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Troubleshooting Fused Silica Capillary Systems

When GC problems arise, a new, high quality, pre-tested capillary column is not generally responsible. The true source of the problem must be pinpointed in order to solve it and prevent a recurrence. Outlined below are some of the most commonly observed problems and a logical procedure for isolating the cause and correcting these problems. Remember, whenever any part of the GC system (lines or column) is opened to the atmosphere, flush the system thoroughly with clean carrier gas at room temperature for 20 minutes to ensure the lines and column are free of oxygen.

Problem: Rising Baseline with Increasing Oven Temperature: Bleed or Noise

A steadily rising baseline with an increase in oven temperature is generally due to contamination or column phase degradation. It is necessary to isolate the source of the bleed by examining the system using the following schedule:

(1) Detector:
- (i) Remove column.
- (ii) Cap the detector.
- (iii) Monitor the detector signal (it may be necessary to add make-up gas to the detector to run it without the column).

If the bleed persists the problem is most likely a result of a build up of contaminants at the detector. If the bleed is eliminated disregard (iv) and check the injector.
- (iv) Disassemble the detector and clean, as per the manufacturer's instructions

(2) Injector:
- (i) Remove column.
- (ii) Install a 1meter length of clean deactivated fused silica tubing between the injector and detector.
- (iii) Run a temperature programmed baseline and monitor the detector signal.

If bleed persists there is a contamination at the injection end of the system.
- (iv) Disassemble the injection port and thoroughly clean all surfaces and liners.
- (v) Replace the septum.
- (vi) Check gas lines and purifiers to ensure that the carrier gas is clean.
- (vii) With a clean detector and injector and a piece of deactivated tubing, a steady detector signal should be observed.

If the column being used has a bonded phase, rinse (backflush) the column before reinstalling. Any contamination in the injection port may have been transferred to the column. The column should be rinsed from detector to injector to remove any build up of low volatility compounds. Do not rinse unbonded columns.

If the bleed persists with the rinsed column installed in a clean system, check the column.

(3) Column:
Non-volatile residues can build up in the column and slowly elute at high temperatures. Residues, if allowed to remain in the column, can catalyze the degradation of the column phase.
- (i) Cut 1/2 meter off the front end of the column, since residues tend to accumulate in the front portion. Since column resolution is proportional to the square root of the column length, several meters can be removed without seriously affecting the separation.
- (ii) Backflush the column with a series of solvents to dissolve any remaining residues.
- (iii) Reconnect the injector end of the column and dry carefully with carrier gas at room temperature. Connect the detector end and monitor the signal.

If bleed persists it is possible that degradation of the stationary phase has begun. At elevated temperatures oxygen can cleave the bond holding the non-polar stationary phase to the fused silica surface. This can occur in more polar columns at much lower temperatures. This is a self-sustaining reaction, and once initiated, will continue even in the absence of oxygen.

Problem: "Ghost Peaks"

Follow the previously outlined steps to determine where the peaks are originating, as they may well be a result from sample flashback. Capillary systems have smaller vaporizing chambers than packed columns. If large sample volumes are used and there is insufficient space for volatilization the sample may flash back to the septum face and condense, only to be re-eluted in successive runs. This is also the case with extremely hot injection ports. The sample vaporizes too quickly and flashes back to the septum face.

To minimize this problem:
(1) Keep the injection port temperature only as high as necessary to vaporize the sample.
(2) Keep injection volumes to the size of the vaporizing chamber in the system.
(3) Keep the injection port free of sample residues and septum debris.

Problem: Septum Bleed

Most commercially available septa are a silicone rubber material. At high temperatures (both oven and injection port) residues can bleed out of the septum and into the column. Repeated piercings may result in an increased amount of bleed.

To minimize this problem:
(1) Keep the injection port temperature only as high as necessary to vaporize the sample
(2) Solvent rinse the septum face before using and handle with tweezers. Skin oils are often released at high temperatures.

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(3) Use a good quality, low bleed septum designed for high temperature work. For septa recommendations for a particular set of conditions, contact our Technical Service Team.
(4) Replace the septum regularly.
(5) When using a PTFE-faced septum, keep the injection port temperature low. Although PTFE is a very inert material it may show signs of chemical breakdown as low as 200°C.

Problem: Badly Tailing Solvent Peak

Generally, this is representative of improper flowrates at the inlet, a leak at the front of the column, or a poorly positioned column.

(1) Inlet flow rates:
(i) If working with split injections, it may be necessary to adjust the split ratio.
(ii) If working with splitless injections, it may be necessary to adjust the purge time. A badly tailing solvent peak could indicate the purge was activated too late.

Consult the GC instruction manual for the recommended flow rates of the different injectors.

(2) Leak:
This can often be corrected by removing the inlet end of the column, recutting the column end to ensure an even, clean cut and repositioning the end in the inlet, making sure a leak-tight seal is obtained.

(3) Improper Column Positioning:
Different injection modes have different specifications for column end positioning. Check the GC instruction manual for the proper positioning of the column in the injection port or liner.

Column Care and Maintenance:
Preventing Premature Column Degradation

Fused silica capillary columns are designed to perform properly under certain specified conditions. Operating the capillary column within the manufacturer's guidelines will help to reduce the time spent troubleshooting problems.

Outlined below are several tips to prevent premature column degradation and to help extend column life.

1) Oxygen-Free System
Bonded phase capillary columns should be operated with a high purity carrier gas. Even on ultra-pure gas lines, oxygen scrubbers should be installed to trap any oxygen from the cylinder or from leaks in the GC system.

2) Temperature Limitations
Each bonded phase column has its own set of temperature limits. For the best performance, the column should be operated within the temperature range given. Some users report routine analyses at both high and lower temperatures under carefully controlled and monitored conditions but a shorter column life usually results.

Never "bake-out" a fused silica capillary column. Heat puts stress on the column, and prolonged exposures to excessive heating will shorten the useful lifetime of a capillary column.

3) Column Rinsing
Only bonded phase columns can be rinsed. Rinsing non-bonded columns will result in the stationary phase being removed from the column. When working with bonded-phase capillary columns, a Capillary Column Rinsing Kit is a vital accessory. It provides a convenient method for backflushing columns to greatly extend their longevity. Residues built up in a column can catalyze the destruction of the phase and should be removed from the column.

Regular solvent backflushing will improve chromatographic runs and ultimately increase the useful lifetime of the column. All bonded phase columns can be rinsed. A good general purpose rinsing would consist of:
(i) methanol followed by;
(ii) dichloromethane followed by;
(iii) hexane

These solvents range from polar to non-polar and each is miscible in the previous solvent.

Capillary Column Rinsing Kit: RK20612

4) Guard Columns
For samples known to be "dirty", a guard column should be used. Attach a 1 meter length of deactivated fused silica tubing to the inlet of the column using either a stainless steel union or a glass connector. Non-volatile compounds will be trapped in this tubing and will not have the opportunity to reach the column and degrade the stationary phase. The guard column can easily be replaced as necessary.

5) Test Mixes
Test mixes provide valuable information about the condition of the column. A test mix (containing the solutes separated on the test chromatogram) is available for every column. Immediately after installation it is good practice to run the test mix to ensure the column is performing as the test chromatogram indicates. Periodic injections of the test mix will help to monitor column performance.