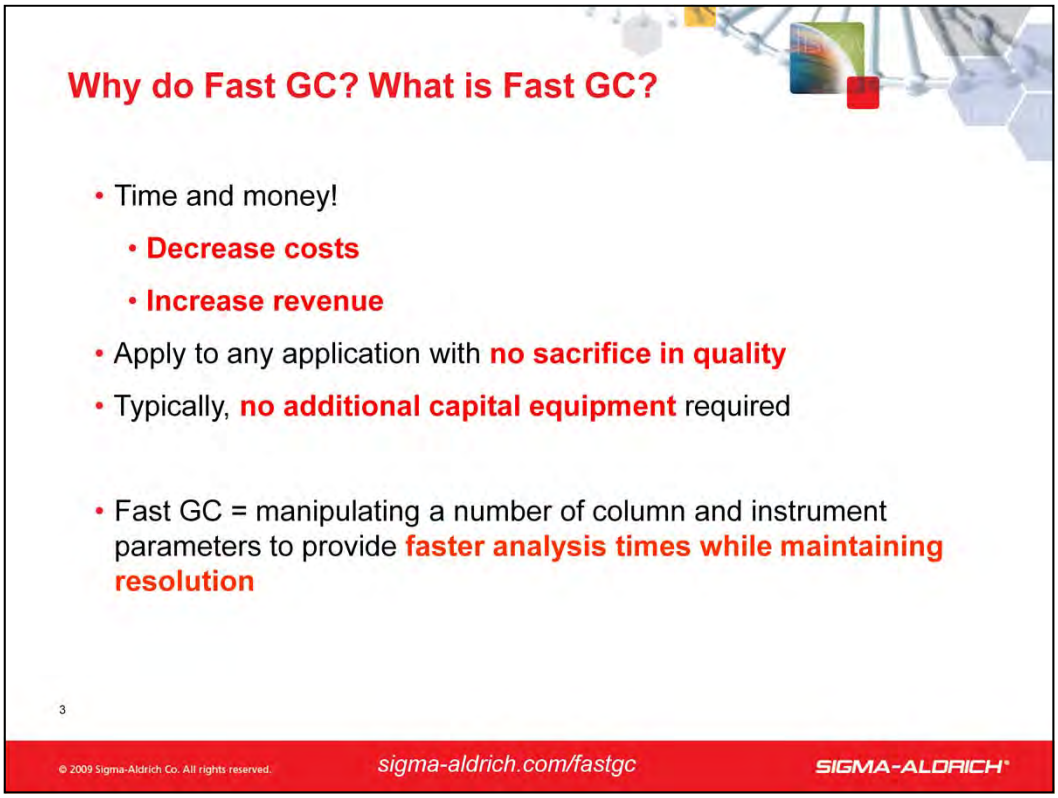
Abstract:
Shorter analysis times allow increased sample throughput, which translates to the completion of more samples per shift. However, any decrease in analysis time must not diminish the resolution necessary to adequately resolve peaks of interest, or to identify specific elution patterns.

The information presented will show you how to apply the Principles of Fast GC to any application, using your existing instrumentation. It will provide a background in the basic theory behind Fast GC, and highlight the practical aspects of making it work. Popular applications, such as BTEX, PAHs, FAMEs, volatiles, and semivolatiles, will be shared.

We begin by answering “Why” Fast GC should be considered, defining “What” it is, and providing the six Principles of Fast GC.



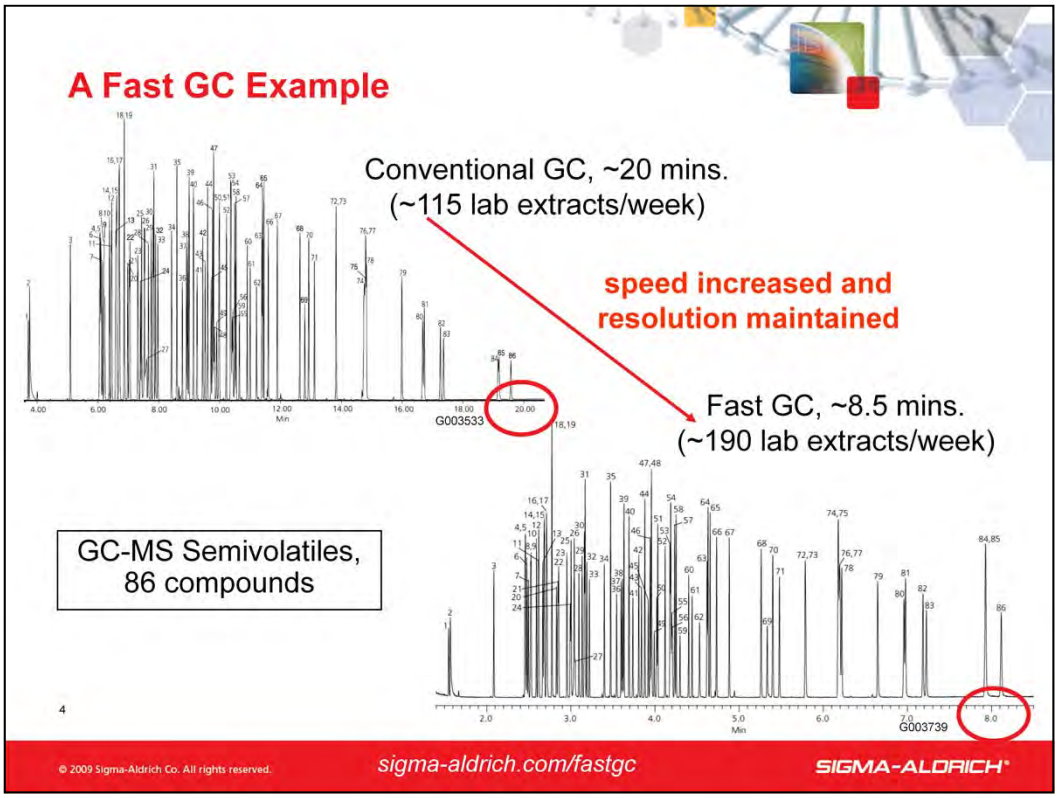
Why should you consider implementing Fast GC into your lab? Time and money!

Fast GC yields faster analysis times than conventional GC, often three to ten times faster. The main benefits to a laboratory are that:

- Costs can be decreased if fewer analysts and/or instruments are needed
- Revenue can be increased if more samples are analyzed
- Fast GC can be applied to any application with no sacrifice in quality!
- Lastly, Fast GC typically does not require any additional capital equipment.

So, what exactly is Fast GC? Simply stated, Fast GC is the manipulation of a number of parameters to provide faster analysis times while maintaining resolution. These parameters include:

- Column dimensions, such as the inside diameter (I.D.), length, and film thickness
- Oven temperature ramp rates
- The carrier gas type and/or linear velocity
- The type of stationary phase



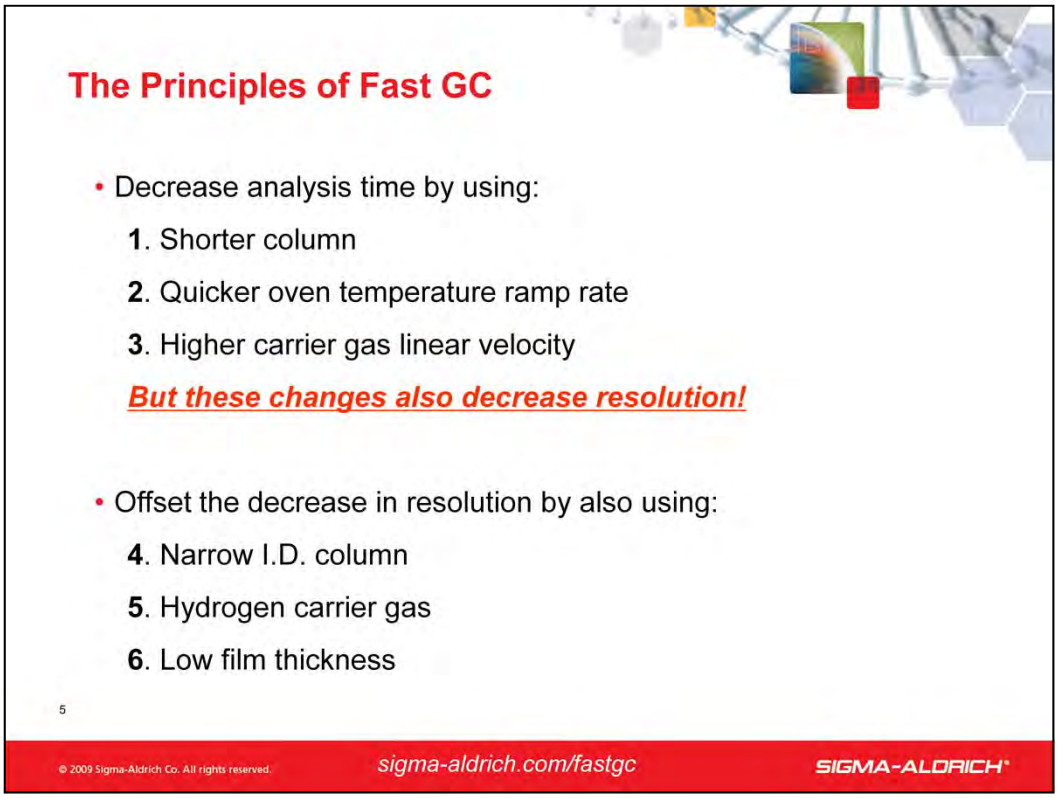
Here is an example of why Fast GC should be considered. Both of these chromatograms are the analysis of GC-MS semivolatiles, an application routinely performed in environmental laboratories. This method requires the GC-MS to be 'tuned' and calibrated prior to the analysis of any lab extracts (blanks, QA samples, and billable samples), and that all lab extracts must be injected within 12 hours of when the 'tune' solution was injected. The shorter the run time, the more lab extracts that can be run within the 'tune' window.

The top chromatogram was obtained using conventional GC. Assuming a single 'tune' window is set-up per day, each instrument can analyze ~115 lab extracts per week after taking into account the cool down period between runs.

The bottom chromatogram was obtained after applying the Principles of Fast GC. Assuming a single 'tune' window is set-up per day, each instrument can now analyze ~190 lab extracts per week after taking into account the cool down period between runs. This increase of 75 lab extracts per week does not require any increase in staff or equipment. Additionally, the quality of the analysis is not diminished!

Conditions (top chromatogram):
- column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
- oven: 40 ° C (2 min.), 22 ° C/min. to 240 ° C, 10 ° C/min. to 330 ° C (1 min.)
- inj.: 250 ° C
- MSD interface: 330 ° C
- scan range: m/z 40-450
- carrier gas: helium, 1.0 mL/min (11 min.), 10 mL/min2 to 1.5 mL/min. (hold remainder of run)
- injection: 0.5 µL, splitless (0.50 min.)
- liner: 2 mm I.D., straight
- sample: 80 component semivolatile standard at 50 ppm plus 6 internal standards (at 40 ppm) in methylene chloride

Conditions (bottom chromatogram):
- column: SLB-5ms, 20 m x 0.18 mm I.D., 0.18 µm (28564-U)
- oven: 40 ° C (0.7 min.), 55 ° C/min. to 240 ° C, 28 ° C/min. to 330 ° C (2 min.)
- inj.: 250 ° C
- MSD interface: 330 ° C
- scan range: m/z 40-450
- carrier gas: helium, 40 cm/sec, constant
- injection: 0.5 µL, 10:1 split
- liner: 2 mm I.D., fast FocusLiner™ inlet liner with taper (2879501-U)
- sample: 80 component semivolatile standard at 50 ppm plus 6 internal standards (at 40 ppm) in methylene chloride



The six underlying Principles of Fast GC are pretty simple.

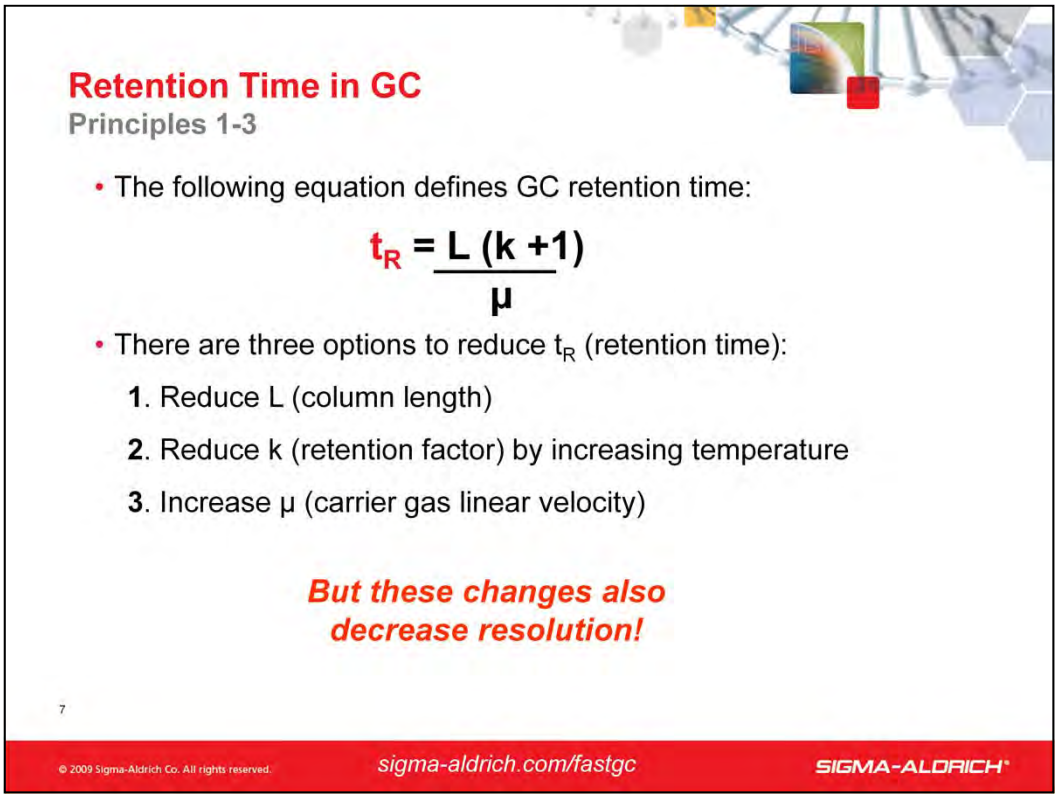
Analysis times can be decreased by using:
- Short columns
- Fast oven temperature ramp rates
- High carrier gas linear velocities

The loss in resolution caused by the above steps can be offset by using:
- Narrow I.D. columns
- Hydrogen carrier gas
- Low film thickness

The more Principles that are applied, the greater the benefit!

Note:
Many of these parameters being manipulated are related to each other. Changing just one may produce a shorter analysis, but may result in a loss in quality. Therefore, all parameters must be evaluated to make sure they are set correctly.

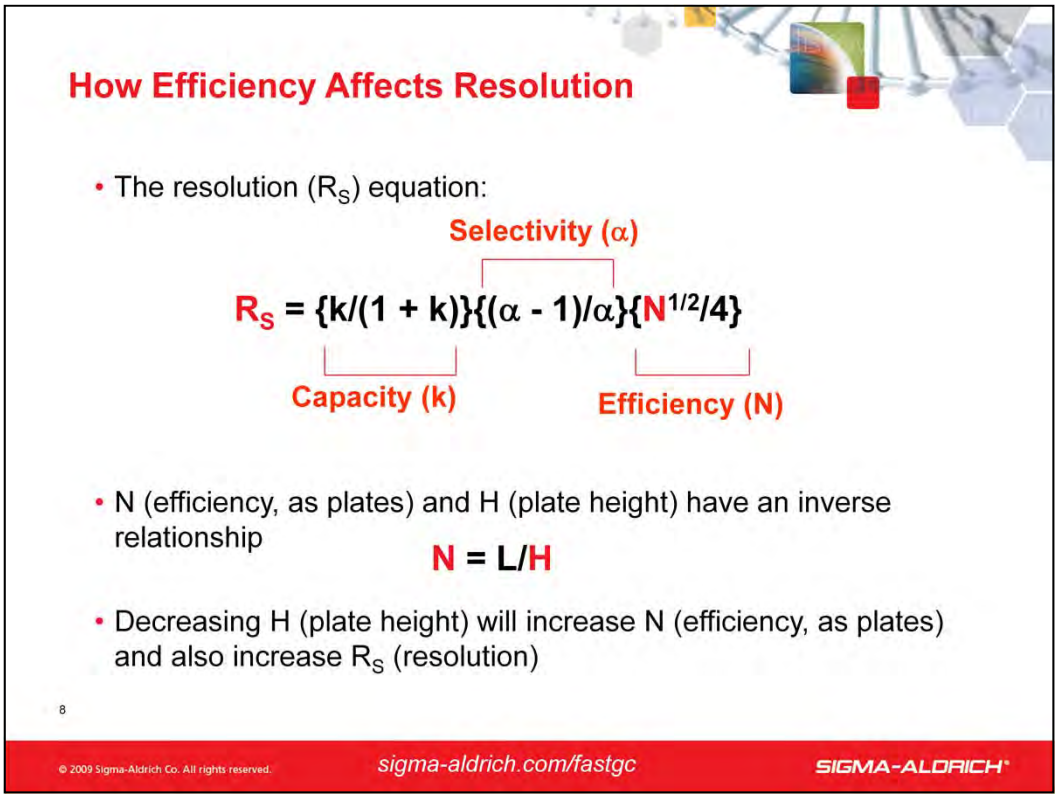
Before we look at how to perform Fast GC, let's take a step back and look at why it works through a short theoretical discussion.



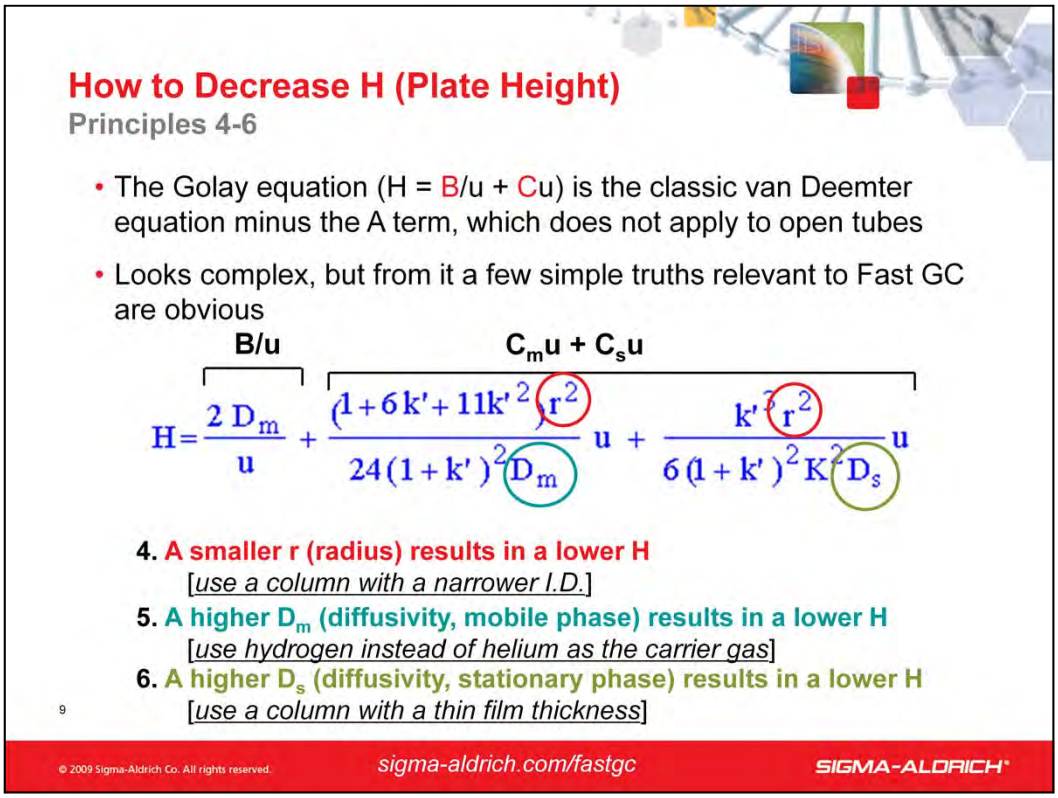
How long analytes are retained in a column dictates the overall analysis time. Simply logic tells us that if retention times can be shortened, the result will be a shorter overall analysis time. The retention time (t_R) of an analyte is a function of column length (L), retention factor (k), and carrier gas linear velocity (u). This equation defines those relationships. For this discussion, we do not need to worry about the correct units for each term. Rather, we are interested in the relationships (cause and effect).

There are three options for reducing retention time:
- Use a shorter column
- Increase oven temperature to reduce analyte partitioning into the stationary phase
- Increase the carrier gas linear velocity to move analytes through the column quicker

These three steps comprise the first half of the Principles of Fast GC. They accomplish shortening analysis time, but sacrifice resolution in doing so. The second half of the Principles of Fast GC focus on gaining back the resolution.



Before we look at the second half of the Principles of Fast GC that focus on gaining back the resolution, we need to understand the relationships between resolution and plate height. The resolution equation tells us that resolution (R_s) is the result of capacity times selectivity times efficiency. Focusing on efficiency (expressed as plates), we see that it is inversely related to plate height (H). If we can decrease plate height (H), we will increase efficiency (N), which in turn will increase resolution (R_s). Therefore, the second half of the Principles of Fast GC deal with decreasing plate height (H) as the means to gain back the resolution lost when the first half of the Principles of Fast GC were applied.



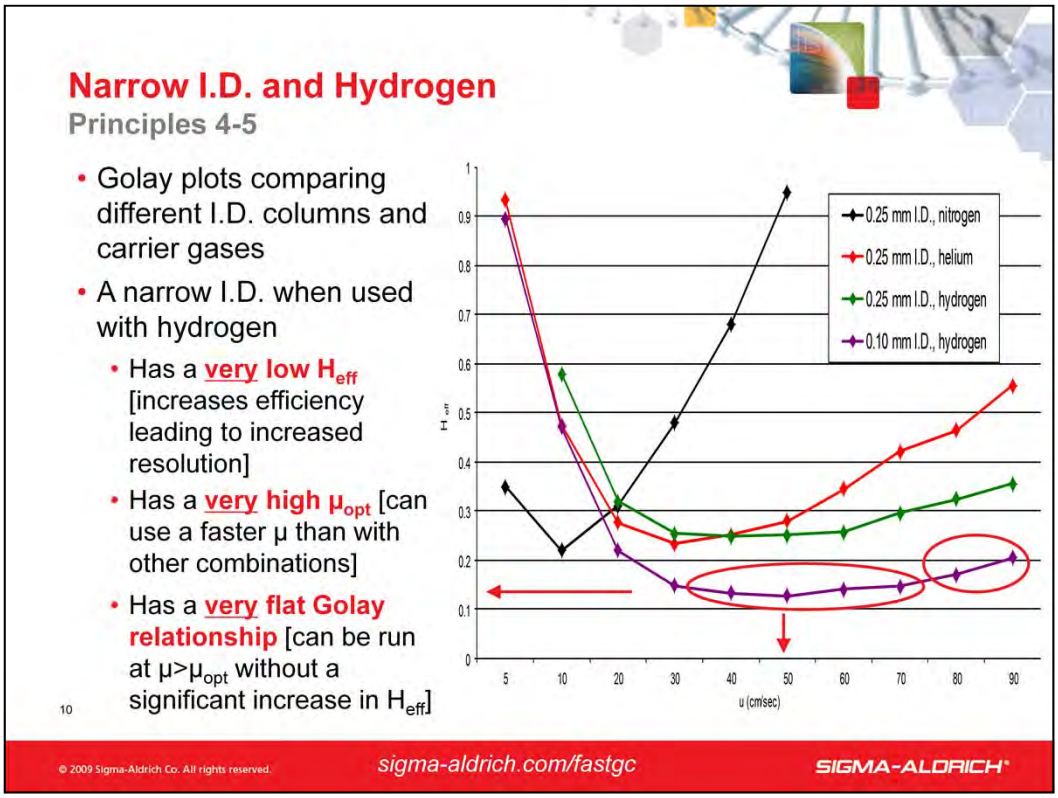
So, how do we decrease plate height (H)? The Golay equation (H = B/u + Cu) is the classic van Deemter equation minus the A term, which does not apply to open tubes. The Golay equation is useful for us because it describes plate height (H), and its relationships to several terms. It looks complex, but from it a few simple truths relevant to Fast GC are obvious:

A smaller radius (r) results in a lower plate height (H) – tells us to use a column with a narrower I.D.

A mobile phase with a higher diffusivity (D_m) results in a lower plate height (H) - tells us to use hydrogen instead of helium as the carrier gas

A stationary phase with a higher diffusivity (D_s) results in a lower plate height (H) - tells us to use a column with a thinner film thickness

van Deemter Review:
- The A term concerns eddy (axial) diffusion [not applicable to open tubes]
- The B term concerns longitudinal diffusion
- The C term concerns resistance to mass transfer

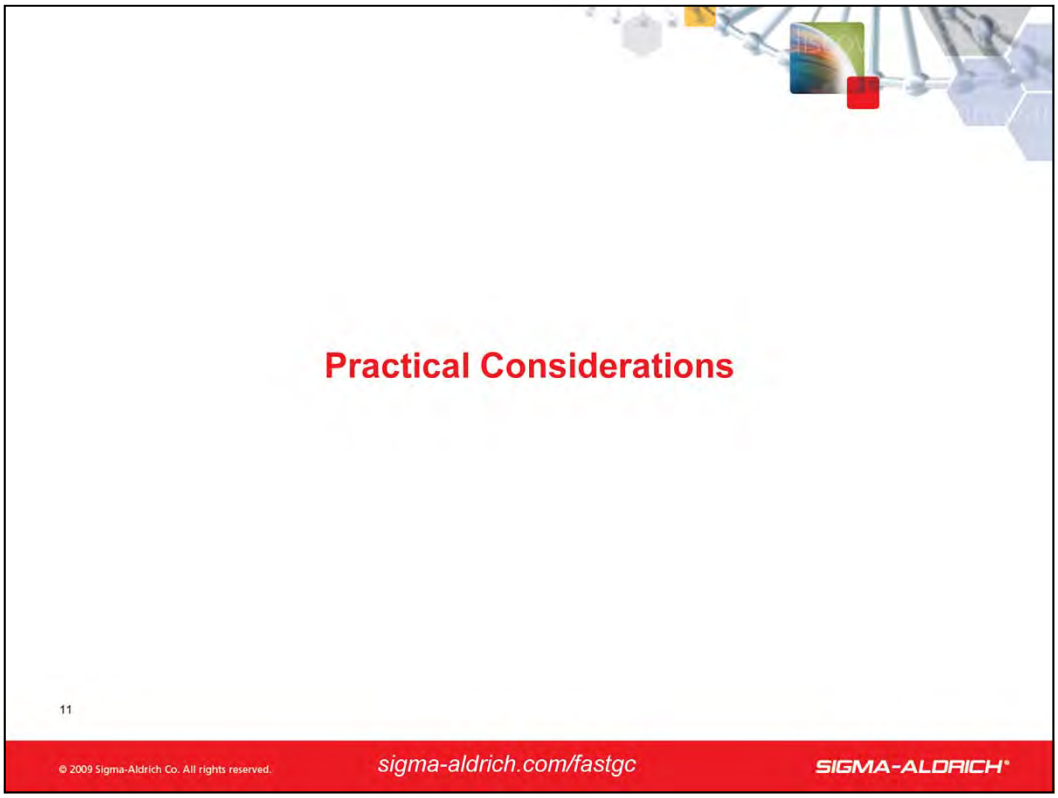


Let's consider column I.D. and carrier gas together. Shown are Golay plots of five combinations of column I.D. and carrier gas. The X-axis shows linear velocity (u), and the Y-axis shows effective plate height (H_{eff}). The phrase optimal linear velocity (u_{opt}) is used to define the linear velocity value when the Golay plot is at its lowest. Lower plate height (H) values result in higher resolution, and that higher linear velocity (u) values result in shorter analysis times. From a Fast GC point of view, we want to choose a column I.D. whose Golay plot reaches low and to the right.

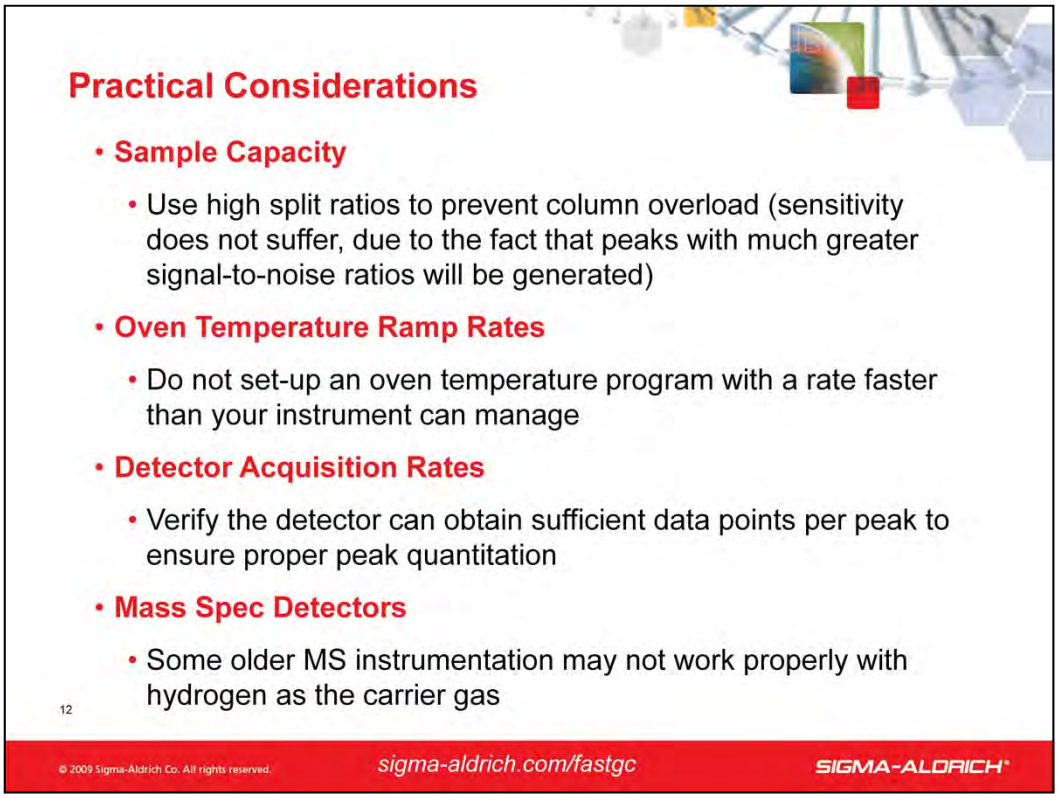
For Fast GC, a narrow I.D. column used with hydrogen is the best choice, because:
- It has a very low effective plate height (H_{eff}) compared to other combinations, which increases efficiency and leads to increased resolution
- It has a very high optimal linear velocity (u_{opt}) compared to other combinations, which allows faster analysis
- It has a very flat Golay relationship compared to other combinations, which allows the use of a liner velocity (u) greater than optimal (u_{opt}) without a significant increase in effective plate height (H_{eff})

* Most impressively is that this combinations can be used with a linear velocity of 80-90 cm/sec, and still exhibits a H_{eff} lower than other combinations run at their u_{opt} values

Note: Data for a 0.10 mm I.D. column with helium carrier gas could not be obtained due to high backpressure.



Before we get into a walk-through of applying Fast GC to an application, there are a few practical considerations we need to be aware of.



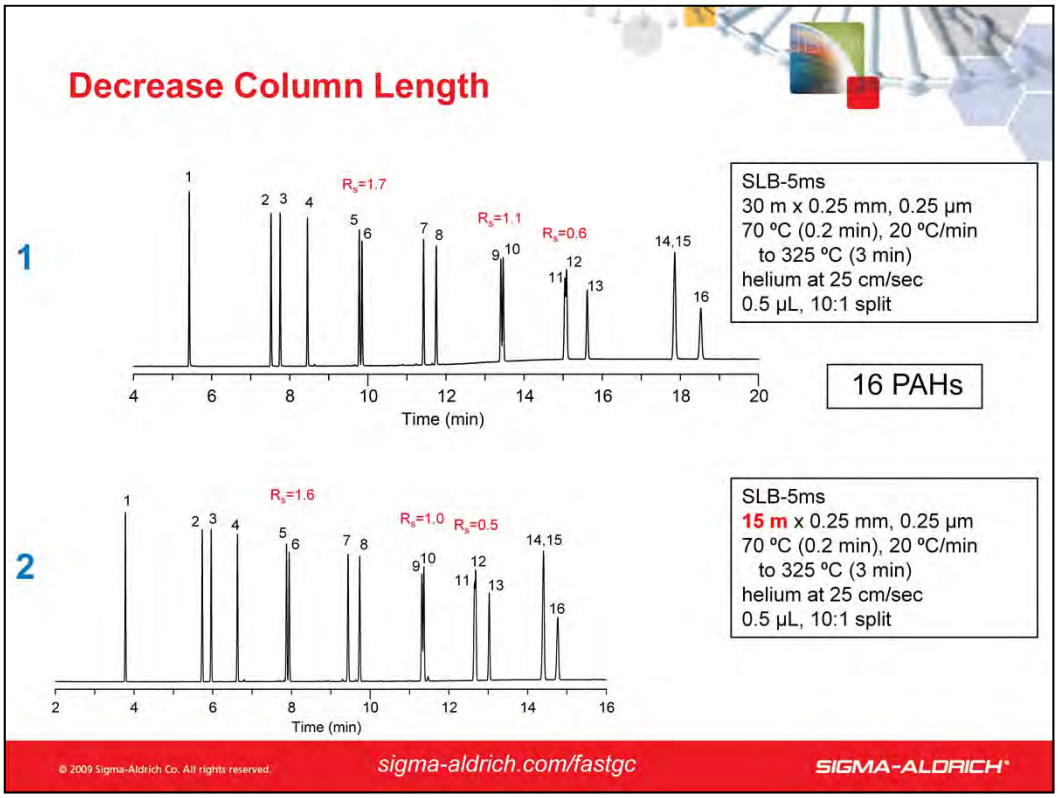
Fast GC column dimensions (narrow I.D. and thin film) have lower sample capacities compared to conventional column dimensions. That is, a smaller amount of sample can be introduced onto the column before peak shapes become distorted. Therefore, high split ratios may be required to prevent column overload.

As we have already discussed, fast oven temperature ramp rates are essential to decreasing analysis time. However, it is important to know the ramp rate abilities of your GC for the temperature ranges in which you will be operating. Programming a ramp rate that is faster than your GC can handle may result in variations in retention time. Make sure to check your instrument manual or manufacturer's web site for a listing of maximum ramp rates over the temperature ranges you plan to operate in. Many newer GC instruments have faster ramp rate abilities due to decreased oven volume or 240V power connections. On older GCs, decreasing the internal oven volume through the use of an insert is an inexpensive and simple way to increase ramping ability.

Because Fast GC produces rapid and narrow peaks, the detector must be able to obtain sufficient data points per peak to ensure proper peak quantitation. Most new detectors are able to work with Fast GC.

We already discussed that the more Principles that are applied, the greater the benefit. However, the instrumentation being used may prohibit applying all of the Principles. An example of this is when working with GC-MS. Some older MS instrumentation may not work properly with hydrogen as a carrier gas. To find out whether your MS is compatible with hydrogen carrier gas, check your instrument manual or manufacturer's web site. If it is not, you will not be able to apply this Principle.

Now for the fun stuff! In this section we will put it all together, taking an application using conventional GC and observing the chromatographic changes while applying the Principles of Fast GC.

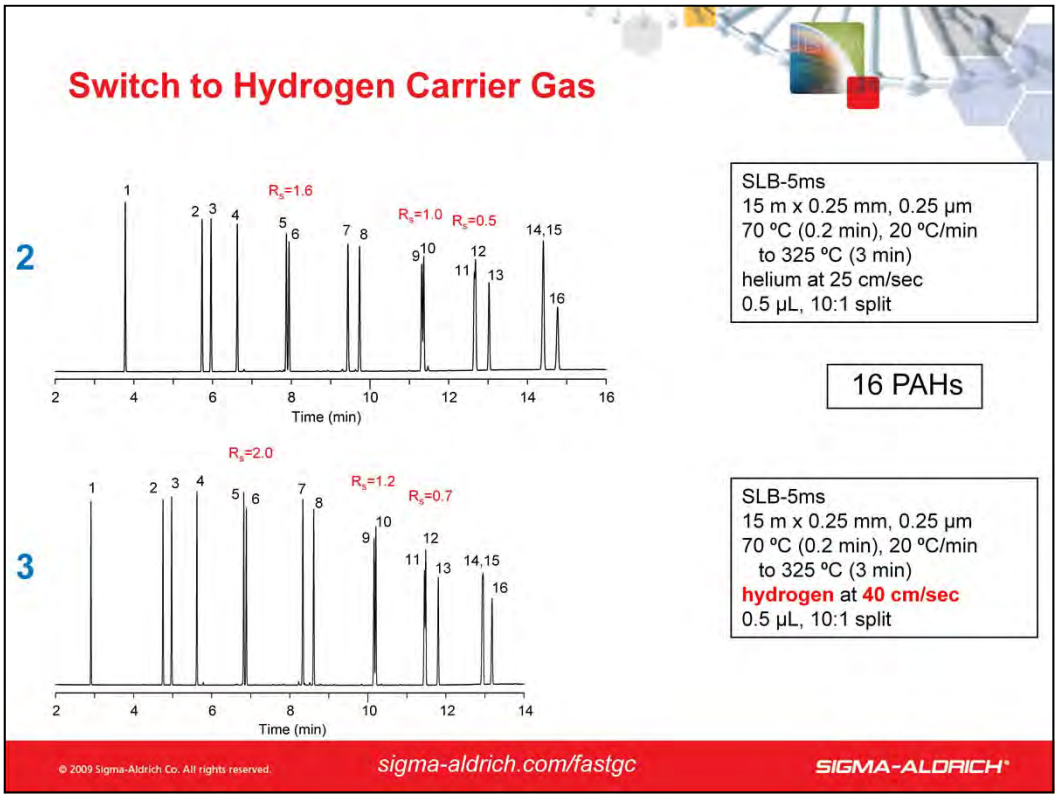


We start with Chromatogram 1, a conventional GC analysis of 16 PAH analytes on a 30 m x 0.25 mm I.D. column. The oven temperature ramp rate of 20 ˚ C/min. was used because this is the maximum single rate that can be used over the 70 – 325 ˚ C temperature range. The difficult separations are phenanthrene/anthracene (peaks 5/6), benzo(a)anthracene/chrysene (peaks 9/10), the isomers benzo(b)fluoranthene/benzo(k)fluoranthene (peaks 11/12), and indeno(1,2,3-cd)pyrene/dibenzo(a,h)anthracene (peaks 14/15). Resolution values for the first two pairs are 1.7 and 1.1, which is borderline acceptable. These should be baseline resolved (value of 1.5 or greater). Resolution for the isomer pair is 0.6, which is generally acceptable. Peaks 14/15 show no separation. Analysis of these analytes is usually by GC-MS where they can be resolved by mass. To achieve better resolutions, a lower initial oven temperature is required, extending the analysis time even longer than the ~19 minutes shown.

Chromatogram 2 shows the same application with a shorter column (15 m instead of 30 m). As expected, analysis time decreases (~19 minutes to ~15 minutes), and resolution values are lower. This is a shorter run, but the resolution is unacceptable.

Conditions (other than those on the slide):
inj.: 250 ˚ C
det.: FID, 325 ˚ C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

Peak IDs:
1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



Chromatogram 2 is where we ended on the previous slide.

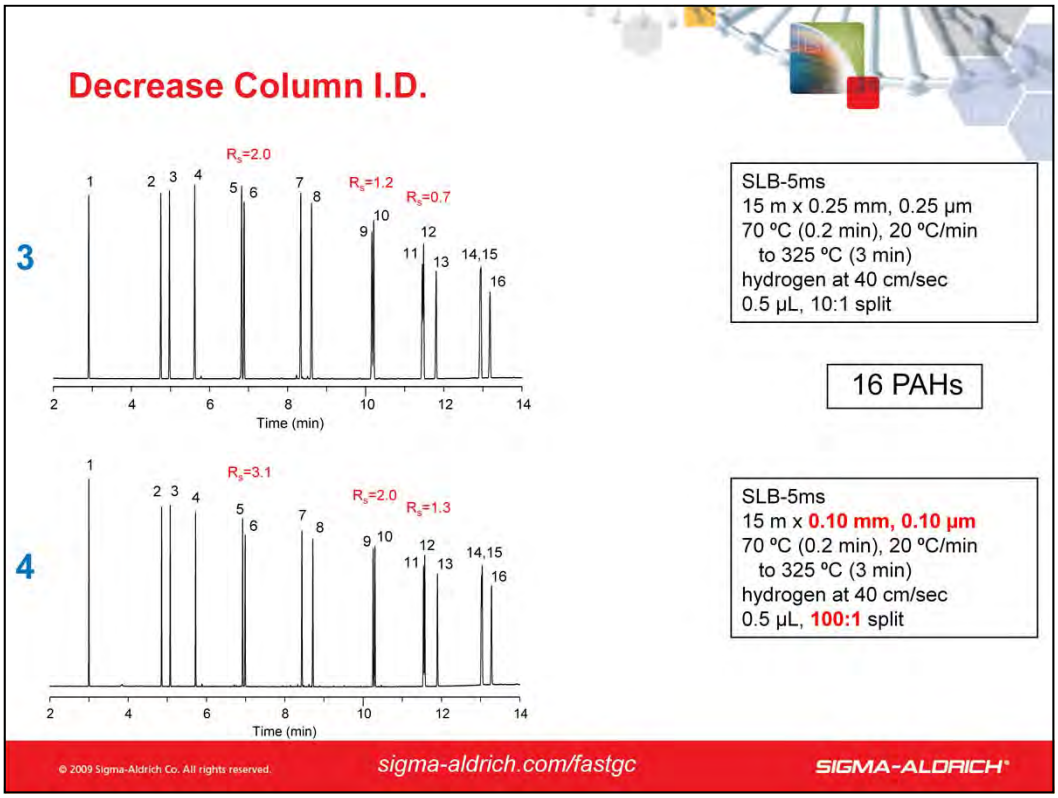
Chromatogram 3 shows what happens when we change the carrier gas (hydrogen at 40 cm/sec instead of helium at 25 cm/sec.. As expected, analysis time is even shorter (~13.5 minutes compared to ~15 minutes). Now look at the resolution values (2.0, 1.2, and 0.7). Why did they get better? Hydrogen at its optimal linear velocity for a 0.25 mm I.D. column (u_{opt} = 40 cm/sec) has a lower effective plate height (H_{eff}) than helium at its optimal linear velocity for a 0.25 mm I.D. column (u_{opt} = 25 cm/sec).

Conditions (other than those on the slide):

inj.: 250 ° C
det.: FID, 325 ° C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

Peak IDs:

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



Chromatogram 3 is where we ended on the previous slide.

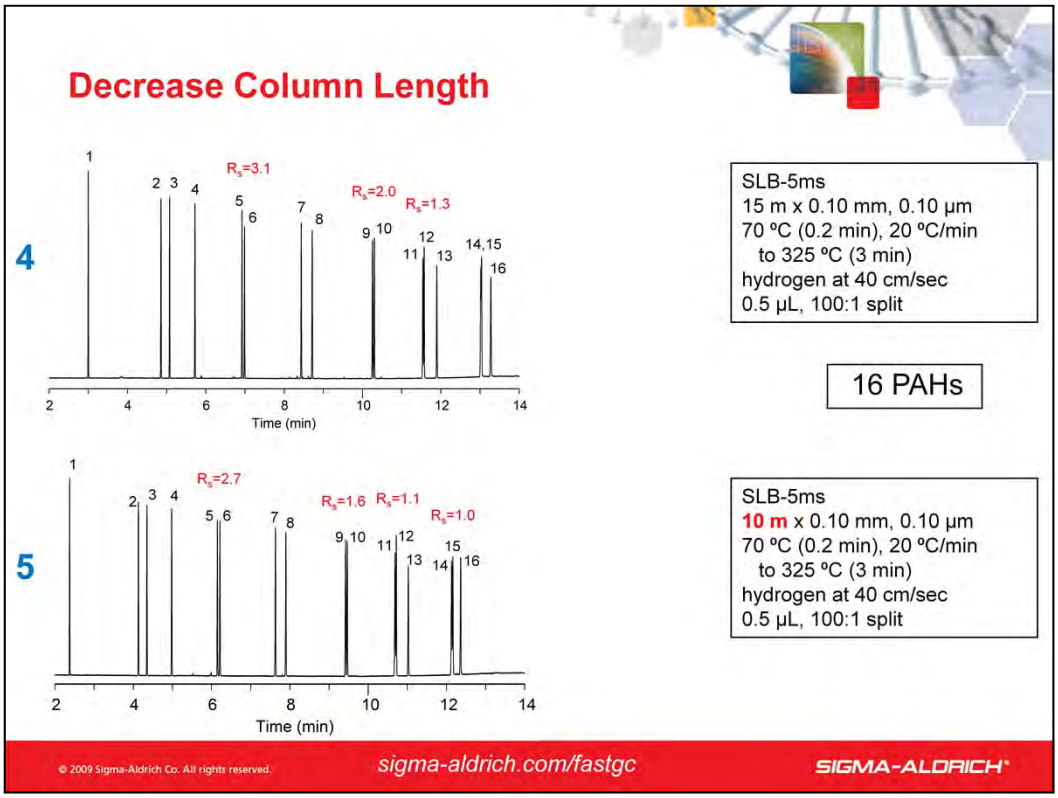
We discussed earlier that decreasing column I.D. was a way to decrease plate height (H), which increases efficiency (N) and subsequently resolution (R_s). We're scientists, so let's experiment and see what happens! Chromatogram 4 shows the same application on smaller I.D. column (0.10 mm I.D. instead of 0.25 mm I.D.). The film thickness was also lowered from 0.25 µm to 0.10 µm to keep the same ratio of stationary phase film to column cross-sectional area. To minimize the risk of column overload, we increased the split ratio from 10:1 to 100:1. Observe that resolution increased as we theorized (3.1, 2.0 and 1.3 for the first three pairs).

Conditions (other than those on the slide):

inj.: 250 ° C
det.: FID, 325 ° C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

Peak IDs:

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene

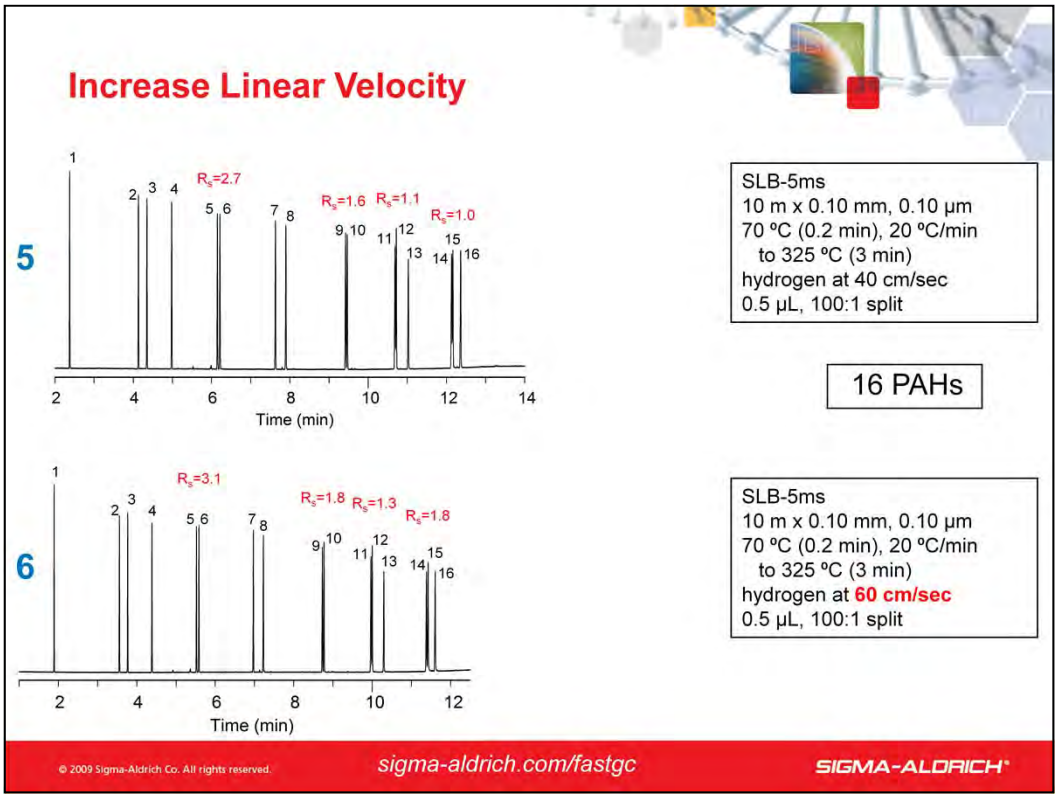


Chromatogram 4 is where we ended on the previous slide.

We decreased column length again, this time from 15 m to 10 m, resulting in Chromatogram 5. As expected, analysis time decreases (~13.5 minutes to ~12.5 minutes), and resolution values are lower (2.7, 1.6, and 1.1). Note that resolution of the fourth pair actually increased (now at 1.0). How is this possible? Because this pair now elutes on the temperature ramp and not the isothermal portion of the run, resulting in sharper peak shapes. Sharper peak shapes is another way to increase resolution.

Conditions (other than those on the slide):
inj.: 250 ° C
det.: FID, 325 ° C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

Peak IDs:
1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



Chromatogram 5 is where we ended on the previous slide.

Chromatogram 6 results after the linear velocity is increased from 40 cm/sec to 60 cm/sec. As expected, analysis time did decrease (from ~12.5 min. to ~11.8 min.). But, why did resolution increase (3.1, 1.8, 1.3, and 1.8)? Was this expected? Remember the Golay plots we looked at during the theoretical discussion about optimal linear velocity? We stated that as column I.D. decreases, the plots tend to move more to the right, and that while hydrogen has an optimal linear velocity (u_{opt}) of 40 cm/sec on a 0.25 mm I.D. column, it has an optimal linear velocity (u_{opt}) of 65 cm/sec on a 0.10 mm I.D. column.

Conditions (other than those on the slide):
inj.: 250 ° C
det.: FID, 325 ° C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

Peak IDs:
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2. Acenaphthylene
3. Acenaphthene
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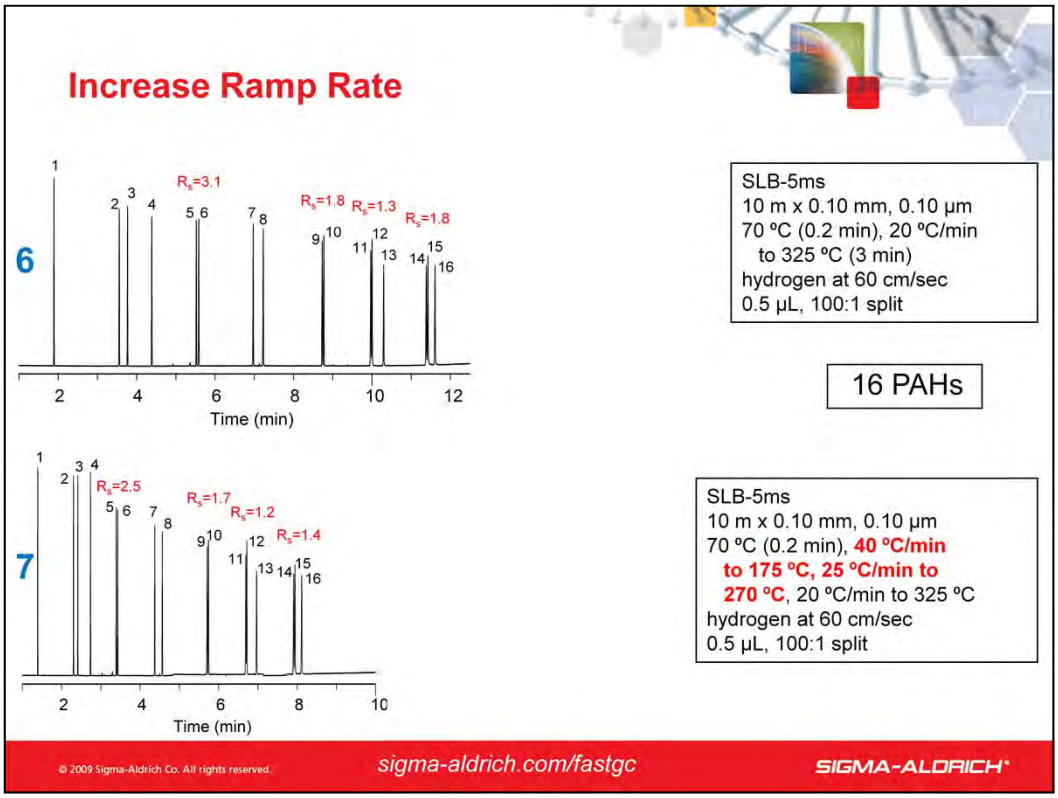
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



Chromatogram 6 is where we ended on the previous slide.

We haven't looked at oven temperature ramp rate yet. What if we pushed the oven to the highest ramp rate possible over each temperature range? After consulting our instrument manual and setting our ramp rates accordingly, we achieved Chromatogram 7. Note that 40 ˚ C/min., 25 ˚ C/min. and 20 ˚ C/min. are used over different temperature ranges throughout the run. These are the maximum rates over these ranges, as found in our instrument manual. Look at the decrease in run time (from ~11.8 min. to ~8.2 min.). While resolution did decrease (2.5, 1.7, 1.2, and 1.4), all values are still acceptable. Who expected the decrease in resolution to be greater? We discussed a few slides ago that sharper peak shapes and better resolution occur if a pair elutes on the temperature ramp and not the isothermal portion of the run. Another way to obtain sharper peak shapes is with a steeper temperature ramp. So, even though the faster temperature ramp will create a decrease in resolution, its effect is minimized due to the sharper peak shapes that are produced.

Conditions (other than those on the slide):
inj.: 250 ˚ C
det.: FID, 325 ˚ C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

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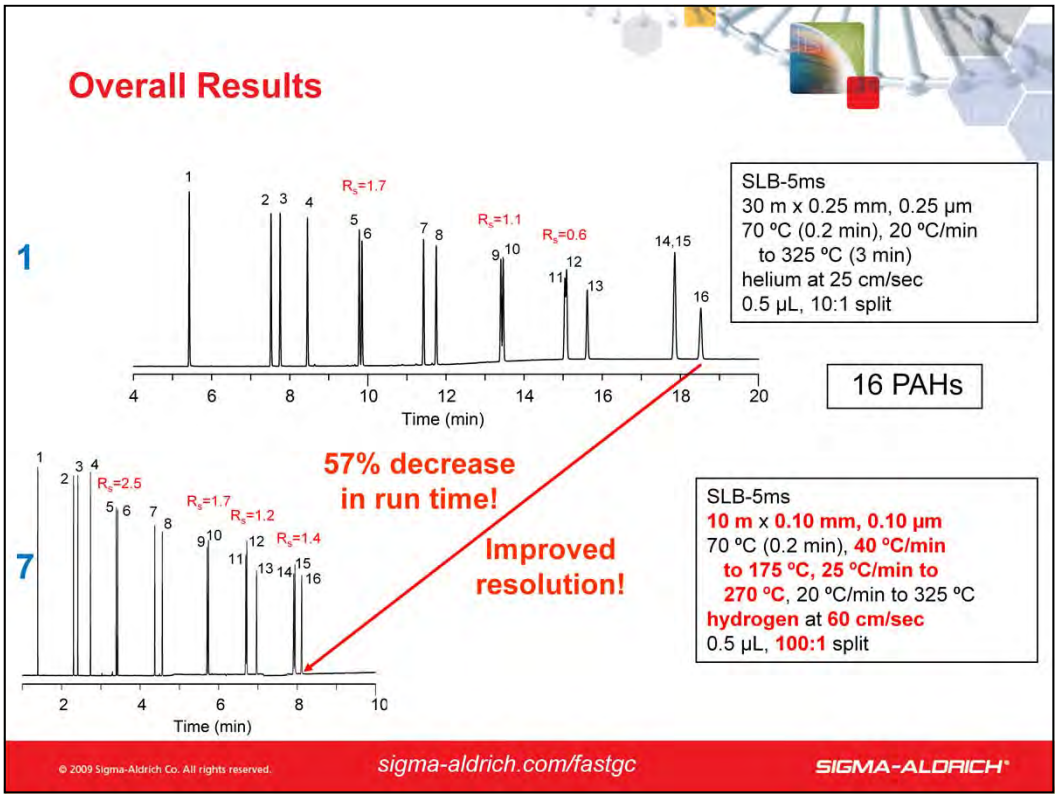
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



Let's stop and have a look at the overall results. Chromatogram 1 is where we started, a conventional GC analysis that may be considered acceptable in many laboratories. Resolution of the first two pairs are borderline acceptable (values of 1.7 and 1.1). Resolution of the isomer pair is generally considered good if the valley is half the height of the taller peak (here we have a value of 0.6). The last pair is typically measured by GC-MS because the MS can resolve by mass.

Chromatogram 7 is where we ended on the previous slide. Compared to Chromatogram 1, it is a very short run (57% decrease in run time) with improved resolution (2.5, 1.7, 1.2, and 1.4).

Conditions (other than those on the slide):
inj.: 250 ° C
det.: FID, 325 ° C
injection (0.25 mm I.D. columns): 0.5 µL, 10:1 split
injection (0.10 mm I.D. columns): 0.5 µL, 100:1 split
liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner design
sample: 16 PAHs, each at 100 µg/mL in methylene chloride

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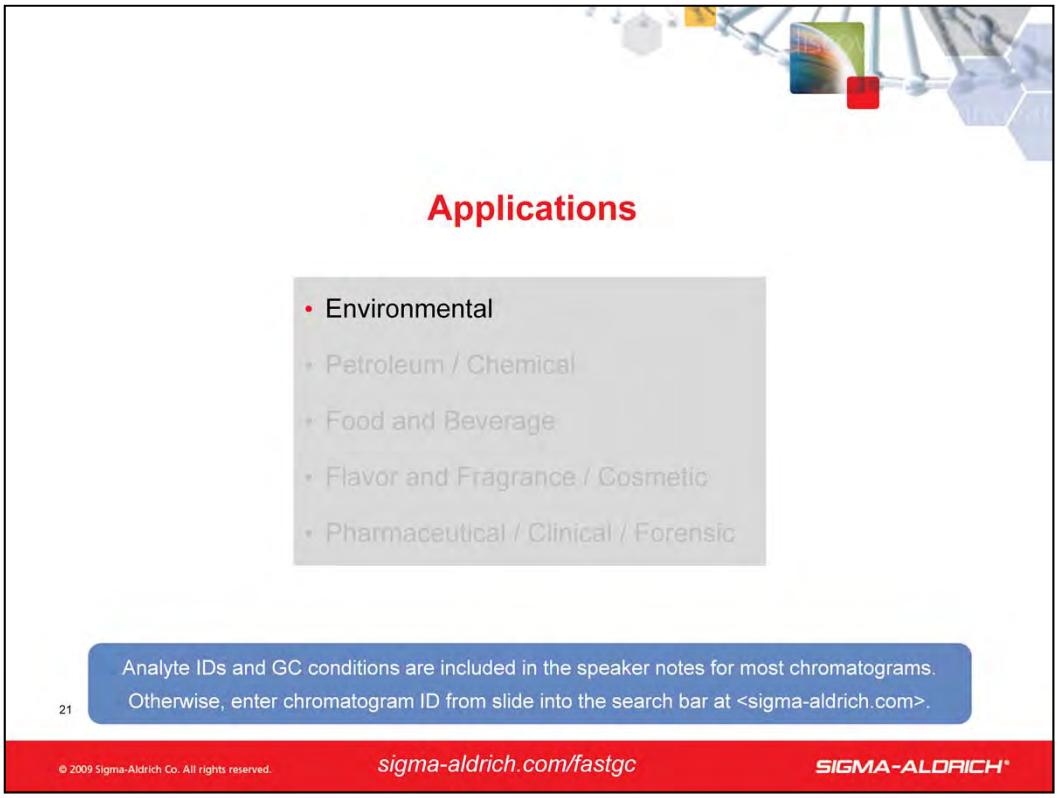
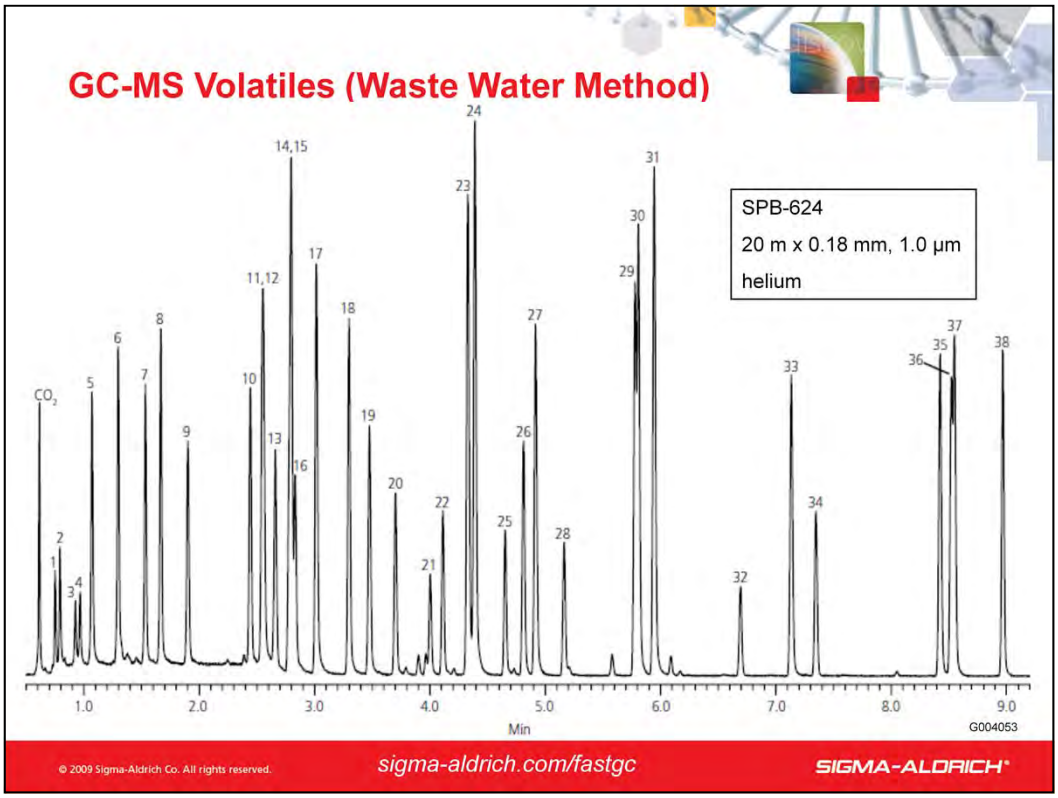
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



This section contains a handful of applications. Remember, the more of the Principles of Fast GC that are applied, the greater the benefit. Due to instrumentation limitations (such as when using GC-MS), some of these chromatograms use helium and not hydrogen as the carrier gas. In these instances, the slide will contain this information.



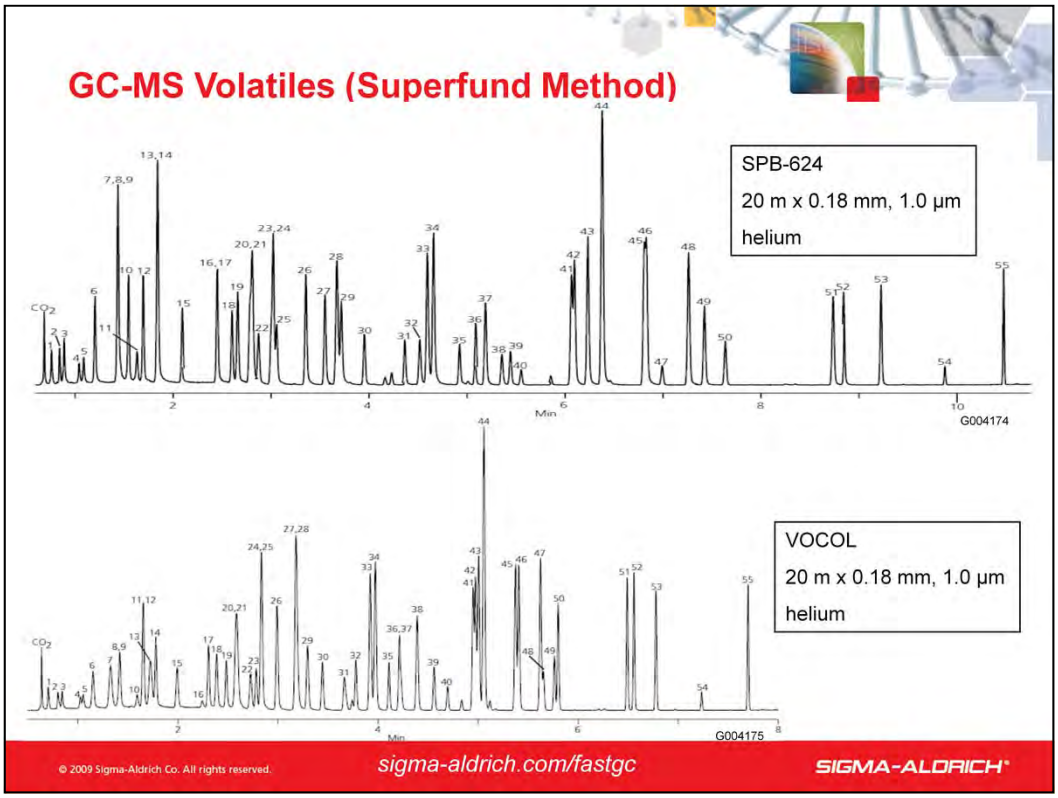
In the environmental industry, the GC-MS analysis of volatiles from drinking water, waste water, ground water, solid waste, and air samples is widely performed. The analyte lists is pretty varied; the 'light' analytes being very volatile gases and the 'heavy' analytes being dichlorobenzenes. This analysis uses helium as the carrier gas, and was used to analyze 38 analytes in ~ 9 min. Note the great peak shapes of the gases (peaks 1-5). The film thickness of 1.0 µm is necessary to retain the volatile gases. Where analytes are not resolved, the MS is used to resolve by mass.

Note:

The raised baseline at the beginning of the run is caused by water. This water is transferred to the GC column during the purge and trap step. When it elutes from the column, it reaches the MS.

Conditions:

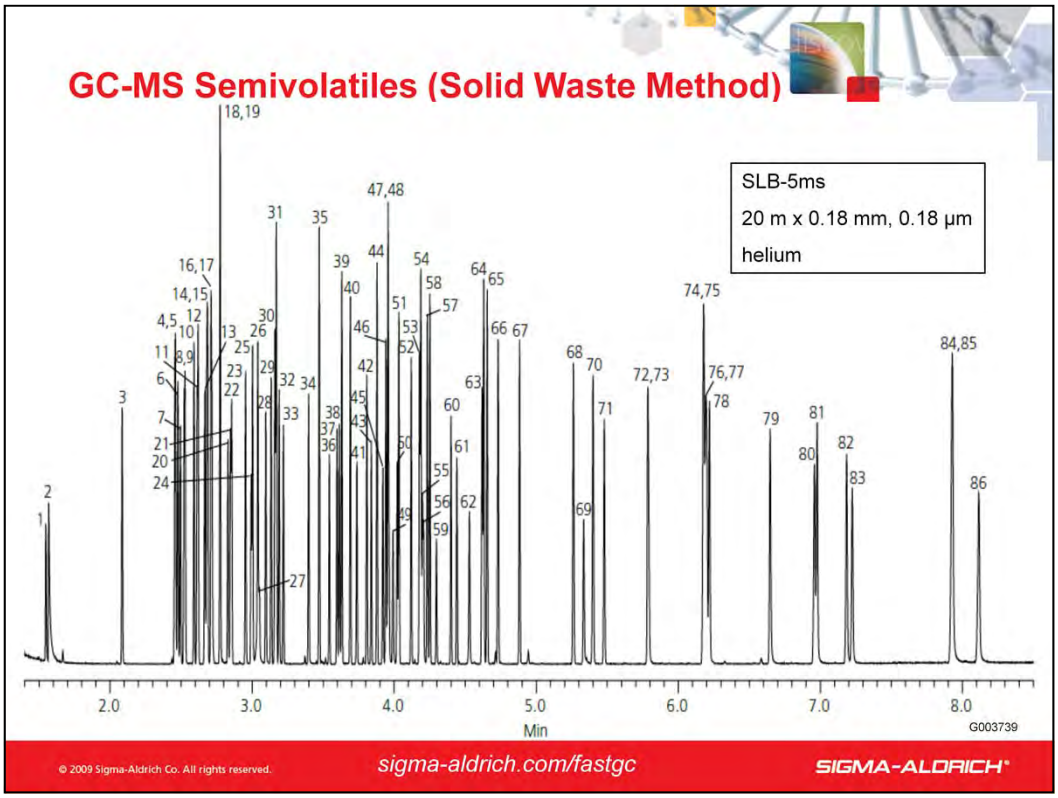
sample/matrix: each analyte at 50 ppb in 5 mL water
purge trap: VOCARB 3000 "K" (24940-U)
purge: 40 mL/min. at 25 ° C for 11 min.
dry purge: 2 min.
desorption pre-heat: 205 ° C
desorption: 150 mL/min. at 210 ° C for 2 min.
bake: 260 ° C for 10 min.
transfer line/valve temp.: 110 ° C
column: SPB-624, 20 m x 0.18 mm I.D., 1.0 µm (28662-U)
oven: 40 ° C (1 min.), 11 ° C/min. to 125 ° C, 35 ° C/min. to 230 ° C (2 min.)
inj.: 150 ° C
MSD interface: 200 ° C
scan range: m/z = 35-400
carrier gas: helium, 1.5 mL/min.
injection: 100:1 split
liner: 0.75 mm I.D. SPME



Another environmental GC-MS volatile application is for samples that originate from Superfund hazardous waste sites. This application requires a different, longer analyte list. Here, two columns with different selectivity are shown. In addition to the Principles of Fast GC, changing column selection may allow a shorter analysis time. The SPB-624 was used to analyze 55 analytes in ~10.8 min., whereas the VOCOL was able to analyze the same 55 analytes in ~7.8 min.

Conditions (top chromatogram):
- sample/matrix: each analyte at 50 ppb in 5 mL water
- purge trap: VOCARB 3000 “K” (24940-U)
- purge: 40 mL/min. at 25 ° C for 11 min.
- dry purge: 2 min.
- desorption pre-heat: 205 ° C
- desorption: 124 mL/min. at 210 ° C for 2 min.
- bake: 260 ° C for 10 min.
- transfer line/valve temp.: 110 ° C
- column: SPB-624, 20 m x 0.18 mm I.D., 1.0 µm (28662-U)
- oven: 40 ° C (1 min.), 11 ° C/min. to 125 ° C, 35 ° C/min. to 230 ° C (2 min.)
- inj.: 150 ° C
- MSD interface: 200 ° C
- scan range: m/z = 35-400
- carrier gas: helium, 1.2 mL/min.
- injection: 100:1 split
- liner: 0.75 mm I.D. SPME

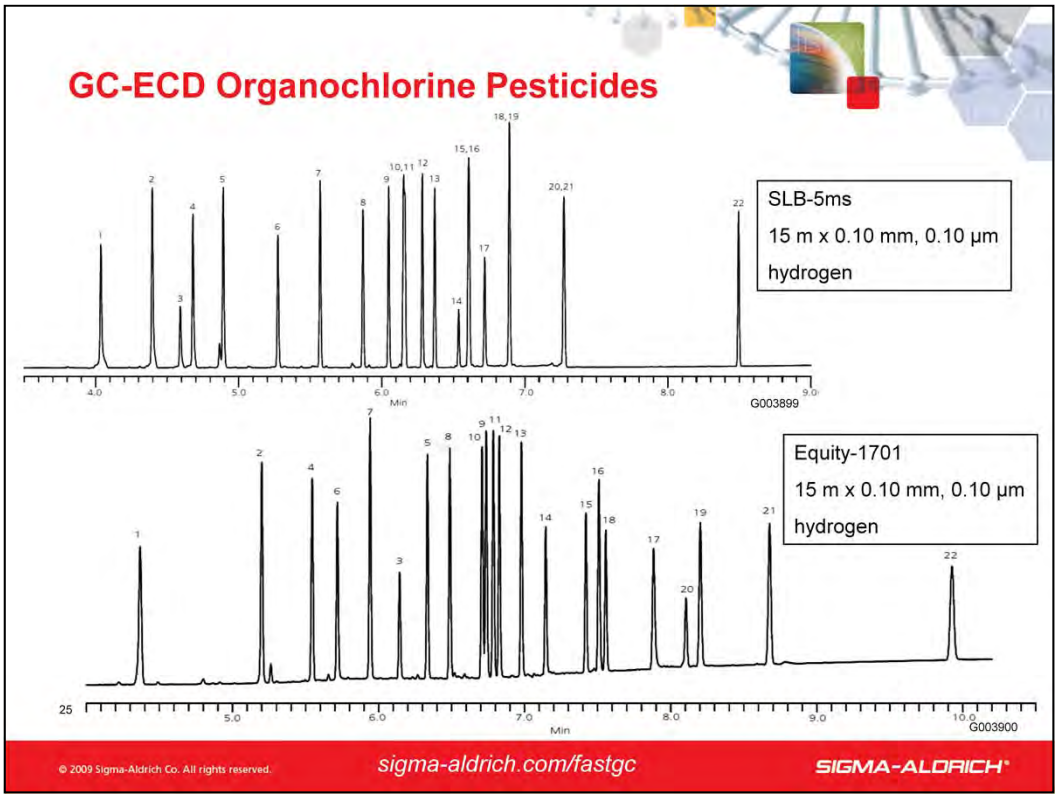
Conditions (bottom chromatogram):
- sample/matrix: each analyte at 50 ppb in 5 mL water
- purge trap: VOCARB 3000 “K” (24940-U)
- purge: 40 mL/min. at 25 ° C for 11 min.
- dry purge: 2 min.
- desorption pre-heat: 205 ° C
- desorption: 150 mL/min. at 210 ° C for 2 min.
- bake: 260 ° C for 10 min.
- transfer line/valve temp.: 110 ° C
- column: VOCOL, 20 m x 0.18 mm I.D., 1.0 µm (28463-U)
- oven: 40 ° C (0.8 min.), 19 ° C/min. to 125 ° C, 32 ° C/min. to 220 ° C (1 min.)
- inj.: 150 ° C
- MSD interface: 200 ° C
- scan range: m/z = 35-400
- carrier gas: helium, 1.4 mL/min.
- injection: 100:1 split
- liner: 0.75 mm I.D. SPME



Another common GC-MS application in the environmental industry is the analysis of semivolatile analytes, ranging from N-nitrosodimethylamine and pyridine to the multi-ringed PAHs, such as benzo(g,h,i)perylene. Even with helium as the carrier gas, the other Principles of Fast GC were used to produce a chromatogram with 86 analytes in ~8.2 min. Very impressive!

Note:
The decreasing baseline at the beginning of the chromatogram is the tail end of the methylene chloride solvent peak.

Conditions:
column: SLB-5ms, 20 m x 0.18 mm I.D., 0.18 µm (28564-U)
oven: 40 ° C (0.7 min.), 55 ° C/min. to 240 ° C, 28 ° C/min. to 330 ° C (2 min.)
inj.: 250 ° C
MSD interface: 330 ° C
scan range: m/z 40-450
carrier gas: helium, 40 cm/sec, constant
injection: 0.5 µL, 10:1 split
liner: 2 mm I.D., fast FocusLiner™ inlet liner with taper (2879501-U)
sample: 80 component semivolatile standard at 50 ppm plus 6 internal standards (at 40 ppm) in methylene chloride

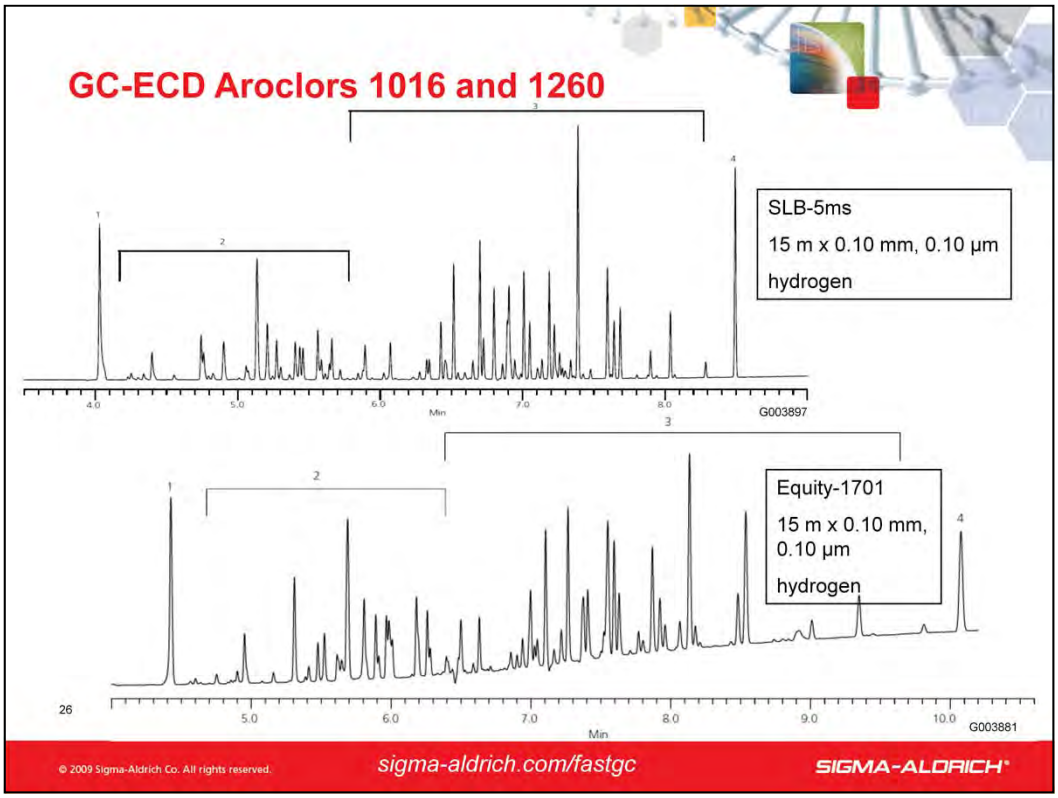


The analysis of 20 chlorinated pesticides and 2 surrogate compounds by GC-ECD is another application routinely performed in the environmental industry. It is common to analyze each sample extract on two columns with differing selectivity for confirmation of identifications. Here are Fast GC chromatograms showing this application on two different columns, an SLB-5ms and an Equity-1701. Run times of ~8.5 min. and 10 min. were achieved.

Conditions (top chromatogram):
column: SLB-5ms, 15 m x 0.10 mm I.D., 0.10 µm (28466-U)
oven: 100 ° C, 25 ° C/min. to 325 ° C
inj.: 225 ° C
det.: ECD, 300 ° C
carrier gas: hydrogen, 40 cm/sec constant
injection: 2 µL, splitless (0.75 min.)
liner: 4 mm I.D., single taper
sample: 50 ppb of a 22 component chlorinated pesticide standard in n-hexane

Conditions (bottom chromatogram):
column: Equity-1701, 15 m x 0.10 mm I.D., 0.10 µm (28343-U)
oven: 100 ° C, 25 ° C/min. to 280 ° C
inj.: 225 ° C
det.: ECD, 300 ° C
carrier gas: hydrogen, 40 cm/sec constant
injection: 2 µL, splitless (0.75 min.)
liner: 4 mm I.D., single taper
sample: 50 ppb of a 22 component chlorinated pesticide standard in n-hexane

Peak IDs:
1. Tetrachloro-m-xylene (surr.)
2. α-BHC
3. β-BHC
4. γ-BHC
5. δ-BHC
6. Heptachlor
7. Aldrin
8. Heptachlor epoxide
9. γ-Chlordane
10. Endosulfan I
11. α-Chlordane
12. 4,4'-DDE
13. Dieldrin
14. Endrin
15. 4,4'-DDD
16. Endosulfan II
17. Endrin aldehyde
18. 4,4'-DDT
19. Endosulfan sulfate
20. Methoxychlor
21. Endrin ketone
22. Decachlorobiphenyl (surr.)

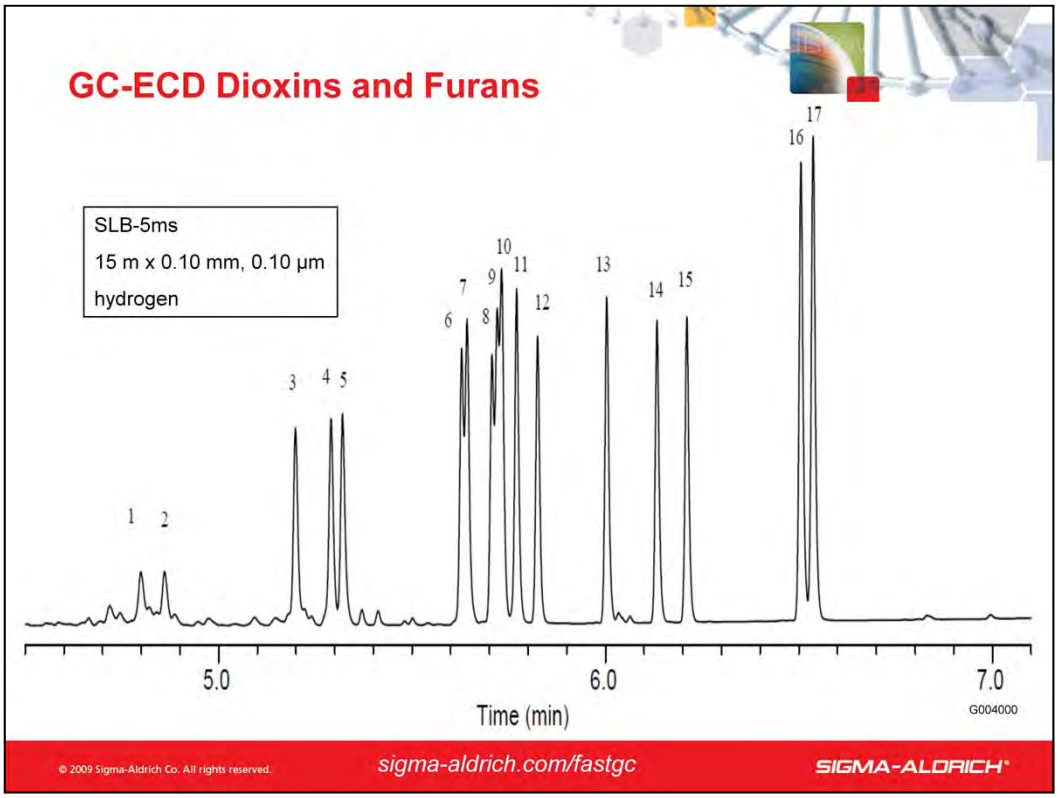


Polychlorinated biphenyls (PCBs) are typically analyzed in the environmental industry using pattern recognition to Aroclor mix standards. Similar to the organochlorine pesticide application, each sample extract is typically analyzed on two columns with differing selectivity for confirmation of identification. Analysis times of ~8.5 min. and ~10.2 min. were achieved on an SLB-5ms and an Equity-1701 column by applying the Principles of Fast GC.

Conditions (top chromatogram):
column: SLB-5ms, 15 m x 0.10 mm I.D., 0.10 µm (28466-U)
oven: 80 ° C (0.5 min.), 50 ° C/min. to 200 ° C, 35 ° C/min. to 360 ° C (2 min.)
inj.: 225 ° C
det.: ECD, 360 ° C
carrier gas: hydrogen, 40 cm/sec constant
injection: 2 µL, splitless (0.75 min.)
liner: 4 mm I.D., single taper
sample: Aroclor standard mix 1 (46846-U) diluted to 500 ppb / 50 ppb (Aroclors / surrogates) in n-hexane

Conditions (bottom chromatogram):
column: Equity-1701, 15 m x 0.10 mm I.D., 0.10 µm (28343-U)
oven: 90 ° C, 35 ° C/min. to 280 ° C (3 min.)
inj.: 250 ° C
det.: ECD, 280 ° C
carrier gas: hydrogen, 50 cm/sec constant
injection: 2 µL, splitless (0.75 min.)
liner: 4 mm I.D., single taper
sample: Aroclor standard mix 1 (46846-U) diluted to 200 ppb / 20 ppb (Aroclors / surrogates) in n-hexane

Peak IDs:
1. Tetrachloro-m-xylene (surr.)
2. Aroclor 1016
3. Aroclor 1260
4. Decachlorobiphenyl (surr.)



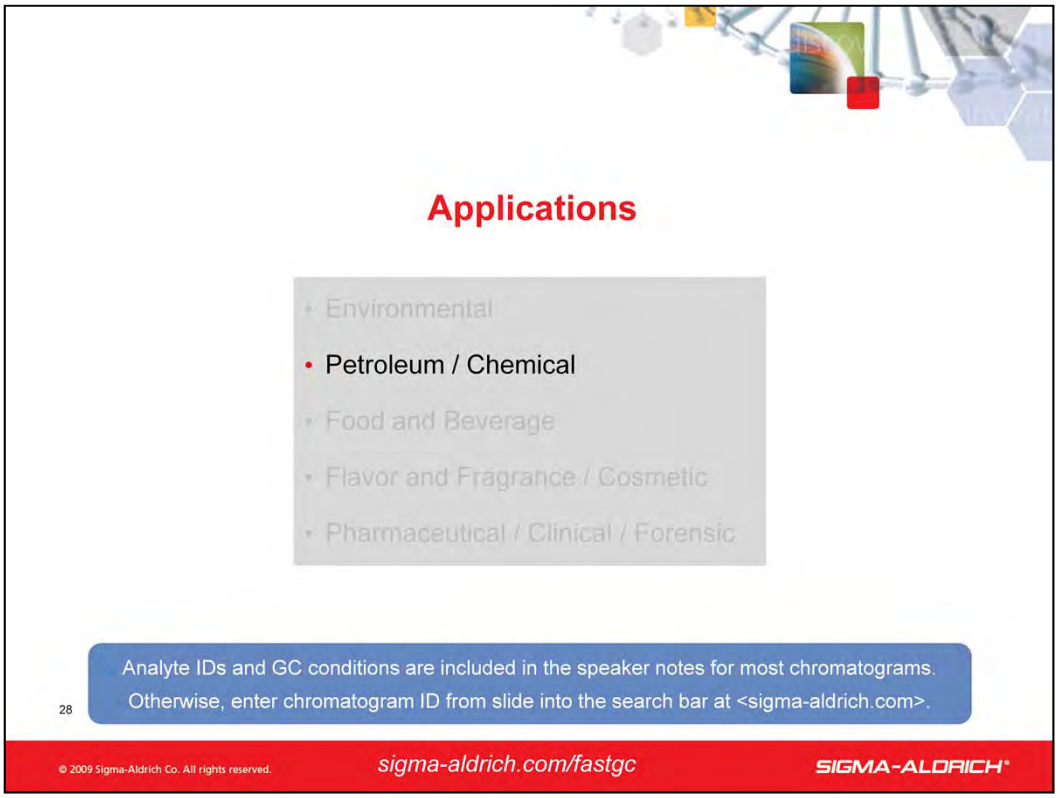
GC-ECD is often used in the environmental industry to screen for the presence of dioxins and furans that have chlorine substitution in the 2, 3, 7, and 8 positions. If detected, the sample extract is then analyzed using high resolution mass spectrometry to verify identifications and perform quantitative measurements. Here, Fast GC was used to shorted the time required for the screening portion of the application to ~6.5 min.

Conditions:

column: SLB-5ms, 15 m x 0.10 mm I.D., 0.10 µm (28466-U)
oven: 150 ° C (1 min.), 35 ° C/min. to 340 ° C (1 min.)
inj.: 250 ° C
det.: ECD, 340 ° C
carrier gas: hydrogen, 45 cm/sec, constant
injection: 1 µL, splitless (1 min.)
liner: 4 mm I.D., single taper
sample: 17 component 2,3,7,8-substituted dioxin standard, 100-500 ppb in n-nonane

Peak IDs:

1. 2,3,7,8-TCDF, 100 ppb
2. 2,3,7,8-TCDD, 100 ppb
3. 1,2,3,7,8-PCDF, 250 ppb
4. 2,3,4,7,8-PCDF, 250 ppb
5. 1,2,3,7,8-PCDD, 250 ppb
6. 1,2,3,4,7,8-HxCDF, 500 ppb
7. 1,2,3,6,7,8-HxCDF, 500 ppb
8. 2,3,4,6,7,8-HxCDF, 250 ppb
9. 1,2,3,4,7,8-HxCDD, 500 ppb
10. 1,2,3,6,7,8-HxCDD, 500 ppb
11. 1,2,3,7,8,9-HxCDD, 250 ppb
12. 1,2,3,7,8,9-HxCDF, 250 ppb
13. 1,2,3,4,6,7,8-HpCDF, 250 ppb
14. 1,2,3,4,6,7,8-HpCDD, 250 ppb
15. 1,2,3,4,7,8,9-HpCDF, 250 ppb
16. OCDD, 500 ppb
17. OCDF, 500 ppb



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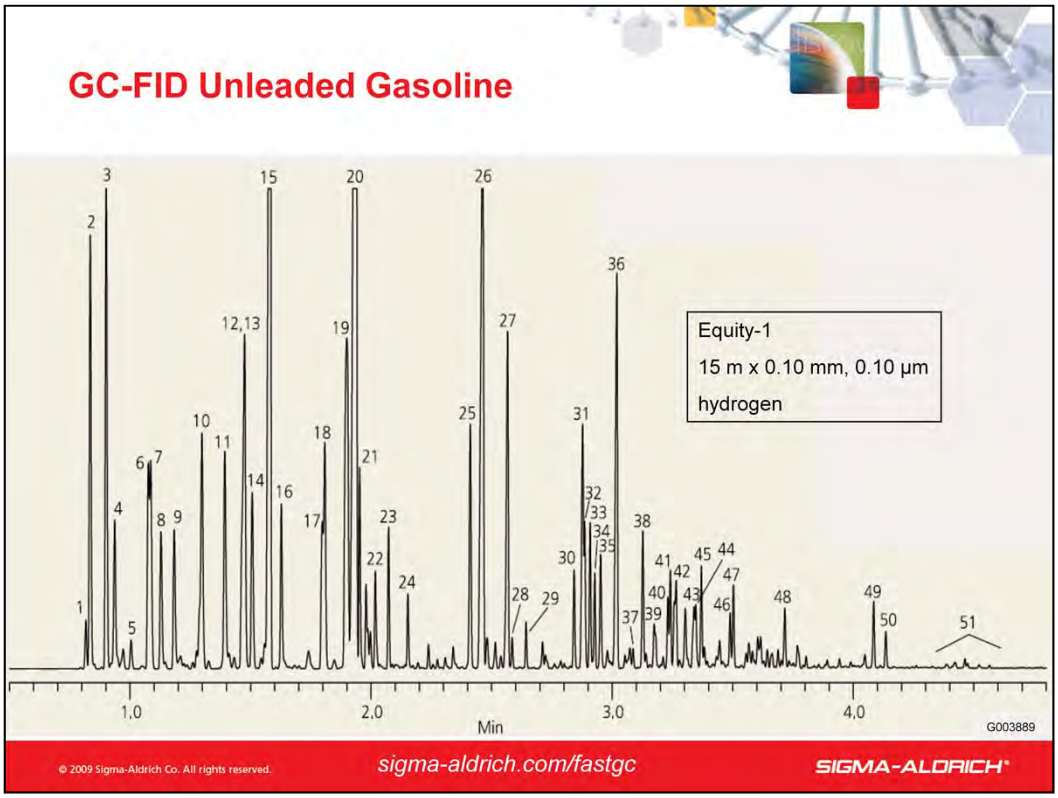
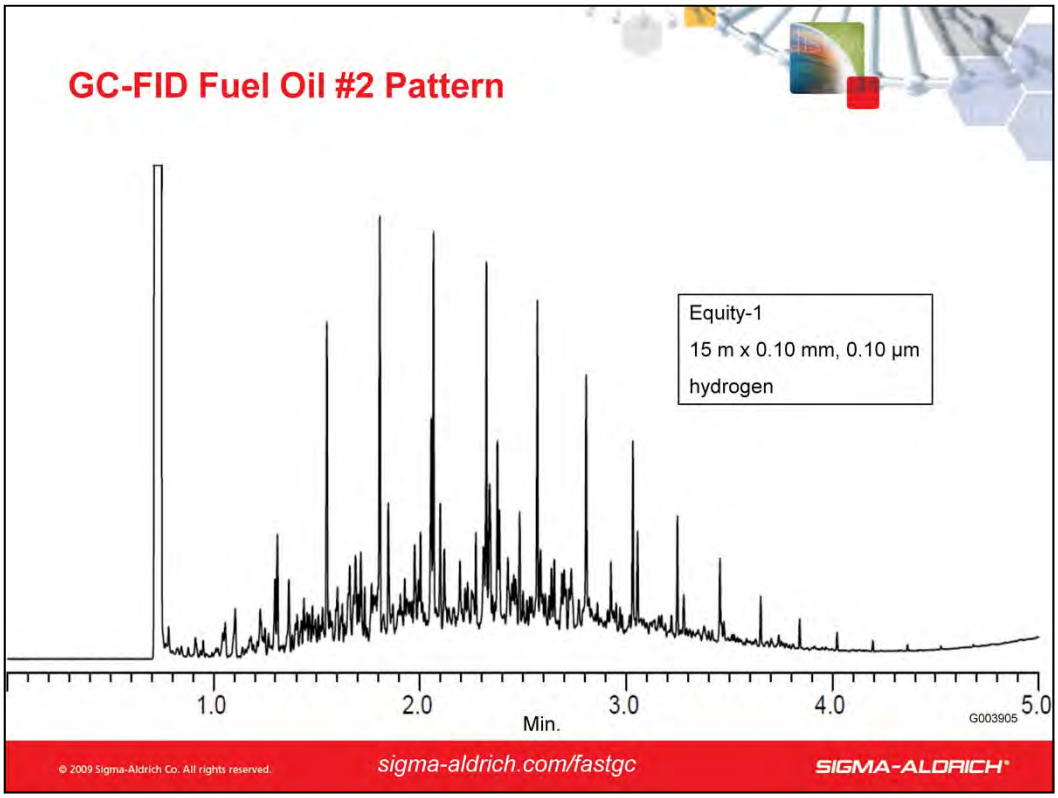


The petroleum industry must deal with very complex fuel samples, that may contain hundreds of individual compounds. Here is the Fast GC analysis of an unleaded gasoline sample with the major peaks identified. An Equity-1 column was used to obtain an analysis time of ~5 min.

Conditions:
column: Equity-1, 15 m x 0.10 mm I.D., 0.10 µm (28039-U)
oven: 40 °C (1 min.), 45 °C/min. to 150 °C (2 min.)
inj.: 175 °C
det.: FID, 175 °C
carrier gas: hydrogen, 45 cm/sec constant
injection: 0.1 uL, 300:1 split
liner: 2 mm I.D., straight
sample: Unleaded gasoline (refinery standard), neat

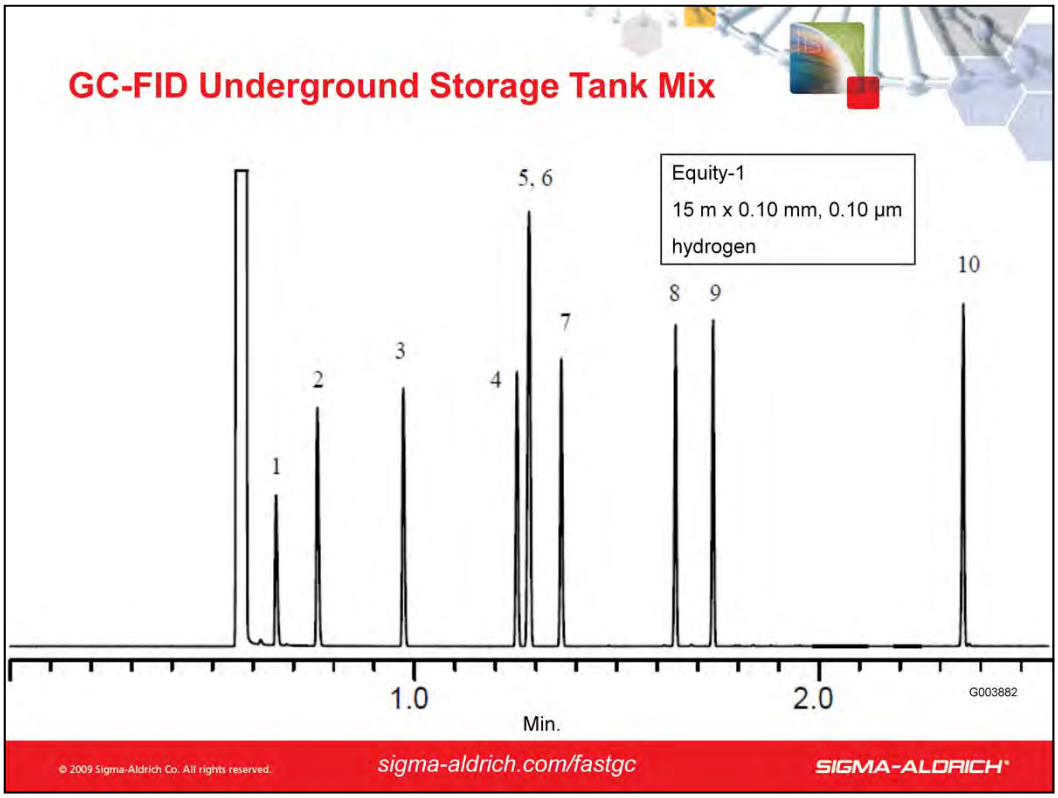
Peak IDs:
1. Isobutane
2. Butane
3. Isopentane
4. Pentane
5. 2,2-Dimethylbutane
6. 2,3-Dimethylbutane
7. 2-Methylpentane
8. 3-Methylpentane
9. Hexane
10. 2,4-Dimethylpentane
11. Benzene
12. 2-Methylhexane
13. 2,3-Dimethylpentane
14. 3-Methylhexane
15. Isooctane
16. Heptane
17. 2,5-Dimethylhexane
18. 2,4-Dimethylhexane
19. 2,3,4-Trimethylpentane
20. Toluene
21. 2,3-Dimethylhexane
22. 2-Methylheptane
23. 3-Methylheptane
24. Octane
25. Ethylbenzene
26. m-/p-Xylene
27. o-Xylene
28. Nonane
29. iso-Propylbenzene
30. Propylbenzene
31. 1-Methyl-3-ethylbenzene
32. 1-Methyl-4-ethylbenzene
33. 1,3,5-Trimethylbenzene
34. 3,3,4-Trimethylheptane
35. 1-Methyl-2-ethylbenzene
36. 1,2,4-Trimethylbenzene
37. iso-Butylbenzene
38. sec-Butylbenzene
39. 1,2,3-Trimethylbenzene
40. Indane
41. 1,3-Diethylbenzene
42. N-Butylbenzene
43. 1,4-Dimethyl-2-ethylbenzene
44. 1,3-Dimethyl-4-ethylbenzene
45. 1,2-Dimethyl-4-ethylbenzene
46. 1,2,4,5-Tetramethylbenzene
47. 1,2,3,5-Tetramethylbenzene
48. Naphthalene
49. 2-Methylnaphthalene
50. 1-Methylnaphthalene
51. Dimethylnaphthalenes



Because fuel samples are so complex, especially the more unrefined they are, the petroleum industry may evaluate product based on pattern recognition rather that the identification and quantitation of individual analytes. Here, the Principles of Fast GC were applied to the analysis of a fuel oil #2 sample, with analysis completed in ~4.5 min. on an Equity-1 column.

Conditions:

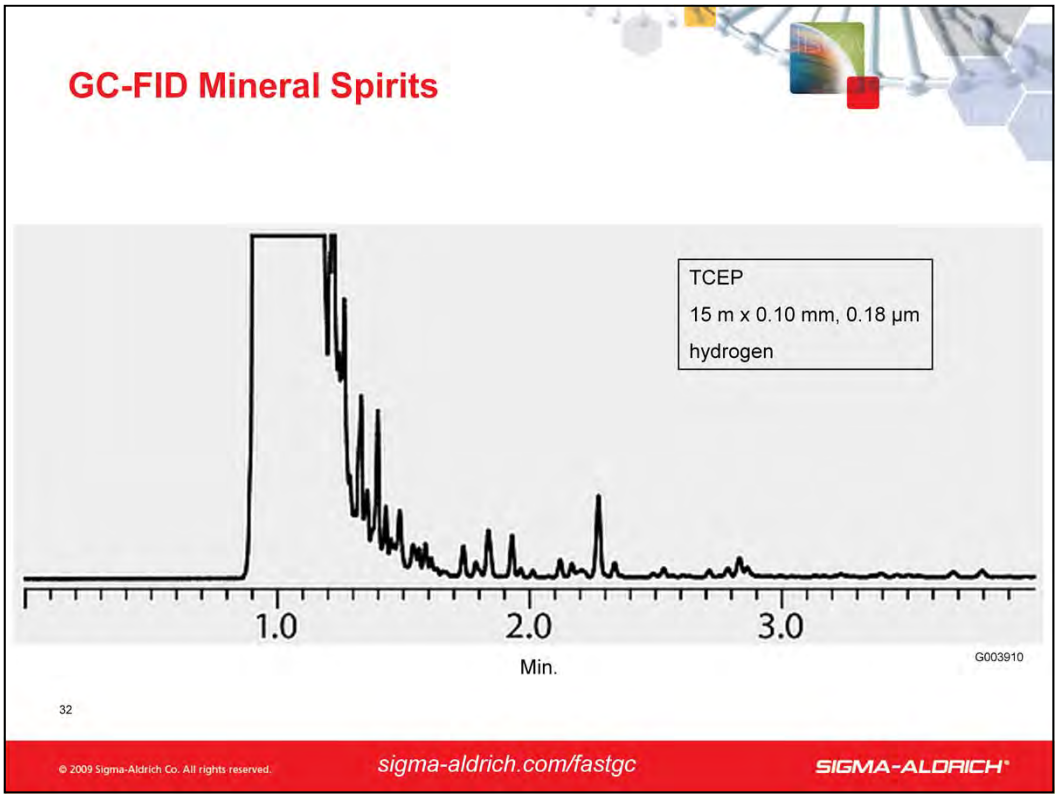
column: Equity-1, 15 m x 0.10 mm I.D., 0.10 µm (28039-U)
oven: 80 ° C, 50 ° C/min. to 325 ° C
inj.: 250 ° C
det.: FID, 350 ° C
carrier gas: hydrogen, 45 cm/sec constant
injection: 0.3 µL, 100:1 split, 0.02 min. pre-injection dwell time
liner: 2 mm I.D., straight
sample: No.2 Fuel Oil standard, 20 mg/mL in methanol (47515-U)



This chromatogram shows an application performed by both the petroleum and environmental industries. Purity of product in an underground storage tank is of interest to the petroleum industry. These analytes may serve as indicator analytes. When an underground storage tank leaks, the contaminated soil is tracked by the environmental industry by looking for these same indicator analytes. The soil that is deemed to be contaminated is remediated. This application was accomplished in ~2.5 min. using an Equity-1 column and the Principles of Fast GC.

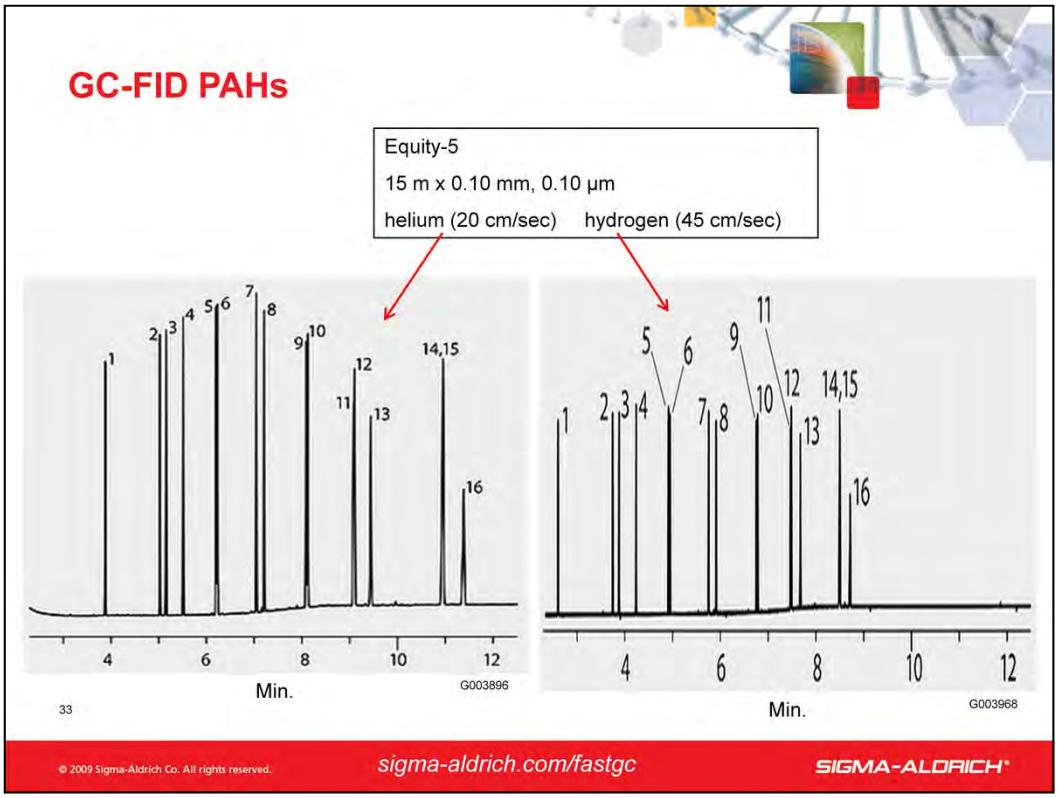
Conditions:
column: Equity-1, 15 m x 0.10 mm I.D., 0.10 µm (28039-U)
oven: 75 ° C, 40 ° C/min. to 110 ° C, 7.5 ° C/min. to 190 ° C
inj.: 200 ° C
det.: FID, 250 ° C
carrier gas: hydrogen, 57 cm/sec @ 75 ° C
injection: 0.5 µL, 200:1 split
liner: 4 mm I.D., split, cup design
sample: UST Modified GRO Mix, each analyte at 1000 ppm in methanol (48167)

Peak IDs:
1. MTBE
2. Benzene
3. Toluene
4. Ethyl benzene
5. m-Xylene
6. p-Xylene
7. o-Xylene
8. 1,3,5-Trimethylbenzene
9. 1,2,4-Trimethylbenzene
10. Naphthalene



Mineral spirits typically contain 75% aliphatic hydrocarbons (most >C10), and 25% aromatic hydrocarbons. This product is used as a paint thinner and a degreaser, plus as a solvent in a wide range of consumer products, including paint. The chromatogram shown on this slide was generated using a TCEP column. This highly polar column results in the elution of the aliphatic portion prior to the aromatic constituents.

Conditions:
column: TCEP, 15 m x 0.10 mm I.D., 0.18 µm (28348-U)
oven: 100 ° C
inj.: 220 ° C
det.: FID, 220 ° C
carrier gas: hydrogen, 43 cm/sec
injection: 0.04 µL, 200:1 split
liner: 4 mm I.D., split, cup design
sample: Low odor mineral spirits (from a local hardware store)



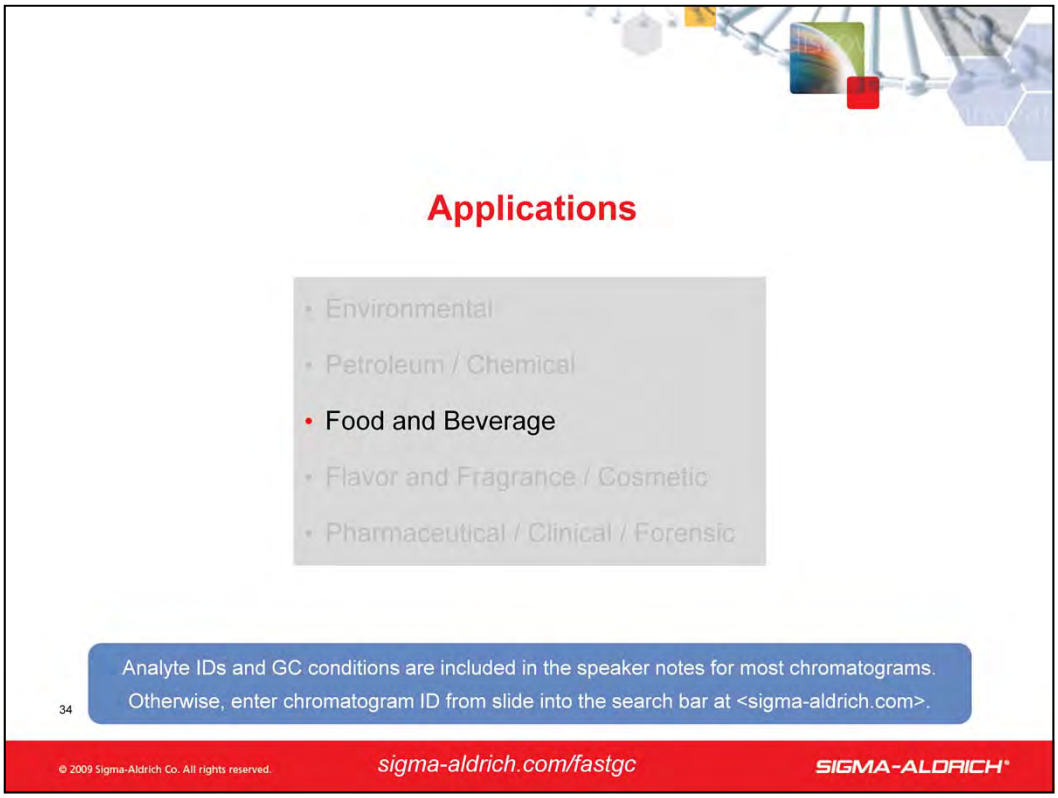
Several polycyclic aromatic hydrocarbons (PAHs) are identified as carcinogens. The PAHs of interest include several isomer sets. These chromatograms of 16 PAHs on the Equity-5 highlight the ability of hydrogen to provide a shorter analysis. A short, narrow column was used for the timely elution of the heavier PAHs.

Conditions:

column: Equity-5, 15 m x 0.10 mm I.D., 0.10 µm (28083-U)
oven: 100 ° C (1 min), 35 ° C/min to 325 ° C (5 min)
inj.: 250 ° C
det.: FID, 350 ° C
carrier gas: helium, 20 cm/sec constant
carrier gas: hydrogen, 45 cm/sec constant
injection: 1 µL, 200:1 split
liner: 4 mm I.D., split, cup design
sample: Each analyte at 200 ppm in methylene chloride

Peak IDs:

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Indeno(1,2,3-cd)pyrene
15. Dibenzo(a,h)anthracene
16. Benzo(g,h,i)perylene



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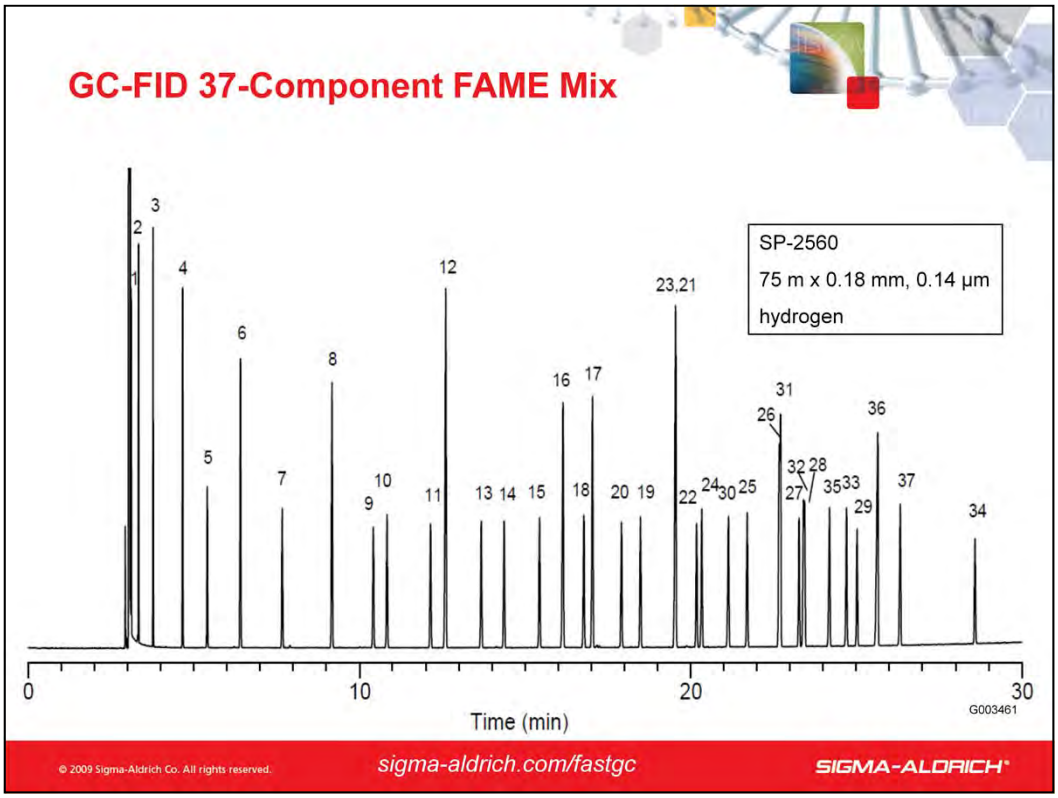
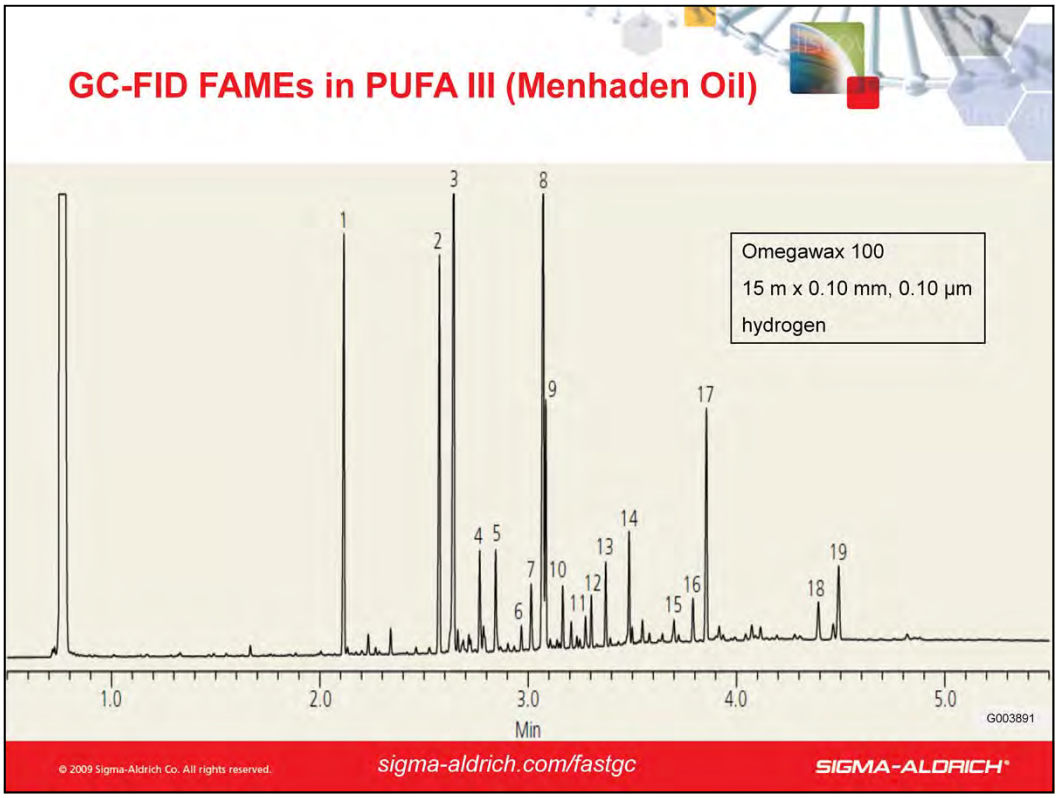


Most people would not consider a 75 m long column to be short. It is in the food & beverage industry for analysts performing fatty acid methyl ester (FAME) applications. The conventional GC column required to perform these separations has a 100 m x 0.25 mm I.D. dimension, and requires over 40 min. to elute the last peak. Here, a 75 m x 0.18 mm I.D. SP-2560 column plus other Fast GC Principles were applied to reduce the analysis time to ~29 min., without any sacrifice in resolution.

Conditions:
column: SP-2560, 75 m x 0.18 mm I.D., 0.14 µm (23348-U)
oven: 140 ° C (5 min.), 4 ° C/min. to 240 ° C (2 min.)
inj.: 250 ° C
det.: FID, 250 ° C
carrier gas: hydrogen, 40 cm/sec @ 175 ° C
injection: 1 µL, 100:1 split
liner: 4 mm I.D, split, cup design
sample: Supelco 37-Component FAME Mix (47885-U), analytes at concentrations indicated in methylene chloride

Peak IDs:
1. Butyric Acid Methyl Ester (C4:0) at 4 wt %
2. Caproic Acid Methyl Ester (C6:0) at 4 wt %
3. Caprylic Acid Methyl Ester (C8:0) at 4 wt %
4. Capric Acid Methyl Ester (C10:0) at 4 wt %
5. Undecanoic Acid Methyl Ester (C11:0) at 2 wt %
6. Lauric Acid Methyl Ester (C12:0) at 4 wt %
7. Tridecanoic Acid Methyl Ester (C13:0) at 2 wt %
8. Myristic Acid Methyl Ester (C14:0) at 4 wt %
9. Myristoleic Acid Methyl Ester (C14:1) at 2 wt %
10. Pentadecanoic Acid Methyl Ester (C15:0) at 2 wt %
11. cis-10-Pentadecenoic Acid Methyl Ester (C15:1) at 2 wt %
12. Palmitic Acid Methyl Ester (C16:0) at 6 wt %
13. Palmitoleic Acid Methyl Ester (C16:1) at 2 wt %
14. Heptadecanoic Acid Methyl Ester (C17:0) at 2 wt %
15. cis-10-Heptadecenoic Acid Methyl Ester (C17:1) at 2 wt %
16. Stearic Acid Methyl Ester (C18:0) at 4 wt %
17. Oleic Acid Methyl Ester (C18:1n9c) at 4 wt %
18. Elaidic Acid Methyl Ester (C18:1n9t) at 2 wt %
19. Linoleic Acid Methyl Ester (C18:2n6c) at 2 wt %
20. Linolelaidic Acid Methyl Ester (C18:2n6t) at 2 wt %
21. γ-Linolenic Acid Methyl Ester (C18:3n6) at 2 wt %
22. α-Linolenic Acid Methyl Ester (C18:3n3) at 2 wt %
23. Arachidic Acid Methyl Ester (C20:0) at 4 wt %
24. cis-11-Eicosenoic Acid Methyl Ester (C20:1n9) at 2 wt %
25. cis-11,14-Eicosadienoic Acid Methyl Ester (C20:2) at 2 wt %
26. cis-8,11,14-Eicosatrienoic Acid Methyl Ester (C20:3n6) at 2 wt %
27. cis-11,14,17-Eicosatrienoic Acid Methyl Ester (C20:3n3) at 2 wt %
28. Arachidonic Acid Methyl Ester (C20:4n6) at 2 wt %
29. cis-5,8,11,14,17-Eicosapentaenoic Acid Methyl Ester (C20:5n3) at 2 wt %
30. Heneicosanoic Acid Methyl Ester (C21:0) at 2 wt %
31. Behenic Acid Methyl Ester (C22:0) at 4 wt %
32. Erucic Acid Methyl Ester (C22:1n9) at 2 wt %
33. cis-13,16-Docosadienoic Acid Methyl Ester (C22:2) at 2 wt %
34. cis-4,7,10,13,16,19-Docosahexaenoic Acid Methyl Ester (C22:6n3) at 2 wt %
35. Tricosanoic Acid Methyl Ester (C23:0) at 2 wt %
36. Lignoceric Acid Methyl Ester (C24:0) at 4 wt %
37. Nervonic Acid Methyl Ester (C24:1n9) at 2 wt %



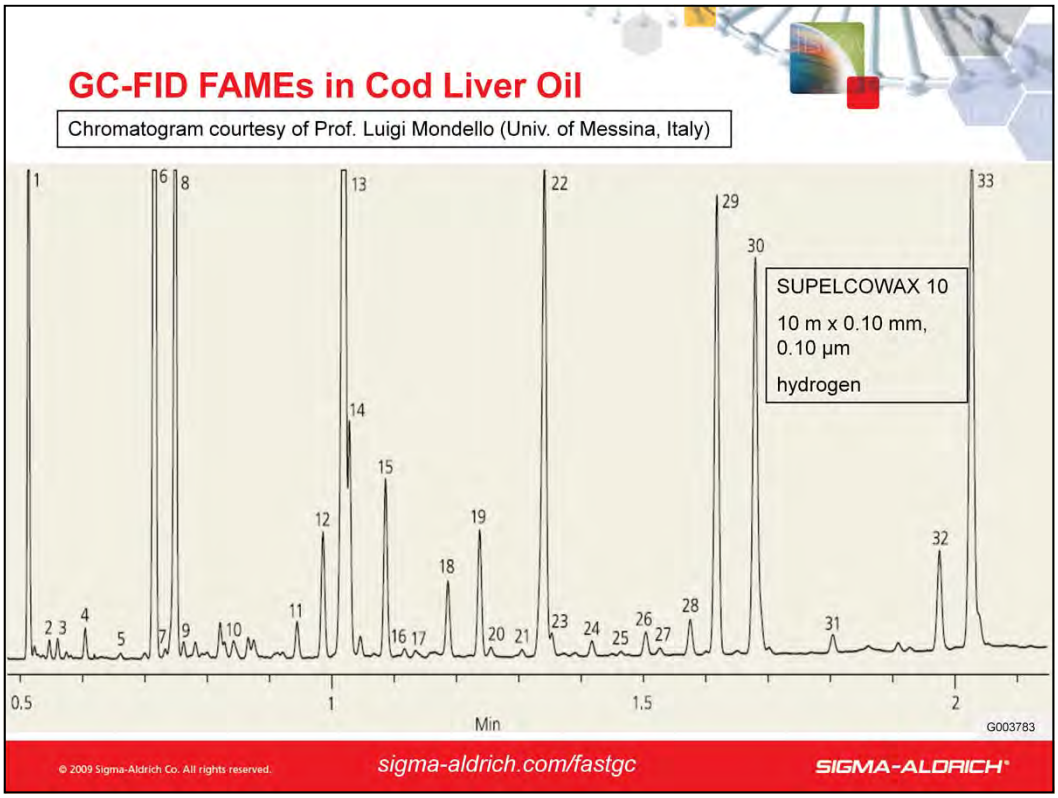
The food & beverage industry must also be concerned with measuring saturated fat content versus monounsaturated and polyunsaturated fat content for nutritional purposes. Here, the short analysis (~4.5 min.) of a polyunsaturated fatty acid (PUFA) mix was accomplished using Fast GC and an Omegawax 100 column.

Conditions:

column: Omegawax 100, 15 m x 0.10 mm I.D., 0.10 µm (23399-U)
oven: 140 ° C, 40 ° C/min. to 280 ° C (2 min.)
inj.: 250 ° C
det.: FID, 280 ° C
carrier gas: hydrogen, 50 cm/sec constant
injection: 0.2 µL, 200:1 split
liner: 4 mm I.D., split, cup design
sample: PUFA No. III – Menhaden Oil (47085-U), diluted to 50 mg/mL in methylene chloride

Peak IDs:

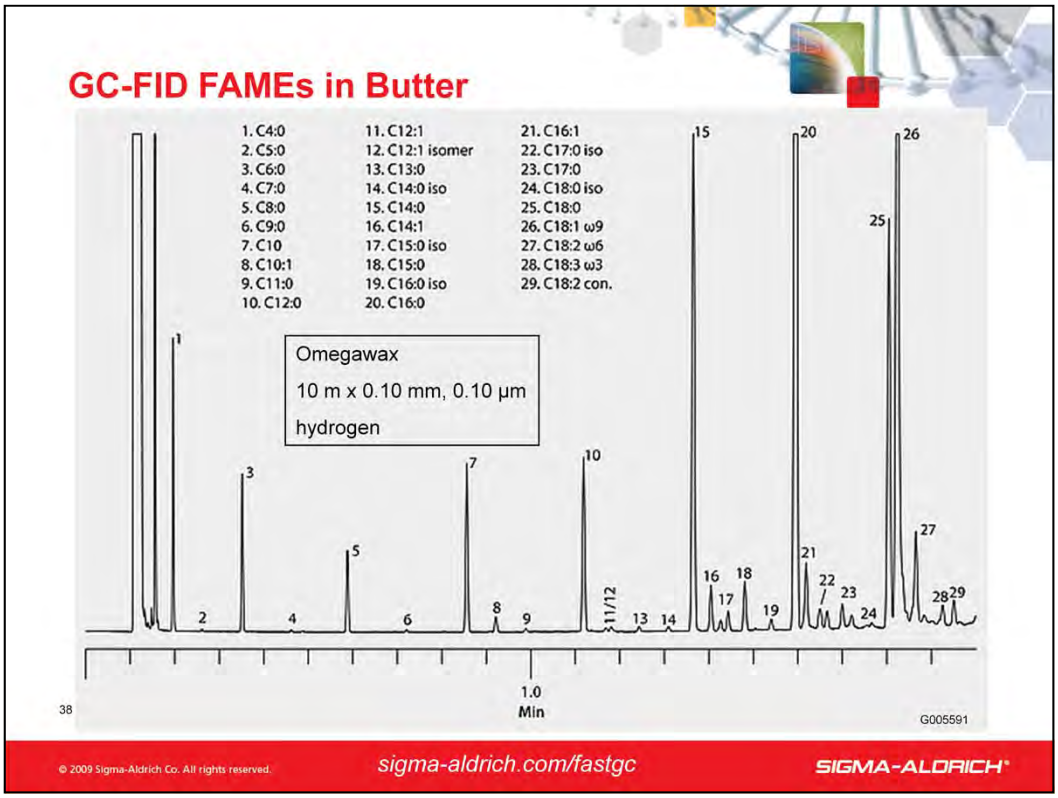
1. C14:0
2. C16:0
3. C16:1n7
4. C16:2n4
5. C16:3n4
6. C16:4n1
7. C18:0
8. C18:1n9
9. C18:1n7
10. C18:2n6
11. C18:3n4
12. C18:3n3
13. C18:4n3
14. C20:1n9
15. C20:4n6
16. C20:4n3
17. C20:5n3
18. C22:5n3
19. C22:6n3



The health benefits of omega 3 and omega 6 fats are well-publicized. Because the human body cannot produce these, they must be obtained through our diet. Data suggests that the omega 3 group provides the greater health benefit. Fish oils tend to be high in omega 3 fat content. The monitoring of the omega 3 and omega 6 amounts and ratios in many foods is routinely performed in the food & beverage industry. This application, provided courtesy of Prof. Luigi Mondello at the University of Messina, Italy, shows the Fast GC analysis of the FAMEs in a cod liver oil sample. A short 10 m x 0.10 mm I.D. SUPELCOWAX 10 column, rapid oven heating, and a high hydrogen linear velocity was used to complete the analysis in ~2.1 in.

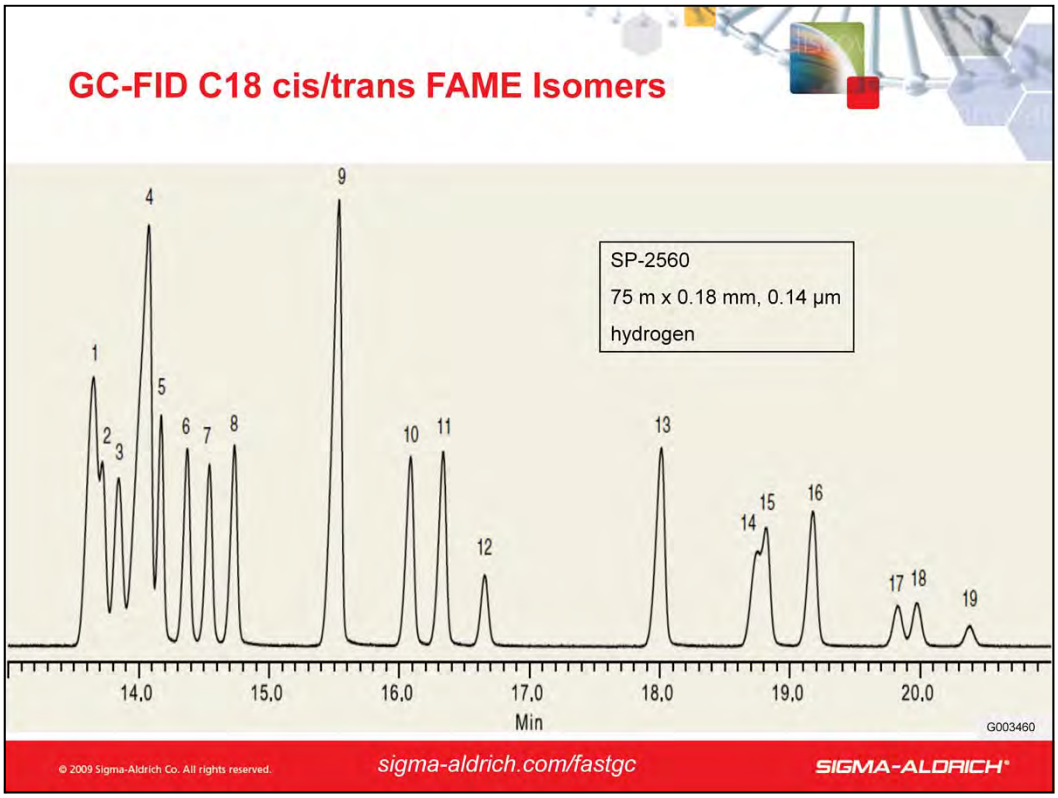
Conditions:
column: SUPELCOWAX 10, 10 m x 0.10 mm I.D., 0.10 µm (25026-U)
oven: 180 ° C , 40 ° C/min. to 270 ° C (0.5 min.)
inj.: 280 ° C
det.: FID, 280 ° C
carrier gas: hydrogen, 100 cm/sec constant
injection: 0.2 µL, 200:1 split
sample: cod liver oil FAMEs in hexane

Peak IDs:
1. C14:0
2. C15:0 anteiso
3. C15:0 iso
4. C15:0
5. C16:0 iso
6. C16:0
7. C16:1ω9
8. C16:1ω7
9. C16:1ω5
10. C16:3ω4
11. C16:4ω4
12. C18:0
13. C18:1ω9
14. C18:1ω7
15. C18:2ω6
16. C18:2ω4
17. C18:3ω6
18. C18:3ω3
19. C18:4ω3
20. C18:4ω1
21. C20:0
22. C20:1ω9
23. C20:1ω7
24. C20:2ω6
25. C20:3ω6
26. C20:4ω6
27. C20:3ω3
28. C20:4ω3
29. C20:5ω3
30. C22:1ω9
31. C21:5ω3
32. C22:5ω3
33. C22:6ω3



Butter contains a concentration of the fat found in the milk source. This is because almost all of milk's nutrients are retained during the manufacturing process. This chromatogram shows the analysis of fatty acids (as fatty acid methyl esters) extracted from a butter sample. The Omegawax is able to provide separation of many FAME isomers.

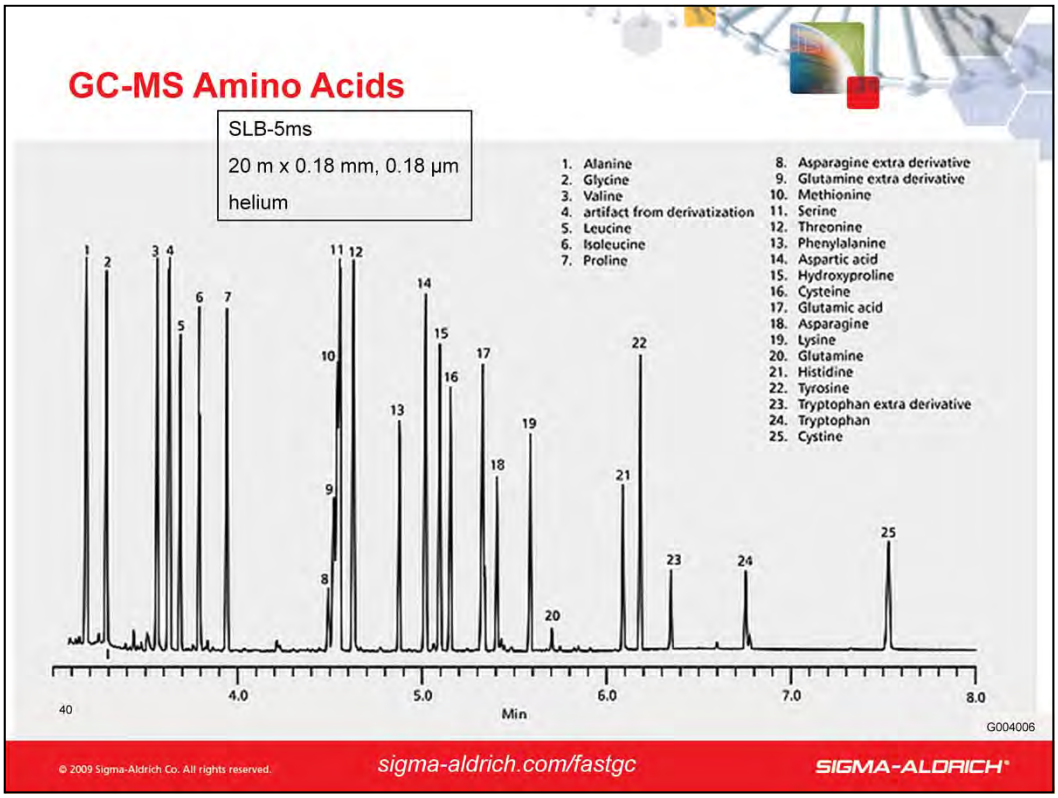
Conditions:
column: Omegawax, 10 m x 0.10 mm I.D., 0.10 µm (custom)
oven: 50 °C, 90.0 °C/min to 250 °C
inj.: 250 °C
det.: FID, 250 °C
carrier gas: hydrogen, 116.0 cm/sec, 400 kPa
injection: 0.2 µL, 200:1 split
sample: butter FAMES, diluted 20:1 in hexane



The cis/trans FAME isomers are difficult to separate because they chemically alike, requiring long columns with highly polar stationary phases, such as the SP-2560. For nutritional purposes, the food & beverage industry needs to identify the type and amount of trans fats. Recent laws governing the information on food labels has increased the number of samples that must be processed. Here, the detailed analysis of the C18 cis/trans FAME isomers was accomplished in ~20.5 min. by applying the Principles of Fast GC.

Conditions:
column: SP-2560, 75 m x 0.18 mm I.D., 0.14 µm (23348-U)
oven: 180 ° C
inj.: 220 ° C
det.: FID, 220 ° C
carrier gas: hydrogen, 25 cm/sec @ 180 ° C
injection: 0.5 µL, 100:1 split
liner: 4 mm I.D., split, cup design
sample: Mixture of C18:1, C18:2, and C18:3 FAMEs in methylene chloride

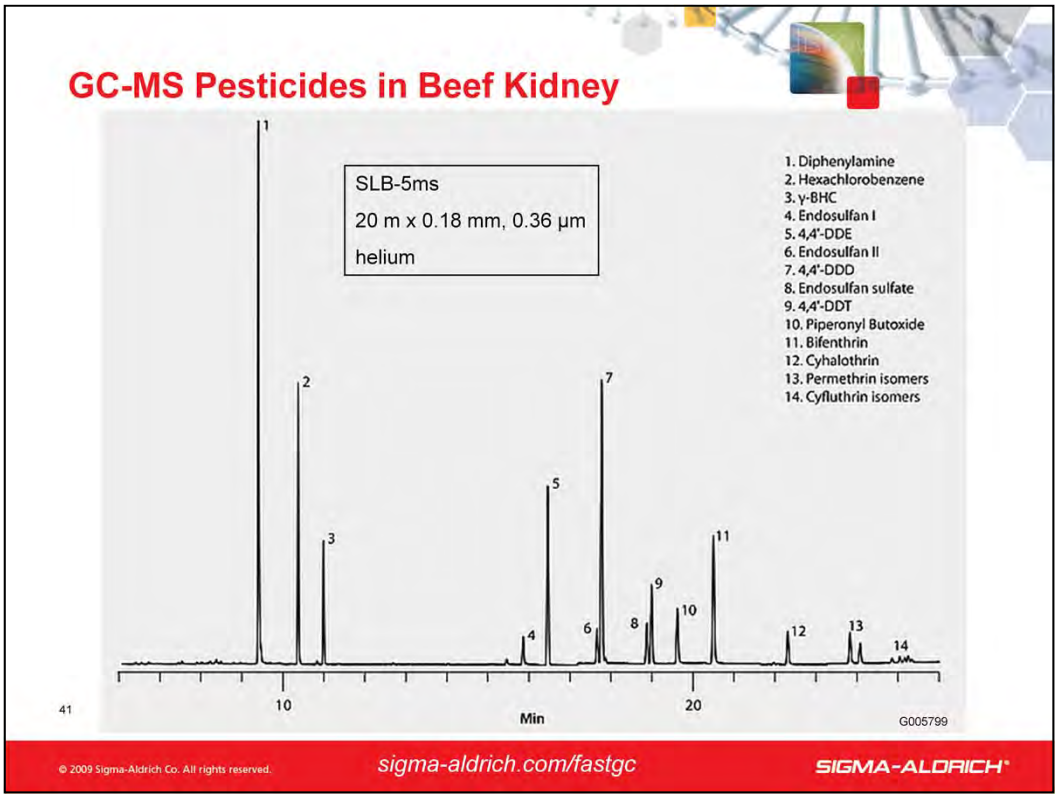
Peak IDs:
1. C18:1 Δ 7t and C18:1 Δ 6t
2. C18:1 Δ 9t
3. C18:1 Δ 11t
4. C18:1 Δ 12t, C18:1 Δ 6c, C18:1 Δ 7c and C18:1 Δ13t
5. C18:1 Δ 9c
6. C18:1 Δ 11c
7. C18:1 Δ 12c
8. C18:1 Δ 13c
9. C18:2 Δ 9t, 12t
10. C18:2 Δ 9c, 12t
11. C18:2 Δ 9t, 12c
12. C18:2 Δ 9c, 12c
13. C18:3 Δ 9t, 12t, 15t
14. C18:3 Δ 9t, 12t, 15c
15. C18:3 Δ 9t, 12c, 15t
16. C18:3 Δ 9c, 12t, 15t and C18:3 Δ 9c, 12c, 15t
17. C18:3 Δ 9c, 12t, 15c
18. C18:3 Δ 9t, 12c, 15c
19. C18:3 Δ 9c, 12c, 15c



Proteins are a major source of energy in foods. They contain essential amino acids (such as lysine, methionine, and valine) that cannot be synthesized in the body. Typically, high performance liquid chromatography (HPLC) is used for the analysis of amino acids. However, GC can also be used, and in some cases availability of instrumentation or operation costs can make it a better choice. The polar nature of amino acids requires derivatization prior to GC analysis. The goal of derivatization is to make an analyte more volatile, less reactive, and thus improve its chromatographic behavior. In the case of amino acids, derivatization replaces active hydrogens on OH, NH2, and SH polar functional groups with a nonpolar moiety.

Conditions:

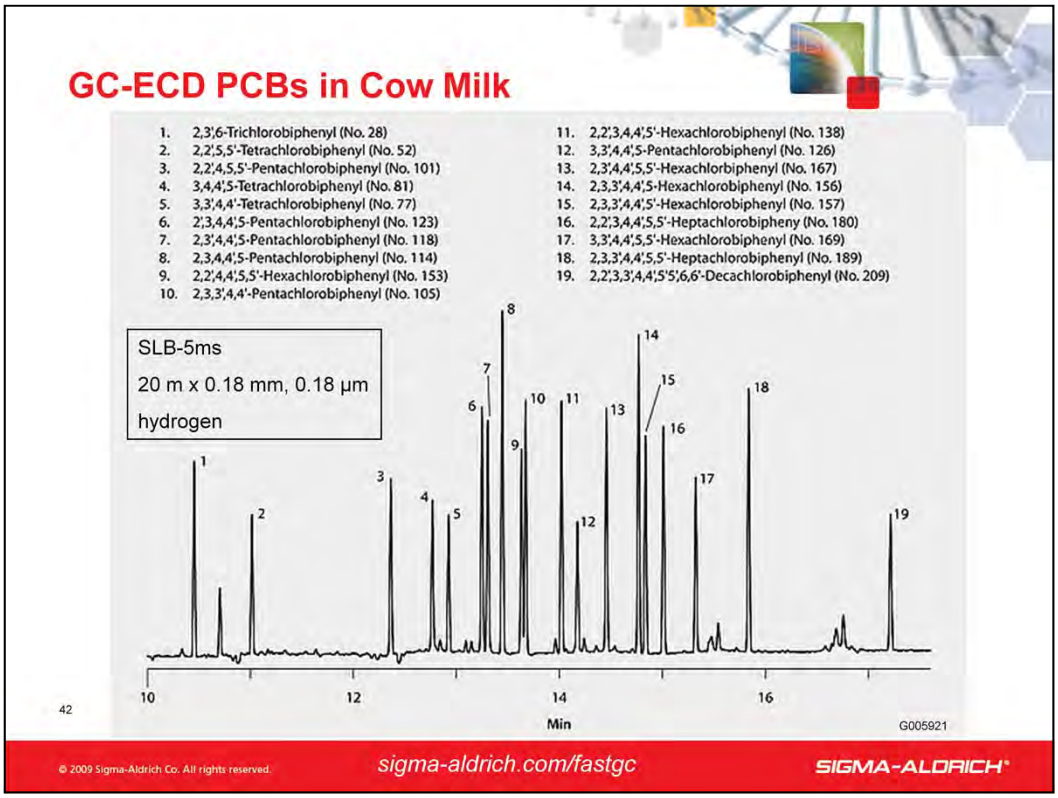
column: SLB-5ms, 20 m × 0.18 mm I.D., 0.18 µm (28564-U)
oven: 100 ° C (1 min), 35 ° C/min to 290 ° C (3 min), 40 ° C/min to 360 ° C
inj.: 250 ° C
MSD interface: 325 ° C
scan range: m/z = 40-450
carrier gas: helium, 1 mL/min, constant
injection: 0.5 µL, splitless (1.0 min)
liner: 2 mm I.D., straight
sample: TBDMS derivatives of amino acids, each approximately 23 ug/mL



Pesticides are sprayed on crops to minimize insect damage. When the crops are used as animal feed, they can end up in meat. Analysis of pesticide levels in food and beverage products is important not only to insure low levels for human consumption, but also to avoid international trade problems. At the present time, more than 1000 pesticide and metabolite residue compounds are identified as associated with food crops, either in current use, or used in the past. A beef kidney sample was analyzed for pesticides by GC-MS (SIM) after extraction and cleanup.

Conditions:

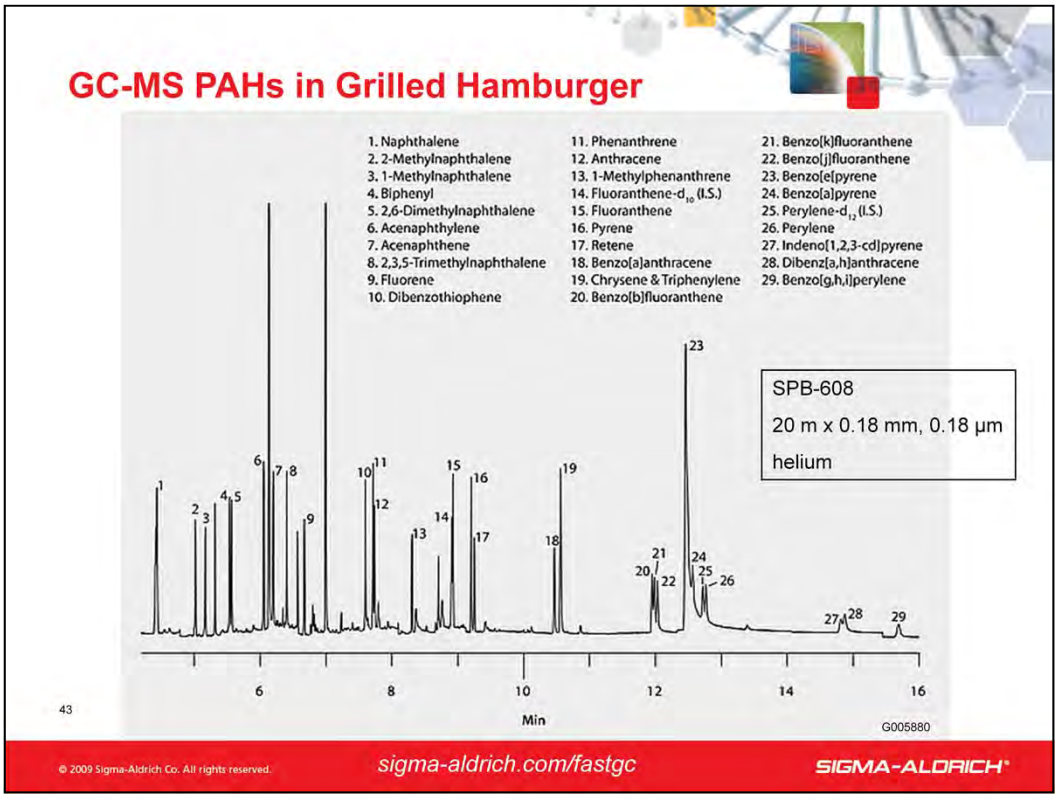
- sample/matrix: 10 gm homogenized beef kidney spiked at 50 ng/g with pesticides
- extraction tube: Supel Que Acetate (AC) Tube (55234-U)
- extraction process: add 10 mL of acetonitrile; shake for 1 minute; add contents of Supel Que acetate extraction tube (55234-U); shake immediately for 1 minute; centrifuge at 3200 rpm for 5 minutes; (Sample Pretreatment: Homogenize 10 gm beef kidney and spike with pesticides at 50 ng/g.)
- clean-up tube: Supel QuE Z-Sep+ tube (55296-U)
- clean-up process: transfer 3 mL of the acetonitrile layer into a Supel QuE Z-Sep+ cleanup tube (55296-U); shake for 1 minute; centrifuge at 3400 rpm for 3 minutes; draw off supernatant for LVI-GCMS analysis
- column: SLB-5ms, 20 m x 0.18 mm I.D., 0.36 µm (28576-U)
- oven: 70 ° C (0.5 min), 25 ° C/min to 125 ° C, 10 ° C/min to 200 ° C, 5 ° C/min to 300 ° C/min (1 min)
- inj.: programmed, 60 ° C (0.28 min), 600 ° C/min to 325 ° C (5 min)
- det.: MSD
- MSD interface: 325 ° C
- carrier gas: helium, 1 mL/min constant flow
- injection: 10 µL LVI, PTV solvent vent, rapid injection speed
- liner: 4 mm ID FocusLiner with taper



Similar as pesticides, PCBs can end up in animal meat and additional products if the animal feed is tainted with PCBs. Such is the case with PCBs in cow milk. This application demonstrates the use of dispersive SPE or "QuEChERS" for the extraction and cleanup of whole cow′s milk for GC-ECD analysis of polychlorinated biphenyl (PCBs).

Conditions:

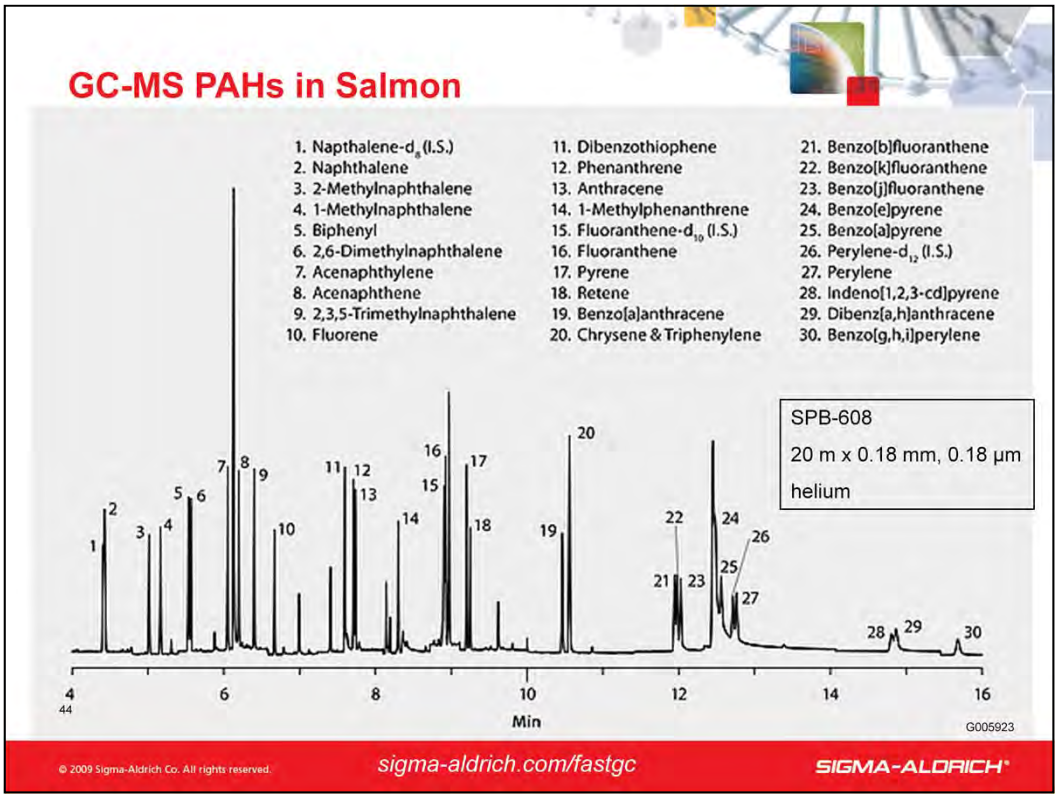
- sample/matrix: 10 g whole cow′s milk
- extraction tube: Supel™ QuE Non-buffered tube (4 g magnesium sulfate, 1 g sodium chloride) (55294-U)
- clean-up tube: Supel™ QuE Z-Sep 150 mg, magnesium sulfate (150 mg, part no. 63135) (55299-U)
- column: SLB-5ms, 20 m x 0.18 mm I.D., 0.18 µm (28564-U)
- oven: 75 ° C (1 min), 12 ° C/min to 340 ° C (10 min)
- inj. temp.: 250 ° C
- detector: µ-ECD, 340 ° C
- carrier gas: hydrogen, 1.2 mL/min, constant
- injection: 1 µLsplitless (0.75 min)
- liner: 4 mm I.D. FocusLiner™ with taper



Polycyclic aromatic hydrocarbons (PAHs) are produced as by-products of cooking. Several PAHs are identified as carcinogens. The PAHs of interest include several isomer sets. This chromatogram of 29 PAHs on highlights the ability of Fast GC to provide resolution with difficult matrix, such as a fatty hamburger. A 20 m x 0.18 mm I.D., 0.18 µm column was used for the timely elution of the heavier PAHs.

Conditions:

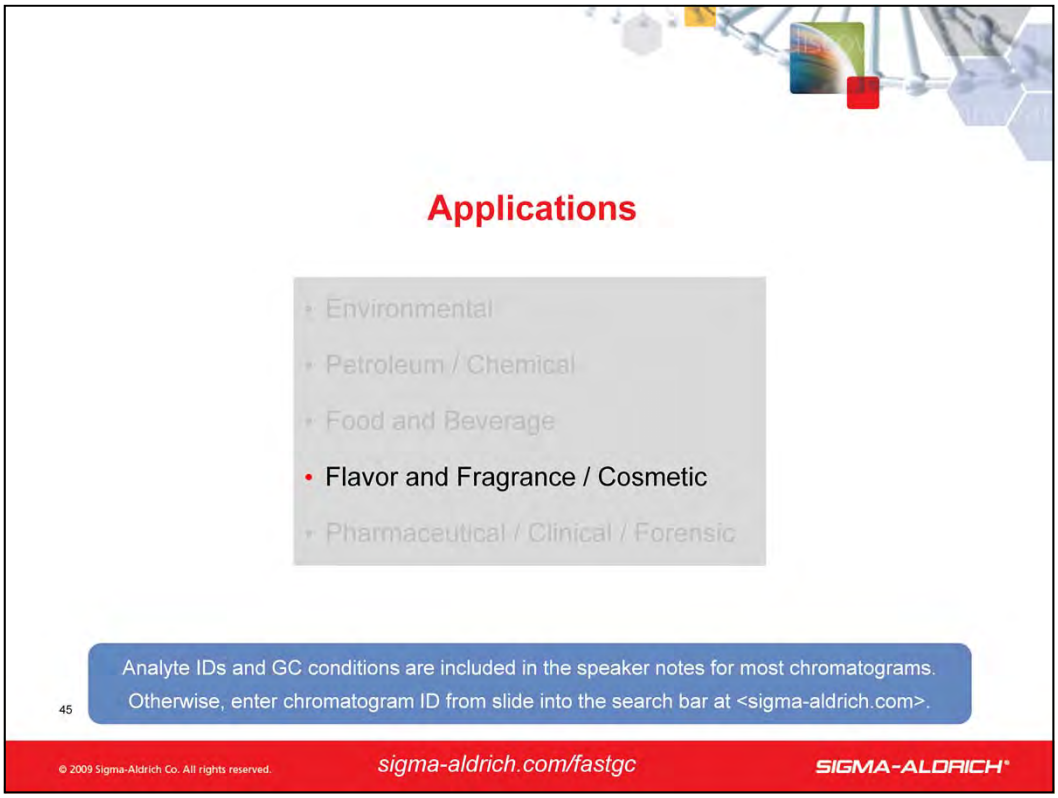
- sample/matrix: grilled hamburger spiked with PAHs at 100 ng/g
- extraction tube: Sodium chloride/magnesium sulfate (55294-U)
- extraction process: add 25 mL acetonitrile, contents of unbuffered salt tube (55294-U); shake for 5 minutes; centrifuge for 5 minutes at 3200 rpm
- clean-up tube: Supel QuE Z-Sep tube (55403-U)
- clean-up process: transfer 3 mL of acetonitrile layer into a Supel QuE Z-Sep cleanup tube (55403-U); shake 1 minute; centrifuge for 3 minutes at 3400 rpm; remove 1 mL supernatant for GC-MS analysis
- column: SPB-608, 20 m x 0.18 mm I.D. x 0.18 µm
- oven: 60 ° C (1 min), 25 ° C/min to 275 ° C, 10 ° C/min to 300 ° C (13 min)
- inj. temp.: 265 ° C
- detector: MS-SIM
- MSD interface: 300 ° C
- carrier gas: helium, 1.5 mL/min constant flow
- injection: 1 µLsplitless (1 min.)
- liner: 4 mm I.D. FocusLiner™ with taper



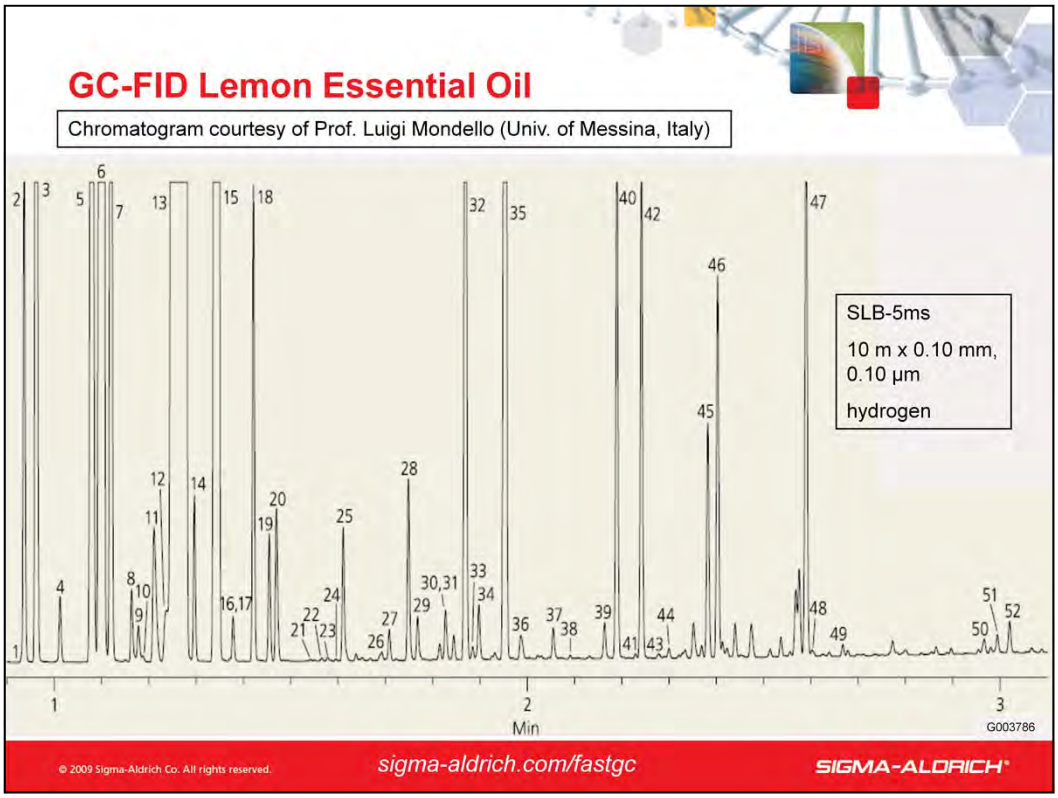
This application also demonstrates the ability of Fast GC to provide resolution with difficult matrix, raw salmon in this case. A 20 m x 0.18 mm I.D., 0.18 µm column was used for the timely elution of the heavier PAHs.

Conditions:

- sample/matrix: raw salmon spiked with PAHs at 100 ng/g
- extraction tube: Supel™ QuE Non-buffered tube, magnesium sulfate/sodium chloride (55294-U)
- extraction process: add 25 mL acetonitrile and the contents of the Supel™ QuE Non-buffered tube (55294-U); shake for 5 minutes; centrifuge for 5 minutes at 3200 rpm
- clean-up tube: Supel™ QuE Z-Sep (500 mg, part no. 55299-U), magnesium sulfate (450 mg, part no. 63135) (55299-U, 63135)
- clean-up process: transfer 3 mL of acetonitrile layer, Supel QuE Z-Sep (500 mg) and MgSO4 (450 mg) into an extraction tube; (shake 1 minute; centrifuge for 3 minutes at 3400 rpm; remove 1 mL supernatant for GC-MS analysis)
- column: SPB-608, 20 m x 0.18 mm I.D. x 0.18 µm
- oven: 60 ° C (1 min), 25 ° C/min to 275 ° C, 10 ° C/min to 300 ° C (13 min)
- inj. temp.: 265 ° C
- detector: MS, SIM
- MSD interface: 300 ° C
- carrier gas: helium, 1.5 mL/min, constant
- injection: 1 µL, splitless (1 min)
- liner: 4 mm I.D. FocusLiner™ with taper



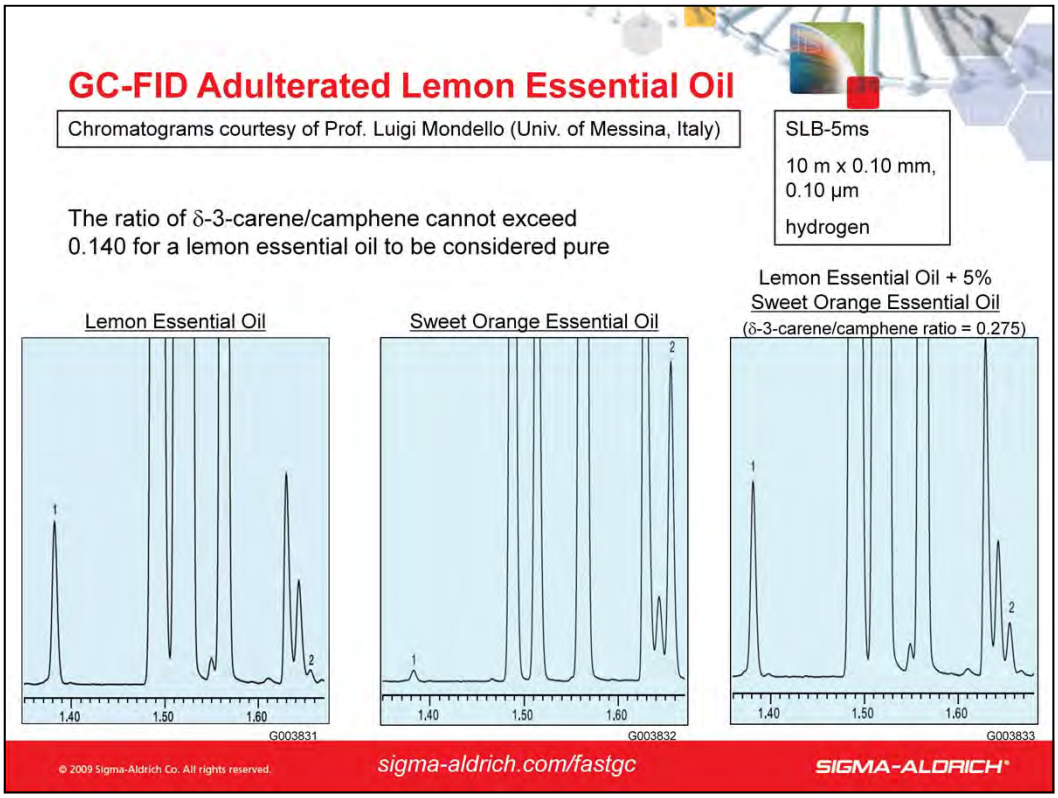
This section contains a handful of applications. Remember, the more of the Principles of Fast GC that are applied, the greater the benefit. Due to instrumentation limitations (such as when using GC-MS), some of these chromatograms use helium and not hydrogen as the carrier gas. In these instances, the slide will contain this information.



The flavor & fragrance industry needs to test raw materials to ensure purity, and to monitor finished goods to detect any adulteration with less expensive products. Here, the Fast GC analysis of a lemon essential oil sample was accomplished in ~3 min. on an SLB-5ms column.

Conditions:

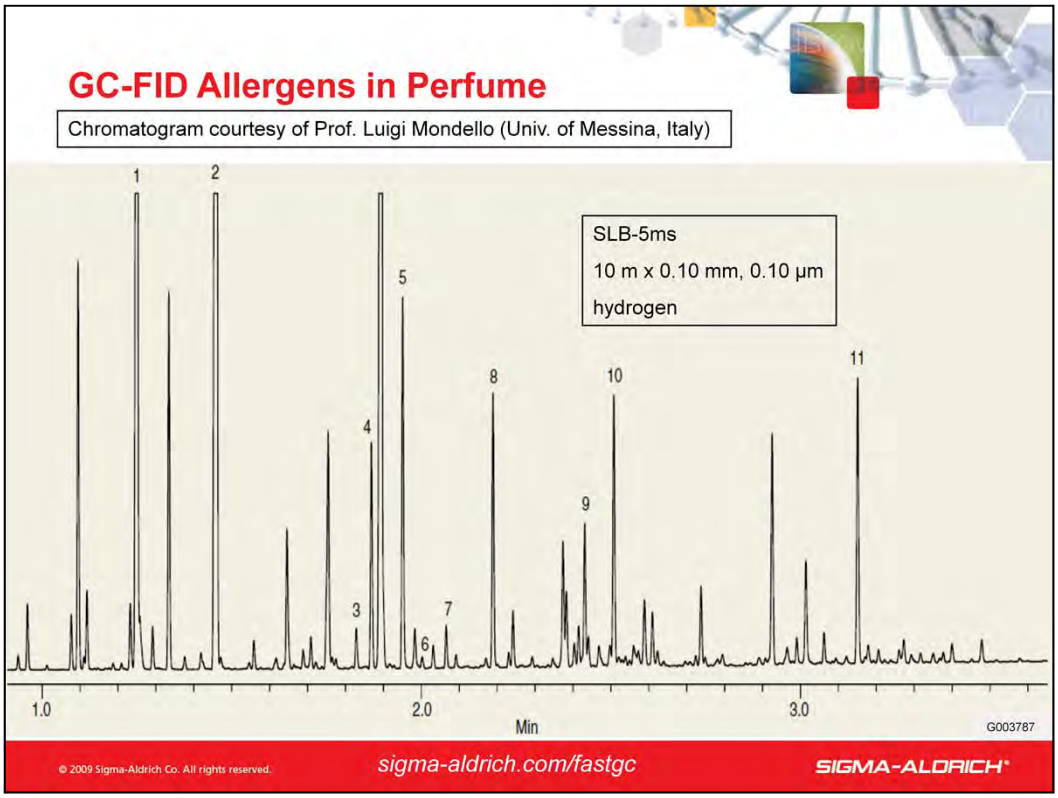
column: SLB-5ms, 10 m x 0.10 mm I.D., 0.10 µm (28465-U)
oven: 40 ° C, 50 ° C/min. to 320 ° C
inj.: 320 ° C
det.: FID, 320 ° C
carrier gas: hydrogen, 81.5 cm/sec constant
injection: 0.4 µL, 300:1 split
sample: lemon essential oil in hexane



Here is an example of a product adulteration that the flavor & fragrance industry looks for. A less expensive citrus essential oil (such as sweet orange essential oil) is mixed with a more expensive citrus essential oil (such as lemon essential oil) to increase the perceived volume of the more expensive citrus essential oil. Smell and taste alone may not be adequate to detect this adulteration. GC can be used by determining the ratios of two compounds, δ-3-carene/camphene. The ratio of δ-3-carene/camphene cannot exceed 0.140 for a lemon essential oil to be considered pure. In these chromatograms provided by Prof. Mondello, the Fast GC analyses of pure and adulterated products are shown on an SLB-5ms column.

Conditions:
column: SLB-5ms, 10 m x 0.10 mm I.D., 0.10 µm (28465-U)
oven: 40 ° C, 30 ° C/min. to 85 ° C, 80 ° C/min. to 320 ° C
inj.: 320 ° C
det.: FID, 320 ° C
carrier gas: hydrogen, 70 cm/sec constant
injection: 0.4 µL, 300:1 split
sample 1: lemon essential oil in hexane
sample 2: sweet orange essential oil in hexane
sample 3: lemon essential oil + 5% sweet orange essential oil in hexane

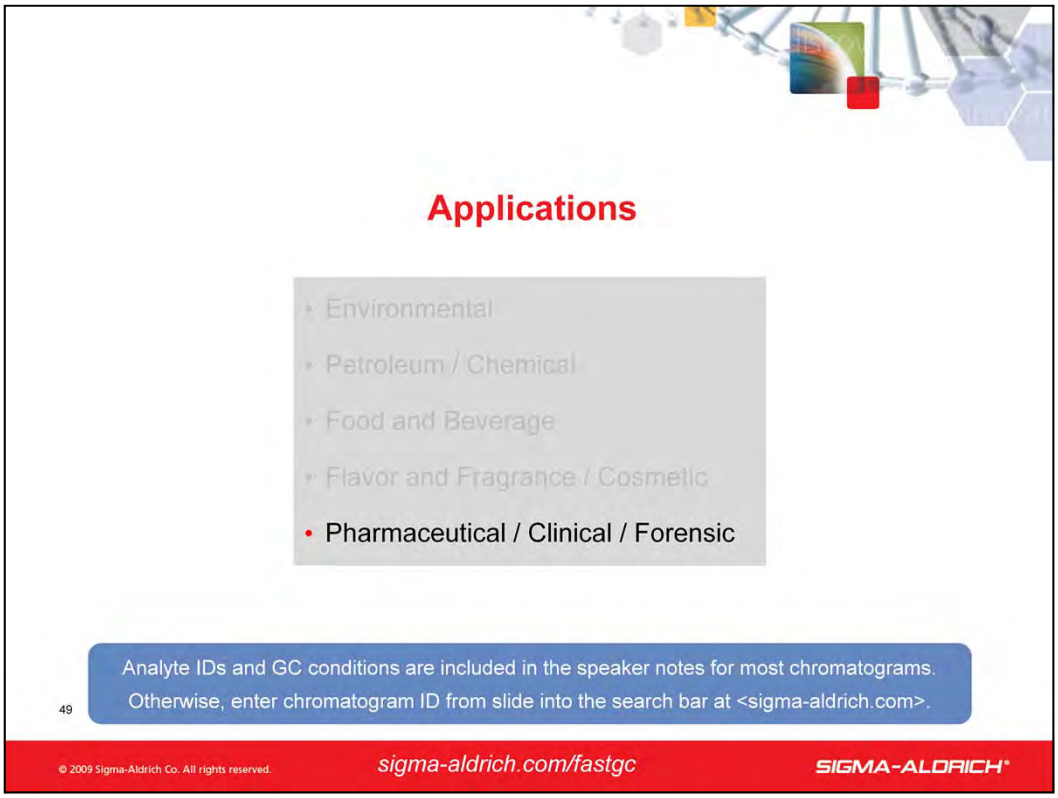
Peak IDs:
1. Camphene
2. δ-3-Carene



The personal care product industry is interested in determining the type and levels of allergens in many of their commercial items. In this chromatogram provide by Prof. Mondello, the allergens eugenol (peak 8) and coumarin (peak 9) are identified along with the some of the major fragrance components in a commercially available perfume. Using Fast GC, the analysis was completed in ~3.5 min. using an SLB-5ms column.

Conditions:
column: SLB-5ms, 10 m x 0.10 mm I.D., 0.10 µm (28465-U)
oven: 40 ° C , 50 ° C/min. to 320 ° C
inj.: 320 ° C
det.: FID, 320 ° C
carrier gas: hydrogen, 81.5 cm/sec constant
injection: 0.2 µL, 500:1 split
sample: Neat perfume

Peak IDs:
1. Limonene
2. Linalool
3. Citronellol
4. Neral
5. Geranial
6. Hydroxycitronellal
7. Cinnamyl alcohol
8. Eugenol
9. Coumarin
10. α-Isomethylionone
11. Hexyl cinnamylaldehyde



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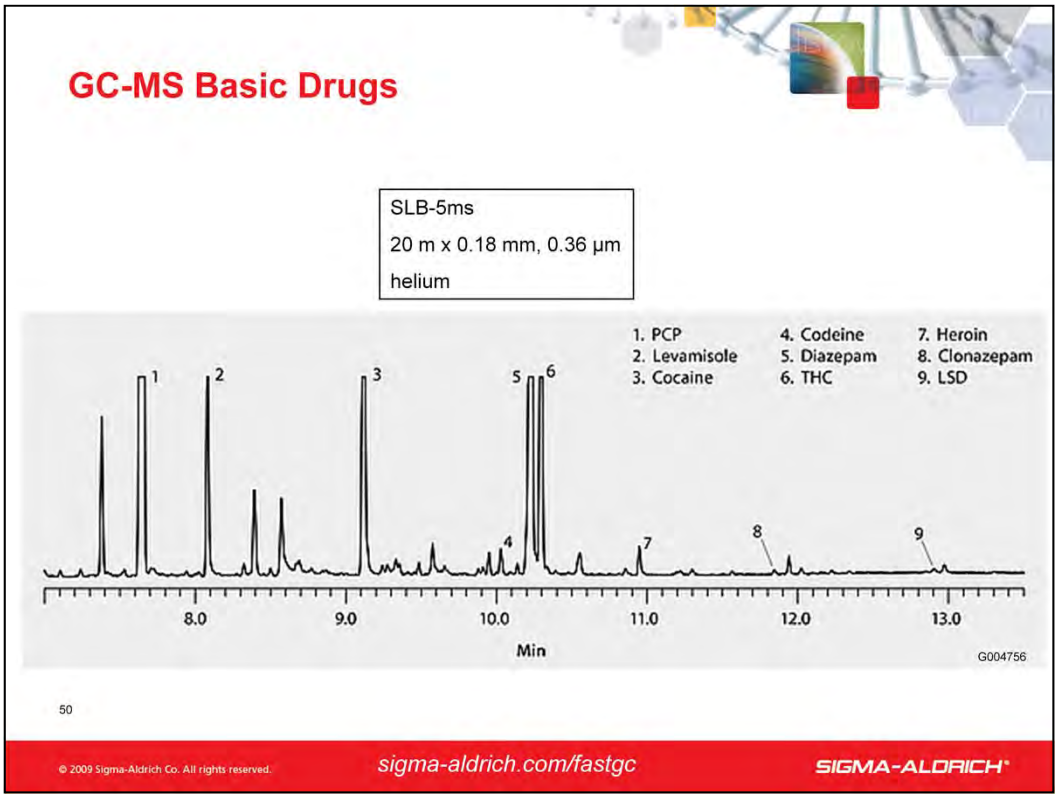
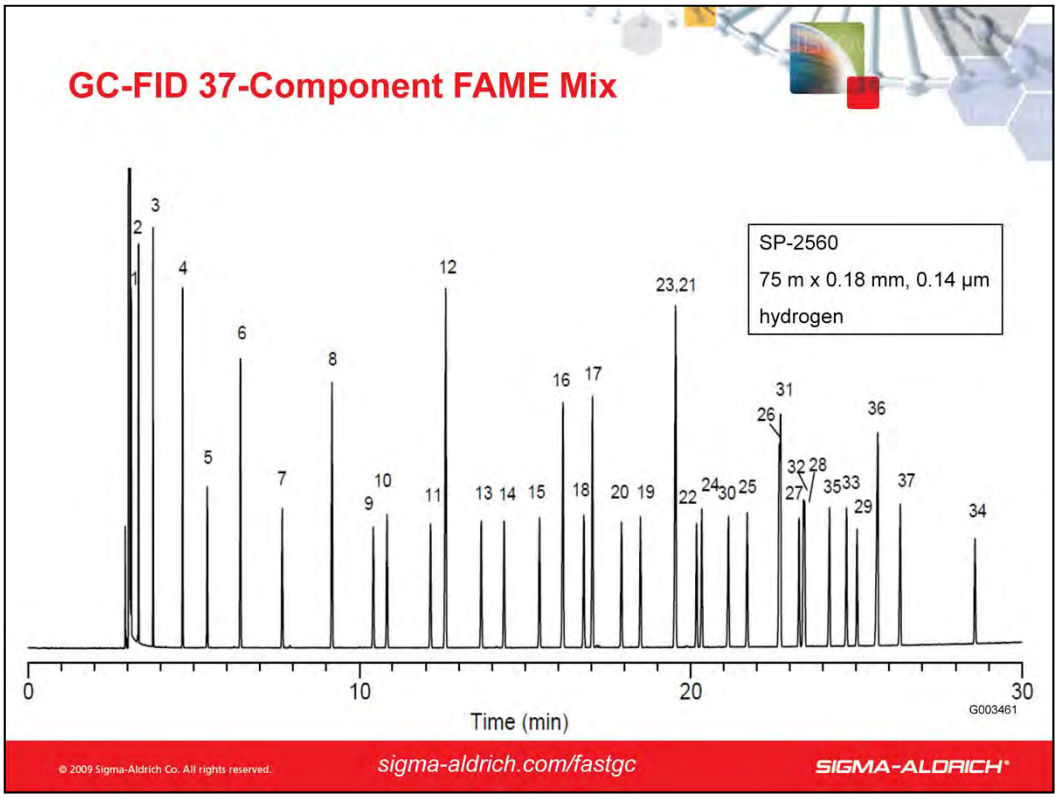


Many drugs of abuse are basic in nature. Analysis typically begins with a sample matrix of urine. Solid phase microextraction is a quick sample preparation technique for aquous-based matrices. This application shows that Fast GC is compatible with SPME.

Conditions:

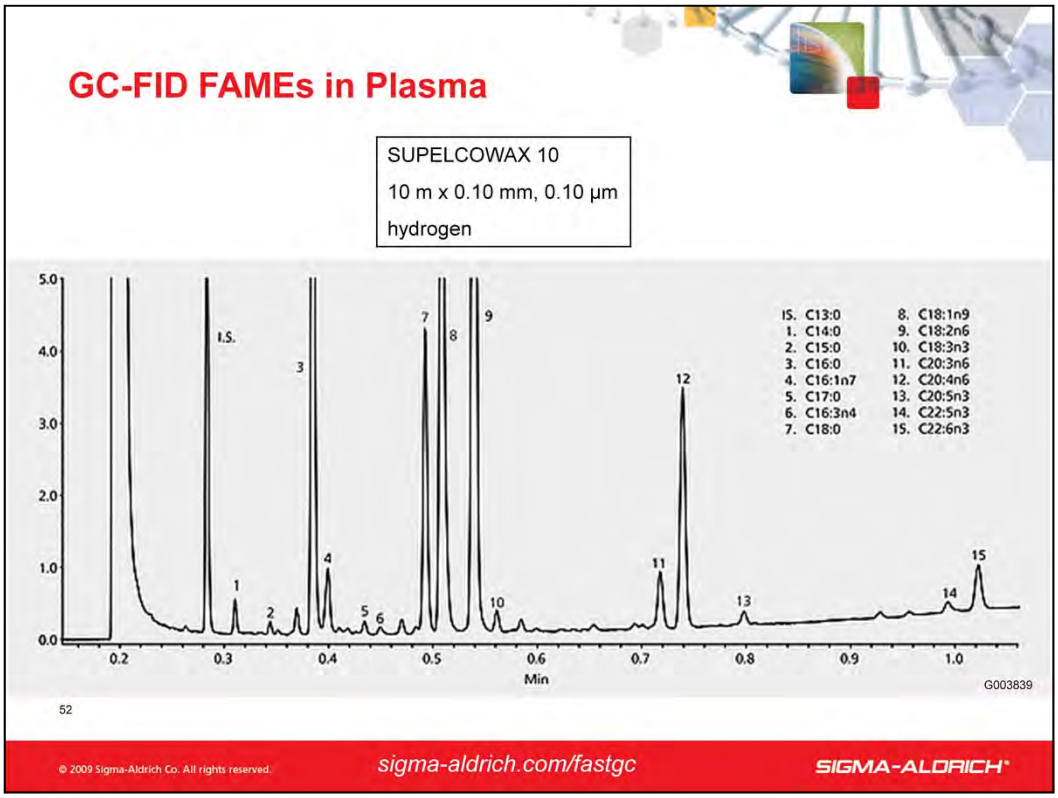
- sample/matrix: 500 ug/L of each compound in 2 mL 0.05M phosphate buffer, pH=11, plus sodium chloride (NaCl) to 25% (w/v)
- SPME fiber: 85 µm Polyacrylate (57304)
- extraction: immersion, 450 rpm stirring at room temp. for 15 min
- desorption process: 250 ˚ C for 3 min
- column: SLB-5ms, 20 m x 0.18 mm I.D. x 0.36 µm (28576-U)
- oven: 100 ˚ C (1 min), 20 ˚ C/min to 325 ˚ C (5 min)
- MSD interface: 325 ˚ C
- carrier gas: helium, 1.5 mL/min. constant
- liner: 0.75 mm I.D. SPME



One indicator of a person's wellness is their fatty acid profile. This involves determining the ratios and amounts of various saturated, monounsaturated, and polyunsaturated fatty acids. This is accomplished by extraction of fatty acids from plasma, conversion to FAMEs, and analysis by GC. This chromatogram of C4 to C24 FAMEs on the SP-2560 allows clinical labs to deliver fatty acid composition information.

Conditions:
column: SP-2560, 75 m x 0.18 mm I.D., 0.14 µm (23348-U)
oven: 140 ° C (5 min.), 4 ° C/min. to 240 ° C (2 min.)
inj.: 250 ° C
det.: FID, 250 ° C
carrier gas: hydrogen, 40 cm/sec @ 175 ° C
injection: 1 µL, 100:1 split
liner: 4 mm I.D, split, cup design
sample: Supelco 37-Component FAME Mix (47885-U), analytes at concentrations indicated in methylene chloride

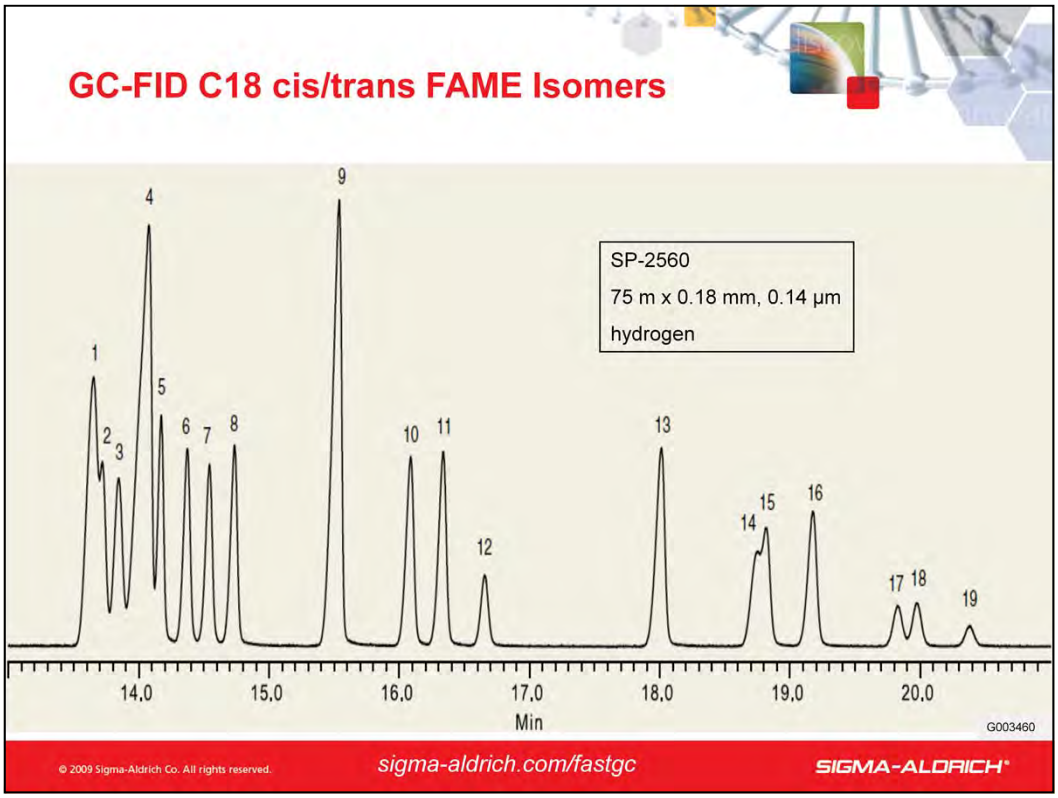
Peak IDs:
1. Butyric Acid Methyl Ester (C4:0) at 4 wt %
2. Caproic Acid Methyl Ester (C6:0) at 4 wt %
3. Caprylic Acid Methyl Ester (C8:0) at 4 wt %
4. Capric Acid Methyl Ester (C10:0) at 4 wt %
5. Undecanoic Acid Methyl Ester (C11:0) at 2 wt %
6. Lauric Acid Methyl Ester (C12:0) at 4 wt %
7. Tridecanoic Acid Methyl Ester (C13:0) at 2 wt %
8. Myristic Acid Methyl Ester (C14:0) at 4 wt %
9. Myristoleic Acid Methyl Ester (C14:1) at 2 wt %
10. Pentadecanoic Acid Methyl Ester (C15:0) at 2 wt %
11. cis-10-Pentadecenoic Acid Methyl Ester (C15:1) at 2 wt %
12. Palmitic Acid Methyl Ester (C16:0) at 6 wt %
13. Palmitoleic Acid Methyl Ester (C16:1) at 2 wt %
14. Heptadecanoic Acid Methyl Ester (C17:0) at 2 wt %
15. cis-10-Heptadecenoic Acid Methyl Ester (C17:1) at 2 wt %
16. Stearic Acid Methyl Ester (C18:0) at 4 wt %
17. Oleic Acid Methyl Ester (C18:1n9c) at 4 wt %
18. Elaidic Acid Methyl Ester (C18:1n9t) at 2 wt %
19. Linoleic Acid Methyl Ester (C18:2n6c) at 2 wt %
20. Linolelaidic Acid Methyl Ester (C18:2n6t) at 2 wt %
21. γ-Linolenic Acid Methyl Ester (C18:3n6) at 2 wt %
22. α-Linolenic Acid Methyl Ester (C18:3n3) at 2 wt %
23. Arachidic Acid Methyl Ester (C20:0) at 4 wt %
24. cis-11-Eicosenoic Acid Methyl Ester (C20:1n9) at 2 wt %
25. cis-11,14-Eicosadienoic Acid Methyl Ester (C20:2) at 2 wt %
26. cis-8,11,14-Eicosatrienoic Acid Methyl Ester (C20:3n6) at 2 wt %
27. cis-11,14,17-Eicosatrienoic Acid Methyl Ester (C20:3n3) at 2 wt %
28. Arachidonic Acid Methyl Ester (C20:4n6) at 2 wt %
29. cis-5,8,11,14,17-Eicosapentaenoic Acid Methyl Ester (C20:5n3) at 2 wt %
30. Heneicosanoic Acid Methyl Ester (C21:0) at 2 wt %
31. Behenic Acid Methyl Ester (C22:0) at 4 wt %
32. Erucic Acid Methyl Ester (C22:1n9) at 2 wt %
33. cis-13,16-Docosadienoic Acid Methyl Ester (C22:2) at 2 wt %
34. cis-4,7,10,13,16,19-Docosahexaenoic Acid Methyl Ester (C22:6n3) at 2 wt %
35. Tricosanoic Acid Methyl Ester (C23:0) at 2 wt %
36. Lignoceric Acid Methyl Ester (C24:0) at 4 wt %
37. Nervonic Acid Methyl Ester (C24:1n9) at 2 wt %



This 'real-world' application is of FAMEs extracted from plasma then converted to FAMEs prior to analysis. It highlights that Fast GC is not just for clean standards, but can handle dirtly samples in production settings.

Conditions:

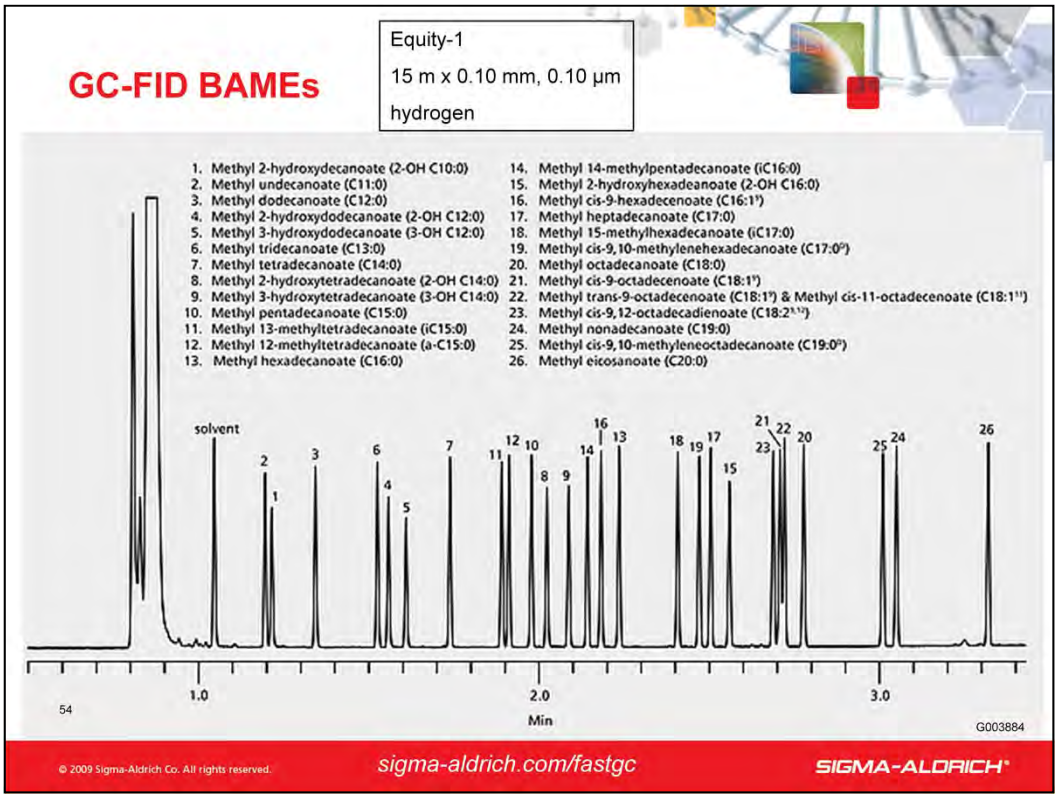
- column: SUPELCOWAX 10, 10 m × 0.10 mm I.D., 0.10 µm (25026-U)
- oven: 220 ° C, 60 ° C/min to 280 ° C (1 min)
- inj. temp.: 280 ° C
- detector: FID, 280 ° C
- carrier gas: hydrogen, 120 cm/sec constant
- injection: 0.5 µL, 30:1 split
- sample: plasma FAMEs in hexane



In addition to general lipid tests (such as HDL, LDL, and triglycerides), health care providers may also be interested in individual analytes. Trans fatty acids cause adverse health consequences. Some of the most common unsaturated fatty acids are the C18 series. Testing is accomplished by extraction of fatty acids from plasma, conversion to FAMEs, and analysis by GC. This chromatogram shows a detailed analysis of C18:1, C18:2, and C18:3 fatty acid isomers, as fatty acid methyl esters (FAMEs) on a SP-2560 provides decent resolution in a relatively short run.

Conditions:
column: SP-2560, 75 m x 0.18 mm I.D., 0.14 µm (23348-U)
oven: 180 ° C
inj.: 220 ° C
det.: FID, 220 ° C
carrier gas: hydrogen, 25 cm/sec @ 180 ° C
injection: 0.5 µL, 100:1 split
liner: 4 mm I.D., split, cup design
sample: Mixture of C18:1, C18:2, and C18:3 FAMEs in methylene chloride

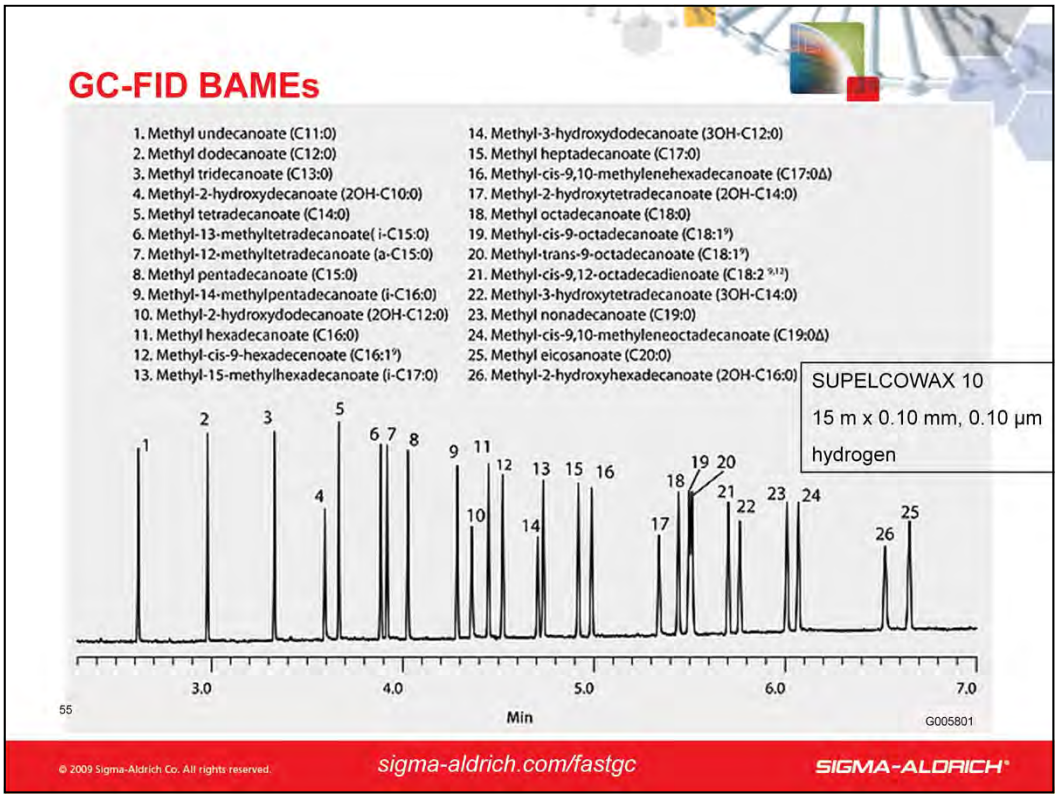
Peak IDs:
1. C18:1 Δ 7t and C18:1 Δ 6t
2. C18:1 Δ 9t
3. C18:1 Δ 11t
4. C18:1 Δ 12t, C18:1 Δ 6c, C18:1 Δ 7c and C18:1 Δ13t
5. C18:1 Δ 9c
6. C18:1 Δ 11c
7. C18:1 Δ 12c
8. C18:1 Δ 13c
9. C18:2 Δ 9t, 12t
10. C18:2 Δ 9c, 12t
11. C18:2 Δ 9t, 12c
12. C18:2 Δ 9c, 12c
13. C18:3 Δ 9t, 12t, 15t
14. C18:3 Δ 9t, 12t, 15c
15. C18:3 Δ 9t, 12c, 15t
16. C18:3 Δ 9c, 12t, 15t and C18:3 Δ 9c, 12c, 15t
17. C18:3 Δ 9c, 12t, 15c
18. C18:3 Δ 9t, 12c, 15c
19. C18:3 Δ 9c, 12c, 15c



Food-borne microbial pathogens can cause illness in humans. Each bacteria has a unique cellular fatty acid profile. Bacterial acid methyl ester (BAME) analysis is the determination of the cellular fatty acid profiles to assist with identification.

Conditions:

- column: Equity-1, 15 m × 0.10 mm I.D., 0.10 µm (28039-U)
- oven: 175 ° C, 30 ° C/min to 275 ° C (1 min)
- inj. temp.: 280 ° C
- detector: FID, 280 ° C
- carrier gas: hydrogen, 45 cm/sec constant
- injection: 0.5 µL, 200:1 split
- liner: 4 mm I.D., split, cup design
- sample: Bacterial Acid Methyl Ester (BAME) Mix (47080-U), methyl ester derivatives, total concentration of 10 mg/mL in methyl caproate

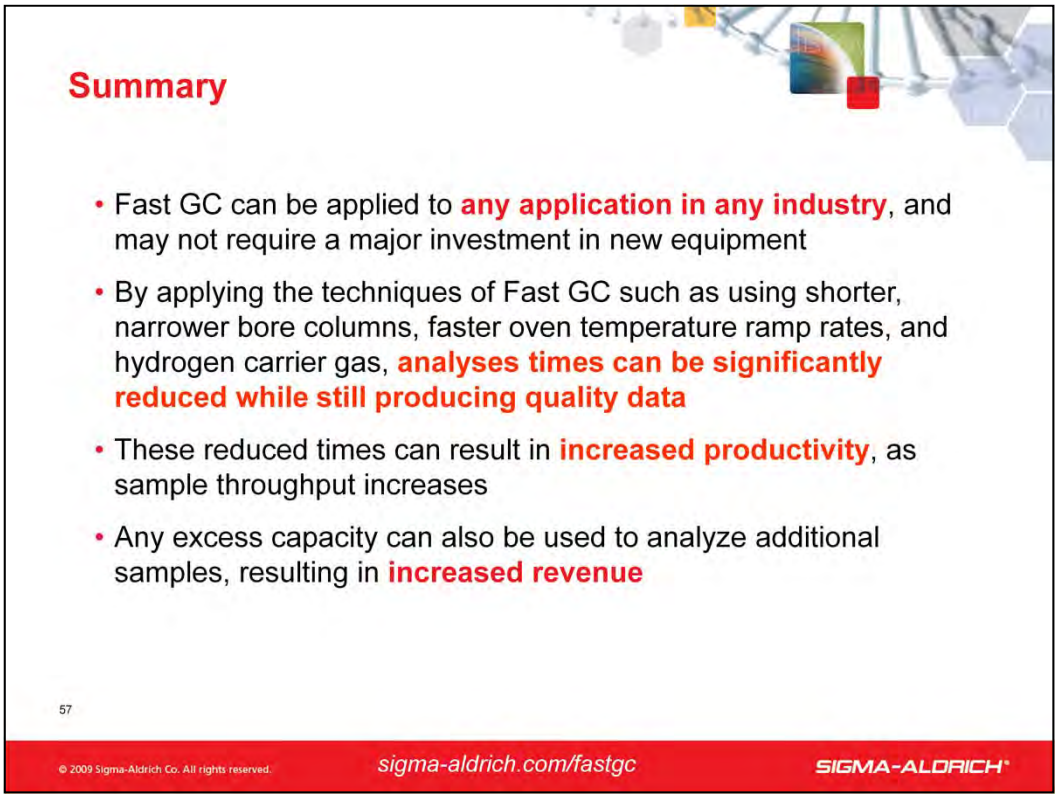


Food-borne microbial pathogens can cause illness in humans. Each bacteria has a unique cellular fatty acid profile. Bacterial acid methyl ester (BAME) analysis is the determination of the cellular fatty acid profiles to assist with identification.

Conditions:

- column: SUPELCOWAX 10, 15 m x 0.10 mm I.D., 0.10 µm (24343)
- oven: 80° C (0.3 min), 30 ° C/min to 180 ° C (0.5 min), 10 ° C/min to 200 ° C (1 min)
- inj. temp.: 250 ° C
- detector: FID, 250 ° C
- carrier gas: hydrogen, 45 cm/sec, constant
- injection: 0.1 µL, 200:1 split
- liner: 2 mm I.D., straight
- sample: bacterial methyl ester standards in methyl caproate, 10 mg/mL (47080-U)

Let's do a quick summary of Fast GC, and mention some helpful resources.



Fast GC can be applied to any application in any industry, any may not require a major investment in new equipment. By applying the techniques of Fast GC such as using shorter, narrower bore columns, faster oven temperature ramp rates, and hydrogen carrier gas, analyses times can be significantly reduced while still producing quality data. These reduced times can result in increased productivity, as sample throughput increases. Any excess capacity can also be used to analyze additional samples, resulting in increased revenue.

If you are looking for additional information, we have two great resources.

Visit our Fast GC web landing page at <sigma-aldrich.com/fastgc> to find over 75 chromatograms, as well as links to educational materials.

You can also request our comprehensive Fast GC brochure. It contains a theoretical discussion, practical considerations, a list of capillary GC columns offered in Fast GC dimensions, 26 chromatograms, and a nice listing of literature and references.

Thank you for your time today.