Search

# Archive for the 'Faster Analyses' Category

« Older Entries

#### Using the Restek EZGC Method Translator to Assist in GC Column Trimming Maintenance

Monday, August 25th, 2014 by Jack Cochran

It is well known by people who analyze environmental and food safety matrices for semivolatile organic compounds like pesticides and PAHs that you occasionally have to trim the GC column to restore peak shapes degraded by nonvolatile matrix material that builds up on the inlet side of the column. (As an aside, <u>change that liner and seal</u>, too!) What gets a bit murky is how you change the GC conditions after trimming to maintain the same elution orders for peaks of interest. Consider using our new <u>EZGC<sup>TM</sup></u>Method Translator and Flow Calculator (MTFC).

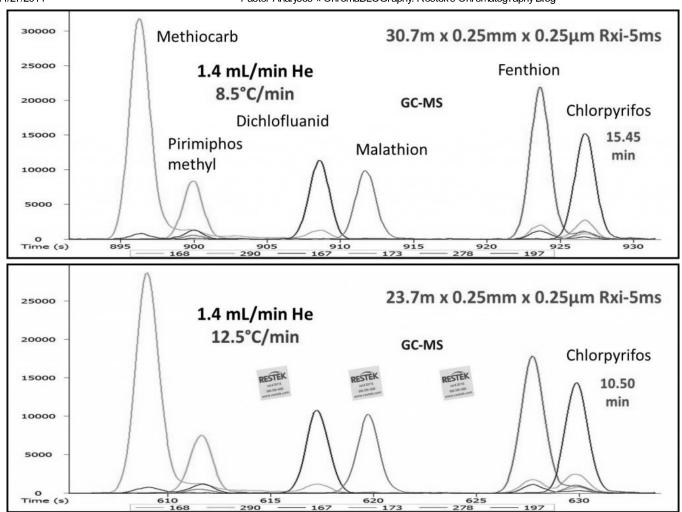
In the example below, I've made three (exaggerated) trims on a GC column that had an original nominal length of 30m, our 30m x 0.25mm x 0.25mm x 0.25mm x 0.25mm, which resulted in a column length of 23.7m. Using the MTFC, enter the Original column dimensions (determine the original length accurately using MTFC, too!), and the original GC conditions. Keeping the flow rate the same, enter the Translation column length, and simply watch MTFC spit out the updated oven program rate for you, which it will do when you have "Translate" selected under Results. Note that the 30.7m and 23.7m chromatograms look almost identical, except that the 23.7m run is faster due to the increased oven program rate necessary to keep compounds eluting at the same temperature (the thing they need to do to avoid elution order flip-flops).

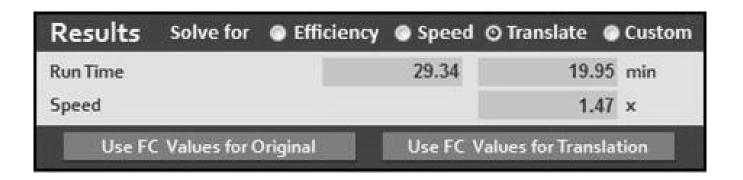
That faster run creates a bit of a problem, the need to update any Selected Ion Monitoring / Selected Reaction Monitoring windows and/or Calibration Table Expected Retention Times. Again, MTFC to the rescue! Just use the "Speed" factor in Results to calculate the expected retention times, as follows:

Predicted retention times for Translation = Actual Original retention times divided by "Speed".

The table below indicates the calculation above works very well. Now, just cut and paste the new retention times into the method and calibration areas and keep using that same column and save money.

If you want to follow along with this example, the Original Method and the Translation Method are:
30.7m x 0.25mm x 0.25 mm Rxi-5ms, 1.4 mL/min constant flow He, GC-MS, split injection  Oven: 90°C (0.1 min), 8.5°C/min to 330°C
——————————————————————————————————————
$23.7m \times 0.25mm \times 0.25 \mu m$ Rxi-5ms, 1.4 mL/min constant flow He, GC-MS, split injection
Oven: 90°C (0.1 min), 12.5°C/min to 330°C





# Using Method Translator Speed Factor for Retention Time Update

- Actual retention times (t<sub>R</sub>s) from 30.7m column
- Actual t<sub>R</sub>s from 23.7m column
- Method Translator (MT) predicted t<sub>R</sub>s from 23.7m column
- Difference (Diff) actual and predicted t<sub>R</sub>s

Pesticide	t <sub>R</sub> 30.7m	t <sub>R</sub> 23.7m	MT t <sub>R</sub> 23.7m	t <sub>R</sub> Diff
Dichlorvos	5.72	3.85	3.89	-0.04
o-Phenylphenol	9.48	6.42	6.45	-0.03
gamma-BHC	12.79	8.68	8.70	-0.02
Vinclozolin	14.31	9.72	9.74	-0.01
Pirimiphos methyl	15.00	10.19	10.20	-0.01
Chlorpyrifos	15.45	10.50	10.51	-0.01
Myclobutanil	16.37	11.13	11.13	-0.01
Fenhexamid	17.68	12.03	12.03	0.00
Iprodione	19.23	13.08	13.08	0.00
Fenpropathrin	20.08	13.68	13.66	0.02
trans-Permethrin	20.49	13.95	13.94	0.02
Deltamethrin	22.47	15.31	15.28	0.02

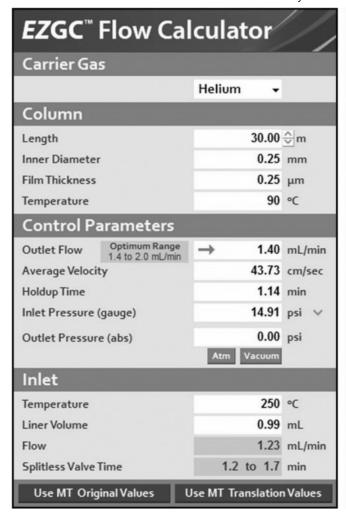
Posted in Enviro, Faster Analyses, Method Translator and Flow Calculator, Pesticides | Add a Comment >>

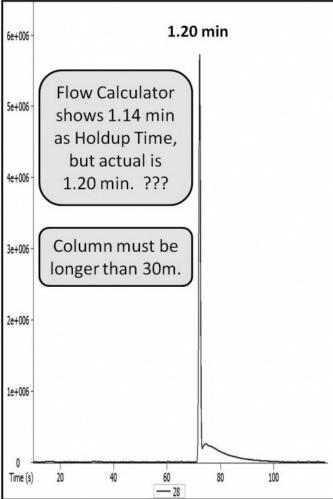
#### Calculating your GC Column Length with the Restek EZGC Method Translator and Flow Calculator

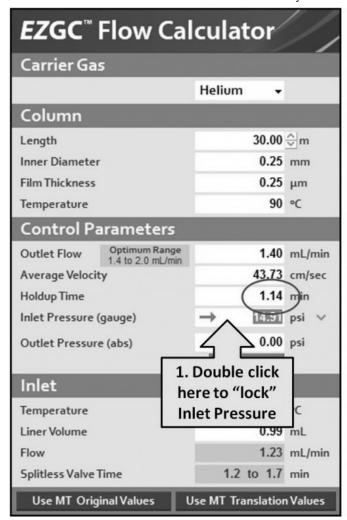
Saturday, July 19th, 2014 by Jack Cochran

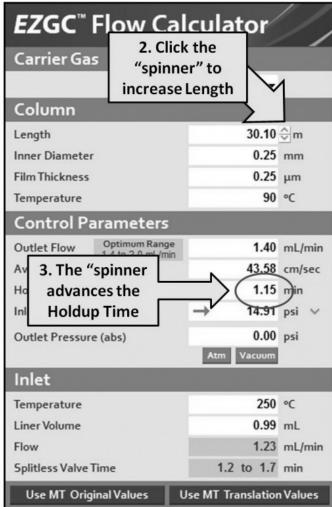
Determining your GC column length is important so that electronic pneumatic control of carrier gas flow is accurate, whether during initial installation of the column or after maintenance column trimming. Otherwise, you can have more flow going into the detector than you think (especially problematic in MS as you might lose sensitivity) or even see elution order changes in your chromatography. Calculation of GC column length is easy (or EZ!) with the EZGC Method Translator and Flow Calculator, just by performing a holdup time determination. Holdup time, the amount of time it takes an unretained compound to traverse the GC column from inlet to detector, can be determined by split injecting air (GC-MS), methane (GC-FID), or methylene chloride headspace (GC-ECD) and noting the "retention time". In the last case, GC-ECD, make sure the oven temperature is at least 250° since methylene chloride will show some retention at lower column temperatures. Don't overload the column with the compound, i.e., the peak should be symmetrical for an accurate determination. Start your work by entering the nominal value (e.g. 30m) for length in your GC control software, enter a flow, and then do the holdup time determination. I usually take the average "retention time" of three analyses.

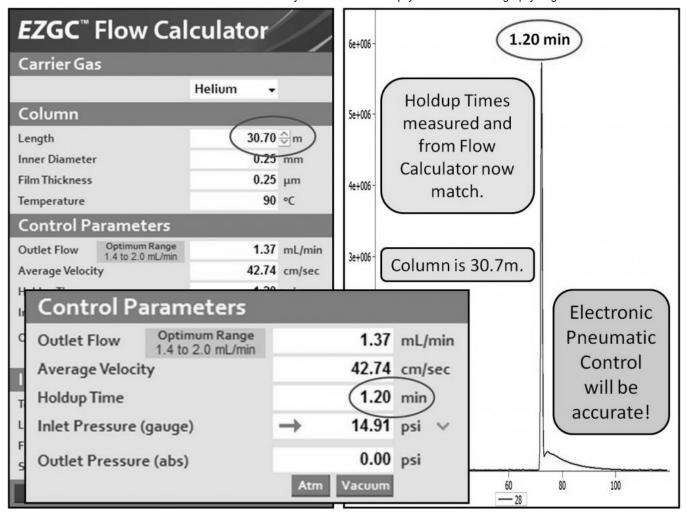
I've outlined the rest of the procedure in the figures below. Note that I've used the Download-ed version, which has the "spinner" for easy adjustment of column length, but it will work for the web-based version, too, just by entering column length values instead of "spinning". Shoot me an e-mail if you have any questions.











Posted in Faster Analyses, GC/MS, Lab Hacks, Method Translator and Flow Calculator, Optimizing Applications, Tips & Tricks | Add a Comment >>

#### Multi/Multi Methods (MMM) in Food Analysis

Thursday, July 17th, 2014 by Hansjoerg Majer

More and more laboratories are facing more and more pressure in both, time and price. More work has to be done in shorter time. Customers are deciding on a cent-to-cent base per analysis whether they will place an order to one contract laboratory or to another. Production laboratories are requested for shorter Turn-Around-Times (TAT) to let production be more precise or to shorten delay times for logistics. Due to increased potential of instrumental infrastructure like Triple Quads, Q-TOFs and other detection techniques as well as automated sample preparation possibilities there is a trend observable towards Multi/Multi Methods (MMM). A Multi/Multi Method is a combination of two or more Multi-Methods (Screening Methods).

During EPRW, Dr. Anna Romanotto and co-workers from the well known Eurofins-Sophia Food Safety Laboratory in Berlin published a poster with their method of "Simultaneous Clean Up and Measurement of 190 Pesticides, EPA PAHs, 18 Plasticizers, Bisphenol A and Non-Dioxine-Like PCBs in Fat and Oil Samples".

Such a highly sophisticated method demands high end instrumentalization and has very dedicated demands to the chromatographic system, especially to the separation column. A suitable column needs to have sufficient separation power to produce an evenly spread chromatogram, with no clusters of compounds to make MS determination difficult, and must separate difficult pairs of compounds with isobaric fragmentation patterns.

For this method is designed as a high sample throughput method, dealing with difficult matrices, also a long term stability and a batch to batch reliability is requested.

For this fully validated method Dr. Romaotto and her crew has chosen a Restek Rxi-17 Sil MS column (L=30m; ID=0,25 mm; dF=0,25 µm) as best fit for this challenging task.

Please find more details about this Time and Cost saving method and the original published poster here

Posted in Faster Analyses, Food, GC/MS, Optimizing Applications, PAH Analysis, Pesticides | Add a Comment »

#### Fast Determination of PAH and PCB in one Run

Thursday, July 17th, 2014 by Hansjoerg Majer

Environmental contract labs face a hard price pressure. To overcome this pressure, a trend into the direction of Multi/Multi Methods can be observed. If possible, more than one parameter group shall be determined and measured with one instrument without changing hardware. This implements a specific request for the separation power of the column used and the detection power of the installed instrument base.

The appearance of PAH and/or PCB contamination is a suitable indicator for industrial contamination of different matrices like Water, Sludge, Soil and Dust. Both parameters are among the most measured compound classes in environmental analysis in Europe.

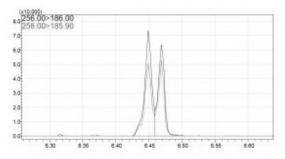
To optimize this measurement and to improve their new TQ-8030 GC/Q³ system, Shimadzu Germany recently showed an application which determines both compound classes in one run within 11 minutes by using Hydrogen as carrier gas and a Pseudo-MRM technique to increase PAH sensitivity.

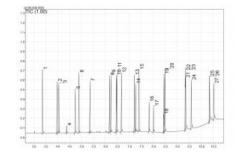
German and International Standards are requesting some frame conditions for the determination of these compounds.

The German Dump Regulation names ISO 18287 with GC/MS detection as method of choice. The alternative EN 15308 names GC/ECD detection and GC/MS detection as suitable, so the Q³ approach of the Shimadzu Scientists is according to the existing norms. The German sewage sludge ordinance asks for no interference between PCB 101 and o,p'-DDE or alpha-Endosulphane and between PCB 138 and p.p'-DDT. Most challenging, a good separation between PCB 28 and PCB 31 is required. The developed application used DIN 38 414–20 as base for sample preparation.

A lot of instrument companies rely on Restek Products, when it really matters. So, in this case, a Restek Rxi-XLB column ( $\models 20 \text{ m}$ ; ID = 0.18 mm; dF= 0.18  $\mu$ m) was chosen to run the separation.

The original paper shows the good separation between the two PCBs 28 and 31 (R>0.94), as shown in Picture 1, and no Interferences between the named PCB and PAH compounds, although measured in one run.







Picture 1: Separation between PCB 28 and 31

Picture 2: Chromatogram of a PCB/PAH mixture (all 5 ng/µl)

More details about the method, the usage of Hydrogen as carrier gas and how to optimize a Q³ by setting Pseudo MRMs for PAHs can be looked up in the original paper (in German), published in Nachrichten aus der Chemiel 62 | Mai 2014 |

Posted in Alternate GC Carrier Gases, Enviro, Faster Analyses, GC/MS | Add a Comment »

# <u>Using the Restek EZGC Method Translator and Flow Calculator to Support Shoot-and-Dilute GC Method Development – Going from GC-ECD to GC-MS</u>

Wednesday, July 16th, 2014 by Jack Cochran

Hopefully some of you are following the Shoot-and-Dilute GC work (split injection) we've been doing in our lab, as it offers a way to keep your GC systems up longer by reducing the impact of dirty samples on inlet liner and column integrity. After giving a lecture on the technique at the recent European Pesticide Residue Workshop in Dublin, Ireland, I was challenged by an audience member to tackle Captan and Folpet using Shoot-and-Dilute GC. Captan and Folpet are two notoriously unstable pesticides that must be determined by GC because they don't ionize well under ESI conditions for LC-MS/MS. By unstable, I especially mean under hot splitless GC inlet conditions. In addition, they fragment heavily by electron ionization GC-MS, so selectivity in complex matrices can be poor enough to mandate their determination using GC-ECD. I've achieved some very promising results already for Captan and Folpet with Shoot-and-Dilute GC-ECD, but that's not what I'm here to talk about...

I'm here to announce, for the first time on ChromaBLOGraphy, the release of our EZGCTM Method Translator and Flow Calculator (MTFC), a super cool tool for GC method development. And, I want to show you how I used it in my Shoot-and-Dilute GC work by translating a method from GC-ECD to GC-MS so I could confirm some Captan and Folpet results for strawberry extracts. In short, I'm using the same nominal length column, a 15m x 0.25mm x



Posted in Enviro, Faster Analyses, GC Injection Techniques, GC/MS, Method Translator and Flow Calculator, Optimizing Applications, Pesticides, Tips & Tricks | 3 Comments »

#### Half the GC Column, Three Times Faster Analysis, Same Chromatogram!

Saturday, June 28th, 2014 by Jack Cochran

### $Good\ things\ that\ happen\ when\ you\ go\ from\ a\ 30m\ x\ 0.25mm\ x\ 0.25\mu m\ GC\ column\ to\ a\ 15m\ x\ 0.25mm\ x\ 0.25\mu m\ GC\ column\ to\ a\ 15m\ x\ 0.25mm\ x$

- You do NOT lose resolution by a factor of 2 by with the 15m column.
- The sample loading capacity is about the same for the 15m column.
- With GC-MS, you gain speed of analysis by a factor of about 3 with the 15m column.
- With proper Method Translation, you get the same chromatogram with the 15m column.
- The peaks are narrower and taller and better detected with the 15m column.
- The cost of a 15m column is about half the cost of a 30m column.

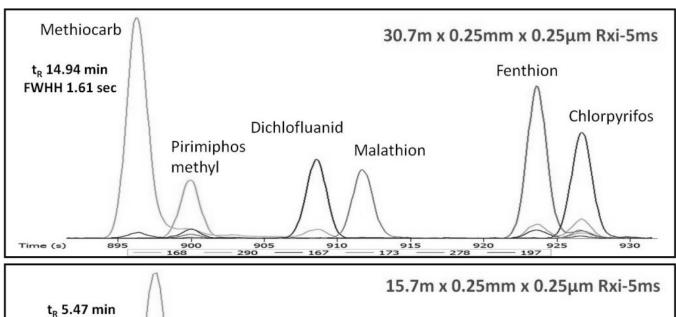
See the chromatograms below. Enough said...

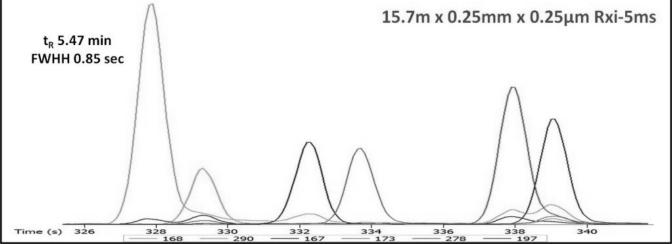
#### Additional reading:

Half the Column, Same Chromatogram. Trimming your GC Column and Maintaining Resolution.

Half the Column, Same Chromatogram: Maintain Resolution of BDE 49 and BDE 71 With Proper Method Translation After Trimming an Rtx®-1614 Column for

Maintenance





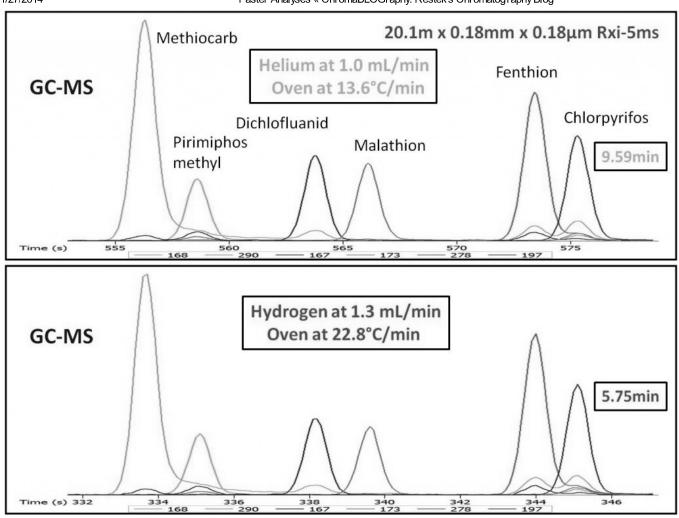
Posted in Faster Analyses, GC/MS, Method Translator and Flow Calculator, Optimizing Applications, Pesticides, Tips & Tricks | 4 Comments >>

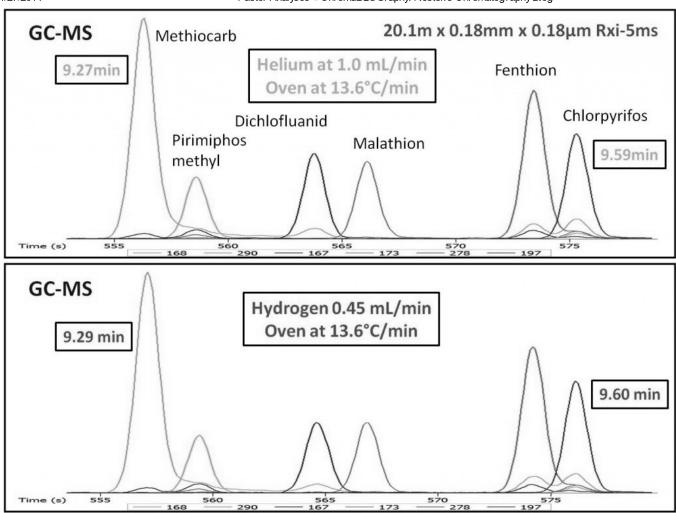
# <u>Using Hydrogen Carrier at a Lower Flow Rate for GC-MS – Separations Compromised?</u>

Sunday, June 22nd, 2014 by Jack Cochran

In the previous ChromaBLOGraphy post, Simple translation of GC methods from helium to hydrogen carrier gas, I demonstrated two Method Translation approaches to switching GC carrier gas from helium (He) to hydrogen (H2) for GC-ECD of organochlorine pesticides. The first approach involved going from speed-optimized flow (SOF) for He to SOF for H2, which is faster, and that requires a faster GC oven program rate to maintain the same chromatographic elution pattern for the compounds of interest. The second approach was to start with SOF for He and then just set the holdup time (or linear velocity) for H2 to match that for He. Yes, we're below optimum flow now for H2, but an advantage is being able to use exactly the same GC oven program and get essentially the same component retention times as we got with He.

The second approach, that of matching holdup times for He and H2 upon a carrier gas switch, can be used to some advantage with GC-MS where hydrogen is not easily pumped and a higher (optimum) flow would lead to a more drastic detectability loss. I give examples of both approaches below for GC-MS from the starting point of efficiency-optimized flow for He. Hopefully you'll come to the same conclusion I did that running at a sub-optimum flow for H2 carrier did not substantially degrade the separations of example pesticides.





Posted in Alternate GC Carrier Gases, Detection Techniques, Enviro, Faster Analyses, GC/MS, Method Translator and Flow Calculator, Optimizing Applications, Pesticides, Tips & Tricks | 4 Comments »

# World Record Set for Longest Gas Chromatography Retention Time!

Monday, April 7th, 2014 by Jack Cochran

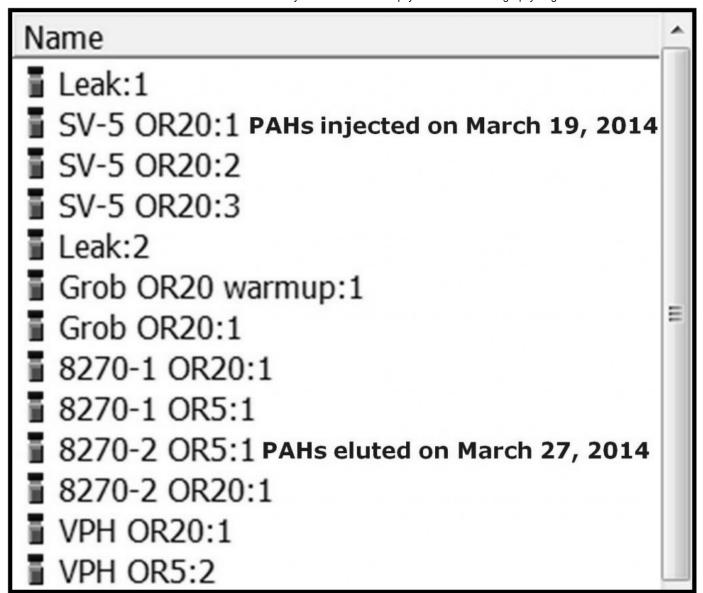
My colleague Jaap de Zeeuw holds a Guinness World Record certificate for making and applying the longest GC column ever, 1300m. That's quite a feat and I've often wondered what the retention times were for that column when you consider the holdup time was probably on the order of hours instead of minutes. No matter how long the retention times were though on that 1300m GC column, I may have exceeded them on a simple 30m x 0.25 mm x 0.25 mm x 0.25 mm Stabilwax, which I've been using for a selectivity study conducted by colleagues James Harynuk and Teague McGinitie at University of Alberta. This work will be presented at the 11th GCxGC Symposium (and 38th International Symposium on Capillary Chromatography) in Riva del Garda, Italy.

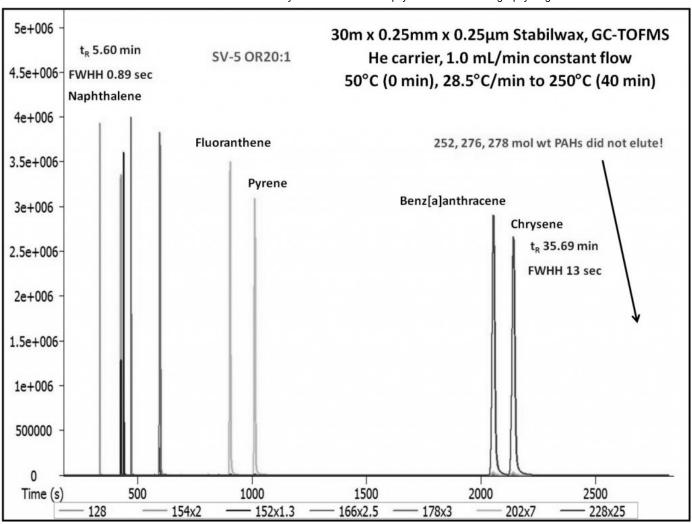
The study includes, Rxi-17Sil MS, Rxi-17Sil MS, Rtx-200, and Stabilwax GC columns, which represent a variety of stationary phase polarities and selectivities. The first 3 have temperature stabilities of 350, 360, and 340°C, respectively, but the Stabilwax only goes to 260°C. I can chromatograph all of the molecules chosen for the study on the first 3 phases, but when I get to the polycyclic aromatic hydrocarbons (PAHs), some of which are notoriously involatile, I struggle on the Stabilwax.

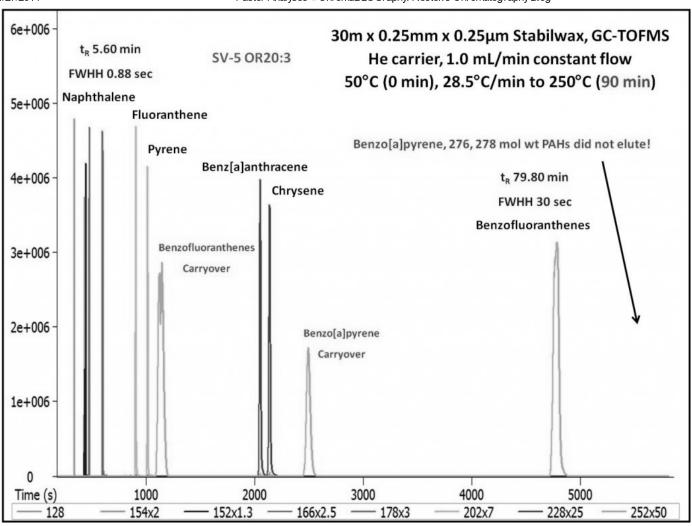
The first figure below shows the Acquired Sample list for the Stabilwax work, and I purposely started with <u>SV Calibration Mix #5 / 610 PAH Mix</u> because I knew some of the compounds would be hard to elute from the wax GC column. Well, how about almost impossible to elute? As you can see in the first chromatogram, even though I had a 40 min final oven temperature hold time at 250°C, none of the PAHs with molecular weights of 252, 276, and 278 eluted. In chromatogram 2, which is run 3 from the queue, I see carryover peaks for 252 PAHs (benzofluoranthenes, benzo[a]pyrene), but still didn't get complete elution of the PAHs even though I pushed the final hold time up to 90 min. OK, this isn't going so well, this part of the selectivity study...

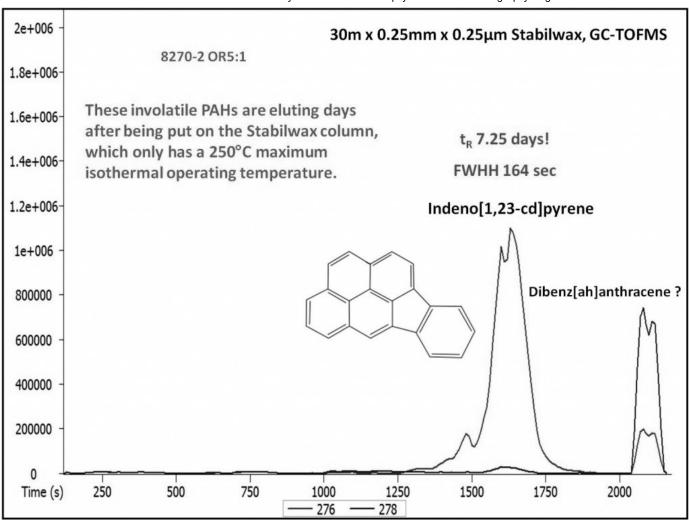
Eventually, after uninstalling the Stabilwax column, putting it in my office for a few days, and then reinstalling it to run different standards, I finally saw the first 276 and 278 PAHs eluting as massively broad peaks, almost 5 min wide at base. When I calculate retention time (yeah, sure, I included the time in my office!) for indeno[1,2,3-cd]pyrene, it's 7.25 days!

Your move, Jaap...









Posted in Conferences, Faster Analyses, Miscellaneous, PAH Analysis | Add a Comment »

# Faster GC Analysis of Medical Cannabis Terpenes with Same 624Sil MS Selectivity

Tuesday, March 25th, 2014 by Jack Cochran

The chromatograms below show what happens when you translate a GC method (previously used for medical cannabis terpenes <u>here</u> and <u>here</u>) from a  $30m \times 0.25mm \times 1.40 \mu m Rxi-624Sil MS$  GC column to a  $30m \times 0.25mm \times 1.00 \mu m Rxi-1301Sil$  MS column. Both of these columns have arylene-modified cyanopropylphenyl dimethyl polysiloxane-type stationary phases. As should be expected, the separation is approximately the same, but faster, due to the thinner film on the 1301Sil MS.

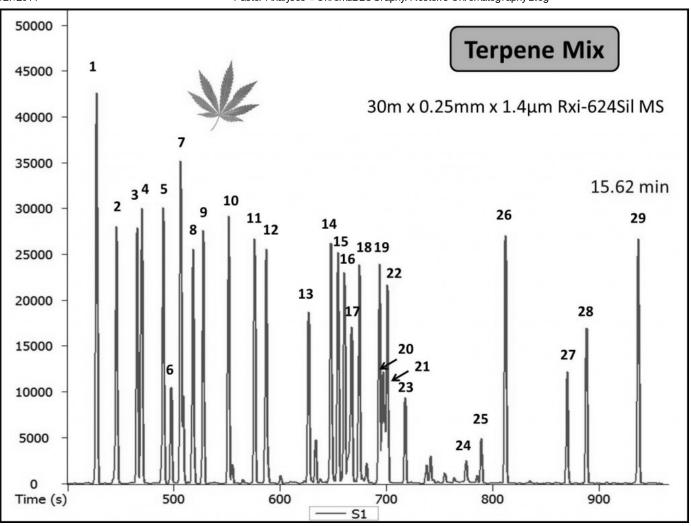
Another thing that the thinner film gives users is the ability to elute less volatile compounds without long isothermal hold times. The example here is for Phytol, an acyclic diterpene alcohol that may be a therapeutically-active compound in cannabis. Phytol wasn't in the terpene mix I analyzed on the 624Sil MS, and in fact, I added several "new" terpenes to my qualitative medical cannabis terpenes standard for the 1301Sil MS work, including Sabinene, alpha-Phellandrene, Ocimene, p-Cymene, alpha-Humulene, alpha-Bisabolol, and Phytol. I've labelled some of those additional terpenes with small letters in the first 1301Sil MS chromatogram and the analyte list, and I show the later eluting additions by name in the second 1301Sil MS chromatogram.

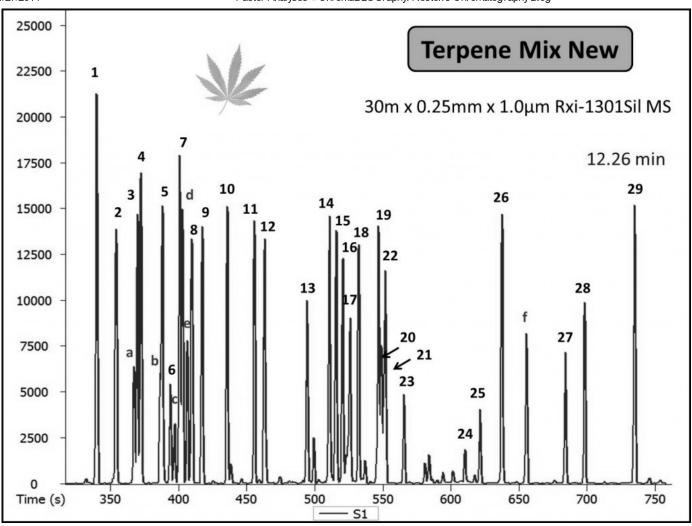
I am continuing this work along several lines, including running on even thinner-film cyanopropylphenyl dimethyl-type columns. Why do that? Because we can pick up efficiency, i.e. more separation power through narrower peaks. Chromatographic efficiency is EXTREMELY important in this type of analysis because there are many, many terpenes in medical cannabis and *high efficiency GC is the only way to go* to achieve the best separations and avoid coelutions that will lead to inaccuracies in terpene quantification for medical marijuana.

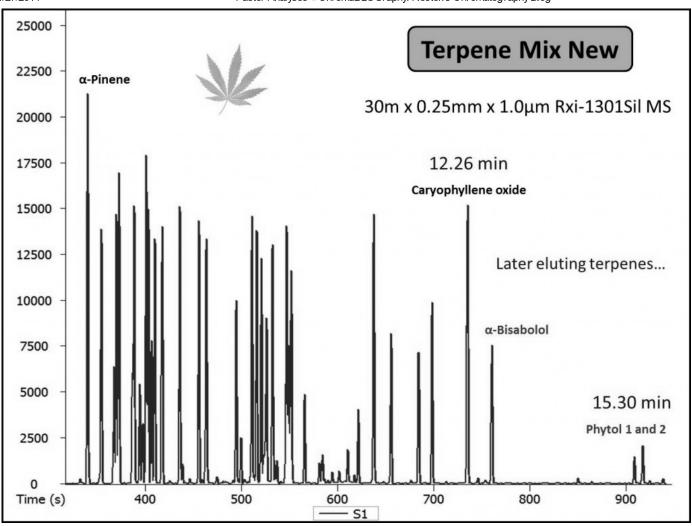
I'd like to acknowledge Andrew Goldsmith of <u>SRI Instruments</u> for providing some of the terpene standards, and Don Rhoads of Restek for making the betaversion Rxi-1301Sil MS GC column.

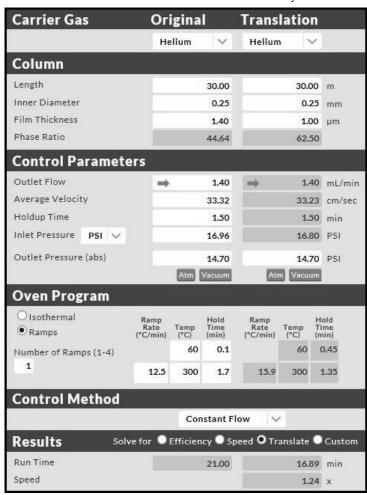
Please, check out our Medical Marijuana web page.

Analyte	Name	
1	alpha-Pinene	
2	Camphene	a Sabinene
3	Myrcene	b slubs Dhallanduana
4	beta-Pinene	b alpha-Phellandrene
5	delta-3-Carene	c Ocimene 1
6	alpha-Terpinene	e demene i
7	Limonene	d p-Cymene
8	1,8-Cineole	
9	gamma-Terpinene	e Ocimene 2
10	Terpinolene	
11	Linalool	
12	Fenchone	
13	1-Isopulegol	
14	dl-Menthol	
15	1-Borneol	Terpene
16	alpha-Terpineol	icipene
17	Dihydrocarveol	Mix New
18	Citronellol	( With teets )
19	Geraniol	
20	2-Piperidone	
21	Citral 1	
22	Pulegone	
23	Citral 2	
24	Citral 3	
25	Citral 4	
26	beta-Caryophyllene	E alaba Himmilana
27	Nerolidol 1	f alpha-Humulene
28	Nerolidol 2	
29	Caryophyllene oxide	







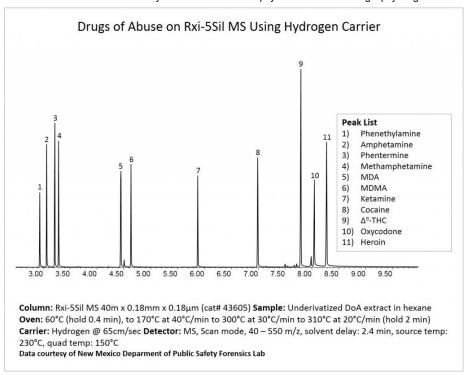


Posted in Faster Analyses, Medical Marijuana, Method Translator and Flow Calculator, New GC Columns, Optimizing Applications | Add a Comment >>

#### Who Says There's Nothing New Happening in GC Forensics?

Monday, February 24th, 2014 by Amanda Rigdon

From an analytical standpoint, GC/MS has been routinely used in forensics labs for decades. It was referred to as the 'gold standard' in analyte identification, until the 'platinum standard' (I'm sure some vendor coined that moniker) of MS/MS became commercially available. Even though we now have a lot of fancy new instruments at our disposal, GC/MS remains the workhorse in forensic laboratories, running methods that have been handed down from one generation of analysts to the next. In most cases, these methods work just fine, but there's always room for improvement. One of our favorite pastimes here at Restek is optimizing chromatographic methods for the best performance possible – yes, we're a population of glorious nerds. Fortunately, there are others in industry that enjoy optimization as much as we do. The folks at the New Mexico Department of Public Safety Forensics Lab let their geek flags fly and improved their GC/MS method for drugs of abuse by switching to hydrogen carrier and using a super-efficient small-bore Rxi-5Sil MS column. The result is some beautiful chromatography in half the time of their original method.



In addition to doubling their throughput, they're now better prepared for the upcoming helium crisis. In an industry where method development time is at a bare minimum, I'm really happy to see that customers are still actively improving their old GC/MS analyses. I'd also like to give my sincere thanks to the New Mexico Department of Public Safety Forensics Lab for allowing me to share their data and story.

For more information on alternative carrier gases and fast GC, check out these blogs:

Simple translation of GC methods from helium to hydrogen carrier gas: http://blog.restek.com/?p=11102

BAC Analysis Using Hydrogen Carrier Gas: Get the Same Results at a Lower Cost! <a href="http://blog.restek.com/?p=6374">http://blog.restek.com/?p=6374</a>

Fast Organochlorine Pesticide Analysis Using Hydrogen Carrier Gas with Split Injection GC-ECD: http://blog.restek.com/?p=7815

Fast(er) GC: How to Decrease Analysis Time Using Existing Instrumentation? Part V: Using Smaller Bore Capillary Columns: http://blog.restek.com/?p=3549

Half the Column, Same Chromatogram. Trimming your GC Column and Maintaining Resolution: http://blog.restek.com/?p=7899

Posted in Alternate GC Carrier Gases, Drugs of Abuse, Faster Analyses, GC/MS, Optimizing Applications | Add a Comment »

#### « Older Entries

Copyright © 2014 Restek. All Rights Reserved. Entries (RSS) and Comments (RSS).