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Pulseless High-Pressure Pump for Liquid Chromatography

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IN HIGH-PRESSURE liquid chromatography, the eluting solvent is generally pumped into the column by a reciprocating piston or bellows pump. Pulses generated by this pump can often be discerned by the detector. Besides introducing difficulties in the interpretation of the detector output, pulses also have a detrimental effect on the separating efficiency of the column. A major portion of these pulses can be eliminated by the use of surge tanks—e.g., spring-loaded bellows connected to tees in the line between the pump and the column inlet. But even careful design will not remove all pulses from the flow pattern and rather complicated and/or large surge tanks, with concurrent large holdup of material, are necessary to reduce the level of the flow variations to a negligible level. In addition, damage to this pump and surge system is feasible if the outlet line is accidentally blocked.

With the pump described here, high pressures up to 1000 p.s.i. can be generated at the outlet of the pump. In comparison, many of the commercially available pumps are incapable of operating above 100-p.s.i. pressure. Delivery into the chromatographic system can be carried out without any discernible pulses for any length of time and any amount of solvent. Accidental blocking of the outlet line does not damage the pump at all. Construction is simpler than reciprocating plunger pumps of equivalent rating.

DESCRIPTION AND OPERATION OF PUMP

A schematic drawing of the unit is given in Figure 1. Four 2-inch by 8-inch stainless steel reservoirs, constructed from pipe nipples, are half-filled with mercury, the displacing agent. High pressure air is used as the source of operating pressure. The use of mercury as the displacing agent prevents the dissolution of the air in the solvent.

The outlet of the pump is on the top of Vessel *A*. For operation up to 250 p.s.i., a dual back-reference flow controller with integral micrometering valves (Millaflow No. 9986 220) is mounted between this outlet and the inlet of the chromatographic column. Because these type flow controllers are currently not available for higher pressure ratings, a fine metering valve is used for work at higher pressures.

The pressure-reducing valve in the air line is adjusted to yield a pressure about one and one-half to two times the desired inlet pressure to the column, but not less than 100 p.s.i. to minimize the effect of the variations in the mercury levels. Prior to introduction into the pump, the solvent in the external reservoir is heated by an infrared lamp. This degases the liquid sufficiently to prevent air from coming out of solution in the column and creating artifacts in the chromatogram. At the normal mode of operation, neither of the three-way solenoid valves is energized. The air pressure, which is now connected to *B*, is transmitted through the mercury to the solvent in *A*. There is vacuum on *D*, and *C* is being refilled from the external solvent reservoir. Mercury rises in *A* as solvent is delivered to the chromatographic column. As soon as the mercury in Vessel *A* comes in contact with the upper electrode, the solenoids are energized. *B* is now connected to atmospheric pressure or to a regulated pressure somewhat lower than the operating air pressure in *D*. The latter is the desired mode of operation at high pressures. The air pressure, which is now on *D*, refills *A* from *C* and, at the same time, keeps up the uninterrupted delivery of the solvent at the

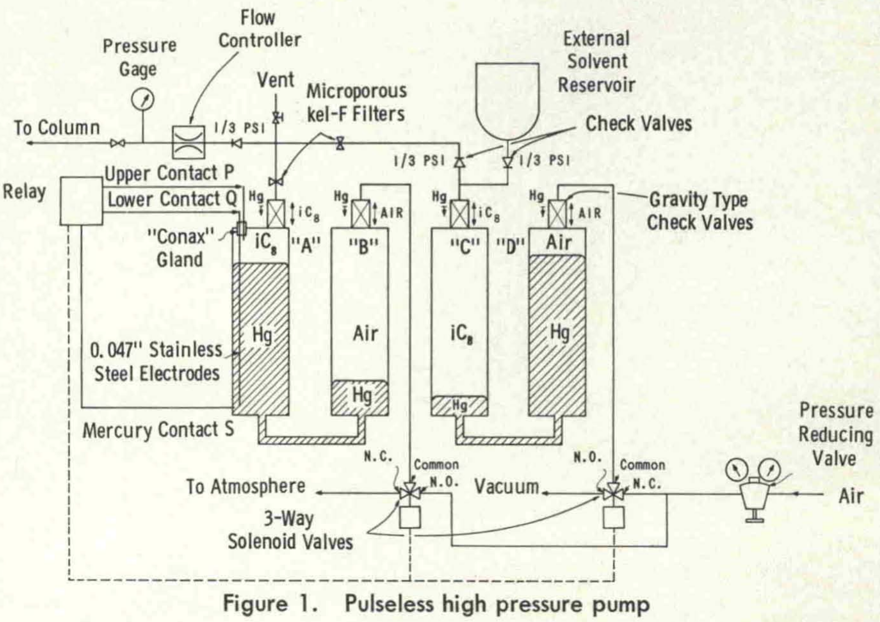

Figure 1. Pulseless high pressure pump

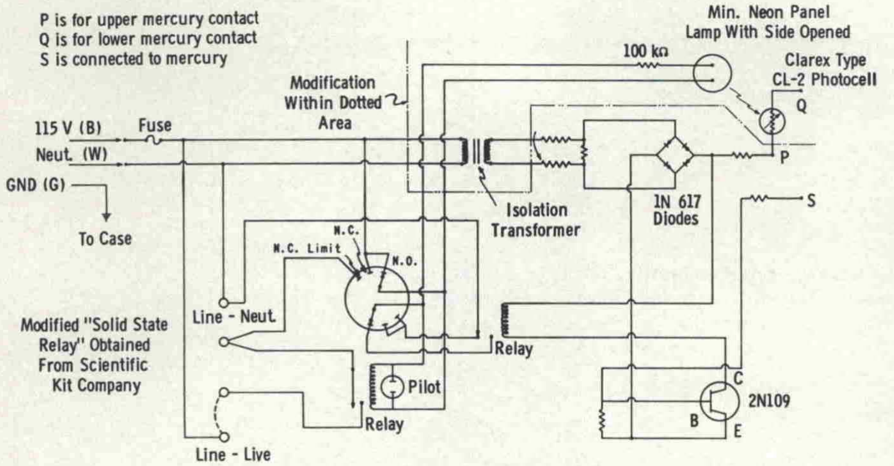

Figure 2. Wiring diagram of relay

adjusted pressure to the chromatographic system. As soon as the mercury in A drops below the lower electrode, the solenoids are de-energized, and the pump assumes operation at the normal mode.

Spring-loaded check valves in the solvent lines ensure the correct direction of flow. Gravity-type check valves above the cylinders prevent mercury from getting out but pass solvents in both directions. We have found it necessary to introduce an impedance to flow between the output lines and the solvent reservoirs, among others, to prevent these check valves from slamming shut during the change between the cycles. Microporous disks of Kel-F have been found satisfactory for this purpose. Figure 1 shows the locations of these disks.

The electrodes in A are 0.047-inch stainless steel rods which are mounted through a modified Conax packing gland assembly which is sealed at the base with epoxy cement. To provide for the cycling between the two mercury levels in A, we have modified a commercially available "Solid State Relay," obtained from Scientific Kit Co., so the relay can be locked up. This alteration is required because the initial electrical contact, which starts the refilling of A, is broken as soon as the refilling operation is initiated. The wiring diagram is given in Figure 2.

As the solvent in C should account for both the filling of A and the delivery to the chromatographic column during the time of filling, the displacement in this vessel should be larger than the amount displaced in A between the two electrodes. For the stated dimensions, flows up to 100 ml. per minute have been attained. For larger capacities, C and D should be larger than indicated. No major problems should be encountered during scaling up. The output pressure will vary with the heights of the mercury levels. To keep this variation negligible in relation to the output pressure, increase in size should preferably be found in increasing the reservoir diameter instead of the length. The limit is reached when the pressure rating of the vessel falls below the desired operating pressure.

The 2-inch stainless steel nipples are rated at 3000 p.s.i. All the Swagelok connections used throughout the pump and the thick-walled $^1/_4$-inch stainless steel tubing have ratings far in excess of this number. The maximum operating pressure is determined by the accessory equipment—i.e., the solenoids and the flow controller. Commercially available items are rated up to 250–500 p.s.i.; and to go to higher pressures, specially constructed equipment may be necessary. It is, however, not difficult to achieve operating pressures up to 1000 p.s.i. In comparison, a piston pump with the equivalent rating would have to be much heavier in construction.

Operation of the pump has been carried out at pressures up to 1000 p.s.i. and flow rates up to 100 ml. per minute with no discernible changes in output flow or output pressure during the different cycles of the pump.

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