

New Equity Capillary GC Columns: The Resolution You Need and the Analyte Response You Require

■ A widespread and demanding application that requires the use of a nonpolar capillary column is the analysis of solid waste samples for semivolatile organic compounds by US EPA Method 8270. This method utilizes GC/MS for the trace analysis of some 240 compounds of varying classes with a wide range of functionalities. To meet the stringent demands of this method it is imperative that the analytical system be optimized, including the capillary GC column chosen to perform the analysis.

Resolution Challenges

Most laboratories performing US EPA Method 8270 typically analyze a list of 80-100 compounds in a single run. Analyzing such a long compound list in a single run presents special challenges with regards to resolution. The mass spectrometer resolves many coeluting pairs based on spectral differences. However, compounds with the same or similar mass spectra still require chromatographic resolution for proper analysis. An example are the commonly analyzed and closely eluting isomers benzo(b)fluoranthene and benzo(k)fluoranthene. These compounds have the same mass spectra and are quantified using the same ion ($m/z=252$.) In order for most GC/MS data systems to perform proper identification and quantification, these isomers must be adequately resolved. Figure A shows the resolution of benzo(b)fluoranthene and benzo(k)fluoranthene in a 50ng standard on the 30m x 0.25mm ID x 0.50 μ m Equity-5 column. The column was able to achieve this separation in a run time of < 23 minutes. Similar resolution was achieved in a 160ng standard as well. In both cases, the resolution achieved was sufficient for proper identification and quantification by the GC/MS data system.

Response Challenges

Performing US EPA Method 8270 presents special challenges with regards to response. A typical target list includes both acidic and basic compounds, with many being quite active and difficult to achieve good response at low levels. In addition, calibration curves are often run as low as 10ng on-column. Achieving good response for active compounds such as benzoic acid, 2,4-dinitrophenol and pentachlorophenol at this low level can be especially difficult. Assuming the GC inlet and mass spectrometer have been properly maintained and

Figure A. Resolution of Closely Eluting Isomers on the Equity-5

Inj. Temp.: 280°C
MS Interface Temp.: 325°C
Oven: 40°C, 2 min; 50°C/min to 100°C;
10°C/min to 200°C; 30°C/min to 325°C
Flow: (He) 20psi, 0 min; to 80psi; to 16.5psi,
3 min.; to 25psi
Sample: 1 μ L, splitless, 50ng

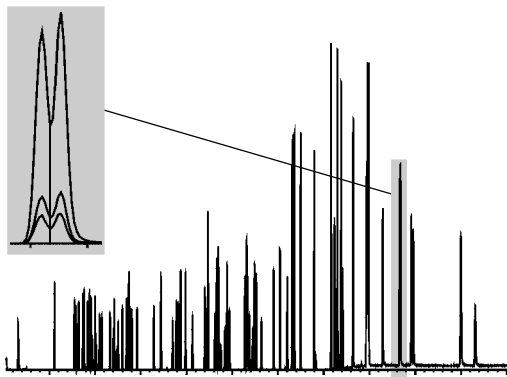
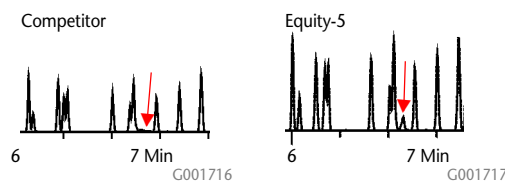


Figure B. Benzoic Acid 10ng on Equity-5 and a Competitor's Column



are optimized for the analysis, the remaining requirement for success is a column with a high level of inertness. The Equity-5 achieves this goal for the difficult to analyze semivolatile compounds. Figure B illustrates the response and peak shape of benzoic acid at 10ng on-column. For comparison, the same analysis was also performed on another brand of capillary column commonly used for this application. The response of benzoic acid was so poor on the competitor's column that the data system used did not detect it. In this case, the analyst would be required to manually identify and integrate the peak. Table 1 compares the response factors for several problematic compounds at 10ng on-column

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Equity Capillary GC Columns



NEW PRODUCTS

Equity Capillary GC Columns

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

Supelco's new and improved Equity Capillary GC Columns deliver the capillary GC column performance you demand for your nonpolar applications. Significant improvements in the polymer chemistry are at the heart of the enhanced performance you will receive with our new Equity Capillary GC Columns.

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 GC 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)	Cat. No.
0.25mm ID	15	0.25	28045-U
	30	0.25	28046-U
	15	1.0	28048-U
0.32mm ID	30	1.0	28049-U
	15	0.25	28054-U
	30	0.25	28055-U
0.53mm ID	30	1.0	28057-U
	15	1.5	28072-U
	30	1.5	28073-U
	15	3.0	28075-U
	30	3.0	28076-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)	Cat. No.
0.25mm ID	15	0.25	28088-U
	30	0.25	28089-U
	30	0.5	28092-U
0.32mm ID	15	0.25	28096-U
	30	0.25	28097-U
	15	0.5	28252-U
0.53mm ID	30	0.5	28259-U
	15	1.5	28265-U
	30	1.5	28267-U
	15	5.0	28278-U
	30	5.0	28279-U

Syringes for CTC Autosamplers



FEATURED PRODUCTS

Syringes for CTC Autosamplers

A comprehensive listing of syringes for CTC Autosamplers has been added to our Hamilton line of Autosamplers. These new syringes are specifically for the CTC Autosamplers. We offer a selection for the A200S, PAL Instruments for GC Sampling, the COMBI PAL for GC Headspace sampling, and the HTS PAL Instruments for HPLC sampling. These syringes come with a 5mm length needle in a variety of gauges and volumes. We have included both Microliter™ and GASTIGHT® syringes for your convenience.

For more information request T402117.

Hamilton Syringes for CTC/Leap Technologies Autosamplers

Capacity	Model	Gauge	Length	Pt.	Cat. No.
GC Sampling: A200S, PAL Instruments					
<i>Modified Microliter</i>					
1.2µL ...	7701.2	26g	51mm	#1	28617-U
<i>Microliter Syringe, Fixed Needle</i>					
5µL ...	75N	26s	51mm	#1	28613-U
10µL ...	701N	26s	51mm	#2	28614-U
10µL ...	701N	26s	51mm	#1	28615-U
<i>GASTIGHT Syringe, Fixed Needle</i>					
25µL ...	1702N	26s	51mm	#1	28649-U
100µL ...	1710N	26s	51mm	#1	28651-U
250µL ...	1725N	26s	51mm	#1	28652-U
500µL ...	1750N	26s	51mm	#1	28653-U

GC Headspace Sampling: COMBI PAL

<i>GASTIGHT Syringe</i>					
1mL ...	1000LTN	23	56mm	#5	28621-U
1mL ...	1001LTN	26	56mm	#5	28622-U
2.5mL ...	1002LTN	23	56mm	#5	28626-U
2.5mL ...	1002LTN	26	56mm	#5	28627-U
5mL ...	1005LTN	23	56mm	#5	28628-U
5mL ...	1005LTN	26	56mm	#5	28629-U

HPLC Sampling: HTS PAL Instruments

<i>Microliter Syringe, Fixed Needle</i>					
10µL ...	701N	22s	51mm	#3	28618-U
<i>GASTIGHT Syringe</i>					
10µL ...	1701N	22s	51mm	#3	28632-U
25µL ...	1702N	22s	51mm	#3	28633-U
100µL ...	1710N	22s	51mm	#3	28634-U
100µL ...	1710N	22	51mm	#3	28635-U
250µL ...	1725N	22	51mm	#3	28636-U
1mL ...	1001LTN	22	56mm	#3	28637-U
2.5mL ...	1002LTN	22	56mm	#3	28638-U
5mL ...	1005LTN	22	56mm	#3	28639-U

Replacement Plungers

Model	Description	Cat. No.
1701CTC	for 10µL CTC Syringe	28641-U
1702CTC	for 25µL CTC Syringe	28642-U
1710CTC	for 100µL CTC Syringe	28643-U
1725CTC	for 250µL CTC Syringe	28644-U
1750CTC	for 500µL CTC Syringe	28645-U
1001CTC	for 1mL CTC Syringe	28646-U
1002CTC	for 2.5mL CTC Syringe	28647-U
1005CTC	for 5mL CTC Syringe	28648-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.

FEATURED PRODUCTS (contd.)

Hamilton and SGE Syringes for Autosamplers

We offer a large selection of syringes from Hamilton and SGE for your autosampler needs. Both single gauge and dual gauge needles are available. We offer both fixed needle and removable needle in a range of needle styles for your application. The needles are narrow-bore stainless steel to minimize liquid dead volume. The following are our most popular autosampler part numbers in the Hamilton and SGE lines.

☎ For more information request T402117.

Syringes for Autosamplers

Capacity	Model	Gauge	Point	Cat. No.
Hamilton				
10µL ...	701ASN (6 pk)	23s	#1	21317
10µL ...	701 ASN (6 pk)	23s-26s	#1	24574
10µL ...	701N (6 pk)	26s	#2	20779
SGE				
5µL ...	SK-5F-HP-0.63 (6 pk)	23	#1	21911
10µL ...	SK-10F-HP-0.63 (6 pk)	23	#1	21544
10µL ...	10FX	26	#2	23966

SEMINARS

Equity Capillary GC Columns

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

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.

LITERATURE

Equity Capillary GC Columns

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

This brochure introduces Supelco's new line of improved Equity non-polar capillary columns. Equity-1 and Equity-5 columns offer the consistent resolution, analyte response, low bleed, and column life you demand for your non-polar applications.

☎ For more information request T402049.

SPME of Odors in Drinking Water Update

A new method involving the use of Solid Phase Microextraction (SPME) has been developed for the analysis of odor compounds in drinking water at parts per trillion (ppt) concentration for analysis by gas chromatography/mass spectrometry (GC/MS). 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. In addition, ten tips for good quantification and low detection limits are presented.

☎ For more information, request T398147.



GC PERFORMANCE TIP

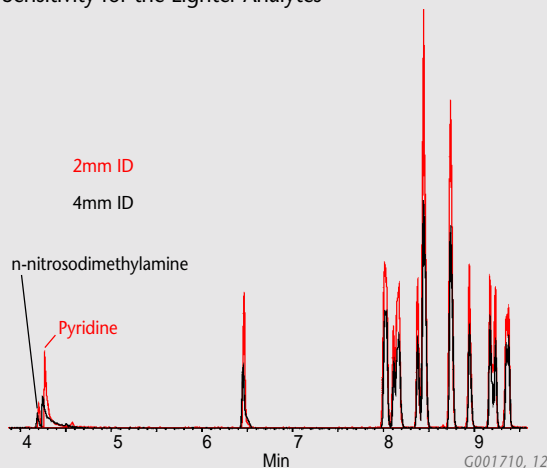
Choosing the Correct Splitless Liner

One of the more popular GC inlet liner dimensions is the 4mm ID. It is frequently used for less volatile, high and low molecular compounds. Other liner choices are also available which can help optimize your analysis for these types of compounds. If your application involves trace analysis and includes somewhat volatile components, consider the use of a 2mm ID liner. This type of liner is recommended for splitless injections of less than 2µL. The reduced volume of this ID liner increases the linear velocity of the carrier gas through the liner. This produces a more rapid introduction of analytes onto the column in a narrow band. The improved focussing provides a better response, especially for lighter analytes. Figure D illustrates the difference in response obtained with a typical semi-volatile standard run on the same column, in the same GC system, but using a 4mm ID liner vs. a 2mm ID liner. Note the difference in response, especially for the peaks pyridine and n-nitrosodimethylamine. Standard practice for this application usually involves the use of a 4mm ID liner. The chromatograms, in Figure D, show how the use

of a 2mm ID liner will increase sensitivity for the lighter analytes.

☎ For more information, request T196899.

Figure D. The Use of a 2mm ID Liner will Increase Sensitivity for the Lighter Analytes



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Gastight, Hamilton, Microliter - Hamilton Co.

Equity, Supelco - Sigma-Aldrich Co.

SPME-Technology licensed exclusively to Supelco. US patent #5,691,206; European patent #523092.

New Equity Capillary GC Columns:...

(continued from page 1)

between the Equity-5 and the competitor's column from Figure B. Overall, the Equity-5 was found to have better response.

The above examples clearly illustrate the ability of the new Equity-5 column to provide the resolution you require and the response you expect for demanding applications. Its ability to resolve closely eluting isomers and provide acceptable peak shape and response at low levels make it a good choice for successfully running semivolatile methods such as US EPA Method 8270.

For more detail see sigma-aldrich.com/thereporter or request T402049.

Table 1: Response Factors at 10ng on-column; Equity-5 vs. Competitor's Column

Compound Name	Equity-5	Competitor
Benzoic Acid	.074	.035
2-Nitrophenol	.164	.137
2,4-Dinitrophenol	.049	.034
2-Methyl-4,6-dinitrophenol	.063	.033
Pentachlorophenol	.079	.052

CASE STUDY 3

Detecting Trace Levels of Odor Compounds

Background

During warm weather, musty odors are often noticed in the water systems. These odors are caused by the presence of blue green algae. Because the human nose can detect these odors at <5ppt, water utilities must monitor for the odor compounds down to <1ppt. A simple, reliable analytical method is needed to routinely detect these odors at 1ppt.

Solution

A method using solid phase microextraction (SPME) offers a simple and reliable means of detecting trace organic compounds, including odor compounds. A 2cm DVB-Carboxen SPME fiber has high extraction efficiency for these analytes. The fiber rapidly releases them when desorbed in the hot injection port. Detection levels at 1ppt can be obtained when the fiber is used in conjunction with a high-resolution, low bleed capillary column connected to a mass spectrometer. The Equity-5 column meets the demanding requirements for this analysis. For low detection limits, single ion mode must be used for quadrupole instruments. A narrow mass range is needed for ion traps.

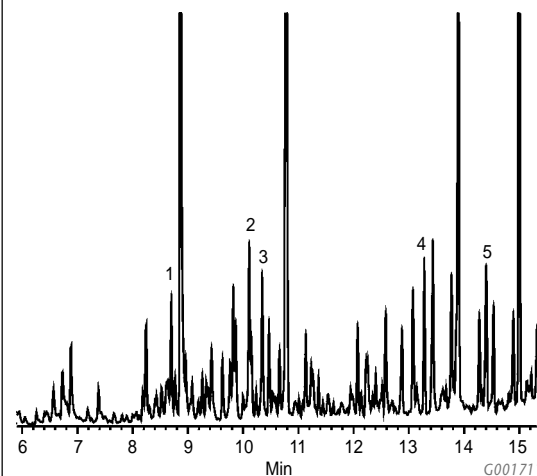
Figure C shows the analysis of common odor components at 2ppt each in water with an internal standard of 2,4,6-trichloroanisole at 8ppt. Good resolution is needed to separate MIB from IPMP for proper quantification. Ion 95 is used to quantify MIB when working with mass spectrometry. Therefore, it must be resolved from siloxane impurities that commonly contain this ion. The Equity-5 column is ideal for this type of analysis with its stable baseline and consistent low bleed.

For detailed information on method linearity, optimizing extraction and analysis conditions, and availability of odor standards, please ask for T398147.

Figure C. Trace Odors in Drinking Water (SPME/GC)

Column: Equity-5, 30m x 0.25mm, 0.25µm film, Cat. No.: 28089-U
SPME Fiber: 2cm StableFlex coated with 50/30µm DVB/Carboxen/PDMS, Cat. No. 57348-U
Extraction: headspace, 65°C (30 min.)
Desorption: 3 min. at 260°C
Oven: 60°C (2 min) to 200°C at 8°C/min
GC Liner: 0.75mm SPME liner
Detector: MS, selected ions (SIM) 95, 112, 124, 137, 197; interface at 280°C
Flow: Helium, 37cm/sec @ 60°C (1mL/min constant flow)
Inj.: SPME fiber, splitless opened at after 1 min at 50mL/min.
Sample: 25mL of water containing 25% NaCl and drinking water odors kit, Cat. No. 46729-U

1. 2-Isopropyl-3-methoxy-pyrazine (IPMP), 2ppt
2. 2-Isobutyl-3-methoxy-pyrazine (IBMP), 2ppt
3. 2-Methylisoborneol (MIB), 2ppt
4. 2,4,6-Trichloroanisole (Internal Standard), 8ppt
5. (±)Geosmin, 2ppt



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