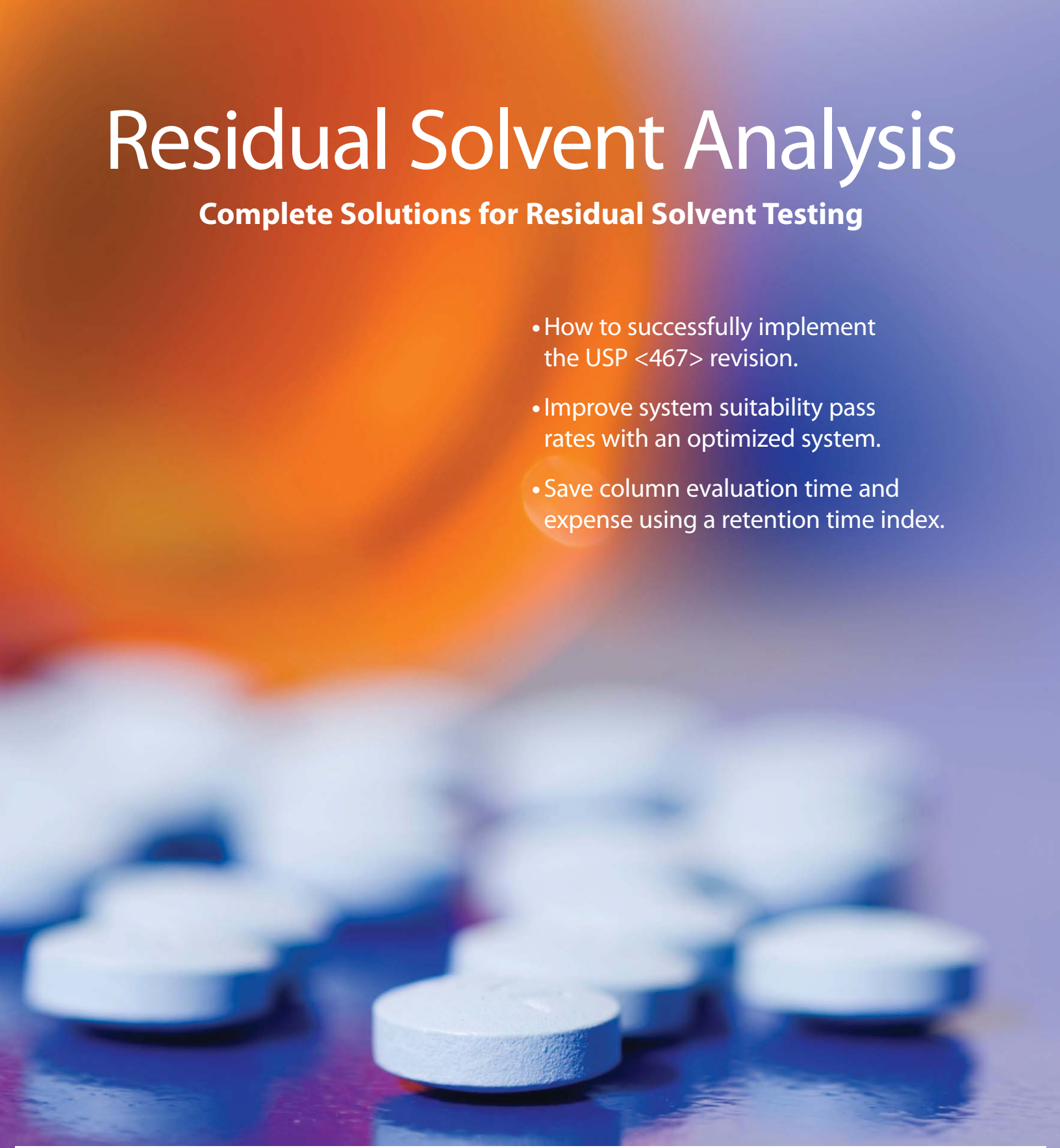
Residual Solvent Analysis

Complete Solutions for Residual Solvent Testing

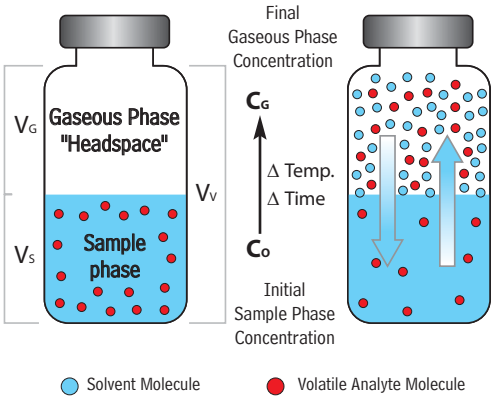
- How to successfully implement the USP <467> revision.
- Improve system suitability pass rates with an optimized system.
- Save column evaluation time and expense using a retention time index.



The Chemistry of Static Headspace Gas Chromatography

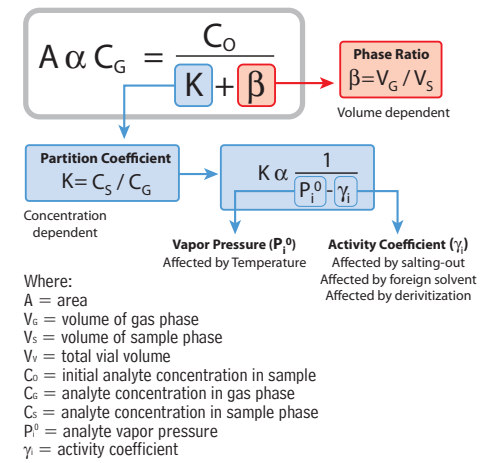
Improve Method Performance with Fundamentals

Figure 1 Volatile components partition into gaseous phase until equilibrium is reached.



Once the sample phase is introduced into the vial and the vial is sealed, volatile components diffuse into the gas phase until the headspace has reached a state of equilibrium as depicted by the arrows. The sample is then taken from the headspace.

Figure 2 Fundamental headspace relationship.



Where:
A = area
V_G = volume of gas phase
V_S = volume of sample phase
V_V = total vial volume
C_O = initial analyte concentration in sample
C_G = analyte concentration in gas phase
C_S = analyte concentration in sample phase
P_i^0 = analyte vapor pressure
γ_i = activity coefficient

Organic volatile impurities (OVIs), commonly referred to as residual solvents, are trace level chemical residues in drug substances and drug products that are byproducts of manufacturing or that form during packaging and storage. Drug manufacturers must ensure that these residues are removed, or are present only in limited concentrations. The International Conference on Harmonization (ICH) Q3C guideline lists the acceptable amounts of solvent residues that can be present. Methodology, both independently developed and compendial, should strive to coincide with this guideline. In this guide, we will take a comprehensive look at residual solvent analysis, in both theory and practice, and illustrate options for the practicing chromatographer.

The analysis of residual solvents is commonly performed using static headspace gas chromatography (HS/GC). The basic premise behind headspace analysis begins with the addition of an exact, known volume or weight of sample into a closed, sealed vial. This creates two distinct phases in the vial—a sample phase and a gaseous phase, or "headspace". Volatile components inside the sample phase, whether a solid or solution, can be extracted, or partitioned, from the sample phase into the headspace. An aliquot of the headspace can then be taken and delivered into a GC system for separation and detection. If we look at the anatomy of a headspace vial (Figure 1), we can begin to see the relationship of the vial components and how we can control these parameters to create analytical methods.

Residual solvent analysis by static HS/GC can be enhanced by careful consideration of two basic concepts—partition coefficient (K) and phase ratio (β). Partition coefficients and phase ratios work together to determine the final concentration of volatile compounds in the headspace of sample vials. Volatile components partition from the sample phase and equilibrate in the vial headspace. Striving for the lowest values for both K and β when preparing samples will result in higher concentrations of volatile analytes in the gas phase and, therefore, better sensitivity (Figure 2).

Controlling the Partition Coefficient

The partition coefficient (K) is defined as the equilibrium distribution of an analyte between the sample and gas phases. Compounds that have low K values will tend to partition more readily into the gas phase, and have relatively high responses and low limits of detection. K can be further described as a relationship between analyte vapor pressure (p_i^0) and activity coefficient (γ_i). In practice, K can be lowered by increasing the temperature at which the vial is equilibrated (vapor pressure) or by changing the composition of the sample matrix (activity coefficient) by adding an inorganic salt or a solvent of lesser solubility, often referred to as a foreign solvent. High salt concentrations and foreign solvents decrease analyte solubility in the sample phase (decrease activity) and promote transfer into the headspace, thus resulting in lower K values. The magnitude of this effect on K is not the same for all analytes. Compounds with inherent low K values in the matrix will experience little change in partition coefficient in response to the addition of a salt and temperature, while volatile compounds in a matrix of similar polarity will show the largest responses.

Adjusting the Phase Ratio

The phase ratio (β) is defined as the volume of the headspace over the volume of the sample in the vial. Lower values for β (i.e., larger sample sizes) will yield higher responses for compounds with inherently low K values. However, decreasing β will not always yield the increase in response needed to improve sensitivity. When β is decreased by increasing sample size, compounds with high K values will partition less into the headspace compared to compounds with low K values and yield correspondingly smaller changes in sensitivity.

Achieving USP<467> Compliance

Your Guide to Successfully Implementing the Revised Method

The USP general chapter <467> Residual Solvents is a widely used compendial method for identifying and quantifying residual solvents when no information is available on what solvents are likely to be present. In an attempt to harmonize with the ICH guidelines, the USP has proposed a more comprehensive method in the current USP 30/NF 25. This revision significantly increases the number of residual solvents to be routinely tested and includes three distinct procedures.[1]

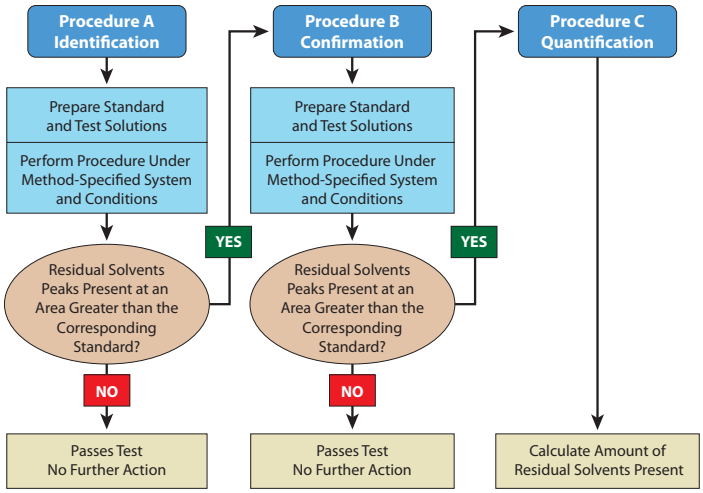
Initially set to become effective July 1, 2007, the implementation of the current version of USP <467> has been delayed until July 1, 2008. Until that time, the Other Analytical Procedures section of the previous version will be retained. However, in preparation for the implementation of the revised method, this application will comply with the procedure and criteria set forth in the USP30/NF25, second supplement (effective December 1, 2007) and the interim revision announcement.

Overview of Method

The revised USP <467> method consists of a static headspace extraction coupled with a gas chromatographic separation and flame ionization detection. In this guide we demonstrate the USP <467> application using two different types of headspace autosamplers. Procedure A was performed using a pressured loop autosampler and transfer line. Procedure B was performed using a heated syringe injection. Either system can be used to meet method requirements.

USP <467> is divided into two separate sections based upon sample solubility: water-soluble and water-insoluble articles. The methodology for both types of articles is similar, but the diluent used in both standard and sample preparations differs based upon the solubility of the test article. The test method consists of three procedures (A, B, and C), that are designed to identify, confirm, and then quantify residual solvents in drug substances and products (Figure 3).

Figure 3 Analytical flow chart for residual solvent testing under the revised USP <467> method.



[1] This number of analytes to be tested represents the sum of Class 1 and 2 residual solvents that can be effectively assayed using HS/GC. The actual number of analytes may be more if xylenes, ethyl benzene and *cis/trans* 1,2 dichloroethylene are differentiated, or if circumstances require the quantification of specific Class 3 residual solvents.

tech tip

Compatibility concerns?
Refer to the Septum Selection Guide at **www.restek.com/septaguide**

free literature

Download your free copy of our Technical Guide for Static Headspace Analysis from **www.restek.com/ovi**

lit. cat.# 59895A

Residual Solvents - Class 1

benzene	10mg/mL	1,1-dichloroethene	40
carbon tetrachloride	20	1,1,1-trichloroethane	50
1,2-dichloroethane	25		

In dimethyl sulfoxide, 1mL/ampul
cat. # 36279 (ea.)

Quantity discounts not available.

Residual Solvents Class 2 - Mix A (15 components)

acetonitrile	2.05mg/mL	methylcyclohexane	5.90
chlorobenzene	1.80	methylene chloride	3.00
cyclohexane	19.40	tetrahydrofuran	3.45
cis-1,2-dichloroethene	4.70	toluene	4.45
trans-1,2-dichloroethene	4.70	*m*-xylene	6.51
1,4-dioxane	1.90	*o*-xylene	0.98
ethylbenzene	1.84	*p*-xylene	1.52
methanol	15.00		

In dimethyl sulfoxide, 1mL/ampul
cat. # 36271 (ea.)

Residual Solvents Class 2 - Mix B (8 components)

chloroform	60µg/mL	nitromethane	50
1,2-dimethoxyethane	100	pyridine	200
n-hexane (C6)	290	tetralin	100
2-hexanone	50	trichloroethene	80

In dimethyl sulfoxide, 1mL/ampul
cat. # 36280 (ea.)

Quantity discounts not available.

Residual Solvents Class 2 - Mix C (8 components)

2-ethoxyethanol	800µg/mL	2-methoxyethanol (methyl Cellosolve®)	250
ethylene glycol	3,100	N-methylpyrrolidone	2,650
formamide	1,100	sulfolane	800
N,N-dimethylacetamide	5,450		
N,N-dimethylformamide	4,400		

In dimethyl sulfoxide, 1mL/ampul
cat. # 36273 (ea.)

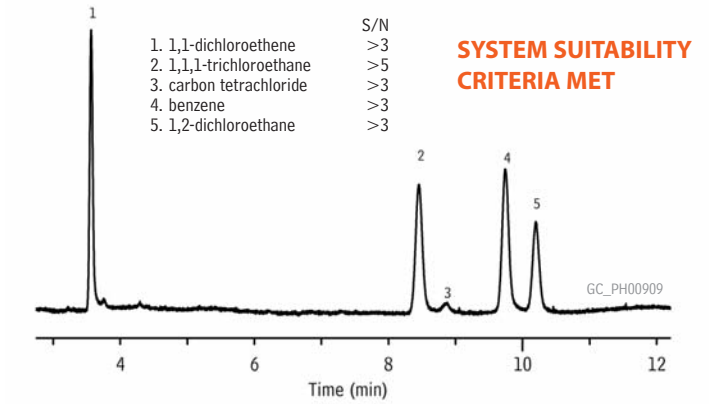
Analytical Reference Materials

The ICH guideline classifies residual solvents by class according to toxicity. Class 1 compounds are carcinogenic and pose a risk to both the consumer and the environment. The use of these solvents must be avoided or tightly controlled. Class 2 compounds are nongenotoxic animal carcinogens and their concentration should be limited. Both Class 1 and 2 compounds require chromatographic determination and are separated into 3 test mixes: Class 1 Mixture, Class 2 Mixture A, and Class 2 Mixture B. Class 3 compounds have low toxic potential. Concentration levels of up to 0.5% are acceptable and, therefore, they can be assayed by nonspecific techniques, such as weight loss on drying. Class 2 Mixture C is not used in the second supplement of USP 30/NF 25, but contains solvents that are not readily detectable by headspace analysis. These solvents should be assayed by other appropriately validated procedures.

Procedure A - Identification

Procedure A is the first step in the identification process and is performed on a G43 column to determine if any residual solvents are present in the sample at detectable levels. First, Class 1 standard and system suitability solutions and Class 2 Mix A standard solutions are assayed under the method-specified operating conditions to establish system suitability. All peaks in the Class 1 system suitability solution must have a signal-to-noise ratio not less than 3, the Class 1 standard solution must have a 1,1,1-trichloroethane response greater than 5, and the resolution of acetonitrile and dichloromethane must be not less than 1 in the Class 2 Mixture A solution. When system suitability has been achieved, the test solutions are assayed along with the Class 1 and Class 2 Mixtures A and B standard solutions. If a peak is determined in the sample that matches a retention time and has a greater response than that of a corresponding reference material, then Procedure B is performed for verification of the analyte. In the second supplement of USP 30/NF 25, an exemption is made for 1,1,1-trichloroethane, where a response greater than 150 times the peak response denotes an amount above the percent daily exposure limit. Figures 4 through 6 illustrate the analysis of Class 1, Class 2 Mixture A, and Class 2 Mixture B residual solvent mixes by Procedure A. The resolution between acetonitrile and dichloromethane was easily achieved using an Rtx®-1301 column.

Figure 4 USP residual solvent Class 1 standard solution on an Rtx®-1301 column (G43).

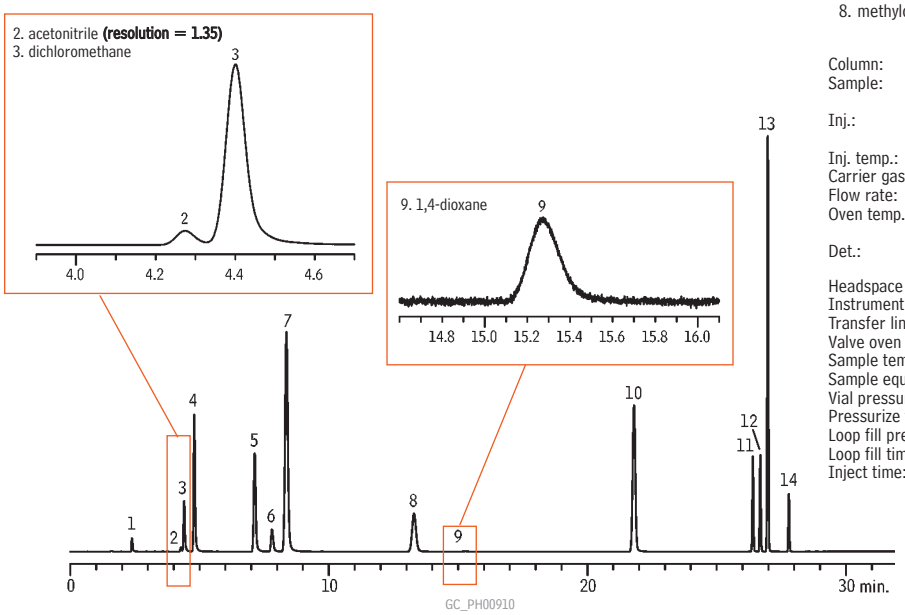


Column:	Rtx®-1301, 30m, 0.32mm ID, 1.8µm (cat.# 16092)	Headspace Conditions	
Sample:	USP <467> Class 1 standard solution (cat.# 36279) in 20mL headspace vial	Instrument:	Tekmar HT3
		Transfer line temp.:	105°C
Inj.:	headspace injection (split ratio 1:5), 1mm split liner, Siltek® deactivated (cat.# 20972-214.1)	Valve oven temp.:	105°C
		Sample temp.:	80°C
Inj. temp.:	140°C	Sample equil. time:	45 min.
Carrier gas:	helium, constant flow	Vial pressure:	10psi
Flow rate:	2.16mL/min., 35.3cm/sec.	Pressurize time:	0.5 min.
Oven temp.:	40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)	Loop fill pressure:	5psi
		Loop fill time:	2.00 min.
Det.:	FID @ 240°C	Inject time:	1.00 min.

Figure 5 USP residual solvent Class 2 Mixture A standard solution on an Rtx®-1301 column (G43).

SYSTEM SUITABILITY CRITERIA MET

1. methanol
2. acetonitrile (resolution=1.35)
3. dichloromethane
4. *trans*-1,2-dichloroethene
5. *cis*-1,2-dichloroethene
6. tetrahydrofuran
7. cyclohexane
8. methylcyclohexane
9. 1,4-dioxane
10. toluene
11. chlorobenzene
12. ethyl benzene
13. *m*-xylene/*p*-xylene
14. *o*-xylene



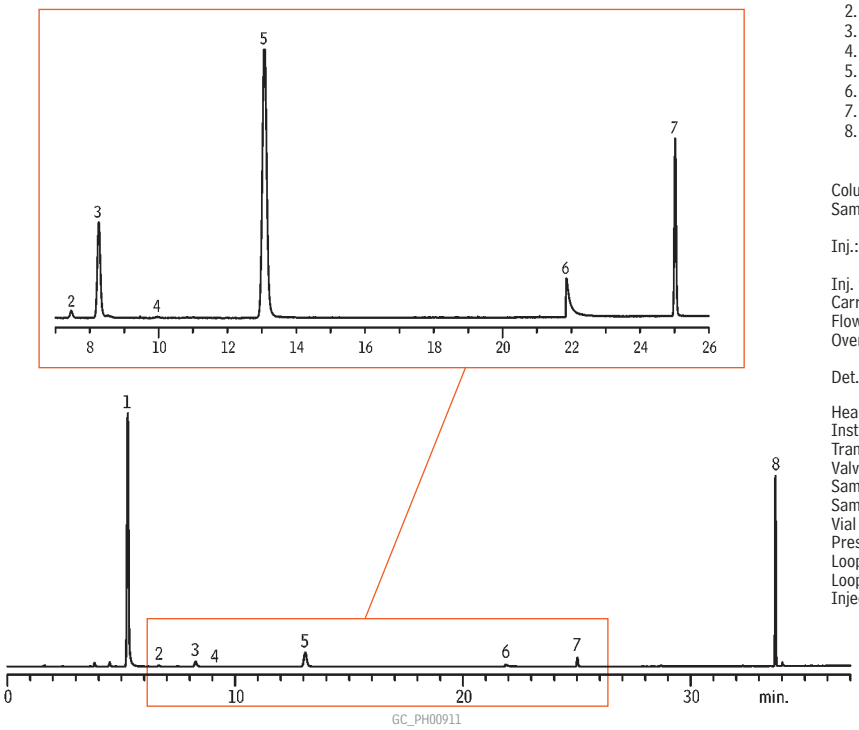
Column:	Rtx®-1301, 30m, 0.32mm ID, 1.8µm (cat.# 16092)
Sample:	USP <467> Class 2 Mixture B standard solution (cat.# 36280) in 20mL headspace vial
Inj.:	headspace injection (split ratio 1:5), 1mm split liner Siltek® deactivated (cat.# 20972-214.1)
Inj. temp.:	140°C
Carrier gas:	helium, constant flow
Flow rate:	2.16mL/min., 35.3cm/sec.
Oven temp.:	40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)
Det.:	FID @ 240°C

Headspace Conditions

Instrument:	Tekmar HT3
Transfer line temp.:	105°C
Valve oven temp.:	105°C
Sample temp.:	80°C
Sample equil. time:	45 min.
Vial pressure:	10psi
Pressurize time:	0.5 min.
Loop fill pressure:	5psi
Loop fill time:	2.00 min.
Inject time:	1.00 min.

Figure 6 USP residual solvent Class 2 Mixture B standard solution on an Rtx®-1301 column (G43).

1. hexane
2. nitromethane
3. chloroform
4. 1,2-dimethoxyethane
5. trichloroethylene
6. pyridine
7. 2-hexanone
8. tetralin



Column:	Rtx®-1301, 30m, 0.32mm ID, 1.8µm (cat.# 16092)
Sample:	USP <467> Class 2 Mixture B standard solution (cat.# 36280) in 20mL headspace vial
Inj.:	headspace injection (split ratio 1:5), 1mm split liner Siltek® deactivated (cat.# 20972-214.1)
Inj. temp.:	140°C
Carrier gas:	helium, constant flow
Flow rate:	2.16mL/min., 35.3cm/sec.
Oven temp.:	40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)
Det.:	FID @ 240°C

Headspace Conditions

Instrument:	Tekmar HT3
Transfer line temp.:	105°C
Valve oven temp.:	105°C
Sample temp.:	80°C
Sample equil. time:	45 min.
Vial pressure:	10psi
Pressurize time:	0.5 min.
Loop fill pressure:	5psi
Loop fill time:	2.00 min.
Inject time:	1.00 min.

Achieving USP<467> Compliance *(continued from page 5)*

Capillary Column—Procedure A

Rtx®-1301 (G43) Columns (fused silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	1.80	-20 to 240°C	30-Meter	16092
0.53mm	3.00	-20 to 240°C	30-Meter	16085

Capillary Column—Procedure B

0.32mm	0.25	40 to 250°C	30-Meter	10624
0.53mm	0.25	40 to 250°C	30-Meter	10625

Procedure B - Confirmation

Once a residual solvent is identified and found to be above the percent daily exposure limit, Procedure B is performed to confirm analyte identity. A G16 capillary column is used here as a confirmation column, because it yields an alternate selectivity compared to a G43 column. The same standard and system suitability preparations are used in Procedures A and B. The system suitability requirements differ here in that the Class 1 standard solution must have a benzene response greater than 5 and the resolution of acetonitrile and *cis*-dichloroethene must not be less than 1 in the Class 2 Mixture A solution, a change from the original version. If the analyte identified in Procedure A again matches the retention time and exceeds the peak response of the reference materials (with the same exception to 1,1,1-trichloroethane), the analyst must quantify the analyte using Procedure C. Figures 7 through 9 illustrate the analysis of Class 1, Class 2 Mixture A, and Class 2 Mixture B residual solvent mixes on a Stabilwax® column. Again, the system suitability requirements were easily met.

Procedure C – Quantification

Once a residual solvent has been identified and verified, Procedure C is used to quantify the analyte by analyzing the sample against compound-specific reference materials. Individual standards are prepared by diluting the analyte in solution to a concentration of 1/20 of the concentration limit given under concentration limit Table 1 or 2 of the method. Following the procedure and instrument conditions in either Procedure A or B (whichever provides the most definitive results), a quantifiable result is produced. For water-insoluble articles, the same procedure is followed, except dimethylformamide or dimethylsulfoxide is used as the diluent.

Figure 7 USP residual solvent Class 1 standard solution on a Stabilwax® column (G16).

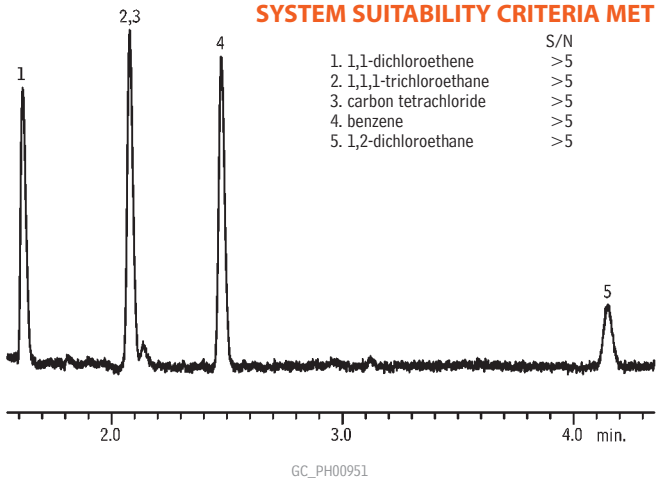


Figure 8 USP residual solvent Class 2 Mixture A standard solution on a Stabilwax® column (G16).

SYSTEM SUITABILITY CRITERIA MET—
RESOLUTION BETWEEN PEAKS 7 & 8 > 1.0

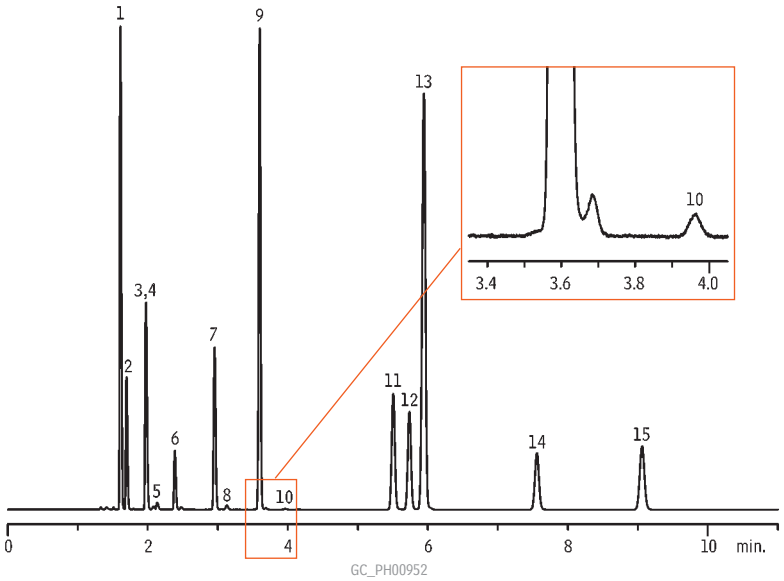
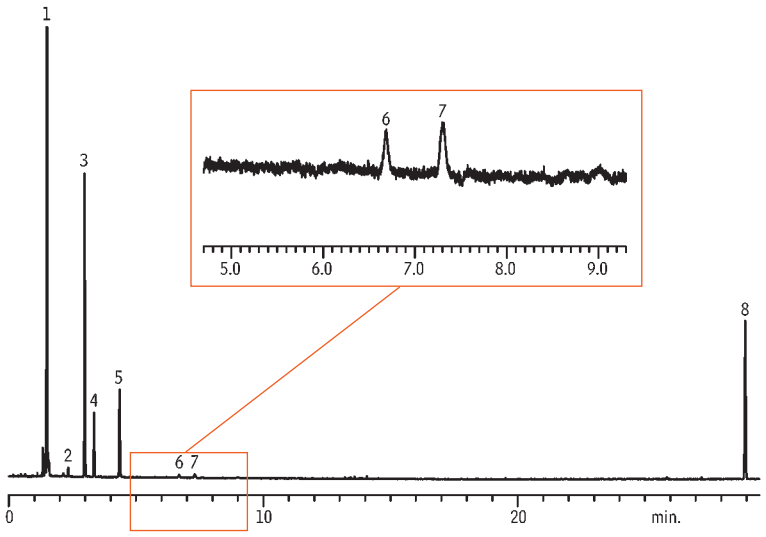


Figure 9 USP residual solvent Class 2 Mixture B standard solution on a Stabilwax® column (G16).



1. hexane
2. 1,2-dimethoxyethane
3. trichloroethylene
4. chloroform
5. 2-hexanone
6. nitromethane
7. pyridine
8. tetralin

Column:	Stabilwax®, 30m, 0.32mm ID, 0.25µm (cat.# 10624)
Sample:	USP Stock Standard Residual Solvents Class 2 Mix B (cat.# 36272) in 20mL headspace vial (cat.# 24685), water diluent
Inj.:	headspace injection (split ratio 1:5), 2mm splitless liner IP deactivated (cat.# 20712)
Inj. temp.:	140°C
Carrier gas:	helium, constant flow
Flow rate:	2.15mL/min., 35.2cm/sec.
Oven temp.:	50°C for 20 min. to 165°C @ 6°C/min. (hold for 20 min.)
Det.:	FID @ 250°C

Headspace Conditions

Instrument:	Overbrook Scientific HT200H
Syringe temp.:	100°C
Sample temp.:	80°C
Sample equil. time.:	45 min.
Injection vol.:	1.0mL
Injection speed:	setting 8
Injection dwell:	5 sec.

Optimize Your Testing Procedure

Tools, Tips, & Techniques for Improving Method Performance

Use Smaller Bore Liners for Better Efficiency

1mm Split Liners for Agilent GCs

ID* x OD & Length	qty.	cat.#
1mm Split**		
1.0mm x 6.3mm x 78.5mm	ea.	20972
1.0mm x 6.3mm x 78.5mm	5-pk.	20973

2mm Splitless Liners for Agilent GCs

ID* x OD & Length	qty.	cat.#
2mm Splitless		
2.0mm x 6.5mm x 78.5mm	ea.	20712
2.0mm x 6.5mm x 78.5mm	5-pk.	20713
2.0mm x 6.5mm x 78.5mm	25-pk.	20714

Split Liners for Varian 1075/1077 GCs

ID* x OD & Length	qty.	cat.#
1mm Split		
1.0mm x 6.3mm x 72mm	ea.	20970
1.0mm x 6.3mm x 72mm	5-pk.	20971

Split Liners for Shimadzu GCs

ID* x OD & Length	qty.	cat.#
1mm Split		
1.0mm x 5.0mm x 95mm	ea.	20976
1.0mm x 5.0mm x 95mm	5-pk.	20977
1.0mm x 5.0mm x 95mm	25-pk.	20978

SPME Liners for Shimadzu 17A, 2010, and 2014 GCs

ID* x OD & Length	qty.	cat.#
SPME Liner		
.75mm x 5.0mm x 95mm	ea.	22278
.75mm x 5.0mm x 95mm	5-pk.	22279

Zero Dilution Liners for PerkinElmer Auto SYS™ and Clarus GCs

ID* x OD & Length	qty.	cat.#
Zero Dilution Inner Liner		
1.0mm x 2.0mm x 73mm	ea.	22990
1.0mm x 2.0mm x 73mm	5-pk.	22991

Zero Dilution Outer Liner		
2.5mm x 6.2mm x 90mm	ea.	22992
2.5mm x 6.2mm x 90mm	5-pk.	22993

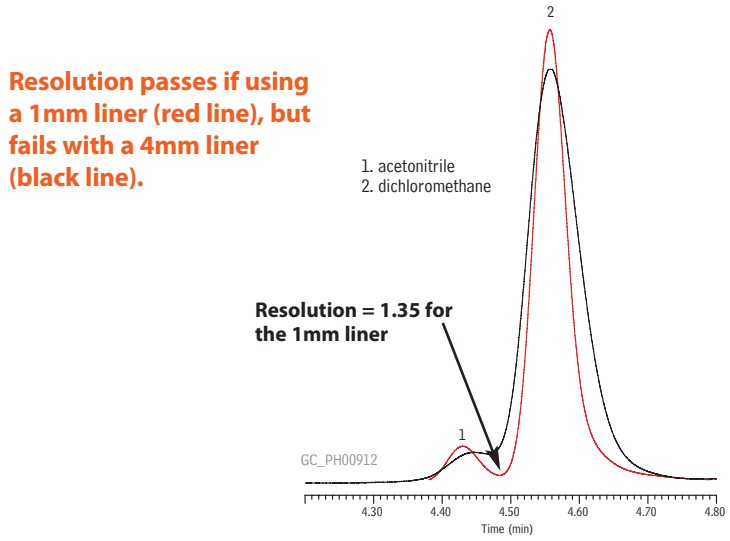
*Nominal ID at syringe needle expulsion point.
**Use this liner for increased sensitivity.

Implementing the revised method for USP<467> can be difficult if the instrument is not optimized correctly. Key issues to address when setting up headspace GC systems include minimizing system dead volume, maintaining inert sample flow paths, and achieving efficient sample transfer. While the second supplement contains a change that allows for modifications to the split ratio, column and liner choices are critical to analytical success.

Use Smaller Bore Liners for Better Resolution

The function of an injection port in headspace analysis is very different than in direct liquid injection. In direct injection, the sample is vaporized in the injection port and larger volume liners (e.g., 4mm) are typically used since the liner must be able to accommodate the solvent expansion volume. In contrast, in headspace analysis, the sample is vaporized inside the headspace vial and the resulting gas sample is simply transferred into the injection port via a transfer line or syringe injection. Since solvent vaporization does not occur in the liner, a large volume liner is not needed and, in fact, the use of one can cause deleterious effects such as band broadening and decreased peak efficiency. For headspace applications, a smaller bore liner, preferably 1mm, is recommended. The smaller liner volume reduces band broadening by increasing linear velocity in the liner allowing faster sample transfer and improving resolution (Figure 10).

Figure 10 Improve system suitability pass rates using smaller bore liners.



Speed Up Method Development Using a Retention Time Index

ICH guideline Q3C states that residual solvents need only be tested when production or purification processes are known to result in the presence of such solvents. Therefore, in many cases exhaustive testing is not needed and individual validated methods for smaller, specific analyte lists are an option. To simplify column selection and reduce method development time, Restek has created a retention time index for ICH Class 1, 2, and 3 residual solvents on various phases (Table I). To use this index, simply locate the analytes of interest on the list and determine which phase gives the optimal amount of resolution—or difference in retention time—between your target compounds. A critical coelution is indicated by a failure to achieve a retention time difference of greater than 1.5 minutes.

Table I Reduce method development time—use a retention time index for column selection.

Retention time data collected using the following conditions:
G16 Stabilwax®: 30m, 0.25mm ID, 0.5µm df, Phase ratio: 125, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.2mL/min., Dead time: 1.38 min. @ 45ºC
G16 Rtx®-WAX: 30m, 0.25mm ID, 0.5µm df, Phase ratio: 125, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.2mL/min., Dead time: 1.40 min. @ 45ºC
G43 Rtx®-1301: 30m, 0.25mm ID, 1.0µm df, Phase ratio: 63, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.2mL/min., Dead time: 1.40 min. @ 45ºC
G27 Rxi®-5ms: 30m, 0.25mm ID, 1.0µm df, Phase ratio: 63, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.1mL/min., Dead time: 1.49 min. @ 45ºC
G1 Rtx®-1: 60m, 0.53mm ID, 3.00µm df, Phase ratio: 43, Oven program: 30ºC, hold 4 min., to 220ºC @ 4ºC/min., Carrier flow: 6.3mL/min., Dead time: 2.54 min. @ 35ºC
Rtx®-200: 60m, 0.53mm ID, 3.00µm df, Phase ratio: 43, Oven program: 30ºC, hold 4 min., to 220ºC @ 4ºC/min., Carrier flow: 7.8mL/min., Dead time: 2.22 min. @ 35ºC

Carrier gas: helium Compound	ICH Class	G16 Stabilwax® Retention Time	G16 Rtx®-WAX Retention Time	G43 Rtx®-1301 Retention Time	G27 Rxi®-5ms Retention Time	G1 Rtx®-1 Retention Time	NA Rtx®-200 Retention Time
1,1,1-trichloroethane	1	3.96	3.49	5.43	5.40	10.82	8.35
1,1,2-trichloroethene	2	15.72	14.28	10.99	9.77	16.75	14.94
1,1-dichloroethene	1	2.23	2.04	2.79	4.41	5.73	4.16
1,2-dichloroethane	1	8.80	7.68	6.15	5.46	10.38	9.74
cis-1,2-dichloroethene	2	6.50	5.65	4.79	2.88	8.71	7.11
trans-1,2-dichloroethene	2	3.63	3.20	3.55	3.54	7.17	5.16
1,2-dimethoxyethane	2	4.80	4.18	6.03	5.54	10.98	10.63
1,4-dioxane	2	8.55	7.49	7.86	7.26	13.54	14.34
1-butanol	3	11.13	10.08	7.18	5.76	11.49	10.13
1-pentanol	3	14.95	13.75	11.19	9.44	16.99	14.95
1-propanol	3	7.69	6.80	4.20	3.37	6.81	6.13
2-butanol	3	7.25	6.44	5.08	4.16	8.51	7.69
2-ethoxyethanol	2	13.99	12.70	8.69	7.36	13.91	13.99
2-methoxyethanol	2	12.42	11.11	6.02	5.14	9.83	10.74
2-methyl-1-propanol	3	9.32	8.40	6.00	4.79	*	*
2-propanol	3	4.81	4.25	3.00	2.55	4.91	4.69
3-methyl-1-butanol	3	13.42	12.25	9.86	8.26	15.28	13.55
acetic acid	3	22.47	20.34	6.52	4.61	8.84	8.96
acetone	3	3.02	2.64	2.89	2.50	4.64	7.68
acetonitrile	2	6.91	5.83	3.28	2.47	4.32	8.89
anisole	3	18.65	17.09	17.12	16.28	25.00	22.84
benzene	1	5.23	4.54	5.98	3.83	11.63	9.17
butyl acetate	3	8.86	7.88	12.12	11.38	19.43	19.63
carbon tetrachloride	1	3.96	3.49	5.61	5.90	11.89	7.42
chlorobenzene	2	13.91	12.54	13.55	13.14	21.56	18.48
chloroform	2	7.31	6.41	5.23	4.64	9.18	6.66
cumene	3	12.36	11.17	16.66	16.69	25.88	20.90
cyclohexane	2	2.16	2.01	5.37	5.89	*	*
dichloromethane	2	5.01	4.33	3.31	3.06	5.87	4.88
dimethylsulfoxide	3	26.47	24.43	16.62	13.01	18.81	30.95
ethanol	3	4.98	4.37	2.52	2.19	4.03	3.80
ethyl acetate	3	4.08	3.56	4.87	4.44	9.04	10.35
ethyl benzene	2	10.72	9.58	13.86	13.81	22.54	18.18
ethyl ether	3	1.72	1.63	2.58	2.67	5.34	3.87
ethyl formate	3	3.16	2.78	3.00	2.78	5.46	6.48
ethylene glycol	2	28.06	26.23	10.77	6.63	12.59	13.86
formamide	2	32.99	30.93	11.85	7.30	12.72	19.93
formic acid	3	24.64	22.09	5.19	2.60	5.59	5.06
heptane	3	1.98	1.86	6.34	6.98	14.18	7.84
hexane	2	1.65	1.58	3.77	4.11	9.06	4.86
isobutyl acetate	3	6.99	6.18	10.39	9.69	17.35	18.02
isopropyl acetate	3	4.26	3.74	6.19	5.71	11.47	12.38
methanol	2	4.23	3.64	1.96	1.80	3.14	2.93
methyl acetate	3	3.19	2.80	3.17	2.93	5.80	7.10
methylbutyl ketone	2	9.10	8.05	11.81	10.50	17.94	20.81
methylcyclohexane	2	2.50	2.30	7.31	7.95	15.49	9.21
methylethyl ketone	3	4.33	3.76	4.90	4.09	7.99	11.55
methylisobutyl ketone	3	6.84	5.97	9.64	8.49	15.35	18.41
m-xylene	2	11.21	10.04	15.46	14.17	23.01	18.78
N,N-dimethylacetamide	2	20.75	19.01	12.95	13.96	21.42	30.00
N,N-dimethylformamide	2	18.04	16.26	13.09	10.23	16.52	26.19
nitromethane	2	11.82	10.31	4.84	3.53	6.30	12.01
N-methylpyrrolidone	2	29.84	27.86	25.09	21.85	29.99	38.08
o-xylene	2	12.79	11.51	15.46	15.26	24.23	20.33
pentane	3	1.49	1.45	2.39	2.62	5.36	3.29
propyl acetate	3	5.98	5.29	8.03	7.44	*	*
p-xylene	2	10.98	9.82	14.29	15.27	22.99	18.69
pyridine	2	12.64	11.24	9.60	8.57	15.40	16.45
sulfolane	2	47.62	43.31	34.02	28.90	36.76	48.67
tert-butylmethyl ether	3	1.94	1.82	3.50	3.59	7.52	5.73
tetrahydrofuran	3	3.63	3.19	5.12	4.90	9.81	9.48
tetralin	2	25.12	23.48	27.49	27.44	37.27	31.72
toluene	2	7.86	6.91	9.80	9.66	17.36	14.00
1,1-diethoxypropane	—	5.42	4.84	11.39	11.38	19.82	15.08
2,2-dimethoxypropane	—	3.11	2.79	5.48	5.55	11.37	8.67
2-chloropropane	—	1.96	1.82	2.67	2.66	5.20	4.61
2-methylpentane	—	1.58	1.52	3.22	3.56	7.72	4.32
acetaldehyde	—	2.05	1.85	1.86	1.84	3.14	3.90
chloroethane	—	1.83	1.71	2.14	2.10	3.97	3.55
chloromethane	—	1.63	1.55	1.70	1.70	3.01	2.73
ethylene oxide	—	2.05	1.86	1.89	2.02	3.59	3.92
formaldehyde	—	2.25	1.57	1.68	1.58	2.66	2.59
isoamyl acetate	—	10.51	9.43	14.84	14.18	22.80	22.62
isooctane	—	1.85	1.75	5.84	6.59	13.66	8.07
isopropyl ether	—	1.86	1.76	4.03	4.23	9.03	5.83
methyl cyclopentane	—	1.91	1.79	4.50	4.93	10.41	5.81
methyl isopropyl ketone	—	4.93	4.29	6.58	5.69	11.04	14.47
methylal	—	2.26	2.06	2.84	2.82	5.65	5.09
trichloroethene	—	6.50	5.70	7.07	7.05	13.58	9.75
water	—	8.24	7.18	1.74	1.68	2.75	2.57

* Not determined

Restek Offers An Extensive Selection of Capillary Columns

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(lit. cat.# 59224A)

Rtx®-624 Columns (fused silica) G43

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	1.40	-20 to 240°C	10968	10969
0.32mm	1.80	-20 to 240°C	10970	10972
0.53mm	3.00	-20 to 240°C	10971	10973
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**
0.18mm	1.00	-20 to 240°C	40924	40925

Rtx®-1301 Columns (fused silica) G43

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits*	30-Meter	60-Meter
0.25mm	0.50	-20 to 270°C	16038	16041
	1.00	-20 to 260°C	16053	16056
	1.40	-20 to 240°C		16016
0.32mm	0.50	-20 to 270°C	16039	16042
	1.00	-20 to 260°C	16054	16057
	1.50	-20 to 250°C	16069	16072
	1.80	-20 to 240°C	16092	16093
0.53mm	0.50	-20 to 270°C	16040	16043
	1.00	-20 to 260°C	16055	16058
	1.50	-20 to 250°C	16070	16073
	3.00	-20 to 240°C	16085	16088

Stabilwax® Columns (fused silica) G16

(Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.25	40 to 250°C	10623	10626
	0.50	40 to 250°C	10638	10641
0.32mm	0.25	40 to 250°C	10624	10627
	0.50	40 to 250°C	10639	10642
	1.00	40 to 240/250°C	10654	10657
0.53mm	1.00	40 to 240/250°C	10655	10658
	1.50	40 to 230/240°C	10669	10672
	2.00	40 to 220/230°C	10670	

also **available**

Custom Column Lengths:
If you do not see the column dimension you need, call our customer service team, and we will make the column for you.

Rxi®-5ms Columns (fused silica) G27

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.50	-60 to 330/350°C	13438	13441
	1.00	-60 to 330/350°C	13453	13456
0.32mm	0.50	-60 to 330/350°C	13439	13442
	1.00	-60 to 330/350°C	13454	13457
0.53mm	1.00	-60 to 330/350°C	13455	
	1.50	-60 to 330/350°C	13470	
ID	**df (µm)**	**temp. limits**	**20-Meter**	
0.18mm	0.18	-60 to 330/350°C	13402	
	0.30	-60 to 330/350°C	13409	
	0.36	-60 to 330/350°C	13411	

Rtx®-1 Columns (fused silica) G1

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.50	-60 to 330/350°C	10138	10141
	1.00	-60 to 320/340°C	10153	10156
0.32mm	1.00	-60 to 320/340°C	10154	10157
	1.50	-60 to 310/330°C	10169	10172
	3.00	-60 to 280/300°C	10184	10187
	4.00	-60 to 280/300°C	10198	
	5.00	-60 to 260/280°C	10178	10180
0.53mm	1.50	-60 to 310/330°C	10170	10173
	3.00	-60 to 270/290°C	10185	10188
	5.00	-60 to 270/290°C	10179	10183
	7.00	-60 to 240/260°C	10192	10193
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**
0.18mm	0.20	-60 to 330/350°C	40102	40103
	0.40	-60 to 320/340°C	40111	40112

Rtx®-200 Columns (fused silica)

(Crossbond® trifluoropropylmethyl polysiloxane)

ID	df (µm)	temp. limits*	30-Meter	60-Meter
0.25mm	0.50	-20 to 310/330°C	15038	15041
	1.00	-20 to 290/310°C	15053	15056
0.32mm	1.00	-20 to 290/310°C	15054	15057
	1.50	-20 to 280/300°C	15069	15072
0.53mm	1.00	-20 to 290/310°C	15055	15058
	1.50	-20 to 280/300°C	15070	15073
	3.00	-20 to 260/280°C	15085	15088
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**
0.18mm	0.20	-20 to 310/330°C	45002	45003
	0.40	-20 to 310/330°C	45011	45012

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

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- Vespel® ring embedded in bottom surface eliminates need for washer.
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Siltek Treated	21242	21243
Stainless Steel	21238	21239
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Gold-Plated	21246	21247
Siltek Treated	21248	21249
Stainless Steel	21244	21245

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- Ideal for high-flow split applications.
- Washerless, leak-tight seals.

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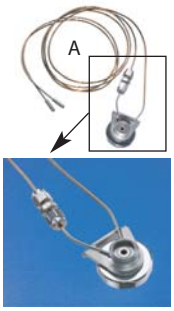
Gold-Plated	22083	22084
Siltek Treated	22085	22086
Stainless Steel	22087	22088

Injection Port Weldments for Agilent GCs

Easily attach your autosampler with pre-installed low dead volume fittings.

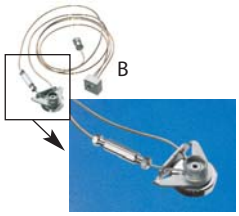


For Agilent GCs with Tekmar Transfer Lines

Description	qty.	cat.#
A) Weldment for Agilent 6890 GCs	ea.	22664
Weldment for Agilent 6890 GCs with optional canister filter	ea.	22668
Weldment for Agilent 5890 GCs	ea.	22666

For Agilent GCs with OI Purge and Trap Systems

Description	qty.	cat.#
B) Weldment for Agilent 6890 GCs	ea.	22665
Weldment for Agilent 6890 GCs with optional canister filter	ea.	22669
Weldment for Agilent 5890 GCs	ea.	22667



FID Replacement Jets

Standard Version

- Engineered with a fluted tip to guide the capillary column into the jet.
- Threads specially coated for easy installation and removal.
- Special processing ensures the highest degree of cleanliness.

High-Performance Version

- Similar to the standard version, but Siltek® treated.
- Extremely inert, for use with active compounds.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs

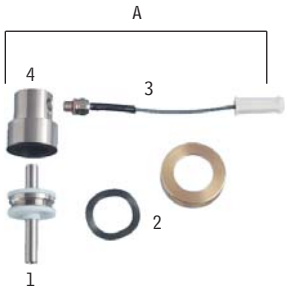
0.011-Inch ID Tip	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard, 0.011-Inch ID Tip	19244-80560	ea.	20670	3-pk.	20671
High-Performance Siltek Treated, 0.011-Inch ID Tip	19244-80560	ea.	20672	3-pk.	20673

Capillary Dedicated FID Replacement Jet for Agilent 6890/6850/7890 GCs

0.011-Inch ID Tip	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard, 0.011-Inch ID Tip	G1531-80560	ea.	21621	3-pk.	21682
High-Performance Siltek Treated, 0.011-Inch ID Tip	G1531-80560	ea.	21620	3-pk.	21683

did you **know**?

Restek carries a full line of FID replacement jets. Visit **www.restek.com** for a complete selection.



Direct Replacement FID Collector Assembly Kit for Agilent 6890/6850/7890 GCs

- Constructed of high-quality stainless steel.
- Meets or exceeds manufacturer's performance.

Description	Similar to Agilent part #	qty.	cat.#
A) FID Collector Assembly Kit (includes insulator)	G1531-60690	kit	21699
FID Collector Assembly Kit w/Siltek Ignitor Castle	—	kit	21132

Replacement FID Parts for Agilent 6890/6850/7890 GCs

- Meets or exceeds manufacturer's performance.

Description	Similar to Agilent part #	qty.	cat.#
1) FID Collector (includes insulators)	G1531-20690 G1531-20700	ea.	21139
2) FID Collector Nut and Washer	19231-20940 5181-3311	set	21136
3) FID Ignitor*	19231-60680	ea.	21001
4) FID Ignitor Castle	19231-20910	ea.	21137
Siltek FID Ignitor Castle	—	ea.	21135

*Also fits OI Analytical 4410 detector (similar to OI part # 191833).

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Description	qty	cat.#
Leak Detector with Universal Adapter Set	ea	22839

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