



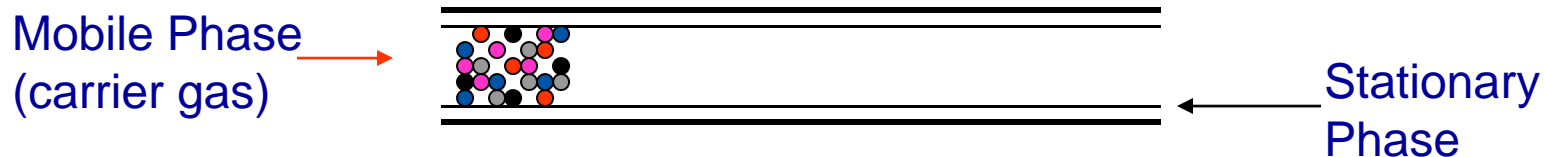
Optimizing Column Selection for Gas Chromatography Applications and Speed of Analysis

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PerkinElmer

Ask Several Questions before Selecting a Column

- ▶ What is the component composition
 - Homologous series of non-polar or slightly polar or polar analytes
 - Mixture of polar and non-polar analytes
 - Volatile components (volatility less than naphthalene)
 - Semi-volatile components (volatility greater than naphthalene)
 - A composite of both volatile and semi volatile components
- ▶ What is the analyte concentration
- ▶ Is sample throughput a concern
- ▶ Is resolution a concern
- ▶ Are there many compounds to separate
- ▶ Is your sample “clean” or “dirty”
- ▶ How is your Gas Chromatograph configured
 - Capillary, packed or PTV injector
 - Detector
 - Backflushing capability

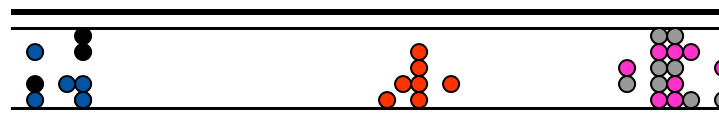
Compounds volatilized by sample introductory system are introduced into column and focus at the head of the analytical column



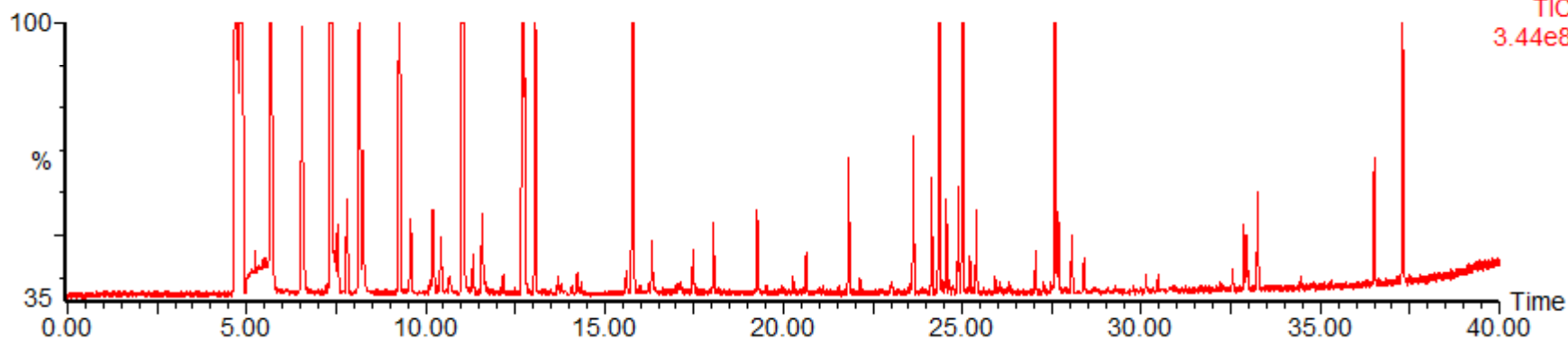
► Separation

- Stationary (liquid) Phase
- Compound Boiling Point (oven temperature program)
- Carrier Flow Rate

- ▶ Analytes more soluble in the stationary (liquid) phase will be retained longer
- ▶ As column temperature increases, less volatile compounds which remained focused will become volatile again and travel down column interacting with stationary phase
- ▶ Analogy - Vinegar and water or Vinegar and oil



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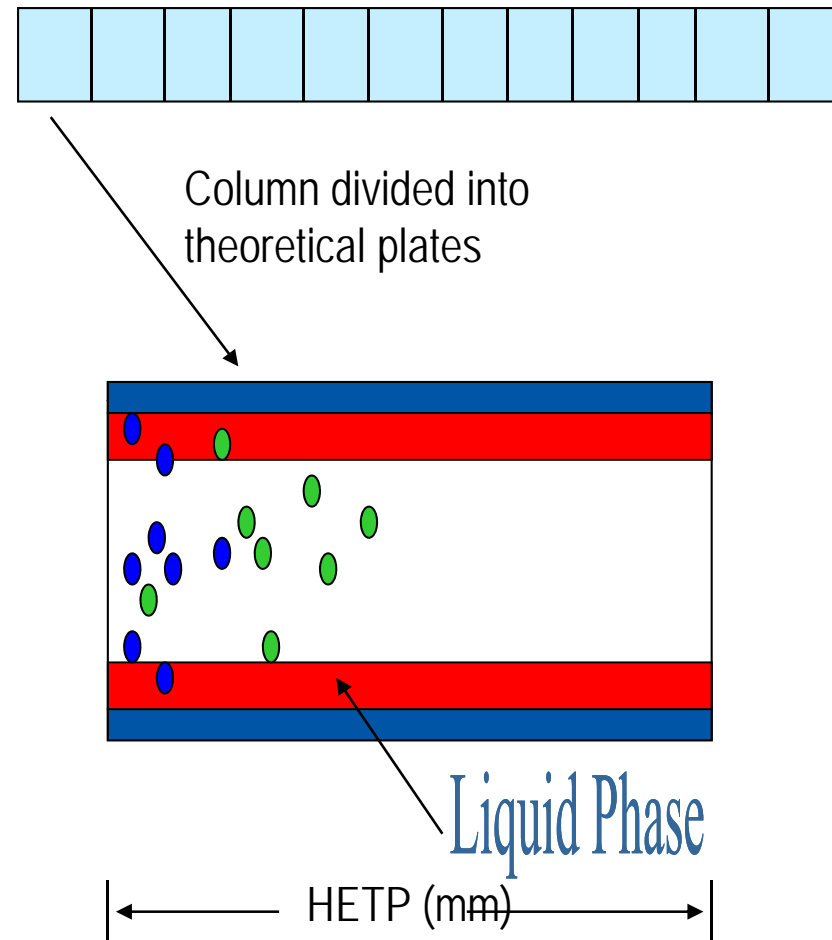




Optimizing for Productivity Using Efficient Columns

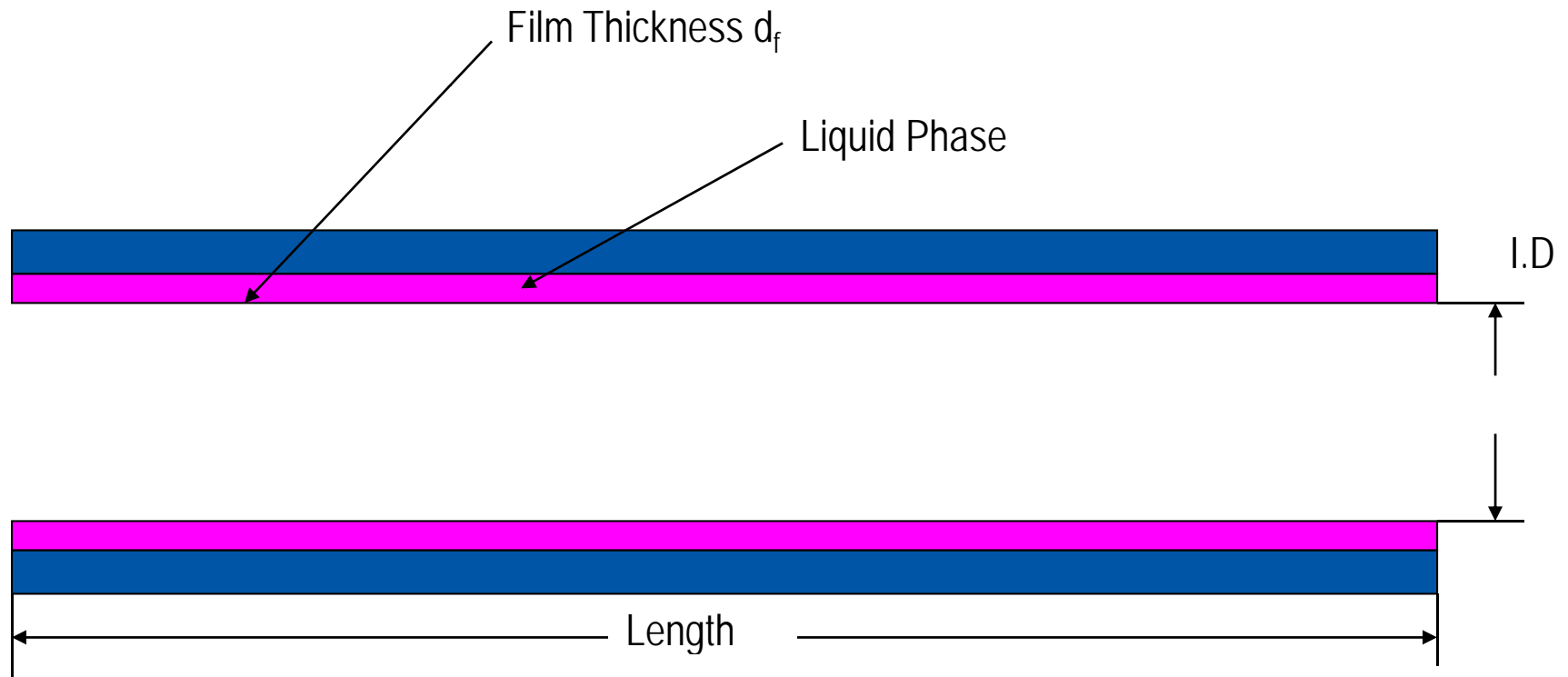
Theoretical Plates: $N = 5.545 (tr/Wh)^2$

- ▶ Benefits of efficiency
 - Enhanced resolution
 - Improved detection limits
 - Faster analysis
- ▶ The column of choice
 - Narrow bore (id) column
 - Thinner film

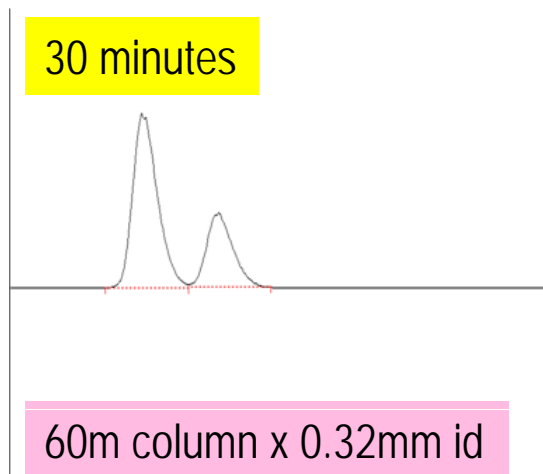
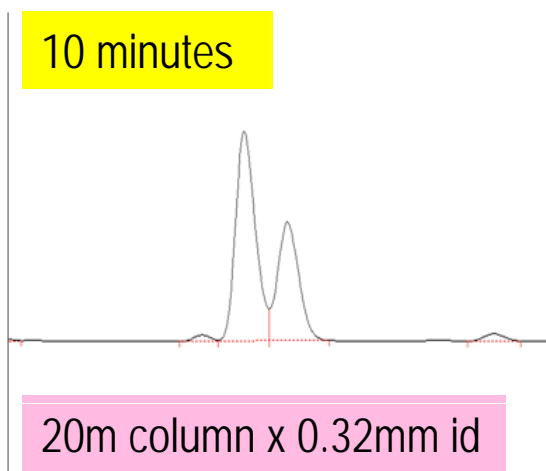


Variables affecting Resolution: $R_s = 2(t_{r2} - t_{r1}) / W_1 + W_2$

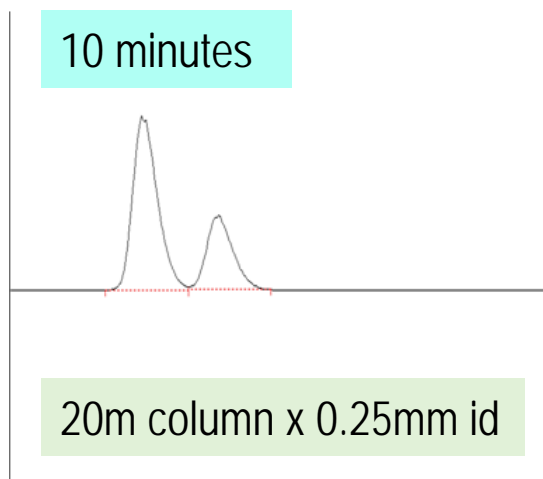
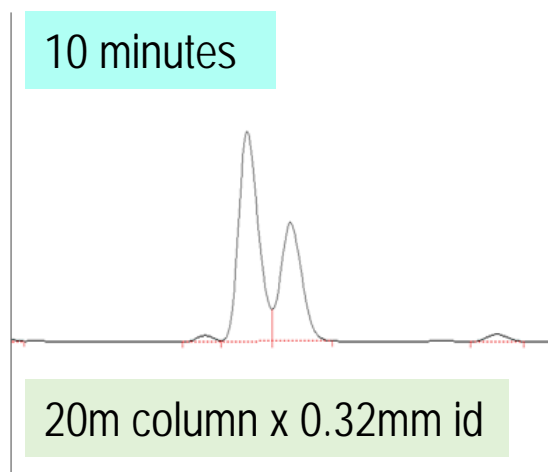
$$R = \left(\frac{1}{4} \sqrt{\frac{L}{H}} \right) \left(\frac{k}{k+1} \right) \left(\frac{\alpha-1}{\alpha} \right)$$



Resolution: Narrower is Better than Longer



$$R \sim L^{1/2}$$

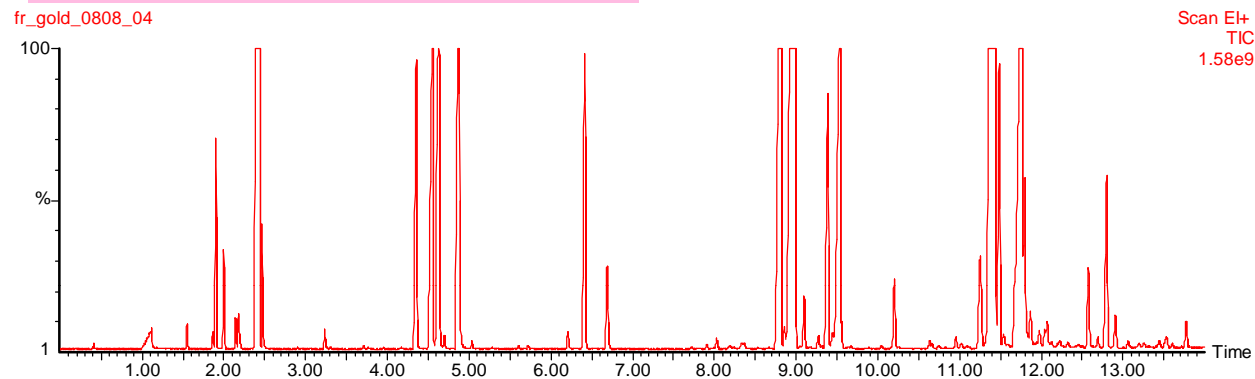


$$R \sim 1/I.D.$$

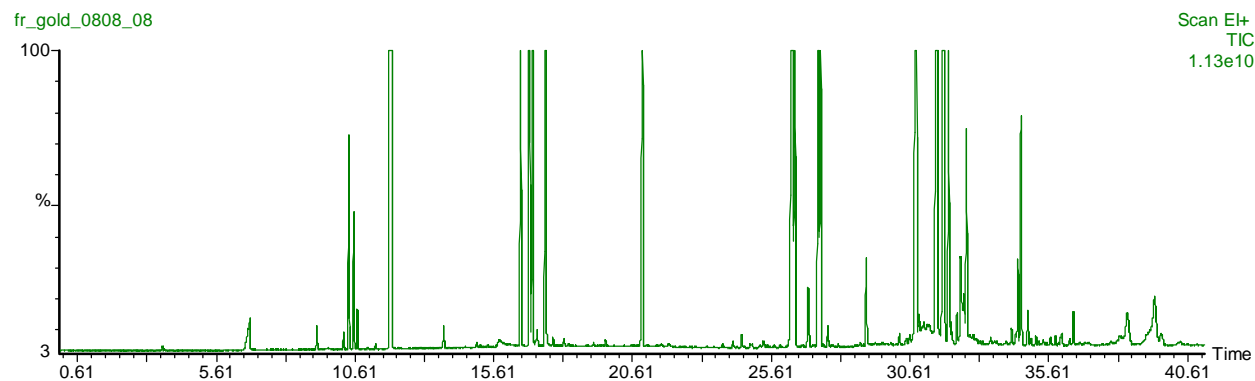
14 minutes versus 45 minutes – Fragrance

Faster
Improved Resolution

Elite 1 – 10m x 0.18mm x 0.4µm

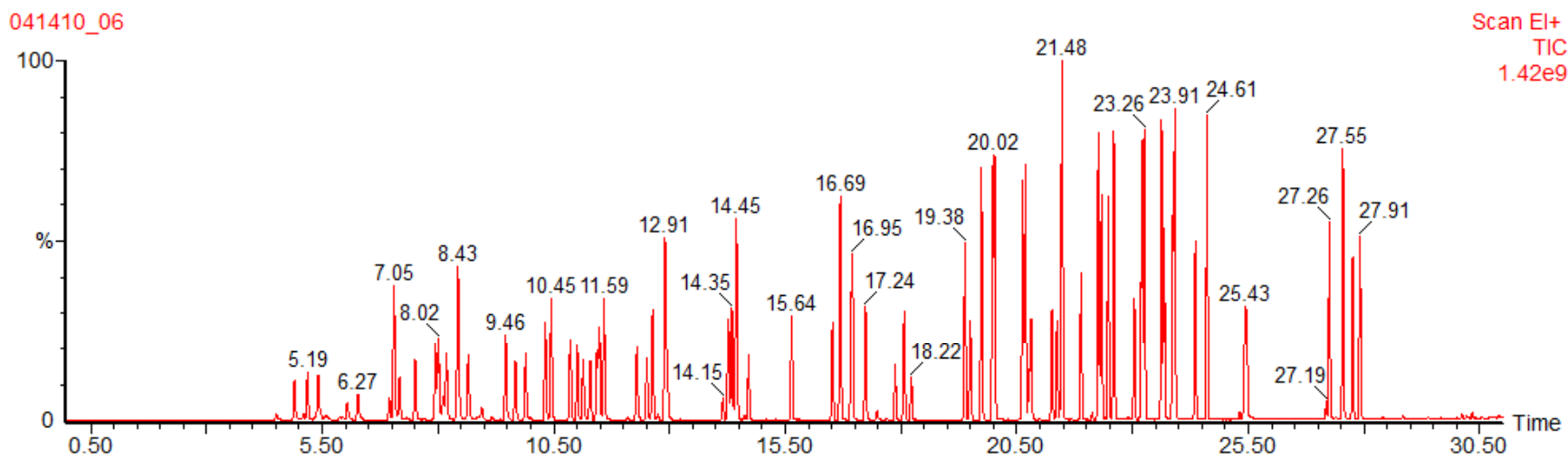


Elite 1 – 60m x 0.25mm x 0.5µm

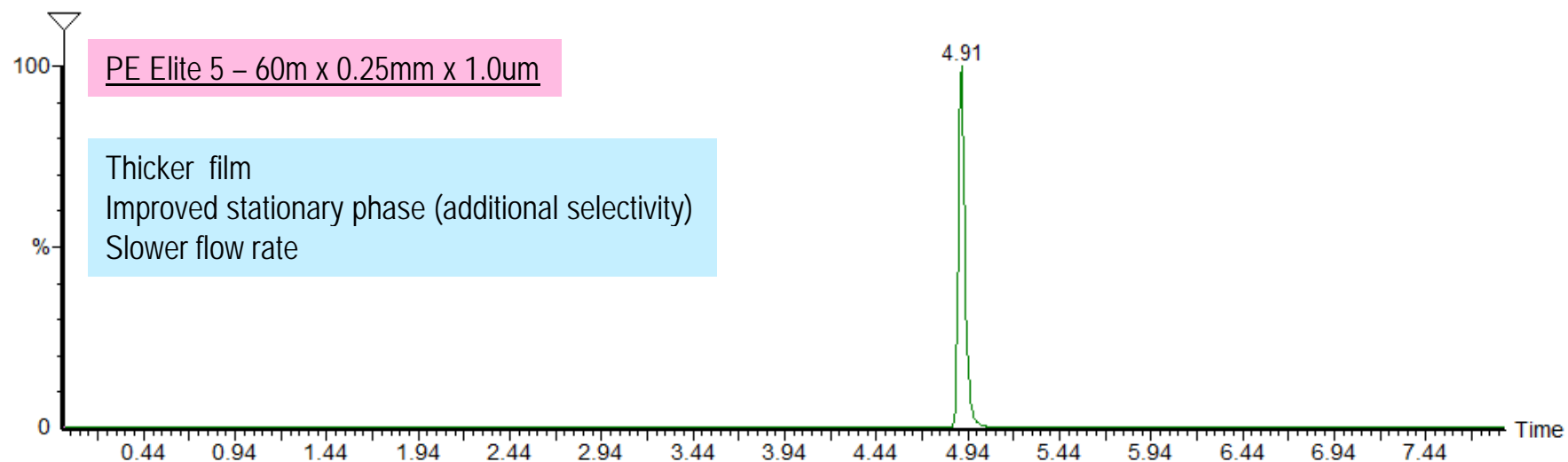
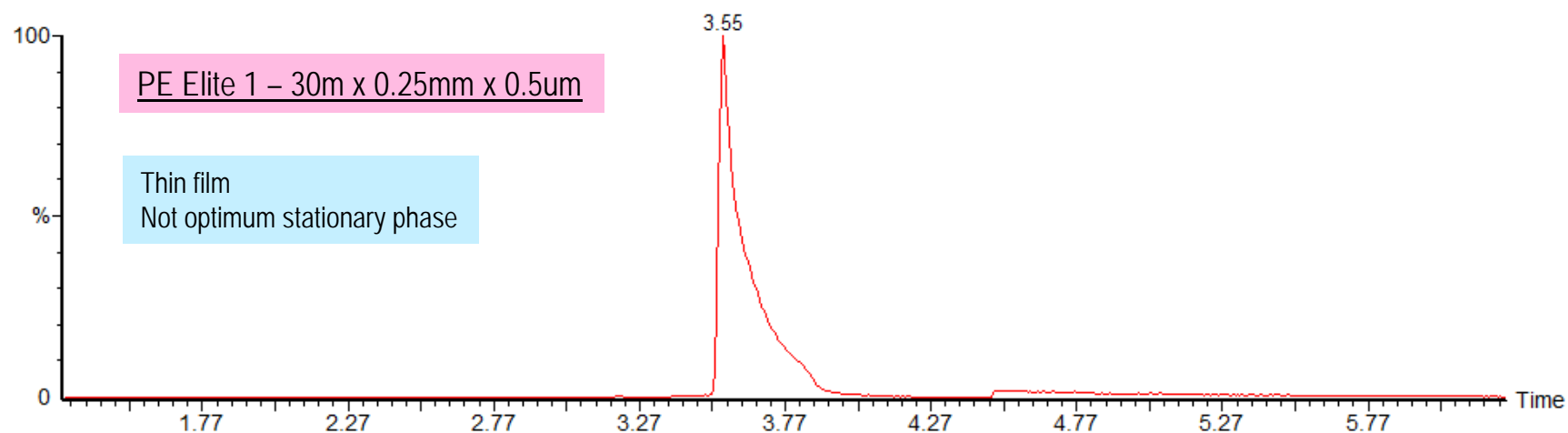


EPA Method TO-17: Volatile Organic Compounds in Air

PE Elite 5 – 60m x 0.25mm x 1.0um

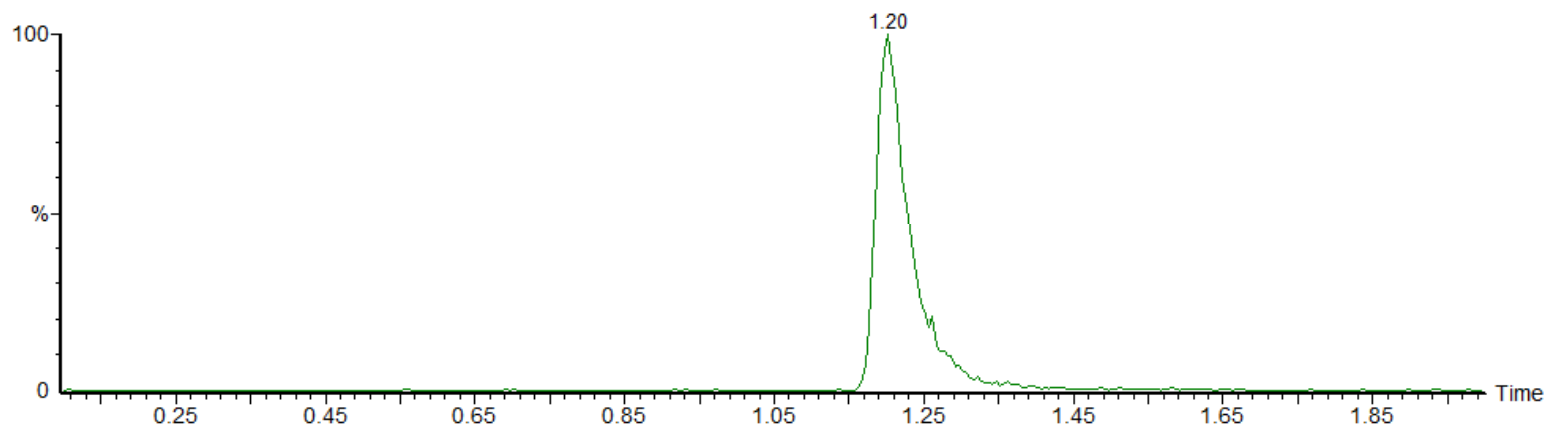


Focusing of volatile components



Shorter, more efficient column with optimum stationary phase

- ▶ Elite 624 – 20m x 0.18mm x 1.0µm
 - Able to focus very volatile gases
 - Reduced analysis time from 30min to 14min
 - Didn't need to use thicker film – reduces MS maintenance

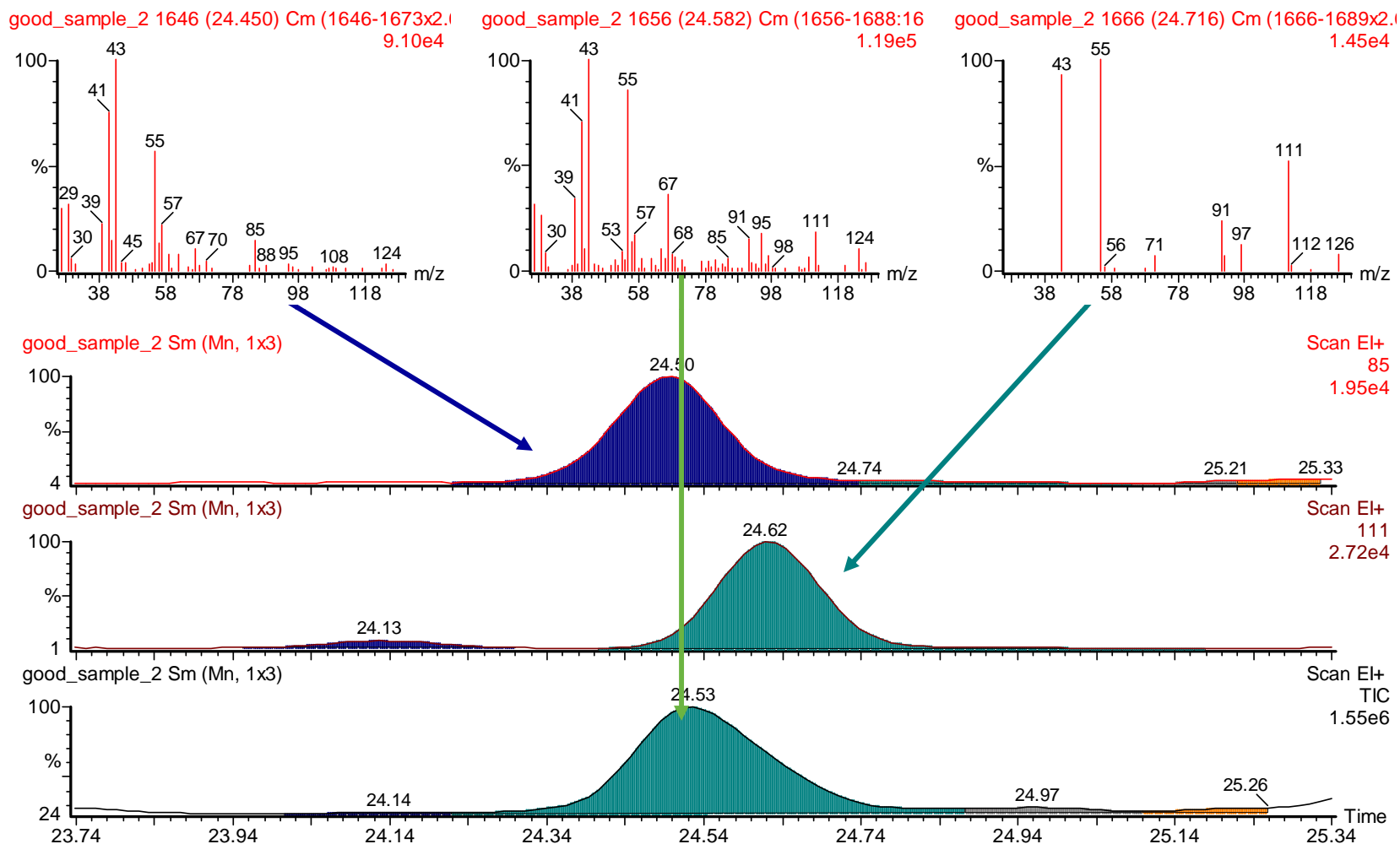




Optimizing for Productivity Using Mass Spectrometry

- ▶ Both a Universal and Selective Detector
- ▶ Enables aggressive detection limits
- ▶ Positive Identification with standard or user library spectrum
- ▶ Interference Free Quantification – chromatographic separation may not be necessary
- ▶ Combines separation and identification techniques
 - 3 dimensional data – *Intensity, Time, Identity*
- ▶ Provides both quantitative and qualitative information about your sample

Universal and Specific typically Interference Free Integration





Optimizing for Productivity

Using Backflush and Dean Switch
Techniques in Conjunction with
Column Technology

Benefits:

- Extends column life
- Increases interval before required maintenance
- Faster analysis
- More samples analyzed in a day



Methanol in Crude Oil
(ASTM D-7059)

Benzene and Toluene in
Gasoline
(ASTM D-3606)

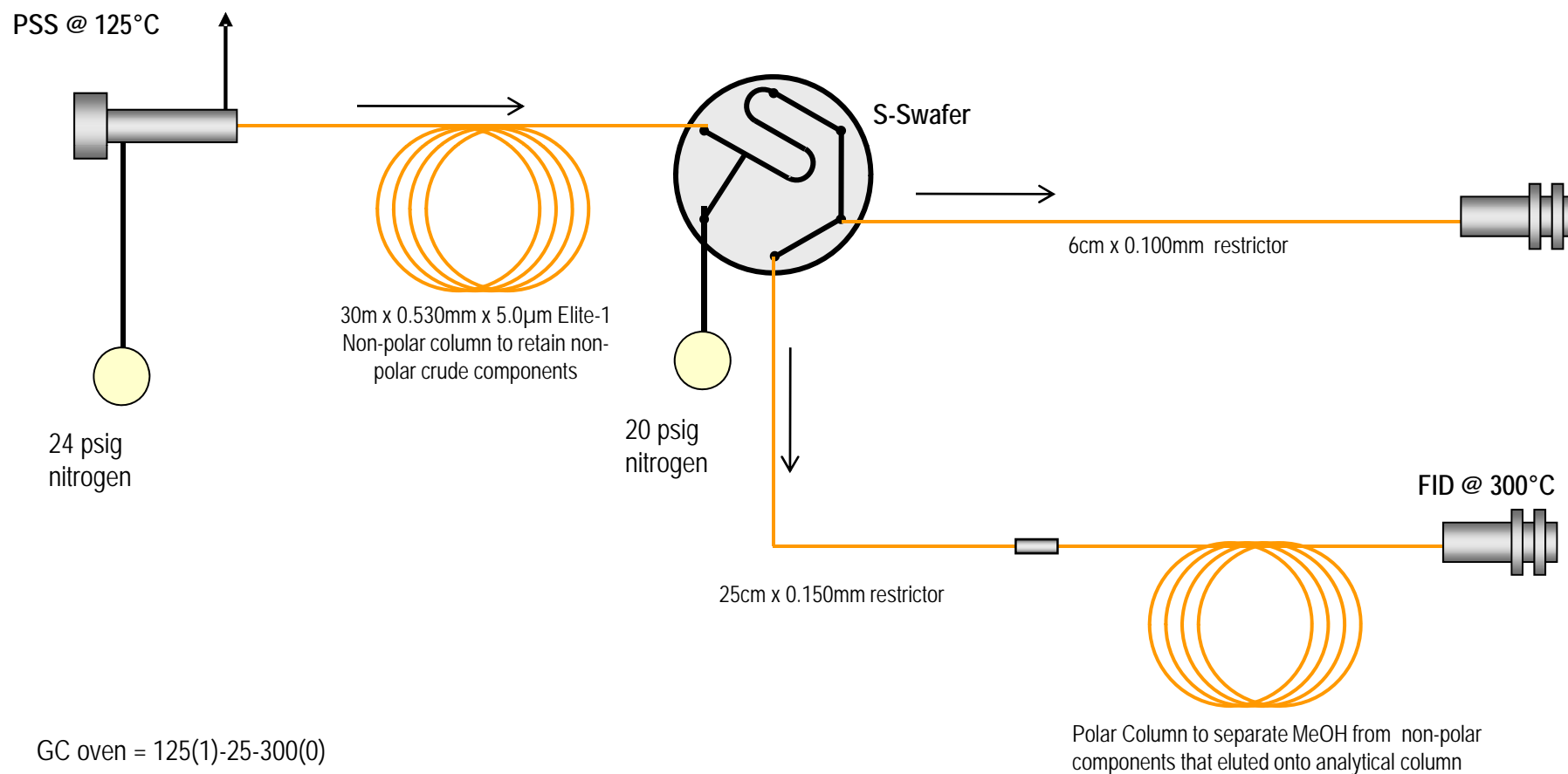
Methanol in Crude Oil Analytical Configuration-Automated

Temperature Programmable (PSS) Injector

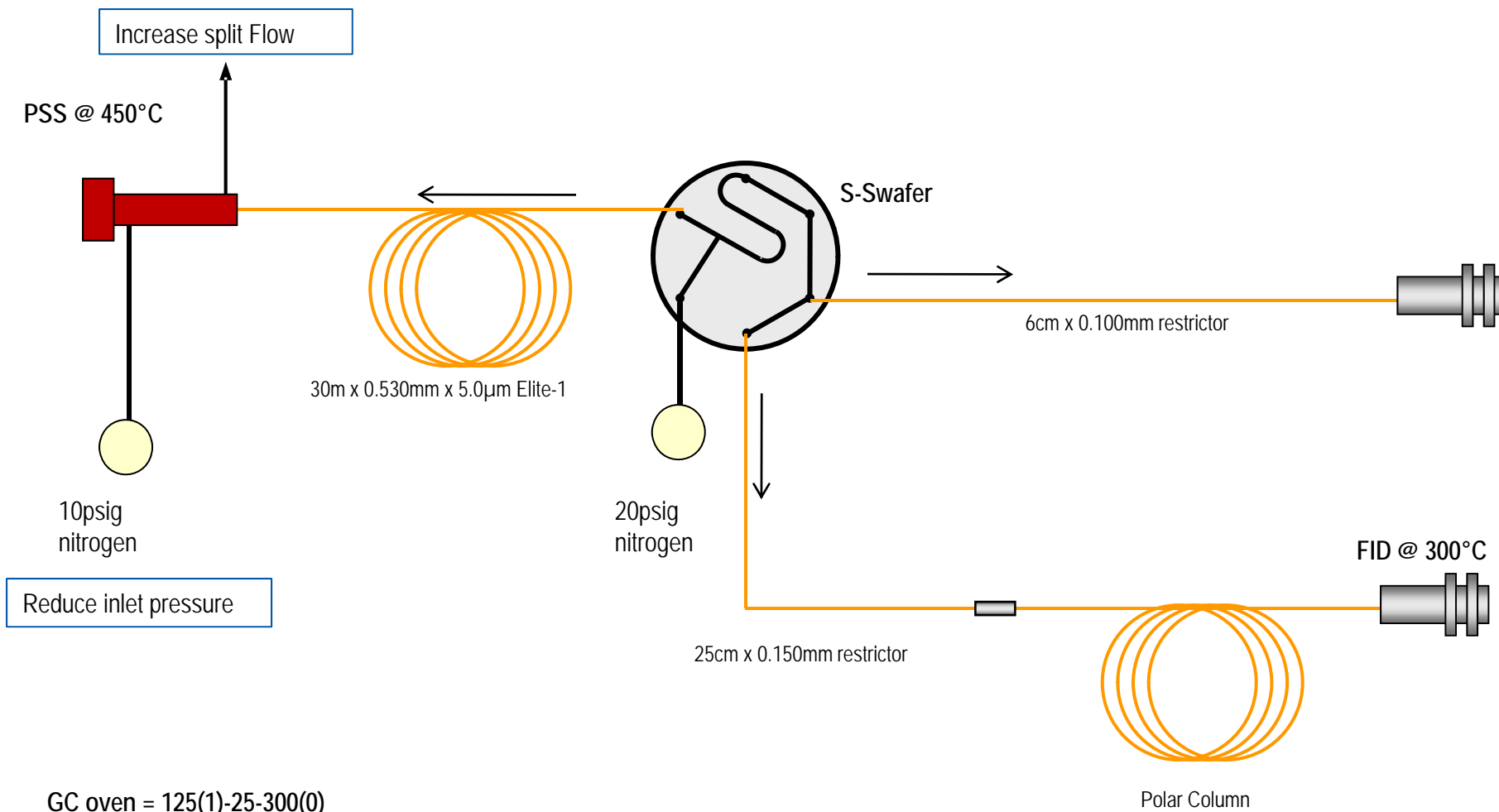
- Select a starting inlet temp which will retain most of the high boiling point crude components in inlet while volatilizing the required lower boiling point targets of interest
- Prevents most of the crude from entering the pre-column

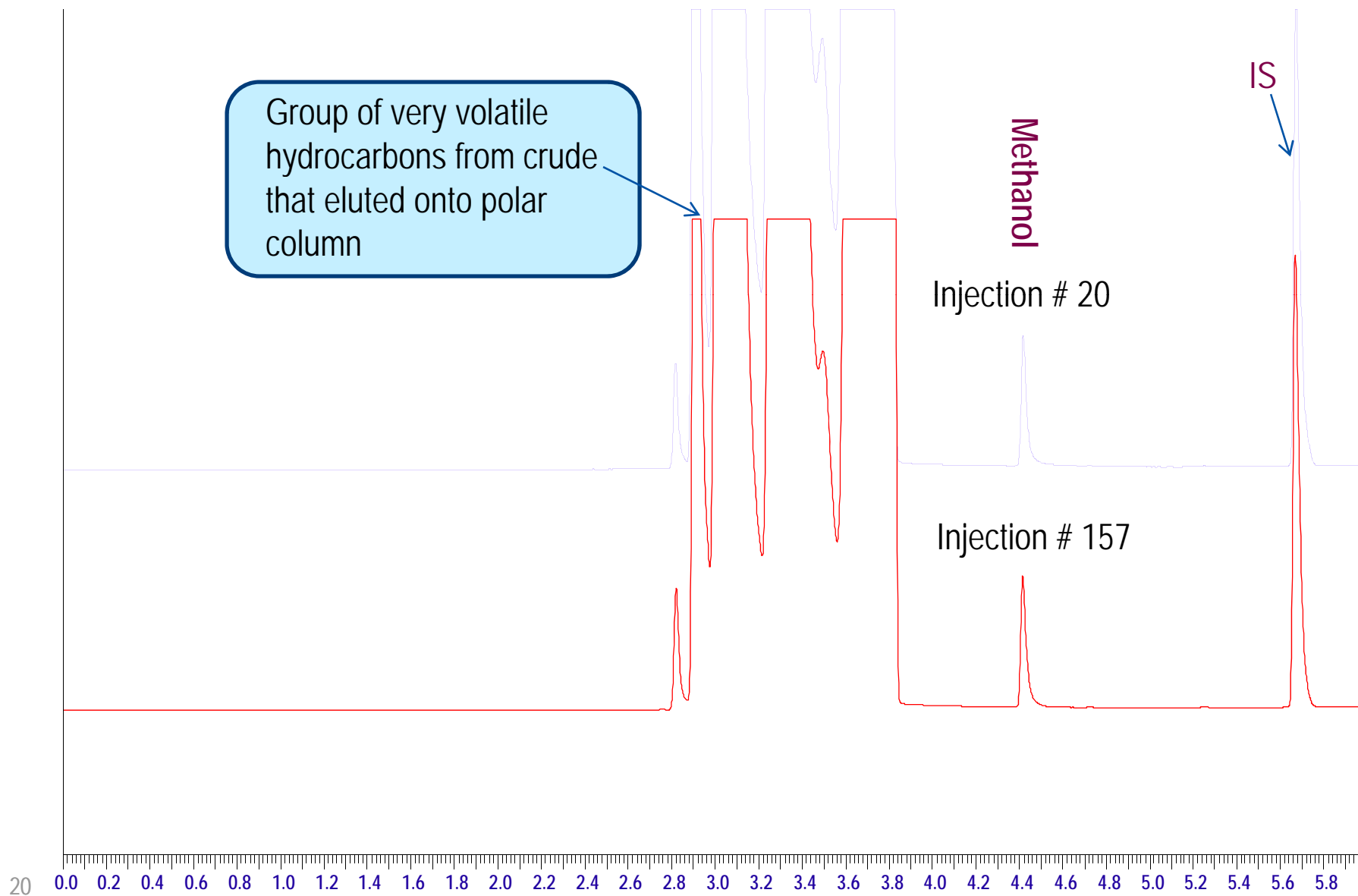
Swafer™ (micro channel flow switcher)

- Provides ability to pressure tune
- Enables backflushing during chromatography
- Prevents unwanted analytes to enter analytical column
- Easy to use – Pressure and time

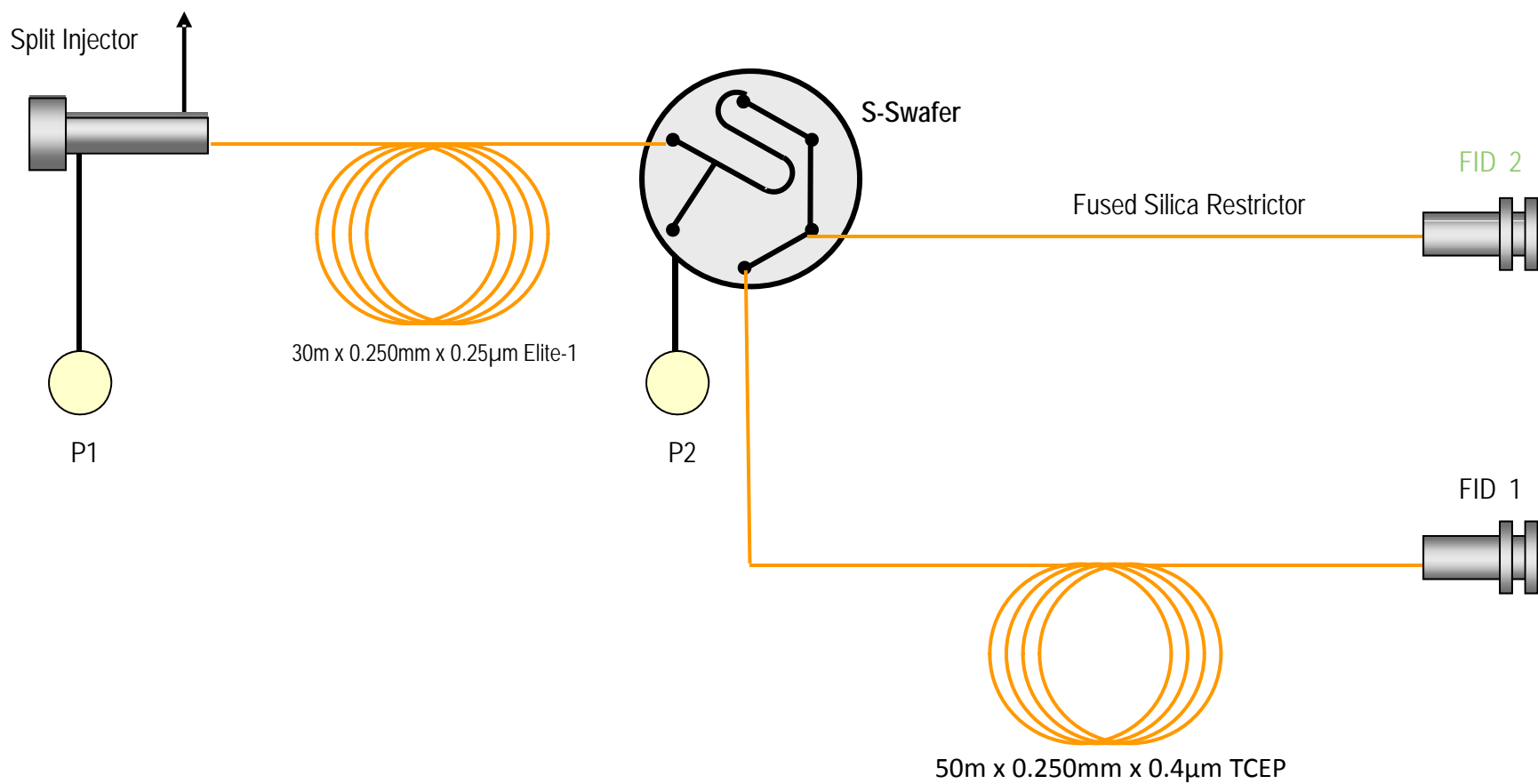


Conditions to Backflush



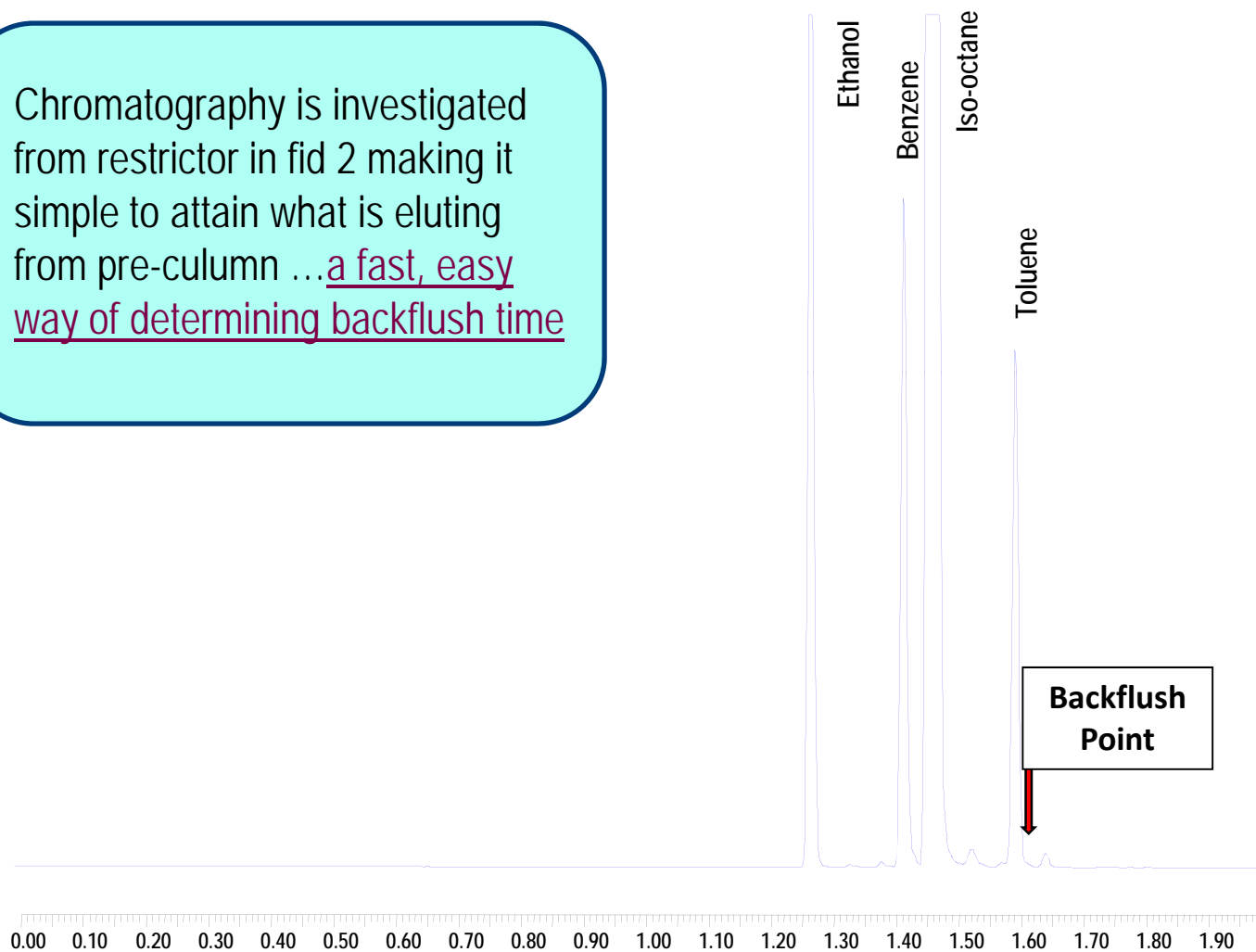


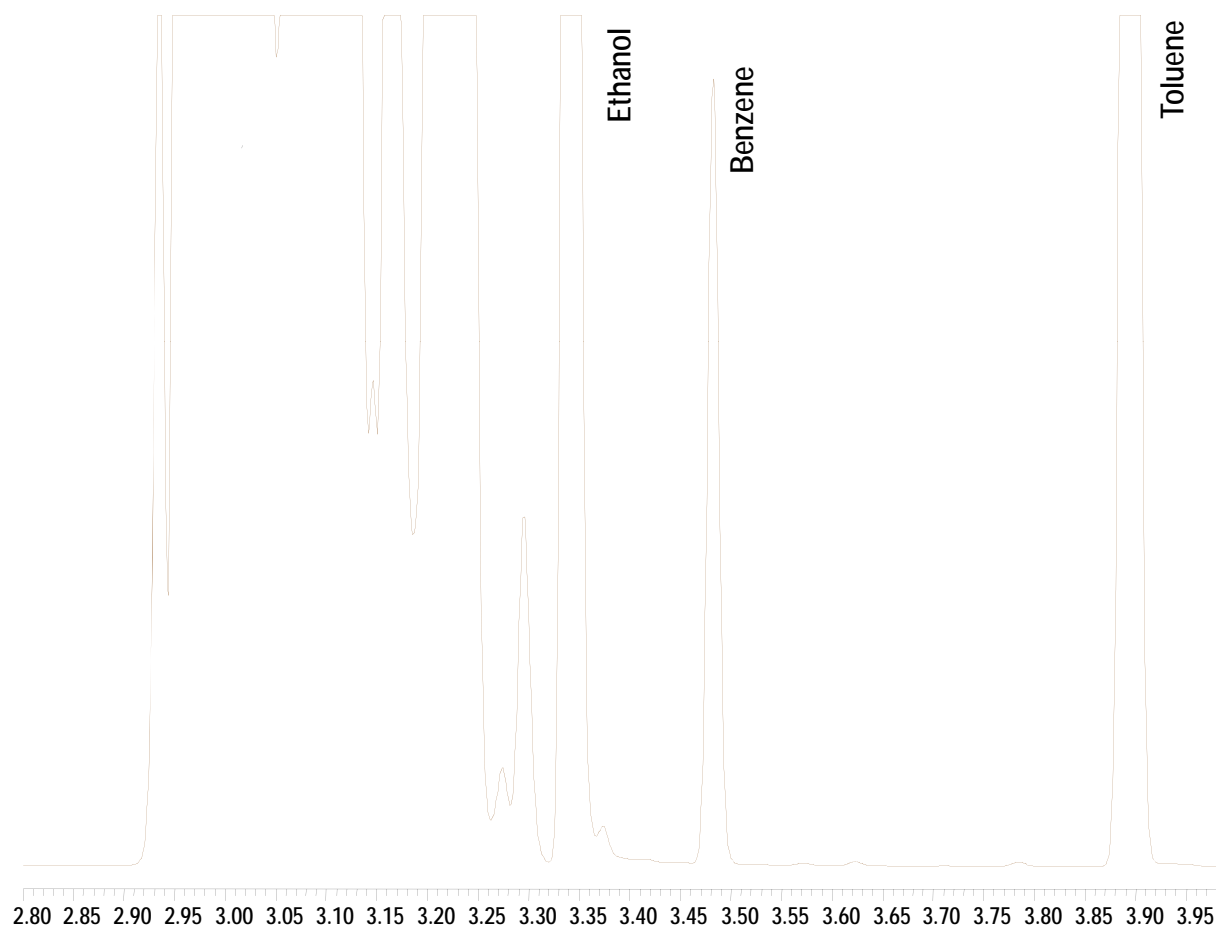
ASTM D-3606: Benzene and Toluene in Gasoline



Determining Backflush Time

Chromatography is investigated from restrictor in fid 2 making it simple to attain what is eluting from pre-column ...a fast, easy way of determining backflush time





- ▶ A brief general statement
 - For semi-volatiles (environmental), PE Elite 5 – 20m x 0.18mm x 0.4um
 - For high molecular weight non-polar compounds, PE Elite 1 – 10m x 0.25mm x 0.1um is great!
 - For focusing volatiles on a non-polar column, a thicker film is preferred. If semi-volatiles are not present, using a volatile stationary phase like an Elite 624, 1301, volatiles, etc. will allow a thinner film
 - Polar compounds may not chromatograph properly on a non-polar stationary phase. Concern ... polar stationary phases like a Wax is not as robust as non-polar columns and have strict temperature limits ie 240 degrees.
 - Most columns have temperature limits. This may dictate your column selection
 - There are specialty columns for active compounds like Amines, Carboxylic Acids, sulfur components, etc. that may require consideration
- ▶ Disadvantages to narrow id columns
 - Stationary phase capacity (increase split or dilute sample)
 - Linearity
- ▶ All products discussed in this presentation are available on www.perkinelmer.com

Thank you ...

we appreciate your time
Questions?



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