Sampling Techniques Handbook

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Contents

Vials and Bottles

1

Caution

Filling sample vials

Figure 1 shows the recommended fill volumes for sample vials of:

- 1 mL for the 2 mL vial
- 50 µL for the 100µL vial

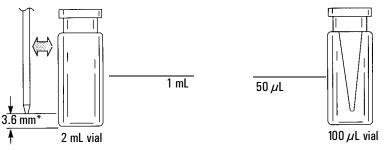
The air space in the vial is necessary to avoid forming a vacuum when sample is withdrawn. This could affect reproducibility.

Do not inject air into the vials to prevent this vacuum. This often damages the cap seal.

When developing your method, keep the following in mind:

- If you need to test a large amount of sample over repeated injections, divide the sample among several vials to obtain reliable results.
- When sample volume in the vial is low, contaminants from the previous sample injection or solvent washes may have a greater impact on the sample.

If you change suppliers, you may need to re-develop your method. Differing manufacturing practices for vial hardware sometimes cause variances in your results.



* Needle position based on standard sampling depth.

Figure 1 Recommended fill volumes for sample vials

Capping sample vials

When a tray is not installed, you may be able to use sample vials with no caps, snap-on caps, or screw-on caps depending on your application. If a tray is used, sample vials must have caps installed. See Figure 2.

To install the airtight crimp caps:

- 1. Clean the inside surfaces of the crimper jaws.
- 2. Place the crimp cap over the top of the vial.
- 3. Lift the vial into the crimper. Squeeze the handle until it reaches the adjuster screw.

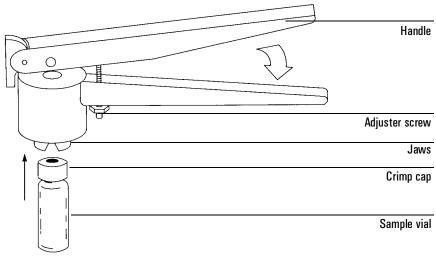


Figure 2 Crimping caps

Figure 3 shows acceptable and unacceptable vial caps.

Check each vial for proper crimping:

- 1. Be sure there are no folds or wrinkles on the part of the cap that wraps under the neck of the vial. To remove folds or wrinkles, turn the vial about 10° and crimp it again. Adjust the crimper for a looser crimp by turning the adjusting screw clockwise.
- 2. Check that the cap cannot be turned by hand. If the cap is loose, adjust the crimper for a tighter crimp by turning the adjusting screw counterclockwise. Crimp the cap again.
- 3. Be sure that each cap has a flat septum centered over the top of the vial.
 - If the septum is not flat, remove the cap, turn the crimper adjusting screw clockwise, and try again.
 - If the cap is not centered, remove the cap and make sure the new cap is flat on the top of the vial before you squeeze the crimper.

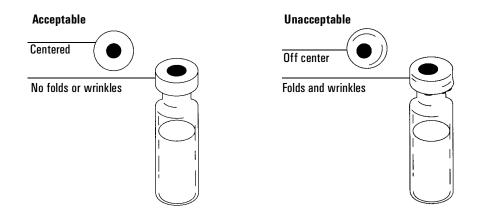


Figure 3 Acceptable and unacceptable caps

Preparing the solvent and waste bottles

The solvent bottles hold solvent for rinsing the syringe between injections. The injector dispenses the solvent washes and sample washes into waste bottles.

Selecting the bottles

The injector uses 4-mL bottles to hold the solvent and waste. You can use either diffusion caps (a plastic cap with a hole; it retards evaporation while letting the needle enter freely) or septa on these bottles. Agilent Technologies recommends diffusion caps (See Figure 4) over septa for two reasons:

- The diffusion cap allows multiple entrances into a bottle without contaminating the liquid inside the bottle with small pieces of septum material.
- For common solvents, the rate of diffusion out of the bottle is less with a diffusion cap than with a septum that has been punctured with a standard syringe needle.

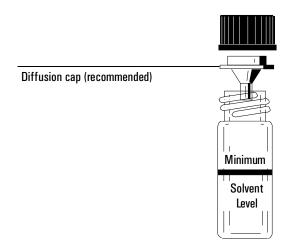


Figure 4 4 mL bottle used for solvent or waste

Filling and placing the bottles

Before each sequence or group of sequences, prepare your solvent and waste bottles as described in your sampler operating manual. Consider the following points:

- Rinse each solvent bottle and fill with 4 to 4.5 mL of fresh solvent. The liquid level should be near the shoulder of the bottle. If the solvent bottle is filled with 4.5 mL of solvent, the syringe can reach about 2 mL (about 250 washes for a 10-µL syringe).
- Do not refill a solvent bottle that still has solvent left in the bottle. The solvent may be contaminated from the last analysis. Discard the remaining solvent, rinse, and fill.
- Empty and rinse each waste bottle. The syringe can discard about 4 mL of waste into the waste bottle (about 500 washes for a 10-µL syringe).

Place the bottles in the appropriate positions on the injector turret according to your model type.

How many sample vials can I run?

The number of sample vials that you can run at one time may be limited by the volumes of the solvent and waste bottles as shown in Table 1 and examples 1, 2 and 3 below. If your application requires more than the maximum number of washes or discards listed in Table 1 below, estimate the maximum number of sample vials using equations described later in this section.

After reaching either limit, you must replace the solvent bottles or empty the waste bottles before running any more samples.

Table 1Maximum Number of Washes (Pre- and Post-Injection) or Waste
Discards

Number of Bottles					Maximum Number of Waste Discards (Sample plus solvent wash discards)					
Syringe size	5 μL	10 µL	25 μL	50 µL	100 µL	5 μL	10 µL	25 µL	50 µL	100 µL
Two bottles	1,000	500	200	100	50	2,000	1,000	400	200	100
One bottle	500	250	100	50	25	1,000	500	200	100	50

Note: Wash volume is 0.8 times the syringe volume.

Caution

Do not exceed the solvent and waste limits of the bottles. If you do, sample carryover may affect your analysis.

Example 1 (tray not installed): Your application uses a 10 µL syringe to inject from 3 sample vials, making 5 injections per sample. Each analysis uses 10 sample washes and 10 solvent washes. You will need 150 solvent washes and you will discard 300 syringe volumes of waste (150 from solvent washes and 150 from sample washes). Because a tray is not present, you are limited to one bottle for solvent and one bottle for waste.

Table 1 shows that you can have up to 250 washes from one solvent bottle and can discard up to 500 washes into the waste bottle. You are well within the limits and can proceed with your analyses.

Example 2a (tray installed): Your application uses a 10-µL syringe to inject from 40 sample vials, making 2 injections per vial. Each analysis uses 3 sample washes and 3 solvent washes. You will need 240 solvent washes and you will discard 480 syringe volumes of waste. You are using two bottles for solvent and two bottles for waste.

Table 1 shows that you can have up to 250 washes from each solvent bottle and can discard up to 1,000 washes into the waste bottles. Only 1 solvent bottle is needed. You are within the limits and can proceed with your analyses.

Example 2b (tray installed): This is the same as **Example 2a** except that you are running 60 samples. You will need 360 washes from the solvent bottles. You must use 2 solvent bottles and must set the run parameters for solvent washes from both positions (e.g., one from solvent A and two from solvent B). The wash requirement is still well within the table limits.

Example 3 (tray installed): Your application requires three sample washes, three solvent A washes, and three solvent B washes with a 10-µL syringe. For 100 sample vials (two injections per vial), you will need 600 solvent washes and will discard 1,200 syringe volumes of waste. With this example, you are using two bottles for solvent and two bottles for waste.

Table 1 shows that you can have up to 250 washes from each solvent bottle and can discard up to 1,000 washes into the waste bottles. You exceed both the solvent and waste limits. The job will have to be split into two runs. Read the next section to estimate the maximum number of sample vials you can run at one time.

Estimating the maximum number of sample vials

CautionThe number of sample vials given by these equations are estimates. Solvent
characteristics such as evaporation rate and surface tension may affect the
capacity of the bottles.

To use equations **S** and **W** described below, you must know the following parameters for your application:

• The number of injections per vial.

- The number of solvent washes (both pre- and post-injection) required from each solvent bottle.
- The number of sample washes and solvent washes per injection that the injector discards into each waste bottle.
- The syringe size used: 5 µL, 10 µL, 25 µL, 50 µL, or 100 µL.
- The number of waste bottles used. When using two bottles, the injector discards the waste equally between them unless you have specified differently.

Using equations S and W

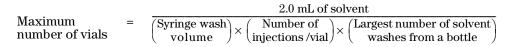
- 1. Substitute the parameters of your application into both equations.
 - Substitute the wash volume, listed in Table 2, into both equations.
 - If two waste bottles are being used, substitute 8.0 mL of waste for the 4.0 mL of waste in **Equation W**.
- 2. Calculate the answers for both equations. Use the smaller of the two answers for the estimate.

Syringe Size µL	Wash Volume µL/wash
5	0.004
10	0.008
25	0.020
50	0.040
100	0.080

Table 2Syringe Wash Volumes

Equation S

Equation **S** estimates the maximum number of vials you can run from the volume of solvent available in the bottle associated with the largest number of washes.



Equation W

Equation \mathbf{W} estimates the maximum number of vials you can run from the waste bottle capacity.

		4.0 mL of waste
Maximum number of vials	=	$\underbrace{\left(\begin{array}{c} \text{Syringe wash} \\ \text{volume} \end{array}\right) \times \left(\begin{array}{c} \text{Number of} \\ \text{injections /vial} \end{array}\right) \times \left(\begin{array}{c} \text{Number of solvent plus} \\ \text{sample washes /injection} \end{array}\right)}$

Equation example

Assume a tray is installed and your application parameters are:

- Two injections per vial
- Three washes from solvent bottle A
- Two washes from solvent bottle B
- Two sample washes
- 10-µL syringe
- Two waste bottles used
- 1. Substitute the parameters of your application into both equations:
 - **S:** Maximum number of vials = $2.0 / (0.008 \times 2 \times 3) = 41$
 - **W:** Maximum number of vials = $8.0 / (0.008 \times 2 \times 7) = 71$
- 2. Use 41, the smaller of the two results, as your estimate.

$\overline{2}$

Syringes and Needles

Selecting syringes

1. Select the syringe **type** based on the inlet (injection port) you are using and the volume of sample you want to inject.

Caution Failure to use an on-column syringe when injecting into an on-column inlet could damage the injector, syringe and column.

- 2. Select a syringe. Refer to your automatic liquid sampler operating documentation for available syringe sizes and corresponding injection volumes. Also refer to the Agilent catalog for consumables and supplies for part numbers and ordering information.
- 3. Select the appropriate syringe needle gauge. Refer to Table 3 below.

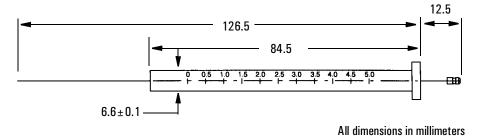


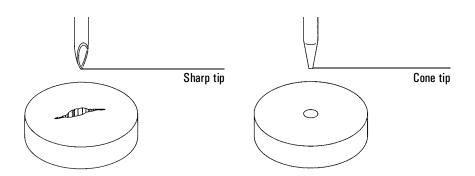
Figure 5 shows some of the critical syringe dimensions.

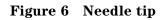
Figure 5 Syringe dimensions

Table 3 Needle Gauge Selection

Inlet	Needle Gauge	Column Type
Packed, split or splitless (including PTV)	23 Gauge or 23/26 Gauge tapered	Any Applicable
Cool on-column	23/26 Gauge tapered or 26 gauge 26/32 Gauge tapered 26/32 Gauge tapered	530 μm 320 μm 250 μm

Use syringe needles with a conical tip. Do not use sharp-tipped needles. They tear the inlet septum and cause leaks. Also, a sharp-tipped needle tends to wipe off on the septum as it exits resulting in a large solvent tail on the chromatogram. See Figure 6 and Figure 7.





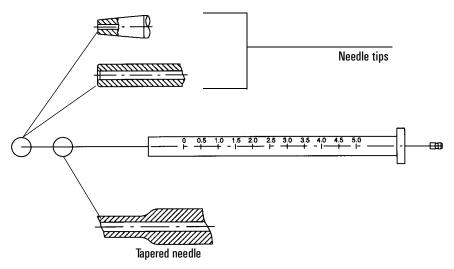


Figure 7 Needle shapes

Replacing syringe needles

The stainless steel needles used for 250-µm and 320-µm injections must be inserted into a glass syringe barrel (the 5 µL syringe barrel is part no. 5182–0836). Select the correct size needle for the column you plan to use. Needles for 250-µm injections (part no. 5182-0833, 3/pk) have silver-colored stops. Needles for 320-µm injections (part no. 5182-0831, 3/pk) have gold-colored stops. See your Agilent catalog for consumables and supplies for a complete list of syringes and needles.

To insert a needle into a syringe barrel:

- 1. Unscrew the syringe barrel cap and remove the spring.
- 2. Make sure the needle has the Teflon disk as shown in Figure 8. If the syringe barrel does not have the Teflon disk, use the instructions in the syringe box to wrap the needle yourself.
- 3. Slide the spring and the cap down over the needle.
- 4. Insert the needle into the syringe barrel.
- 5. Screw the cap back on the syringe barrel.

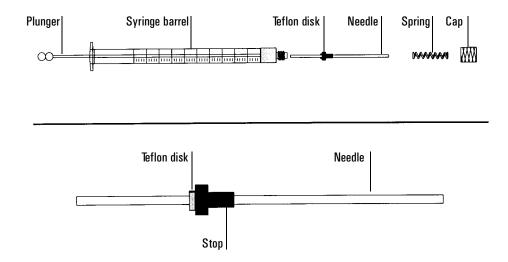


Figure 8 Syringe parts and assembly

Syringes and Needles **Replacing syringe needles** 3

Troubleshooting

Chromatographic symptoms

This chapter deals only with sampler-related problems. However, many of the symptoms described here could also come from other sources, particularly the stability of the gas chromatograph temperature and its gas supplies.

If you cannot correct the problem, obtain Agilent service.

Symptom: Variability



Retention times or areas are not reproducible.

Figure 9 Retention times or areas are not reproducible

Possible

Cause: Inlet septum is leaking

Action: If the septum is leaking, replace it. If the septum you replaced experienced less than 200 injections, check for the following possible problems to prevent premature septum failure:

- The septum retainer nut is too tight.
- The syringe needle is not straight.
- The syringe is not installed correctly.

Troubleshooting **Variability**

Possible Cause:	Syringe is worn or dirty
Action:	If the syringe looks dirty or the plunger is sticking, clean the syringe with an appropriate solvent or follow the syringe manufacturer's cleaning instructions.
Possible Cause:	Sample volume is too low or too high
Action:	Check sample level. If the sample vials are not filled correctly, evaporation or contamination may affect the analysis. The sample level should be approximately half the volume of the vial. See <i>Filling sample vials</i> on page 2.
Possible Cause:	Vial caps are loose
Action:	Check vial caps. If you can turn the vial crimp caps by hand, they are too loose. Loose caps may cause volatile samples to change concentration over time. See <i>Capping sample vials</i> on page 3.
Possible Cause:	Sample is not stable
Action:	Check the sample stability. Some samples change with heat or ultraviolet light. There are several ways to reduce changes to unstable samples:
	 Use the tray quadrants to cool the sample. Use amber sample vials. Store the samples in a protected environment.
Possible Cause:	Sample size varies
Action:	Install a new syringe. If the sample size varies, the syringe is probably not precise or the plunger is worn. Variations may be due to syringes with removable needles because of dead volume or needle-to-needle variation.
Possible Cause:	Air bubbles are in the needle
Action:	If air bubbles are in the needle, increase the run parameter that controls the number of sample pumps. See your controlling device operating manual.

If this does not help and the sample is viscous, try the following:

- Increase the viscosity delay time.
- Use the tray quadrants to warm the sample.
- Dilute the sample in an appropriate low-viscosity solvent.

Symptom: Contamination or ghost peaks

Possible Cause:	Vial cap septum is dissolving in solvent Ghost peaks sometimes appear when small pieces of septum material dissolve in the sample. Make several blank runs to determine the presence or absence of the ghost peaks.
Action:	 Check for the following: Be sure the vial septum is flat. If the vial septum is not flat, the needle tends to core the septum and drop pieces into the sample. See <i>Capping sample vials</i> on page 3. Check the needle. If the syringe needle has burrs, it could cut pieces of the septum and push them into the sample. Check the vial septum. If the vial septum is not resistant enough to the solvent you are using, try a more resistant type.
Possible Cause:	Sample vials are contaminated
Action:	Ghost peaks are sometimes caused by contaminated sample vials. Try new or clean vials to see if ghost peaks disappear. Store new vials in a contamination- free location.
Possible Cause:	Injection port septum is giving off volatiles
Action:	Make several blank runs with a small piece of aluminum foil backing the inlet septum. If the contamination peaks disappear, they were probably due to the septum. Try replacing the septum you usually use with another type.

Possible	
Cause:	Column is contaminated
	High molecular weight samples that contain residues may cause the syringe, the inlet liner, or the first few inches of column to become contaminated.
Action:	Do the following:
	• Replace or clean and deactivate the inlet liner.
	• Examine the first few inches of a capillary column for foreign material by holding a light behind it. If possible, remove the contaminated section.
Possible	
Cause:	Sample is not stable
	Some samples change with heat or ultraviolet light.
Action:	Check the sample's stability. There are several ways to reduce the change:
	• Use the tray quadrants to cool the sample.
	• Use amber sample vials.
	• Store the samples in a protected environment.

Symptom: Smaller or larger peaks than expected

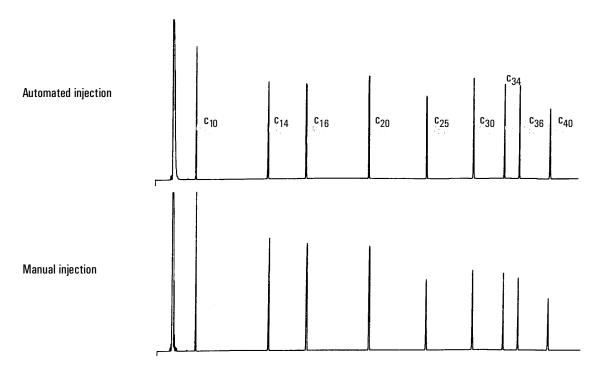


Figure 10 Smaller or larger peaks than expected

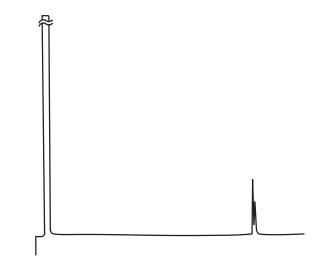
Possible

Cause: You are comparing a chromatogram without needle fractionation against one with needle fractionation

Action: Check your injection mode. In the normal injection mode, the sampler uses fast injection to deliver a representative amount of the sample. Fast injection minimizes needle fractionation. Chromatograms from manual injection or slower auto injection devices show higher levels of low molecular weight materials versus higher molecular weight materials because the volatiles boil out of the needle faster than the higher weight materials.

Possible Cause:	You are using a packed inlet and a 530-µm column
Action:	Check your inlet. Capillary columns used with packed inlets have some inherent sample discrimination characteristics. See <i>Suggestions for packed inlets with</i> 530 - μm columns on page 31.
Possible Cause:	There is a leak in the GC system
Action:	Replace the septum and check the fittings for leaks. If the leaking septum has experienced less than 200 injections.
	To prevent future premature failures, be sure that:
	 The septum retainer nut is not too tight. The syringe needle is straight. The syringe is installed correctly. The injector is aligned to the injection port (7673 only). See the appropriate manual that describes the installation.
Possible Cause:	Sample is not stable
Action:	Some samples change with heat or ultraviolet light. Check the sample stability. There are several ways to reduce the change:
	Use the tray quadrants to cool the sample.Use amber sample vials.Store the samples in a protected environment.
Possible Cause:	Vial caps are loose
Action:	Check the vial caps. Loose vial caps can cause selective loss of lighter materials from a sample. The caps should not rotate easily if installed properly. See <i>Capping sample vials</i> on page 3.

Symptom: Sample carryover



Blank run showing carryover peaks.

Figure 11 Blank run showing carryover peaks

Possible Cause:	Number or type of washes is insufficient
Action:	Check the run parameters for the number of sample and solvent washes. The number of washes needed depends on your application. See <i>Controlling sample carryover</i> on page 30.
Possible Cause:	You ran out of solvent
Action:	Check the solvent bottles. If the solvent level is below 2.5 mL, the syringe cannot reach the solvent. Replace the remaining solvent with 4 to 4.5 mL of fresh solvent. See <i>Filling and placing the bottles</i> on page 6.

	Check the waste bottles. If the waste level is near the neck of the bottle, replace it with an empty bottle.
Possible Cause:	Syringe is worn or dirty
Action:	If the syringe looks dirty or the plunger is sticking, clean the syringe with an appropriate solvent or follow the syringe manufacturer's cleaning instructions. If the syringe seems worn, replace it.
Possible Cause:	Samples (vial-to-vial) are of immiscible types
Action:	In this situation, the sample and solvent washes may not rinse the syringe properly. Increase the number of wash cycles or use a solvent that rinses a variety of sample types.

Symptom: No signal/no peaks

Possible Cause:	Syringe plunger is malfunctioning
Action:	Verify that the syringe plunger is secured by the plunger screw. If the plunger screw is loose, tighten it. See your operating manual.
	Check the syringe needle for plugging. If the syringe is plugged, replace or clean the syringe.
Possible Cause:	Sample level is too low in vial
Action:	If there is no or very little sample in the vial, the needle may not be able to reach it. See <i>Filling sample vials</i> on page 2.
	Alternately, with some injectors you may edit your method to adjust the needle sampling depth. See your operating manual.
Possible Cause:	Sample is viscous
Action:	If the sample is viscous, try the following:
	 Increase the viscosity delay time. Use the true was depute to grow the neurophysical second sec
	Use the tray quadrants to warm the sample.Dilute the sample in an appropriate low-viscosity solvent.
	 Turn the tower fan off (selected models).

$\overline{4}$

Special Topics

Controlling sample carryover

This section describes the features of the injector used to control carryover, the presence of peaks from an earlier injection in the present analysis.

By reducing or eliminating carryover, you may be able to adjust your application for a more efficient use of solvent and sample, and increase the number of sample vials you can run at one time.

What your injector can do to reduce sample caryover

Your injector can perform solvent washes, sample washes, and sample pumps to control carryover. Each of these actions reduces the concentration of sample left in the syringe after an injection. The effectiveness of each depends on your application.

Solvent wash

During a solvent wash, the injector fills the syringe to 80% of its volume (for example, 4 μ L with the 5 μ L syringe and 8 μ L with the 10 μ L syringe) from either the solvent A or solvent B position. Then it discards the syringe contents into one of the waste bottles. Solvent washes can be set to occur before taking a sample (pre-injection solvent wash) or immediately after the injection (post-injection solvent wash).

Sample wash

During a sample wash, the injector fills the syringe to 80% of its volume with the next sample and discards the contents into one of the waste bottles. Sample washes occur before the injection. When sample is limited, you can use a solvent prewash to wet the syringe before drawing sample.

Sample pump

During a sample pump, the injector fills the syringe to 80% of its volume with the next sample and returns it to the sample vial. Pumps occur after the sample washes and immediately before the injection. Pumps serve to eliminate bubbles. If the needle contains solvent from a previous wash, the pumps may add a small amount of solvent that mixes with the sample and can dilute a small volume.

Number and type of washes

Under ideal conditions, four washes reduce the carryover to one part in 10,000. The actual number and type of washes you need depends on many factors, including:

- The percentage of carryover that you can accept
- The viscosity and solubility of the analyte(s)
- The viscosity and volatility of the solvent(s)
- The degree of wear in the syringe barrel

The number and type of washes is often set for you as a standard method. You can also determine the number and type of washes experimentally.

To measure the percentage of carryover in your procedure, run a solvent blank after a sample and compare the peak areas of the components.

Suggestions for packed inlets with 530-µm columns

When using a heated, packed inlet with a 530-µm column, do the following:

- Install the column so that no more than 1 to 2 mm of the column extends past the ferrule. This avoids large unswept volumes at the base of the inlet.
- Use polyimide ferrules (Vespel) instead of graphite. A small portion of the column ferrule is exposed to sample vapor.
- Insulate the part of the inlet that projects into the oven. If the oven is programmed to increase temperature, the lower part of the inlet could become a cold spot.

Special Topics **Suggestions for packed inlets with 530**-µm columns

Glossary

B

Bleed:

See septum bleed.

Bottle:

The 4 mL glass bottles placed in the injector turret to hold solvent or waste. See also **vial**.

С

Capillary column:

Low resolution capillary columns have internal diameters of 0.4 mm to 0.75 mm. High resolution capillary columns have internal diameters of 0.1 mm to 0.4 mm.

Configuration:

The specific arrangement of automatic liquid sampler modules, gas chromatograph, and data handling devices that operate as a system.

Cool on-column:

An injection technique which places all of the sample directly onto the column without vaporization.

\mathbf{F}

Fast injection:

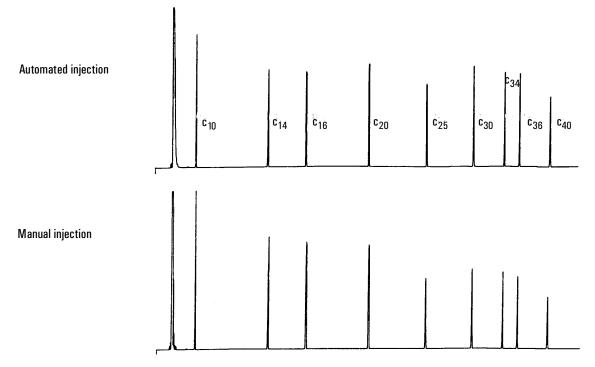
A patented method of introducing a sample to a heated inlet without the negative affects of needle fractionation.

If you are using the automatic liquid sampler for the first time, you may see some changes in the resulting chromatograms. Most of the changes are due to reducing the amount of vaporization from the needle during injection.

- The peak areas of your chromatograms may be smaller. Automatic fast injection delivers the desired setpoint volume of sample.
 Without fast injection, residual amounts of sample boil out of the needle and enter the inlet. This extra amount could measure up to 1 µL.
- The peak areas of your chromatograms may show less differentiation between the low boiling and high boiling components.

Without fast injection, the sample you introduce is richer in low boiling components than in high boiling ones because of fractional distillation in the needle. Not only does residual sample in the needle enter the inlet, but the low boiling components boil off first. This is called needle fractionation or discrimination.

The following chromatograms compare manual injection with automatic fast injection from the automatic liquid sampler for a 1 μL sample of C_{10} to C_{40} in hexane.



Automated vs. manual injection

For more information on the performance of the automatic liquid sampler, order the following technical papers from your Agilent representative:

Publication No. 43-5953-1843: Snyder, W. Dale. Fast Injection with the 7673A Automatic Injector: Chemical Performance, Technical Paper 108, June 1985.

Publication No. 43-5953-1878: Snyder, W. Dale. Performance Advantages of the 7673A Automatic Injector Over Manual Injection, Technical Paper 109, August 1985.

Publication No. 43-5953-1879: Kolloff, R. H. C. Toney, and J. Butler. Automated On-Column Injection with Agilent 7673A Automatic Injector and 19245A On-Column Capillary Inlet -Accuracy and Precision, Technical Paper 110, August 1985.

Fractionation

See Needle fractionation.

Ghost peaks:

Small peaks that do not originate in the sample. They usually indicate that contamination is entering the system.

H

Home positions:

Each of the moving parts has a reference point from which it moves. The parts—for example, the plunger, syringe carriage, and the tray arm—will return to their home positions at various times during operation to insure accurate movements.

Homing:

The process of the turret, syringe carriage, syringe plunger, vial gripper, or tray arm moving to its home position.

I

Injection:

The injector motion that delivers the sample to the inlet or column of the gas chromatograph.

Injection port:

See inlet.

Inlet:

The terms **inlet** and **injection port** are used interchangeably in this manual.

Interaction:

The attraction or repulsion between two chemical species in a specific chemical environment. For example, some sample components interact with a glass inlet liner unless the liner is deactivated.

Ν

Nanoliter injection:

A feature of Agilent injectors that enables an injection volume of 2% of the syringe volume, as well as 10%, 20%, 30%, 40%, and 50% (on selected models).

Needle fractionation:

The boiling off of sample in the syringe needle during injection. Not only does residual sample in the needle enter the inlet, but the low boiling components boil off first. See **fast injection**.

Р

Parameter:

A control value or set point, sometimes optional, used to define an activity.

Peak area discrimination:

Used to describe a chromatogram with peak areas that are not reproducible.

R

Retention time:

The time it takes from the moment of injection until a compound is detected at the peak maximum.

Run:

A single analysis performed under a given set of conditions on one instrument.

Run time:

The time, during the run, that has elapsed from the initiation of data acquisition (or time of injection). For completed runs, this term refers to the total time from injection (or initiation of data acquisition) to the end of the run (or termination of data acquisition).

S

Sample:

A liquid, gas, solid, or heterogeneous portion of material that is representative of the whole.

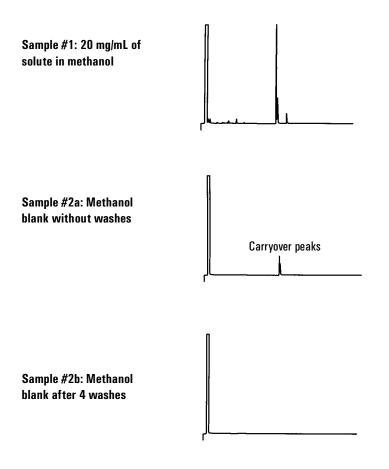
Sample carryover:

Any traces of the previous sample shown in the chromatogram.

After an injection, the syringe retains a volume of sample in the needle and between the plunger and barrel that ranges from 0.6 to 1 μ L. Normal laboratory practice requires washing the syringe with a solvent and/or the next sample to dilute and wash away the remaining sample.

The first two chromatograms below show the effect of carryover when $1 \mu L$ from a vial of methanol is injected after $1 \mu L$ from a vial of a solute is dissolved in methanol. The peaks in the second chromatogram are from the solute left in the syringe from the first injection.

The third chromatogram shows the result of washing the syringe with four solvent washes. The carryover peaks disappear.



Sample carryover

Sequence:

A set of instructions that defines how a piece of equipment, for example the GC, performs more than one automated run. These instructions usually include the automatic liquid sampler parameters, instrument equilibration time, method name, and a sample information table. Sequences can be recursive, that is, one sequence can contain another sequence.

Split injection:

An injection technique in which the GC inlet allows only a portion of the sample to be routed into the column. The rest of the sample is vented. This technique compensates for the low capacity of high resolution capillary columns.

Splitless injection:

An injection technique in which the GC inlet directs all of the sample onto the column after it has vaporized in the injection port.

Standalone:

The controlling method for certain Agilent injectors which uses the injector's own electronics to set the run parameters and operate the automatic liquid sampler.

Т

Tailing:

A chromatographic peak which is skewed, usually due to active sites in the column or the chromatographic system.

V

Variability:

Used to describe chromatogram retention times and peak areas that are not reproducible.

Vial:

The 2 mL or 100 µL bottles used for holding samples. See also **bottle**.

Viscosity:

A flow characteristic of a liquid that may influence the reproducibility of the injection volume.