



Using Alternative Carrier Gases with Accelerated ASTM D2887 Simulated Distillation Analysis

ASTM Method D2887 now allows for alternate carrier gases, so hydrogen or nitrogen can replace helium carrier gas for simulated distillation. Here, we show that with an MXT-1HT SimDist column and Restek's EZGC online method translator, existing methods using helium can be easily converted to either hydrogen or nitrogen carrier gas. Because retention times are preserved with proper method translation, there are minimal changes to peak identification tables, which significantly simplifies method validation.



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Method #	Method Title	Restek® Column(s)	Restek® Reference Standard(s)
(High-Temperatu	re) Simulated Distillation (SimDist)		
D2887	Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography (C5–C44)	MXT®-2887, Siltek®-treated stainless steel, 10 m x 0.53 mm x 2.65 μm - cat.# 70199 or MXT®-1HT SimDist, Siltek®-treated stainless steel, 10 m x 0.53 mm x 2.65 μm - cat.# 70132	ASTM D2887-12 Calibration Standards - cat.# 31674 - cat.# 31675 Polywax® Standards - cat.# 36224–36227 D2887 Calibration Mix - cat.# 31222
D7213	Standard Test Method for Boiling Range Distribution of Petroleum Distillates in the Boiling Range from 100 to 615 °C by Gas Chromatography (C5–C60)	MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.10 μm - cat.# 70112 or MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.20 μm - cat.# 70115 or MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.88 μm - cat.# 70131	E-mail standards@restek.com for more information.
D6352	Standard Test Method for Boiling Range Distribution of Petroleum Distillates in Boiling Range From 174 to 700 °C by Gas Chromatography (C10–C90)	MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.10 μm - cat.# 70112 or MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.20 μm - cat.# 70115	Polywax® Standards - cat.# 36224–36227
D7398	Standard Test Method for Boiling Range Distribution of Fatty Acid Methyl Esters (FAME) in the Boiling Range from 100 to 615 °C by Gas Chromatography	MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.88 μm - cat.# 70131	Polywax® Standards - cat.# 36224–36227
D7500	Standard Test Method for Determination of Boiling Range Distribution of Distillates and Lubricating Base Oils in Boiling Range From 100 to 735 °C by Gas Chromatography (C7–C110)	MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.10 μm - cat.# 70112 or MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.20 μm - cat.# 70115	Polywax® Standards - cat.# 36224–36227
D7169	Standard Test Method for Boiling Point Distribution of Samples with Residues Such as Crude Oils and Atmospheric and Vacuum Residues by High-Temperature Gas Chromatography	MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.10 μm - cat.# 70112 or MXT®-1HT SimDist, Siltek®-treated stainless steel, 5 m x 0.53 mm x 0.20 μm - cat.# 70115	Polywax® Standards - cat.# 36224–36227
D7096 (replaces D3710)	Standard Test Method for Determination of the Boiling Range Distribution of Gasoline by Wide-Bore Capillary Gas Chromatography	MXT®-1, Siltek®-treated stainless steel, 15 m x 0.53 mm x 5.00 μm - cat.# 70177 or MXT®-1, Siltek®-treated stainless steel, 30 m x 0.53 mm x 5.00 μm - cat.# 70179	E-mail standards@restek.com for more information.
Detailed Hydroca	rbon Analysis (DHA)		
D5134	Standard Test Method for Detailed Analysis of Petroleum Naphthas through <i>n</i> -Nonane by Capillary Gas Chromatography	Rtx®-DHA-50, 50 m x 0.20 mm x 0.50 μm - cat.# 10147	E-mail standards@restek.com for more information.
D6729	Standard Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 100-Meter Capillary High-Resolution Gas Chromatography	Rtx®-DHA-100, 100 m x 0.25 mm x 0.50 μm - cat.# 10148	DHA Standards - cat.# 33034 - cat.# 30725–30731
D6730	Standard Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 100–Meter Capillary (with pre-column) High-Resolution Gas Chromatography	Rtx $^{\circ}$ -DHA-100, 100 m x 0.25 mm x 0.50 µm - cat.# 10148 and Rtx $^{\circ}$ -5 DHA Tuning, 5 m x 0.25 mm x 1.00 µm - cat.# 10165	DHA Standards - cat.# 33034 - cat.# 30725–30731
D6733	Standard Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 50-Meter Capillary High-Resolution Gas Chromatography	Rtx®-DHA-50 , 50 m x 0.20 mm x 0.50 μm - cat.# 10147	DHA Standards - cat.# 33034 - cat.# 30725–30731
D5501	Standard Test Method for Determination of Ethanol Content of Denatured Fuel Ethanol by Gas Chromatography	Rtx $^{\circ}$ -DHA-150, 150 m x 0.25 mm x 1.00 μ m - cat.# 10149	E-mail standards@restek.com for more information.





Transport Tran	Method #	Method Title	Restek® Column(s)	Restek® Reference Standard(s)
Standard Teal Method for International of Researce and Distance (Information of Section Section 1) Standard for International Conference by Car Chromotography (Information of Mills, ETB, TMM) Conference of Section 1) Conference o				
Diff. Lettings-phyly (Dayspeates) 0.56 m p. 0.75 m m p. 1/36* 00 0.56 m p. 1/36* 00 0.07 m m p. 1/36* 00			- cat.# 83606-800 Specified in the D3606 method addendum - includes: - Rtx $^{\circ}$ -1 , 6' (1.8 m), 1/8" OD, 2.0 mm ID and	
Ellyblenzene, pin-Wylene, O-Xylene, C-3 and Neswier Aromatics. and Total Aromatics in Finished Casoline by Gas Chromatography Biodiesel Biodiesel Test Method for Determination of Free and Total Clycerin in 8-100 Biodiesel Methyl Estren by Gas Chromatography AMT* Biodiesel TC, 3 the x 0.53 mm x 5.00 µm vmth 2 m integra- Gap* - call 8 10020 AMT* Biodiesel TC, 3 the x 10.51 µm with 2 m integra- Gap* - call 8 10020 AMT* Biodiesel TC, 3 the x 10.51 µm with 2 m integra- Gap* - call 8 10020 AMT* Biodiesel TC, 3 the x 10.51 µm with 2 m integra- Gap* - call 8 10020 AMT* Biodiesel TC, 3 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 3 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 3 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm retention gap - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51 µm with 2 m x 0.53 mm r x 10 µm - call 8 10020 AMT* Biodiesel TC, 5 the x 10.51	D4815	DIPE, tertiary-Amyl Alcohol, and C1 to C4 Alcohols in Gasoline	Micropacked with 20% TCEP on 80/100 Chromosorb PAW 0.56 m x 0.75 mm ID x 1/16" OD - cat.# 19040 and Rtx®-1, 30 m x 0.53 mm x 3.00 μm	E-mail standards@restek.com for more information.
De584 Post Method for Determination of Free and Total Glycer in in B-100 Bodiese Methyl Extens by Gas Chromatography MXT*-Biodiesel TG, 14 m x 0.53 mm x 0.16 µm with 2 m x 0.53 mm retention gap - cat # 318026 - 3003 shows 2002 and 100 n x 0.32 mm x 0.10 µm with 2 m x 0.53 mm retention gap - cat # 31002-3005 - cat # 33002-3003 shows 2002 mm x 0.10 µm with 2 m x 0.53 mm retention gap - cat # 3002-3005 - cat # 3002-3003 shows 2002 mm x 0.00 µm with 2 m x 0.53 mm retention gap - cat # 3002-3005 - cat # 3002-3003 shows 2002 mm x 0.00 µm with 2 m x 0.53 mm x 50 µm - cat # 34438-34440 shows 2002 mm x 0.00 µm with 2 m x 0.53 mm x 20 µm - cat # 34438-34440 shows 2002 mm x 0.00 µm with 2 m x 0.53 mm x 20 µm - cat # 34438-34440 shows 2002 mm x 0.00 µm x 0.53 µm	D5580	Ethylbenzene, p/m-Xylene, o-Xylene, C9 and Heavier Aromatics,	0.56 m x 0.75 mm ID x 1/16" OD - cat.# 19040 and Rtx $^{\circ}$ -1, 30 m x 0.53 mm x 5.00 μ m	E-mail standards@restek.com for more information.
Part Dispose	Biodiesel			
Standard Test Method for Analysis of Natural Gas by Gas Chromatography MATI*-Msieve 5A, Siltek*-treated stainless steel, 30 m x 0.53 mm x 50 µm	D6584		- cat.# 70289 or MXT®-Biodiesel TG, Siltek®-treated stainless steel 10 m x 0.32 mm x 0.10 μm with 2 m x 0.53 mm retention gap - cat.# 70290 or Rtx®-Biodiesel TG, 10 m x 0.32 mm x 0.10 μm with 2 m x 0.53 mm retention gap	- cat.# 31880 - cat.# 33020–33026
Cat.# 7972-2773	Natural Gas			1
Standard Test Method for Determination of Hydrocarbons in Liquefied Petroleum (LP) Gases and Propane/Propene Mixtures by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Standard Fast Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography Ref. Alumina BOND/MAPD, 50 m x 0.53 mm x 10 μm	D1945		- cat.# 79723-273 and MXT®-Q-BOND, Siltek®-treated stainless steel, 30 m x 0.53 mm x 20 μm	
Liquefied Petroleum (LP) Gases and Propane/Propene Mixtures by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Standard Practice for Analysis of Reformed Gas by Gas Chromatography Cat. # 88000-875 or MXT*-Msieves 5A, Siltek*-treated stainless steel, 30 m x 0.53 mm x 50 μm - cat. # 79723-273 and MXT*-O-BOND, Siltek*-treated stainless steel, 30 m x 0.53 mm x 50 μm - cat. # 79723-273 and MXT*-O-BOND, Siltek*-treated stainless steel, 30 m x 0.53 mm x 20 μm - cat. # 19716	Refinery Gas			I.
Cat.# 88000-875	D2163	Liquefied Petroleum (LP) Gases and Propane/Propene Mixtures		1
D2593 Standard Test Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography Refinery Gas Standard #5 - cat.# 19778 Refinery Gas Standard #5 - cat.# 19778 Refinery Gas Standard #5 - cat.# 19756 Rt*-Alumina BOND/Na ₂ SO ₄ , 50 m x 0.53 mm x 10 μm Refinery Gas Standard #5 - cat.# 19756 Rt*-Alumina BOND/Na ₂ SO ₄ , 50 m x 0.53 mm x 10 μm Refinery Gas Standard #5 - cat.# 19756 Rt*-Alumina BOND/KCl, 50 m x 0.53 mm x 10 μm Refinery Gas Standard #5 - cat.# 19756 Rt*-Alumina BOND/KCl, 50 m x 0.53 mm x 10 μm Refinery Gas Standard #5 - cat.# 19760 - cat.# 19760			- cat.# 88000-875 or MXT®-Msieve 5A, Siltek®-treated stainless steel, 30 m x 0.53 mm x 50 μm - cat.# 79723-273 and MXT®-Q-BOND, Siltek®-treated stainless steel, 30 m x 0.53 mm x 20 μm	E-mail standards@restek.com for more information.
Impurities by Gas Chromatography	Impurities			
Concentrates by Gas Chromatography - cat.# 19756 - cat.# 19756 - cat.# 34443 Rt*-Alumina BOND/KCl, 50 m x 0.53 mm x 10 μm - cat.# 19760 and Rtx*-1, 30 m x 0.53 mm x 5.00 μm - cat.# 10179 Sulfur D6228 Standard Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Flame Photometric Detection Standard Test Method for Sulfur Compounds in Light Petroleum Rtx*-1, 50 m x 0.53 mm x 7.00 μm - cat.# 10179 E-mail standards@restek.com more information.	D2593			1
Impurities in Ethylene by Gas Chromatography - cat.# 19760 and Rtx*-1, 30 m x 0.53 mm x 5.00 µm - cat.# 10179 Sulfur	D2712		"	
D6228 Standard Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Flame Photometric Detection Rtx®-1, 60 m x 0.53 mm x 7.00 μm - cat.# 10193 or MXT-®-1, Siltek®-treated stainless steel, 60 m x 0.53 mm x 7.00 μm - cat.# 70193 D5623 Standard Test Method for Sulfur Compounds in Light Petroleum Rtx®-1, 30 m x 0.32 mm x 4.00 μm E-mail standards@restek.com	D6159		- cat.# 19760 and Rtx®-1, 30 m x 0.53 mm x 5.00 μm	
in Natural Gas and Gaseous Fuels by Gas Chromatography and Flame Photometric Detection - cat.# 10193 or MXT-®-1, Siltek®-treated stainless steel, 60 m x 0.53 mm x 7.00 µm - cat.# 70193 D5623 Standard Test Method for Sulfur Compounds in Light Petroleum Rtx®-1, 30 m x 0.32 mm x 4.00 µm E-mail standards@restek.com	Sulfur			
		in Natural Gas and Gaseous Fuels by Gas Chromatography and	- cat.# 10193 or MXT-®-1, Siltek®-treated stainless steel, 60 m x 0.53 mm x 7.00 μm	E-mail standards@restek.com for more information.
	D5623		· ·	E-mail standards@restek.com for more information.







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Using Alternative Carrier Gases with Accelerated ASTM D2887 Simulated Distillation Analysis

By Katarina Oden, Barry Burger, and Amanda Rigdon

Introduction

ASTM Method D2887 is a widely used test procedure for simulated distillation (SimDist) analysis. This method is applicable to petroleum fractions with a final boiling point between 55 °C and 538 °C at atmospheric pressure and with vapor pressures that allow sampling at ambient temperatures. ASTM D2887 is used to evaluate process stream samples in order to provide accurate data for optimizing refinery operations, and it can also be used for product specification testing as allowed by contractual agreements. Most often, the method is run using a wide-bore (0.53 mm ID) column with a 0.88 to 2.65 μ m polydimethylsiloxane (PDMS) layer. Thickfilm columns offer more sample loading capacity and are preferred over traditional packed columns because of their comparatively low-bleed characteristic and reduced analysis times.

Method D2887 includes an accelerated option (procedure B) for use when faster analyses are desired. Recently, the method also included an allowance for alternate carrier gases so that the standard helium carrier gas can be replaced with either hydrogen or nitrogen. Using these alternate carrier gases allows companies to take advantage of significant cost savings and to employ reliable and more easily available carrier gases. The easiest way to switch carrier gases is to use method translation, which is well known to preserve elution order and compound separation. An interesting derivative of method translation can be used to preserve retention times. Here, our approach was to translate the original method to alternative carrier gases so that the original retention times would be maintained. We show that with an MXT*-1HT SimDist column and Restek's EZGC* online method translator, existing methods based on helium carrier gas can be easily converted to either hydrogen or nitrogen carrier gas without sacrificing the accuracy of the method. Because retention times are preserved with proper method translation, there are minimal changes to peak identification tables and method validation is a much simpler task.

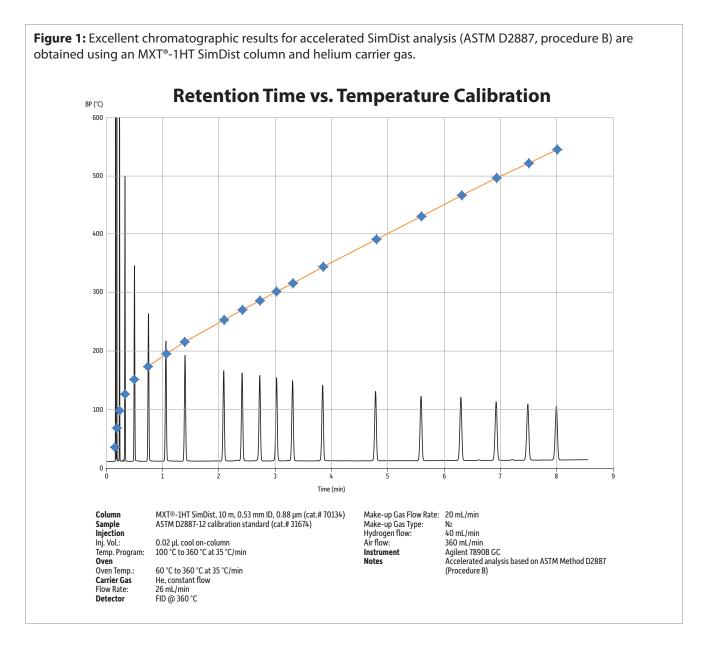
How to Match Your Helium Method

Helium carrier gas is traditionally used for ASTM D2887 because it is nonflammable and gives good resolution at an acceptably fast flow rate. However, helium is expensive and it can fluctuate in availability. Using methodology based on the accelerated parameters (procedure B) for helium carrier gas, along with an MXT $^{\circ}$ -1HT SimDist column, provided very good chromatographic results (Figure 1). Requirements for resolution and skewness were met. In addition, excellent linearity was obtained for the C10-C44 calibration curve of retention time versus boiling point. Note that to extend the linearity of the curve down to C5, the initial column temperature can be lowered to subambient temperatures, or a thicker film column (2.65 μ m) can be used.

Pure Chromatography

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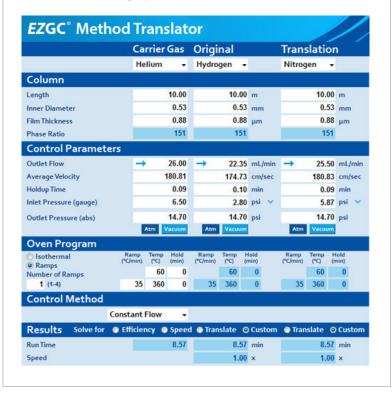
Both hydrogen and nitrogen are attractive alternatives to helium carrier gas because they are less costly and readily available. In fact, both gases can easily be generated in-house, and many labs opt to use a hydrogen or nitrogen generator so they are assured of a reliable and cost-effective supply. In order to match the results obtained with helium when using hydrogen or nitrogen, we used Restek's *EZGC** online method translator to adjust method parameters. This free, online software allowed us to easily establish instrument conditions that would give equivalent chromatographic results using either hydrogen or nitrogen carrier gases. Figure 2 shows the interface of the tool as well as the translated conditions for hydrogen and nitrogen.

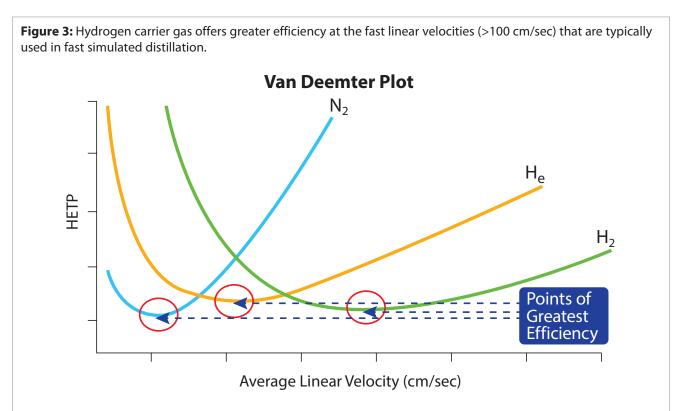


Performance of Hydrogen Carrier Gas

Hydrogen is a good alternative carrier gas because you can use higher linear velocities while still obtaining adequate efficiency and, thus, resolution (Figure 3). Usually, fast SimDist methods use much higher linear velocities (>100 cm/sec) than are used in methods that focus on obtaining highresolution separations. These high linear velocities are well beyond the optimal range for all carrier gases, but there is less efficiency loss with hydrogen than with either helium or nitrogen. Further, the drop in resolution at high linear velocities is much more subtle than the drop in efficiency. The reason the resolution differences are not as dramatic as Van Deemter plots would predict is that resolution is a function of the square root of efficiency.

Figure 2: Simplify the switch to hydrogen or nitrogen by using Restek's *EZGC*® online method translation software to establish conditions that produce the same oven parameters, analysis times, and chromatographic results.



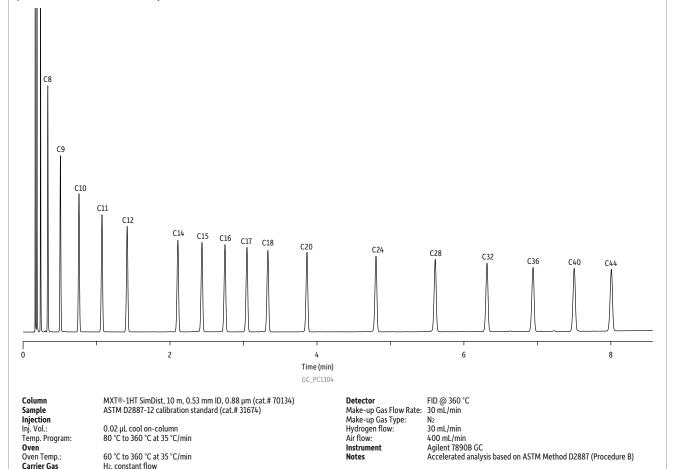


Using the EZGC[®] method translator, conditions were established for hydrogen carrier gas that would give the same retention times as when using helium carrier gas. A partial validation was performed to evaluate the performance of hydrogen as an alternate carrier gas. The data for a retention time calibration standard in Figure 4 and Table I demonstrate that excellent results were obtained even at the 150 cm/sec linear velocity used in the accelerated section of ASTM Method D2887 (procedure B). Peak relative response factors all had a deviation of <10%, as specified in the method. In addition, peak shapes were very good with asymmetry values ranging from 0.96 to 1.19. Note that the translated conditions generated by the EZGC° software provided virtually identical retention times compared to those obtained using helium.

Table I: Evaluation of retention time calibration standard peak shapes and relative response factors when using hydrogen carrier gas.

Carbon Number	% Mass	Asymmetry	Response Factor	Response Factor Deviation
C5	5.0	-	-	-
C6	5.0	0.98	0.95	5%
C7	5.0	1.09	1.01	-1%
C8	5.0	1.01	1.01	-1%
C9	5.0	0.98	1.01	-1%
C10	5.0	1.00	1.00	0%
C11	5.0	0.98	0.99	1%
C12	5.0	0.97	0.98	2%
C14	5.0	0.96	0.94	6%
C15	5.0	0.97	0.95	5%
C16	5.0	0.98	0.94	6%
C17	5.0	0.98	0.94	6%
C18	5.0	0.99	0.93	7%
C20	5.0	0.97	0.92	8%
C24	5.0	1.03	0.98	2%
C28	5.0	1.19	1.01	-1%
C32	5.0	0.98	0.97	3%
C36	5.0	1.00	0.97	3%
C40	5.0	1.01	0.96	4%
C44	5.0	1.05	0.96	4%

Figure 4: Analysis of a retention time calibration standard using hydrogen carrier gas and translated method parameters established by *EZGC*® online software.





Flow Rate:

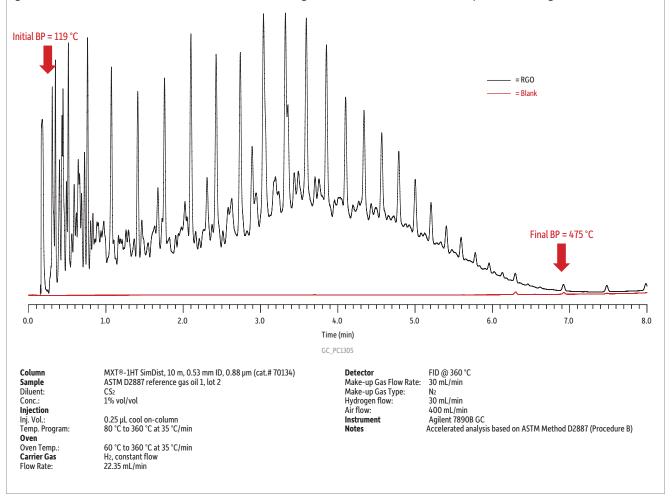
22.35 mL/min

In addition to evaluation of a retention time standard, a blank and reference gas oil (RGO) were also analyzed. The overlay in Figure 5 shows the method requirement was met in that the blank chromatogram does not cross the sample chromatogram, even toward the end of the run. The boiling point distribution data (Table II) were also assessed using SimDist software. The SimDist software establishes a correlation between the boiling points of the hydrocarbons and their retention times in the chromatogram (Figure 1). Based on this calibration, RGO boiling point values at different offsets from the initial boiling point are calculated and compared to the RGO reported values (Table II). The results obtained from the translated SimDist method using hydrogen agree with the ASTM D2887 consensus boiling point values within the allowable percent of windows.

Table II: Comparison of reference gas oil analyzed using hydrogen carrier gas to allowable values.

	ASTM D2887 Values Obs			Observed Values	
%Offset	RGO Standard BP Temp. (°C)	Allowable Difference (°C)	Actual Measured BP Temp. (°C)	Difference (Measured Value - Standard Value)	Result
IBP	115.6	7.6	119.4	3.8	Pass
5	151.1	3.8	151.2	0.1	Pass
10	175.6	4.1	178.6	3.0	Pass
15	200.6	4.5	204.2	3.6	Pass
20	223.9	4.8	227.4	3.5	Pass
30	259.4	4.7	262.4	3.0	Pass
40	288.9	4.3	291.2	2.3	Pass
50	312.2	4.3	313.1	0.9	Pass
60	331.7	4.3	331.3	-0.4	Pass
65	342.8	4.3	343.1	0.3	Pass
70	353.3	4.3	354.0	0.7	Pass
75	365.6	4.3	365.9	0.3	Pass
80	377.8	4.3	378.4	0.6	Pass
85	391.1	4.3	391.2	0.1	Pass
90	406.7	4.3	407.5	0.8	Pass
95	428.3	5.0	429.9	1.6	Pass
FBP	475.6	11.8	475.2	-0.4	Pass

Figure 5: Overlay of blank and reference gas oil analyses show the stable baseline obtained using hydrogen carrier gas. Even at the end of the run, the blank chromatogram does not exceed the sample chromatogram.



Performance of Nitrogen Carrier Gas

A potential drawback to using hydrogen carrier gas is that it is an explosive gas and safety can be a concern if it is not handled properly. Nitrogen can also be used as a carrier gas and some people prefer it because of its nonexplosive properties and affordability. Additionally, reliable nitrogen generators can now be purchased at a relatively low cost. The only drawback to nitrogen is its slow optimal linear velocity. Although the linear velocity used for accelerated SimDist analysis significantly exceeds the optimum level for nitrogen (as well as the optimum level for the other carrier gases), good chromatographic separations were still obtained (Figure 6). Peak width is broader under nitrogen due to the relatively large loss of efficiency, but resolution between target hydrocarbons is still well within the method parameters, and retention times were virtually identical to those obtained using helium carrier gas. Furthermore, the results reported in Table III demonstrate the excellent performance of the method in terms of response factor and peak asymmetry.

Table III: Evaluation of retention time calibration standard peak shapes and relative response factors when using nitrogen carrier gas.

Carbon Number	% Mass	Asymmetry	Response Factor	Response Factor Deviation
C5	5.0	-	-	-
C6	5.0	1.15	0.95	5%
C7	5.0	1.05	0.99	1%
C8	5.0	1.01	0.99	1%
C9	5.0	1.01	0.99	1%
C10	5.0	1.0	1.00	0%
C11	5.0	1.01	0.99	1%
C12	5.0	1.01	0.99	1%
C14	5.0	1.02	0.96	4%
C15	5.0	0.97	0.98	2%
C16	5.0	0.99	0.98	2%
C17	5.0	0.98	0.98	2%
C18	5.0	0.98	0.98	2%
C20	5.0	1.04	0.97	3%
C24	5.0	1.05	0.97	3%
C28	5.0	1.10	0.97	3%
C32	5.0	1.05	0.98	2%
C36	5.0	1.15	0.98	3%
C40	5.0	1.11	0.98	2%
C44	5.0	1.03	0.97	3%

Figure 6: Analysis of a retention time calibration standard using nitrogen carrier gas and translated method parameters established by EZGC® online software. C8 C9 C10 C11 C12 C24 Time (min) GC PC1306 MXT®-1HT SimDist 10 m 0.53 mm ID 0.88 µm (cat # 70134) Detector FID @ 360 °C Column Sample ASTM D2887-12 calibration standard (cat.# 31674) Make-up Gas Flow Rate: 20 mL/min Injection Make-up Gas Type: Ini. Vol.: 0.015 uL cool on-column Hydrogen flow: 40 mL/min 80 °C to 360 °C at 35 °C/min Air flow: Temp. Program: Oven Instrument Agilent 7890B GC 60 °C to 360 °C at 35 °C/min Oven Temp.: Accelerated analysis based on ASTM Method D2887 (Procedure B) Notes Flow Rate: 25.5 mL/min

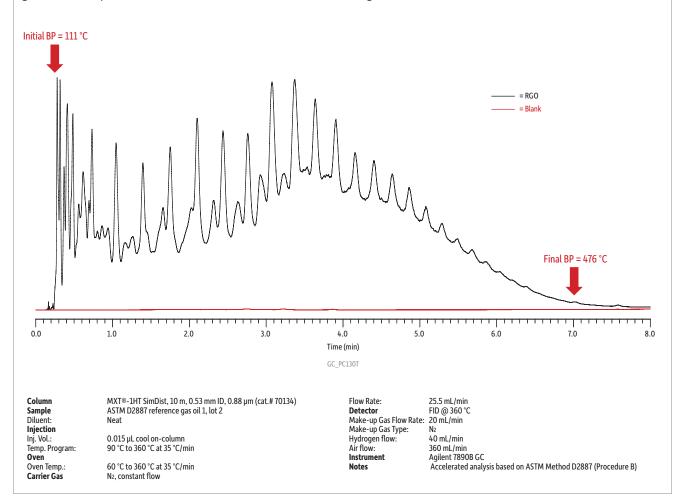


As with the hydrogen carrier gas experiment, a blank and reference gas oil were also analyzed using nitrogen carrier gas and the method parameters established for nitrogen using EZGC* method translation software. Figure 7 shows the chromatographic overlay of the reference gas oil and the blank when using nitrogen carrier gas. As was the case with helium and hydrogen, good chromatographic results were obtained as indicated by the lack of overlap between the blank and the RGO. Further, the quantitative data obtained using Sim-Dist software in Table IV show that the use of nitrogen carrier gas still produced boiling point values that met method requirements.

Table IV: Comparison of reference gas oil analyzed using nitrogen carrier gas to allowable values.

	ASTM D2887 Val	ues		Observed Values	
%Offset	RGO Standard BP Temp. (°C)	Allowable Difference (°C)	Actual Measured BP Temp. (°C)	Difference (Measured Value - Standard Value)	Result
IBP	115.6	7.6	111.4	-4.2	Pass
5	151.1	3.8	151.1	0.0	Pass
10	175.6	4.1	178.1	2.5	Pass
15	200.6	4.5	204.0	3.4	Pass
20	223.9	4.8	227.4	3.5	Pass
30	259.4	4.7	262.7	3.3	Pass
40	288.9	4.3	291.8	2.9	Pass
50	312.2	4.3	313.7	1.5	Pass
60	331.7	4.3	332.0	0.3	Pass
65	342.8	4.3	343.3	0.5	Pass
70	353.3	4.3	354.8	1.5	Pass
75	365.6	4.3	366.9	1.3	Pass
80	377.8	4.3	379.3	1.5	Pass
85	391.1	4.3	392.6	1.5	Pass
90	406.7	4.3	409.8	3.1	Pass
95	428.3	5.0	431.6	3.3	Pass
FBP	475.6	11.8	473.6	-2.0	Pass

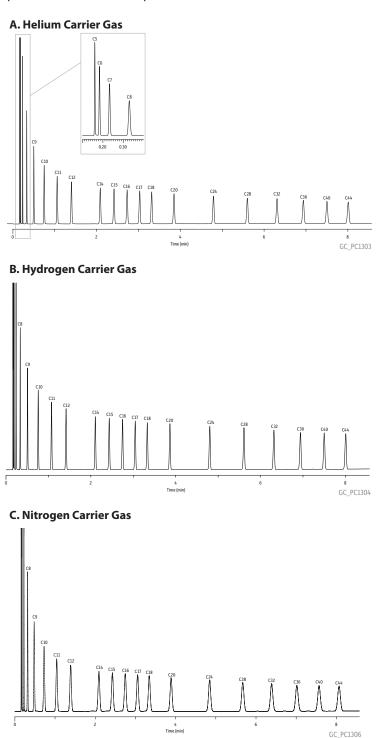
Figure 7: Overlay of blank and reference gas oil analyses show the stable baseline obtained using nitrogen carrier gas. No overlap is seen between the blank and RGO chromatograms.



In Summary

ASTM Method D2887 was modified to include the use of alternative carrier gases. Companies running simulated distillation analyses now have the opportunity to use either hydrogen or nitrogen carrier gas, both of which are inexpensive and easy to produce in-house using a generator. When replacing helium carrier gas with either of these alternatives, revalidation and recalibration can be significantly simplified by using EZGC® method translation software to establish method parameters that give equivalent retention times. Figure 8 directly compares the retention times for C5-C44 hydrocarbons that were presented earlier in the article. When viewed side by side, it is easy to see that the new parameters provided equivalent retention times for all three carrier gases. As would be expected, peaks are sharpest when using hydrogen and widest when using nitrogen; however, all three carrier gases perform well and provide an opportunity for companies to run accelerated SimDist testing to assure product quality and to optimize operations using less expensive and readily available carrier gases.

Figure 8: Comparison of ASTM D2887-B using helium, hydrogen, and nitrogen carrier gases. Note that at high carrier gas velocities nitrogen is the least efficient gas and produces the widest peaks while hydrogen, the most efficient at high carrier gas velocities, produces the narrowest peaks.



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Pure Chroma





Petrochemical Applications

Virtually Particle-Free Rt®-Silica BOND Columns

Provide Reliable PLOT Column Performance With Less Time Lost for Maintenance

By Amanda Rigdon, Bill Bromps, Tom Vezza, and Jaap de Zeeuw

- Optimized manufacturing process practically eliminates particle release, reducing downtime due to system obstructions and damage from particles.
- Bonded silica stationary phase minimizes impact of water, resulting in reproducible retention times for water-containing samples.
- Versatile, highly retentive column ideal for analysis of light hydrocarbons, sulfur gases, halocarbons, and carbon dioxide at temperatures above ambient.
- Individually QC tested with sensitive unsaturated C4 probes to ensure consistent selectivity.

Porous layer open tubular (PLOT) columns are very useful to GC analysts working on a wide variety of applications, and the unique selectivity of PLOT columns makes them particularly good for separating gaseous compounds without cryogenic cooling. However, the overall utility of traditional PLOT columns is hampered by the characteristic instability of the porous layer that coats the inside of the column. With most PLOT columns, particles that shed from the porous layer create significant problems because they can form obstructions inside the column that can alter flow, causing retention time instability. In addition, particle build-up makes frequent maintenance necessary as jets become obstructed and detectors become contaminated. In contrast, new Rt®-Silica BOND columns from Restek are exceptionally robust due to optimized manufacturing and phase bonding steps that practically eliminate particle release. This exceptional stability—in combination with high loadability, inertness, and consistent selectivity—makes these new columns extremely reliable and ideal for the analysis of light hydrocarbons, sulfur gases, and halocarbons. In addition, carbon dioxide and other permanent gases can be retained at ambient temperature on this silica-based column. This article demonstrates the robustness of the Rt®-Silica BOND column and its performance for many of the applications relevant to testing natural gas and light hydrocarbon streams.

Virtually Particle-Free and Water Resistant PLOT Performance

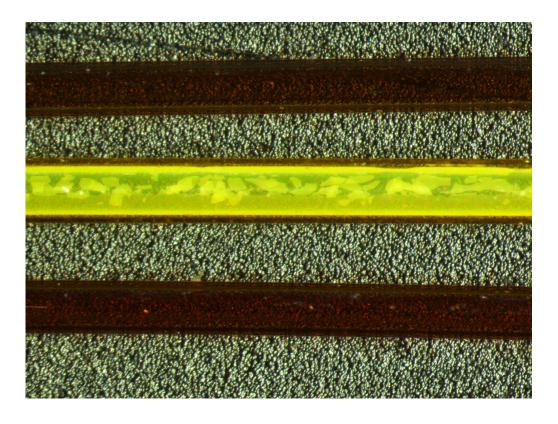
Restek's proprietary manufacturing technique for the Rt®-Silica BOND column results in an extremely stable porous layer with traditional PLOT column loadability and retention without loose particles that can damage valves and foul FID jets.





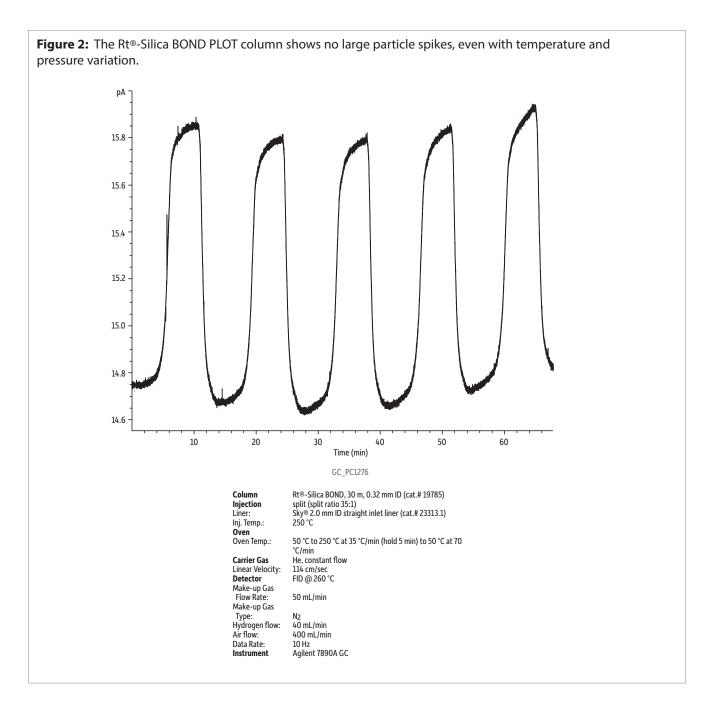
Figure 1 shows a magnified picture of three fused silica columns. The middle column is a traditionally-manufactured PLOT column, the bottom column is a wall-coated open tubular column, and the top column is an Rt®-Silica BOND PLOT column. Note the uneven layer of particles on the middle column, as well as areas where the particles have completely detached from the column wall; this causes irregularities in the internal diameter of the column that can cause retention time instability. In comparison, the Rt®-Silica BOND column looks identical to the wall-coated open tubular column, with no visible shedding of particles or peeling of the coating layer. While the Rt®-Silica BOND column does contain a porous layer, the structure of this layer is extremely fine and well-adhered to the column wall, ensuring virtually particle-free operation over the lifetime of the column.

Figure 1: Traditional PLOT columns (middle) have an uneven coating of particles that can shed, fouling instrument parts. Rt®-Silica BOND columns (top) have a very fine porous layer with no visible particles and look very similar to wall-coated open tubular columns (bottom).

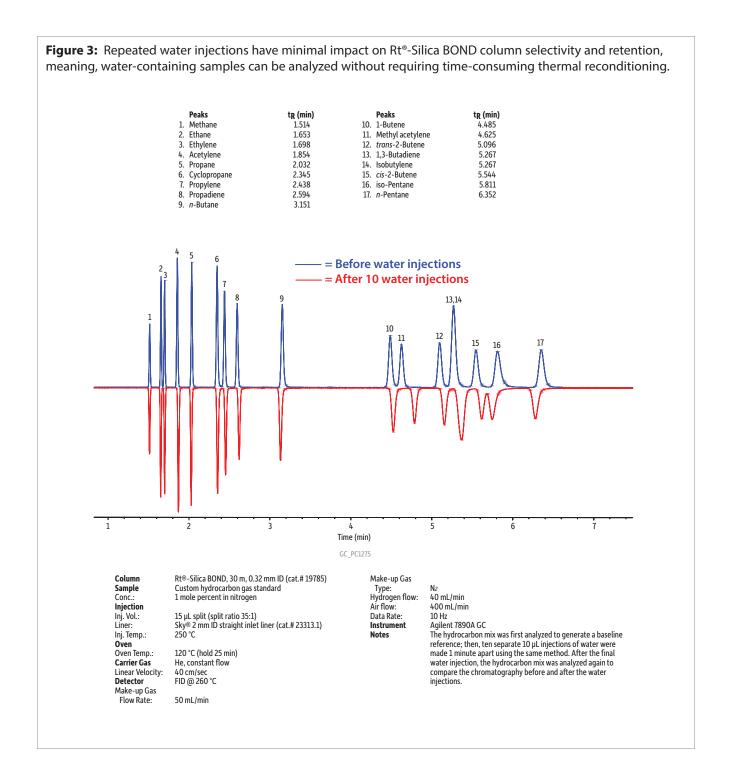


The manufacturing process used to make Rt®-Silica BOND columns results in a PLOT column with high selectivity, retention, and capacity without the particle shedding associated with conventional PLOT columns. This provides improved column robustness and less downtime for maintenance. The particle-free nature of this column is evidenced by a particle-generation experiment in which a column was temperature and pressure ramped multiple times. Changes in temperature cause changes in pressure, which result in particle shedding in traditional PLOT columns. Free particles generate large spikes when they hit the flame ionization detector (FID), interfering with quantification. In addition, the particles themselves can obstruct FID jets and damage valves. Note that no large particle spikes were generated when this experiment was carried out on a brand new Rt®-Silica BOND column (Figure 2).





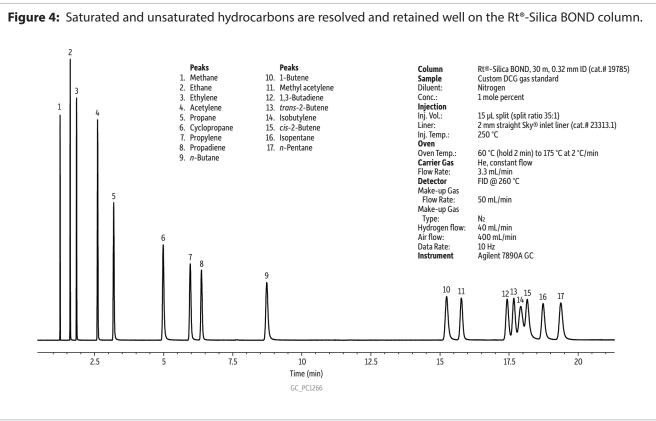
Another benefit of Restek's proprietary manufacturing process for the Rt®-Silica BOND column is that the stationary phase of the column is composed almost entirely of silica. While silica retains water, it does not adsorb it. Some PLOT materials adsorb water, which changes the retention and selectivity of the column. After analyzing samples containing water, these PLOT columns require extensive thermal conditioning (bakeout) to return their original retention and selectivity. Figure 3 shows a mixture of saturated and unsaturated hydrocarbons analyzed on the Rt®-Silica BOND column both before exposure to water and then immediately after 10 large volume water injections. Even under these experimental conditions of extreme overwetting, the retention and selectivity of the column remain very similar and under normal use conditions would be effectively identical. This consistent water-resistant performance allows analysts to save time by minimizing maintenance and eliminating the extensive bakeout periods associated with other PLOT columns.

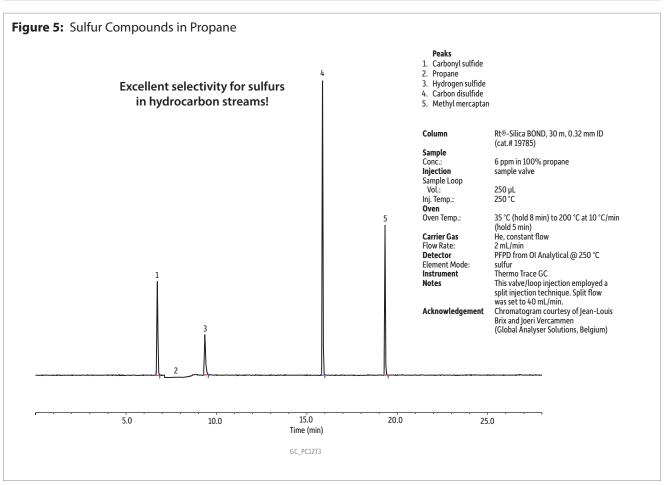


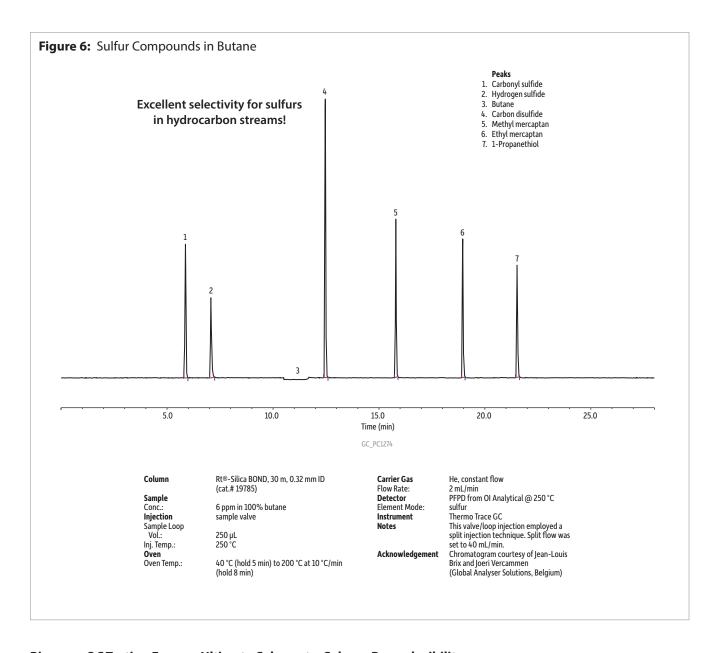
Versatile Column for Many Applications

The new Rt®-Silica BOND column combines the retention, capacity, and selectivity of traditional PLOT columns with virtually particle-free, water-resistant performance. The bonded silica surface provides excellent retention for light hydrocarbons (Figure 4), permanent gases, and halocarbons, allowing for easy analysis of impurities in light hydrocarbon streams. In addition to light hydrocarbon analysis, the Rt®-Silica BOND column is especially selective for sulfur compounds in hydrocarbon streams. Figures 5 and 6 illustrate good separation of sulfur compounds in propane and butane, respectively.





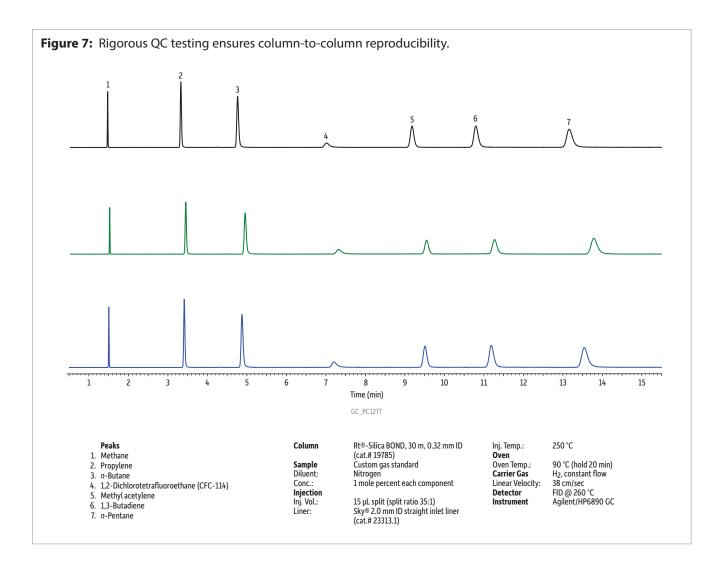




Rigorous QC Testing Ensures Ultimate Column-to-Column Reproducibility

While column-to-column reproducibility is a must for all analysts, it is especially important in valve, backflushing, or column-switching applications. With this in mind, a special QC test was designed for the Rt®-Silica BOND column. Performance parameters, including efficiency, selectivity (RI), retention (k), and inertness are evaluated for each and every column. While the QC tests from some manufacturers include some of these parameters, the compounds used to measure RI are not well-retained and not sensitive to changes in column selectivity. The RI compounds used in the QC test for the Rt®-Silica BOND are 1,3-butadiene and methyl acetylene, which are not only very sensitive probes for selectivity, but are of high interest to many analysts. Additionally, while some commercially available PLOT columns are not evaluated for inertness, Rt®-Silica BOND column inertness is measured with propylene, which is a more active, unsaturated hydrocarbon. This QC testing ensures the highest level of column-to-column reproducibility available in the industry for PLOT columns. Figure 7 shows QC results from three separate lots of Rt®-Silica BOND columns.





Conclusion

The Rt®-Silica BOND column gives you the retention and capacity you need from PLOT columns, along with virtually particle-free and water-resistant operation. The combination of rugged manufacturing and rigorous QC testing ensures every Rt®-Silica BOND column will provide optimal performance and reliable results for every analysis, while minimizing downtime due to maintenance from particle shedding or time-consuming bakeouts due to water contamination. The column's unique selectivity makes it ideal for analysis of hydrocarbons, halogenated compounds, and sulfur gases.





Pure Chromat

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Aura[™] Personal Air Sampler Kit

cat.# 26484 (Electropolished) and cat.# 26485 (Siltek®-Treated)

Overview

The Aura™ personal air sampler (PAS) was developed to help environmental and occupational health experts monitor personal exposure to airborne volatile organic compounds (VOCs). The Aura™ PAS was specifically designed as an alternative to diffusive sampling badges and/or active sampling with thermal desorption (TD) tubes. The Aura™ PAS was engineered to avoid some of the significant shortcomings associated with the aforementioned personal sampling devices, and it delivers the following advantages:

- 1. A passive sampling device that does not require a pump and manages variations in face velocity, temperature, and humidity better than traditional sampling approaches.
- 2. A whole-air sampling approach that affords multiple analyses of over 100 VOCs.
- 3. A simple, quick connection to start and stop flow; does not require flow calibration.

The Aura™ PAS passively collects an 8-hour, whole-air sample via vacuum in a 400 cc canister. The Aura™ PAS does not use a flow controller like other canister sampling approaches; rather, flow is controlled by a proprietary critical orifice. Each Aura™ PAS is delivered with a pre-calibrated starting flow that is approximately 0.310 mL/min. The Aura™ PAS will maintain a near-constant flow (i.e., the ending flow will be within 15% of the starting flow) throughout the 8-hour sampling duration. This flow will result in a fully evacuated (e.g., 29-30" Hg) canister being filled to ~35% full (i.e., 140 mL). All of the aforementioned flows, ranges, and volumes have been established to ensure the Aura™ PAS is compliant with OSHA's 25% bias requirement.

Note: Kit components must be used together to ensure proper sampling. Correct flow may not be obtained if sampling lines of different dimensions or canisters of different volumes are used.

This document provides step-by-step instructions on how to use the Aura™ PAS.



- A 400 cc miniature canister with ¹/₄" quick-connect stem (quantity 1)
- $B \frac{1}{8}$ " quick-connect body (quantity 1)
- C disposable 8-hour sampler lines (quantity 10)
- D holster (quantity 1)
- E sampler line clip (quantity 1)
- F belt (quantity 1)
- G -clean-cut tubing cutter (quantity 1)

Prior to Use

The 400 cc canister with quick-connect stem (part A) is shipped under 30 psig of pressure. Prior to use, you must depressurize the canister using the following steps:

- 1. Unpack the 400 cc canister with quick-connect stem (part A) and remove the black rubber protective cap from the stem. Do not discard the black rubber protective cap; you will need to place it back on the stem after sampling.
- 2. Unpack the quick-connect body (part B).
- 3. Attach the quick-connect body (part B) to the 400 cc canister with quick-connect stem (part A). To obtain a complete connection, you will need to pull back the knurled end of the quickconnect body (part B).

You should hear nitrogen release as the canister depressurizes. If you do not hear this, please contact Restek Technical Service at support.restek.com or 1-814-353-1300, ext. 4.





Step 1

Step 2



Step 3





Cleaning and Evacuating the Sampling Canister

If the 400 cc canister with quick-connect stem (part A) has already been cleaned/evacuated, then proceed to step 1 in the Sampler Setup section.

Otherwise, perform the following steps for cleaning/evacuating the 400 cc canister with quick-connect stem (part A).

- 1. Connect the 400 cc canister with quick-connect stem (part A) to your canister cleaning/evacuating system using the quick-connect body (part B).
 - The quick-connect body (part B) has a $^{1}/8$ " end; therefore, if your cleaning/evacuating system is set up for $^{1}/4$ " connections, a $^{1}/8$ " tube to $^{1}/4$ " tube end reducer (cat.# 23178) is required.
 - To save time, we recommend you buy an additional quick-connect body (part B, cat.# 26482) and attach it to your canister cleaning/evacuating system and/or autosampler.
- 2. Clean the 400 cc canister with quick-connect stem (part A) following your internal standard operating procedure (SOP), but do not exceed 110 °C.
- 3. After cleaning, fully evacuate (e.g., 29-30" Hg) the 400 cc canister with quick-connect stem (part A).
- 4. Disconnect the 400 cc canister with quick-connect stem (part A) from the cleaning system by disconnecting the quick-connect body (part B) from the canister stem.
- 5. Verify and record the vacuum of the 400 cc canister with quick-connect stem (part A) with the use of the quick-connect body (part B) attached to an appropriate vacuum gauge.
- 6. Remove the quick-connect body (part B) from the cleaning system.

Assembling the Sampler

- The quick-connect body (part B) ships with a nut and a twopiece ferrule that you need to remove and discard.
- 2. Screw the quick-connect body (part B) onto a sampler line (part C) using the nut and pre-swaged ferrule on the end of the sampler line (part C).
- Use two ⁷/16" wrenches to tighten the quick-connect body (part B) onto the sampler Line (part C).



Step 1



Step 2



Step 3

Mounting the Personal Air Sampler on the User

- 1. Insert the end of the sampler line (part C) into the opening on the underside of the holster (part D) strap. The opening is adjacent the Restek label.
- Slide the sampler line (part C) through the holster (part D) strap until the end protrudes from one of the eight exit holes on the other end of the strap. The exit holes allow the user to customize the fit. Once the holster (part D) is on, the user can adjust the fit by changing which exit hole is used.
- 3. If you have not already done so, be sure to verify and record the vacuum of the 400 cc canister with quick-connect stem (part A) using the quick-connect body (part B, cat.# 26482) attached to an appropriate vacuum gauge. Restek offers a vacuum gauge preassembled with the proper quick-connect fitting for use with the Aura™ PAS (cat.#24224).
- 4. Insert the 400 cc canister with quick-connect stem (part A) into the holster (part D).
- 5. Have the user place his or her head and one arm through the holster (part D) strap.
 - The exit holes in the holster (part D) strap should be across the wearer's chest and facing out, so that the end of the sampler line (part C) is on the front of the user.
- 6. Using the adjustment on the holster (part D) strap, adjust the fit so the holster (part D) lies on the side of the hip. Restek offers an extension strap (cat.#26481) if additional length is needed for a comfortable fit.
- 7. Connect the quick-connect body (part B) that is attached to the sampler line (part C) to the 400 cc canister with quick-connect stem (part A).
 - Sampling has now begun, so be sure to record the start time.
- 8. The sampler line (part C) needs to protrude from a hole in the strap near the breathing zone. Move the tubing to a new exit hole, if necessary, so that the end of the sampler line (part C) is placed correctly.
 - Make sure there is no slack in the sampler line (part C) and that all of the line has been pulled through holster (part D) strap.
- 9. Using a Restek® clean-cut tubing cutter (cat.# 25069), cut the sampler line (part C) so approximately 2-3" protrude from an appropriate hole in the holster (part D) strap.
 - Remember that the end of the sampler line (part C) is required to be in the breathing zone.





Step 1

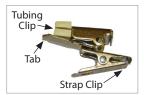






Step 9

- 10. Unpack the sampler line clip (part E), which is shipped in the closed position. This clip is composed of two clips that are attached together: a tubing clip and a strap clip. Open the entire sampler line clip (part E) by pulling the padded end of the tubing clip up off of the tab. Once the entire sampler line clip (part E) is in the open position, the padded end of the tubing clip will be open and ready for insertion of the sampler line (part C).
- 11. Place the sampler line (part C) between the tubing clip pads and close the entire sampler line clip (part E).
- 12. Attach the sampler line clip (part E) to the strap on the holster (part D) with the strap clip.
- 13. Connect belt (part F) to holster (part D) and adjust length for a comfortable fit.







Closed Position

Step 10

Open Position







Steps 11-12

Step 13

After Sampling

- 1. After 8 hours of sampling, disconnect the quick-connect body (part B) from the 400 cc canister with quick-connect stem (part A). Sampling has now stopped, so be sure to record the end time.
- 2. Remove the sampler from the wearer.
- 3. Verify and record the vacuum of the 400 cc canister with quick-connect stem (part A) using an appropriate vacuum gauge that is equipped with a quick-connect body (part B).
- 4. Place the black rubber protective cap back on the stem of the 400 cc canister with quick-connect stem (part A).
- 5. Ship the 400 cc canister with quick-connect stem (part A) to a laboratory capable of conducting U.S. EPA TO-15 analyses.

In the Laboratory

- 1. If you have not already done so, verify and record the vacuum of the 400 cc canister with quick-connect stem (part A).
- 2. Download the testing data template Excel file from the Aura kit product page.
- 3. Enter your information into the cells marked "enter data." The mean flow based on vacuum and volume will be automatically calculated for you using the data you entered.

	End user must enter data in green cells.						
Serial Number of 400 cc Canister: Enter Data Sampling Start Time*: Enter Data							
Serial Numb	per of Sampler Line:	Enter Data	San	npling End Time*:	Enter Data		
	Date: Enter data Sampling Elapsed Time: #VALUE						
	<u>Mear</u>	n Flow Based on	Vacuum and Volu	<u>ıme</u>			
Starting Vacuum (" Hg)							
Enter Data	Enter Data	#VALUE	#VALUE	Enter Data	#VALUE		

*Enter hours and minutes based on a 24-hour clock (e.g., 22:00 for 10 p.m.)

^Enter hours and minutes to the nearest quarter hour (e.g., 8 hours and 10 minutes = 8.25)

This example shows typical results for an 8-hour sampling event:

Starting Vacuum (" Hg)	Ending Vacuum (" Hg)	Vacuum Remaining (%)	Volume Consumed (mL)	Volume Remaining (mL)	Mean Flow (mL/min)
28.65	18.64	65.1	139.8	260.2	0.29

4. Analyze field samples following your internal SOP for the analysis of canisters (e.g., fill canister to desired pressure and analyze via U.S. EPA Method TO-15).

Note: The Aura™ PAS canister will be partially filled ~35% (i.e., 140 mL); therefore, most analytical laboratories will need to fill the canister in order to conduct a TO-15 analysis. We recommend that the canister be filled to 7.5 psig and 200 mL of sample be analyzed. If larger sample volumes are desired, the canister may be filled to 15 psig. The filling of the canister to 7.5 psig will generally result in a 4x dilution of your sample.

Replacement Parts and Accessories

Aura[™] **Personal Air Sampler Kits**

Description	Electropolished cat.#	Siltek Treated cat.#
Aura Personal Air Sampler Kit		
(Includes: 400 cc miniature canister with 1/4" quick-connect stem, quick-connect body,	26484	26485
holster and belt, 10-pk. of disposable 8-hour sampler lines, tubing cutter, and lapel clip)		



Aura[™] Personal Air Samplers (Disposable 8-Hour Sampler Lines)

Description	qty.	cat.#
Aura Personal Air Samplers (Disposable 8-Hour Sampler Line)		26475
	10-pk.	26476



Replacement Filter with 2 μm Frit for Aura[™] Personal Air Samplers

Description	qty.	cat.#
Filter with 2 µm Frit for Aura Personal Air Samplers	ea.	26479



Replacement 2 μm Frits for Aura[™] Personal Air Samplers

Description	qty.	cat.#
2 μm Frit for Aura Personal Air Sampler	ea.	26477
	10-nk	26/178



Accessories for Aura[™] Personal Air Samplers

Description	qty.	cat.#
Holster and Belt for Aura Personal Air Sampler	ea.	26480
Belt Extension for Aura Personal Air Sampler	ea.	26481
Lapel Clip	ea.	26486



Replacement ¹/8" Quick-Connect Body for Aura[™] Personal Air Samplers

Description	qty.	cat.#
Replacement ¹ / ₈ " Quick-Connect Body for Aura Personal Air Sampler	ea.	26482
Replacement 1/8" Quick-Connect Body with 1/4" Adaptor for Aura Personal Air Sampler	ea.	26483



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