

## Tech Tips

### How to Find and Fix Common Leaks

Leaks cause valuable sample loss. Nobody wants that. The key of the valve holding pressure is the integrity of the sealing surfaces. If there is a scratch on the sealing surface, or the needle seal in the rotor seal is damaged, a leak may appear. It is also important to realize what appears to be a leak can instead be a result of siphoning. The following are three most common situations in which fluid leaks occur.

1. If fluid leaks out of the needle port only while loading the loop (i.e., while pushing down on the plunger of the syringe), the problem is most likely that the needle seal or the needle port fitting in the loop filler port is not gripping the syringe needle tightly enough to prevent leakage around the syringe needle. Tighten the needle seal grip by pushing down on the needle port (see Fig. 1) or replace the needle port fitting to make a tighter

grip on the needle. The tightening reduces the hole diameter of the needle seal and port fitting.

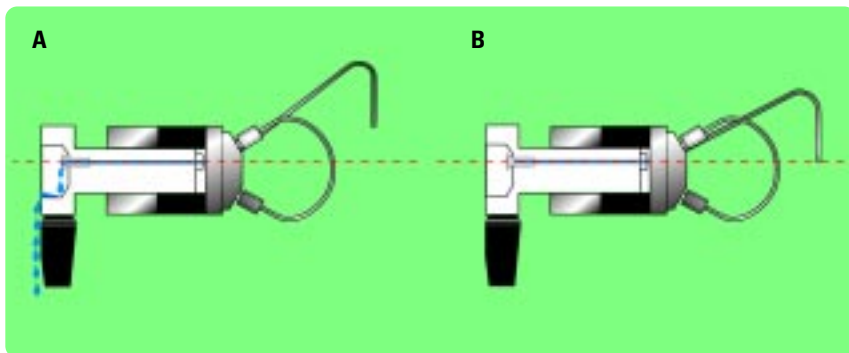
2. If fluid leaks continuously from the needle port or vent lines and/or from the stator or stator ring interface, the rotor seal and/or stator face assembly needs to be replaced. Scratches on the rotor seal or cracks in the stator face assembly allow mobile phase to escape and cause cross port leakage. Genuine Rheodyne replacement rotor seals are listed on page 6.

3. If fluid from the needle port and/or vent lines but eventually stops, the cause is most likely siphoning and not a leak. Siphoning occurs if the vent lines are lower or higher than the needle port. Adjust the vent line(s) so that the outlet is at the same horizontal level as the needle port to prevent siphoning. See Fig. 2.

For other leakage or injection troubleshooting, refer to the Rheodyne *Troubleshooting Guide for HPLC Injection Problems*. You may download the *Guide* from the Agilent website: [www.chem.agilent.com/cag/cabul/lcref.htm](http://www.chem.agilent.com/cag/cabul/lcref.htm)



**Fig. 1**  
To reform the needle seal, push the eraser end of a pencil against the needle port.



**Fig. 2**  
Needle port level compared to the level of vent line outlet: (A) siphoning occurs when the vent line outlet is above the needle port level; (B) siphoning does not occur if the vent line outlet is the same horizontal level as the needle port.

## Burst Pressure of PEEK Tubing

PEEK tubing can be used instead of stainless steel tubing in most applications. PEEK is inert to almost all organic solvents and is biocompatible. Unlike metals, plastics are viscoelastic, and therefore, the yield strengths are not well defined. Many factors affect the burst pressure of PEEK tubing. PEEK tubing will burst at a lower pressure when you:

1. Increase the inside diameter.
2. Increase the temperature.
3. Increase the time of exposure.
4. Increase the concentration of organic solvents.
5. Expose tubing to specific solvents. DMSO, THF, and methylene chloride cause the PEEK tubing to swell. Concentrated nitric acid and sulfuric acid weakens the tubing.

## How to Select the Right Rotor Seal

The standard rotor seal in many Rheodyne manual valves is made from a Vespel blend. This polyimide has low wear and high chemical resistance. Vespel tolerates a pH range of 0 to 10 (see Fig. 3). Solutions more basic than pH 10 dissolve Vespel which damages the rotor seal. If you use any solutions above pH 10, Rheodyne recommends a PEEK blend rotor seal. PEEK offers a high chemical resistance and versatility, and will tolerate the entire pH range from 0 to 14. Tefzel blend rotor seals may be appropriate for some applications. Replacement rotor seals are listed on page 6.

Genuine Rheodyne rotor seals are matchless in performance and product life. For a quarter of a century they have exceeded the needs and expectations of chromatographers. The rotor seals are products of rigid manufacturing and quality assurance procedures before they are incorporated into our valves or

shipped to our customers. Only genuine Rheodyne parts ensure the continued precision performance of Rheodyne valves.

Rheodyne's engineers develop exacting product specifications and designs including the factory-installed rotor seal ring which optimizes rotor seal efficiency. Rheodyne rotor seals must pass tougher-than-real-world standards of performance. Rheodyne rotor seals are made from proprietary-blended polymers, formulated specifically for resistance to repetitive chemical and physical stresses of the entire 0 to 14 pH range.

Tested under actual laboratory conditions, Rheodyne rotor seals fully meet the demanding requirements of day-to-day manual instrument use as well as the operating conditions found in today's automated laboratories.

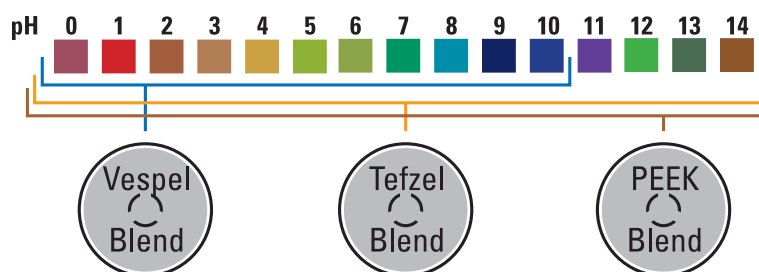


Fig. 3  
pH range of various rotor seal materials. pH colors are for illustration only.

## Tech Tips

### Sample Loop Loading: Partial-filling vs. Complete-filling

#### Partial-filling

Use the partial-filling method if you need to conserve sample, or if you want to vary sample volume frequently.

In partial-filling, the syringe sets the volume injected onto the column. There is no sample waste, and the volume injected onto the column is equal to that dispensed from the syringe. Reproducibility is 1.0% relative standard deviation (RSD). The volume of the sample loaded is limited to half the sample loop volume. For example, the most you can load into a 200  $\mu\text{l}$  sample loop is 100  $\mu\text{l}$ . See Fig. 4.

This limitation is due to the manner in which fluids move in tubes. Fluidic movement in tubes affects reproducibility.

#### Complete-filling

Use the complete-filling method if you have a sufficient amount of sample with which to work, you do not vary sample volume, or you need high reproducibility.

In complete-filling, the loop sets the volume loaded onto the column. You use excess sample (two to five in loop volumes) to replace all the mobile phase in the loop. See Fig. 5.

Change the loop to vary the sample volume. Reproducibility is typically 0.1% RSD for loop sizes  $\geq 5 \mu\text{l}$ . Accuracy is limited as loop volumes are nominal.

#### Q: "Which method should I use and which Rheodyne sample injectors use this method?"

A: There are two types of injectors available: dual mode and single mode. Dual mode injectors allow both partial- and complete-filling whereas single mode injectors allow only complete-filling. See Sample Injectors on pages 4–5.

If you are collecting experimental data, sample is scarce, and/or you want to use different sample volumes, a dual mode injector with a large volume sample loop is appropriate. Only dual mode injectors allow the partial-filling method with which you can easily vary your volumes (up to half your sample loop volume) by setting the syringe volume. Once you begin routine analysis, and/or you have an abundance of sample, either a dual mode or single mode injector is appropriate. Both types of injectors allow complete-filling method with which you fill the sample loop in excess. Complete-filling maximizes the reproducibility of your results.

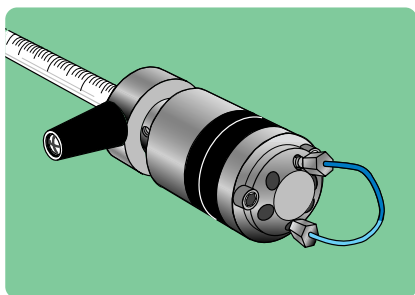


Fig. 4  
The sample loop can fill up to half the loaded volume in partial-filling method.

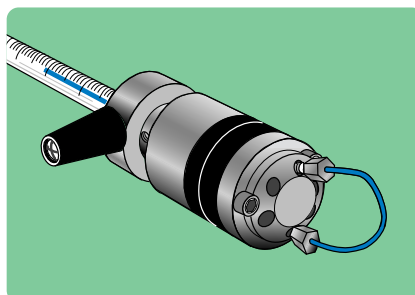


Fig. 5  
The sample loop is filled in excess in complete-filling method

## How to Properly Install Sample Loops



Fig. 6  
Cut-away view of stainless steel sample loop installation.

### Stainless Steel

Stainless steel sample loops are supplied with fittings that are unswaged onto the tube. It is important that the loop be completely bottomed in the injector port before the ferrule is swaged onto the tube. The depth of the tubing holes may vary slightly from port to port and from valve to valve. A fitting made up in one port may leave a small cavity in another port. The cavity causes high dispersion and peak distortion such as fronting, tailing, or broadening. It is good practice to label loop ends so they will be replaced in the same, respective ports that were used in swaging the ferrules. Hint: swaging ferrules separately on each side, into each respective valve port makes loop installation easier.

### To install the sample loop:

a) Take one end of the loop and place the nut (1) and ferrule (2) onto the tubing (3) with the threaded portion of the nut and tapered portion of the ferrule toward the end. See Fig. 6A.

b) Insert the tubing into port (4). Confirm that the tubing is bottomed in the valve port as shown in Fig. 6A.

c) While firmly pressing down on the tubing, hand-tighten the nut as tight as possible.

d) With the slotted socket wrench (see page 11), designed especially for fittings, tighten three 90° turns past finger tight. Remove the loop to confirm that the ferrule is swaged onto the tube.

e) Repeat steps a–d with the other end of the loop while the swaged end remains outside the valve port. See Fig. 6B.

f) Reinstall each end of the loop to their respective ports. See Fig. 6C.

### RheFlex PEEK Fittings and PEEK Tubing

PEEK loop installation requires steps a–c in the stainless steel section above. Finger tightening of PEEK fittings is adequate to make a leak-free connection. The slotted backside

of the ferrule (1) is squeezed down onto the tube (2) by the mating conical surface in the nut (3). See Fig. 7. The nut and ferrule can both be reused many times. Unlike ordinary fittings, the unique RheFlex PEEK design, specifically the angles and surface contact between the ferrule and nut, prevents the nut from gripping the ferrule and twists both the ferrule and the tube during tightening. Otherwise, such twisting stresses the PEEK tubing and lowers the pressure rating of the tubing.

The ferrule can slide and reposition itself along the tube when the fitting is reinserted into a port. It is important that the PEEK tubing is completely bottomed in the injector port before the fittings are tightened to avoid leaving an undesired cavity.

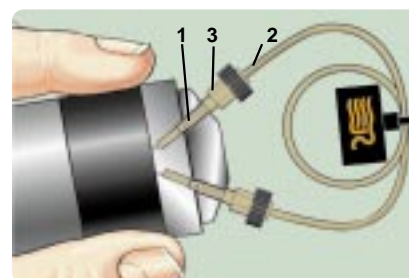


Fig. 7  
Cut-away view of PEEK sample loop installation