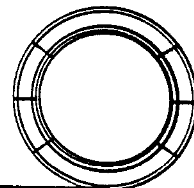
Hints for the Capillary Chromatographer



Becoming Proficient at Troubleshooting Capillary Chromatography Systems

The key to good troubleshooting is to methodically, logically, and quickly pinpoint problems that arise. Good problem solving techniques are essential in a laboratory to minimize down time.

The first and most important step to becoming proficient at troubleshooting is to read the instrument manuals. Instrument manufacturef s invest a lot of time and expense in writing manuals to provide a better understanding of the GC system. They include many of t& basic concepts tfi;y have learned over the years that help avoid many common pitfalls. They also provide detailed flow l%tth diagrams and instructions for disassembling injectors, detectors, and other parts that require customer servicing. Spend some time and review the manual. Learn about the inlet and detector systems. Understand the basic pneumatics and flow paths and know where the critical seals are located to quickly identify the source of most problems that arise.

To Begin Troubleshooting

First, determine if the problem is column or system related. Frequently, analysts call our technical service department with what they believe is a column problem. However, after some basic troubleshooting questions, it often turns out to be a bad inlet sleeve or improperly set carrier gas.

To determine if the system or column is the problem, simply install a column of known performance. If the problem remains, then it is most likely a system related problem, or a problem with both the system and the column. If the problem goes away, then it could have been column related or simply that the problem was corrected during re-installation. To be certain that the problem was column related, re-install the problem column again to make sure that the problem reappears.

Routine Instrument Maintenance

Usually, a careful methodical approach to troubleshooting is not attempted until the common instrument problems are addressed. Common instrument maintenance procedures performed are:

- changing dirty or contaminated inlet sleeves
- replacing the septum
- checking the inlet seal (o-ring or ferrule) for leaks
- confirming proper column insertion distances
- leak checking all column connections and external fittings
- replacing spent purifiers
- checking for properly set flow rates and linear velocity
- inspecting gauge pressures, electrometer settings, all temperatures, signal levels, integrator settings, and any other parameters that could be suspect.

Routine Column Maintenance

While the column is out of the instrument, perform routine column maintenance. Common maintenance procedures performed are:

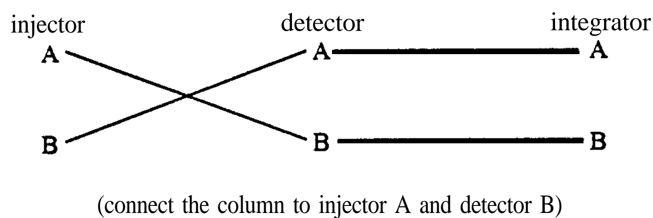
- inspecting the column for spontaneous breakage, discoloration, or contamination
- cutting two loops off of the inlet and one loop off of the detector end of the column

If Routine Maintenance Does not Work, Begin Diagramming and Documenting

Now troubleshooting gets tough. If the problem is not solved after routine maintenance, immediately begin documenting what has been done and start diagramming what should be done. This aids in communicating to others what effect changing variables have on solving the problem. Document the procedures in chronological order listing times, dates. and important instrument parameters. Label all troubleshooting chromatograms. These steps help to inform anyone else that may be working on the system of the troubleshooting procedures that have been completed.

Start with a simple instrument diagram (Figure 1), and try switching Column A to Detector B and vice versa. If the problem moves to detector B, then the problem is most likely occurring in the injector.

Figure 1 - Begin problem isolation when system is at fault.



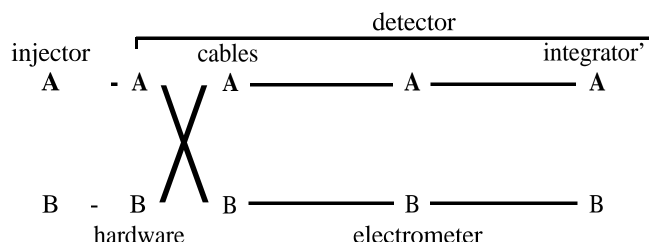
(connect the column to injector A and detector B)

Some common injector problems we have observed are:

- wrong size graphite o-ring on HP inlet sleeve
- wrong sleeve type (using splitless sleeve for split analyses)
- leaking or contaminated metal disk on HP inlet
- bad solenoid valve containing split flow
- knife edge not cutting septum in Varian systems
- wrong length sleeve used in Varian systems
- not using glass wool with fast injecting autosamplers

If the problem stays on detector A when the column outlet is switched, then suspect a detector problem. Begin isolating detector problems by switching hardware, cables, electrometers, integrators, or any suspect part in the pathway. Figure 2 shows the detector hardware being isolated. If the problem goes away from the A side when the detector base is changed, then that detector is most likely the cause and should be replaced.

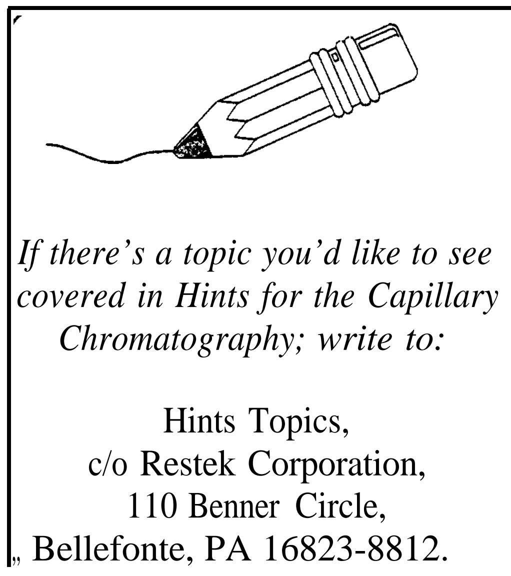
Figure 2 - Isolating Detector Problems



Some common detector problems we have observed are:

- broken or leaking jets
- column or ferrule fragment located inside the jet
- plugged jet orifice
- column installed too far into the detector
- oxidized polarizer or signal contacts
- shorted insulator on the collector assembly
- leak at the detector base
- bad needle valve or regulator
- incomplete or oxidized ground
- bad heater or heater controller
- air conditioning air currents blowing on the detector
- bad or contaminated carrier or combustion gasses
- bottled air with less than 21% O_2
- detector gasses not set properly or optimized

The complexity of a capillary GC system almost guarantees that one day you will be faced with troubleshooting a difficult problem. If you have read the manuals and follow a logical troubleshooting sequence, you can quickly isolate the cause of most problems.



If there's a topic you'd like to see covered in Hints for the Capillary Chromatography; write to:

Hints Topics,
c/o Restek Corporation,
110 Benner Circle,
Bellefonte, PA 16823-8812.

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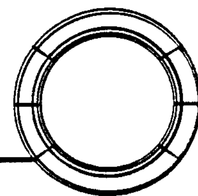
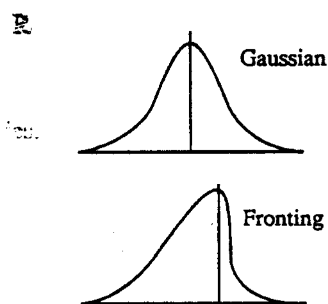


Sample Capacity and Column Overload

What is column overload?

Column overload occurs when the amount of sample injected exceeds the column's capacity for that component. Overload is normally observed as a fronting, non-gaussian peak shape (Figure 1). A column's capacity is a function of several parameters including the columns internal diameter (ID), its film thickness (df), the solubility of the compound in the columns stationary phase, and capacity factor (k).

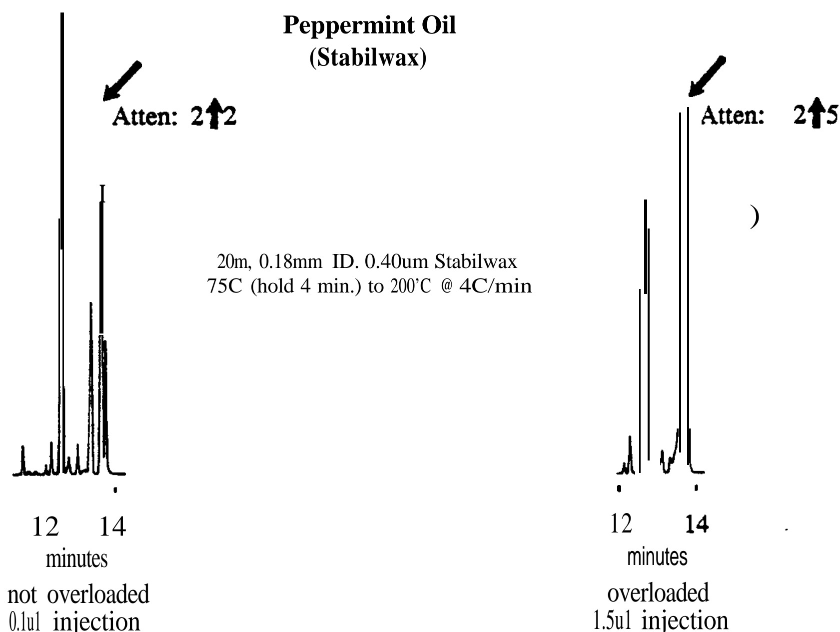
Figure 1 - Normal Gaussian vs. Overloaded Fronting Peak Shapes



Why is **it important not to exceed a column's capacity?**

Capillary columns have much lower sample capacities than packed columns, therefore, it is extremely important to optimize the amount of sample injected. When sample capacity is exceeded, peak symmetry is lost and resolution is affected. Because the peak shape will be *much broader, resolution between two closely eluting peaks can be lost. Figure 2 shows the loss of peak symmetry and resolution in the analysis of peppermint oil on a Stabilwax column. In the first chromatogram, 0.1 ul of neat peppermint oil was injected. At these low concentrations, very good resolution between the menthyl acetate, neo-menthol, b-caryophyllene, and terpinene-4-ol is obtained. In the second chromatogram, 1.5ul of neat peppermint oil was injected. Because the sample concentration exceeded the column's capacity, a significant loss in resolution occurred.

Figure 2 - Minimize the amount of sample injected to maximize resolution.



How can overload be prevented?

Two choices are available to prevent overload:

- ▼ reduce the sample concentration reaching the column
- ▼ choose a column and run conditions that will allow greater sample capacity

To reduce the sample concentration reaching the column, the sample components can be diluted by increasing the split ratio, diluting with additional solvent, or by introducing a smaller amount.

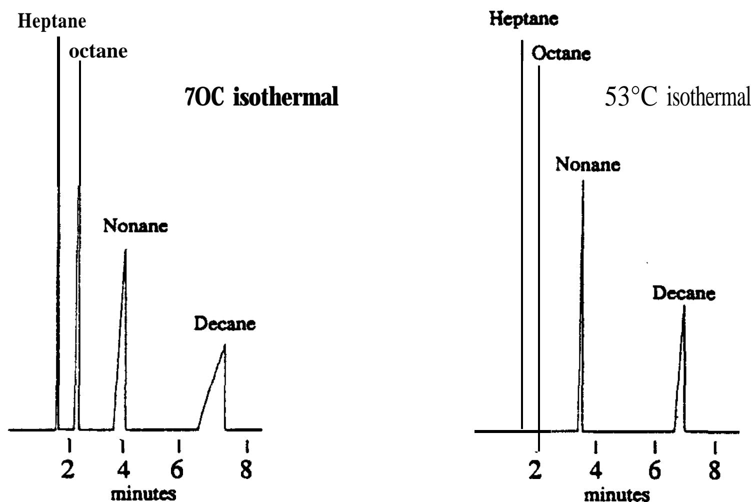
How does column ID affect sample capacity?

As the column ID increases so does sample capacity. Table 1 shows typical column capacity for several different diameter columns. Figure 3 compares sample capacity on 0.25 and 0.53mm ID columns. Four hydrocarbons Oleptane, octane, nonane, and decane) were analyzed at a concentration of 1000ng on

Table 1

Column ID	0.18mm	0.25mm	0.32mm	0.53mm
Sample Capacity	<50ng	50-100ng	400-500ng	1000-2000ng

Figure 3 - Increase sample capacity by increasing column ID.



15m, 0.25mm ID, 0.25um Rtx-1 (cat.10120)

15m, 0.53mm ID. 0.25um Rtx-1 (cat.# 10122)

both a 0.25mm ID and 0.53mm ID column. The 0.25mm ID column exhibits overload and severe peak fronting for nonane and decane. In comparison, the 0.53mm ID column shows symmetrical peak shapes for nonane and only slight overload for decane. This illustrates the effect of increasing sample capacity by increasing column ID.

How does column film thickness affect sample capacity?

Increasing the column's stationary phase film thickness also increases sample capacity. Figure 4 shows this effect. Again, we show the same series of hydrocarbons at the 1000ng concentrations on 30 meter, 0.25mm ID, 0.25um and 1.0um Rtx-I columns. On the 0.25um column, the nonane peak shows some overload and the decane peak shows severe fronting. By increasing the film thickness to 1.0urn, the peak symmetry of nonane is restored and the decane peak shows only slight fronting.

How does solubility affect sample capacity?

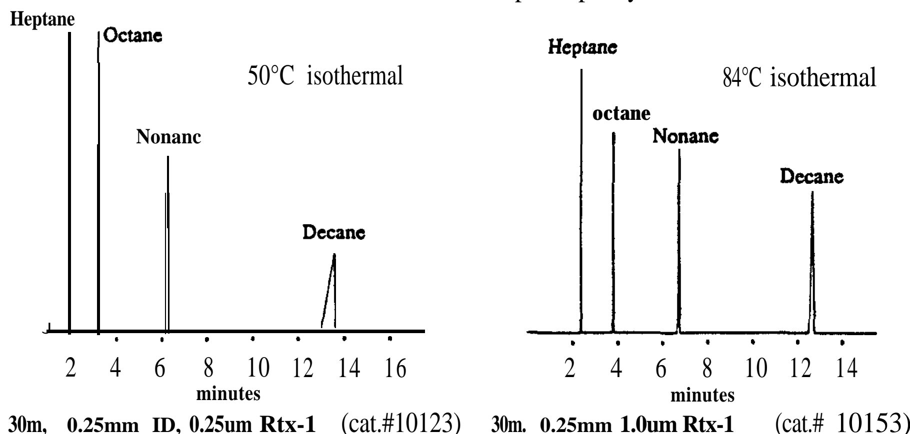
The solubihty of a sample component in the column's stationary phase also has an effect on sample capacity. The more soluble a component is in the stationary phase, the greater the column capacity for the solute. For example, a polar compound will have greater solubility in a polar stationary phase than in a non-polar stationary phase. For environmental analysts, this phenomenon is very common when analyzing acid and base-neutral extracts on a non-polar, 5% diphenyl stationary phase. Benzoic acid, a polar compound, always exhibits very poor peak symmetry, demonstrating overload on this non-polar stationary phase. Even though it is at the same concentration, it is less soluble in this phase than the other priority pollutants and exceeds the column's capacity at a much lower concentration.

How does component retention affect sample capacity?

Sample capacity is also affected by how long the component remains in the stationary phase. The capacity factor or k gives us an indication of how long the component remains on the stationary phase. The longer a sample component remains in the stationary phase, the greater the chance for overload. The capacity for a component can be increased by selecting run conditions that will create lower *k* values by causing the component to elute faster from the column (faster flow rates or faster temperature programming).

When choosing a column, the analyst must keep in mind the range of component concentrations. By optimizing column ID and film thickness, and by matching the solubilities of sample components with the stationary phase, samples can be analyzed without overload. Also, by optimizing run conditions, the ***k*** value for components can be minimized resulting in better sample capacity.

Figure 4 - Increasing the stationary phase film thickness increases column sample capacity.



30m, 0.25mm ID, 0.25um Rtx-1 (cat.#10123)

30m. 0.25mm 1.0um Rtx-1 (cat.# 10153)

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