1150 Troubleshooting

Most of the problems associated with sampling valves are related to peak broadening in transfer lines and inlets, sample adsorption by the valve or transfer lines, leaks, and perturbations in the baseline.

Chromatographic symptoms

Troubleshooting valves and their related plumbing is primarily a matter of systematic checking and verification of unimpaired mechanical operation of any moving part. This requires an understanding of how the valve functions internally and how the plumbing is configured. A plumbing diagram is essential for effective troubleshooting.

The following “symptom-cause” list gives the most commonly encountered problems found with valves and their solution.

Table 1150-1 Troubleshooting valve related chromatographic problems

Symptom	Possible cause	Solution
Lost peaks (degradation)	Valve or transfer lines too hot	Reduce temperature 50°C, reevaluate
	Transfer line activity	Use nickel or Hastelloy tubing
Lost or tailing peaks	Valve or transfer line too cold	Increase temperatures 50°C, reevaluate
Baseline perturbation	Slow valve rotation	Increase actuator pressure
	Rotor distorted	Replace rotor
	Sample/column pressure too different	Add back-pressure regulator to sample drain
Peak tailing broad peaks	Column overload	Use smaller sample loop Increase split flow
	Flow too slow	Increase column flow Increase split flow
	System voids	Check connections Reduce volume of connecting tubing

Loss of sensitivity or excessive drift

Several possible causes exist for overall deterioration of the chromatogram.

- Contamination in the valve requires a thorough cleaning.
- Internal leakage requires a complete disassembly and inspection of the mating surfaces.
- Poor temperature control may require a full check of electronic and thermal components.
- Lack of proper conditioning techniques, columns, etc.
- Failure or deterioration of other components (columns, detectors, etc.).

Loss of peaks in specific areas of the chromatogram

Entire sections of chromatographic data can be lost due to a valve that does not rotate or one that rotates improperly. Other than obvious component failures (solenoid, actuator, etc.), improper adjustments and misalignments cause most problems.

- Check that adequate air (about 482 kPa or 70 psi) is supplied.
- Check the valve. Is it rotating?
- If the valve rotates, check for proper alignment of the actuator, mechanical binding or slippage of connecting parts.
- Check for blocked flow paths with valve in both positions.

Extraneous peaks

Air peaks are sometimes seen in a chromatogram when leakage occurs because the valve rotor does not seal properly. These leaks may not be detectable using the soap-bubble method.

If a leak is suspected but cannot be located with soap bubbles, a pressure check will determine definitely if a leak exists. Extraneous peaks can occur due to contamination or improper conditioning of the valve. If leaks are not apparent, clean or condition the valve.

Other causes, totally unrelated to the valve, may produce similar symptoms. Impure carrier gas (i.e., containing water) can cause extraneous peaks.

Peak broadening and tailing

Voids in the flow system (valve and connecting tubing) cause tailing and peak broadening. Use inlets and liners with small internal diameters and connect the valve to the inlet or column with short lengths of connecting tubing of narrow inner diameter.

If early-eluting peaks are too broad, stationary phase or thermal focusing effects should be used with packed-column ports or increased split flows when capillary split inlets are used. Inlets should be equipped with narrow inner diameter liners, and narrow-bore connecting tubing should be used between the valve and inlet.

Baseline shifts

Baseline perturbations are caused by changes in column flow as the valve is rotated and as the sample loop equilibrates to system pressure. Slow valve rotation momentarily stops carrier gas flow and, when the valve stops rotating, a sudden increase in flow occurs which slowly returns to the set point. Check actuator pressure (usually 40 to 75 psi), valve rotor tension, and valve temperature to ensure that the valve rotates as quickly as possible. A restrictor or backpressure regulator can be added to the sample vent line to maintain the sample loop at system pressure. This will reduce the time it takes for the flow to stabilize after the valve is switched.

Baseline upsets

Frequently, baseline upsets may be seen on chromatograms when valves are switched. These upsets are caused by pressure changes within the system, injections of large volume samples, or by changing the amount of restriction in the flow path. These upsets will become more of a problem when high sensitivity is required. Addition of a fixed restriction downstream from the valve may help minimize the upset. Changes in column length may also help reduce the upsets.

Fixed restrictors are used immediately before flame detectors to prevent flameout and are used in some instances to prevent pressure surges from damaging TCD filaments. An adjustable restrictor (needle valve) can also be used where a matched restriction is desired but not for preventing pressure or flow surges.

Often confused with baseline upsets, an offset is a shift in the baseline that does not return quickly to the original level. Baseline offsets may be caused by air leaks but more commonly are due to a change in gas purity or flow rate in the detector. Poor carrier gas or improperly conditioned filters and traps should be suspected whenever offsets occur.

Variation in peak area and retention time

The amount of sample contained in the loop and, therefore, the amount injected onto the column is affected by loop pressure and temperature. Variations in pressure and temperature lead to variability in peak areas. Flow restrictors or back-pressure regulators help to maintain constant loop pressure, and valve boxes help maintain temperature.

Leaks can occur in the valve itself or at any of the connecting points with transfer lines. Leaks usually cause area irreproducibility, retention times changes, and increases in the area of air peaks (with thermal conductivity detectors). Leaks in rotors can sometimes be fixed by tightening the nuts holding the rotor in the valve body. Leaks in connections are usually found with an electronic leak detector or with a liquid leak detection fluid (e.g., Snoop).

Pressure check

Leak checking the plumbing involved in a valve system must be done carefully and methodically. The pressure check method below will indicate, but sometimes not isolate, a leak in the flow path. Since this method does not necessarily isolate the leak, other leak check methods may be needed to locate the leak specifically.

Note Each valve in a system has two flow paths, ON and OFF. A leak sometimes occurs in only one of these two positions. Check both.

1. Disconnect the detector from the valve system.
2. Cap the valve system at its outlet and pressurize to 689 kPa (100 psi). Allow 2 to 5 minutes for pressure to equilibrate. If your instrument has flow control, it should read zero flow.
3. Turn off the gas supply at the source.
4. Generally, the pressure will drop quickly for approximately 30 to 60 seconds, then stabilize. After this initial pressure drop, the gauge should not indicate more than a 7 to 14 kPa (1 to 2 psi) drop during a 10 minute period.

5a. If no leak is indicated, actuate all valves and repeat steps 2 to 4.

5b. If a leak does show up, try to pinpoint the source using a soap bubble meter. Do not assume that the leak exists only at the valve. Often plumbing connections such as unions or bulkhead fittings are at fault. See Valve Box should it become necessary to expose the valve system.

6. If the leak cannot be found easily, divide the system in half and repeat the pressure check. Continue dividing in halves, and pressure check until the leak is isolated.