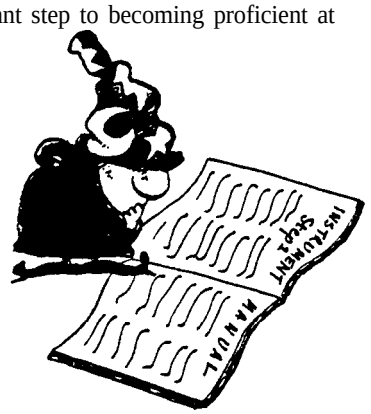
Hints for the Capillary Chromatographer

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:

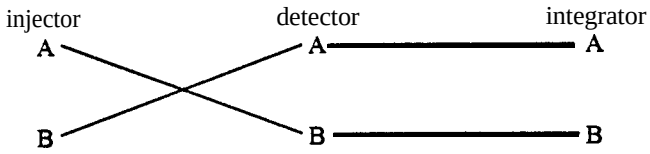
- inspecting the column for spontaneous breakage, discoloration, or contamination
- cutting two loops off of the inlet and one loop off of the detector end of the column

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 l), 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.



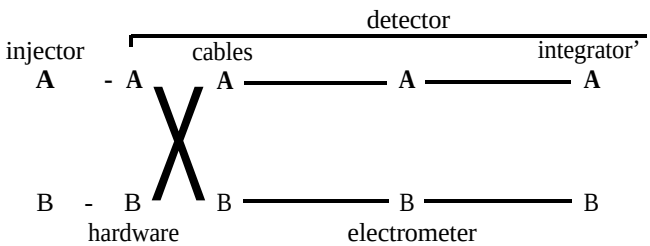
(connect the column to injector A and detector B)

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.

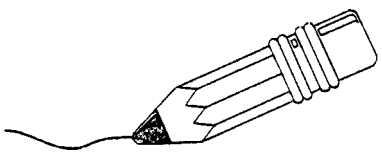
Figure 2 - Isolating Detector Problems



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% 02
- 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.