TROUBLESHOOTING

Piston-Diaphragm Pumps

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In last month's column we reviewed troubleshooting of reciprocating-piston pumps. These pumps are characterized by: solvent-piston contact, the need for a high-pressure seal around the piston, relatively slow pumping speeds (10–100 cycles/min), and reduced flow pulsations through the use of pulse dampeners, multiple heads, or shaped driving cams to move the pistons at constant linear speed (as opposed to sinusoidal speed). Although reciprocating piston pumps were some of the first to be used for HPLC, the piston-diaphragm (or membrane) pump has also been used since about 1970. Diaphragm pumps are used in the chemical industry to add precise but small amounts of fluids to chemical processes — especially those in which the chemicals are corrosive, no contamination is allowed, or long-term operation without maintenance is desired. These pumps are especially useful for salt solutions because seals, which can be worn down by crystals, are not used.

This month's column describes methods for troubleshooting piston-diaphragm pumps in LC systems. Because many of the same problems can occur in different pump designs, the diagnostic and corrective procedures described for the diaphragm pump will also be of value to those using other kinds of pumps.

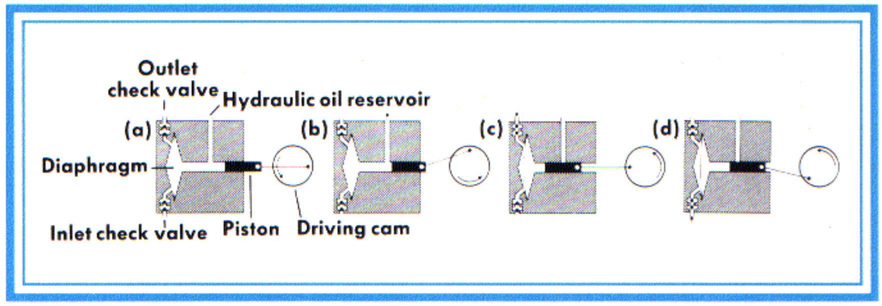

FIGURE 1: Cross-section of a piston-diaphragm pump head. (a) Piston in fully retracted position at end of suction stroke; (b and c) delivery stroke; (d) suction stroke.

PISTON-DIAPHRAGM PUMP OPERATION

Characteristics of a piston-diaphragm pump include:

- a flexible metal diaphragm that isolates the eluent that is being pumped from the actual pumping piston
- the absence of seals
- fast pumping speeds (approximately 300 cycles/min)
- reduced pulsations by using fast pumping speeds or pulse dampeners
- the absence of shaped driving cams.

The operation of a diaphragm pump is shown in Figure 1. As the piston moves forward, it traps a volume of hydraulic pumping oil from the oil reservoir partway through the stroke (Figure 1, positions b to c). As this oil is moved forward it causes the metal diaphragm to flex forward and forces the bottom check valve closed, which causes the eluent to move out of the pump head through the upper check valve (position c). As the piston withdraws, the lower check valve opens and eluent is pulled into the front chamber (position d). The metal diaphragm completely isolates the eluent from the hydraulic pumping oil and the piston. The piston fits closely in the piston chamber, but because seals are not used, oil can leak between the piston and piston chamber. Because of this oil leakage, it is necessary to operate the pistons at high speeds so that flow does not drop significantly with increased back pressure.

This high-speed operation generates high frequency pulsations that can affect spectrophotometric or refractive index detectors. Early diaphragm pumps used three or more pump heads that operated out of phase so that the sinusoidal pulses of each pump head overlapped to give a more constant flow. Flow pulsations were dampened out by pulse dampeners and by the natural expansion and contraction, or the *compliance*, of the system.

In the currently available diaphragm-pumping systems, the same high-speed pumping is used, but only one head is used for each solvent. The flow from each pump head goes through a purge valve that permits solvent to be drawn through the heads for priming, for flushing out old solvent, and for observing flow from each head. The same membrane technology used in the pump is used in the damper-sensor assembly to simultaneously dampen pulsations and measure pressure and flow. In the damper-sensor of each pump, the eluent passes over one side of a flexible metal membrane and the other side is filled with hexane (thermostated to 40°C). Information from a pressure transducer in the hexane chamber, combined with the known compressibility of hexane, is used by the microprocessor to calculate flow from the decay of each pressure pulse.

Flow is not controlled by changing the frequency of pulsations, as with piston pumps, but rather by changing the depth to which the piston pushes into the oil chamber. If frequency were changed, the slowest flows would allow all the hydraulic oil to squeeze around the piston and the result would be no flow. The pump heads are advanced onto or retracted from the piston with a stepping motor to control the flow from each pump head (there is one pump head for each eluent). The relative position of the two heads, under the control of the microprocessor, determines the percent of eluent A in eluent B. From the damper-sensor, eluent is fed to a mixing chamber, which has a third pressure sensor to measure combined ouput pressures, and then eluent is fed to the injector and column.

While we are discussing troubleshooting for diaphragm pumps, it should be emphasized that all parts of the pumping system interact for final pump performance, just as in any other modern liquid chromatograph.

These parts include the pump heads, the individual damper-sensors, the mixing-chamber pressure sensor, and the microprocessor that conditions and feeds back data. Thus, problems in the pump may mean problems in these other components. As was discussed in February's column, the problem should be isolated to the pump itself; in other words, first eliminate problems external to the pump such as solvent problems, leakage, or blockages, before working on the pump itself. If the following pump troubleshooting efforts fail, malfunctions of the damper-sensors, mixing-chamber pressure sensor, or control electronics are indicated and generally require a service call.

Before examining the most common pump problems, be sure to check that a problem really does exist by making sure that the flow and pressure settings are correct and that the columns and eluents are what you expect. If we now assume that these other elements have been eliminated as the source of the malfunction, then that leaves us with the diaphragm pump to consider as the source of the problem.

CHECK FOR AIR ENTRAPMENT

If the pump is switched on and you can hear it operating, but there is no flow, entrapment of small bubbles of air in either the pump-head inlet or the outlet check valves may be the cause of pump malfunction. (This problem can also occur with piston-displacement pumps, as described in the February issue of *LC*.) There are several indicators for this problem, the most obvious of which is low flow. If a mixture of A and B solvents is used, the %B will be either 0% B (indicating that the B pump is inoperative) or 100% B (indicating that the A pump is inoperative). This phenomenon can be confirmed by checking to be sure that the inoperative pump head is fully inserted over the piston (that is, no threads are showing). A detailed account of methods for finding and eliminating air entrapment problems by eliminating leaks and degassing eluent, and the proper start-up procedures were discussed in the February issue.

REMOVING AIR

Many of the same methods for removing air bubbles from piston-displacement pumps can also be used with piston-diaphragm pumps. These methods include solvent degassing, replacing check valves, and bleeding air from the system. Some precautions, however, must be observed with diaphragm pumps to avoid ruining the columns, creating leaks, or rupturing the pump diaphragm itself. A special problem can develop if the damper-sensor pressure transducer reads zero pressure: it signals the microprocessor to increase the pump output. The pump head is then screwed onto the piston to produce the maximum flow (10 ml/min). Unlike most conventional piston pumps that can respond to flow changes instantly, the diaphragm pump takes approximately 60 sec to go from the 0% to 100% flow positions. The pump flow may still be set at 1 ml/min; however, the pump will attempt to pump 10 ml/min. If, by undertaking any of the approaches described below, the check valves in the pump head are restored to operation, a flow of 10 ml/min will result. This high flow may be adequate to flush bubbles from the pump. The system will, however, momentarily experience a high-pressure surge before the pressure transducer can shut down the pump. This pressure surge can damage parts of the system — especially the columns. Before attempting to restore the operation of the check valves, it is essential to open the purge valve on the high-pressure side of the pump. Once flow is restored through the check valves, it is essential to shut off the pump and wait for up to 60 sec to let the heads retract before starting up the pump again.

Flow through the check valves can be restored in any of several ways, but be sure the purge valve on the high-pressure side of the pump is open. If the pump is on, flow will occasionally be restored just by opening the high-pressure purge valve. If the flow problem is caused by a check valve stuck open with air bubbles, the easiest and safest approach is to tap the upper and lower check valves firmly with a plastic screwdriver handle. If the pump is running, this action will often release air bubbles from the check valves. If an inlet gas ballast/bubble trap is used with an in-line, 0.45-μm filter (*LC*, Vol. 1, pp. 542–544, 1983), the 25-ml syringe can be used to readily apply positive and negative eluent pressure through both the inlet and outlet check valves; this method will frequently start flow. Another method for starting flow is to attach a syringe to the purge valve and pull solvent through the pump. As an alternative, unscrewing each check valve one-quarter turn often starts flow, but take care not to overtighten check valves because this could destroy them. Because flow may start abruptly at any time when these techniques are used, standard laboratory safety precautions, such as eye and skin protection, should be taken.

If the above approaches do not start flow, clean or replace the check valves. If an overpressure condition exists and run conditions, column, and eluent are correct, then the problem may be that the pressure transducer in the damper-sensor is blocked or has electronic problems that require a service call. This problem can be diagnosed further by switching pump outlets (which will also switch damper-sensor transducers) in order to see if the overpressure problem also changes.

CHECK FOR DIAPHRAGM BREAKAGE

In the rare case in which the outlet valve sticks in the closed position, an overpressure condition will develop in the pump head itself and the diaphragm may break. Diaphragms can also break from the fatigue of long use. A perforation in the diaphragm often is indicated by milky emulsion coming out of the column or the high-pressure purge valve (only if the hydraulic oil is immiscible with the eluent). A perforated diaphragm is also indicated when there is no hydraulic oil in the oil reservoir. If more oil is added to the reservoir and it rapidly disappears, this indicates that the membrane has a major perforation. Smaller holes may be harder to detect. It is recommended that a record of the oil use of each pump head be kept in the logbook. It should not be necessary to use more than 25 ml of oil per month per head. If the pump requires more, a leak around the membrane on the oil side should be suspected.

OTHER PROBLEMS

Unlike reciprocating-piston pumps, seal deterioration and piston breakage are not problems associated with the membrane pumps. As explained above, the piston operates rapidly in a bath of hydraulic oil, and therefore no seals are needed around the piston. Because the piston is made of steel and is tightly attached to the pump body, it is unlikely that a piston will break. The piston and seat can wear out, and replacement may be required after prolonged use. If erratic, low, or high flows persist after check-valve purging and replacement, and the diaphragm appears to be intact, the pump problem may lie with electronic parts of the system and a service call may be required.

CONCLUSIONS

Diaphragm-piston pumps require the same general troubleshooting techniques as reciprocating-piston pumps. First of all, isolate the pump problem and then use procedures recommended specifically for your pump type to eliminate the problem. The pump operations manual contains valuable guidelines for troubleshooting a particular brand of pump. Most manufacturers also provide telephone consultation to help you solve a problem before a service call is required. As with any liquid chromatograph, follow factory-recommended preventive maintenance schedules and use clean solvents for maximum return on your LC investment.

ACKNOWLEDGMENT

We wish to thank Hewlett-Packard for reviewing this article and for permission to use the figure. ■

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