Application Brief

Environmental

Agilent

Trusted Answers

Selecting a GC Liner to Decrease Column Trim Frequency

Author

Angela Smith Henry, PhD
Agilent Technologies, Inc.

Abstract

GC inlet liner selection is an important part of reduced column maintenance. A liner with a frit or wool can trap the nonvolatile matrix of injected samples compared to an open, barrierless liner, such as a single taper liner. Using an EPA 8270 style analysis, three liner styles were evaluated with a series of soil matrix injections. The evaluation was used to determine the required frequency of column trimming on an Agilent 8890 gas chromatograph (GC) and 5977B Inert Plus mass spectrometer (MS). Single taper liners required a column trim three times more often than sintered glass fritted liners and glass wool packed liners.

Introduction

The performance of EPA 8270 analyses depends on GC inlet and column performance. The GC inlet liner selection is an important part of reduced column and system maintenance and better column performance. Inlet liner selection was evaluated with a series of soil matrix injections using three liner styles. The evaluation was used to determine the needed frequency of column trimming on a 8890 GC and 5977B Inert Plus MS with repeated injections.

Experimental

The 8890 GC was configured with a single MS flow path interfaced with an extractor electron ionization (EI) source and a 30 m Agilent J&W DB-5ms Ultra Inert column. The 5977B GC/MSD was installed with a 9 mm drawout plate. Table 1 summarizes GC method settings, consumables, and MS conditions. Table 2 lists the tested liners.

Using EPA method 8270 as the basis for this testing, a tuning standard and representative standard were used for MS calibration, tuning settings, and quality control. The tuning standard (part number GCM-154-1) containing a mixture of benzidine, pentachlorophenol, 4,4'-diphenyltrichloroethane (4,4'-DDT), and decafluorodiphenyltrichloroethane (DFTPP) was diluted to 25 µg/mL in dichloromethane (DCM) and used as a quality control (QC) check. The EPA 8270 short mixture (part number 5191-3905) was diluted to 5 µg/mL, and 40 µg/mL of deuterated-polycyclic aromatic hydrocarbons (d-PAHs) were added as internal standards. The EPA 8270 short mixture is comprised of the more challenging and active compounds, such as 2,4-dinitrophenol, benzoic acid, and 3,3'-dichlorobenzidine, and resolution pair of benzo(b) fluoranthene and benzo(k)fluoranthene.

Table 1. GC and MSD instrument conditions and consumables.

Parameter	Value
Injection Volume	1 µL
Inlet	Split/splitless 280 °C Pulsed splitless 30 psi until 0.6 min Purge 50 mL/min at 0.6 min Switched septum purge 3 mL/min
Column	Agilent J&W DB-5ms UI, 30 m × 0.25 mm, 0.25 µm (p/n 122-5532UI)
Column Temperature Program	40 °C (hold for 0.5 min), 10 °C/min to 100 °C, 25 °C/min to 260 °C, 5 °C/min to 280 °C, 15 °C/min to 320 °C (hold 2 min)
Carrier Gas and Flow Rate	Helium at 1.30 mL/min, constant flow
Transfer Line Temperature	320 °C
Ion Source Temperature	300 °C
Quadrupole Temperature	150 °C
Scan	*m/z* 35 to 500
Gain Factor	0.4
Threshold	0
A/D Samples	4

Table 2. Liner styles and abbreviated names used throughout text.

Liner Information	Name to be Used in Text
Agilent Ultra Inert Splitless Low Fritted Liner (p/n 5190-5112)	Fritted liner
Agilent Ultra Inert Splitless Single Taper with Wool Liner (p/n 5190-2293)	Wool liner
Agilent Ultra Inert Splitless Single Taper Liner (p/n 5190-2292)	Single taper liner

A composite mixture of soils extracted with dichloromethane was prepared for method 8270 analysis. The mixture was a representative matrix residue that is typically encountered in the lab and was procured from Pace Analytical (Mt. Juliet, TN).

A sequence of injections was run to determine the working lifetime of the liners and columns, which have been described in previous application notes as parts of matrix studies.[1,2] The sequence of injections includes a blank injection of dichloromethane, GC/MS tuning standard, EPA 8270 short mixture, and 10 replicate soil matrix injections. The sequence is repeated twice before the analysis is paused to check performance criteria and determine if maintenance is required.

Results and discussion

Based on previous work, it was noted that DDT percent breakdown tended to cause QC to fail first. Failures were due to matrix build-up in liners and required liner replacement.[1,2] Pentachlorophenol and benzidine tailing factors could also cause QC failure and typically indicated the need for a column trim. Additionally, the EPA 8270 short mix compounds could cause calibration verification to fail, which normally requires a column trim to recover. Internal standard peak area could also cause calibration failure, but generally occurred less frequently.[1,2] The EPA 8270 short mix contains 12 sensitive compounds. When four or more compounds fell outside of the 80 to 120% range of the calibration set, it was reported as a failure. When the

DDT percent breakdown surpassed 20%, when tailing factors were greater than 2.0, or when the EPA 8270 short mix had more than three compounds fall outside ±20% of the calibration range, the liner and septum were replaced. After inlet maintenance, the system was retested with the QC and EPA 8270 short mixtures. If any of the requirements still failed, the column was trimmed and the system was retested. If the system recovered after column maintenance, experiments continued. If the requirements continued to fail, the GC/MS was retuned, and the requirements were re-evaluated.

Table 3 summarizes the average liner lifetime, number of liners used before a column trim was required, and the number of column trims before the column was replaced. The single taper liner had a slightly longer lifetime over the fritted and wool liners. However, the column had to be trimmed three times more often with the single taper liner than with the fritted or wool liners. Using the single taper liner also required more frequent column replacement compared to the other liners. The replacement was caused by increased transfer of matrix onto the column and transit of that matrix through the column with repeated runs and oven cycles.

The initial DDT percent breakdown illustrates the efficiency of the fritted liner to prevent nonvolatile matrix from getting onto the column. Tables 4 and 5 show the initial DDT percent breakdown for the single taper and fritted liners, respectively. For the single taper liner, initial DDT percent breakdown increases significantly between each liner. For example, in data set number 1, the first single taper has a breakdown of 1.4% (Table 4). The next liner, number 2, has an initial breakdown of 14.2%, indicating significant transfer of the soil matrix on the column. Comparatively in Table 5, the initial DDT percent breakdown for the fritted liner was 0.3%, which slowly increased to 2.4% over seven liners tested in the first data set; a similar pattern was observed in the other fritted liner data sets. The fritted glass barrier prevents a significant amount of matrix from reaching the column. Preventing matrix from reaching the column resulted in extending the column lifetime. The initial DDT percent breakdown for wool liners was more variable and not easy to correlate to matrix transfer onto the column. The variability in initial DDT percent breakdown may be caused by the inherent variability in glass wool packing and deactivation.

Table 3. Average lifetime by number of matrix injections, number of liners used before a column trim, and number of trims before a new column was required for each liner style.

Liner Type	Average Lifetime (Number of Matrix Injections)	Number of Liners Used Before Column Trim	Number of Column Trims Before New Column Required
Fritted	12	7	2
Wool	10	6.67	2
Single Taper	14	2.33	1

Table 4. Tracking the initial DDT percent breakdown for Agilent Ultra Inert single taper liner in the matrix study.

Single Taper Data Set Number	Liner Number	Initial DDT Percent Breakdown	Notes
1	1	1.4	New column
	2	14.2	
	3	18.8	Trim after this liner
2	1	4.5	
	2	17.3	Replace column
3	1	2.9	
	2	13.3	Trim after this liner

Table 5. Tracking the initial DDT percent breakdown for Agilent Ultra Inert splitless single taper with frit liner in the matrix study.

Fritted Liner Data Set Number	Liner Number	Initial DDT Percent Breakdown	Notes
1	1	0.3	New column
	2	0.4	
	3	0.7	
	4	2.0	
	5	2.5	
	6	2.6	
	7	2.4	Trim after this liner
2	1	0.3	

Conclusion

Single taper liners require column trims three times more often than sintered glass fritted liners and glass wool packed liners. Matrix can contaminate more of the column with an open-style liner. The barrier of a frit or wool prevents most of the matrix from reaching the column, leading to longer column lifetimes and fewer column trims. To maximize column lifetime for complex matrix samples, the Agilent Ultra Inert splitless liner with frit will provide more analytical runs before a column trim is required.

References

1. Smith Henry, A. Analysis of Semivolatile Organic Compounds with Agilent Sintered Frit Liner by Gas Chromatography/Mass Spectrometry, *Agilent Technologies*, publication number 5994-0953EN, **2019**.
2. Smith Henry, A. Comparison of Fritted and Wool Liners for Analysis of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, *Agilent Technologies*, publication number 5994-2179EN, **2020**.

www.agilent.com

DE11655128

Printed in the USA, April 18, 2022
5994-4780EN

Agilent
Trusted Answers