Simulated Distillation of Petroleum Products by Packed Column and Capillary Column GC

Simulated distillation by gas chromatography (SIMDIS or GCD) is a technique often employed in the petrochemical industry for quickly determining the boiling range distribution of petrochemical products. Two ASTM test methods—D3710 and D2887—are used for SIMDIS analyses of products with final boiling points of up to 500°F and 1000°F, respectively. Each lot of packing for Petrocol A columns and Petrocol B columns is specifically tested, to meet or exceed ASTM requirements for stationary phase polarity, resolution, and peak symmetry for Method D3710 and Method D2887, respectively. These columns ensure minimal peak tailing and significantly less bleed than other commercially prepared columns. Two capillary columns have also been developed for ASTM SIMDIS methods: Petrocol 3710 columns for Method D3710 and Petrocol 2887.

Key Words:

- Petrocol GC columns ASTM SIMDIS methods
- simulated distillation

Petrocol Packed Columns Ensure More Reliable Information from SIMDIS Analyses

Current methodology published by the American Society for Testing and Materials (ASTM) suggests using packed gas chromatography columns for performing simulated distillation (SIMDIS or GCD) analyses gasoline range materials (Method D3710) or higher boiling petroleum fractions (Method D2887).

ASTM Method D3710 (1) is applicable to samples having a final boiling point of up to 500° F (260° C). There are several critical column performance requirements for this method:

Stationary phase polarity — The phase must be nonpolar, to elute sample components in a true boiling point order.

Column resolution — To properly simulate distillation, a low efficiency separation technique, the column must not display too much resolution. Resolution (R) for nC12 and nC13 must be between the values of 2 and 4.

Peak skewness (asymmetry) — Peak asymmetry affects the accuracy of measurements of boiling point distribution. Therefore, interaction between the sample and the packing support (one cause of peak tailing) must be minimized.

Baseline rise (column bleed) — High column bleed makes it difficult to accurately determine when a sample has completely eluted from the column. Consequently, the final boiling point may be miscalculated.

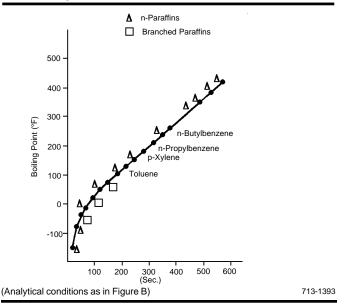
First peak retention time — The first peak must not elute so fast as to prevent establishment of a suitable baseline. ASTM has established a minimum first peak retention time of 15 seconds.

Each lot of packing used in PetrocolTM A columns ($20^{"}/50.8$ cm x $1/8^{"}$ stainless steel) is specifically tested to ensure these columns will meet or exceed ASTM specifications for column polarity, resolution, and peak symmetry.

A totally nonpolar phase is critical for Method D3710. Even slight polarity can affect retention of the more polar sample components — such as aromatics in gasoline. This will cause the apparent boiling point of a polar component to differ from the true boiling point. For Method D3710, this difference, measured by retention time relative to n-paraffins eluting before and after the component, cannot exceed 6°C. Petrocol A columns provide an accurate apparent-to-true boiling point relationship — even for aromatic hydrocarbons (Figure A). For example, the difference between apparent and true boiling point for toluene is less than $\pm 3^{\circ}$ C.

To ensure that analytical results closely represent distillation, ASTM Method D3710 imposes a specific criterion for column resolution of nC12 from nC13 (2<R<4). Each lot of packing used in Petrocol A columns is tested to ensure that the columns will meet this requirement. Sample component retention times among columns

Figure A. Hydrocarbon Boiling Point and Retention on a Petrocol A Column Are Closely Correlated



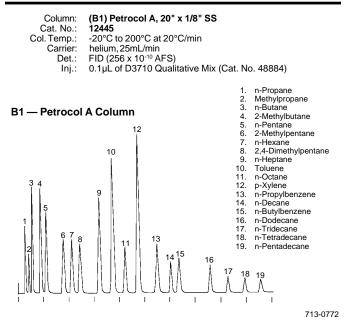


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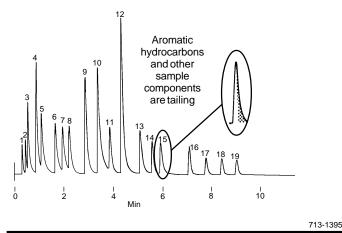
from several lots of packing have been reproducible to within $\pm 4\%$. Thus, column-to-column performance is very consistent.

Skewed peaks can make it difficult to accurately quantify sample components. Commercially prepared columns currently used for Method D3710 often yield tailing peaks for aromatic and aliphatic petroleum components. (This tailing is a result of adsorptive interaction between the sample components and the support used in the packing.) In contrast, the proprietary support used in Petrocol A columns is exceptionally inert, and therefore minimizes peak tailing. This helps ensure more accurate results. Figure B shows chromatograms for a D3710 calibration standard, obtained from a packed column that is often used for SIMDIS analyses and from a Petrocol A column. The degree of peak tailing from the UC-W982/Chromosorb® P AW column exceeds the specification of Method D3710. Peaks from the Petrocol A column, on the other hand, are symmetric.

Figure B. Petrocol A Column Minimizes Hydrocarbon Peak Tailing







Excessive column bleed can interfere with your ability to establish a baseline, and hence to determine when a sample has eluted completely from the column. This is critical in determining the final boiling point (FBP) of a sample. The proprietary support in Petrocol A columns also helps minimize bleed. Petrocol A columns bleed 50-66% less than other columns that have the same phase loading on supports with larger surface areas.

ASTM Method D2887 (2) is similar to Method D3710, except that petroleum fractions with higher boiling points are analyzed in Method D2887. Petroleum fractions having final boiling points of no more than 1000°F (538°C) — such as diesel fuels, lubricating oils, kerosenes, and gas oils — are analyzed by this method. ASTM requirements for proper system performance for Method D2887 include:

Stationary phase polarity — The phase must be nonpolar, as in Method D3710.

Baseline rise — Baseline rise is more important in Method D2887 than in Method D3710, because of the higher boiling points of the sample components involved. Column temperatures up to 350°C are required to elute the heavier compounds from the column. At such temperatures, nearly all stationary phases bleed to some degree, but the ASTM methodology states that a stable baseline is mandatory to establish when a sample has completely eluted from the column.

Column resolution — The column must not display too much resolution, for the same reason as in Method D3710. Resolution (R) for nC16 and nC18 must be between 3 and 8.

First peak retention — As in Method D3710, the first peak must not elute so fast as to prevent establishment of an accurate baseline. In Method D2887, a retention time of no less than 1 minute has been established for the first peak.

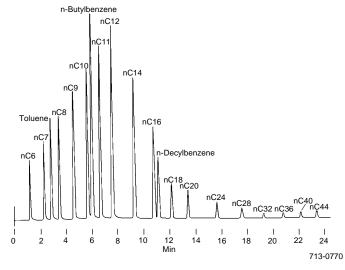
The higher temperatures used in Method D2887 dictate that a column with a low phase loading is needed. Consequently, we prepare Petrocol B columns (20"/50.8cm x 1/8" stainless steel) specifically for Method D2887, to ensure symmetrical peaks, a close relationship between apparent and true boiling points, and low bleed at high temperatures. Relative to other packed columns used for Method D2887, Petrocol B columns offer improved peak symmetry and lower bleed, as shown in Figure C. At 350°C, bleed from Petrocol B columns is negligible (Figures C, D, and E), and higher molecular weight hydrocarbons elute more rapidly. Hydrocarbon boiling points and retention times display a linear relationship over the entire range of the method. For Petrocol B columns, under our test conditions, resolution of nC16 from nC18 is 5.5 exactly between the specified limits of 3 and 8. This permits an analyst greater flexibility when adjusting chromatographic conditions to a specific sample.

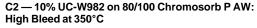
To ensure highly consistent column-to-column performance from Petrocol A and Petrocol B columns, each lot of packing is tested to meet or exceed specifications in the ASTM methodology. Thus, information obtained by using these columns will be highly reliable. Our 20"/50.8cm x 1/8" U-shaped stainless steel columns will fit most GC ovens.

Figure C. Petrocol B Column Minimizes Hydrocarbon Peak Tailing and Column Bleed for Method D2887

Column	(C1) Petrocol B, 20" x 1/8" SS
Cat. No.:	
Col. Temp.:	-25°C to 350°C at 15°C/min
Carrier:	helium, 30mL/min
Det.:	FID (64 x 10 ⁻¹⁰ AFS)
Ini.:	0.1µL of mixed aliphatics and aromatics

C1 — Petrocol B Column: Negligible Bleed at 350°C





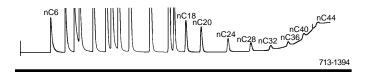


Figure D. Negligible Column Bleed for a Gas Oil Analysis

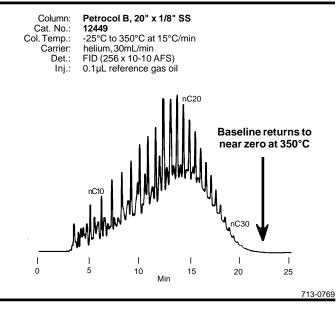
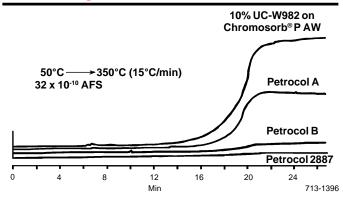


Figure E. Packed Column Bleed Levels at 350°C Using ASTM Method D2887



Capillary Columns Ensure the Most Reliable SIMDIS Information

Although Petrocol A and Petrocol B columns provide a marked improvement in packed column performance for SIMDIS analyses, *all* packed columns have limitations that affect their usefulness for this technique. The support can be adsorptive, causing peak tailing that can reduce accuracy. Subambient initial temperatures must be used to obtain the best correlation between boiling point and retention time. Even so, curves are nonlinear and results are imprecise for the C3-C5 light hydrocarbons. Packed columns must be conditioned for 16 hours or more to ensure a stable baseline. This prolonged conditioning, plus large numbers of samples, can cause loss of column phase.

By determining the optimum combinations of column length, diameter, and phase film thickness — and establishing suitable carrier gas flow rates — we developed wide bore capillary columns that satisfy all column performance criteria in ASTM Methods D3710 and D2887. These wide bore capillary columns offer better performance than any packed column used for this purpose. They are tested to ensure they meet or exceed ASTM performance requirements. They can be used in packed column instruments, with direct injection ports and packed column flow controllers. In addition, the capillary columns can be used with packed column injection systems and detectors commonly used in the petroleum industry.

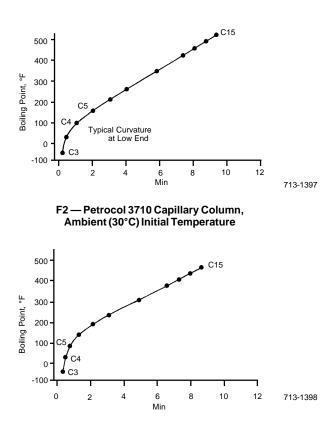
Advantages of using these capillary columns in place of packed columns include:

- Improved boiling point vs. retention time linearity (Method D3710)
- Excellent peak shape (D3710 & D2887)
- Shorter analysis times (D3710 & D2887)
- Reduced column bleed (D3710 & D2887)
- Potential for ambient temperature operation (D3710)
- Potential for extending the analysis range (D2887)
- Very short column conditioning time (D3710 & D288)

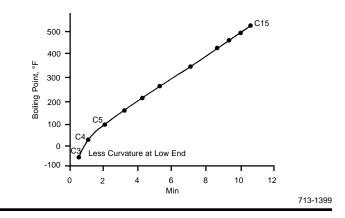
For SIMDIS analyses of gasoline range materials according to Method D3710, Petrocol 3710 capillary columns (10m x 0.75mm ID, 5.0 μ m phase film) offer several significant advantages over even the most reliable packed columns. The C3-C5 hydrocarbons are retained longer on the thick stationary phase film, improving the linearity of the boiling point/retention time curve. In fact, the curve obtained by using a Petrocol 3710 capillary column and an ambient initial temperature is very similar to that

Figure F. Petrocol 3710 Column Provides Near-Linear Correlation Between Hydrocarbon Boiling Point and Retention Time

F1 — Packed Column, -20°C Initial Temperature



F3 — Petrocol 3710 Capillary Column, -20°C Initial Temperature

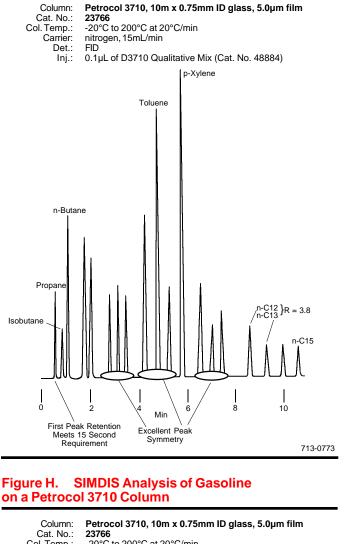


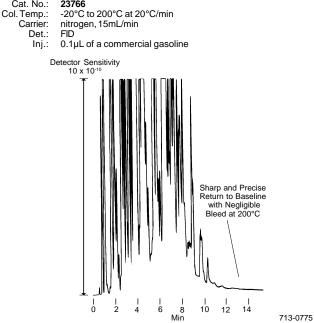
obtained from a packed column and a subambient initial temperature (Figure F).

Using a Petrocol 3710 column, peaks are symmetrical and column bleed is low, while a sharp return to baseline makes determinations of the final boiling point more reliable (Figures G and H). A very short conditioning time for Petrocol 3710 columns (15 minutes to 1 hour), and the fact that the phase is bonded, provide for a potentially longer column lifetime.

Note that nitrogen, rather than helium or hydrogen, is used as the carrier gas in Figures F, G, and H (and in SIMDIS analyses on Petrocol 2887 columns as well). At flow rates most useful for these analyses,

Figure G. Excellent Peak Symmetry, Resolution, and Short Analysis Time from a Petrocol 3710 Column





column efficiency exceeds method requirements when either helium or hydrogen is used. Efficiency is more easily reduced to simulate distillation with nitrogen as the carrier gas (Figure I), ensuring resolution within the specifications of the ASTM methods at much lower flow rates and thus conserving gas.

Petrocol 2887 capillary columns (5m x 0.53mm ID, 0.5µm phase film) satisfy all ASTM column requirements for Method D2887. Figure J shows a D2887 SIMDIS calibration blend of C6-C44 nparaffins analyzed on a Petrocol 2887 column. The hydrocarbons

Figure I. Low Flows of Nitrogen as Carrier Gas Ensure Column Efficiency Falls Within ASTM Specifications

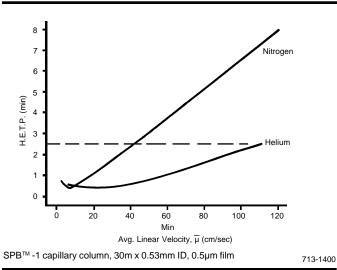


Figure J. D2887 SIMDIS Calibration Blend on a Petrocol 2887 Column

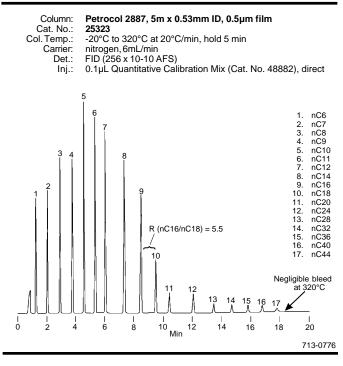
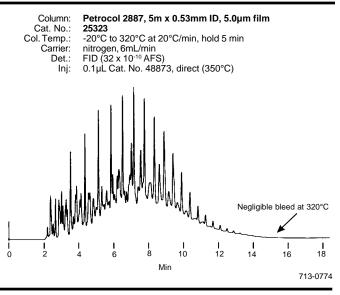


Figure K. Rapid Analysis of Reference Gas Oil with Negligible Bleed

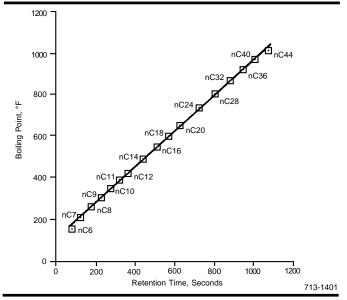


are separated in boiling point order, minimum retention time is at least 1 minute, and the column exhibits a stable baseline with negligible bleed. Peak symmetry is excellent and resolution of nC16 from nC18 is 5.5. Figure K, an analysis of a reference gas oil sample, also shows the bleed from a Petrocol 2887 column is negligible at 320° C.

For best precision in Method D2887, the plot for boiling point versus retention time should be linear for the nC6 through nC44 hydrocarbons. Figure L shows the plot for these hydrocarbons is linear on a Petrocol 2887 column.

Another advantage of the Petrocol 2887 capillary column is the potential to extend the applicable boiling range of the SIMDIS method. Relative to a packed column analysis, a compound having a given carbon number typically elutes from the thin film

Figure L. Calibration Curve for n-Paraffins on a Petrocol 2887 Column



capillary column at a lower column temperature. Table 1 compares typical n-paraffin elution temperatures from a Petrocol 2887 capillary column and a 20' x 1/8" packed column representing current SIMDIS packed column methodology. For a given paraffin, the elution temperature is 20°C or more lower on the capillary column. By taking advantage of the thermal stability of the capillary column (350°C maximum temperature), the applicable test method range can be extended to paraffins with higher boiling points.

Petrocol 2887 columns are conditioned for 1 hour or less at 320°C, compared to 16 or more hours at 350°C for commonly used packed columns. In addition, the thin, bonded phase film produces less column bleed than a packed column. Because the phase is bonded, column lifetime is prolonged for these columns.

Table 1.Typical Elution Temperaturesof n-Paraffins

	Elution	Temp. (°C)	
n-Paraffin	Packed	Capillary	
C20	208	182	
C24	242	215	
C28	271	245	
C32	298	274	
C36	320	300	
C40	340	320	
C44	350	320	

Packed Column: Chromosorb P AW, 20" x 1/8", 10% UCW-982, Oven.: -20°C to 350°C at 20°C/min (5 min), Carrier: helium, 30mL/min, Det.: FID (32 x 10⁻¹⁰ AFS), Inj.: 0.1µL Cat. No. 4-8873, direct, 350°C.

Capillary Column:Petrocol 2887, 5m x 0.53mm ID, 0.5µm film, Oven.: -20°C to 320°C at 20°C/min (5 min), Carrier: nitrogen, 6mL/min, Det.: FID (32 x 10⁻¹⁰ AFS), Inj.: 0.1µL Cat. No. 48873, direct, 350°C Despite the fact that SIMDIS data obtained from Petrocol capillary columns will be more accurate than data obtained from packed columns, the capillary column data and packed column data show very good agreement (Table 2). We believe the discrepancies between values for the early eluting fractions (below 20% of the sample eluted from the column) reflect the reduced curvature (and more accurate correlation) for the capillary column, at this end of the boiling point/retention curve (see Figure F).

Our Quality Assurance department tests each Petrocol 3710 and Petrocol 2887 column to ensure proper resolution, boiling point elution order, boiling point/retention time linearity, and column bleed. By installing a simple conversion kit, you can use these columns in packed column injection ports, with packed column flow controllers. (Contact our Technical Service Department.) Petrocol 3710 columns are made of borosilicate glass, with flexible fused silica transfer lines at both ends. Petrocol 2887 columns are made of fused silica. We recommend connecting a 1-meter retention gap of uncoated, deactivated 0.32mm ID fused silica tubing to a Petrocol 2887 column (Contact our Technical Service Department.). The retention gap will increase column back pressure, for more precise flow control, and will protect the column from nonvolatile contaminants in your samples.

If you are currently using a packed column for D3710 or D2887 SIMDIS analyses, a Petrocol capillary column can improve column performance, save you time, and reduce column replacement costs, without requiring you to purchase major new instrumentation.

We also offer calibration standards that will help you establish conditions for using Petrocol columns, and allow you to monitor their long-term performance. These standards are described in the product listing of this bulletin.

	- · -		-			
Sample Volume % Off $^{ m o}$	Calc. Ten Petrocol 3710 Capillary Column	np.(°F) [▼] Packed Column	Relative Difference %	Boiling Petrocol 2887 Capillary Column	g Pt. (°F)≜ ASTM Packed Column Consensus	Relative Difference %
IBP	20	34	37.0	231	238	2.9
5	76	87	9.5	280	289	3.1
10	95	94	0.7	325	336	3.3
15	138	115	13.0	370	384	3.6
20	163	150	5.9	412	429	4.0
25	186	164	8.9	451	—	_
30	197	185	4.4	485	496	2.2
35	227	203	7.9	515	—	—
40	232	215	5.4	542	548	1.1
45	260	243	4.8	568	—	—
50	278	250	7.5	590	594	0.7
55	283	278	1.3	611	—	—
60	311	295	3.7	630	629	0.2
65	322	308	3.1	650	—	—
70	332	336	0.8	670	669	0.1
75	345	347	0.4	690	—	—
80	364	365	0.2	711	709	0.3
85	387	392	0.9	734	732	0.3
90	405	416	1.9	761	759	0.3
95	440	458	2.8	794	797	0.4
FBP	624	662	4.2	868	887	2.1

Table 2. SIMDIS Data from Petrocol Capillary Columns and Packed Columns Show Good Agreement

Means for 10 injections.

 $^{\rm o}$ Each value represents 5% of the sample eluted from the column.

▼ Data obtained by using a commercial gasoline sample and Spectra-Physics D3710 SIMDIS software.

▲ Data obtained by using ASTM reference gas oil sample and Spectra-Physics D2887 SIMDIS software.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

Ordering Information:

Columns for D2887 SIMDIS Analyses

Petrocol 2887 Capillary Column 5m x 0.53mm x 0.5µm film	25323
Petrocol B Packed Column 20" x 1/8" SS	12449
Columns for D3710 SIMDIS Analyses	
Petrocol 3710 Capillary Column 10m x 0.75mm x 5.0µm film	23766
Petrocol A Packed Column 20" x 1/8" SS	12445

Petrochemical Standards for SIMDIS Analyses **SIMDIS Calibration Blends**

These qualitative and quantitative hydrocarbon blend are prepared according to ASTM recommendations.

When following ASTM D3710, use Catalog No. 48884 to calibrate retention times for boiling point correlation. Use Catalog No. 48879 to determine detector response factors and monitor column and system performance.

When following ASTM D2887, use Catalog No. 48889 to determine column resolution. Use Catalog No. 48882 to calibrate retention times for boiling point correlation.

D3710 Qualitative Calibration Mix

6 x 1mL

48884

48879

This mixture consists of the following components in the approximate proportions (w/w) indicated. Six 1mL ampuls.

n-Propane	1.5	n-Octane	5.4
2-Methylpropane	1.5	p-Xylene	13.0
n-Butane	4.5	n-Propylbenzene	4.3
2-Methylbutane	9.7	n-Decane	3.2
n-Pentane	7.6	n-Butylbenzene	3.2
2-Methylpentane	5.4	n-Dodecane	3.2
n-Hexane	5.4	n-Tridecane	2.2
2,4-Dimethylpentane	5.4	n-Tetradecane	2.2
n-Heptane	9.7	n-Pentadecane	2.2
Toluene	10.8		

3710 Quantitative Calibration Mix

6 x 1mL

This mixture consists of the following components in the proportions (v/v) indicated. Six 1mL ampuls.

2-Methylbutane	10.5	n-Propylbenzene	4.7
n-Pentane	8.1	n-Decane	3.5
2-Methylpentane	5.8	n-Butylbenzene	3.5
n-Hexane	5.8	n-Dodecane	3.5
2,4-Dimethylpentane	5.8	n-Tridecane	2.3
n-Heptane	10.5	n-Tetradecane	2.3
Toluene	11.6	n-Pentadecane	2.3
n-Octane	5.8		
p-Xylene	14.0		

D2887 Quantitative Calibration Mix

•			
6 x 1mL			48882
This mixture consists tions (w/w) indicated:		wing n-paraffins in the	e propor-
Hexane	6	Octadecane	5
Heptane	6	Eicosane	2
Octane	8	Tetracosane	2
Nonane	8	Octacosane	1
Decane	12	Dotriacontane	1

48889

Decane	12	Dotriacontane	1
Undecane	12	Hexatriacontane	1
Dodecane	12	Tetracontane	1
Tetradecane	12	Tetratetracontane	1
Hexadecane	10		

D2887 Column Test Mix

6 x 1mL

This mixture contains 1% (w/v) each of n-hexadecane and n-octadecane in n-octane. Six 1mL ampuls.

Petrocol DH Column Test Mix

1mL 48872 Use this mixture to periodically assess performance of a Petrocol DH 100 meter capillary column. The mixture consists of the following hydrocarbons, by volume, in cyclohexane:

	-		
n-Hexane	1%	Benzene	1%
n-Heptane	1%	Toluene	1%
n-Octane	1%	m-Xylene	4%
n-Nonane	1%	p-Xylene	2%

High Molecular Weight n-Paraffin Standards

For SIMDIS or high temperature GC analyses.	
Hexacontane, 50mg	48893
Pentacontane, 50mg	48595
Polywax [®] 500, 5g	48475
Polywax 500	
10mg/mL in p-xylene 6 x 1mL	48480-U
Polywax 655, 5g	48477
Polywax 655	
10mg/mL in p-xylene 6 x 1mL	48482
Reference Gas Oil Sample	

(1 mal	•	40073
6 x 1mL		48873

This sample is a petroleum fraction with an approximate boiling point range of 250°F-850°F.

Quantitative ASTM Wax Standards

Developed for use with a proposed ASTM method for wax analysis (subcommittee D.02.04L), you can use these n-paraffin mixtures to calibrate your GC for petroleum wax, carbon number distribution, and high temperature SIMDIS analyses.

Available neat in 500mg ampuls or diluted (in cyclohexane) in 1mL ampuls.

PS-12-60N, 500mg	48930-U
PS-12-60D, 1mL	48932

Component	Cat. No. 48930-U Neat (wt. %)	Cat. No. 48932 Dilute (wt./vol. %)
C12	2	0.2
C14	3	0.3
C16	4	0.4
C18	5	0.5
C20	6	0.6
C22	8	0.8
C24	10	1.0
C26	12	1.2
C28	12	1.2
C30	10	1.0
C32	8	0.8
C36	6	0.6
C40	5	0.5
C44	4	0.4
C50	3	0.3
C60	2	0.2

PS-18-44N, 500mg	48926
PS-18-44D, 1mL	48928

Component	Cat. No. 48926 Neat (wt. %)	Cat. No. 48928 Dilute (wt./vol. %)
C18	3	0.3
C20	5	0.5
C22	8	0.8
C24	11	1.1
C26	14	1.4
C28	18	1.8
C30	14	1.4
C32	11	1.1
C36	8	0.8
C40	5	0.5
C44	3	0.3

n-Paraffin Mixtures for Determining Retention Indices

These qualitative standards* are supplied neat (Cat. Nos. 47100-47102) or in 5mL n-octane (Cat. Nos. 47106-47108).

Description	Cat. No.
C5, C6, C7, C8, total 500mg	47100
C7, C8, C9, C10, total 500mg	47101
C10, C12, C14, C16, total 500mg	47102
C18, C20, C22, C24, total 250mg/5mL	47108
C22, C24, C28, C32, total 250mg/5mL	47106
C24, C28, C32, C36, total 250mg/5mL	47107

References

- 1. ASTM 3710, Test Method for Boiling Range Distribution of Gasoline and Gasoline Fractions by Gas Chromatography, 1988 Annual Book of ASTM Standards, Vol. 5.03, pp78-88.
- 2. ASTM D2887, Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography, 1988 Annual Book of ASTM Standards, Vol. 5.02, pp506-513.

Obtain references from ASTM, 1916 Race Street, Philadelphia, PA 19103 USA.

Trademarks

Chromosorb - Manville Corp.

Petrocol - Supelco, Inc.

Polywax - Petrolite Specialty Polymers Group

SPB — Supelco, Inc.

Supelco - Supelco, Inc.

*Qualitative Standard (see our general catalog).

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