Maintenance and Troubleshooting

This section lists procedures to keep your inlet functioning properly, including:

- Cleaning and care
- Pressure and temperature control problems
- Safety shutdown and alarm relay
- Septum problems
- Syringe problems
- Sampler vial cap septum problems
- FID flameout problems
- Proper configuration
- Peak broadening and split peaks
- Useful tools

Cleaning and care

Most laboratories have airborne lint and dust that accumulates on the needle guide and can be carried into the inlet or column on the syringe needle. Particulate matter in the inlet interferes with easy passage of the syringe needle. If dirt enters the column, it can alter the chromatography.

Clean the stainless steel needle guide, spring, and insert by sonication for 1 minute in aqueous detergent, then in distilled water. Rinse with methanol, and air dry. Check the insert for cleanliness with a magnifier.

Clean the fused silica needle guide by forcing methanol through the needle with a squeeze bottle.

Troubleshooting automatic and manual injections

If you have checked these possible causes and still have a problem, call your nearest Hewlett-Packard Service office.

Symptom	Possible Cause	Corrective or Preventive Action
Not enough pressure (safety shutdown activated).	Septum leaks or is missing.	Check system for leaks.
	Column is broken.	
	Column ferrule seal leaks.	
	Gas supply is off.	
	Supply pressure is inadequate.	
	Desired pressure may not be achievable with the column in use.	
Pressure goes to 0 or maximum.	Configuration is wrong.	Check your configuration in “Proper Configuration”.
Not Ready light flickers (oscillating pressure).	Septum or column connection leaks.	
	Pressure set higher than the operating limit.	
Not Ready light flickers (oscillating temperature).	Configuration is wrong.	See “Proper Configuration”.
	Inlet temperature equilibration time is too short.	Increase equilibration time.
Pressure and temperature are not controllable.	Configuration is wrong.	See “Proper Configuration”.
Inlet cools down very slowly.	Fan is either not running or blowing away from inlet.	Check that the fan is running.

Symptom	Possible Cause	Corrective or Preventive Action
Bent needle	Incorrectly installed needle support assembly	Check needle support assembly installation.
	Defective needle	Check each syringe before installation to make sure needle is straight.
	Incorrect insert	Make sure the insert is the correct size for the column and needle you use. Also check that the insert is installed correctly.
	Overcrimped vial caps	See *Capping sample vials* in the *HP 7673 Automatic Sampler Manual* for instructions on crimping vial caps.
	Worn or damaged rubber needle guide	Check the needle guide on the needle support foot every time you change the inlet septum, and replace if necessary.
	Incorrect inlet septum	Use only a 5-mm septum with a through-hole.
	Poor alignment of inlet septum and septum nut	Align the inlet septum and septum nut according to the instructions provided in this manual.
	Incorrect column internal diameter	Check the internal diameter of the column by using the appropriate insert.
	Closed inlet septum hole	Replace the septum.
	Poor alignment of the inlet and the automatic injector	See the *HP 5890 Series II Operating Manual* for alignment instructions.

Symptom	Possible Cause	Corrective and Preventive Action
No peaks or unexpectedly small peaks	Plugged syringe needle	Replace the needle, or clean it with wire.
	Worn syringe barrel	Replace the syringe often, or use a gas-tight syringe. (Under high pressure, the sample is pushed upward through the gap between the plunger and the glass barrel, and a worn plunger can lead to sample loss.)
	Loose removable needle	Make sure syringe barrel caps are screwed on tightly and that the Teflon disk is wrapped tightly.
	Incorrectly placed or missing Teflon disk on the syringe needle	Check every syringe needle to make sure the Teflon disk is present and correctly placed.
Poor precision; poor repeatability; large standard deviation	Worn syringe barrel	Replace the syringe often. (Under high pressure, the sample is pushed upward through the gap between the plunger and the glass barrel, and a worn plunger can lead to sample loss.)
	Loose removable needle	Make sure syringe barrel caps are screwed on tightly and that the Teflon disk is in place.
	Widened holes in vial caps	Replace vial caps when holes widen and leaks develop. (A sample with a low boiling point can escape through a hole that has become too large.)
	Inlet pressure set too low.	Adjust the pressure.
	Incorrectly placed or missing Teflon disk on the syringe needle	Check every syringe needle to make sure the Teflon disk is present and correctly placed.

Safety shutdown

Programmable cool on-column inlets that are equipped with electronic pressure control have a safety shutdown feature to prevent gas leaks from creating a safety hazard. If the system cannot reach a pressure setpoint, the system beeps. After about two minutes, the beeping stops and the following message appears on the display:

Also, a relay signal is set that can trigger an alarm.

A safety shutdown can occur under the following conditions:

1. There is a leak in the system (see *Pressure and temperature control problems*). This includes missing septa or columns!
2. The column is not restrictive enough to reach desired pressure (i.e., 530 µ columns will not go to 100 psi with available flow).

   Note: This may occur during a programmed pressure ramp that is too high a pressure.
3. There is insufficient supply pressure.
4. Configuration is set wrong. Check the mode switch on the inlet controller board (see *Proper configuration*).

To recover from a safety shutdown, turn the GC power off, then on. Then reset temperature and pressure zones to desired values. (After safety shutdown, pressure setpoint is automatically reset to zero.)

Septum problems

The od and thickness of the disk septa are critical for long life and leak-free operation. If the od is too small and the septum is too thick, the septum will fail prematurely. A thick septum can bend needles and make needle insertion difficult.

If the septum drops easily into the retaining well and falls out when the needle guide is turned upright, the od is too small. The od should be 5 mm, and the septum should fit snugly in the retaining well.

When the needle guide is screwed onto the inlet, resistance will be felt when contact is made with the septum. This should occur when the guide is 1/8-or 1/4-turn from bottoming on the inlet body. If contact occurs sooner, the septum is too thick. If no resistance is felt, the septum may be too thin.

Syringe problems

For manual injection with fused silica needles, make sure the needle is clean and free of dust. Wipe with solvent wet tissue, and wipe dry just before injection. Use the same solvent used to dissolve the sample. If the needle should break, replace it. Do not use a needle less than 10 cm long.

For automatic injection, the same cleanliness precautions apply as mentioned earlier. All the areas in the automatic sampler that introduce samples (wash/waste bottles, needle guide) collect dust and should be cleaned periodically.

For longer septum life, you can polish the syringe needle with fine abrasive to remove any concentric ridges left from the needle drawing process. These are visible when magnified, and, if present, can scrape septum particles into the inlet.

Automatic sampler vial cap septum problems

Contamination can occur from vial cap septa, particularly if more than three injections are made from one vial. Repeated punctures may dislodge septum pieces into the sample. To determine if this is a contamination source, cut a 1- x 2-mm segment from a septum and slurry in a vial with 1 ml of the same solvent used for the sample. Analyze this mixture under the same conditions used for sample analysis. If peaks appear that might interfere with your analysis, try other types of septa until you find one compatible with your analysis. A similar test can be run with the injection port septum if you suspect it to be a source of contamination.

Contamination can also occur when small particles are picked up by the sampler syringe and delivered on column. The symptom is sudden onset of a tailing solvent and early eluting component peaks.

The only solution is to remove enough column to eliminate the particles. The easiest way to find them is to backlight the column with a flashlight. Cover the lens with a tissue to diffuse the light. Move the light along the column until the offending particles are found and cut off enough column to eliminate them. Use a magnifier if necessary; a single, very small particle will cause significant tailing.

FID flameout problems

When using pressure programming with large id columns (i.e., 530 µ columns) it is possible to blow out the FID flame if pressure (flow) becomes too high. If this occurs, either lower the pressure ramp or switch to a more restrictive column (longer and/or smaller id).

Peak broadening and split peaks

As a sample is injected on the column at a temperature below the boiling point of the solvent, the carrier gas pushes the liquid farther into the column, creating a flooded zone. The solute components are spread over the length of the flooded zone. This is called *band broadening in space.*

1. Sample on column at moment of injection

2. Carrier gas pushes liquid solvent to create a flooded zone. The solutes distribute themselves evenly across this zone, causing band broadening. The flooded zone is made worse when the solvent is less soluble in the stationary phase.

The length of the flooded zone depends on the solubility of the solvent in the stationary phase. If the solvent is less soluble, the flooded zone will be longer, and peak broadening will be greater.

Further distortion comes from nonuniform distribution of low volatility compounds across the solvent band. This gets worse as sample volume increases, and column length or diameter decreases.

Possible solutions

1. **Check Solubilities.** Make sure the polarity of the solvent is compatible with with polarity of the stationary phase you are using. The length of the flooded zone is affected by the solubility of the solvent in the stationary phase. If the solvent is less soluble, the flooded zone will be longer, and peak broadening will be greater.

2. **Use a Retention Gap (RG).** A retention gap is a deactivated, uncoated (or thinly coated) precolumn in series with the analytical column.

   In general, the length of the retention gap required and type of deactivation depend on injected volume and solvent polarity. A working rule of thumb is to use 0.3-m or 1-m of retention gap per µl injected. For a 3 µl sample, use a 3-m RG. The RG should be wetted by the solvent, which means it should be deactivated with material of like polarity. Fused silica tubing is commercially available in a range of diameters and deactivations.

   Automatic injection requires a 0.53-mm id RG, but the analytical column can be of smaller id.

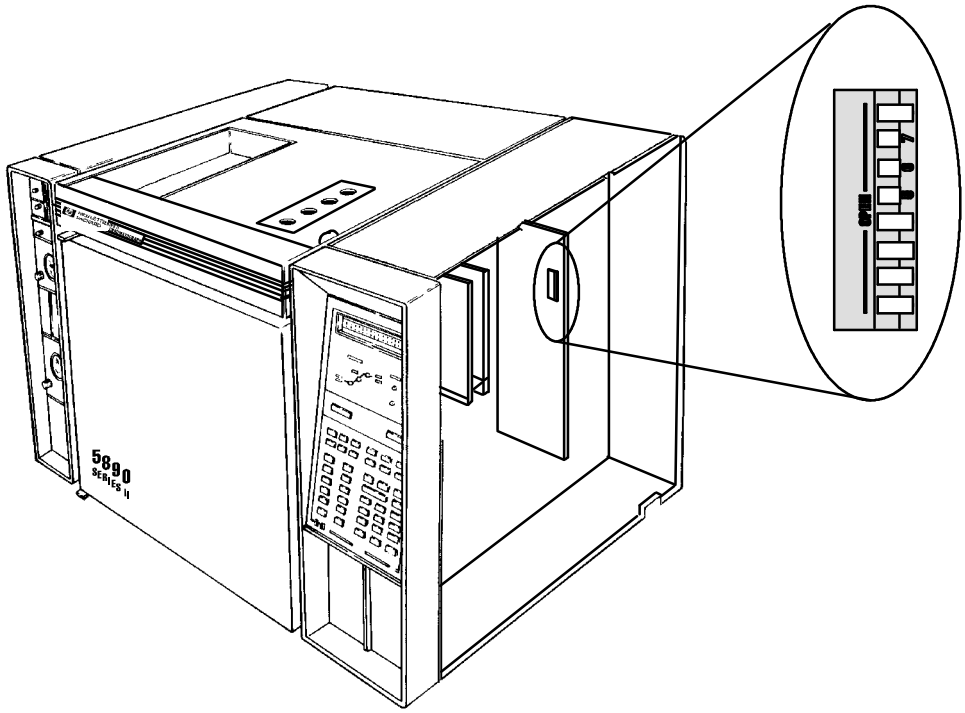
3. **Optimize the Temperature Program and Inlet.** Check the boiling points of the solvent and solutes.

4. **Use the Right Injection Volume.** Know the capacity of the column. Injection volume is important; large volumes may exhibit band broadening, particularly for components whose boiling points are more than 150°C above that of the solvent.

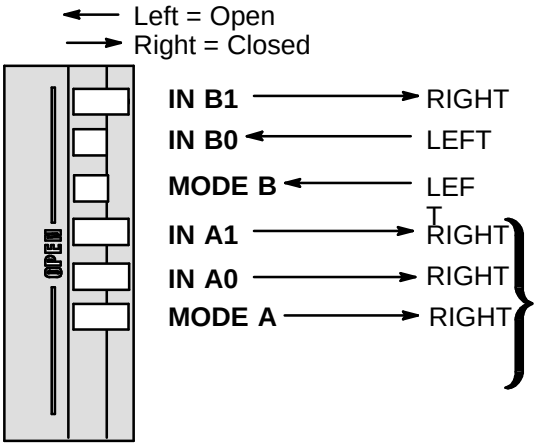
Proper configuration

If the inlet is not working at all, there may be a configuration problem.

1. Turn off the GC power, and remove the side panel of the GC.
2. Check if the switches on the inlet controller board are set for your configuration.
3. Turn on the GC.



Switch setting examples

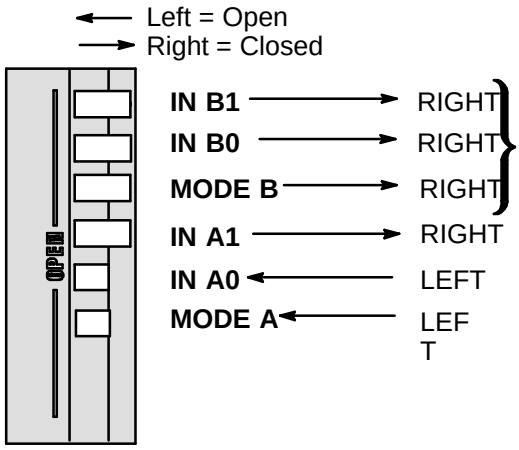


NOTE

This group of switches controls the A position inlet. If another *electronic pressure controlled* inlet is installed in the A position, this group of switches must be set according to the instructions for that particular inlet.

INLET B = Cool On-Column with Electronic Pressure Control

INLET A = Any Non-Electronic Pressure Controlled Inlet

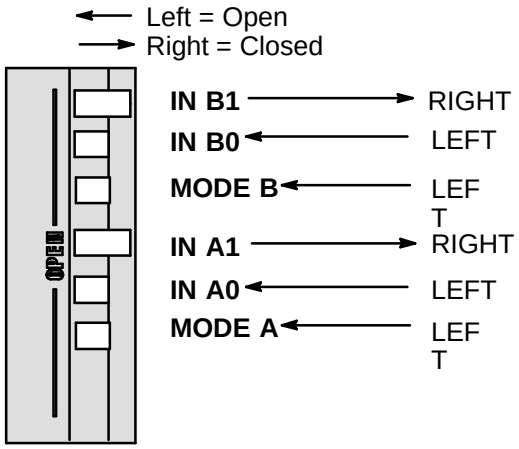


NOTE

This group of switches controls the B position inlet. If another *electronic pressure controlled* inlet is installed in the A position, this group of switches must be set according to the instructions for that particular inlet.

INLET B = Any Non-Electronic Pressure Controlled Inlet

INLET A = Cool On-Column with Electronic Pressure Control



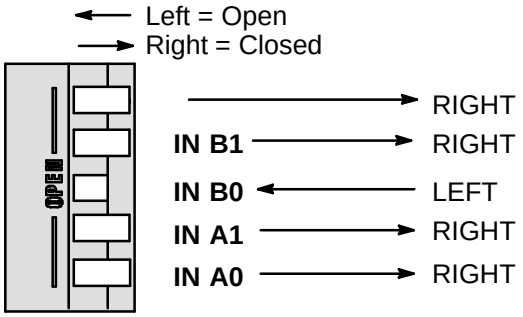
INLET B = Cool On-Column with Electronic Pressure Control

INLET A = Cool On-Column with Electronic Pressure Control

Setting manual pressure control board configuration

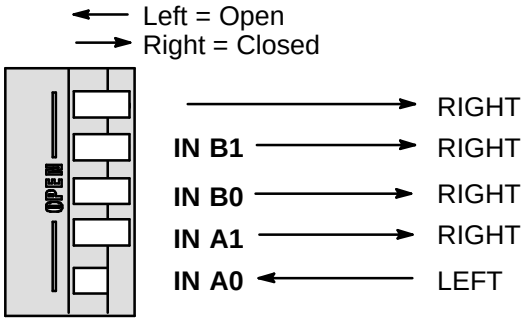
INLET B = Cool On-Column with Manual Pressure Control

INLET A =Any Other Inlet



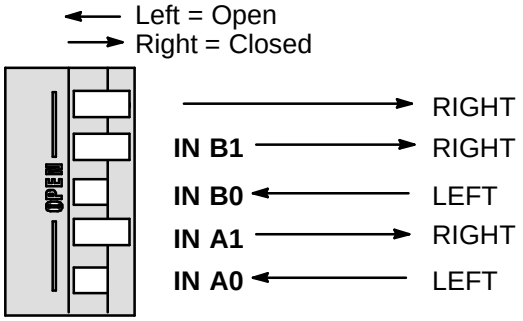
INLET B = Any Other Inlet

INLET A = Cool On-Column with Manual Pressure Control



INLET B = Cool On-Column with Manual Pressure Control

INLET A = Cool On-Column with Manual Pressure Control



Useful tools

- **Ultrasonic cleaning bath•** It is the only effective way to clean small parts.
- **Tungsten carbide knife•** Used for cutting fused silica tubing. The knife edge can be kept sharp with an inexpensive diamond hone, such as those used for touching up carbide-tipped saw blades and router tips.
- **Drill index (#61-80)•** Used for drilling holes in Vespel and Graphite/Vespel ferrules.
- **Reamers•** Used to ream slightly undersized holes drilled in ferrules to match column diameter closely.
- **10x magnifier•** Used for examination of column ends and small hardware.
- **Hemostats** (curved or straight)**•** Very handy for manipulating small parts without losing them or contaminating with fingerprints.
- **Fine abrasive paper** (emery or 600-grit silicon carbide)**•** Used for smoothing rough edges and polishing surfaces.