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Operating Instructions For Model 7125 Syringe Loading Sample Injector

1.0 INTRODUCTION

The Model 7125 Syringe Loading Sample Injector is a rotary valve designed for high performance liquid chromatography. Supplied with the valve in a separate bag are the following items (see Fig. 5B):

a) Two socket wrenches;
b) One open-end wrench;
c) Two 22cm long tubes (0.51mm I.D.) for vents;
d) Four tube fittings (threaded bushing and ferrule) for tubing connections to valve ports;
e) One 7125 needle port cleaner;
f) Two #8-32 screws for valve mounting.

The 7125 is supplied with a #22 ga needle with a CTFE hub (part #7215) which is shipped inserted in the needle port (underneath the red protective cap). This needle should be removed from the port before use of the valve, but it should be left fully inserted in the port during periods of idleness to keep the needle seal properly sized.

The standard sample loop supplied with the 7125 is the 20-microliter size (part #7022).

Following are the standard sample loops available. Each loop is supplied with fittings for direct connection to the valve.

Catalog number	Sample Loop size microliters	Tubing bore mm	Tubing bore inches
7020	5	.18	.007
7021	10	.30	.012
7022	20	.51	.020
7023	50	.51	.020
7024	100	.51	.020
7025	200	.76	.030
7026	500	.76	.030
7027	1000	.76	.030
7028	2000	1.00	.040
7029	5000	1.00	.040

2.0 WARRANTY

Rheodyne products are warranted against defects in materials and workmanship for a period of one year following date of shipment. Rheodyne will make repairs or replacements free of charge upon return to the factory, transportation paid, of the defective item.

3.0 THEORY OF OPERATION

The Model 7125 is a six-port sample injection valve in which loading of the sample loop is accomplished with a syringe through a needle port built into the valve shaft.

Figure 1 shows the flow diagram of the valve. The six small circles represent the ports in the valve stator (rear of injector). The two heavy arcs represent the connecting passages in the rotor seal. The larger circle represents the needle port (in the rotor seal). The needle port is used to fill the sample loop in the LOAD position. In the INJECT position the loop is switched into the solvent stream and the needle port is vented through valve port 5. Rotation of the knob through 60° switches the valve from LOAD (CCW) to INJECT (CW).

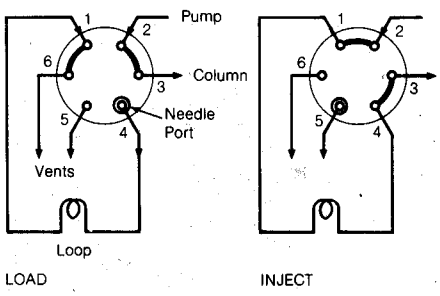

Fig. 1: Model 7125 Flow Diagram

Figure 2 shows the needle port geometry. When the syringe needle is fully inserted, the flat tip of the needle touches the flat face of the stator so that the entire volume of sample discharged from the syringe enters the stator passage which is part of the sample loop. Therefore, all of the sample discharged from the syringe becomes injected onto the column. There is no sample loss.

The flat stator face is polished alumina ceramic — a hard surface which cannot be damaged by the needle tip.

Maximum operating temperature of the Model 7125 is 80°C.

Two methods of loading the sample can be used: The complete loop filling method and the partial filling method. Following is a description of these methods.

3.0.1 FILLING THE LOOP COMPLETELY

This is the conventional method in which an excess of sample is used to insure that the sample loop is completely filled. The volume of sample is determined precisely by the loop volume and the highest degree of precision is obtained. The method is explained in Section 6.0.1.

3.0.2 PARTIALLY FILLING THE LOOP

If only small quantities of sample are available, this is the method of choice.

In this method a microsyringe is used to determine the volume of sample delivered to the loop. The loop has been previously filled with solvent from the last run and the syringe delivery causes the solvent to be displaced by the sample. The displaced solvent exits from vent tube 6.

With this method it is possible to inject samples ranging from less than one microliter up to approximately 50% of the loop capacity. Sample loops can be used on the Model 7125 ranging from 5 microliter capacity up to 5 milliliters. The partial filling method of injection is described in Section 6.0.2.

4.0 GETTING STARTED

To prepare for initial use of the Model 7125, you should follow the installation and start-up instructions in sections 5 and 6. Also be sure to read the **CAUTION** and **WARNING** notes in section 4.1. Sections 7, 8 and 9 give helpful operating suggestions, maintenance and servicing information.

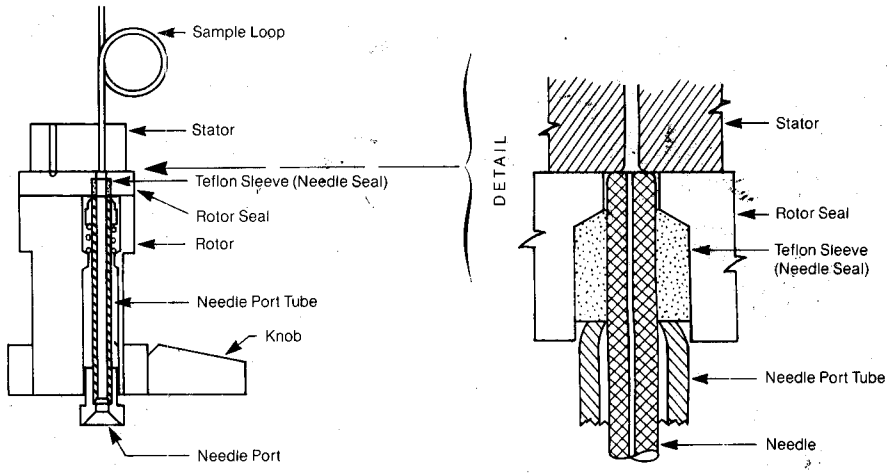

Fig. 2: Model 7125 Needle Port Geometry

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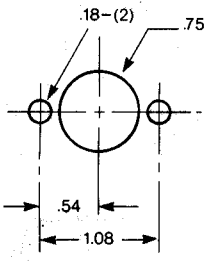

Fig. 3: Panel Holes Required (Inches)

CAUTION: To avoid liquid squirting back at you, discharge syringe slowly.

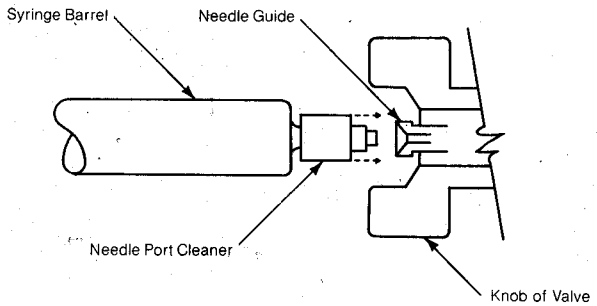

Fig 4: Use of Needle Port Cleaner

4.1 IMPORTANT SAFETY NOTICES

4.1.1 CAUTION: Be sure to use the proper size of syringe needle to avoid damage to the valve. See Section 4.2.

4.1.2 WARNING: When using the Needle Port Cleaner, discharge the syringe slowly to avoid squirting solvent back at yourself. See Section 5.1.

4.1.3 WARNING: When using sample loops larger than the 100 microliter size, protect yourself from the rapid ejection of mobile phase coming out of the needle port when the valve is turned from INJECT to LOAD. See Section 9.9.

4.2 USING PROPER SYRINGES

In both methods of sample loading it is necessary to insert a syringe needle into the needle port. In the complete filling method it is possible to use any syringe with Luer tip together with the needle supplied with the 7125. In the partial filling method, conventional low pressure microsyringes can be used. These are available from several manufacturers and should have the following needle specifications:

Needle dimensions: 0.028″ O.D. x 2″ long, without electro taper.

Point style: 90° (square end).

Failure to use needle of proper size can result in damage to the injector.

Rheodyne also supplies suitable microsyringes. Following are the part numbers:

7201 10ul Syringe
7202 25ul Syringe
7205 50ul Syringe
7210 100ul Syringe
7225 250ul Syringe
7250 500ul Syringe

5.0 INSTALLATION

a) Figure 3 shows the panel holes required for mounting the Model 7125. In addition you may need a hole to run the two vent tubes through the panel. Maximum panel thickness is 0.19″. To mount the valve, first remove the knob by loosening the two knob set screws. Use the two #8-32 screws supplied to fasten the valve to the panel.

If the Rheodyne #7161-020 Valve Position Sensing Switch is to be used, refer to the mounting instructions for that accessory.

b) When replacing the knob be sure that the two set screws are tightened securely on the two flats on the shaft. The third threaded hole in the knob is left blank. The set screws should be tightened only on flats.

c) Connect the two 22cm long tubes (supplied) to valve ports 5 and 6. Use the longer threaded bushing on port 6, and the short one on port 5. Both of these tubes should be bent so that their ends point downward to facilitate collecting the vented liquid.

To prevent siphoning effects, the outlet ends of both tubes should be at the same horizontal level as that of the needle port.

d) Connect pump to port 2 and the column tube to port 3, using the longer threaded bushing for the column tube. Leave the column disconnected from the valve during initial cleaning operations.

The use of a Rheodyne Column Inlet Filter will protect your column inlet frit from plugging from particles in the sample or from pump and valve wear particles.

5.1 USING THE NEEDLE PORT CLEANER

Connect the 7125 Needle Port Cleaner (part #7125-054) to a syringe as shown in figure 4. A syringe of at least 2 ml capacity with Luer tip (not Luer Lock) is recommended — such as Rheodyne part #7252. Use the cleaner to flush out the needle port with mobile phase with the valve in the INJECT position. To do this, push the tip of the cleaner against the conical opening of the Needle Guide and discharge the syringe slowly. Observe **WARNING 4.1.2**. The discharged liquid can squirt back at you if you are not careful.

5.2 INITIAL OPERATION

To clean out the valve and prepare it for connecting to the column, establish solvent flow through the valve. Turn valve to LOAD, then INJECT (to fill the sample loop). Use the 7125 Needle Port Cleaner to flush out the needle port with about 1 ml of mobile phase. This will leave the needle cavity filled with mobile phase. The excess solvent will flow out port 5 and leave vent tube 5 filled with solvent. Repeat the flushing step with the valve in LOAD position. This will fill vent tube 6.

Before connecting the valve to the column, make some practice injections using mobile phase. Follow the instructions in section 6. After practicing, connect the column and proceed with the chromatography.

6.0 MAKING AN INJECTION

6.0.1 FILLING THE LOOP COMPLETELY

This is the conventional method in which the volume of sample injected is precisely determined by the volume of the loop plus valve passages. Use a syringe of suitable capacity together with the #22 ga needle supplied (90° point).

a) Observe **WARNING 4.1.3** and turn valve to LOAD.

b) Load the syringe with sample.

c) Insert the syringe needle into the needle port all the way until the hub almost touches the needle guide. The needle tip will touch the stator face. Do not push hard on the syringe — just be sure it is bottomed.

d) Gently discharge the syringe to completely fill the loop.

e) Leave the syringe in position and turn the valve to INJECT.

f) Remove the syringe.

g) Alternatively, use the syringe to suck up the sample from the vial into which vent tube 6 is dipped.

h) Be sure that the loading passages have been flushed with solvent after the last injection to prevent cross-contamination between runs.

i) In this method, vent tube 5 is not used. Flushing of the needle port and valve passages is done with the valve in LOAD position.

In the complete filling method, an excess of sample must be used because the fluid velocity in the sample loop tubing varies from a maximum at the tube axis to zero at the wall. As sample pushes solvent ahead of it during loading, the locus of the sample-solvent interface becomes diffuse. Solvent remains along the wall. The amount of sample in the loop approaches its maximum value asymptotically. About 2 to 3 loop volumes of sample are required to achieve 95% of maximum. This is the minimum recommended for good precision, but 5 to 10 loop volumes will provide better precision. You should determine this experimentally for yourself.

6.0.2 PARTIALLY FILLING THE LOOP

In this method, the syringe delivery determines the sample volume injected (see Section 3.0.2).

a) With valve in INJECT position, use the 7125 Needle Port Cleaner (part #7125-054) to flush out the needle port with about 1 ml of eluting solvent. This will flush out residual contamination from the previous injection. The flushing liquid will exit out of vent tube 5.

NOTE: See Section 9.3 for possible elimination of this flushing step.

b) Observe **WARNING 4.1.3** and turn valve to LOAD.

*Vespel and Tefion are trademarks of DuPont.

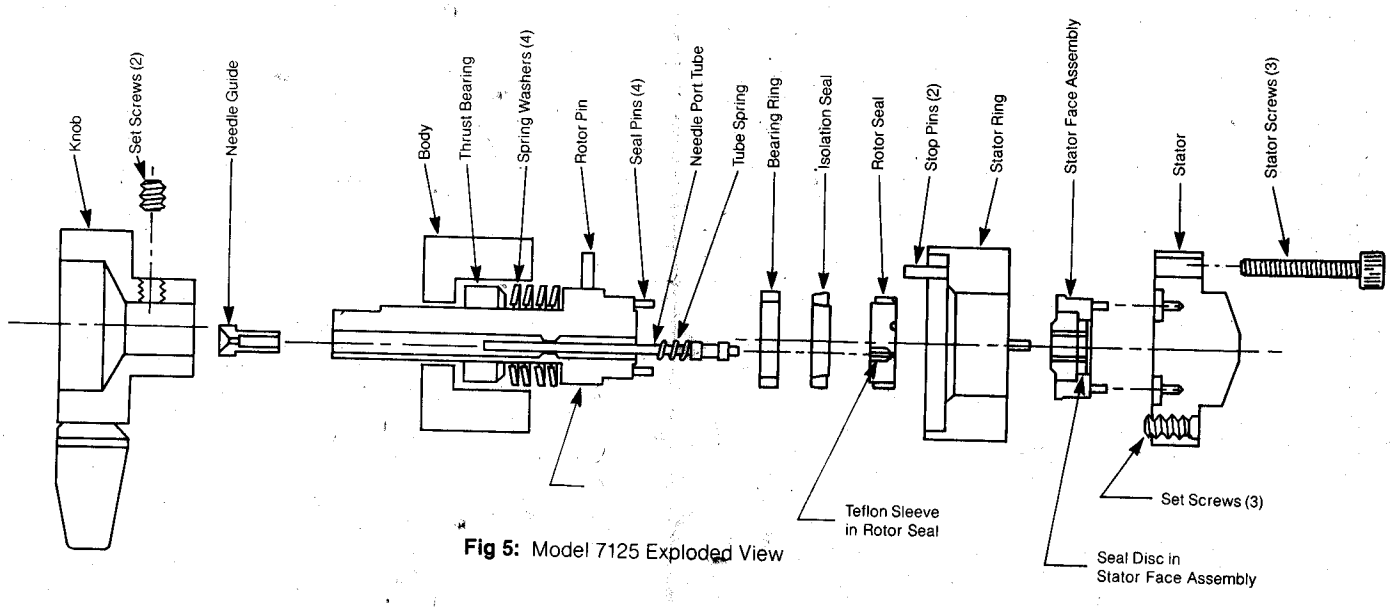

Fig 5: Model 7125 Exploded View

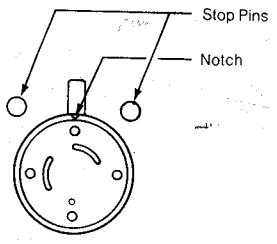

Fig. 5A
Rotor Seal Orientation (Viewed From Stator)

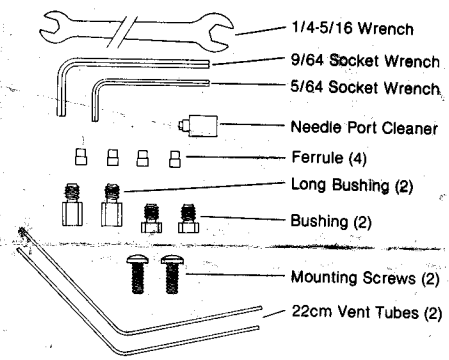

Fig. 5B: Parts supplied in separate bag

c) Load the syringe with the desired sample volume.

d) Insert syringe needle into the needle port all the way until the hub or barrel almost touches the needle guide. The needle tip will touch the stator face. Do not push hard on the syringe — just be sure it is bottomed.

e) Gently discharge the syringe contents.

f) Leave syringe in position and turn valve to INJECT.

g) Remove the syringe and leave the valve in INJECT position.

In the partial filling method, no more than half a loop volume of sample should be passed into the loop in order to maintain volumetric accuracy. With larger volumes some of the sample is lost out vent tube 6. This is because of flow velocity inequalities throughout the loop tubing.

7.0 ADJUSTING FOR HIGHER PRESSURE OPERATION

The three small set screws in the stator (see Figure 3) have been factory-set so that when the three stator screws are fully tightened, the spring force between the valve rotor and stator is sufficient to hold 34 MPa (5000 psi). If leakage is to be corrected, or if operation up to 48 MPa is to be done, proceed as follows: The three set screws should be **loosened** about 1/20 turn each (18° of rotation) and the three stator screws **tightened** an equal amount. If this new setting fails to accomplish leak-free operation, repeat the procedure by an additional 1/20 turn. Avoid excessive tightening which will accomplish nothing but increased wear of the rotor seal. If it is necessary to loosen spring tension, either to lower the operating pressure or to adjust for a new rotor seal which may be thicker than the one being replaced, reverse the above procedures; i.e., first loosen the stator screws. Then tighten the set screws.

8.0 MAINTENANCE

With normal use, the Model 7125 should give thousands of injections without trouble. The main causes of premature failure are:

a) Incorrect needle tip shape can cause the ceramic stator face to chip which then causes deep scratching of the rotor seal surface.

b) Abrasive particles in the sample which cause scratches on the rotor seal surface.

The rotor seal wears with use and is the only part that routinely needs replacement. It can be expected to last between 6 months and 2 years.

8.1 CHANGING ROTOR SEAL

To change the rotor seal proceed as follows:

a) Leave the Model 7125 attached to the panel and leave knob on. If it is necessary to remove the valve from the panel before servicing, then replace the knob after removal from panel (this simplifies the disassembly).

b) Remove the three stator screws (refer to Figure 5). Do not change the setting of the set screws in the stator at this time.

c) Remove stator, stator face, and stator ring from valve body by pulling axially to disengage the various pins.

d) Remove the rotor seal by prying it off of the four seal pins, using a screwdriver or knife blade.

e) The isolation seal and bearing ring usually are left in place because they rarely need changing.

f) Install a new rotor seal (part #7125-047) on the four seal pins by following the reassembly procedure in Section 8.2.

8.2 REASSEMBLY

Reassembly of the valve requires putting all parts back together as shown in Figure 5 while observing the following:

a) Be sure that the rotor seal is correctly oriented as shown in Figure 5A with rotor seal slots facing the stator. The needle port tube will be in line with the Teflon sleeve in the rotor seal when the rotor seal is in the correct position.

b) In replacing the stator ring, be sure that the two stop pins are replaced in their holes in the stator ring, then push the stator ring squarely onto the rotor assembly so that the stop pins enter the mating holes in the body (with the rotor pin between the two stop pins) and so that the isolation seal slips inside the stator ring without hanging up.

c) Make sure that the black seal disc is in place in the stator face assembly and that its holes are aligned with the six holes in the ceramic. This seal disc usually stays tightly in place inside the stator face assembly and should not be removed.

d) In replacing the stator face assembly on the stator, the small notch in the rim of the face assembly should be in line with port 1 of the stator. Make sure that the two pins enter the holes in stator.

e) Mount the stator and stator face assembly on the valve by pushing it onto the two pins on the stator ring and then adding the three stator screws. Be sure that the stator face assembly remains properly in position on the stator. It has a tendency to fall off; so, be careful. Tighten each screw a little at a time to keep the stator surface parallel to the stator ring surface. If the three sets screws in the stator were left unchanged, then tighten the three stator screws until all parts are held firmly in place. The three set screws will insure that the gap between stator and stator ring is uniform and exactly as it was before disassembly.

f) If the set screws need adjusting because a new rotor seal was installed or because leakage has to be stopped, refer to Section 7.0 and be sure that each set screw is turned an equal amount so that after the stator screws are retightened, the gap between the stator and stator ring is uniform all around.

g) Note that there are three threaded holes in the knob but only two set screws are used. The set screws should be tightened only on the two flats milled on the shaft.

h) After installation of a new rotor seal, it is usually necessary to form the needle seal around your syringe needle. See Section 9.2.

8.3 SUGGESTED SPARE PARTS

7125-047 Rotor Seal for 7125/7126
(expected lifetime 6 to 24 months)

Spare fittings for new tubing connections:

7010-009 Bushing
7010-011 Long Bushing
7010-062 Extra Long Bushing
7010-010 Ferrule (one required for each make-up)

9.0 OPERATING SUGGESTIONS AND TROUBLE-SHOOTING

9.1 LEAKAGE

If liquid is observed dripping out between stator and stator ring, the stator screws should be tightened as explained in Section 7.0. Leakage out the needle port or vent tube (other than that caused by loading the loop) is caused by scratches on the rotor seal. Try tightening stator per Section 7.0, or, if this fails to stop the leakage, replace the rotor seal (part #7125-047).

NOTE: If the vent tubes from ports 5 and 6 do not have their outlet ends at the same horizontal level as the needle port, siphoning can occur which is often misinterpreted as "leakage."

9.2 NEEDLE SEAL LEAKAGE

Because the outside diameter of the syringe needle varies from syringe to syringe, the needle seal (Teflon sleeve in the rotor seal) may not immediately seal properly around a needle which is smaller than average. This will result in a loss of accuracy in loading the sample. The spring loaded needle tube will eventually reform the Teflon sleeve to make a good seal, but if you do not want to wait, do the following:

With needle removed from the needle port, push on the plastic needle guide. This will assist the spring in deforming the Teflon sleeve. Do not push so hard that the Teflon sleeve is squashed too much. A few gentle tries will produce the desired result.

To check for a proper liquid seal around the syringe needle, fill the syringe with water and slowly discharge the syringe with the injector in the LOAD position. Notice the lack of resistance to syringe discharge. Now repeat the action with the injector handle half-way between LOAD and INJECT. (The pump must be off in this position.) Now it should feel noticeably harder to discharge the syringe.

The needle seal is designed to seal around the needle only to a few psi of pressure. Since a microliter syringe can produce much higher pressure with just a small force on the plunger, do not expect the needle seal to completely prevent syringe discharge with the handle in the half-way positon.

9.3 FLUSHING BETWEEN INJECTIONS

Measurements have indicated that under proper operating conditions the residual sample left in the needle cavity and on the needle seal surface after an injection varies between 0.001 and 0.01 microliter. This represents 0.01% to 0.1% of a 10 microliter injection. If this amount of cross-contamination between successive injections is acceptable, then you do not need to flush the needle port between injections. Eliminate step (a) in Section 6.0.2.

However, it is wise to check the magnitude of cross-contamination periodically and to use the flushing step when in doubt. Conditions that can produce excessive cross-contamination are:

a) Needle is too short so that the needle tip does not touch the stator face. Minimum length from hub to needle tip should be 5.00 cm (1.97 inch).

b) Syringe is not held in place with needle bottomed in needle port while turning from LOAD to INJECT.

c) Dirt particles or needle seal shavings in the needle port are preventing the needle tip from touching the stator face.

Even when cross-contamination is not a concern, it is good practice to flush the valve (Section 5.1) about every ten injections. This prevents buildup of contamination and also keeps the needle port and vent tube 5 filled with solvent, preventing air from inadvertently entering the sample loop.

9.4 USE OF AQUEOUS BUFFERS OR SALT SOLUTIONS

To prevent the formation of salt crystals in the valve, flush out the flow passages and the needle port with water after usage of salt solutions.

9.5 USE OF HIGH pH SOLUTIONS

The standard rotor seal is Vespel, a DuPont polyimide which has exceptionally good wear resistance. However, it is susceptible to alkaline attack, deteriorating rapidly when used with solutions of pH over 10. An alternative material is available for alkaline applications (consult factory).

9.6 PLUGGED VALVE PASSAGES

If valve passages get plugged, they can be opened by removing the stator as described in Section 8.1 and cleaning the passages with a small wire (.015-inch maximum diameter).

9.7 KEEPING NEEDLE SEAL IN PROPER SHAPE

During periods of idleness of the chromatograph, such as overnight or during weekends, it is advisable to leave the 22 ga needle (supplied) fully inserted in the needle port. This will keep the needle seal (Teflon sleeve in the rotor seal) in proper shape and prevent it from being squeezed down too small.

9.8 CALIBRATING SAMPLE LOOPS

Sample loop sizes are designated by nominal values, which can vary from the actual value by as much as 20%. This is due to the ±.001" tolerance on the tubing I.D. (see table below). Since both standards and unknowns are usually analyzed with the same loop, knowledge of the absolute loop volume is rarely needed. If the actual loop volume must be known, it is best to calibrate it in place on the valve, so that the flow passages in the valve (one in the rotor and two in the stator) are also taken into account.

Tubing bore	Volume tolerance resulting from .001" bore tolerance
.012"	±17%
.020"	±10%
.030"	± 7%

9.9 USE OF LARGE SAMPLE LOOPS

When large sample loops are used, a few microliters of mobile phase will be expelled from the needle port and vent tube 6 when the valve is returned to LOAD. This happens because the compressed fluid in the sample loop expands when it is exposed to atmospheric pressure. Since the compressibility of most solvents is about 10^{-4} per atmosphere, the solvent in a 1-ml sample loop will expand about 20ul upon decompressing from 21 MPa (3000 psi). Observe **WARNING 4.1.3**. A small test tube or absorbent tissue can be placed at the needle port when returning the valve to LOAD, in order to safely catch the expelled liquid.

9.10 CHANGING SAMPLE LOOPS AND COLUMN CONNECTIONS

The depth of the tubing holes in the valve ports may vary slightly from port to port and from valve to valve. A fitting made up in one port may leave a dead space in another port. It is good practice, therefore, to label sample loops so that if they are removed, they will be replaced in the same orientation in the same valve. Also be sure that the column connecting tube at port 3 is made up with no dead space.

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