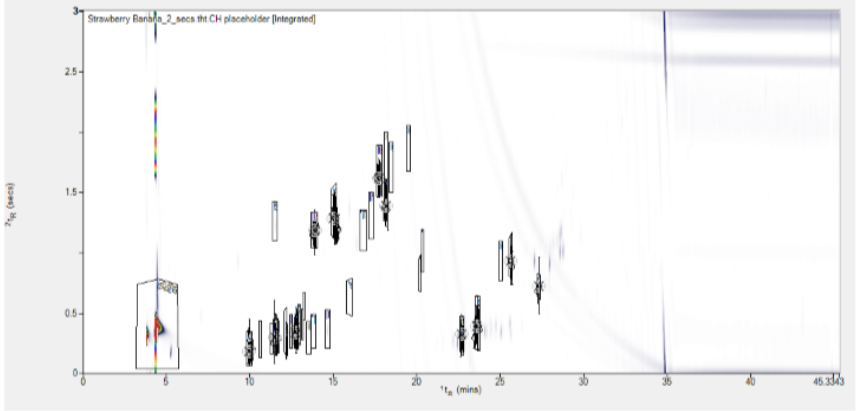
SepSolve Analytical

SepSolve Analytical

SepSolve Analytical
22 Commerce Road
Peterborough
PE2 6LR
UK

Group Type Analysis Report Report Date : - Thursday, November 8, 2018

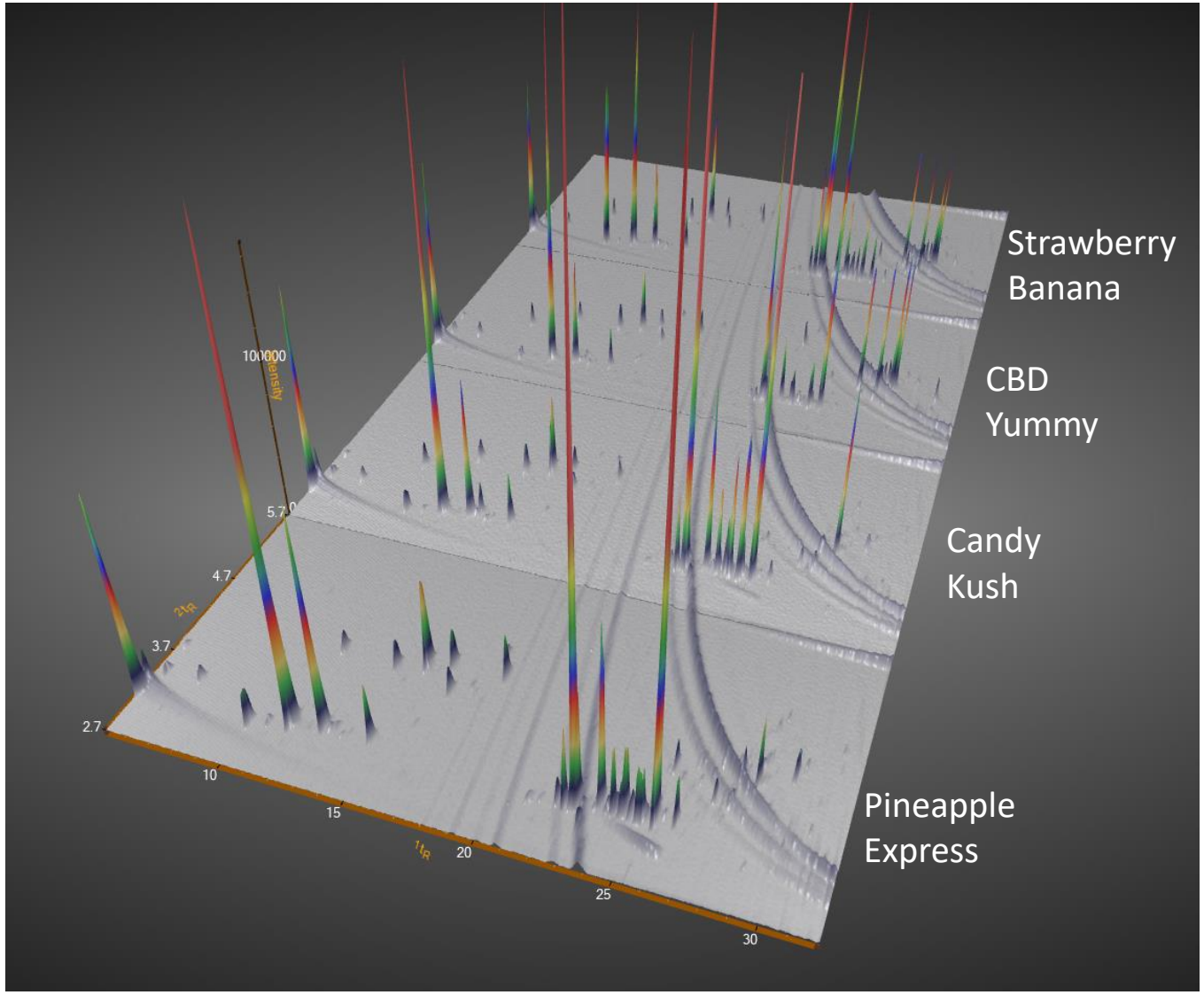
File Name :- C:\Users\laura.mcgregor\Documents\Applications\Cannabis terpenes\New data\Strawberry Banana_2_secs.tht.CH
Import Date :- 12:00:00 AM
Method name :- Cannabis terpenes Nov2018
Method modified :- 08/11/2018 20:28:57



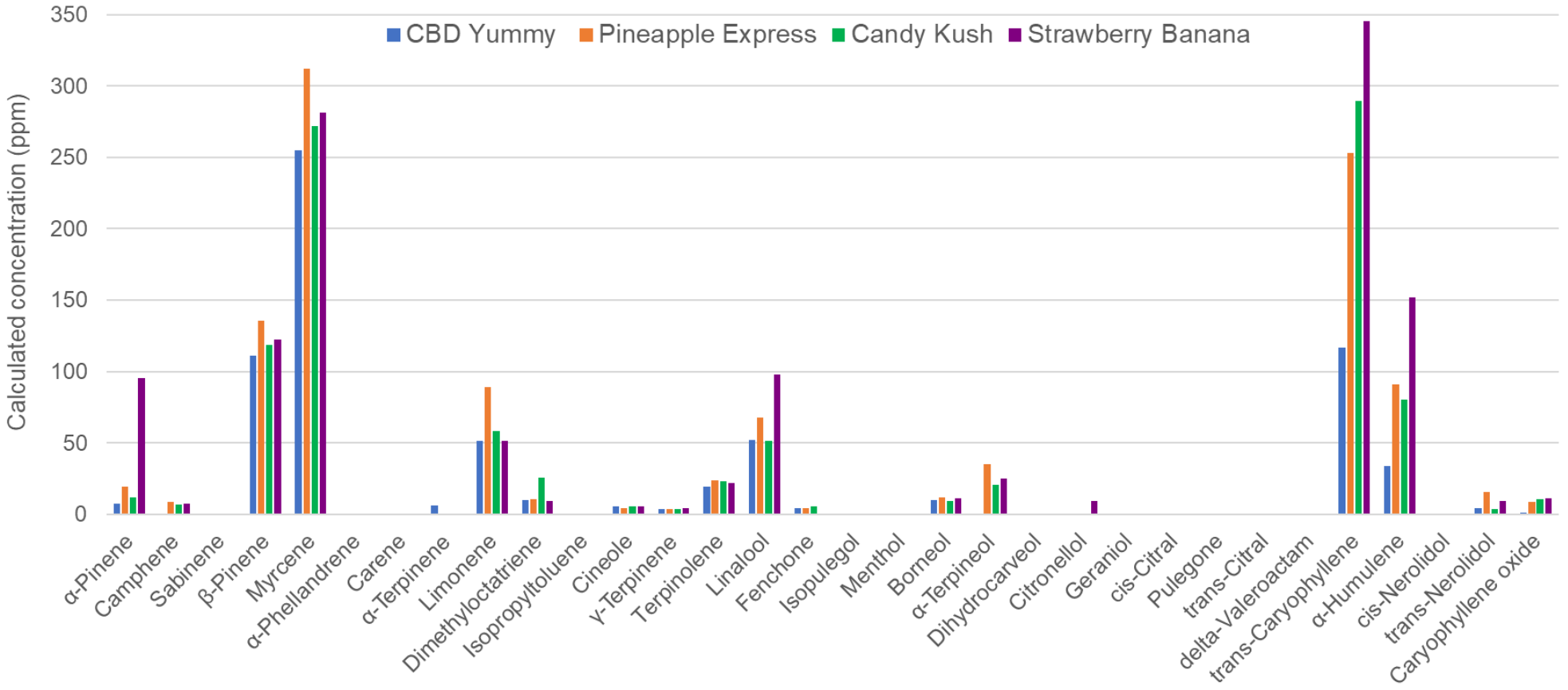
Region	Area	Area %	# peaks
alpha-Humulene	3405939	16.77	1
Camphene	0	0	0
Sabinene	0	0	0
alpha-Phellandrene	0	0	0
alpha-Pinene	1958252	9.64	1
alpha-Terpineol	0	0	0
beta-Pinene	1070679	5.27	1
Borneol	269604	1.33	1
Caryophyllene oxide	214107	1.05	1

Group	Area	Area %	# peaks
Monoterpenes	8177308	40.25	9
Oxygenated monoterpenes	168989	0.83	1
Oxygenated sesquiterpenes	483711	2.38	2
Sesquiterpenes	3405939	16.77	4
Total terpenes	12589259	61.97	16

Comparison of cannabis oils



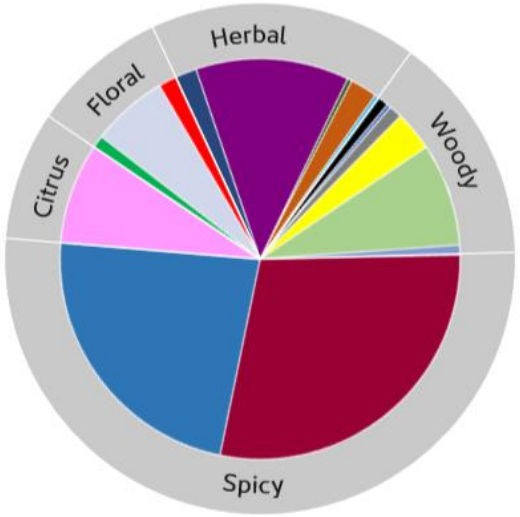
Comparison of cannabis oils



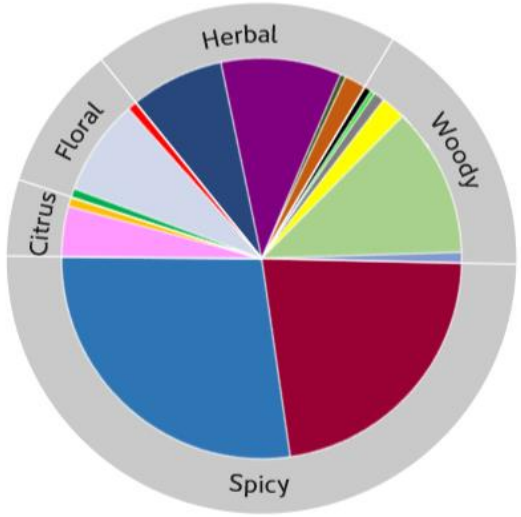
- ‘Strawberry Banana’ has increased levels of α-pinene, trans-caryophyllene and α-humulene, and was the only sample to contain citronelllol
- ‘CBD Yummy’ was the only sample to contain α-terpinene

Improved confidence in aroma profiles

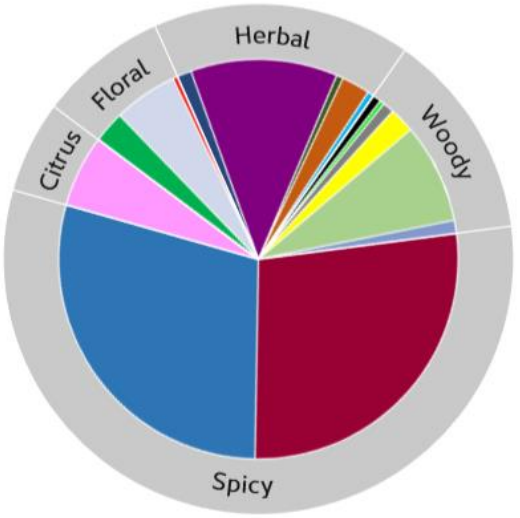
‘Pineapple Express’



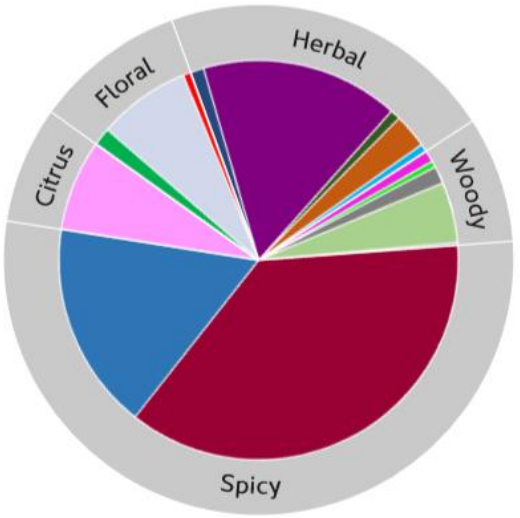
‘Strawberry Banana’



‘Candy Kush’



‘CBD Yummy’



Citrus
- 9: Limonene
- 22: Citronellol

Floral
- 10: Dimethyloctatriene
- 15: Linalool
- 31: *trans*-Nerolidol

Herbal
- 1: α-Pinene
- 4: β-Pinene
- 12: Cineole
- 14: Terpinolene
- 16: Fenchone

Spicy
- 5: Myrcene
- 28: *trans*-Caryophyllene

Woody
- 2: Camphene
- 8: α-Terpinene
- 13: γ-Terpinene
- 19: Borneol
- 20: α-Terpineol
- 29: α-Humulene
- 32: Caryophyllene oxide

Case studies

Cannabis terpenes

Total petroleum hydrocarbons (TPH)

Total Petroleum Hydrocarbons (TPH)

Background

- Commonly split into the Volatile Petroleum Hydrocarbons (VPH) and the Extractable Petroleum Hydrocarbons (EPH)

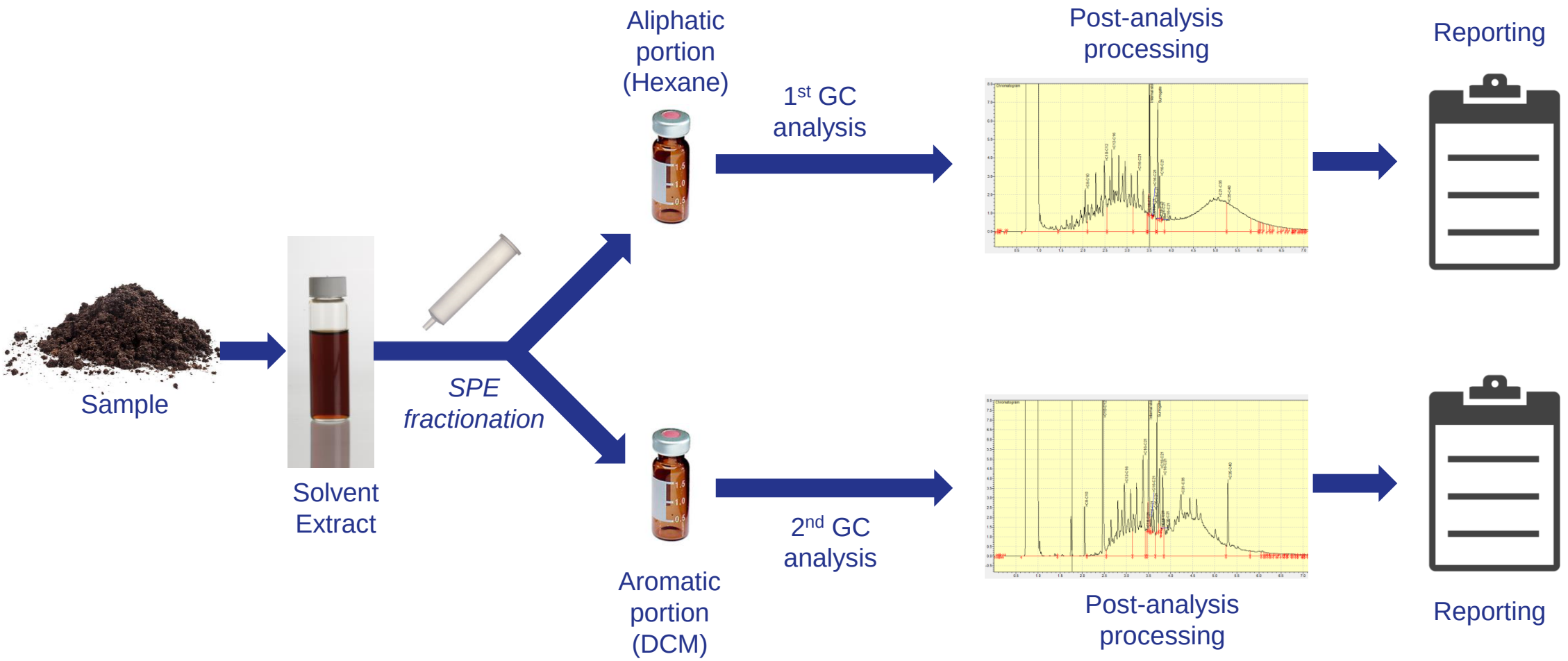
- EPH monitors hydrocarbons from an equivalent carbon number of C_{10}-C_{40} (sometimes C_{44})

- For environmental fate and risk-based analysis the aliphatic and aromatic hydrocarbons must be separated

- Compounds are reported as groups (>C_{10}-C_{12}, >C_{12}-C_{16}…etc) rather than individually

Total Petroleum Hydrocarbons

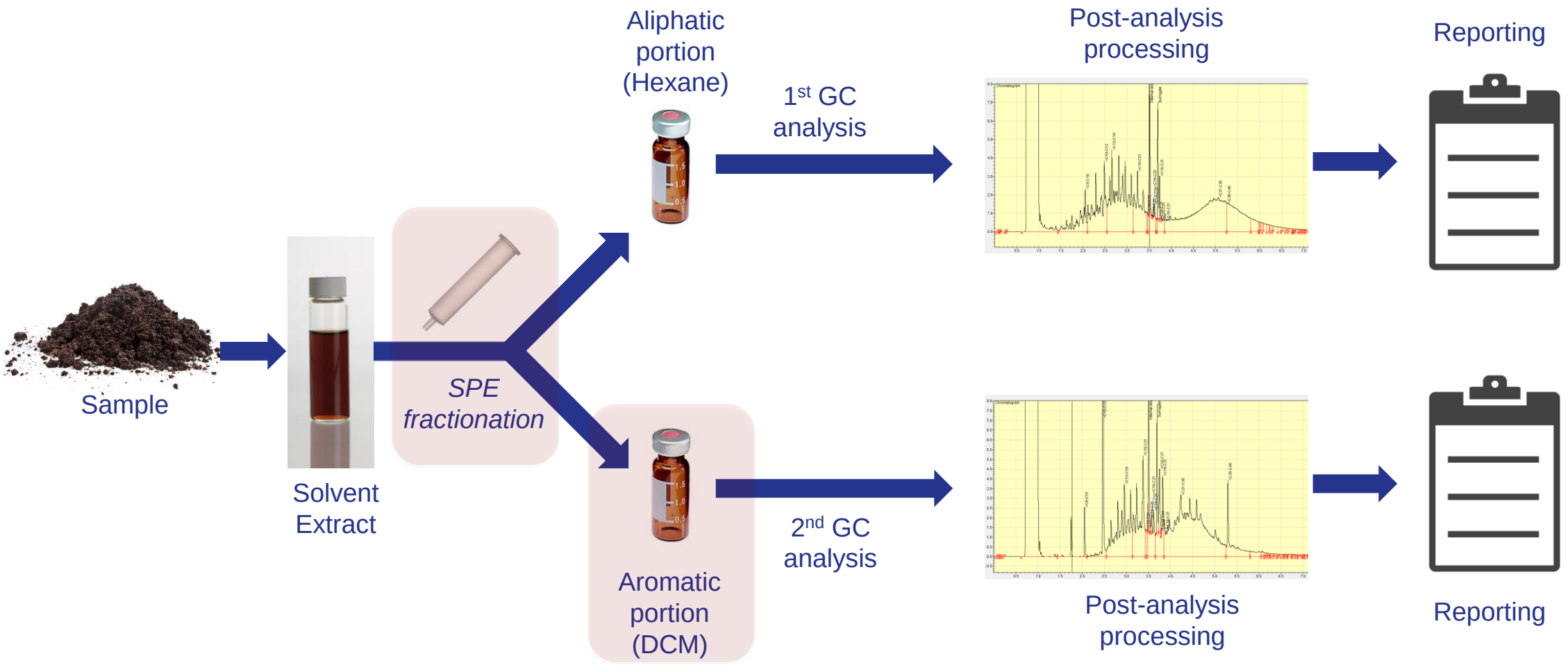
The Traditional Method



Total Petroleum Hydrocarbons

What can we change?

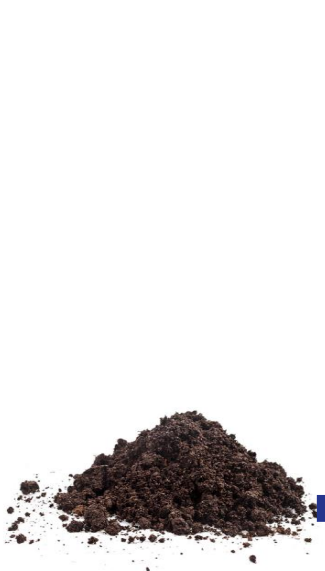
Expensive consumables and waste disposal



Total Petroleum Hydrocarbons

What can we change?

Labour-intensive process



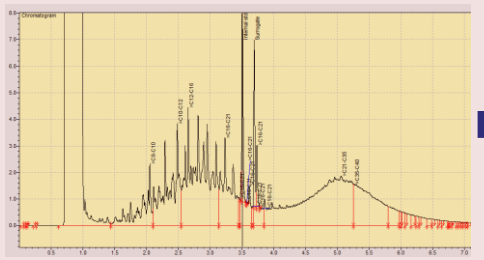
Sample

Solvent Extract

SPE fractionation

Aliphatic portion (Hexane)

1st GC analysis



Post-analysis processing

Reporting

Aromatic portion (DCM)

2nd GC analysis

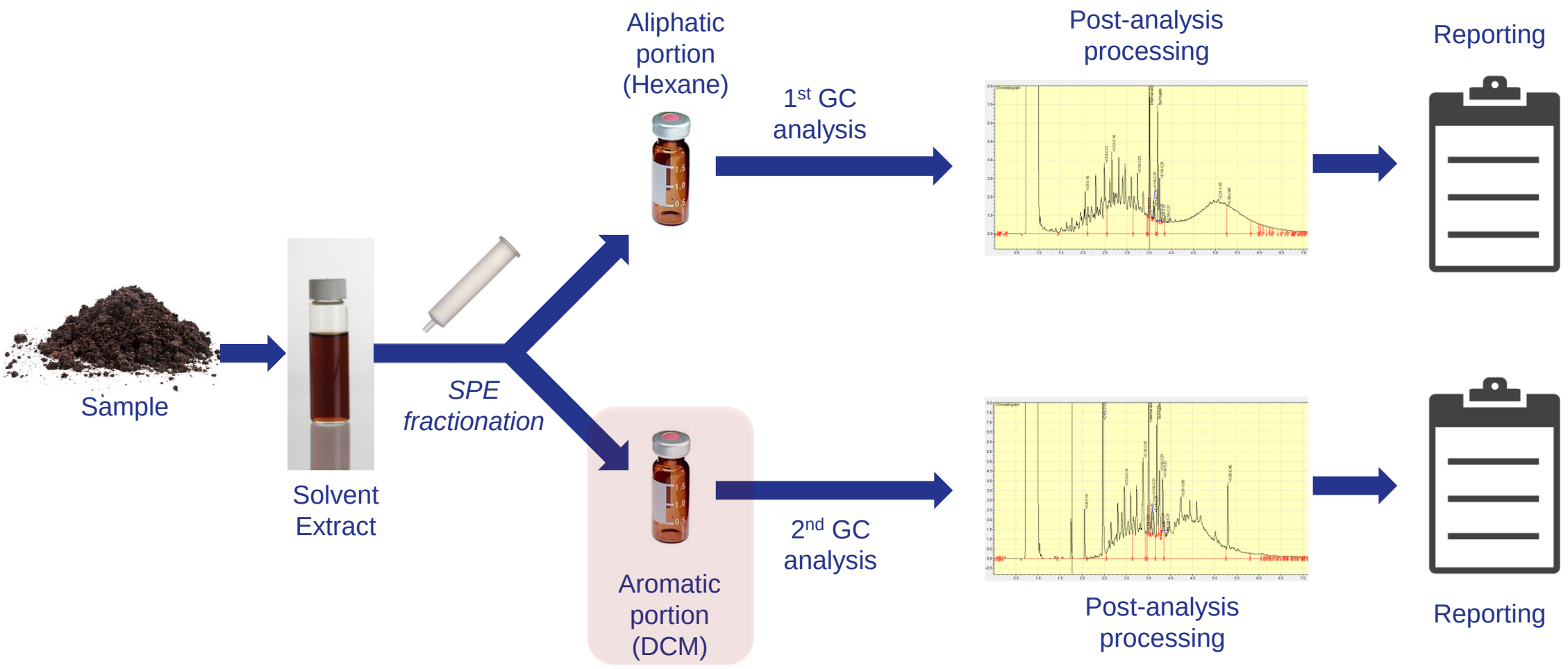
Post-analysis processing

Reporting

Total Petroleum Hydrocarbons

What can we change?

Health and safety concerns



A new approach to TPH…

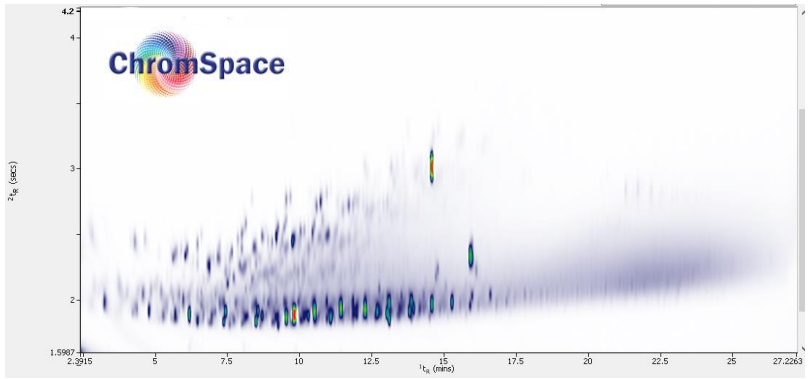
…using GC×GC-FID

- Chromatographic separation of aliphatic and aromatic hydrocarbons in a single run, reducing processing time

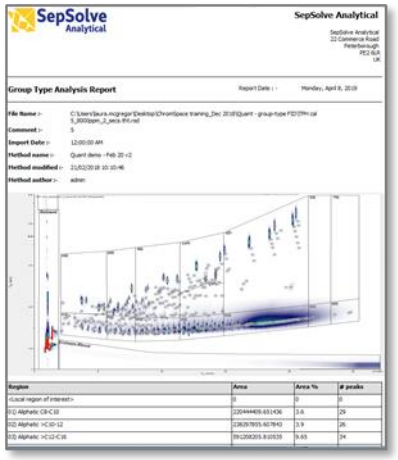
Sample

Traditional solvent extract

Single transfer



Single analysis AND automated real-time data processing



Reporting

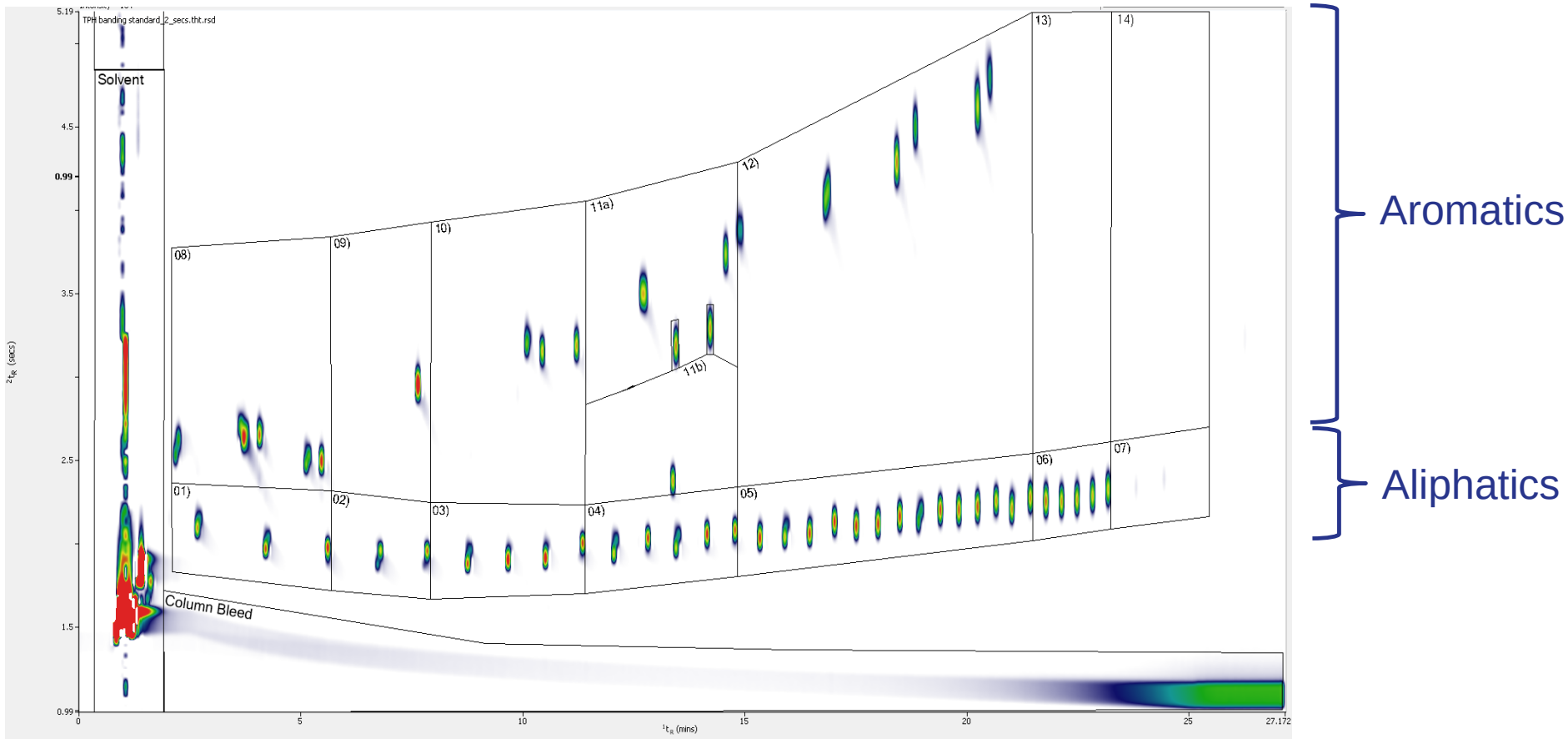
Consumables savings

Fast return on investment

	Small lab	Large lab
Samples per week	100	500
Weekly saving	€280	€1410
Monthly saving	€1120	€5640
Annual saving	€13,440	€67,680

Simple data processing…

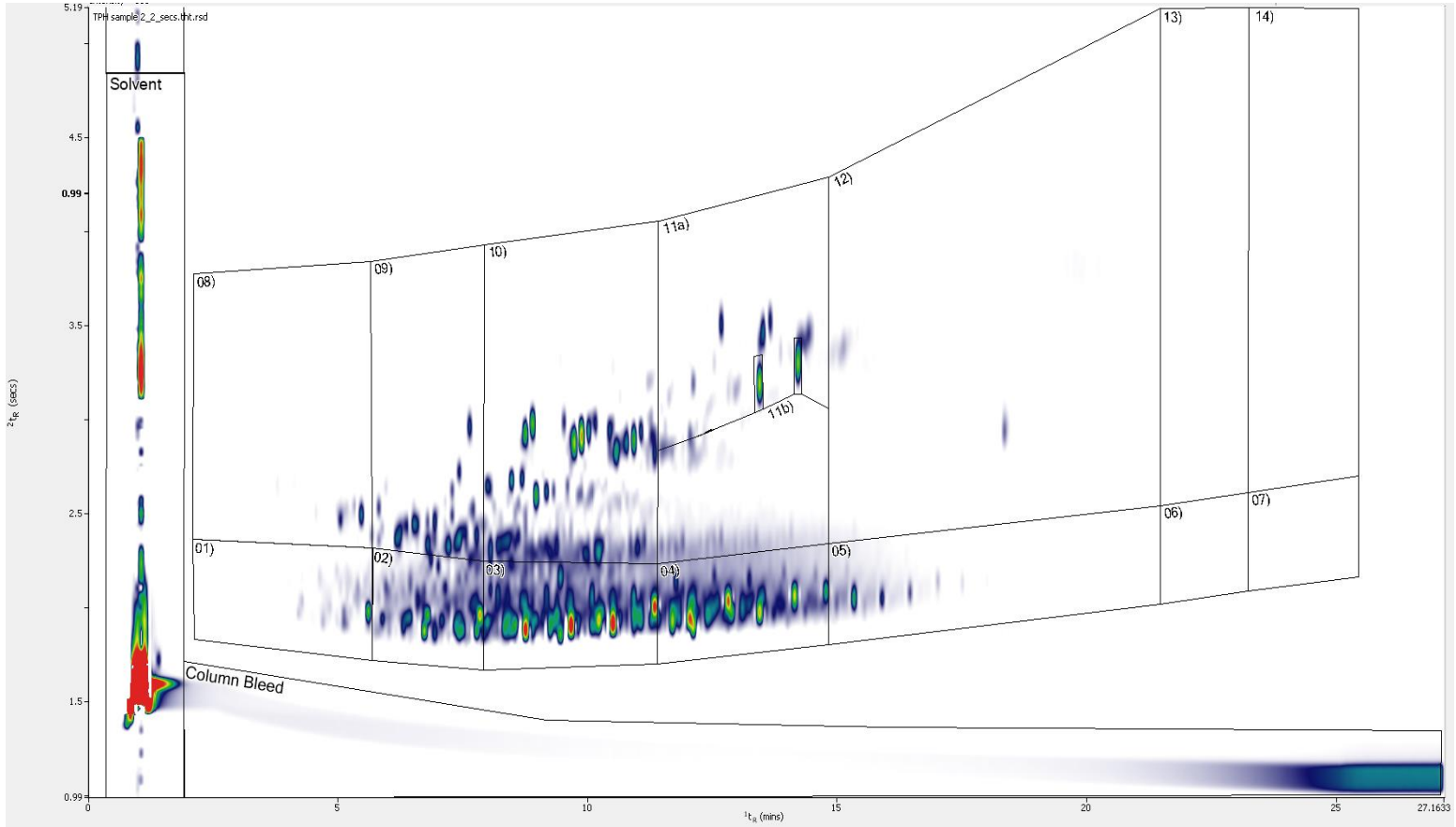
…using stencils



- Regions of interest (Aliphatic >C_{10}-C_{12}….etc) are identified using a banding standard
- Internal standard and surrogate regions can also be added

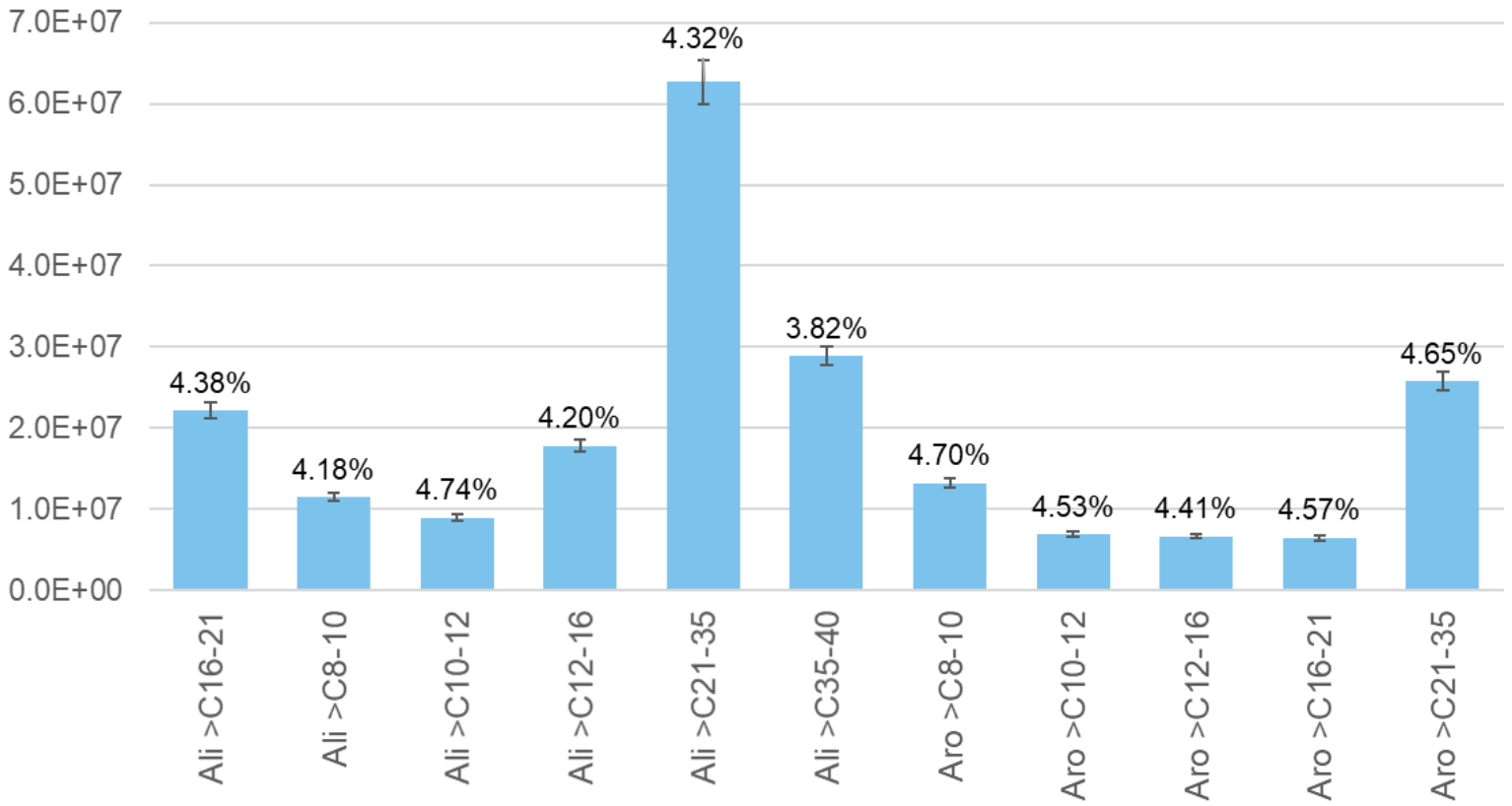
Simple data processing…

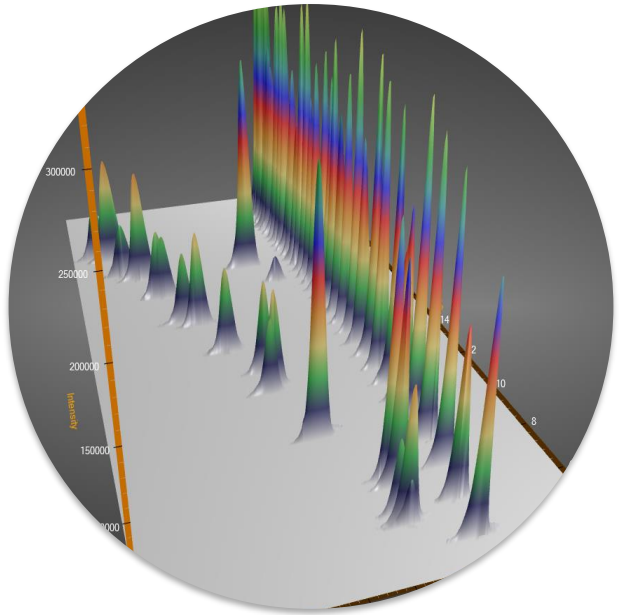
…using stencils



- Stencils are then applied to real samples in **real-time data processing**

Repeatability

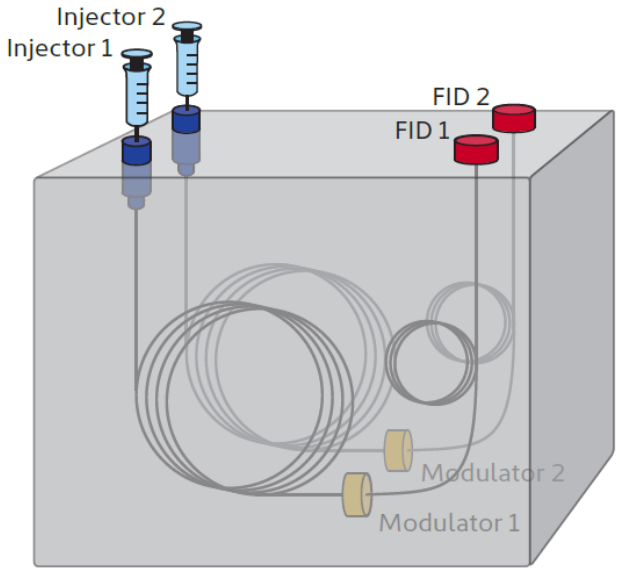


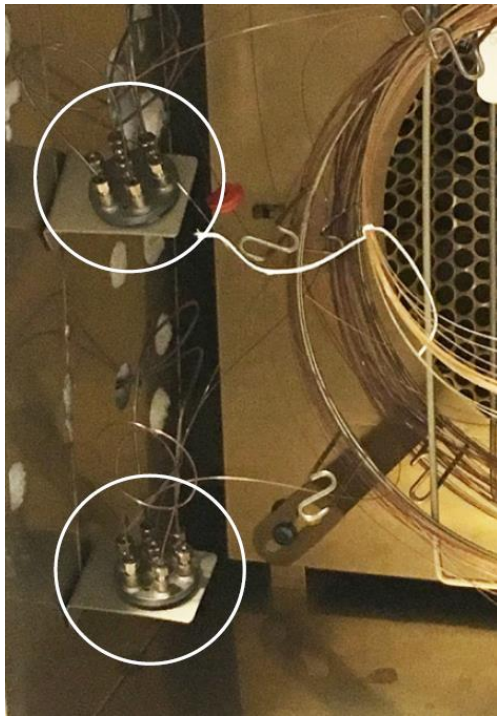


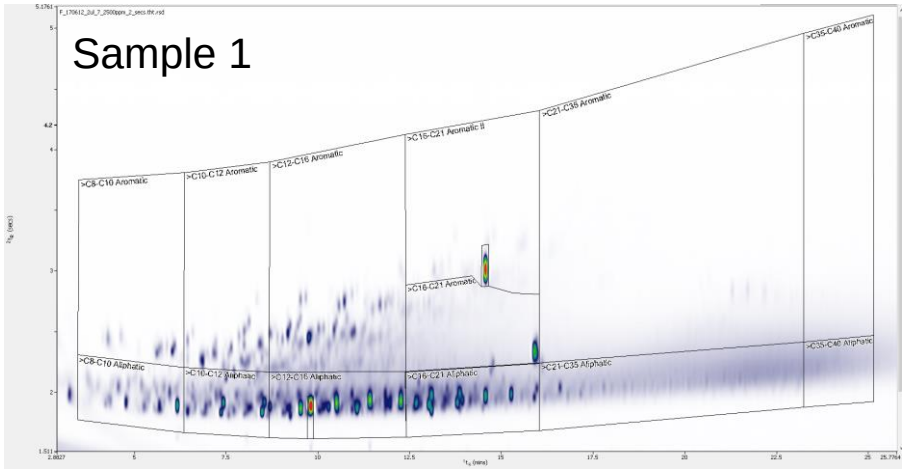
- 15 injections of the TPH marker standard over a **5 day** period
- All RSD <5%

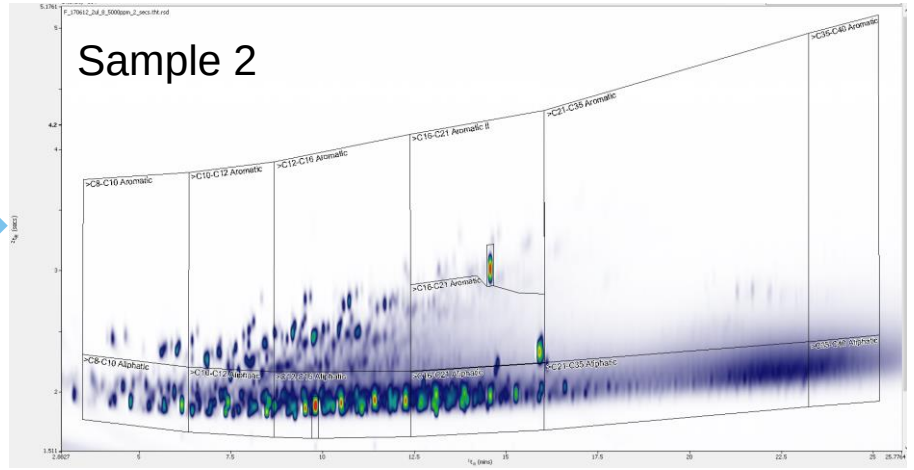
Enhanced productivity…

…through dual-channel GC×GC



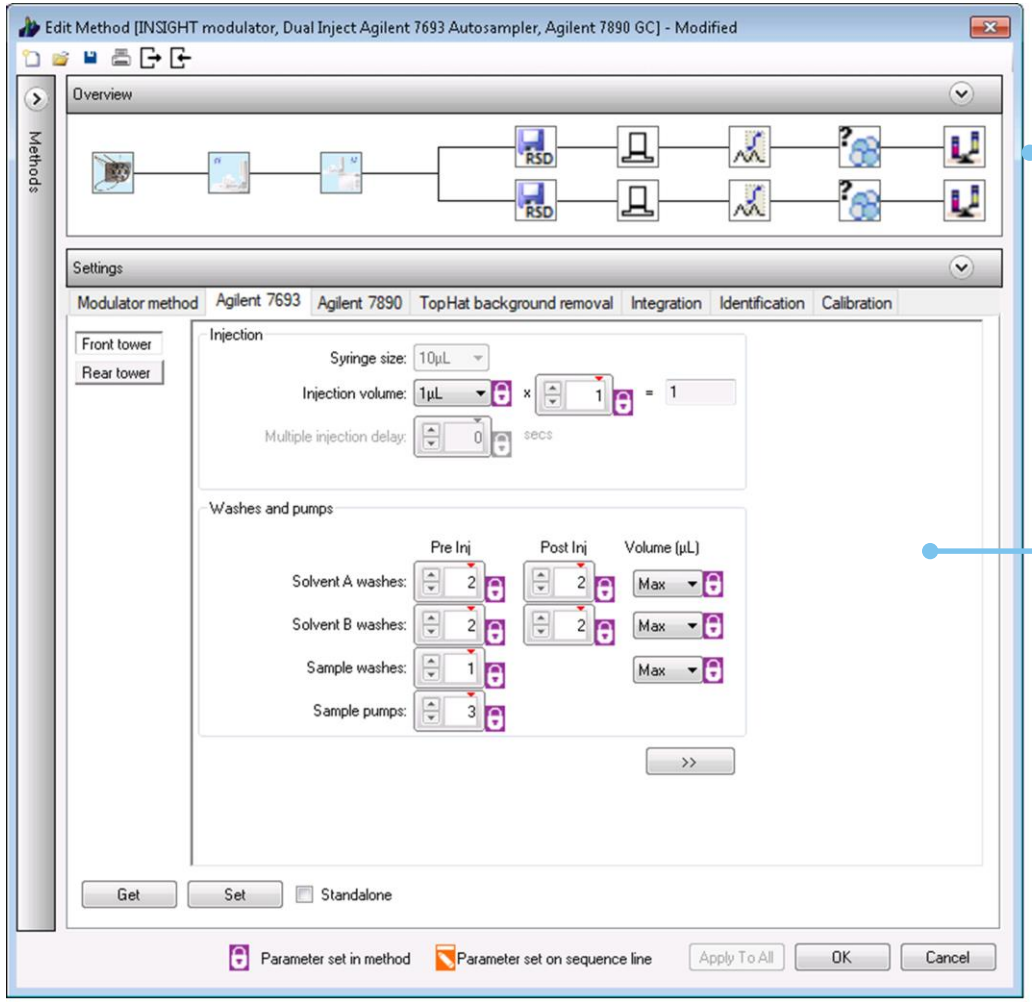






- Run two samples simultaneously, with real-time data processing in ChromSpace
- Compatible with Agilent and Thermo GCs

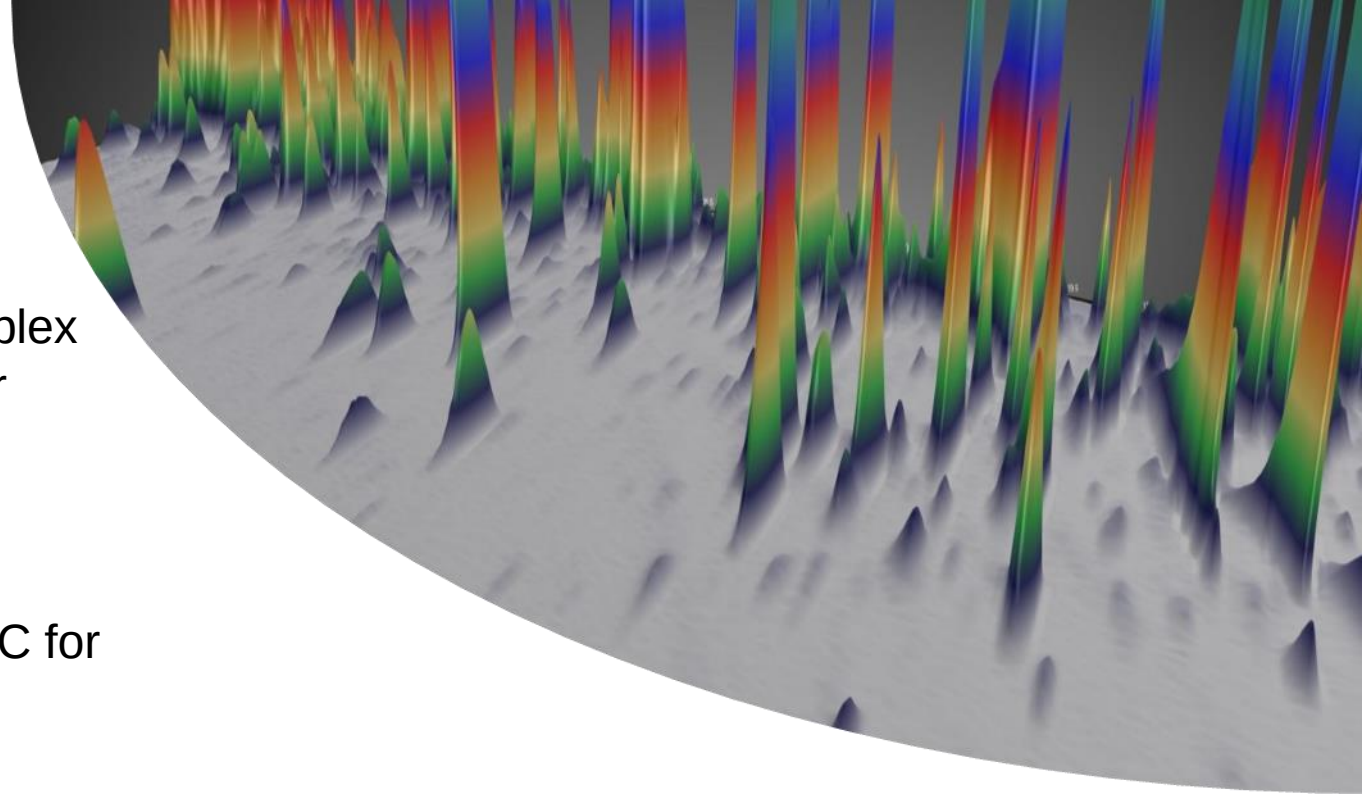
ChromSpace for dual-channel GC×GC



Real-time data processing for both channels

Template methods already configured

Take home messages



- GC×GC provides enhanced separation for complex environmental samples, eliminating the need for sample fractionation
- INSIGHT offers consumable-free, robust GC×GC for the widest range of analytes (VOCs to SVOCs)
- GC×GC is moving from “niche” to “routine” thanks to improvements in hardware and software
- Coupling with TOF MS adds an extra level of information on sample composition

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