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GC Tips and Tricks to Speed Up Your Analysis and Increase Your Throughput

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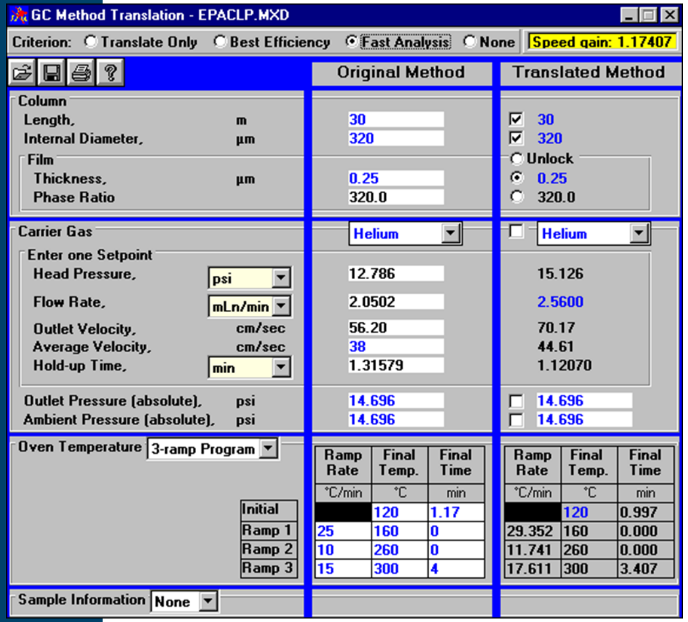
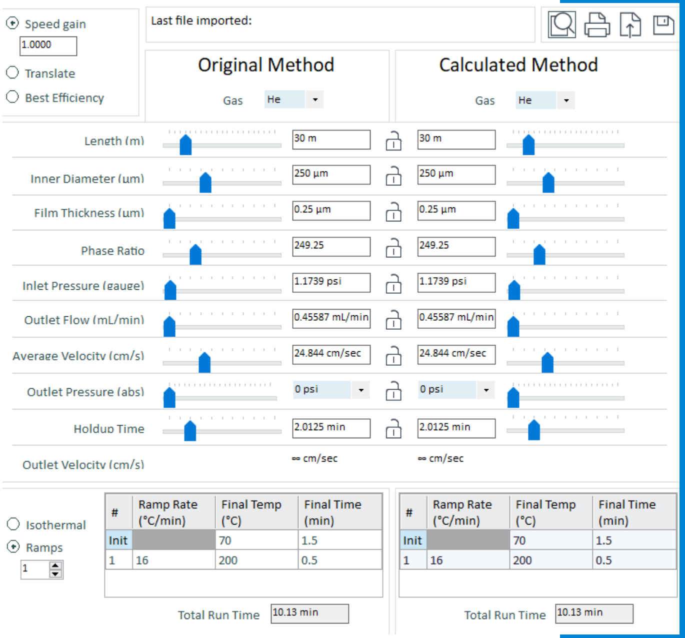
GC Method Translation Software
- decrease analysis time
- optimize GC parameters
- maintain peak patterns and resolution





Method Translation Software (Not New)

Precise Time-Scaling of Gas Chromatographic Methods Using Method Translation and Retention Time Locking

Application

Gas Chromatography

May 1998

Authors

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Abstract

Key Words

Pesticides, GC, GC-AED, retention time locking, RTL, method translation, scalable RT libraries

Introduction

Interest in the analysis of pesticide

the observed element content of the peak. The combination of time and element content narrows rapidly the possible compounds that could have produced the heteroatom response to a few pesticides.

The element-selective detection is done with either gas

Acknowledgements & References

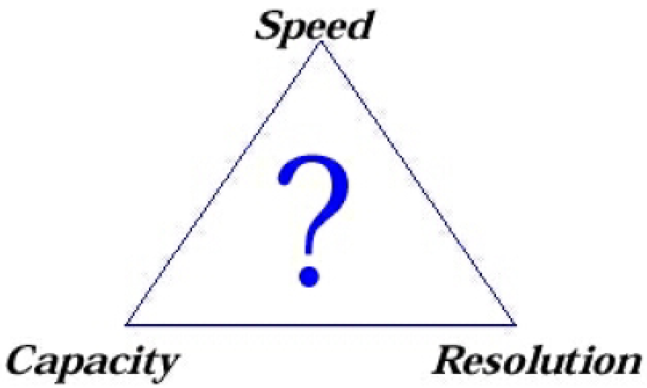
1. M. Klee and V. Giarrocco, "*Predictable Translation of Capillary GC Methods for Fast GC,*" Publication 5965-7673E, March **1997**.
2. V. Giarrocco, B. D. Quimby, and M. S. Klee*, "Retention Time Locking: Concepts and Applications,"* Publication 5966-2469E, December **1997**.
3. P. L. Wylie and B. D. Quimby, *"A Method Used to Screen for 567 Pesticides and Suspected Endocrine Disrupters,"* Publication 5967-5860E, April **1998**.
4. L. Wool and D. Decker, *Practical Fast Gas Chromatography for Contract Laboratory Program Pesticides, J. Chromat. Sci. Vol. 40, September* **2002**.
5. F. Bothe, K. Dettmer, and W. Enewald, "*Determination of Perfume Oil in Household Products by Headspace SPME and Fast Capillary Gas Chromatography", 57,* **2003**.
6. M. Sinnott, S. Jones, "*Rapid Analysis of Food and fragrances Using High Efficiency Capillary GC Columns*", Publication 5989-7509EN, November **2007.**
7. "*Method Translation of HJ679-2013 for Intuvo*", August **2017**.

Can I speed up my runtime?

- What data do I really need for my analysis?
- Are there large amounts of baseline between peaks?
- Do I need to resolve all the peaks in my chromatogram?
- What carrier gas should I use?
- How much sample do I need to place on the column to detect my components of interest?

Variables for Shortening Runtime

- Stationary Phase
- Temperature Programming
- Carrier Gas: type and linear velocity
- Shorten Column Length
- Decrease Film Thickness
- Decrease Internal Diameter



Resolution

$$R_s = \frac{\sqrt{N}}{4}\left(\frac{k}{k+1}\right)\left(\frac{\alpha-1}{\alpha}\right)$$

Efficiency	$N = f$ (gas, L, r_c)	L = Length
Retention	$k = f$ (T, d_f, r_c)	r_c = column radius
Selectivity	$\alpha = f$ (T, phase)	d_f = film thickness
		T = temperature

Selectivity

Represents the separation power of a particular adsorbent to separate a mixture of components.

Stationary Phase - Common Types

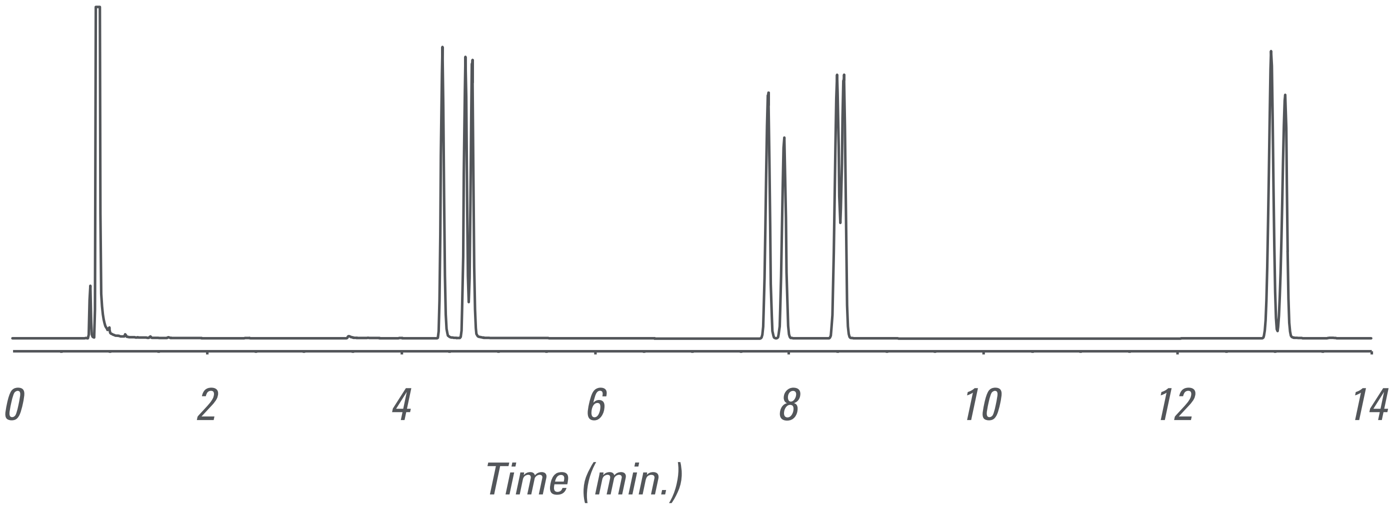
Siloxane polymers

Poly(ethylene) glycols

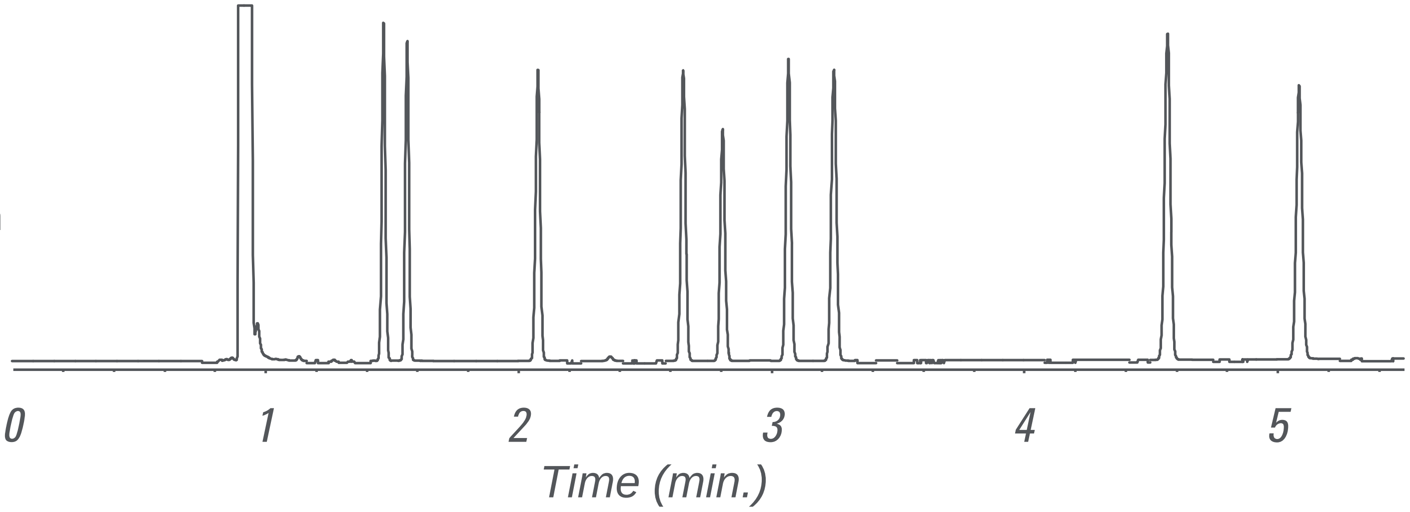
Porous polymers

Start with the Right Phase

DB-1
15m x 0.32mm, 0.25µm
Oven:
40°C for 2 min
40-120°C at 5°C/min



DB-Wax
15m, 0.32mm, 0.25µm
Oven:
80-190°C at 20°C/min



Stationary Phase Selection

Utilize existing Information, Determine critical separations

Selectivity/Polarity, Temperature Limits, SP-Application specific

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Software-Supported Method Development - the Scanview Program

Version 13

Created by don_gage on Apr 12, 2018 10:41 AM. Last modified by noreuter on Mar 20, 2019 4:13 AM.

The New Scanview Application Database, Version 2019

Norbert Reuter*, Ingrid van der Meer, *Agilent Technologies, Middelburg, The Netherlands*

Introduction

The new Scanview version 2019 is an application database that contains full method descriptions and literature references for gas and liquid chromatographical analyses, sample preparation, optical spectroscopy applications (UV, IR, CE, ICP-MS/ICP-OES and AA) and chemical cleanup and reaction monitoring. More than 5900 application notes are contained in the Scanview database. The user-friendly front-end allows you to search/filter on a variety of categories – the top five categories are the most used.

https://community.agilent.com/docs/DOC-2118-software-supported-method-development-the-scanview-program

Choose the column phase that gives the best separation but not at the cost of robustness or ruggedness.

Helium Carrier Gas Alternatives

Important theoretical considerations relating to peak efficiency

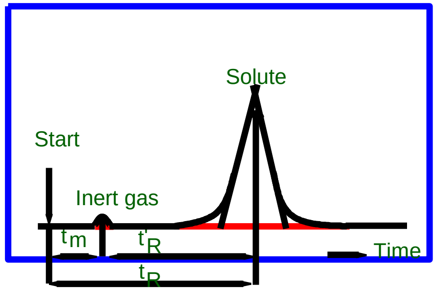
Sharp, narrow peaks in a chromatogram is an indication of **a high efficiency GC column.**

- Remember that **efficiency** is represented mathematically by the symbol "*N*" called *Theoretical Plates*, and that the larger *N* is, the better the resolving power of the column (i.e., higher resolution).
- **Resolution** is described mathematically by the symbol R_s and its numeric value tells how well two adjacent peaks are separated from each other.

$$R_s = \frac{\sqrt{N}}{4}\left(\frac{k}{k+1}\right)\left(\frac{\alpha-1}{\alpha}\right)$$

A resolution value of 1.5 tells us that two peaks are baseline separated. The greater (higher) the R_s value, the more separation that has been achieved.

Calculating efficiency



We would like to know the actual time the component spends in the stationary phase.

$t'_R = t_R - t_m$

$n = 5.545 \left(\frac{t'_R}{W_h}\right)^2$

t'_R = corrected retention time.

n = effective theoretical plates.

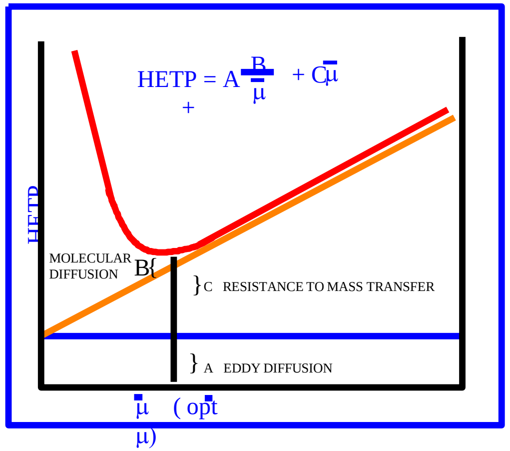
Let's relate "n" to the length of the column.

Plates per meter (N) = $\frac{n}{L}$ or

Height equivalent to a theoretical plate (HETP) = $\frac{L}{n}$

Thus, the more efficient the column, the bigger the "N" the smaller the "HETP".

Efficiency and carrier gas linear velocity



Efficiency is a function of the carrier gas linear velocity or flow rate.

The minimum of the curve represents the smallest HETP (or largest plates per meter) and thus the best efficiency. "A" term is not present for capillary columns.

- Plot of HETP versus linear velocity is know as the **Van Deemter** plot.
- The linear velocity value at the minimum of the curve is the optimum value for achieving the best efficiency.

Helium Carrier Gas Alternatives

Let's make this easy

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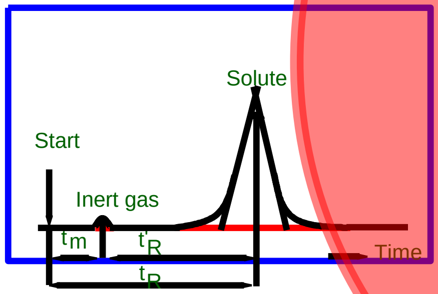
Calculating efficiency



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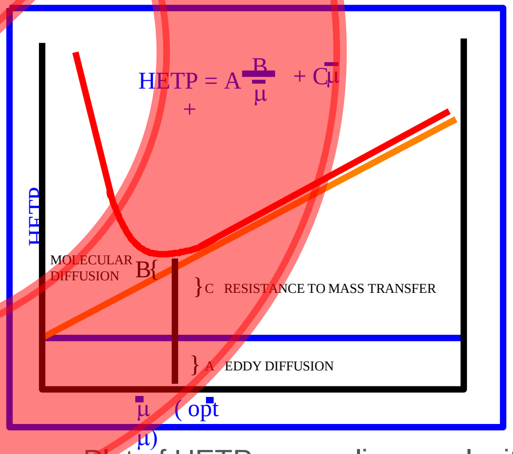
Efficiency and carrier gas linear velocity



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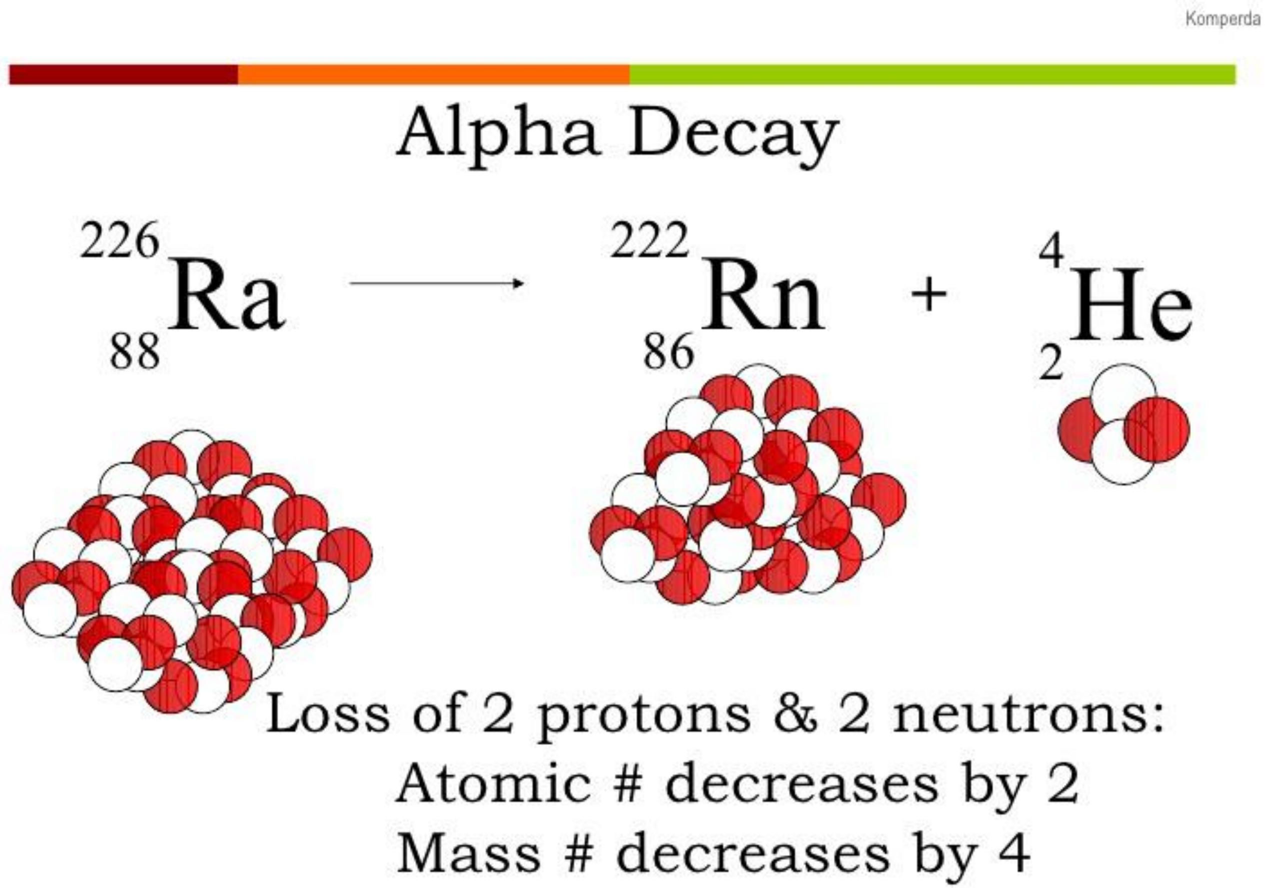
Where does Helium come from?

Where does Helium come from?

The **helium** is **formed** during the natural radioactive decay of elements such as uranium and thorium. These heavy elements were **formed** before the earth but they are not stable and very slowly, they decay. ... This alpha-particle is actually just the heart of a **helium** atom - its nucleus



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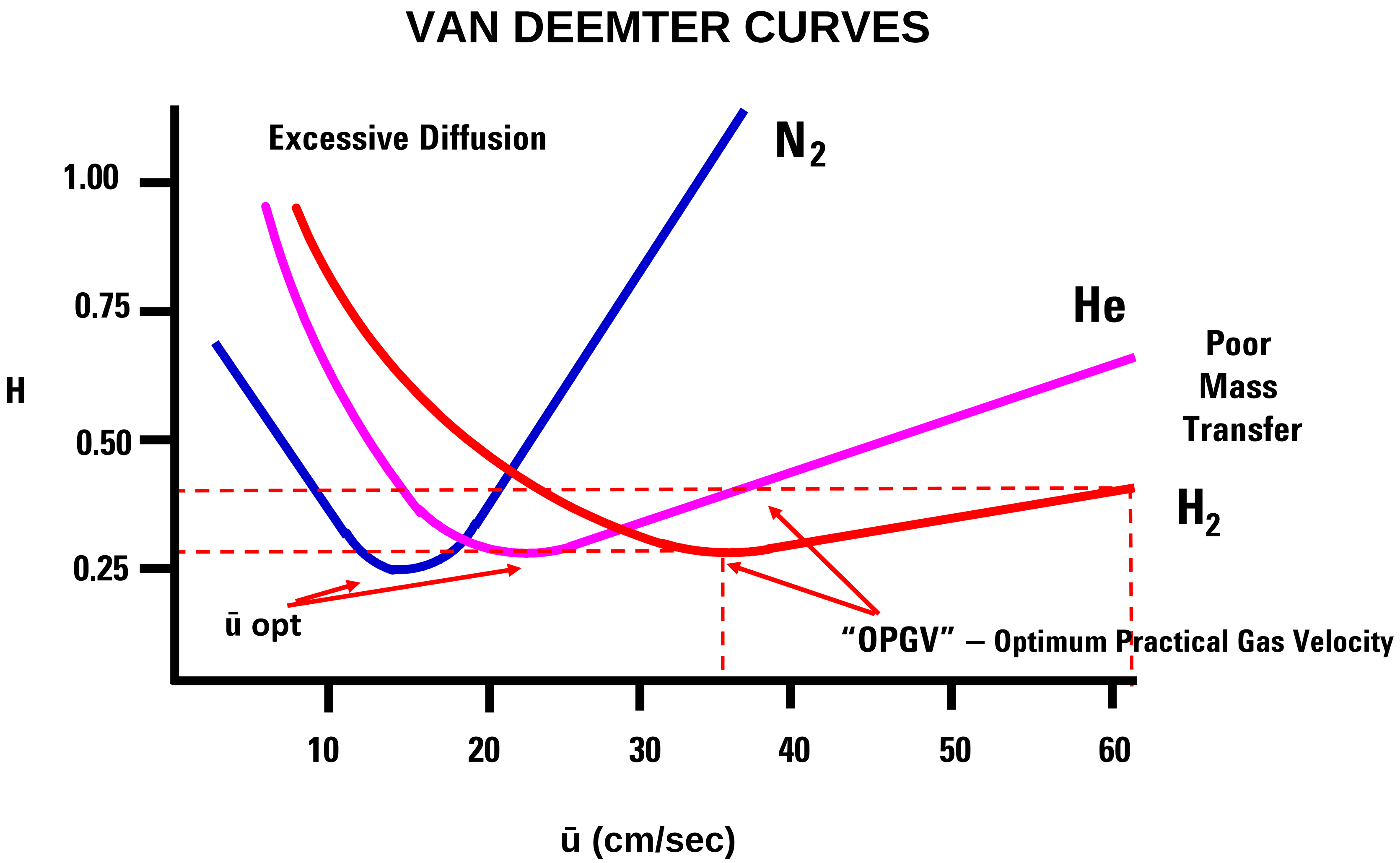
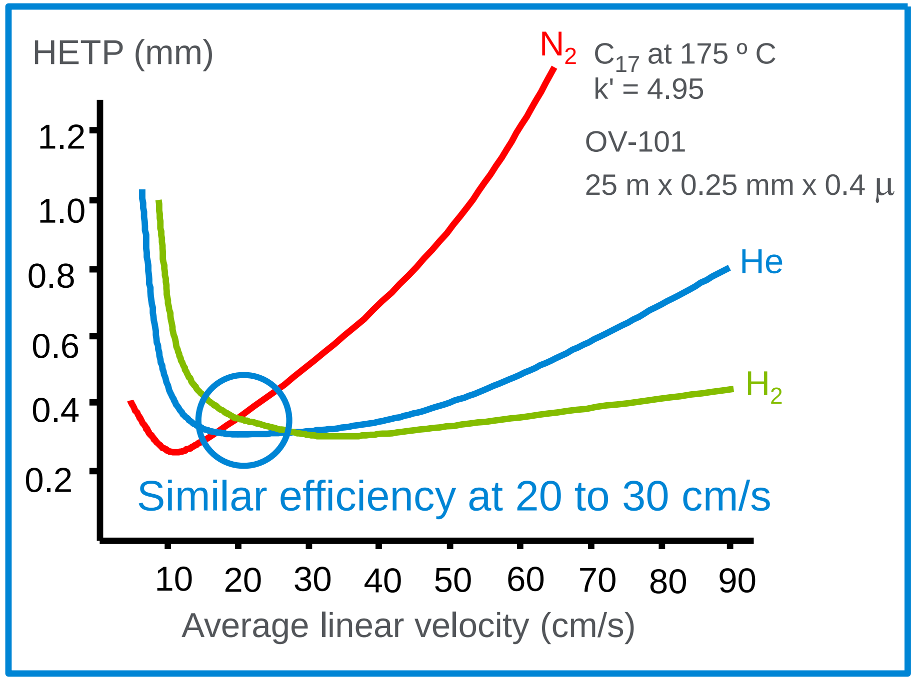
Carrier Gas Selection

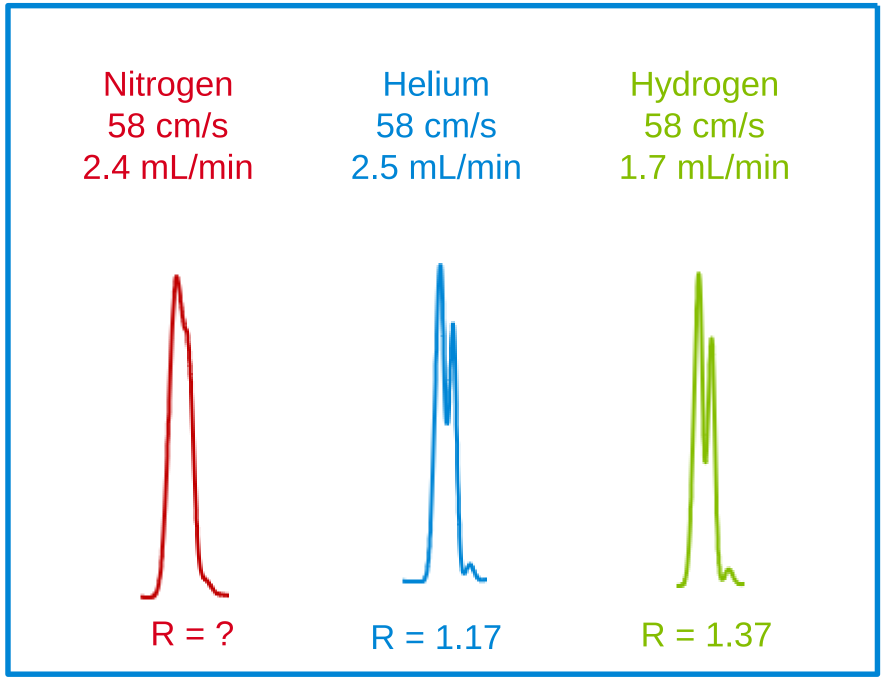


H = height equivalent of theoretical plates (goal is shorter height, to achieve more plates/meter)
ū = velocity

Van Deemter

Why nitrogen gets a bad reputation for capillary GC



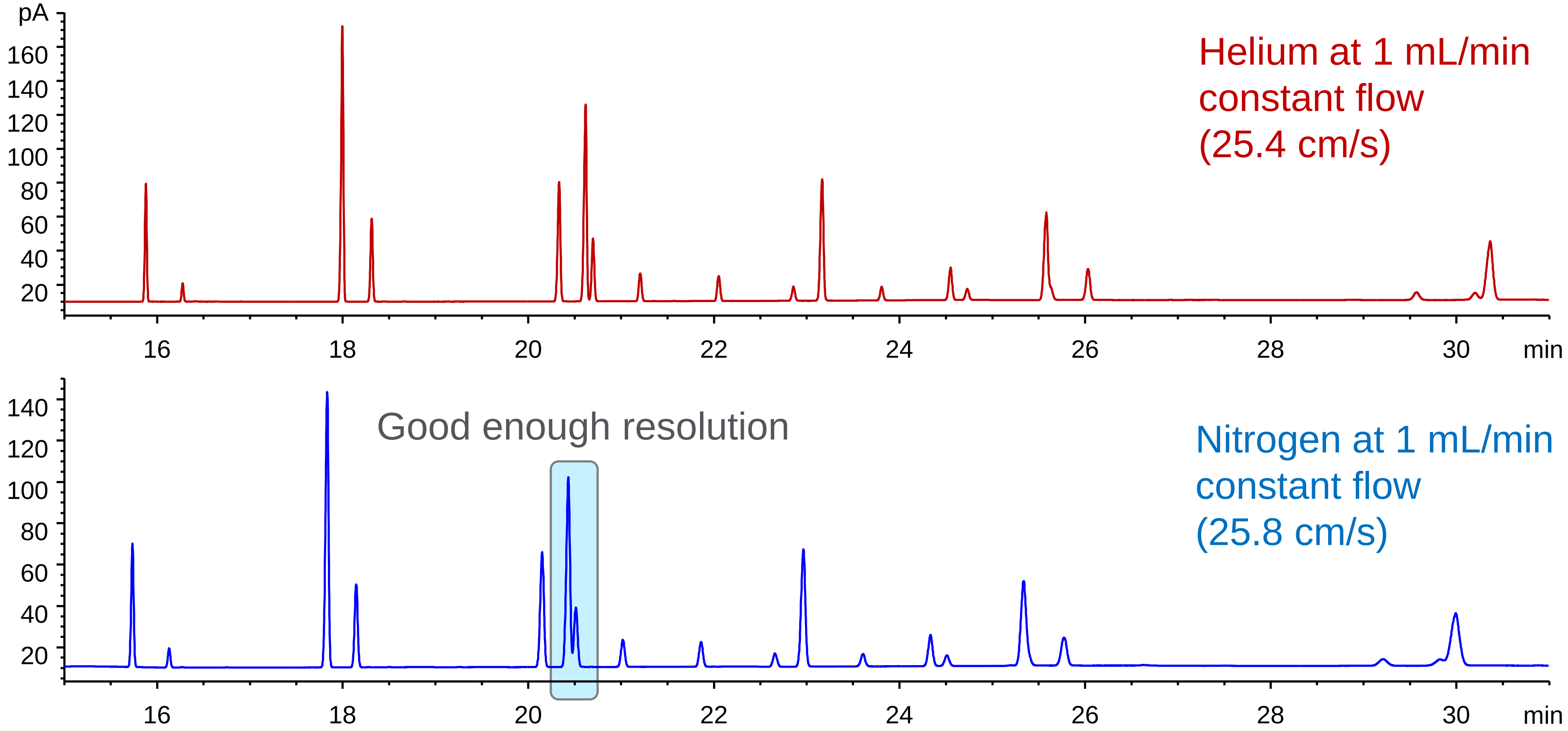


- N_2 actually provides the best efficiency, but at a slower speed
- Most helium methods have too much resolution
  - Lower N_2 efficiency at higher flows can still provide "good enough" resolution
- Most GC methods now use constant flow
  - N_2 efficiency losses with temperature programming are not as severe

Many Helium GC Have Excess Resolution

EN14103 – GC analysis of FAME content in biodiesel

HP-INNOWax, 30 m x 0.25 mm id x 0.25 µm



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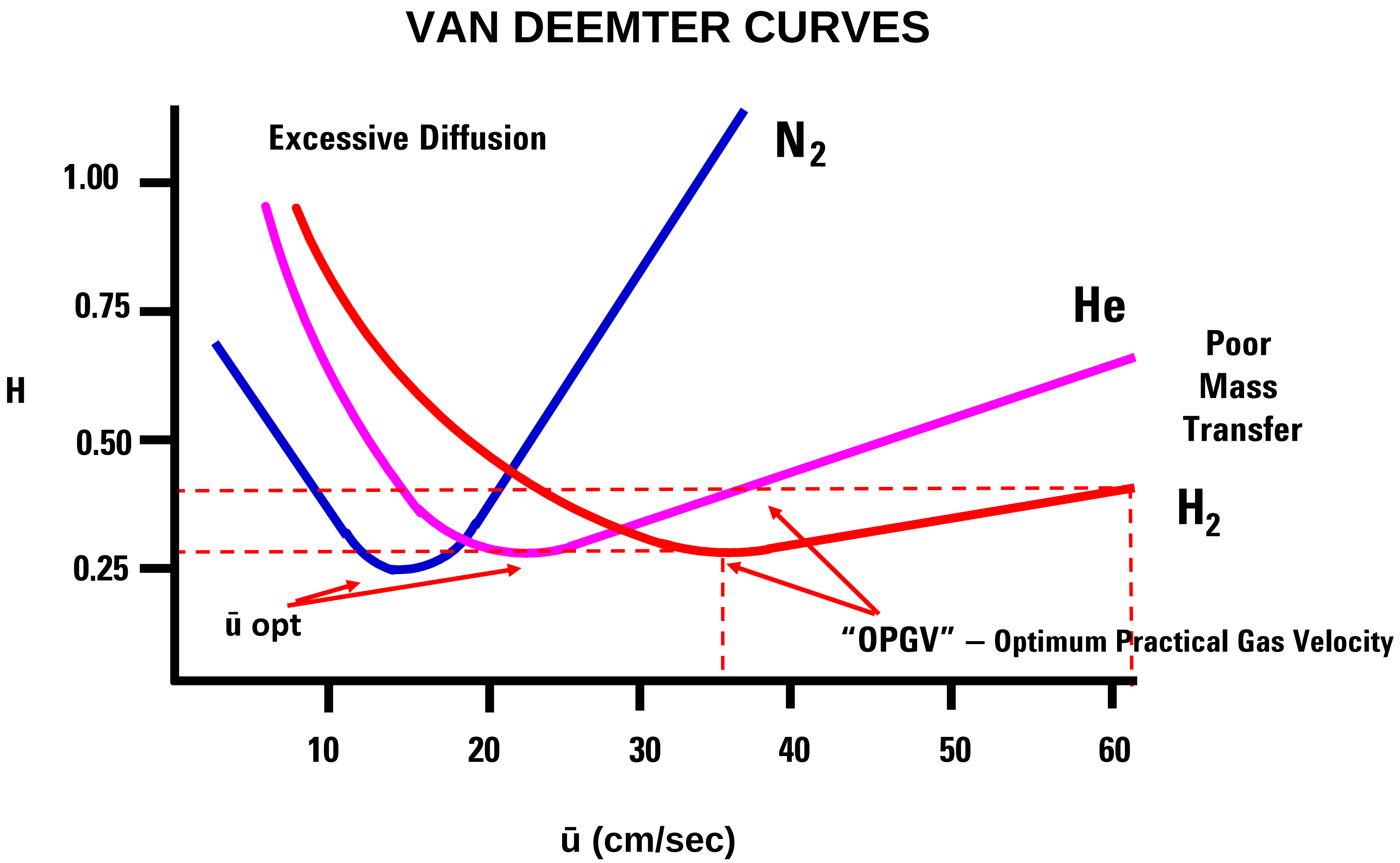
Carrier Gas Selection



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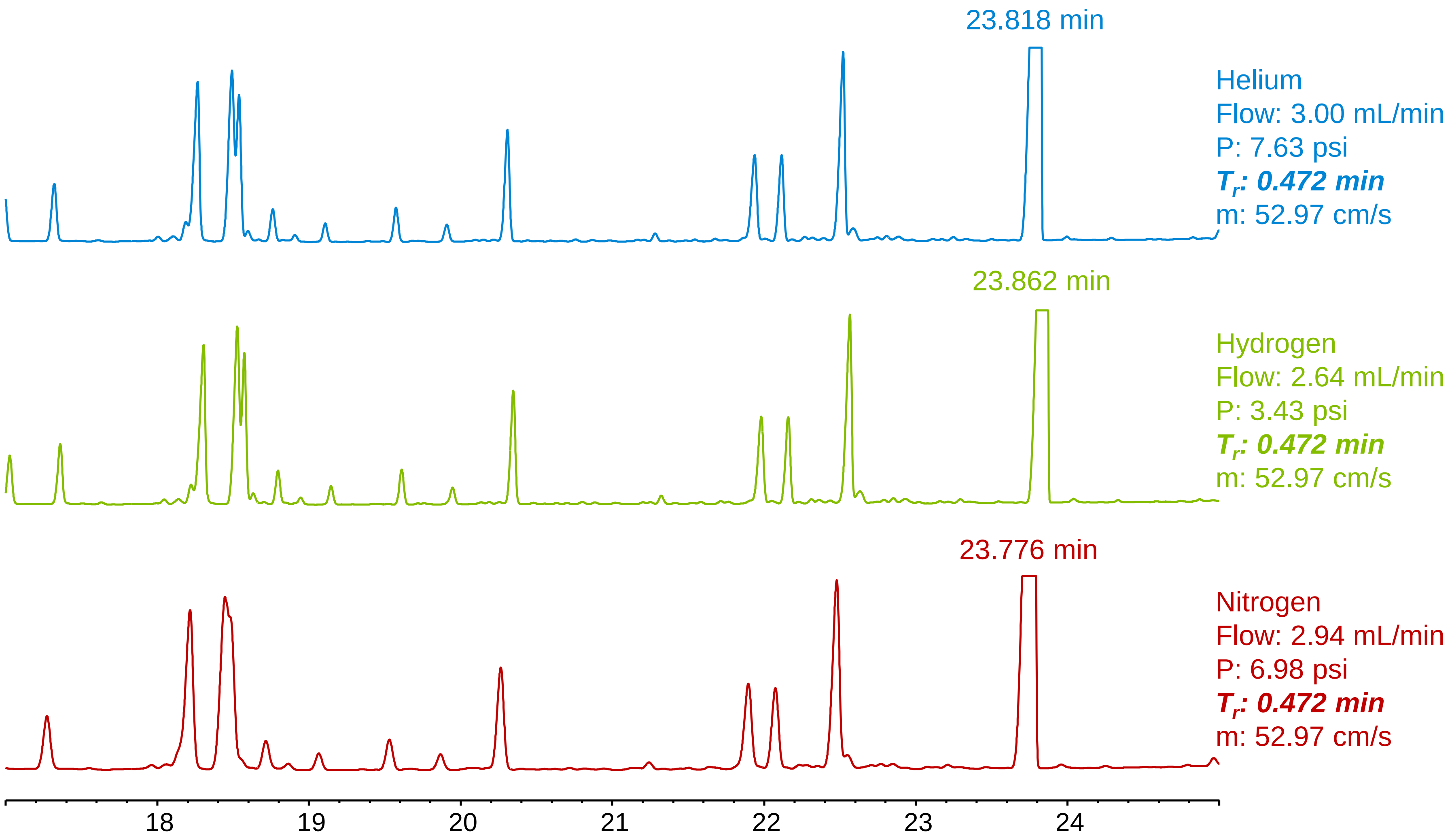
Same Holdup Time (T_r) Gives Consistent Retention Times Compared to Original Helium Method



Column Diameter - Theoretical Efficiency

I.D. (mm)	n/m
0.05	23,160
0.10	11,980
0.18	6,660
0.20	5830
0.25	4630
0.32	3760
0.45	2840
0.53	2060

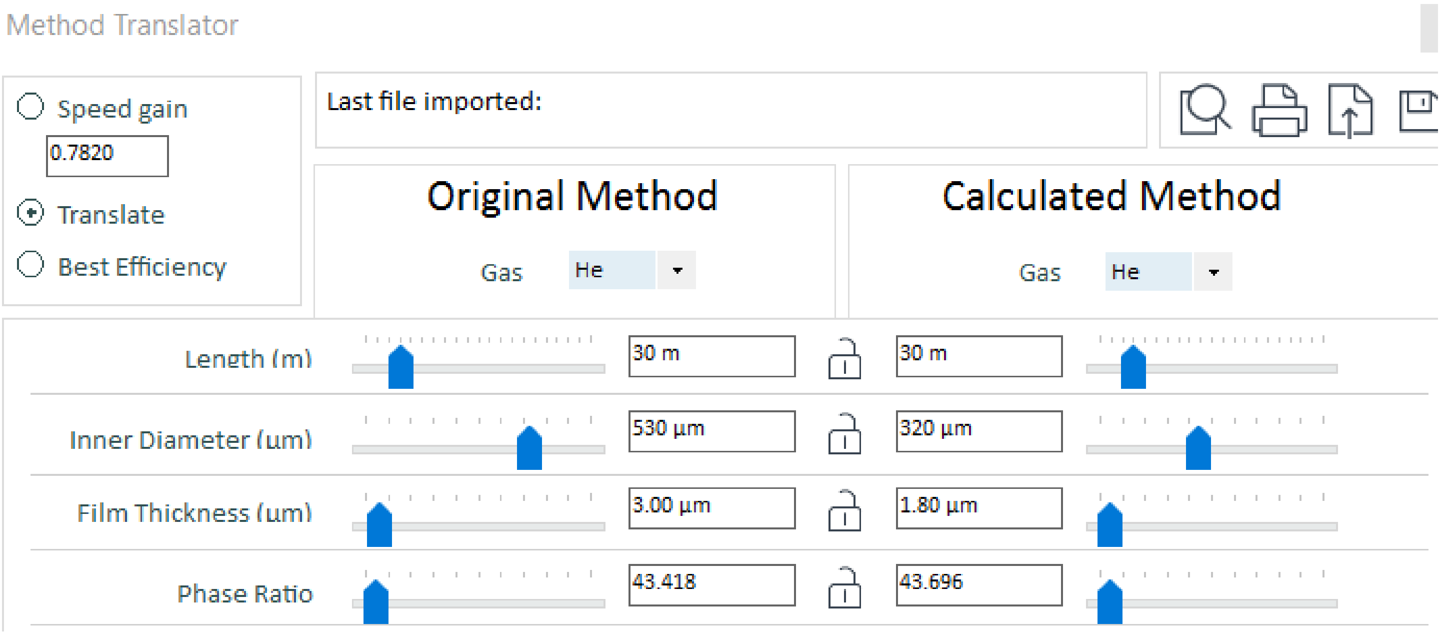
Retention Factor k = 5

PHASE RATIO (β)

Column Dimensions	Phase Ratio β
30 m x .53 mm x 3.0 μm	44
30 m x .32 mm x 1.8 μm	44

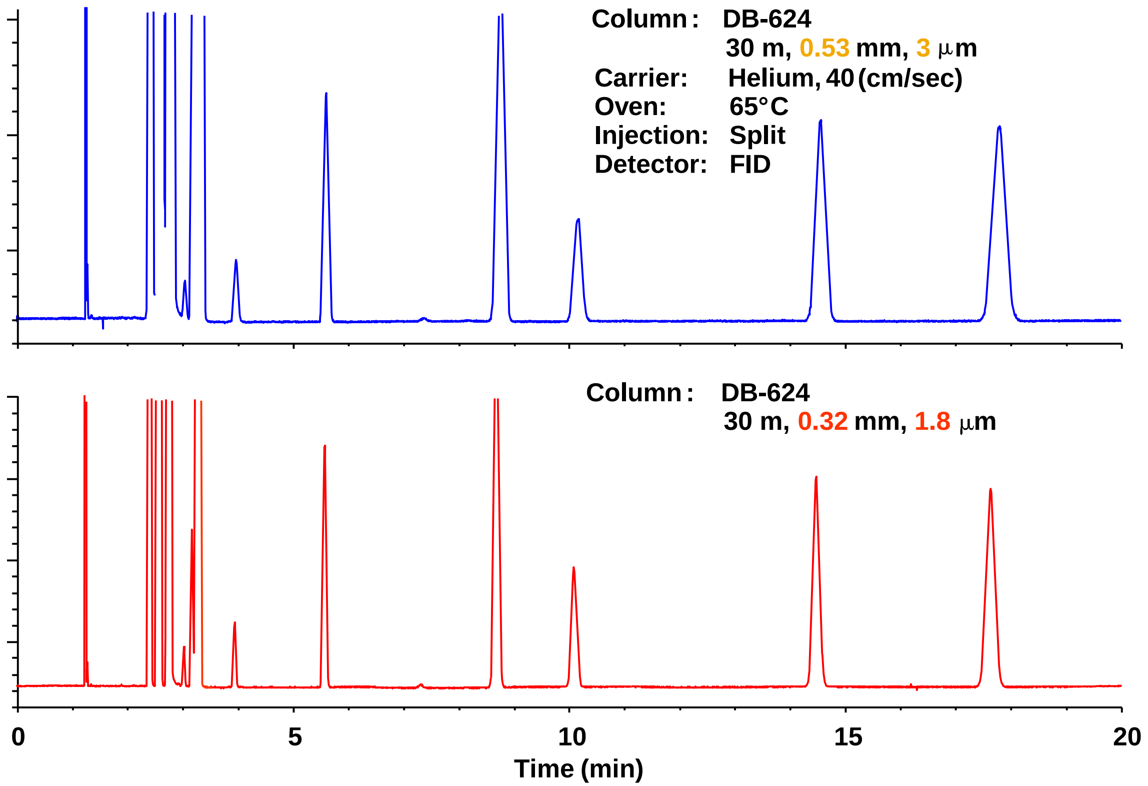
$$\beta = \frac{r}{2d_f}$$

$K_c = k\beta$ *(same phase ratio gives same retention)*

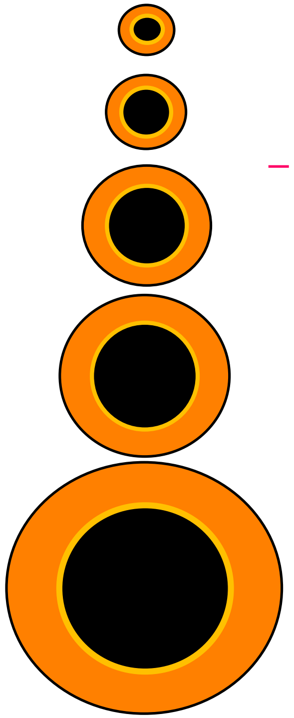


Different Column I. D.

Equal Phase Ratios



Column Diameter and Capacity



I.D. (mm)	Capacity (ng)
0.05	1-2
0.10	6-13
0.18	25-55
0.20	35-70
0.25	80-160
0.32	110-220
0.45	600-800
0.53	1000-2000

Like Polarity
Phase/Solute
0.25 µm film thickness

Column Diameter and Carrier Gas Flow

Lower flow rates: Smaller diameter columns

Low flow rates : GC/MS
High flow rates: Headspace, purge & trap

Higher flow rates: Larger diameter columns

Diameter Summary

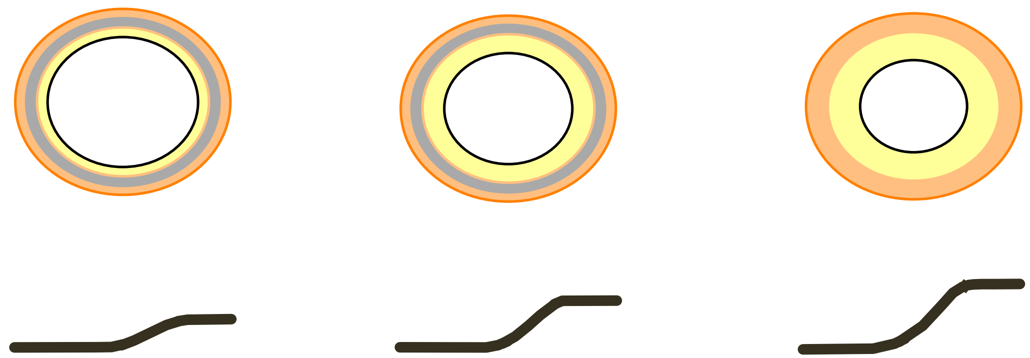
To increase	Diameter
Efficiency	Smaller
Resolution	Smaller
Pressure	Smaller
Capacity	Larger
Flow rate	Larger

Film Thickness and Capacity

Thickness (µm)	Capacity (ng)
0.10	50-100
0.25	125-250
0.50	250-300
1	500-1000
3	1500-3000
5	2500-5000

0.32 mm I.D.
Like Polarity Phase/Solute

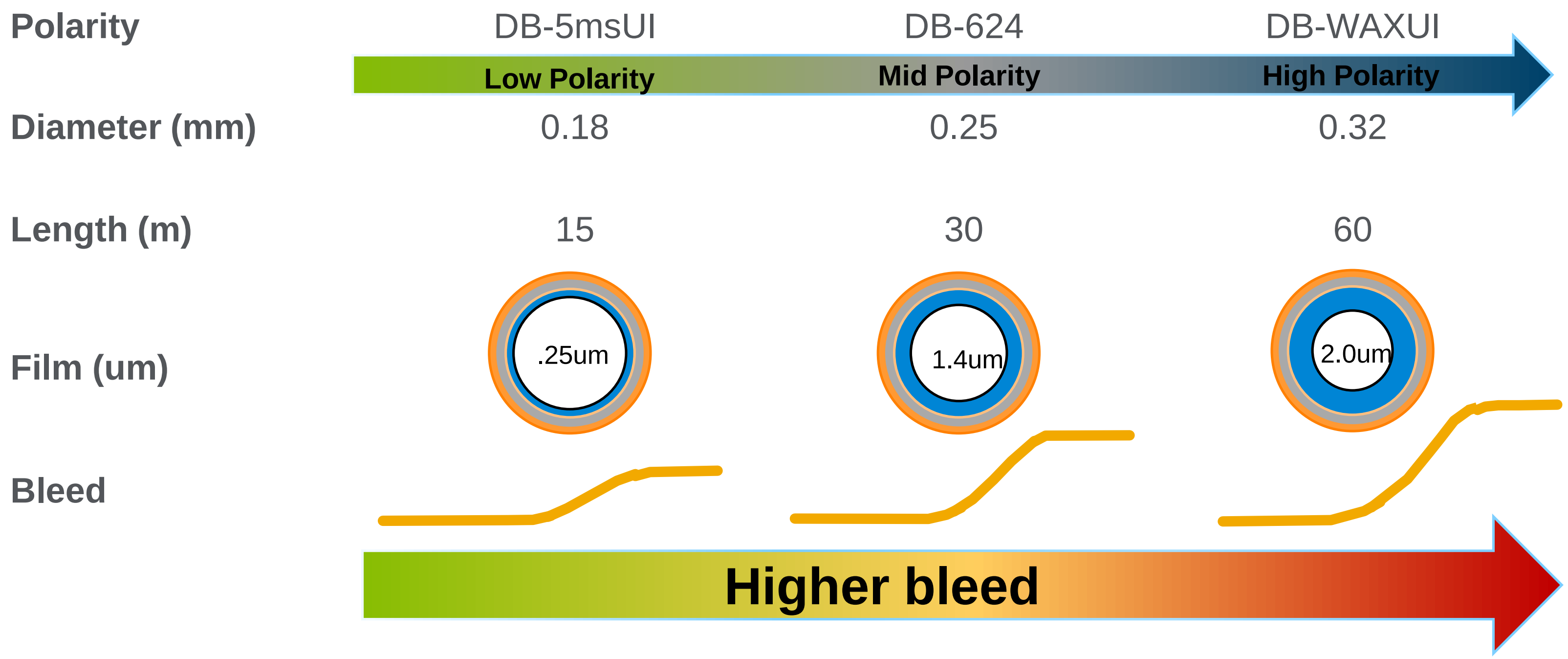
More stationary phase = More degradation products



What column types/dimensions produce higher bleed?

- Polarity: More polar = higher bleed
- Low polarity = More thermally stable
  - Look at temperature limits as a general indicator of thermal stability

- The more total mass of polymer in the column the higher the bleed (within a given phase)
  - Larger diameters
  - Longer columns
  - Thicker films



Column Length and Efficiency (Theoretical Plates)

Length (m)	N
15	69,450
30	138,900
60	277,800

0.25 mm ID
n/m = 4630 (for k = 5)

More Meters = More Plates = More Resolution

Column Length and Resolution

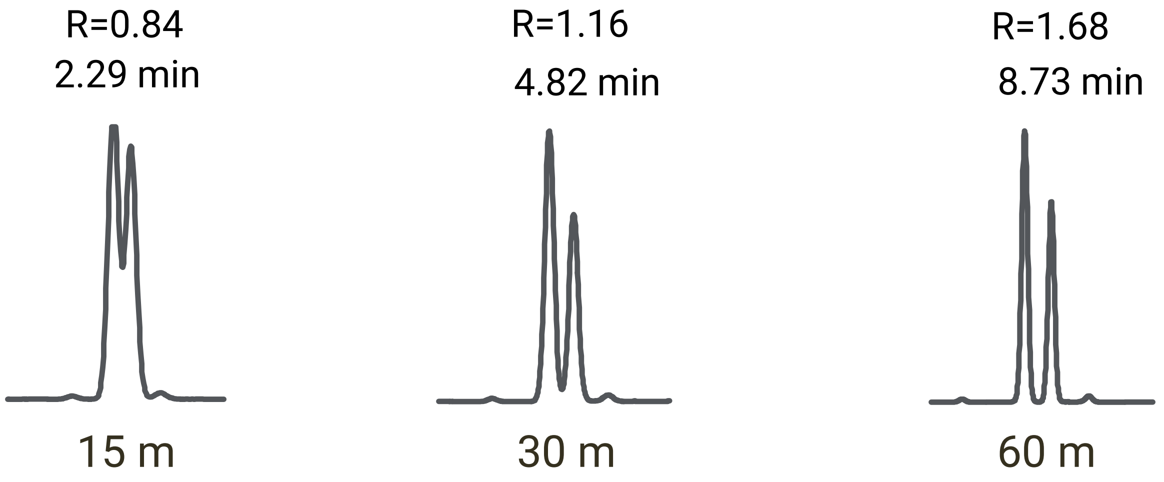
$$R \propto \sqrt{N} \propto \sqrt{L}$$

Length X 4 = Resolution X 2

$$t \propto L$$

Upside = Cut a bunch off during routine inlet maintenance and not lose a lot of Resolution

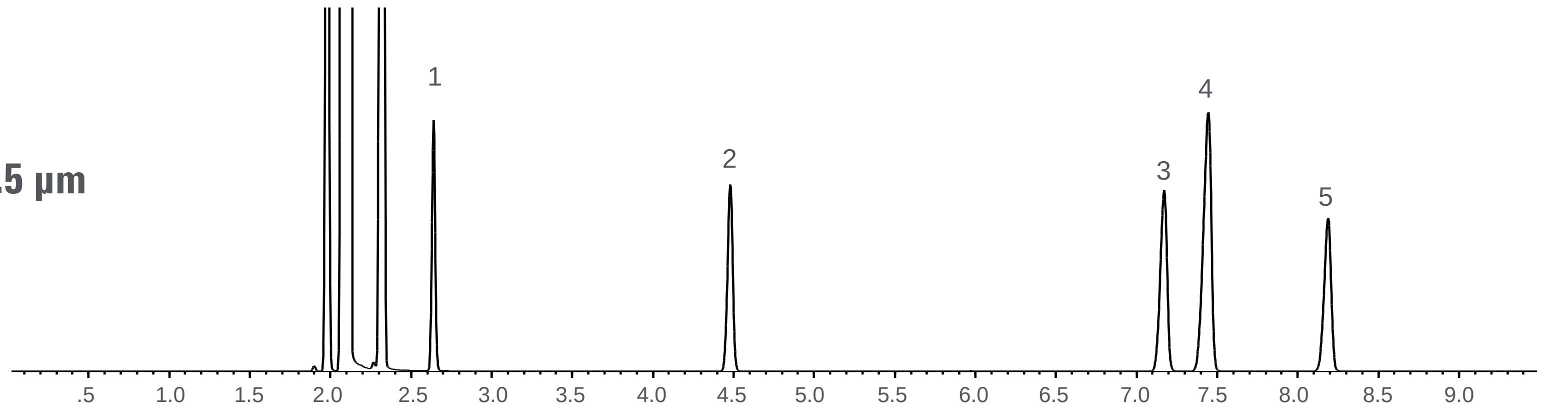
Column Length VS Resolution and Retention: Isothermal



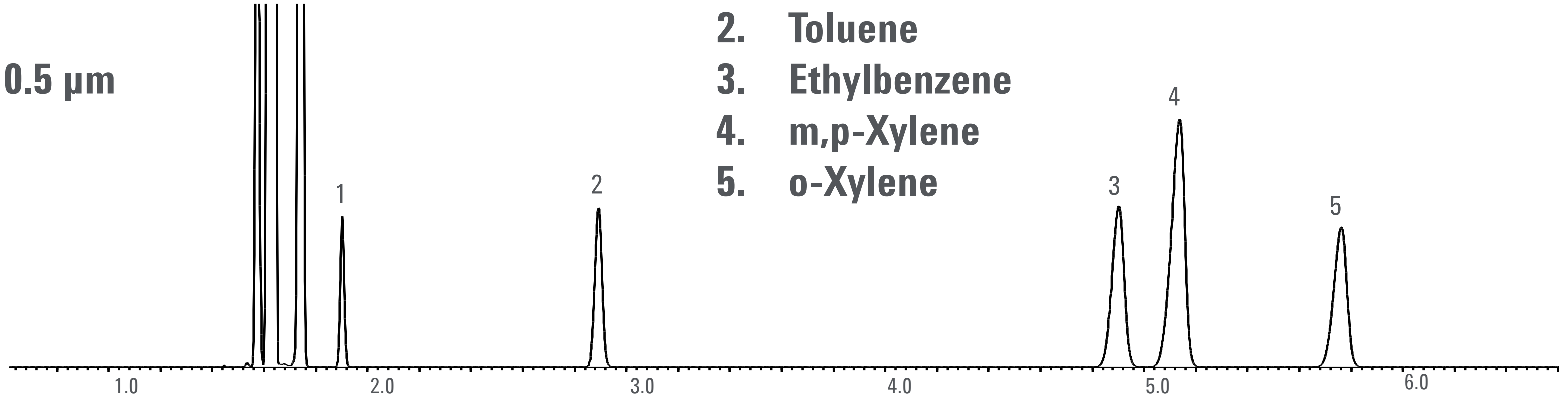
Double the plates, double the time but not double the resolution

DECREASE THE LENGTH

DB-5
30 m
0.53 mm I.D., 0.5 µm



DB-5
15 m
0.53 mm I.D., 0.5 µm



1. Benzene
2. Toluene
3. Ethylbenzene
4. m,p-Xylene
5. o-Xylene

BTEX
Carrier: Helium, 36 cm/sec at 40°c
Oven : 40°C for 3 min, 5°/min to 100°C

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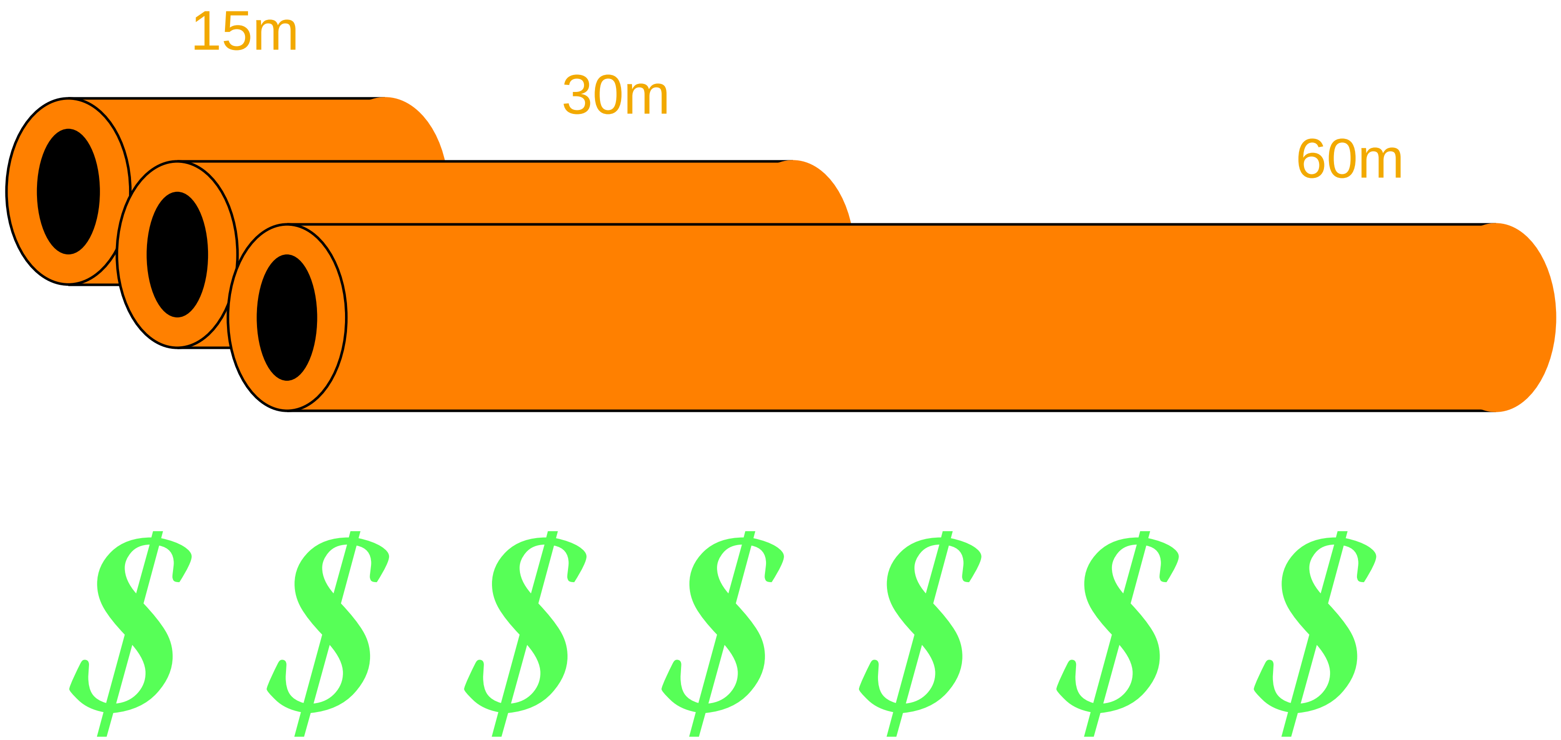
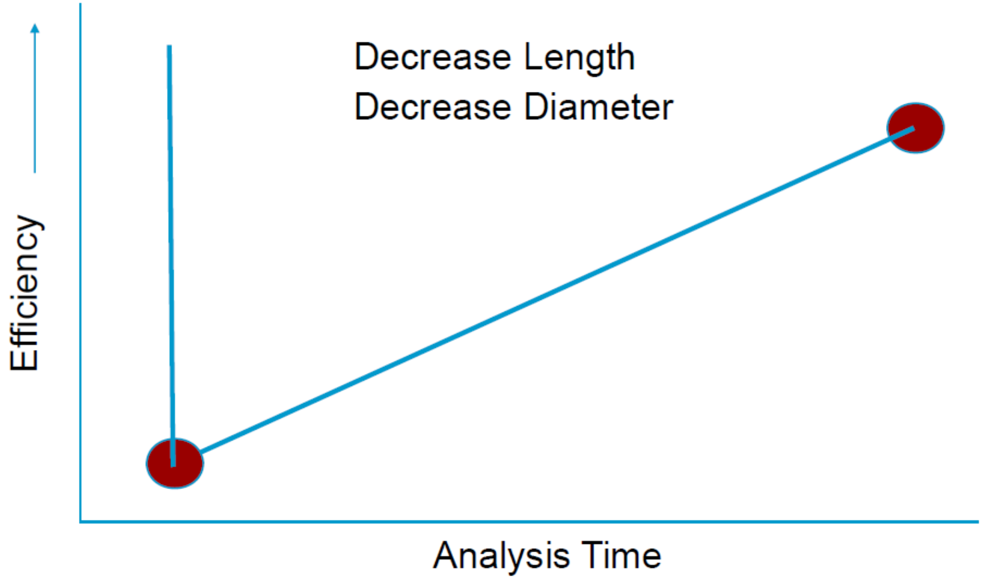
Column Length and Cost



Length Summary

To Increase	Length
Efficiency	Longer
Resolution	Longer
Analysis Time	Longer
Pressure	Longer
Cost	Longer



Column Temperature

Developing Temperature Programs

Most powerful variable

Changes Selectivity and Retention

Natural log (ln) relationship between retention and temperature

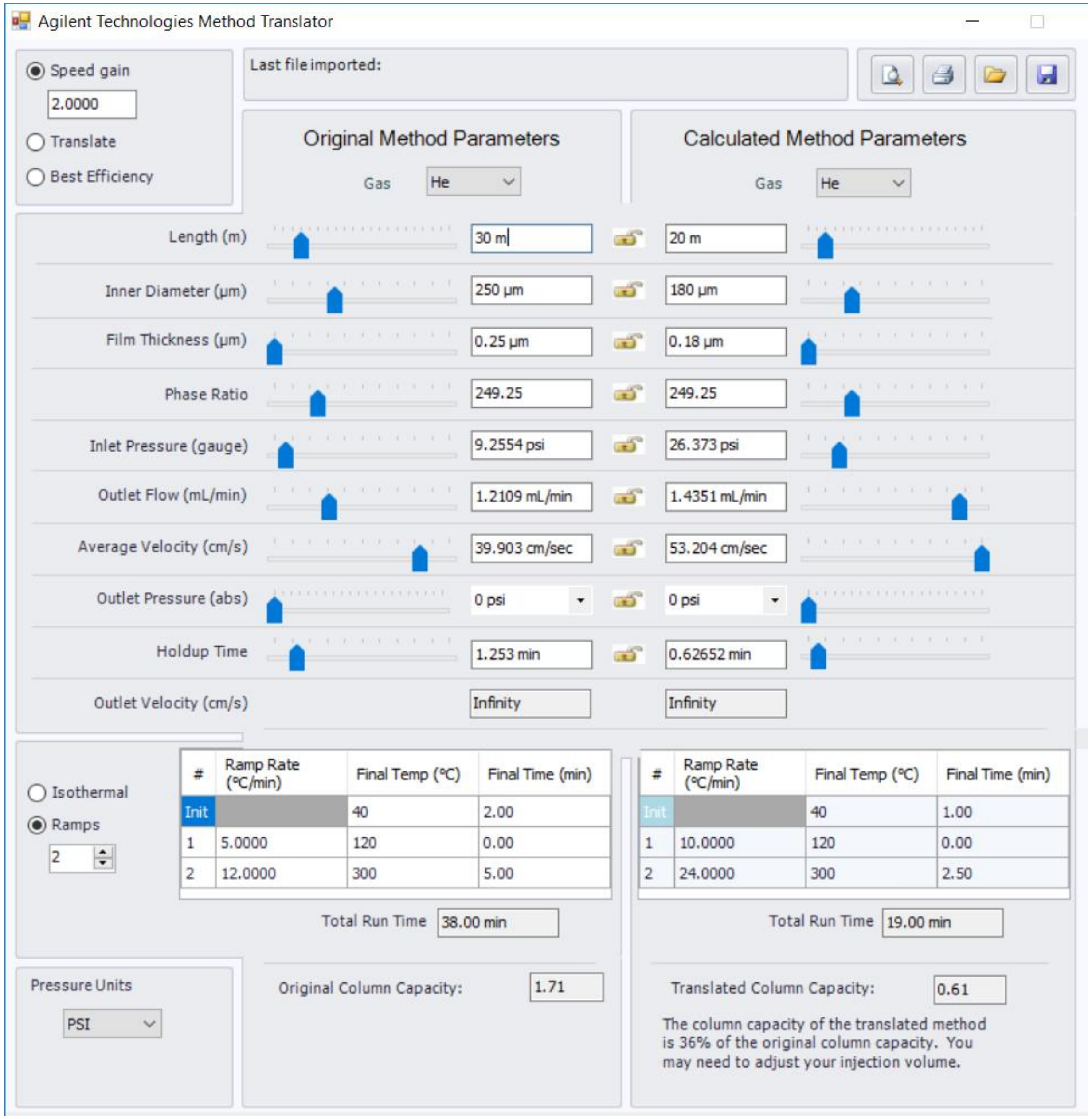
Most difficult to predict and develop

Often involves trial and error

Changes in Column Dimensions, Gas Type or Velocity Require Changes in Temp Program Rates

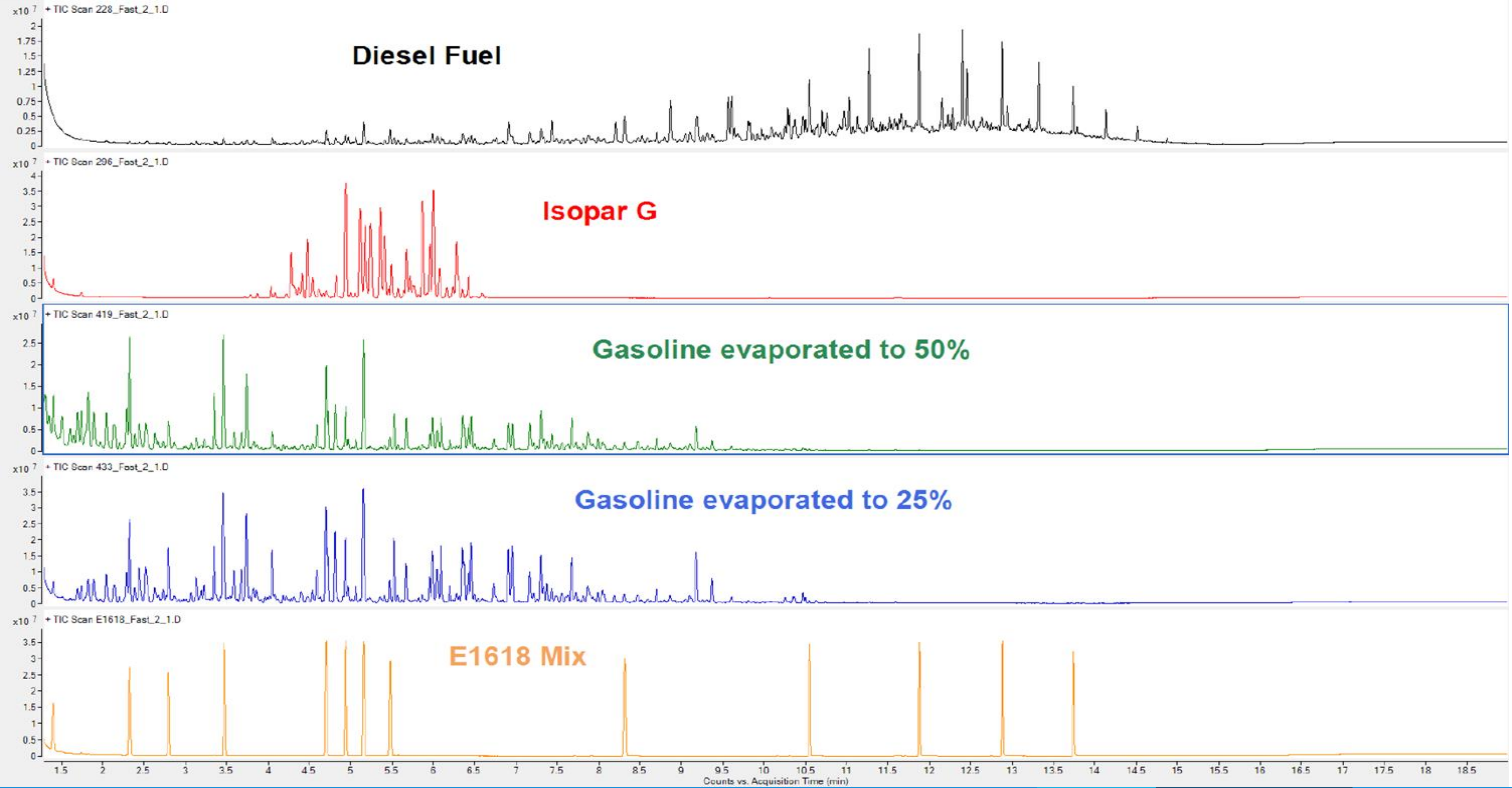
Method Translator

38 min method to 19 minute method



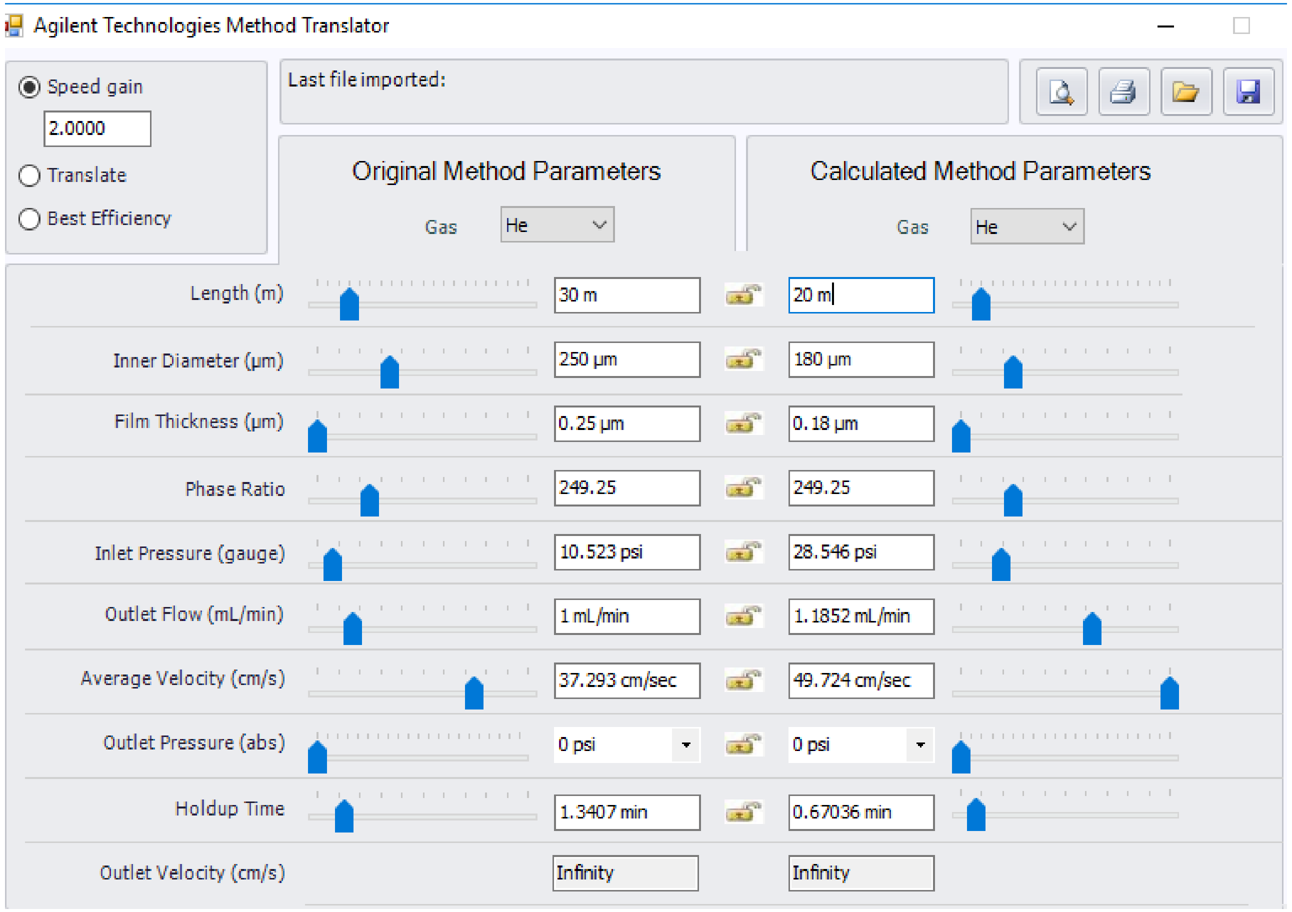
Conventional ASTM Mix E1618 for Fire Debris – 38mins.

Can you tell a difference?



Method Translator QC Mix

Speed Gain



Method Translator QC Mix

Speed Gain

Isothermal

Ramps (selected): 1

#	Ramp Rate (°C/min)	Final Temp (°C)	Final Time (min)
Init		100	2.00
1	20.0000	300	18.00

Total Run Time: 30.00 min

#	Ramp Rate (°C/min)	Final Temp (°C)	Final Time (min)
Init		100	1.00
1	40.0000	300	9.00

Total Run Time: 15.00 min

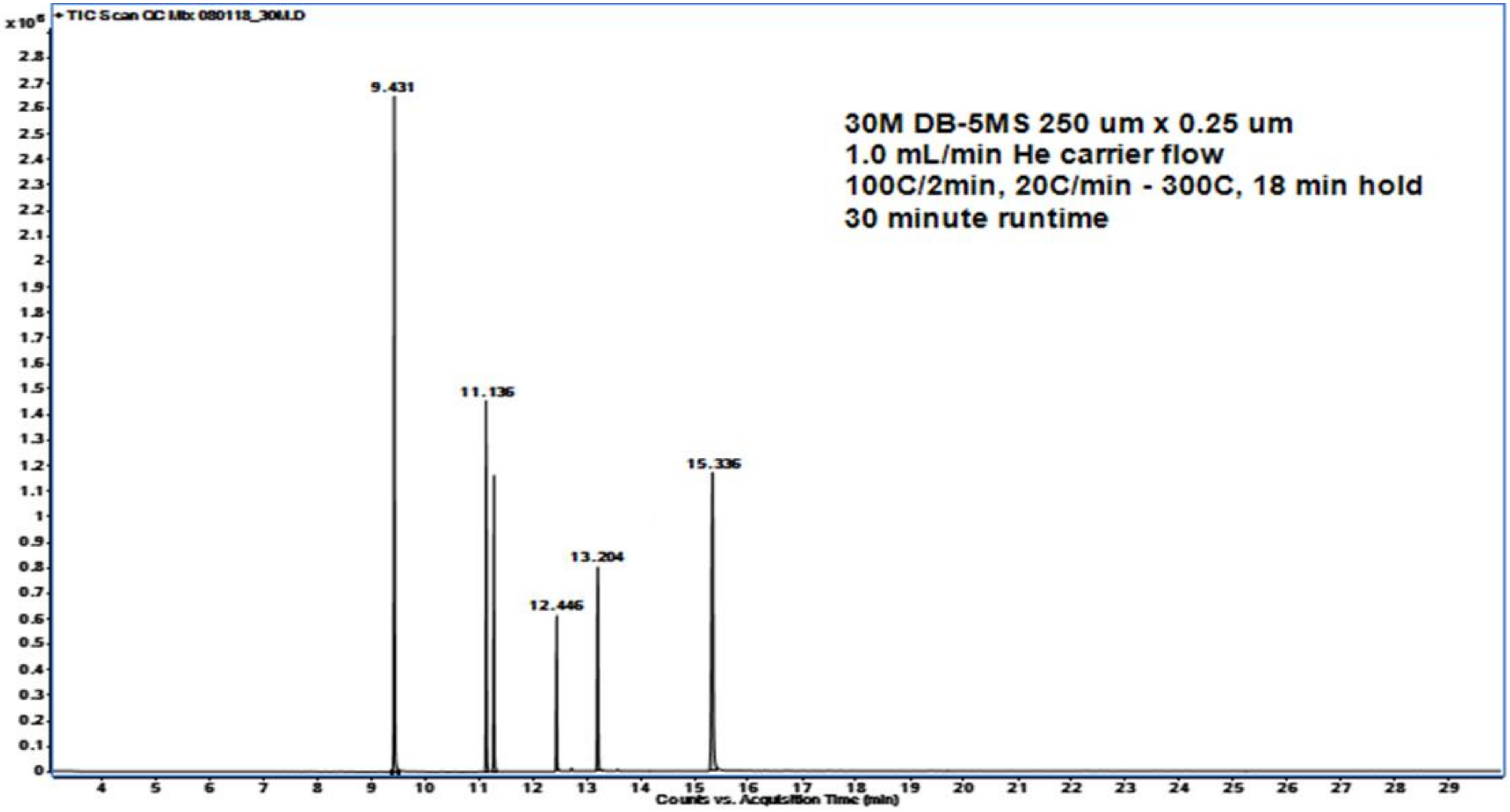
Pressure Units: PSI

Original Column Capacity: 1.71

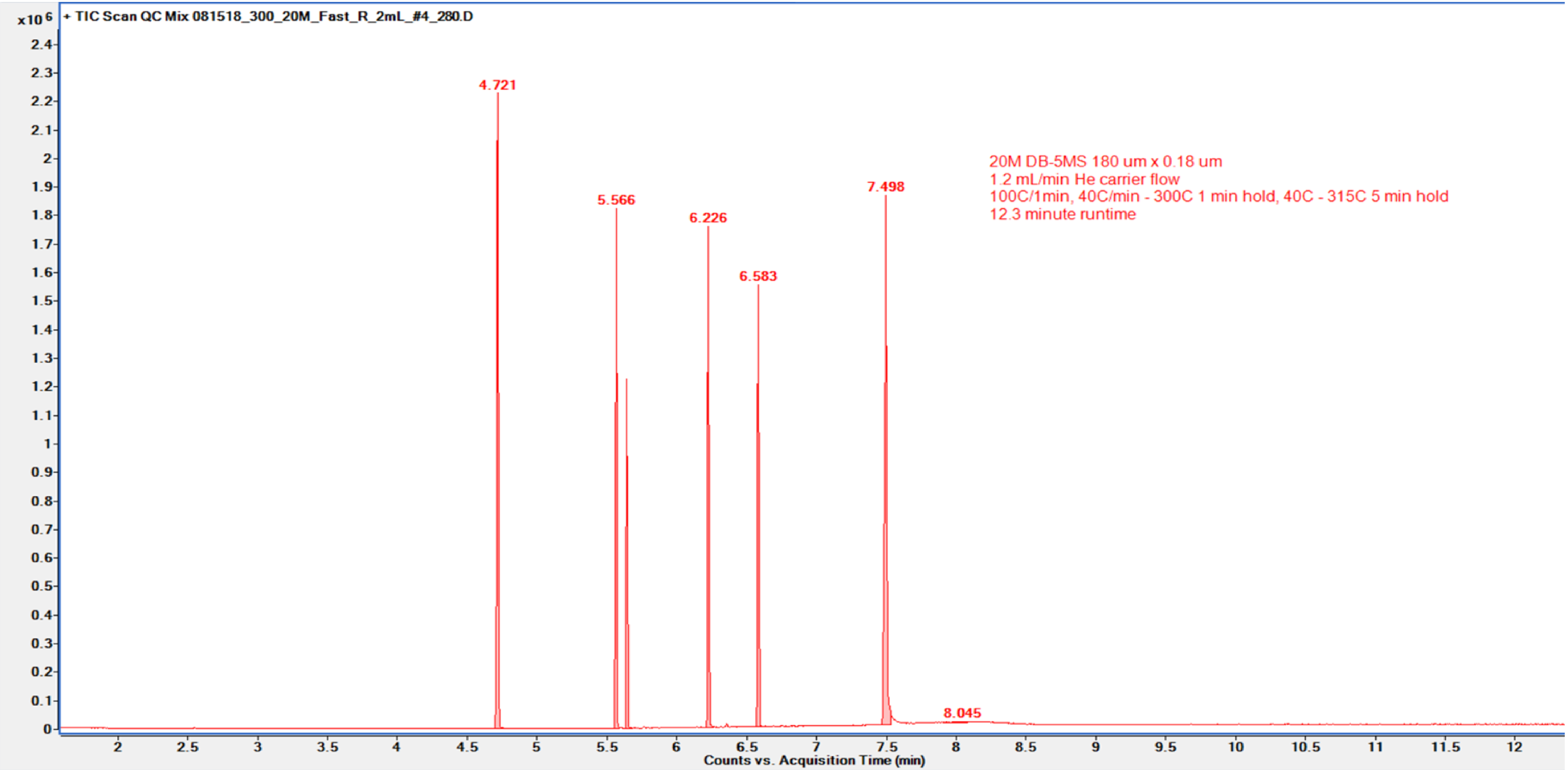
Translated Column Capacity: 0.61

The column capacity of the translated method is 36% of the original column capacity. You may need to adjust your injection volume.

QC Mix 16 minute



QC Mix 8 minute



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In Summary

Diameter – Smaller allows shorter length but has less capacity

Small Change in ID Easier to Translate – Again think capacity

Length – Shorter might be possible without losing a lot of Resolution

Temperature Program – Use Method Translation Software to scale temperatures properly

Method Translation Software – FREE and it works

Hydrogen Carrier – Higher velocities for even faster analyses

Detector Selection – Have realistic expectations for what can be achieved

https://www.agilent.com/en/support/gas-chromatography/gcmethodtranslation