The 8610 gas chromatograph is shipped equipped with a direct injection-type injection port. This port permits on-column manual injections with traditional chromatography syringes. The injection port is simple and highly efficient by design. Swagelok stainless steel hardware is used in the assembly of the injection port. Injection of gas and liquid samples is performed using standard syringes equipped with a 26 ga. needle. Beveled (medical-style), straight, and hooked tips are available from many suppliers in this needle size. For larger needles, such as a side-port, blunt-tipped needle, a 1/8" Swagelok stainless steel nut is used in place of the supplied septa nut. Although several needle tips are available, hooked-tip needles promote septa life by slicing through the septa without "coring" the silicone, as do medical and straight-tipped needles.

Therefore, they are recommended. "Coring" punches shards of septum into the injection port and may result in plugging of the syringe needle and failure to deliver sample. Over an extended period of time, these shards could migrate into the column. In a packed column, this accumulation of septum shards increases the exposed surface of silicone available to produce silicone or "septa" bleed. In a capillary column, these shards could plug the column completely. Routine maintenance of the septum prevents this from occurring. A bad septum may bleed excessively or permit carrier gas to leak out of the system, affecting retention times. It may visibly bulge or show numerous slices or shards of silicone protruding in toward the injection port. This usually occurs when the septum nut has been over-tightened and the physical characteristics of the septum have been altered due to compression of the silicone. If a septum is extremely bad, the user might see a puff of smoke blow out from the injection port after injection. This is the volatized sample blowing back out through the leak on a continuous stream of carrier gas. Septa may become tacky and unusable after extended service. The septa nut should be finger-tight. Once the user feels the septum seat snugly against the bulkhead fitting, the septa nut has been tightened sufficiently. Use the two black rubber O-rings on the injection port as a guide - the nut should barely make contact with the outer O-ring when the nut is properly tightened. NEVER use a wrench to tighten the septa nut. An over-tightened septum will have a markedly decreased lifetime. Larger side-delivery needles also tend to reduce septa life due to the size of the puncture created during injection. This requires more frequent servicing of the septum. Please note that when septum replacement is required during use of the thermal conductivity detector (TCD), the filament current should be turned off at the electrometer located on the right side control panel of the chromatograph, prior to removing the septa nut. Failure to do so could result in the destruction of the detector filaments due to lack of carrier gas flow through the column and into the detector.
When performing analyses using manual direct injection, the method or technique used to prepare the syringe and perform the injection can mean the difference between obtaining chromatograms that are either poorly resolved or clean and sharp. Reproducibility can also be affected if the amount injected varies from injection to injection. This is why it is imperative that a consistent, reproducible method or technique of manual injection be used when performing direct injection.

Sample volume affects the quality of data produced by the gas chromatograph. If too much sample is injected, the column becomes overloaded and the peaks produced will be broad and tailing. Insufficient sample will likely result in quantitation inaccuracies. If the syringe is not properly primed and loaded (or the sample slug contains air bubbles) when injecting liquid samples, or the syringe has not been properly evacuated, purged and loaded when injecting gas samples, the sample amount actually injected will vary, as will the results obtained. The procedure indicated below is just one of many in use today by chromatographers performing direct injection of liquid samples. The syringe and plunger are cleaned. The plunger should not be bent. Then the syringe is flushed thoroughly, primed, and loaded with precision.

1. Rinse the syringe thoroughly in solvent, working the plunger. Then draw sample in, remove needle from sample vial, and eject sample outside of vial wipe needle, and repeat several times to prime syringe.

2. Draw one microliter of air into the syringe.

3. Then draw one microliter of sample into the syringe after the slug of air.

4. Follow the microliter of sample with one microliter of air, sandwiching the sample between the slugs of air.

5. Once the syringe is loaded, insert the needle into the injection port quickly. Press the plunger quickly and remove the needle immediately. Injection should take less than 2 seconds.

Properly prepared, the syringe needle is inserted completely into the injection port in one smooth, quick motion. Then the plunger is driven home immediately. As soon as the plunger tip hits the end of the sample chamber, the syringe needle is withdrawn from the injection port in a quick, smooth motion. This will prevent any sample remaining in the needle from having time to vaporize into the injection port before or during withdrawal (if this were allowed to occur, it would result in peak broadening and tailing). You may currently be using a different technique for direct injection. As long as the method being used is consistent and reproducible, you will obtain reliable, consistent reproducibility from your direct injection analyses of gases and/or liquids.
In order to place a sample into the column of a gas chromatograph without de-pressurizing the injection port and column or interrupting the carrier gas flow, some type of penetrable, resealable membrane must be used. The membrane must be penetrable to permit the introduction of the syringe needle into the injection port, but must also have the ability to re-seal itself. If it could not re-seal itself, each injection would leave a leak that would permit carrier gas to escape from the system. Each subsequent injection would worsen the condition, adversely affecting retention times and sensitivity. Silicone rubber is commonly used to produce injection port septa. Silicone, due to its formulation, is soft yet maintains the ability to seal puncture wounds created by syringe needles. Although septa differ in formulation, proper care will prolong the life of any septum. A silicone septa (CRS 800-327-3800, part number 237740) is installed in all SRI injection ports when shipped. This septum is very soft and resealable. It demonstrates low silicone bleed and does not affect sample component elution times. Additionally, this septum exhibits negligible "coring" for better durability and performance. This septum seals well in the tapered interior of the 1/8" modified Swagelok injection port. The example at right illustrates the difference in physical appearance between this septum and the standard cut septa machine-stamped from silicone sheets. Coated septa are manufactured this way. The coating is intended to reduce septum bleed and increase resealability.

All septa are made of silicone rubber that contains polymerized silicone gums. A catalyst is used to obtain the polymerization that produces the elasticity sought from the septum material. Unfortunately, some silicone oils remain un polymerized in the septum and freely diffuse out of the silicone septum. These oils vaporize into the injection port and are known as septa bleed. Some manufacturers insert additional oils, making the septa softer and easier to remove from their molds. This increases the amount of septa bleed in those pieces. Most low bleed septa are manufactured by extending the duration of polymerization, resulting in a harder silicone with less bleed. The septa used by SRI exhibit extremely low bleed while remaining soft and highly resealable.

When silicone oils bleed into the column over time, the efficiency and performance of the column is degraded. Columns with a silicone liquid phase, such as OV-1 or SE-30 types, will not display the effects of septa bleed as readily as would a phase such as Carbowax 20M, which would be adversely affected by the effects of silicone bleed. In other columns, the condition may go unnoticed initially, especially during isothermal operation until the development of a high unsteady baseline occurs, accompanied in some instances by increased noise. When the column temperature is ramped as occurs during temperature-programmed operations, the silicone oils begin to elute as they are heated to their respective boiling points. These silicone components will elute through several runs, producing spurious peaks with often reproducible retention times. This can influence component identification and measurement negatively.

In some work, where sensitivity is not great, septa bleed is not a concern. To identify septa bleed, especially where temperature programming is employed, cool the unit to ambient temperature and hold for ten to fifteen minutes. Then ramp the temperature up to the maximum running temperature normally used, with the sensitivity set to high. Any peaks or baseline drift can be attributed to septa bleed. One method to minimize bleed is that of baking septa in an oven prior to insertion into the injection port in order to volatize the silicone oils. The septa may also be baked in the injection port overnight, as long as the column oven is maintained at the same temperature as the injection port to avoid the accumulation of bleed products. Regardless of septum type, septa should never be handled except with tools. Finger oils may appear on chromatograms as additional peaks.
All SRI Instruments heated injection ports are equipped with a specially-designed septum nut which dissipates any heat that could be transferred from the heated injection port body (including split-splitless configurations), to the septum nut and septum by contact. Experience indicates that when injection ports are permitted to transfer heat to the silicone septum, that septum bleed can and does occur. Septum bleed is the volatilization under heat of silicone oils used in the manufacture and formulation of today's high-performance, resealing silicone septa.

Silicone bleed produces numerous interference peaks in chromatograms when the septum exceeds 180°C and oil vapors are swept into the column and quantitated. This situation is undesirable and easily avoided when the special septum nut is used.

The special septum nut assembly illustrated above keeps the septum at an adequate distance from the heated injector assembly to maintain the septum at or near ambient temperature with the assistance of the additional mass of the septum nut extension. This prevents any silicone oils present in the septum from volatizing and being carried into the column. This feature may be retrofitted onto earlier versions of the SRI GC heated injection port, as only two new parts are required. The following page contains chromatographs that illustrate the effect of this new injector design.