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The Reporter

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For Durability and Low Bleed, Equity Meets the Challenge

In many of today's analytical laboratories, the trend is towards greater sample throughput while achieving lower detection levels. For most GC analyses, this translates to running an increased number of samples daily while using highly sensitive detectors such as a mass selective detector (MSD). It is important that the column chosen for the analysis be both durable and exhibit low bleed. We evaluated both the Equity-5 and Equity-1 for durability using a special challenge test. We also evaluated the columns for bleed with an MSD. Both evaluations found that the new Equity-5 and Equity-1 met these challenges.

Durability Challenge

A durability test was designed to challenge column performance in a situation that often exists in today's analytical testing laboratory. The test consisted of over 60 runs total, made by alternating injections of a 1µL performance evaluation mix with 5 consecutive 2µL splitless injections of a solid waste extract sample. At the end of the test, column and injector maintenance was performed and a final run was made of the performance mix. The performance evaluation mix contained several very active probes, including pentachlorophenol and benzoic acid. The response factors of each probe were calculated relative to an internal standard (2,2'-difluorobiphenyl.) The Equity-1 was found to maintain very consistent response through the test. The Equity-5 experienced some loss in response, but recovered well after clipping one loop from the column (Figure A). Pentachlorophenol can be especially tough to recover. The chromatography of this compound after column maintenance was found to be acceptable on both columns. The results from the Equity-5 are presented in Figure B.

Bleed Challenge

High sensitivity detectors require the use of stable columns with low bleed levels. We evaluated the performance of the Equity-5 and Equity-1 with an MSD by injecting a low-level standard and examining the quality of the spectrum of the last eluting compound, benzo(ghi)perylene. The mass spectra of eluting peaks should contain minimal interference from extraneous ions produced by the column's phase. This is important for proper identification of the compound. A five nanogram standard was injected on both the Equity-5 and Equity-1. Both columns exhibited low bleed and clean spectra for benzo(ghi)perylene. The primary ion

GC issue

Figure A. Challenge Test on an Equity-5

Column: **30m x 0.25mm ID, 0.25µm**
Cat. No. **28089-U**
Oven: 35°C (4 min) to 325°C @ 10°C/min (15 min)
Inj.: 250°C
Det.: FID, 360°C
Flow: 27psi constant pressure
Injection: 1.0µL, 100:1 split of test mix
Sample: Test Mix – 2.5 to 5ng on-column of a 16 component semivolatile standard

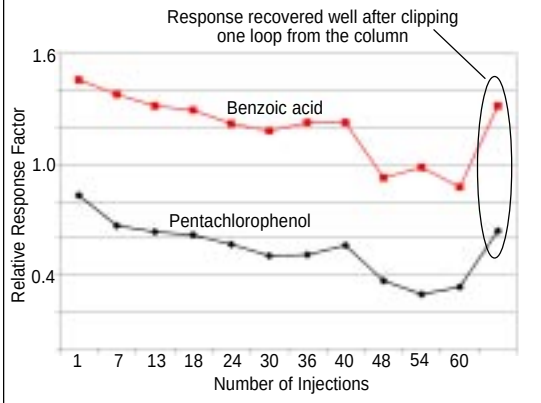

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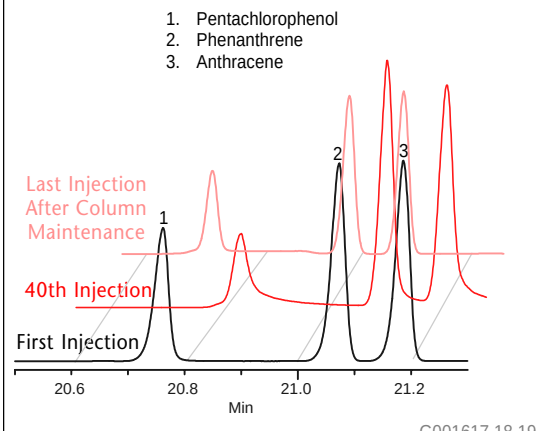
Figure B. Chromatography on the Equity-5 Before, During and After the Durability Challenge Test



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SUPELCO
a member of the Sigma-Aldrich family

T202008

Equity Capillary GC Columns

Equity Capillary GC Columns

The performance you demand…the service you deserve…from the company you trust.

Supelco's new Equity Capillary GC column line has been expanded to include more of the column dimensions that you need for your nonpolar applications. New dimensions that are available include 0.1mm and 0.2mm ID columns, along with new column lengths for our standard 0.25, 0.32, and 0.53mm ID columns. The new smaller ID columns are ideal for analysts preferring fast GC applications. We have enhanced the performance of all Equity Capillary GC columns due to significant improvements in the polymer chemistry. The polymer improvements result in better bonding, higher thermal stability, and superior product reproducibility. If you use a non-polar column, try our new Equity line of improved capillary columns.

For more information request T402049.

Equity-1 Capillary GC Columns

Phase: bonded; poly(dimethylsiloxane)
Temp. Limits: 0.25 and 0.32mm ID: -60°C to 325/350°C
0.53mm ID: -60°C to 300/320°C (≤1.5µm df)
-60°C to 260/280°C (>1.5µm df)

Length (m)	df (µm)	Beta	Cat. No.
0.10mm ID			
15	0.10	250	**28039-U**
0.20mm ID			
12	0.33	152	**28041-U**
25	0.33	152	**28042-U**
10	1.2	42	**28043-U**
0.25mm ID			
30	0.10	625	**28044-U**
15	0.25	250	**28045-U**
30	0.25	250	**28046-U**
60	0.25	250	**28047-U**
15	1.0	63	**28048-U**
30	1.0	63	**28049-U**
60	1.0	63	**28050-U**
100	1.0	63	**28052-U**
0.32mm ID			
30	0.10	800	**28053-U**
15	0.25	320	**28054-U**
30	0.25	320	**28055-U**
60	0.25	320	**28056-U**
30	1.0	80	**28057-U**
60	1.0	80	**28058-U**
100	1.0	80	**28060-U**
30	2.0	40	**28061-U**
30	5.0	16	**28062-U**
60	5.0	16	**28063-U**
0.53mm ID			
15	0.10	1325	**28064-U**
30	0.10	1325	**28065-U**
15	0.5	265	**28067-U**
30	0.5	265	**28068-U**
15	1.0	133	**28069-U**
30	1.0	133	**28071-U**
15	1.5	88	**28072-U**
30	1.5	88	**28073-U**
60	1.5	88	**28074-U**
15	3.0	44	**28075-U**
30	3.0	44	**28076-U**
60	3.0	44	**28077-U**
15	5.0	27	**28079-U**
30	5.0	27	**28081-U**
60	5.0	27	**28082-U**

Equity-5 Capillary GC Columns

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)
Temp. Limits: 0.25 and 0.32mm ID: -60°C to 325/350°C
0.53mm ID: -60°C to 300/320°C (≤1.5µm df)
-60°C to 260/280°C (>1.5µm Df)

Length (m)	df (µm)	Beta	Cat. No.
0.10mm ID			
15	0.10	250	**28083-U**
0.20mm ID			
15	0.20	250	**28084-U**
30	0.20	250	**28085-U**
60	0.20	250	**28086-U**
12	0.33	152	**28087-U**
0.25mm ID			
15	0.25	250	**28088-U**
30	0.25	250	**28089-U**
60	0.25	250	**28090-U**
30	0.5	125	**28092-U**
15	1.0	63	**28093-U**
30	1.0	63	**28094-U**
60	1.0	63	**28095-U**
0.32mm ID			
15	0.25	320	**28096-U**
30	0.25	320	**28097-U**
60	0.25	320	**28098-U**
30	0.32	250	**28099-U**
30	0.5	160	**28195-U**
30	1.0	80	**28199-U**
60	1.0	80	**28251-U**
0.53mm ID			
15	0.5	265	**28252-U**
30	0.5	265	**28259-U**
60	0.5	265	**28263-U**
30	1.0	133	**28264-U**
15	1.5	88	**28265-U**
30	1.5	88	**28267-U**
30	3.0	44	**28268-U**
60	3.0	44	**28269-U**
15	5.0	27	**28278-U**
30	5.0	27	**28279-U**
60	5.0	27	**28293-U**

*All literature mentioned in this issue can be obtained from the website, **sigma-aldrich.com/TheReporter**, by completing the Literature Request section on the reply card, or by calling our Technical Service Dept.*

NEW PRODUCTS (contd.)

CAP Kits for Agilent, PerkinElmer, and Varian GCs

Supelco CAP Kits are designed to reduce the risk of chromatographic problems and instrument downtime. Each CAP Kit contains a Supelco capillary GC column of your choice and the high performance accessories required for your specific gas chromatograph. Our high performance accessory line includes preconditioned, low bleed septa, high temperature ferrules, inlet seals of high purity metal, and inert glass liners. We offer CAP Kits for Agilent, PerkinElmer, and Varian instruments. A free accessory case is available for each CAP Kit ordered during this introduction.

For more information request T402020, T402021, and T402022.

P000919

Description	Cat. No.
GC Accessory Case for Agilent Technologies	**28035-U**
GC Accessory Case for Varian	**28036-U**
GC Accessory Case for PerkinElmer	**28037-U**

LITERATURE

Equity Capillary GC Columns Update

Our 32-page Equity Capillary GC Column brochure has been updated with analytical information relevant to today's analysts working within the clinical, environmental, food & beverage, and pharmaceutical markets. Twenty-five new applications are presented on our improved nonpolar Equity capillary GC columns including solvents, fuel oils, phenols, drugs of abuse, and blood alcohol analyses. Equity-1 and Equity-5 columns offer the consistent resolution, analyte response, low bleed, and column life you demand for your nonpolar applications.

For more information request T402049.

SPME of Odors in Drinking Water Update

Method 6040D, developed by the American Water Works Association (AWWA), describes the use of SPME for the determination of geosmin, methylisoborneol (MIB), isopropylmethoxypyrazine (IPMP) and isobutylmethoxypyrazine (IBMP) in drinking water. SPME offers a simple-to-use and cost effective technique to analyze odor compounds when used in conjunction with a high resolution, low bleed Equity-5 capillary column. This application note has been updated to include details of the new AWWA Method 6040D and to provide data demonstrating achievable linearity when analyzing odor compounds present at 1-10ppt.

For more information, request T398147.

SEMINAR

Equity Capillary GC Columns

This seminar describes the benefits of the new Equity-1 and Equity-5 improved non-polar capillary GC columns. The presentation focuses on the critical performance factors of resolution, analyte response, low bleed, and column life and how Equity columns deliver the performance you demand for these critical factors. Included in the presentation are many applications demonstrating the Equity column performance.

For more information request T402055.

GC PERFORMANCE TIP

Reduction of Water and Methanol Improves Purge and Trap Analyses

Water and methanol can cause problems with the analysis of VOCs using purge and trap units. Water causes baseline disturbances in mass spec systems that can result in elevated area counts. Methanol interference may cause improper quantification of gaseous VOCs particularly bromomethane. Minimizing the amount and the effects of water and methanol can reduce downtime and reanalysis of samples.

Purge traps containing silica gel will release a large amount of water due to the high affinity of silica gel for hydroxylated compounds. Likewise charcoal with tapered, closed pores retains more water and methanol than other carbon molecular sieves. Hydrophobic traps, such as the VOCARB line, retain less water but elute water and methanol more sharply than charcoal and silica gel, so it appears that more water is being released.

To reduce interferences from water, desorb the trap for a shorter period of time. One to 2 min. is generally sufficient. This reduces the amount of water carried over to the column. After desorption, bake the column for at least 6 minutes to adequately remove the remaining water. If you lower the transfer line and valve temperatures to 120°C, this will reduce the impact of water on the chromatography.

Reduce methanol interferences by concentrating your standards and spiking with smaller volumes. Use wire in the needle syringes for good accuracy. If you are not analyzing for gaseous VOCs, do not use a purge trap containing a molecular sieve. Purge traps without sieves will retain much less water and methanol than traps with a sieve or charcoal.

For Durability and Low Bleed...

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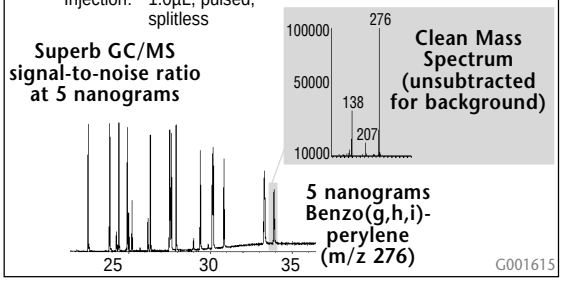
used for identification of this compound (m/z=276) was of significantly higher response than the ion resulting from column bleed (m/z=207). The results of this bleed challenge from the Equity-5 are presented in Figure C.

This data illustrates how the new Equity-5 and Equity-1 columns meet the durability and low bleed challenges presented by many of today's demanding applications. Equity will provide the consistent column life you count on and low bleed levels you expect for success with your nonpolar applications.

For more information, request T402049.

Figure C. GC/MS Bleed on an Equity-5

Column: **30m x 0.25mm ID, 0.25µm**
Cat. No. **28089-U**
Oven: 40°C (3 min) to 100°C @ 10°C/min (2 min) to 325°C @ 8°C/min
Inj.: 250°C
MSD Interface: 325°C
Flow: 14.4psi constant pressure w/pulse to 40psi until 0.25 min
Injection: 1.0µL, pulsed, splitless



CASE STUDY 4

Improving Volatiles Analysis

Background

In purge and trap analysis of volatiles, the flow passing through the purge trap during desorption passes directly to the analytical column. The higher the flow rate through the trap, the more rapidly analytes will be desorbed, and the better the resulting chromatography. Chemists doing this application with a mass selective detector (MSD) are faced with a special dilemma when using smaller ID columns. Traditional set-ups have included a 0.53mm ID column, run in direct or splitless mode, and a GC/MSD equipped with a jet separator. This column dimension requires high flow rates (5-10mL/min). Most MSDs, however, are not compatible with flow rates above 4mL/min. To eliminate this problem, the jet separator reduces the flow before it enters the detector. Smaller ID columns such as 0.32 or 0.25mm do not require flows > 3mL/min, so there is no need for a jet separator. The lower flow results in slower desorption of analytes from the purge trap. This results in broad peaks and tailing. Figure D illustrates a splitless analysis of a typical volatiles mix on a 60m x 0.32mm ID x 1.8µm SPB-624 column.

Solution

A split injection allows a faster desorption flow rate to be used. Figure E illustrates the results of using a split injection. The flow through the trap during desorption was 22mL/min. The flow was then split 18:1 in the GC inlet. This resulted in a faster transfer of analytes to the column, sharper peaks, and better sensitivity. A low volume liner should be used to minimize dead volume in the inlet. The 0.75mm ID SPME liner works well for this purpose.

For more information request T197916.

Figure D. Splitless Analysis of Volatiles

Column: **SPB-624, 60m x 0.32mm, 1.8µm film**
Cat. No.: **24251**
Oven: 35°C (8 min) to 100°C at 5°C/min to 220°C at 15°C/min, hold 3 min
Carrier: Helium, 3.5mL/min
Det.: MSD, scan range 35-300 amu
Inj.: Purge 11 min, dry purge 3 min, desorb 235°C, 2 min, splitless
Sample: 100ppb @ in 5mL water
Trap: VOCARB 3000

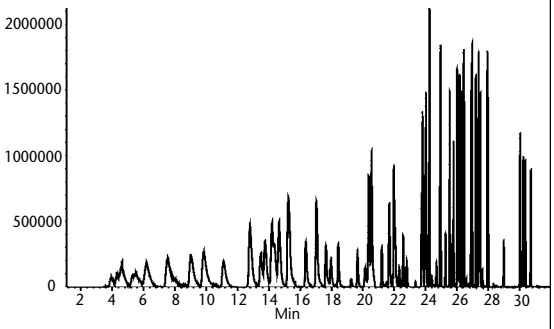


Figure E. Split Analysis of Volatiles (split 18:1 at GC inlet)