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Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part I: Impact of column dimensions.

August 25th, 2011 by Jaap de Zeeuw

When talking with customers, I always hear there is a lot of interest in doing faster GC. Often people think they have to buy new instrumentation to do that. Sometimes it's necessary if you have a 20 year old GC, but for most GC's of younger age, you can easily develop method to go faster. What you need to know is what your objectives are.

There are roughly 2 situations:

1 We have plenty of resolution between the components of interest. Here we trade some efficiency in favor for a faster analysis

2 We have "just" enough resolution. Here we need for the faster solution at least similar efficiency (plate number).

$$R_s = 1/4 \left[\alpha - 1 \right] \left[\frac{k}{1 + k} \right] \sqrt{N_{th}}$$

Fig. 1 generalized resolution equation

In the first case we can manipulate several parameters to reduce the analysis time. Let's first take a look at the column dimensions.

As we have plenty of resolution, we should be able to choose a column with less separation power. **The most easy is to use a shorter column length**. Looking at the resolution equation in fig.1, we see the resolution is linear to the square of the plate number. This means that when I reduce the column length by a factor 2, the resolution only reduces by a factor 1.4. The analysis time however reduces immediately by a factor 2.

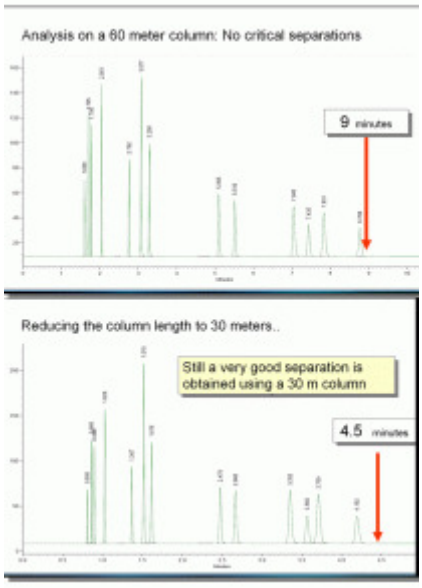

Fig.2 Comparison of analysis time of a 60 and a 30m column; Isothermal analysis on Rt Alumina BOND. The 30m is 2x faster.

Fig 2 shows the effect of length on analysis time using a 60 and a 30 m Rt Alumina BOND column. The alumina BOND is very selective for C1-C5 hydrocarbons, meaning that the components are all very well resolved. The analysis was done in the same GC, same carrier gas and the result shows a very simple reduction of analysis time by a factor 2.

By changing ONLY the column length, the only change we had to make was setting a lower inlet pressure. Very easy to implement for isothermal methods.

For temperature programmed methods, we have to adjust besides the inlet pressure, also the temperature program rate and the iso times. Reason for this is, that we like to have exactly the SAME separation. This only happens if the elution temperatures are kept the same.

If we reduce the column length by a factor 2, we need to **increase the program rate** by a factor 2 and **decrease the iso-times** by a factor 2. Example calculation is shown in figure 3. When we make these changes, we also will get exactly a factor 2 shorter run time while elution temperatures remain the same.

This can be done basically for all methods where there is plenty of resolution. The limitation is usually set by the maximum temperature program the oven can accommodate. Most GC's can program with 25-30C/min, but above 200C it becomes difficult. One can make the GC keep up with the higher temperature program rates by using a 220V OR using an oven –insert.. By reducing the oven size, the systems can heat up much faster.

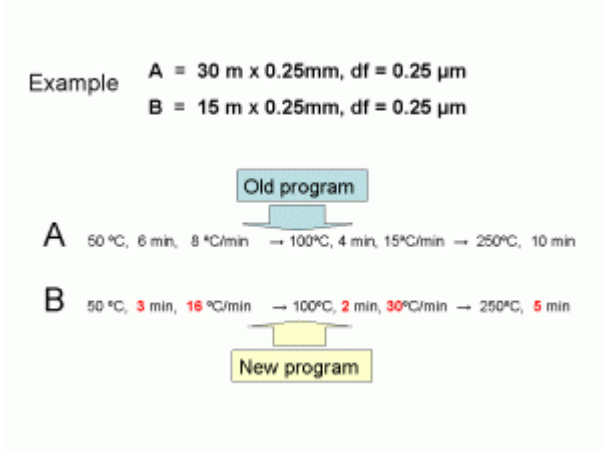

Fig.3 Example of new temperature program when replacing a 30m for a 15 m column: elution temperatures will be similar resulting in same chromatogram, just 2x faster

To produce less plates one can also use a wider diameter column of same or shorter length, or use a much shorter column with smaller diameter. Choosing the wider diameter in shorter length will also be an option, but we have to be more careful with adjusting pressures and oven programming-rate. The much shorter smaller ID column will be faster, but will also challenge the method on setting of operational parameters like pressures, program rates, injection speed and loadability. We will discuss this one later if we are going for speeding up situation 2.

Next time we will discuss how we manipulate run time using flow..

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Part II : Impact of higher column flow: http://blog.restek.com/?p=3376

Part III: Using faster temperature programming: http://blog.restek.com/?p=3414

Part IV: Using hydrogen as the carrier gas: http://blog.restek.com/?p=3520

Part V: Using Smaller bore capillary Columns : http://blog.restek.com/?p=3549

This entry was posted on Thursday, August 25th, 2011 at 1:12 pm and is filed under Faster Analyses, Optimizing Applications, Tips & Tricks. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

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Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part II: Impact of Higher Column Flow.

August 28th, 2011 by Jaap de Zeeuw

In our previous blog we discussed the use of a shorter column to reduce run time. We could do that because in our application we have plenty of resolution. This works very nice, but we have to cut our existing column in 2, or buy a new, shorter column. When analyzing challenging samples, like extracts of biological tissues or sediments, a shorter column usually will "age" faster. That means that we cannot do the same nr of analysis on a short column as we can expect on the long column. This should not be an issue as one can already benefit from a 2x faster analysis time and a lower purchase price.

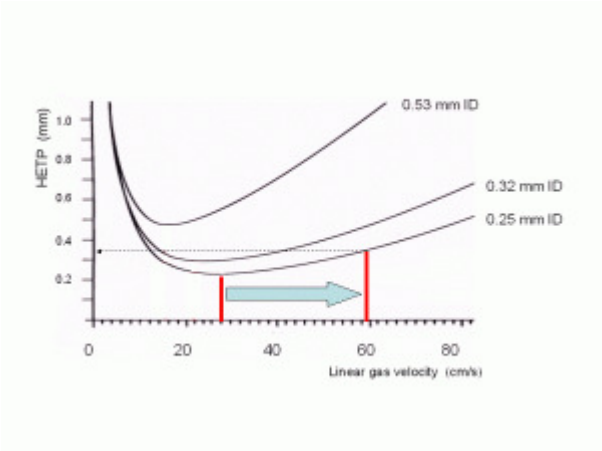

Fig 1: Van Deemter plot. Increasing velocity will cause some efficiency loss. No issue for "simple" separations

There is another way to speed up analysis and that is to operate the column under a higher flow. Now we are not replacing the column, we are only changing the linear gas velocity.

As shown in the van Deemter plot in fig. 1, operating a capillary at higher velocity will result in a loss of efficiency. That's exactly what we needed as we are still discussing situation 1 (see : http://blog.restek.com/?p=3333), where we have enough resolution and we like to speed up the separation at the cost of efficiency.

If we increase the linear velocity a factor 2, we loose efficiency, but the impact is lower then using a column of half the size.

It depends on the carrier gas. The loss of efficiency is the least using hydrogen, followed by helium and nitrogen.

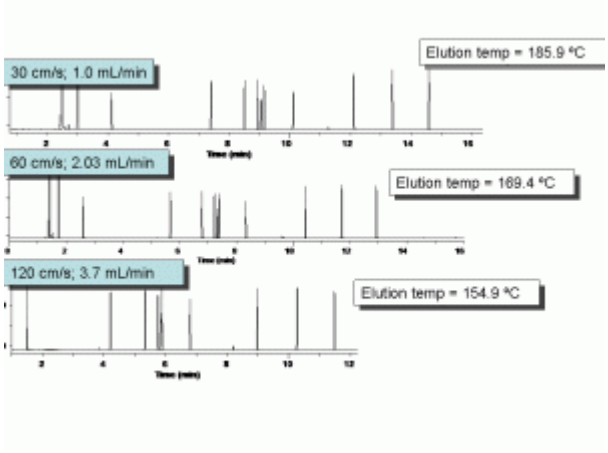

Fig.2 Impact of using Higher column flow rate (same temp program) 60ºC, 2 min → 250 ºC @ 10ºC/min

For isothermal analysis it is pretty straight forward we can reduce run time a factor 2 if we use twice the gas velocity.

In temperature programmed analysis we can also benefit from a factor 2 speed increase, but we have to change the temperature program to get the same elution temperatures. Figure 2 shows a separation of a test mixture where we have set the column at 30, 60 and 120 cm/s using the same temperature program. This is a practical mistake that is made quite often: because of using the same temperature program, we get little gain in analysis time. Here we win only 3 minutes. Additionally by using the same program with a higher linear velocity, the elution-temperatures will decrease, which result in relative peak shifting. If we zoom into the area where we have more peaks eluting, we observe that peaks start to shift relative from each other, see Fig. 3.

This effect will always happen when we change conditions that affect the elution temperature.

If we adjust the program also , we get results as shown in figure 4. By using a faster temperature program we can also reduce analysis time with the same factor as we used to increase the linear gas velocity.

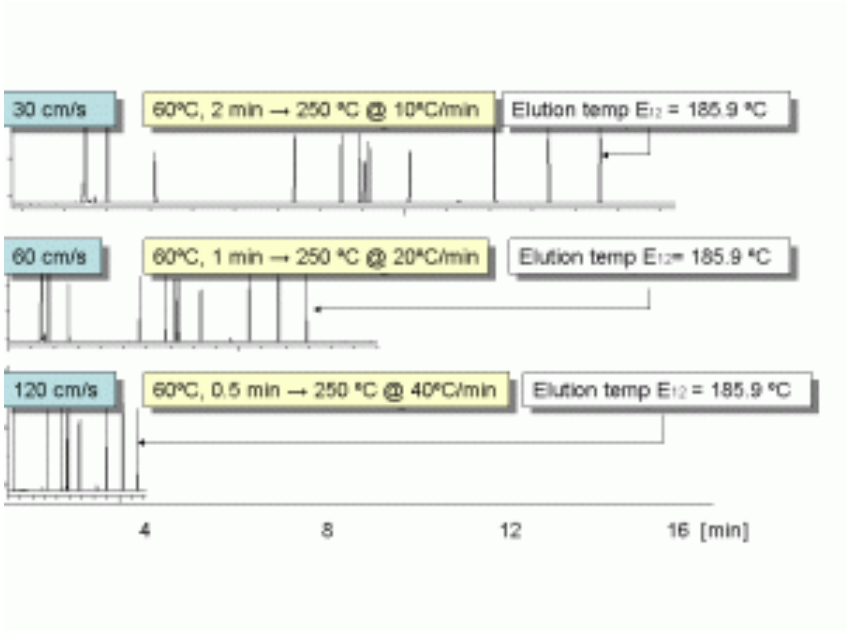

Fig. 4 Temperature programs needed to get the SAME elution temperature: Now the run times are also much shorter

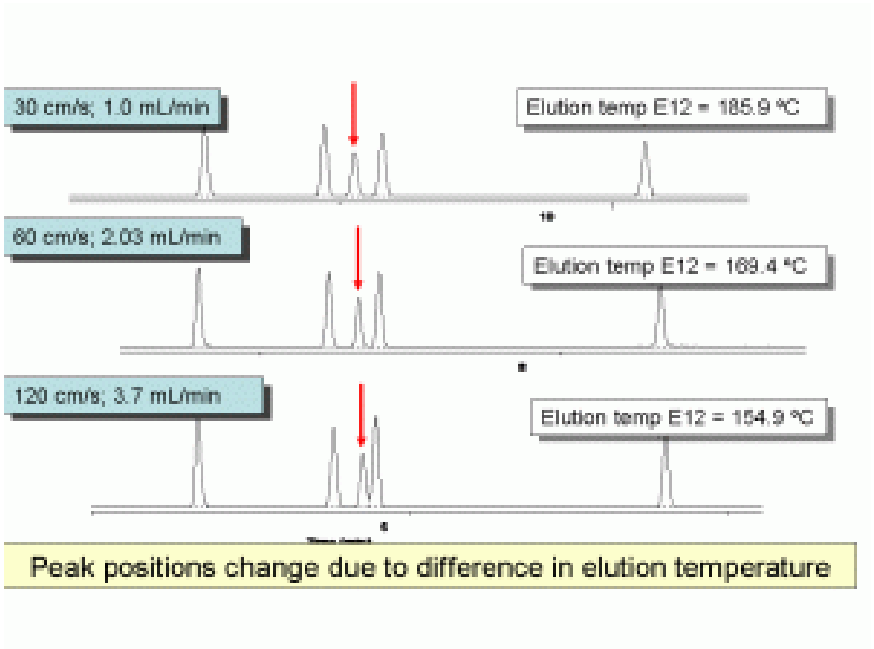

Fig.3 Peak positions change due to difference in elution temperatures

How much separation do we loose?

We only started to do this exercise for separations where we had plenty of resolution. We did an analysis of a complex sample (perfume eternity) on a 30m x 0.25mm Rxi 5Sil MS, using linear velocity of 60 cm/s.

Then we did the same analysis at 2x higher linear velocity, 120 cm/s.

Figure 5 shows the result. Peak elution profile is very similar. Analysis time was a bit longer because the 6890 GC oven could not keep up with the 40C/min temperature program. Fig 6 shows an expansion of a "crowded area". Here we indeed see we have lost some efficiency, but this was also to be expected.

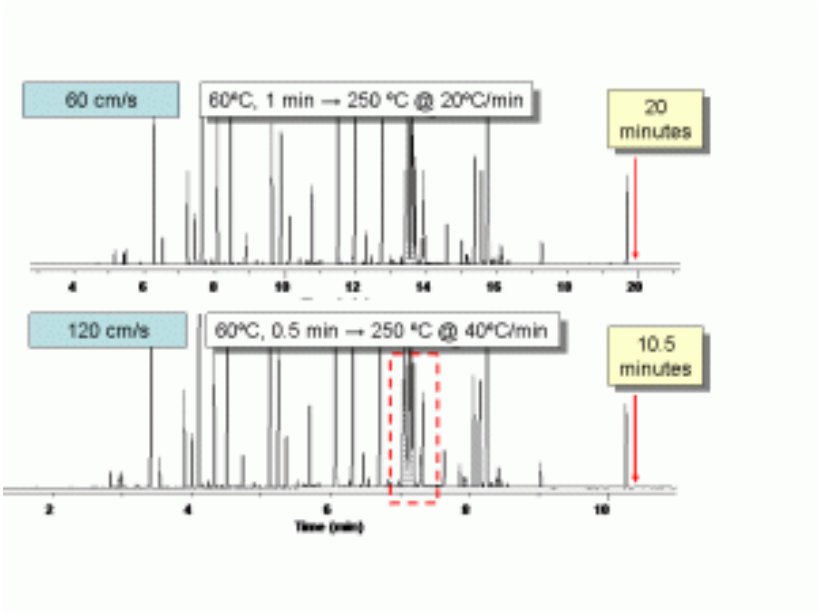

Fig.5 Perfume analysis on Rxi-5Sil MS, 30/0.25/0.25 at 60 and 120 cm/s

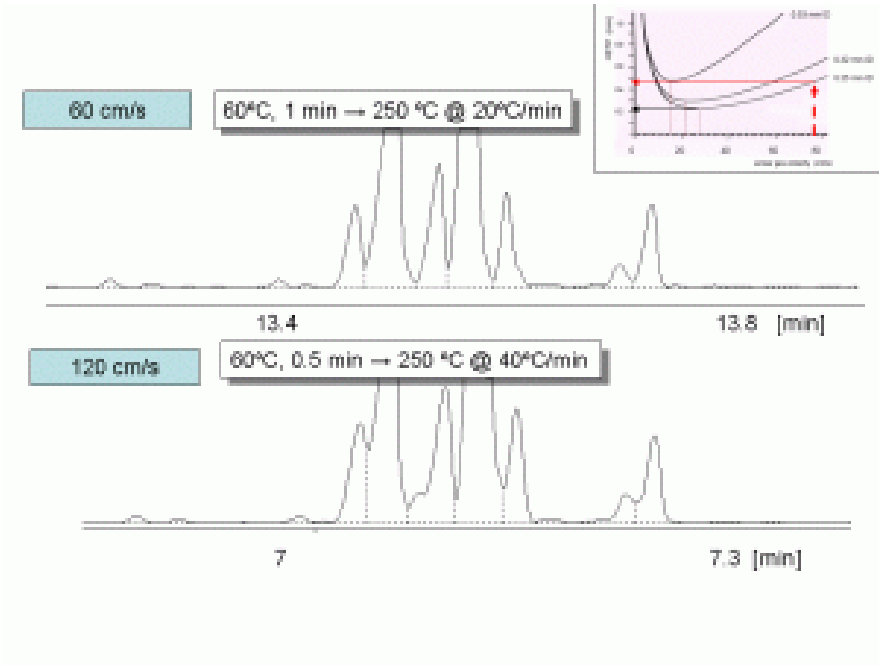

Fig. 6 Detail of peak-cluster from fig. 5

The temperature program for the faster method depends on the increase in gas velocity. In formula, see fig. 7. Fig 8 gives an example calculation.

Interesting advantage of using higher linear velocity, is that eluting peaks will be higher which benefits sensitivity. We can inject less onto the column by reducing sample volume, extra sample dilution or operating at a higher split-ratio. This all will result in increased life time as less contamination will be brought on to the column.

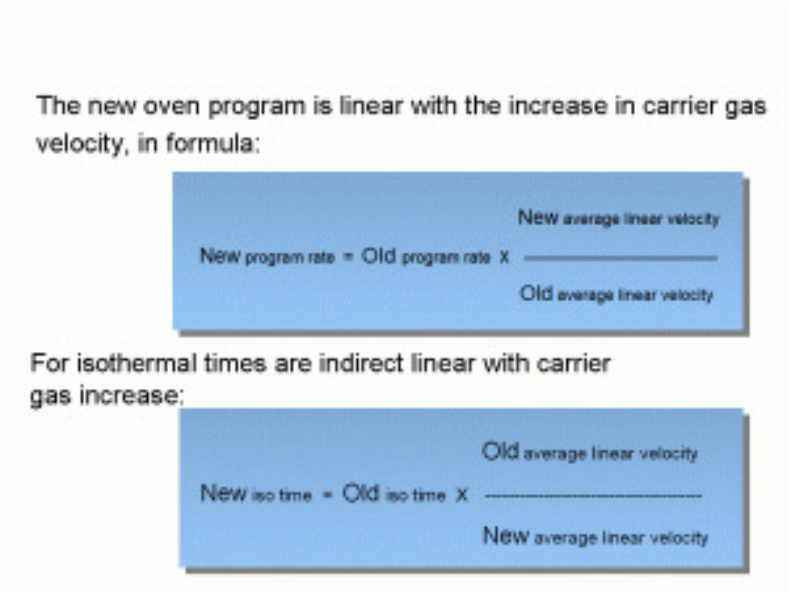

Fig.7 To get the same elution temperatures we have to "calculate" the oven program rate and the Iso-times. (Iso temperatures must remain the same)

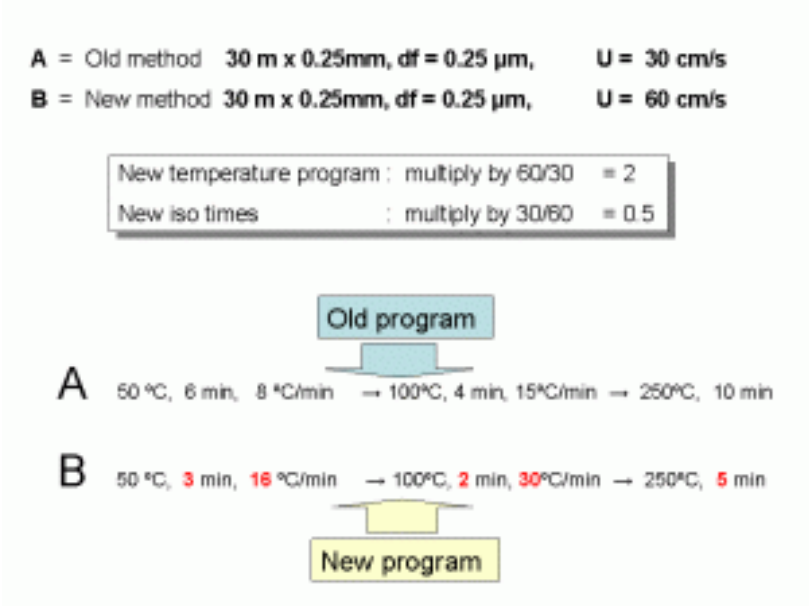

Fig.8 Example of calculation for a 30m column moving from 30 to 60 cm/s

Additionally, if the column ”ages” and efficiency is decreasing, one can decide to operate the column more optimal at a lower velocity and still get the separation.

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2 Responses to “Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part II: Impact of Higher Column Flow.”

1. *jaap* says:
August 29, 2011 at 8:27 pm

   For FID detection this would work very nice. In mass spectroscopy there are sometimes restrictions as max flow may be limited due to pump capacity. Ion traps usually do not like high helium flows. There are however also more quadrupole systems available that deal with higher flows. Few months ago I saw the Shimadzu MS-system launched that could deal with 15 mL/minute and had no problem measuring very narrow peaks. This should allow also the high flow advantage using quadrupole MS systems. I understand most TOF systems already can work with higher flows..

   Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part III: Impact of using Faster Temperature Programming « ChromaBLOGraphy: Restek's Chromatography Blog says:
September 4, 2011 at 10:47 am

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Is the BP Gulf Oil Well Still Spilling? »

Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part III: Impact of using Faster Temperature Programming

August 30th, 2011 by Jaap de Zeeuw

In our previous blogs we discussed the use of a shorter column and higher gas velocity to reduce run time. For both approaches we had to adjust our oven temperature program to benefit maximal in time and to get similar peak elution order.

Sometimes we can also change the oven temperature program using the same column and the same carrier gas velocity.

For instance, if we have a chromatogram where we have no peaks of interest in the first section of the chromatogram (after the solvent has eluted), we can safely ramp the oven to a higher temperature and start the programming from there. Also to elute late, non-interesting peaks, we can "bake-out" the column by running it to a higher temperature. In this case the HT phases are especially of interest as they can be used up to 380/400C. (I would also add a flow program to speed up elution).

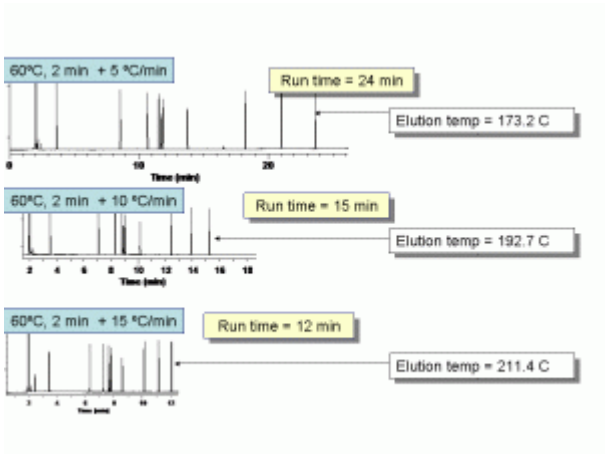

Fig. 1 Enough resolution: using a faster temperature program at the same linear gas velocity; 3x higher temperature program reduces run time a factor 2, but elution temperature increases with more then 30C.

If the temperature program is deliberately changed while keeping other parameters constant, we will get a faster analysis, but also different elution temperatures. Fig.1 shows what happens with the analysis time when I use a program rate of, 5, 10 and 15 C/min. **By increasing the program rate 3 times, the elution time reduces a factor 2**. The elution temperature for the components is now at least 30 ºC higher. For simple mixtures this is no problem at all, but if there are compounds present with a different polarity, there may be a bigger impact. Example is shown in fig.2 where pesticides are separated on a highly selective phase, the Rtx-Cl-Pesticides.

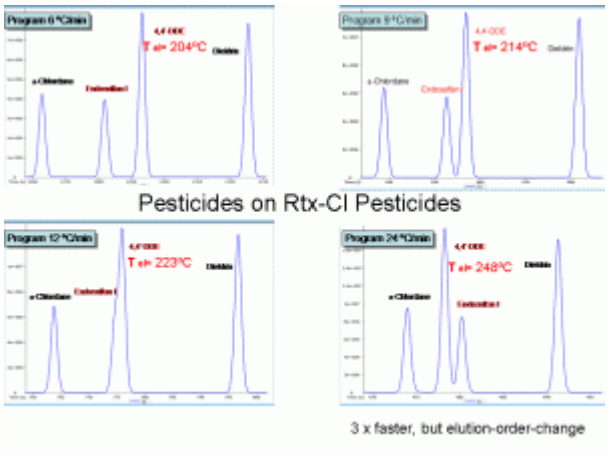

Fig. 2 Peak elution order change of pesticides using different temperature programs. Because elution temperature changes, the peak elution changes.

Because this phase is designed to be very selective for chlorinated pesticides, it is also polarizable by temperature. This means that peak elution can be tuned by using different elution temperatures, which can be achieved using a different flow, a different program rate or a combination.

It's clear to see that with increased temperature program rate, the elution temperature of 4,4'-DDE is increasing. This makes this component elute relative faster compared to the Endosulfan I. Programming with 12ºC/min makes both peaks co-elute, but using a program of 24ºC/min, the 4,4'-DDE elutes BEFORE the Endosulfan I and we get full base line separations and .. **a 3 times shorter run time!**

One needs to be aware that sometimes we cannot operate GC oven at very high ramp-rates, simply because the "real" oven temperature cannot keep up with the "set" value. Fig. 3 shows example of 7 repeated analysis of pesticides using 20, 30, 40 and 50 C/min. temperature programs. Conditions are listed in fig. 4.

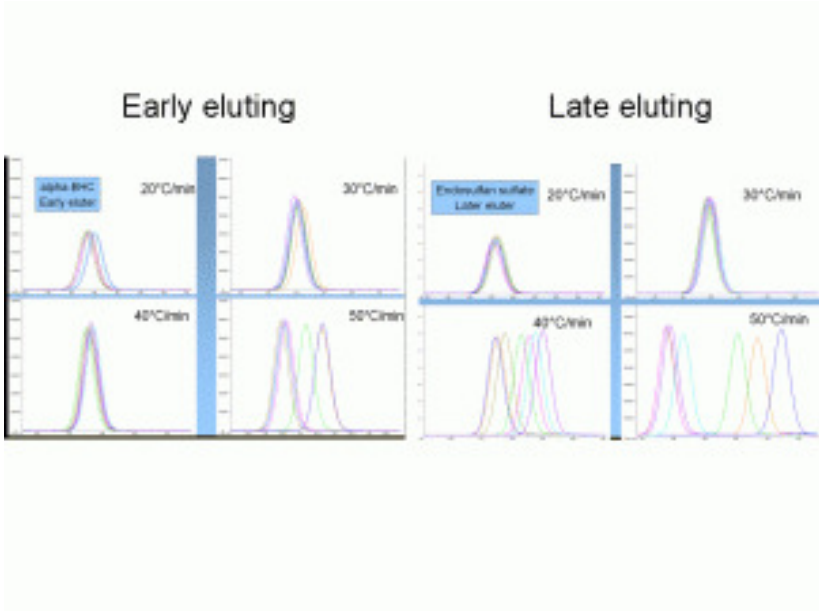

Fig. 3 Impact of programming rate on retention time reproducibility. If “real” oven temperature is not matching the “set” values, retention times will show higher variation. This will be observed especially for late-eluting compounds

GC	: Agilent 6890, 110 V, No oven insert
Injection	: Split, 1 µl, fast injection
Liner	: 4mm liner with wool, Siltek deactivation
Inject. Temp	: 250°C
Split ratio	: 50:1
Column	: 20m x 0.18mm x 0.18µm Rtx-CLPesticides
Carrier gas	: H_2, Constant flow, 0.9 mL/min.
GC oven	: 80°C (1 min), 20, 30, 40, 50°C to 320°C Final hold up to 2 min

Fig.4 Conditions used for oven performance experiment

For the early eluting pesticides, like alpha BHC, retention times are reproducible up to programs of 40ºC/min. The later eluting pesticides, like endosulfan, are eluting under conditions where the real oven temperature cannot meet the set values. As a result there is more deviation on retention times which allows only a max program speed of 30 ºC/min.

If one wants higher speed programming, you have to look at the specifications of the GC. The 220/240V

program speed, there are oven-inserts available. By reducing the oven size, the speed of programming can be increased considerably.

Another development that is related to the faster programming, are the direct heating systems, see fig. 5. Columns are heated directly by an electric heating wire(resistor). Some systems use the Restek MXT – metal columns and direct apply a voltage. This allows temperature programs up to 1000ºC/min. to be controlled. Such systems will produce very short run times. Example in Fig.6 shows a simdist application on a Rtx-5 column, which was realized within 2 minutes. Such high temperature programs will have big impact on peak elution order, which always needs to be verified. Eluting peaks will also be very narrow. (0.1-0.2sec is not unusual), for which the detection systems needs to be able to produce enough data points.

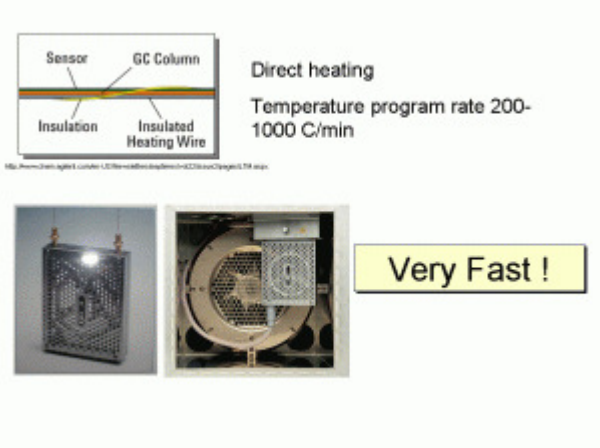

Fig.5 Direct heating modules: shown Interscience (Thermo) Ultra Fast GC

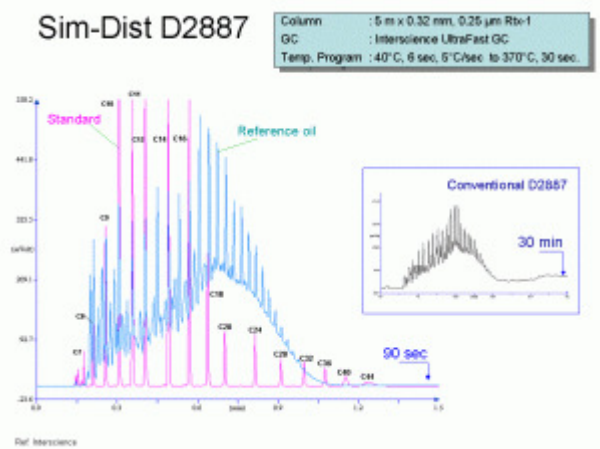

Fig. 6 Direct heating allows very short run times. Conventional methods can be 15x faster

Direct heating systems are good for simple applications where the sample matrix is known. Maintenance can be more challenging as one cannot cut a piece of the analytical column, so one needs to replace the whole unit or work with guard column make extra couplings.

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One Response to "Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part III: Impact of using Faster Temperature Programming"

> *Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part II: Impact of Higher Column Flow. « ChromaBLOGraphy: Restek's Chromatography Blog* says:
> September 4, 2011 at 10:50 am

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Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part IV: Using Hydrogen as the Carrier Gas

September 2nd, 2011 by Jaap de Zeeuw

In our previous blogs we discussed solutions to reduce analysis time in situations where we had excessive resolution. Use of shorter columns, higher flow and faster programming allowed serious faster GC using existing instrumentation

Now we will move into a different situation and that is where we have "just enough resolution" to do the separation. Example chromatogram is shown in Fg.1. Any reduction in resolution will immediately affect the separation of the components. In this situation, we have only 2 options to choose from:

1: use hydrogen as the carrier gas;

2: use a shorter column that produce a higher efficiency;

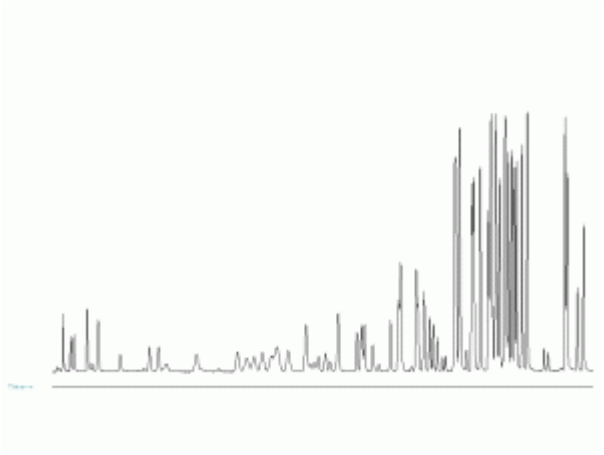


Fig. 1 Example of a complex chromatogram.
Any loss in efficiency will immediately result in
a separation challenge

The most easy way to reduce analysis time is to change the carrier gas. By using hydrogen instead of helium, we can benefit from the higher optimal linear velocity, which is a factor 2 higher, see fig. 2. At Restek, we test all our columns with hydrogen as the carrier gas, which saves us 50% on the investment in gas chromatographs.

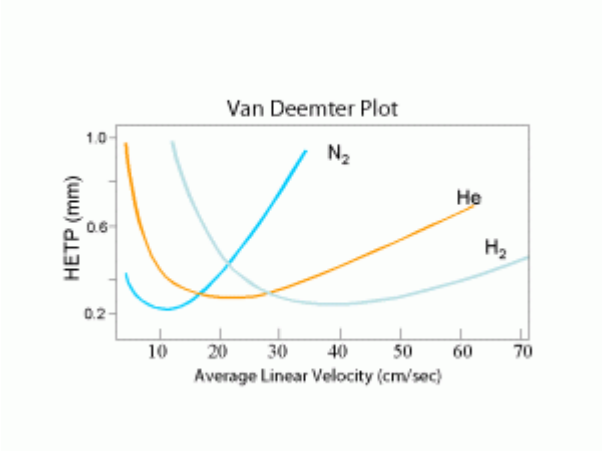


Fig. 2 Van Deemter curve for different gases.
Hydrogen is 2x faster then helium

By changing to hydrogen we not only benefit form a faster analysis, we also benefit from a higher response. As the peaks elute 2x faster, the height will be 2 times higher. For a similar signal/noise, one can inject 50% of the sample, from which we can benefit in less contamination of liners and column inlet. Fig 3 shows an example. The pressures for using hydrogen are the same as we use with helium. Due to the lower hydrogen-viscosity, we get approximate the double linear velocity.

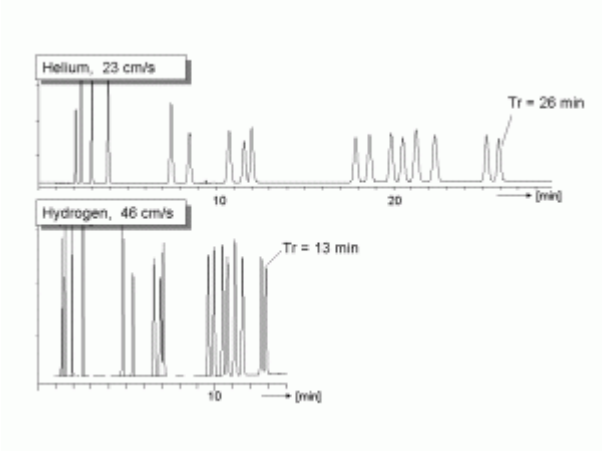


Fig. 3 Using hydrogen instead of helium does the same analysis in half the time and peaks are 2x higher..

For isothermal analysis the conversion is pretty straightforward. For temperature programmed analysis using hydrogen, we have to change the temperature program, to get the same elution temperatures, see fig.4. (this will give us also the factor 2 shorter run time and also makes sure that the peak elution order will not change). The change in temperature program is similar as we had to apply when we run the column at a higher gas velocity using the SAME carrier gas.(see: http://blog.restek.com/?p=3376). Only now, using hydrogen, we maintain the efficiency.

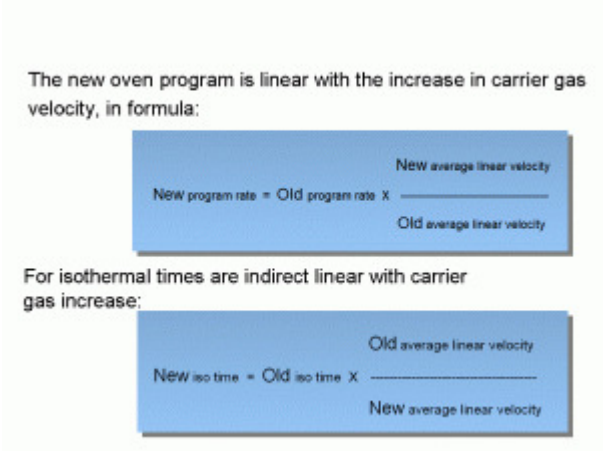
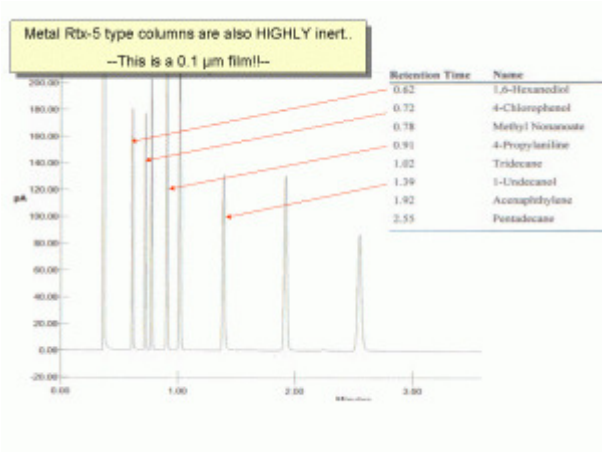


Fig.4 To get the same elution temperatures using hydrogen, we have to "calculate" the new oven program rate and the new Iso-times.

One of the biggest concerns is, that hydrogen is combustible. Indeed, it is combustable over a concentration range of 4% to 74% by volume, but we have to put this in perspective.

1. **The risk** for building up these levels is reduced because of the enormous fast diffusion (dilution).
2. **Many labs already have a hydrogen gas line** in place, used for fueling the FID. One can also use Hydrogen generators. These produce a relative small amount of hydrogen;
3. **The GC's have nowadays digital flow controls.** If one set a flow controlled carrier gas supply, it is impossible to have a large amount of hydrogen released in the oven. As soon as the column breaks, the pressure will be gone(its "flow" controlled), and the max. amount of hydrogen entering the oven is the injector volume and the actual hydrogen flow. It will be very difficult to even reach the 4% level;
4. **One can also buy hydrogen detection system**s that you can install in the GCs. These systems use sensors that measure the air taken from the oven on hydrogen presence. If hydrogen is detected, the oven can be shut off and often an alternate gas can be turned on to protect the column.
5. **To reduce the risk to a minimum one can also use metal columns**, like the MXT series You will be surprised how inert a metal column is. Example shown in figure 5 shows a 0.1 micron of a Rtx-5-coated MXT column. Test compounds are highly polar, as well as acidic and basic. All components elute with impressive peak symmetry. Restek developed and commercialized this series with a range of stationary phases.



MXT column with only a 0.1 micron film..
Shows the unique quality of the Siltek deactivation

Hydrogen can be used with all injection systems. For safety reasons, the split vent is usually vented. In the early days when we used hydrogen. we always made a little "torch" on top of our GC's and we just "lit" the hydrogen. As this is considered "open fire" it is not allowed in today's labs.

The effects of using Hydrogen on detection systems is another concern. For most detection systems like FID, ECD, TCD, FPD, PFPD, SClD, I am not aware of issues. Of course, with FID and high column flows, one has to adjust the hydrogen feed of the detector. However for MS the use of hydrogen is not very clear, see also: http://blog.restek.com/?p=3212. Detectors like the NPD (the beads) do not seem to like hydrogen. Also PDD(HID) and ion traps need helium.

If you have any experiences using hydrogen, positive or negative, please share!

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Part III: Using faster temperature programming: http://blog.restek.com/?p=3414

Part V: Using Smaller bore capillary Columns : http://blog.restek.com/?p=3549

This entry was posted on Friday, September 2nd, 2011 at 9:25 am and is filed under Faster Analyses, Optimizing Applications, Tips & Tricks. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

4 Responses to "Fast(er) GC: How to Decrease Analysis Time using Existing Instrumentation? Part IV: Using Hydrogen as the Carrier Gas"

1. *Lars Kürstein* says:
   September 2, 2011 at 11:54 am

   Dear Jaap -

When using hydrogen as carrier gas in gas chromatography or mass spectrometry, you probably have to investigate for undesirable sample matrix effects as well? In the catalyst business, where I am working, we know that hydrogen and hot surfaces (injection ports), under certain circumstances, not is the best enviroment for some chemicals.
I am just wondering … when analyzing alkenes, alcohols, other oxygenates or components containing sulphur, nitrogen or chloride ….. what happens with theese components in a hot injection port together with lots of high pressure hydrogen?

With kind regards -
Lars Kürstein

2. *Jaap de Zeeuw* says:
September 4, 2011 at 2:51 pm

Hi Lars,
In all the user-contacts I had, I have never heard about a reaction occurring using the hydrogen.

I agree that a "hot" injection port with a catalyst present, something could happen. This will be a time/temperature relation. With slitted injection the time is fraction of a second; Splitless injection may be more challenging. Also here, I have only come across decomposition with some pesticides, brominated flame retardants , carbamates etc. To reduce reactivity with hydrogen, we need to make sure that liners/(and wool) are clean and well deactivated.

n the column side: When we developed alumina PLOT columns, we expected a possible problem using the alumina and hydrogen. We could not proof any reactivity up to 200C.

regards
jaap

3. *Lars Kürstein* says:
September 5, 2011 at 9:22 am

Dear Jaap -

Thanks for your reply!

I am just wondering a little again :-) ….. What about polar stationary phases and hydrogen as carrier gas? Could you imagine hydrogen to be "aggressive" to some other polar stationary phases, e.g. PEG stationary phases?

With kind regards -
Lars Kürstein

4. *Jack Cochran* says:
September 5, 2011 at 12:24 pm

Hi Jaap:

First, I want to agree with Lars and thank you for the wonderful tutorials on fast GC. These are

I'm glad to see you suggesting the use of hydrogen as a carrier gas. Even to this day, it is a completely under-utilized carrier gas in the US, and with the helium shortage, it really surprises me more people haven't moved to it, especially considering you can actually generate hydrogen, unlike helium (unless you have a sun in your basement).

Hydrogen can indeed promote undesirable reactions in GC inlets, especially if there are metals present in the liner, even trace amounts. Aviv Amirav reported on this in a nice article (I don't have the cite with me now, but will get it later). I don't think inlet time, even in split injection, is so short that this couldn't happen, as gas phase reactions occur very, very quickly. I know, for example, that when using hydrogen carrier in a mass spectrometer multiple years ago, that I saw very unusual mass spectra for hexachlorocyclohexanes (HCHs), essentially where they were dominated by 78 m/z ion eventually, an ion they normally don't even have in their spectra. The 181 and 219 ion clusters were essentially gone. Other chlorinated pesticide spectra were also affected; PCBs were NOT, to any appreciable extent (and I've even used hydrogen in an ion trap MS for PCB work). What was really strange was that the HCH spectra took almost a week to return to normal even after helium was restored as the carrier gas. I wondered if somehow H2 was trapped in the ceramics of the source somehow and continuing to cause the spectral issues through surface reactions. OK, that's a bit crazy, but what could it be?!

Thanks again, Jaap!

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Milk Matters – Using QuEChERS extraction approach with GCxGC determination »

Fast(er) GC: How to Decrease Analysis Time Using Existing Instrumentation? Part V: Using Smaller Bore Capillary Columns

September 6th, 2011 by Jaap de Zeeuw

If the use of hydrogen is not an option for speeding up, and we cannot afford to loose any efficiency, the only option we have is to use a shorter column with a smaller diameter.

The efficiency is proportional with decreasing column diameter, meaning that a column with 2x smaller diameter can be 50% shorter and will deliver exactly the same number of theoretical plates.

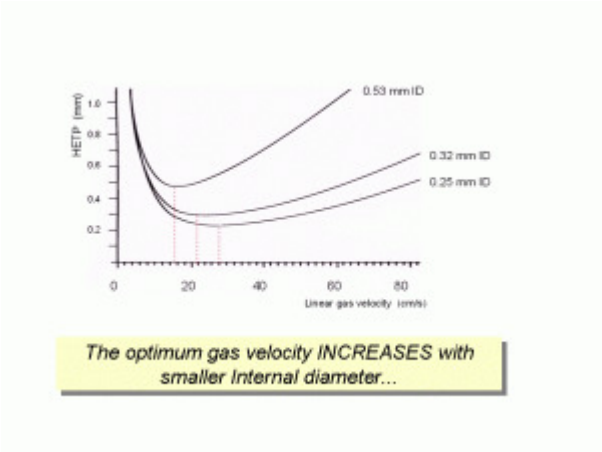

Fig. 1 Influence of column diameter on optimal gas velocity and HETP: The smaller the diameter, the higher the optimum linear velocity

Most widely used are the 0.25mm and 0.32mm ID columns. In order to speed up the analysis, already 25 years ago, the 0.1 mm ID columns were developed and commercialized. Compared with a 25 m column, they showed comparable efficiency and analysis time could be up to 3x shorter. This was also due to the higher optimum linear gas velocity for the smaller bore columns, see fig. 1. The generations of GC's, like 6890) were all developed to accommodate the application 0.1mm columns. Pressures are much higher, gas and oven controls must be more accurate and also the detector sample rate had to fast enough to measure the narrow peaks produced by the 0.10mm.

Practically the use of 0.10mm columns did not meet expectations for many, as these columns have limitations.

1. For compositional analysis where we can use high split-ratio's, the columns work fine. For trace analysis, where we have to use splitless injection, the story is different. In Splitless injections, the liner volume must be transferred on to the column. Column flow in a typical 0.1 is very low, Helium flow at a velocity of 30 cm/s at the outlet is 0.3 mL/min. As the gas is under a pressure of 217 kPa at the inlet, it is compressed a factor 3. That means that the volumetric flow at the inlet is only 0.1 mL/min. To transfer the full liner volume in a splitless injection, will take considerable time. This adds to analysis time but also impacts injection volume. Therefore a "pressure pulse" has to be considered.
2. Sample capacity is very low. The average 0.1 mm column can be coated with max 0.2-0.4 micrometer film. Injection of 5 ng will often already show signs of peak skewing.
3. Using 0.1mm columns, we have to work with relative high inlet pressures: The risk for septum leaks/discrimination will increase, especially with huge pressure pulses;
4. Because columns are very short, for optimal results very fast temperature programs are required. Ovens do have limitations there as max programming rate is dictated by oven size and design.
5. Use of MS is not always possible. Ion traps need a certain minimal flow. Also the eluting peaks can be <0.5 seconds in width. We need enough data collection speed. Newer MS systems will meet this.
6. Because of small ID and thin film, the 0.1mm ID columns need more frequent maintenance as the column inlet will contaminate faster. Guard columns play a bigger role.

If all conditions are considered properly, one can do fast GC using the 0.1 mm columns. Fig. 2 shows a semi-volatiles analysis in only 5.5 minutes using a 10m x 0.1 mm Rxi-5Sil MS column.

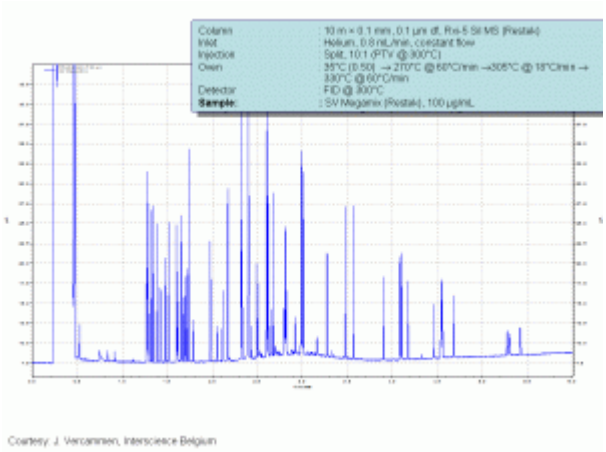

Fig. 2 Semivolatiles in les then 5.5 minutes using
10m x 0.1mm capillary with 0.1 um Rxi-5Sil MS

Many of the issues listed above could be overcome by using columns of 0.15mm ID. This diameter capillary seems to provide a very practical balance between all common column parameters. The reduction in run time we can achieve using 0.15mm, is a factor 2.

Instead of a 30m x 0.25mm, we use a 20m x 0.15mm. The efficiency of a 20m x 0.15mm is about 10% higher then the 30m x 0.25mm. By length only, we will be able to run 66% faster if we would use the same gas velocity. Because we have 10% higher efficiency we will operate the 0.15mm column at a 30% higher velocity (50 cm/s instead of 36 cm/s). By doing this, we will loose some efficiency, but that's acceptable as we end up with similar efficiency as the 30m x 0.25mm, but with 2x shorter run time.

For this conversion, we ideally must use columns with the same phase ratio (beta). A 0.25µm film in a 0.25mm ID column must be replaced by a 0.15µm film in a 0.15mm ID column.

Column 1 : Original column used

Column 2 : new column for faster method

$$\text{New Temp Program (2)} = \text{Old Temp. program(1)} \times \frac{\text{Length column 1}}{\text{Length column 2}} \times \frac{\text{Gas velocity 2}}{\text{Gas velocity 1}}$$

$$\text{New Iso Times (2)} = \text{Old Iso Times (1)} \times \frac{\text{Length column 2}}{\text{Length column 1}} \times \frac{\text{Gas velocity 1}}{\text{Gas velocity 2}}$$

Fig 3. Formula for calculation temp. program and iso-times to get the SAME elution temperatures. Valid for columns having the SAME phase-ratio (Beta)

As we have seen in the previous blogs, when we change column length and linear gas velocity, we need to set a different temperature program, to get similar peak elution order. (we need the same elution temperatures). Fig 3 shows an easy calculation to do that. This is generic calculation as compressibility of gases is not included. Dr. Leonid Blumberg has done a great job making software for such conversions. (available as free-ware from the web).

Fig 4 shows a complex perfume analysis, where we converted the analysis from a 30m x 0.25mm to 20m x 0.15mm column. Conditions are listed in fig.5. We get similar peaks sequence, but 2x shorter run time.

OLD METHOD

Column	: Rxi-5Sil MS, 30m x 0.25mm, df = 0.25µm
Carrier	: H2, 1.2 mL/min, u = 36 cm/s constant flow
Spit inject	: 1:100; 1.0 µl
Oven	: 100°C, 2 min, 5°C/min → 250 °C
GC	: Agilent 6890

NEW METHOD

Column	: Rxi-5Sil MS, 20m x 0.15mm, df = 0.15µm
Carrier	: H2, 0.5 mL/min, u = 50 cm/s constant flow
Spit inject	: 1:100; 1.0 ul
Oven	: 100°C, 0.9 min, 9.75°C/min → 250 °C
GC	: Agilent 6890

Fig. 5 Conditions for 30m x 0.25mm and 20m x 0.15mm Rxi-5Sil MS columns. The 0.15mm column is operated above its optimum linear velocity

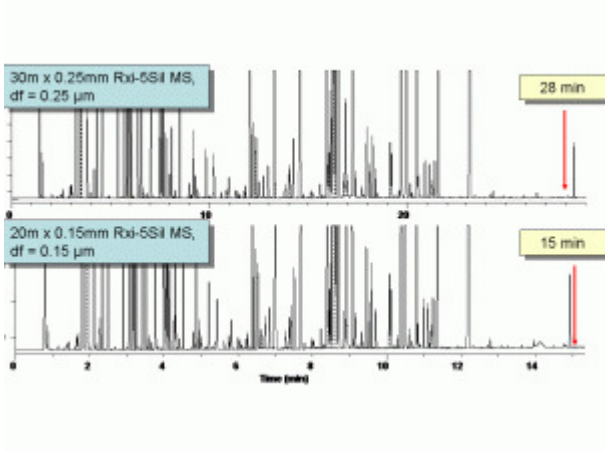

Fig.4 Analysis of Perfume" Eternity Moment" on 2 systems with comparable efficiency. The 20m x 0.15mm column is 2x faster

Interesting detail we also should mention is, that the peaks from the 0.15 mm column will be 2 x higher. We can use that for sensitivity, but better may be, to inject only 50% of the sample.. By doing that, we contaminate our system also 2x less meaning we can do twice the number of analysis before maintenance..

If the sampler cannot inject 0.5 µl (instead of 1 µl), you may consider to dilute the sample 1:1 and still get the benefit.

Related blogs on fast(er) GC :

Part I : Impact of column dimensions: http://blog.restek.com/?p=3333

Part II : Impact of higher column flow: http://blog.restek.com/?p=3376

Part III: Using faster temperature programming: http://blog.restek.com/?p=3414

Part IV: Using hydrogen as the carrier gas: http://blog.restek.com/?p=3520

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