Volume 15 | Number 1 | 2002

Agilent Separation Times

Technically Advanced GC Columns and Supplies for Optimized Performance

Performance is not an Illusion: Maintenance Tips for FIDs
See Page 2.

Agilent Technologies

Sharing Expertise

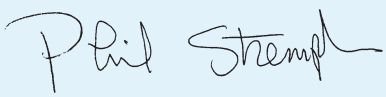
This issue of Separation Times includes articles from our chemists on maintaining FID detectors; the risks in using non industry-standard columns; and lessons in analyzing dirty samples. Eberhardt Kuhn continues his case histories (in "Ask Technical Support") with the case of the disappearing peaks. We also have special guest authors who explain how a faster chromatography method decreased run times by 35% for racehorse drug testing.

"Agilent e-Seminars are an innovative way for us to share knowledge. I particularly like that people can take part in these real-time, interactive courses wherever they are."

Teaching Agilent Technologies' e-Seminars is an important part of our work. These free information-packed courses help participants improve laboratory skills and keep current with technology advances—without leaving their desks. (See Page 12.)

Each instructor is a GC expert. Angelica Reese (on this issue's cover and the author of "Keys to Maintaining FID Detectors") is an authority on detectors, basic GC theory and GC column selection, maintenance and troubleshooting. With a B.S. in Cell and Molecular Biology from San Francisco State University, she began her career as a forensic toxicologist, testifying in county and federal courts as a drug and alcohol expert. In her three years on our staff, she has distinguished herself as a seasoned instructor and frequent presenter at conferences.

I hope that you find this issue valuable. If you want further information or advice, send us an e-mail (see Page 11) or give us a call. And please sign up for an e-Seminar. You may get Angelica as your instructor.



Phil Stremple, Ph.D.
GC Columns Program Manager
phil_stremple@agilent.com

From Start to Finish

The last issue of *Separation Times* (publication number 5988-4232EN) offered a guide to selecting the right inlet parts. In this issue, we spotlight results, with hints for getting optimum detector performance.

Keys to Maintaining FID Detectors

- **Set gas flows within recommended rates**
- **Install the right jet for your application**
- **Maintain detector temperature above 110°C**
- **Maintain gas purifiers**
- **Remove deposits**
- **Clean or replace jet, collector and ignitor glow plug assembly periodically**

By Angelica Reese
Technical Support Chemist

The Flame Ionization Detector (FID) is one of the most widely used detectors in gas chromatography. The simplicity of its operation and the dependability of its performance are the primary reasons for its popularity. It is also because of these reasons that the FID can be, and often times is, neglected when it comes to simple maintenance and optimization.

There are three gases necessary for FID operation: hydrogen, air and makeup gas (usually nitrogen but it can also be helium). The ratio of hydrogen to air is important in the operation of the FID. The ratio should be between 8 and 12% to keep the flame lit.

(Continued on Page 3)

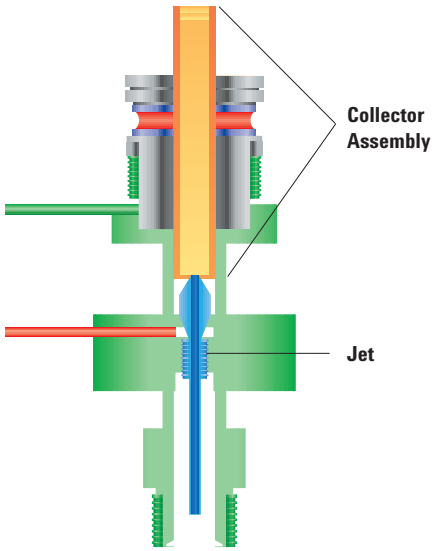


Figure 1. Flame Ionization Detector

Continued from Page 2

Typical flows for these gases are ~30 mL/min for hydrogen, ~300 mL/min for air and ~30 mL/min for make-up gas. While the carrier gas is also necessary, changes in the carrier gas flow rates do not significantly affect sensitivity. It's a good rule of thumb to measure the FID flows with an independent measurement device—bubble meter or electronic flow meter—periodically (use the FID flow adapter, Agilent part number 19301-60660). If the gas flows deviate from the recommended flow rates more than 10%, there may be difficulty lighting the flame. On the other hand, difficulty lighting the flame can also be a symptom of using the wrong jet or a plugged jet.

There are different size jets available for different applications. Packed columns use different jets (18710-20119 or 18789-80070) than capillary columns (19244-80560 or 19244-80620) and dedicated capillary FIDs for the 6890 GC use different jets (G1531-80560 or G1531-80620) than adaptable FIDs. (See Page 8 for details on different jets and their applications.) You must install the proper jet for your application before installing the column. The choice of jet can optimize the flame shape for capillary columns or reduce contamination build-up for high molecular weight eluates. Typically, small-bore jets provide the greatest sensitivity but can be prone to contamination.

Since the FID combustion process results in water formation, the detector temperature must be kept above 110°C to prevent condensation. Such condensation, especially when combined with chlorinated solvents or samples, can cause corrosion and sensitivity loss and/or spiking. The FID is typically operated at 250°C to 300°C to prevent solute condensation.

Figure 2. Plugged FID jet

Certain compounds, including by-products of column bleed, can cause deposits to build up on the detector. These deposits can reduce sensitivity and cause chromatographic noise and spikes. Deposits must be mechanically removed to return sensitivity and performance.

Jets require periodic cleaning or replacement (consult your instrument manual or go to our Web site www.agilent.com/chem to view the on-line instructional how-to videos under Technical Support). Once removed from the detector assembly, the jet can simply be replaced with a new one or cleaned. When cleaning the jet, be careful not to scratch the inside of the jet.

If corrosion or contamination has been confirmed, the collector may also need to be cleaned or replaced. To remove/replace the collector, refer to your instrument manual or our Web site. To clean the collector, follow the same procedures as for the jet, except use a nylon brush to remove any deposits.

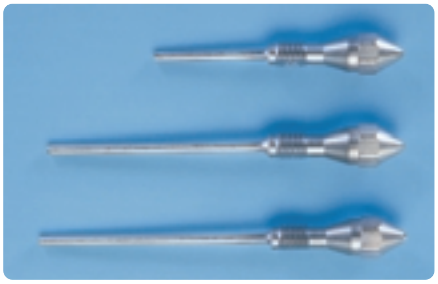
Cleaning the FID Jet

1. Sonicate the jet in a cleaning bath containing aqueous detergent for approximately five minutes.
2. Remove the jet and use a cleaning wire to clean the inside of the jet.
3. Sonicate for another five minutes.
4. Remove the jet from the bath with tweezers and rinse thoroughly with hot water followed by a small amount of methanol.
5. Blow the jet dry with a burst of compressed air and allow the jet to air-dry on a paper towel.

Unexplained detector noise or abnormally high background can be caused by old gas purifiers that need to be replaced on the detector gas lines or indicates the need to install purifiers.

Often the ignitor glow plug assembly may need to be replaced. The easiest way to determine if the ignitor needs replacement is if you have difficulty lighting the flame or if the ignitor does not glow orange when the ignition switch is activated. Refer to the instrument manual or our how-to videos on our Web site for instructions regarding replacement.

To access the how-to videos on the Web, visit www.agilent.com/chem, Technical Support.



Agilent FID Jets (from top to bottom)
Dedicated capillary, Part No. G1531-80560
Adaptable capillary, Part No. 19244-80560
Adaptable packed, Part No. 18710-20119

ORDER GUIDE*

Jet Type	Jet Tip I.D. (mm)	Jet Tip I.D. (in.)	Part Number
Jets for capillary-dedicated FID (6890 GC only)			
Capillary	0.29	0.011	G1531-80560
High Temperature Capillary	0.47	0.018	G1531-80620
Jets for adaptable FID (6890, 6850, 5890 GCs)			
Capillary	0.29	0.011	19244-80560
Packed	0.47	0.018	18710-20119
Wide Bore Packed	0.79	0.030	18789-80070
High Temperature Capillary	0.47	0.018	19244-80620

* Jets for the capillary-optimized FID are 48 mm long, while jets for the adaptable FID are 61 mm (capillary) and 63.5 mm (packed) long.

For more information on detectors, see Page 8.

Considerations When Analyzing Dirty Samples

- **Glass wool packing traps sample residue**
- **Pre-packed liners reduce liner-to-liner variation**
- **The gold-plated inlet seal at the bottom of the injection port needs routine maintenance**

By Jason Ellis
Technical Support Chemist

Samples containing large amounts of nonvolatile or semivolatile residues can quickly contaminate the capillary column and GC system. This contamination can cause retention time shifts, loss of resolution, loss of response and elevated detector signal. Ideally, sample residues should be removed before injection by proper cleanup techniques. Unfortunately, this is not always possible. When dirty samples must be injected, proper inlet maintenance and liner selection can help to avoid more severe problems later, such as the contamination of a capillary column or detector.

The Agilent/J&W Scientific toll free number for Technical Support (800-552-0413) will no longer be valid after August 15, 2002. Please use **800-227-9770**, option # 4, then press # 1 for GC Technical Support. Thank You!

Glass wool in the liner can help to trap some of the sample residues before they enter the capillary column. But quantitative reproducibility can be profoundly affected by variability in the amount and placement of glass wool. Agilent offers liners with carefully controlled amounts of deactivated glass wool located either at the top or the bottom.

Pre-packed liners eliminate the tedious steps of packing and deactivating your own liners. This can improve method reproducibility because liner-to-liner variation is greatly reduced. If you are using a liner with glass wool located at the top, your syringe needle length should not exceed 42 mm. If your needle is longer, then the sample will be injected below the glass wool plug, reducing or eliminating its effectiveness.

While the liners and glass wool listed here are all chemically deactivated, some compounds may still exhibit adsorption or decomposition problems when using glass wool packing. This is typically exhibited by response loss and peak tailing for compounds with active functional groups. Amines, nitrophenols and organic acids tend to fall into this category, as do other active or thermally labile compounds. For this reason, not all methods perform well with glass wool. When performing method development, it is often a good idea to try doing some analyses with and without glass wool packing.

(Continued on Page 5)

Continued from Page 4

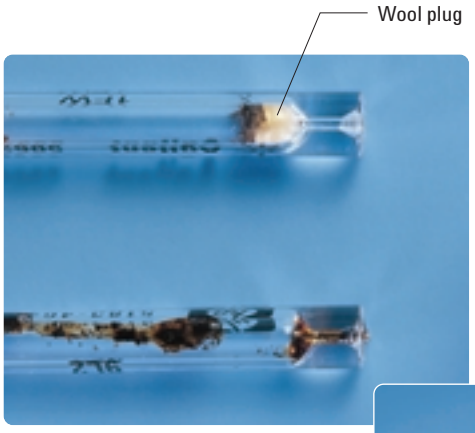


Figure 1. Bottom portion of liners with contamination residue.

Figure 1 shows the bottom portion of liners with contamination residue from injections of a wine sample. Approximately the same amount of sample was injected into both liners for this example. Clearly, the glass wool at the bottom of the liner has prevented the contaminant material from reaching the bottom of the inlet and the front of the column.

Often neglected by GC users, the gold-plated inlet seal at the bottom of the injection port on 5890, 6890 and 6850 split/splitless inlets also needs routine cleaning or replacing. Over time, sample residues will accumulate on this seal and can cause analyte adsorption or decomposition, and can elevate instrument signal background. A clean gold-plated inlet seal is even more important when performing splitless injections because of the increased residence time of the sample in the heated injection port. Figure 2 shows a gold-plated inlet seal contaminated with sample residue from injections of a wine sample.

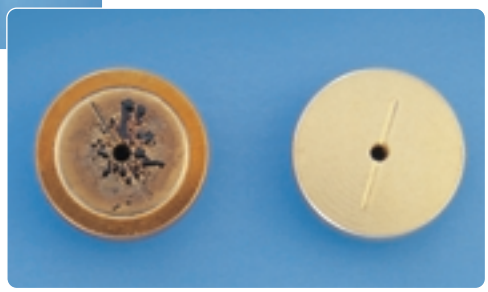
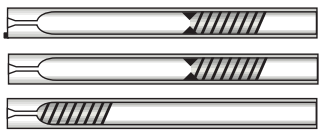


Figure 2. Gold-plated inlet seals: contaminated with sample residue (left) and clean (right).

The message: Keep your instrument running at its very best with routine maintenance and genuine Agilent consumables and replacement parts—the PerfectFit for optimum performance.

ORDER GUIDE

Product	Description	Part Number
Split Liner	Low pressure drop, glass wool at top, bottom taper, deactivated	5183-4647
Splitless Liner	Glass wool at top, bottom taper, deactivated	5183-4711
Splitless Liner	Glass wool at bottom, bottom taper, deactivated	5062-3587
Gold-Plated Seal	For Agilent 5890, 6890, and 6850 GCs	18740-20885



Be Careful! "Replacement" GC Columns May Not Be Equivalent

- **Imitation columns can be very different from brand-name columns**
- **You get what you pay for**

By Mitch Hastings
Technical Support Chemist

Some column manufacturers claim that their capillary GC columns are "replacements" for industry standard columns such as the J&W DB- and HP- brand columns. The implication is that analysts can substitute one of these "replacement" columns for a DB- or HP- column and obtain essentially the same relative elution order and column selectivity under the same conditions. However, as the following data show, "replacement" does not mean equivalent. Not recognizing this semantic dance can jeopardize a laboratory's time and money.

We compared the J&W DB-WAX column with a wax column from another manufacturer who claims that its column can "replace" J&W's industry-standard DB-WAX. We chose a typical application—adulterants and impurities commonly found in distilled alcohol from biological sources. The separation is very simple, containing only 17 compounds of interest, which are listed in the accompanying table. Moreover, the analysis is readily accomplished on a DB-WAX column. We performed the comparison under identical conditions of sample, temperature, column dimensions and carrier gas linear velocity. In addition, we incorporated a few hydrocarbons to check retention indices, a measure of selectivity. If these measurements turned out to be the same (within normal variation, generally +/- 2 RI units for this phase type), then the replacement column really can be considered equivalent and the manufacturer's claim is valid.

(Continued on Page 7)

Table 1. Selectivity Comparison of DB-WAX Columns

Compound	DB-WAX Time (min)	"Replacement" Time (min)	DB-WAX Retention Index	"Replacement" Retention Index	Retention Index Difference
1. Methane	1.184	1.195			
2. Pentane	1.230	1.236	500.00	500.00	n/a
3. Acetaldehyde	1.430	1.436	704.14	715.20	-11.05
4. Methyl Formate	1.587	1.590	764.24	775.23	-10.99
5. Propionaldehyde	1.700	1.694	794.33	803.62	-9.29
6. Acetone	1.818	1.806	819.41	828.23	-8.82
7. Methyl Acetate	1.889	1.872	832.33	840.69	-8.36
8. Butyraldehyde	2.213	2.181	878.37	886.37	-8.00
9. Ethyl Acetate	2.317	2.271	890.09	896.98	-6.89
10. Acetal	2.366	2.271	895.25	896.98	-1.73
11. Nonane	2.413	2.298	900.00	900.00	n/a
12. Methanol	2.446	2.392			
13. Methyl Propionate	2.505	2.450			
14. Isopropanol	2.852	2.744			
15. Ethanol	3.021	2.902			
16. 1-Propanol	5.150	4.879			
17. Isobutanol	7.653	7.117			
18. 1-Butanol	10.642	9.917			
19. Active Amyl Alcohol	16.421	15.303			
20. Isoamyl Alcohol	16.442	15.303			

ORDER GUIDE*

Most Popular Agilent DB-WAX Columns

Description	I.D. (mm)	Length (m)	Film (µm)	Part Number
DB-WAX	0.25	30	0.25	122-7032
DB-WAX	0.25	60	0.25	122-7062
DB-WAX	0.32	30	0.5	123-7033
DB-WAX	0.53	30	1.0	125-7032

* This is a small sampling of the many columns and dimensions available. For more information, call Agilent or go to www.agilent.com/chem

GC Conditions in our Test

Agilent Column:	**DB-WAX**
	Agilent: 30 m x 0.25 mm I.D., 0.25 µm
Agilent Part No.:	**122-7032**
"Replacement" Column:	30 m x 0.25 mm I.D., 0.25 µm
Carrier:	42 cm/sec
Oven:	40°C, Isothermal
Injector:	Split 1:50, 150°C
Sample:	1:5 in H_2O of 0.1-1% of each component in ethanol

Figure 1. See above table for compound identification.

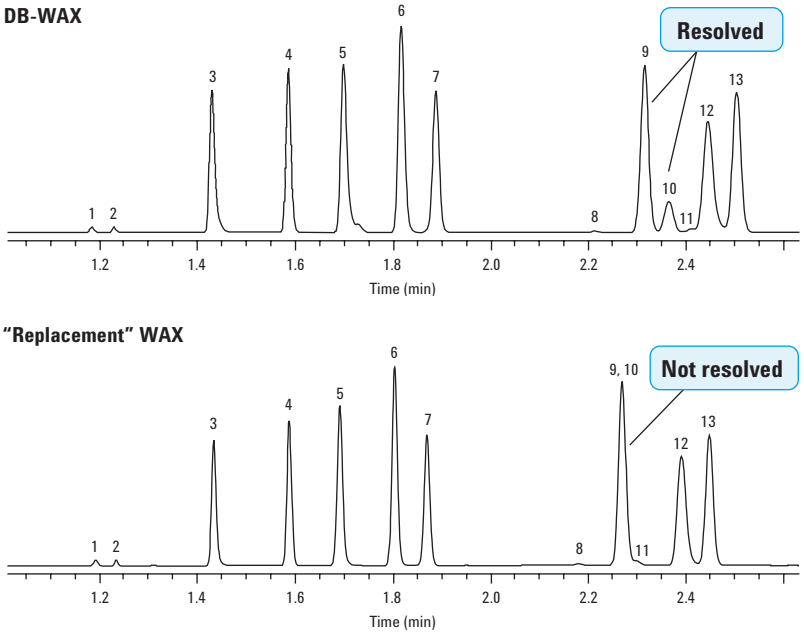
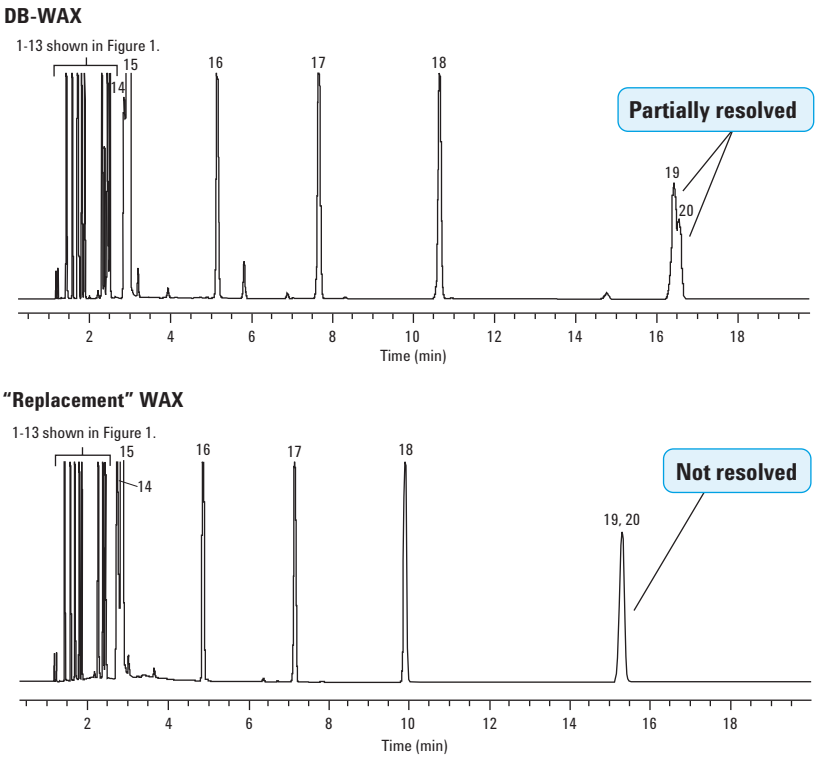


Figure 2.



"Replacement" GC Columns May Not Be Equivalent

Continued from Page 6

However, as the data in the table show, we found that selectivity varied over 11 units in some cases between these columns. The chromatograms shown here also reveal that you cannot just plug in and start using the so-called replacement columns. If you replace a DB-WAX column with one of these other wax columns, you:

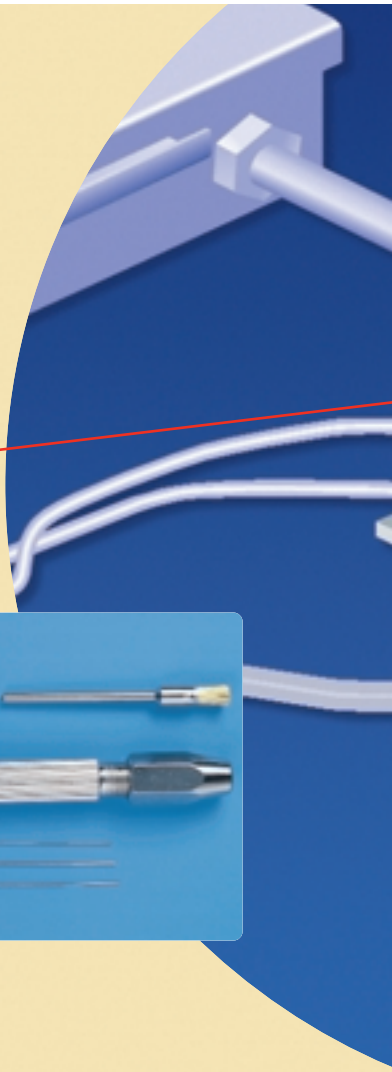
- Lose resolution of ethyl acetate and acetal (Figure 1).
- Lose what little resolution there existed between active amyl and isoamyl alcohol (Figure 2).

The manufacturers of replacement columns often claim that the "only difference is the price." For even simple applications such as the one shown here, you may actually lose money when you calculate the time it takes to re-verify elution orders and the potential risk that either the column will not work for your application or an elution order change might be missed. Any minor savings in the price quickly disappears—to be replaced by major liabilities.

For most analysts, consistent column performance from one column to the next is the most prized column performance characteristic. **To ensure consistency, Agilent tests every single column.** Further, Agilent is still the only column manufacturer who publishes and meets bleed, plates per meter, and retention indices specifications for nearly all the columns we supply. These specifications have only tightened since their initial publication in 1994.

Optimum Detector Performance using Authentic Agilent Replacement Parts and Supplies

It would be a shame to fully optimize all aspects of your chromatographic method and achieve superb chromatography only to elute your analytes of interest into an under-performing detector. The GC detector provides the critical link between the chromatographic separation and the chromatogram. To assure optimal performance, appropriate preventative maintenance is crucial. And be sure to use authentic PerfectFit Agilent replacement parts on your 6890, 6850 and 5890 GCs.



FID Cleaning Kit

FID Common Maintenance Supplies for 6890, 6850 and 5890 GCs

Item	Part Number
Flame Ionization Detector (FID)	
Flow measuring insert	19301-60660
Ignitor glow plug assembly	19231-60680
FID cleaning kit	9301-0985
Jet cleaning wire	19301-20720
FID performance test mixture	18710-60170
FID/NPD 1/4-in. packed column adapter	19231-80530
FID/NPD 1/8-in. packed column adapter	19231-80520
FID/NPD capillary column adapter	19244-80610
Vespel adapter ferrule (required for all adapters)	5080-8774

Agilent Replacement Parts and Accessories for Other Detectors

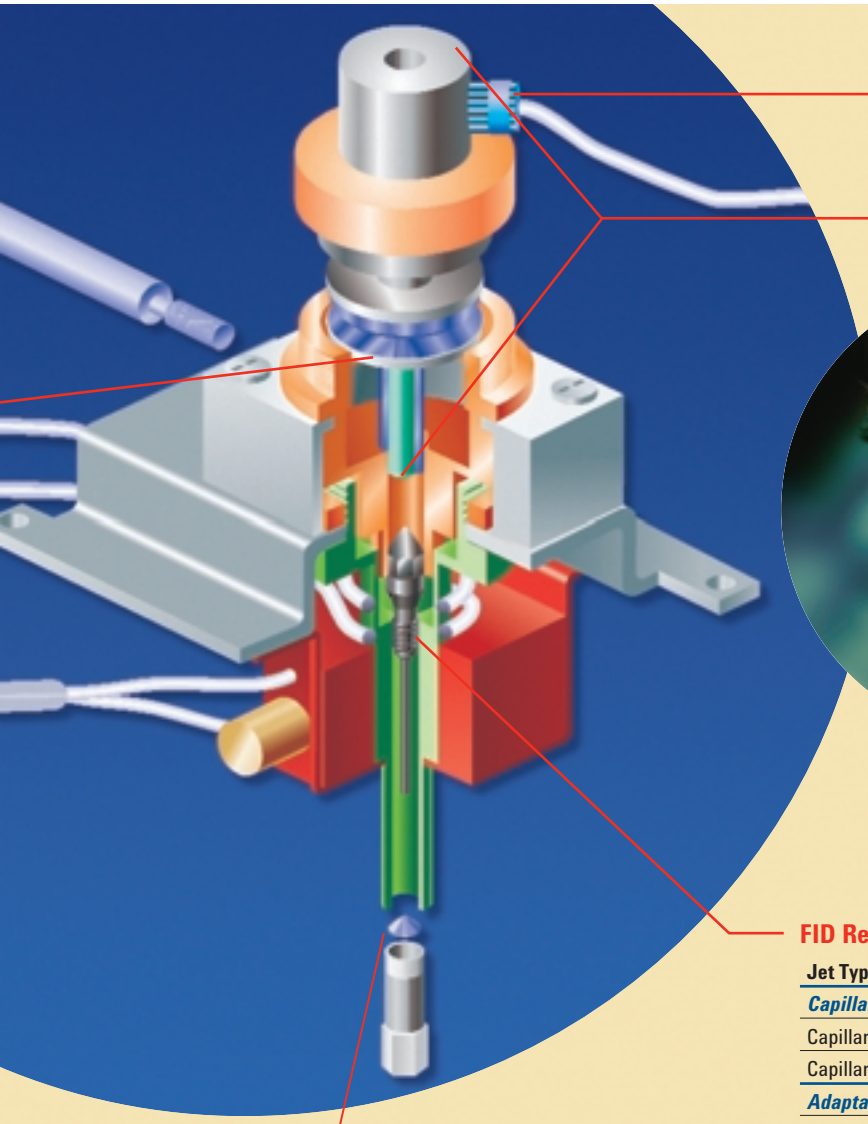
Item	Part Number
Nitrogen Phosphorus Detector (NPD)	
White ceramic bead assembly (6890 GC only)	G1534-60570
Black ceramic bead assembly (6890 GC only)	5183-2007
Collector assembly (bead) (5890 GC only)	19234-60540
Detector jets	See FID listing
Graphite ferrules	See FID listing
NPD performance test mixture	18789-60060
Thermal Conductivity Detector (TCD)	
Replacement cell (6890/6850 GCs only)	G1532-60675
Replacement cell (5890 GC only)	19232-60676
Graphite ferrules	See FID listing
TCD performance test mixture	18711-60060
Electron Capture Detector (ECD)	
Makeup gas adapter	19233-80565
Makeup gas adapter (micro-ECD)	G2397-80520
Fused silica liner	19233-20625
Fused silica liner (micro-ECD)	G2397-20540
Stainless steel makeup gas adapter cap	19233-20755
Graphite/Vespel ferrules	See FID listing
ECD wipe test kit	18713-60050
ECD performance test mixture	18713-60040

Instrument-Specific Miscellaneous FID Supplies

Item	Part Number
FID collector assembly* (5890 GC only)	19231-60690
FID collector assembly* (6890 GC only)	G1531-60690
Collector body (5890 GC only)	19231-20960
Collector body (6850 and 6890 GC only)	G1531-20690
Hastelloy ignitor castle** (5890 GC only)	19231-21060
Hastelloy collector body** (5890 GC only)	19231-21080

* Assembly includes: Gasket, Ignitor Castle, Ignitor Glow Plug Assembly, Spring Washer-Wavy, Collector: Housing, Mount, Nut, Body, Spanner Nut, Insulator (upper and lower)

** Hastelloy components are ideal when analyzing corrosive materials.



FID Replacement Jets

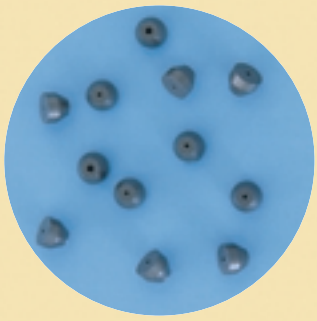
Jet Type	Length (mm)	Tip I.D. (in.)	Part Number
Capillary dedicated FID (capillary columns only; 6890 GC only)			
Capillary	48	0.011	G1531-80560
Capillary*	48	0.018	G1531-80620
Adaptable FID (capillary or packed columns; 6890, 6850 and 5890 GCs)			
Capillary	61	0.011	19244-80560
Capillary*	61	0.018	19244-80620
Packed column	63.5	0.018	18710-20119
Packed column jet*	63.5	0.030	18789-80070

* For high-temperature applications

FID Ferrules

Ferrule Type	Column I.D. (µm)	Ferrule I.D. (mm)	Part Number*
General purpose graphite	180, 200, 250, 320	0.5	5080-8853
General purpose graphite	530	1.0	5080-8773
Graphite/Vespel	180, 200, 250	0.4	5181-3323
Graphite/Vespel	320	0.5	5062-3514
Graphite/Vespel	530	0.8	5062-3512
Universal column nut for Agilent 6890 GC (2/pk)			5181-8830
Universal column nut for Agilent 6850 GC (2/pk)			5183-4732

* All ferrule part numbers represent a quantity of 10/pk.



See Page 2 for FID maintenance information.

Ask TECHNICAL SUPPORT

Now You See It, Now You Don't.

The Case of the Disappearing Peaks

When a customer installed a new column, keeping conditions identical, she noticed that critical analytes were disappearing. She assumed the column was defective, but there were other forces at work here.

By Eberhardt Kuhn, Ph.D.
Technical Support Chemist

Recently, a customer installed a new HP-5ms column in her instrument. She had been running the same application for some time on her old column, and set all the instrument parameters, such as head pressure, to the same values again. She immediately noticed that resolution between a pair of critical analytes had dropped from about 80% with the old column to less than 20% with the new column. This was unacceptable for accurate and reproducible quantitation, and the column was returned as "defective," along with supporting documentation.

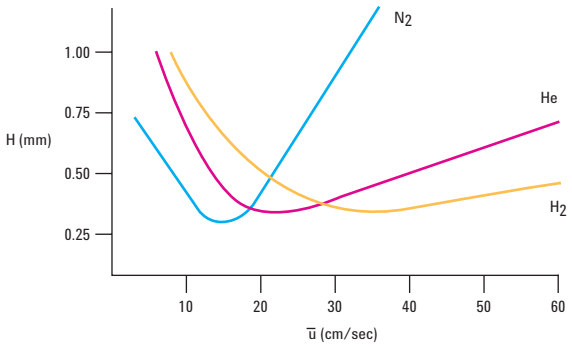
When examining the chromatograms closely, we noticed that the retention times of the poorly resolved peaks on the new column had shifted by over one minute earlier than the original method. This difference indicated a change in linear velocity of the carrier gas, as the run conditions were the same.

The Dutch chemist J.J. van Deemter established the relationship between carrier gas velocity and efficiency of a column (see Figure 1). As you can see, the number of theoretical plates, and thus resolving power, is greatly influenced by linear velocity. In this case, the resolution was indeed quite sensitive to carrier linear velocity. When we adjusted the carrier gas velocity to elute the two compounds at the same retention time as before, resolution was restored to the desired level, as shown in figures 2 and 3. The question remained: Why did the retention times of those compounds shift under identical run conditions?

Actually, the run conditions were NOT identical. This is because the columns were not truly identical. As with any manufacturing process, there are tolerances to which a product is built. In the case of a GC capillary column, these tolerances include diameter and length. And small differences in diameter and length will affect the actual velocity and flow rate through a column at a given head pressure. Gas chromatography instruments do not have a velocity and flow-measuring device built in. The linear velocity displayed is calculated from the head pressure and the column dimensions that the user inputs. If you use nominal values (30 m length, 0.25 mm diameter), the instrument automatically calculates flow and linear velocity data for a 30.00 m x 250.0 um column. Many columns do not have these exact dimensions, which makes it imperative to measure the actual linear velocity by injecting a non-retained compound (such as methane) and using its retention time and the

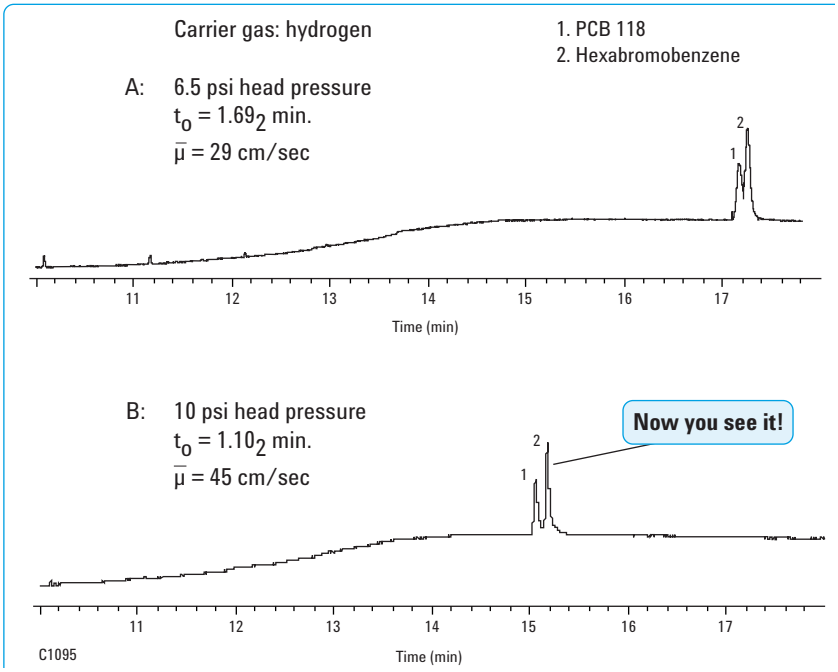
(Continued on Page 11)

Figure 1: van Deemter Curves for Common Carrier Gases*



* The y-axis shows the height of a theoretical plate (H), which is inversely proportional to the number of theoretical plates per meter. The smaller the H, the greater the efficiency of a column.

Figure 2: HP-5ms 30 m x 0.25 mm I.D. x 0.25 µm



Now You See It, Now You Don't.
Continued from Page 10

length of the column (see Equation 1) to calculate average linear velocity. The flow rate can also be calculated by incorporating the column cross-section into the equation (as shown in Equation 2).

Equation 1: $\bar{\mu} = L/t_o$

Equation 2: $\bar{F} = \pi r^2 L/t_o$

- $\bar{\mu}$ average linear velocity (cm/sec)
- $\bar{F}$ average flow rate (mL/min)
- L length of column in cm
- t_o retention time of non-retained peak
- r radius of column in cm

If you want to operate under truly identical conditions, you must adjust the column head pressure to match the retention of a non-retained compound from the previous column. Relying solely on instrument features, such as electronic pneumatics control (EPC), can lead to incorrect settings and cause problems.

To get a better feel for the magnitude of the effect that column dimensions have on the head pressure/linear velocity relationship, you can plug some numbers into the Agilent Flow Calculator, which you can download (for free) from our Web site: www.chem.agilent.com. Click on "Technical Support," then click on "User Contributed Software."

The results may surprise you.

Why Take an Agilent e-Seminar?

One way our GC experts share their expertise is by teaching e-Seminars. Pictured from left to right: Mitch Hastings, Jason Ellis, Cameron George, Jeanee Tollefson, Angelica Reese, Allen Vickers, and Eberhardt Kuhn.

- Agilent e-Seminars **take little of your time** while giving you so much in return.
- You and your co-workers can **take part in real-time,** interactive courses—wherever you are.
- This is an innovative and efficient way to **improve your skills** in the lab.
- Our expert Agilent technical support staff are among the instructors—all **industry leaders** and highly experienced teachers.
- 60- to 90-minute online seminars **keep you current** with today's latest technology.
- Agilent e-Seminars **convey expertise** gained from 35 years of chromatography leadership—as well as cutting-edge information.
- Helpful instructional materials sent to you when you register become **good reference** documents.
- You can **ask questions** online in a chat window or directly by phone during designated question and answer periods.
- Seminars are presented in **various languages.**
- **Plus Agilent e-Seminars are free.***

All you need is a telephone, Internet access and an Internet browser such as Microsoft Internet Explorer™ or Netscape Navigator™.

Practical and Highly Pertinent

Partial List of e-Seminar Topics

- Method validation and robustness testing
- GC column selection
- Characterization of surface metal contaminants
- Optimization of the Agilent 1100 LC system
- Impact of FDA's 21CFR11 validation guidance
- Low-bleed stationary phases for GC
- Instrument control and data acquisition
- Quality control of microarray samples
- Solutions for high sample throughput in HPLC
- Column maintenance and troubleshooting

100% Satisfaction

An online Agilent poll conducted after each e-Seminar reveals that 100% of those polled felt the e-Seminar was a valuable use of their time and they would attend another. One participant noted "It was very helpful and informative. I appreciate the time I saved in not having to physically move out of my office."

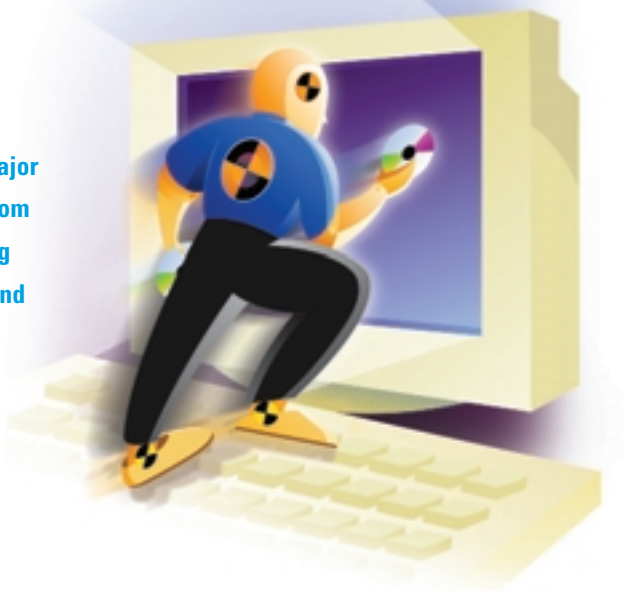
To view Agilent's e-Seminar schedule, get an account or register for a class, go to www.agilent.com/chem and click on "Education" in the left navigation bar. Then click on "e-Seminars."

* Long distance charges may apply. Separate phone and Internet lines are required to access the audio and Web portions of an e-Seminar simultaneously.

3 Reasons Why You Need Agilent's Software Backup Solution

Bounce Back Fast from a Crash

If your system has a hard-drive failure, major virus attack, serious user error, trouble from incompatible software or severe operating system crash, it can take days to reload and reconfigure all of your components.



With Agilent's Software Backup Solution, you have no reason to worry.

1. **You avoid laborious reconstruction after a system crash.** Agilent's Software Backup Solution for single-PC systems saves days of work recapturing all of your unique settings—including network information, printer and other peripheral configurations, user settings, operating system registry, application software settings, operating system customization, analytical hardware configuration, directory structure and security information.

2. **You get full-system protection.** After system installation, an Agilent service representative performs a full-system backup, capturing an exact image of your hard drive onto removable media. These CD-Rs and floppies contain all the information you need to recapture your original configuration, including the operating system, applications and all custom settings. The disks are safeguarded in a protective case along with simple instructions for restoring the system.

3. **You can recover quickly with just a few steps.** With this solution, you can restore your complete system and data—typically within two hours. All you have to do is make a few clicks and follow the easy directions shown on your monitor. If you have any problems, telephone or on-site support is available to help you.

You can get periodic backups to keep your data and configuration current. An Agilent service representative performing scheduled preventative maintenance can repeat the full backup procedure. This periodic backup preserves any new data, applications, upgrades or configuration changes added to your system after the last backup.

Note: This service is only for Agilent-approved PCs with specified Hewlett-Packard CD-Writer. The CD-Writer is not included with the basic solution but is available as an option, with installation included.

ORDER GUIDE

Product	Part Number
Software Backup Solution	H1782A (with or without CD-Writer)

Want to know more?

For more information about this solution:

- See Agilent publication 5988-6126EN
- Visit www.agilent.com/chem

Speeding Racehorse Drug Testing Using DB-5ms and GC/MS

- **Agilent's free method translation software enabled a shorter, smaller-diameter column.**
- **Run times decreased 35%.**
- **Elution order and peak and spectral quality remained the same.**
- **DB-5ms is a tough column for a tough sample.**

Wayne Skinner

Dan McKemie

Jason Ellis

By special guest authors Wayne Skinner, Dan McKemie, and Scott Stanley, Ph.D. (not pictured) of the California Animal Health and Food Safety Laboratory, Kenneth L. Maddy Equine Analytical Chemistry Laboratory, University of California at Davis, and **Jason Ellis,** Technical Support Chemist, Agilent

The rapid development of new analytical technology and applications has established GC/MS as the routine tool for problem solving in horse-racing laboratories—with a tremendous increase in sensitivity of testing. Yet this technique raises system stability and sample throughput complications for post-race analyses.

The Kenneth L. Maddy Equine Analytical Chemistry Laboratory (EACL) at the University of California at Davis replaced thin-layer chromatography with modern drug testing procedures.

At EACL, a vital part of method development was the incorporation of a faster chromatography method. The EACL had been using a 30 m x 0.25 mm, 0.25 µm film (5%-phenyl)-methylpolysiloxane column but wanted to integrate a 20 m x 0.18 mm, 0.18 µm film DB-5ms to improve laboratory throughput. Agilent's method translation software* allowed for easy translation of the existing method to the shorter, smaller-diameter column without affecting elution order. The shorter column with a smaller internal diameter decreased run times by 35% while maintaining the same peak and spectral quality.

This reduction in analysis time is critical, as the laboratory typically needs to screen 40 to 60 samples per instrument every night. Urine specimens are prepared by automated SPE prior to a screening run using an Agilent 6890 GC with a 5973 MSD in full-scan mode. Autosampler vials containing dried sample extracts are loaded into a CTC autosampler attached to the GC. On-line derivatization occurs when the autosampler dispenses derivatization reagent to the vial then injects the sample. This method makes use of an Apex ProSep™ PTV equipped with a packed injection port liner, which acts as a pre-column for the large volume injection (LVI). If a target compound is found through the screening process, a new aliquot of the sample is taken and run through a more specific cleanup method and re-analyzed.

Figure 1 shows a 200 ppb performance mix of 27 analytes used to monitor established method performance. The method calls for a solvent delay of 5.5 minutes to accommodate LVI, with the last analyte emerging from the column at 16.3 minutes. Most analytes form TMS derivatives; only mazindol and pentobarbitol are observed in both neat and TMS forms. Figure 2 shows a chromatogram of a quality control sample of equine urine fortified with 25 analytes at 50 ppb.

Horse urine specimens contribute a substantial amount of biological matrix. Nevertheless, the method and column have handled hundreds of injections while maintaining required performance and resolution. The DB-5ms has proven to be a rugged and reliable tool employed to maintain racing integrity.

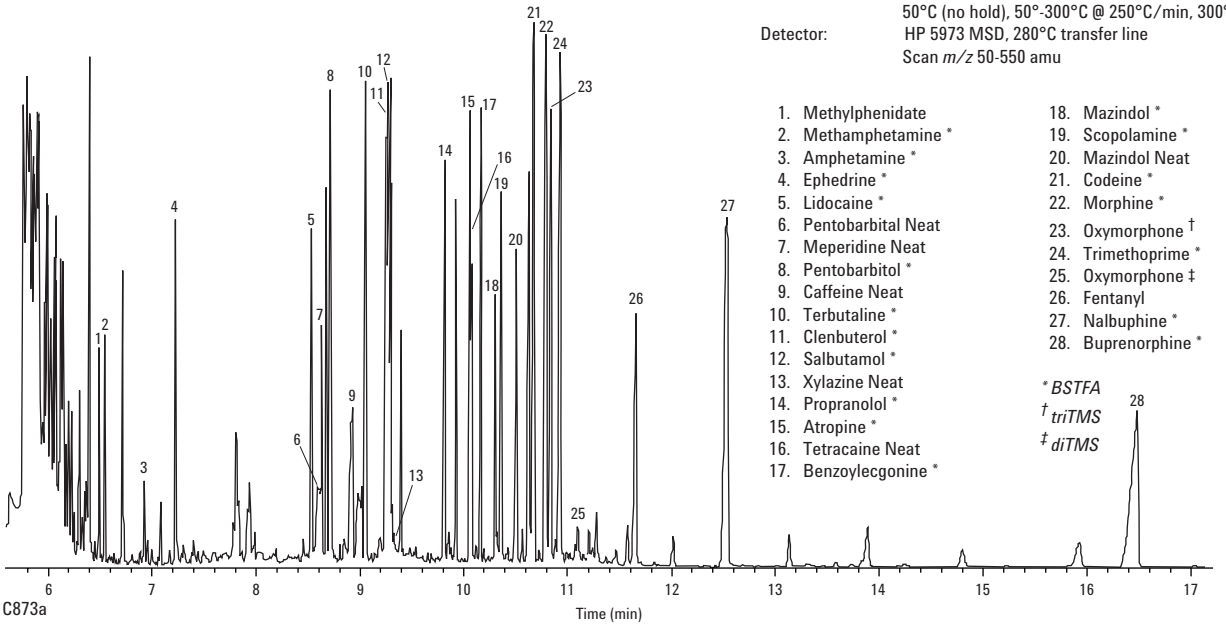
If you go to the races, you can feel confident that the race is fair—thanks in part to gas chromatography.

*Agilent's method translation software is available for free download in the Technical Support section of the Agilent Chemical Analysis Group Web site (www.agilent.com/chem). Select "Technical Support" then select "User Contributed Software." For more details on method translation, please contact Agilent technical support and request application note 5965-7673E: "Predictable Translation of Capillary Gas Chromatography Methods for Fast GC." Or download the note directly from our online literature library.

(Continued on Page 15)

Continued from Page 10

Figure 1. Standards performance check, each drug fortified at 200 ppb.

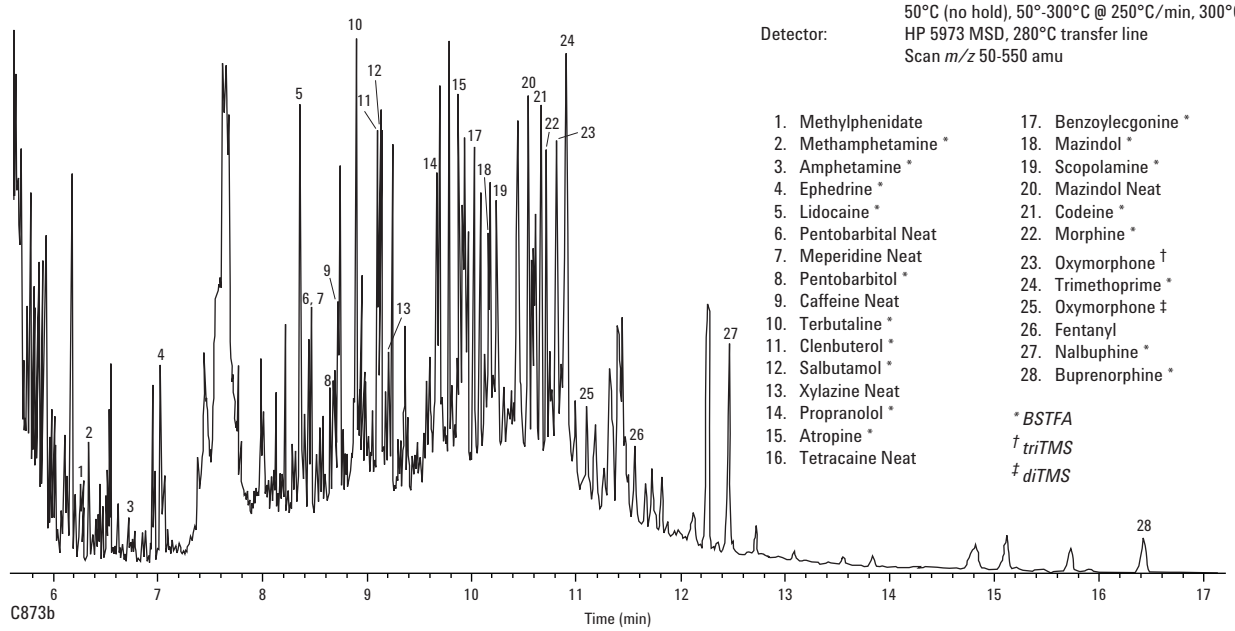


Column:	**DB-5ms**
	20 m x 0.18 mm I.D., 0.18 µm
Agilent Part No:	**121-5522**
Carrier:	Helium at 37 cm/sec, constant flow
Oven:	50°C for 3 min, 35°/min to 300°C, 300°C for 7 min
Injector:	APEX ProSep PTV injector, 40 µL injection size
	50°C (no hold), 50°-300°C @ 250°C/min, 300°C (16 min)
Detector:	HP 5973 MSD, 280°C transfer line
	Scan *m/z* 50-550 amu

1. Methylphenidate
2. Methamphetamine *
3. Amphetamine *
4. Ephedrine *
5. Lidocaine *
6. Pentobarbital Neat
7. Meperidine Neat
8. Pentobarbitol *
9. Caffeine Neat
10. Terbutaline *
11. Clenbuterol *
12. Salbutamol *
13. Xylazine Neat
14. Propranolol *
15. Atropine *
16. Tetracaine Neat
17. Benzoylecgonine *
18. Mazindol *
19. Scopolamine *
20. Mazindol Neat
21. Codeine *
22. Morphine *
23. Oxymorphone †
24. Trimethoprime *
25. Oxymorphone ‡
26. Fentanyl
27. Nalbuphine *
28. Buprenorphine *

* *BSTFA*
† *triTMS*
‡ *diTMS*

Figure 2. Equine urine quality control sample, each drug fortified at 50 ppb.



Column:	**DB-5ms**
	20 m x 0.18 mm I.D., 0.18 µm
Agilent Part No.:	**121-5522**
Carrier:	Helium at 37 cm/sec, constant flow
Oven:	50°C for 3 min, 35°/min to 300°C, 300°C for 7 min
Injector:	APEX ProSep PTV injector, 40 µL injection size
	50°C (no hold), 50°-300°C @ 250°C/min, 300°C (16 min)
Detector:	HP 5973 MSD, 280°C transfer line
	Scan *m/z* 50-550 amu

1. Methylphenidate
2. Methamphetamine *
3. Amphetamine *
4. Ephedrine *
5. Lidocaine *
6. Pentobarbital Neat
7. Meperidine Neat
8. Pentobarbitol *
9. Caffeine Neat
10. Terbutaline *
11. Clenbuterol *
12. Salbutamol *
13. Xylazine Neat
14. Propranolol *
15. Atropine *
16. Tetracaine Neat
17. Benzoylecgonine *
18. Mazindol *
19. Scopolamine *
20. Mazindol Neat
21. Codeine *
22. Morphine *
23. Oxymorphone †
24. Trimethoprime *
25. Oxymorphone ‡
26. Fentanyl
27. Nalbuphine *
28. Buprenorphine *

* *BSTFA*
† *triTMS*
‡ *diTMS*

Exclusive Offers

A Good Tip for **Extra Savings**

Having an Agilent service contract brings valuable benefits:

- **Maximum instrument uptime**
- **High system productivity**
- **Extended instrument life**
- **Fast problem resolution**

You have the flexibility to design your own support package or the convenience of selecting one of our economical pre-configured industry service bundles.

And now, as a service contract customer, you can get even more. Service contract customers are automatically eligible for our **Contract Plus Rewards Program**:

- Introductory offers on new products and services
- Incentives for purchases of laboratory supplies
- Discounts on selected Agilent products and training courses
- Advice on replacement strategies for older instrumentation
- Access to restricted areas of the Agilent Web site where you can find such useful aids as an extensive chromatogram database and how-to maintenance videos
- Notification of information that pertains to your specific interests

Simply by registering at **www.agilent.com/chem/rewards**, service contract customers receive exclusive e-mail notification of special offers designed just for them. If you already have a service contract, please contact your Agilent sales representative for your personal membership identification number.

Stay current!

Subscribe to *Separation Times.*
Online, go to
www. agilent.com/chem/supplies.
Under "Extras," click on
"What's New" then click
"Tap into Agilent's Expertise:
Subscribe to
Separation Times."

Agilent Technologies

Americas Field Support Center
2850 Centerville Road
Wilmington, Delaware 19808-1644

Printed in USA April 1, 2002
5988-5880ENCA