Waters
Lab Highlights
An Internal Communication of Applications and Techniques
LAH 0437 6/90
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The 150CV GPC/Viscometer System
II. System Overview

This is the second in a series of Lab Highlights which will describe the operation and performance of the 150CV GPC/Viscometer System[1].

The block diagram (Figure 1) of the 150CV system indicates the key elements that constitute the viscometer detector. The internal solvent reservoir (rarely used in the 150C) has been eliminated and solvent is supplied from an external source to the main pumping system (pump compartment) and the total system pressure transducer (PT) in standard fashion. In a regular 150C, the outlet of this pressure transducer is routed to the injector compartment. However, the viscometer detector is sensitive enough to detect the minor flow fluctuations due to the piston crossover in a properly operating dual-piston pump. In order to successfully use the detector as a viscometer, we have included a pulse dampening system, or baseline optimization box (BOB), to reduce the fluctuations by a factor of more than 100. Since there is not enough space to locate this dampening system in the injector compartment, the dampeners are located in the pump compartment on the drawer just in front of the main pumping system. The baseline optimization box consists of a series of eight dampening and eight restrictive elements in alternating order and it is enclosed in a dual-wall container so as to minimize the effects of any temperature variations. The flow from the system pressure transducer is routed back into the pump compartment, through the BOB, and then to the injector.

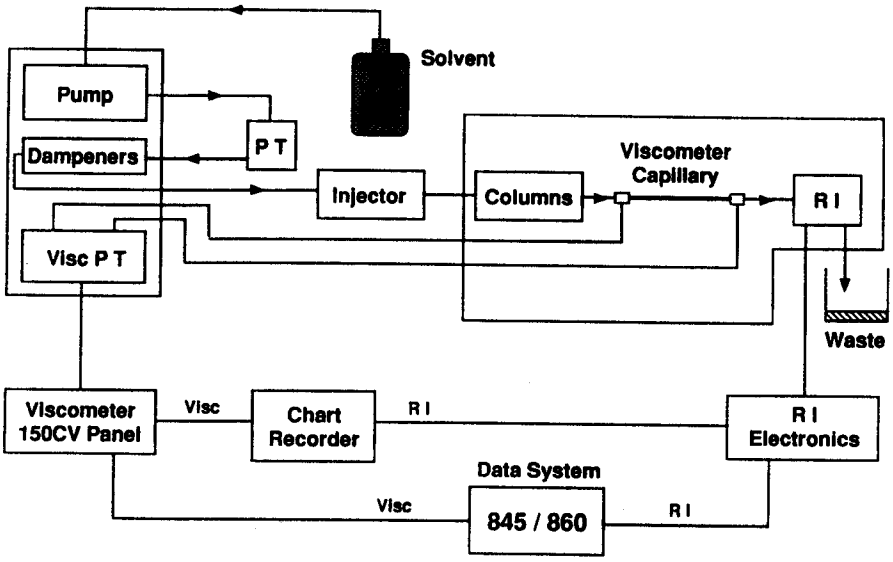

Figure 1. Block diagram of the major components within the 150CV System

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From the injector, the solvent flows into the columns. In the 150CV, the outlet of the last column, which is connected to the inlet of the RI detector in a regular 150C system, is instead connected first to the viscometer capillary and then to the inlet of the RI detector. The viscometer capillary is a 6 inch length of stainless steel tubing (1/16" O.D., 0.014" I.D.). The columns and viscometer capillary are both enclosed in a dual-wall container to isolate them from any temperature fluctuations in the column compartment. This container is located between the refractometer (at the front) and the heating elements (at the rear) and can easily accommodate up to six (6) 30 cm long GPC columns. From tees at either end of the viscometer capillary, connecting tubing (stainless steel, 1/16" O.D., 0.020" I.D.) is routed into the pump compartment and connected to each side of a differential pressure transducer (Visc PT). Under normal operation, the solvent in these connectors is stationary. By opening outlet fittings at each side of the transducer, these lines can be flushed as needed to remove air or to change solvents. This viscometer transducer is also enclosed in a dual-wall container to insulate it from the effects of any temperature variations and the complete transducer assembly is located on the top of the dampening system (BOB).

The outlet of the RI detector is directed into a modified internal waste container in the pump compartment, just below the column compartment. The waste valve on the older container has been removed and the effluent is continually drained to external waste. The output signal from the viscometer and RI detectors is displayed on the electronics control panel for each detector. The viscometer electronics panel is located in the lower left corner of the front of the 150CV in place of the blank panel (150C) that had been reserved for a future detector. The chromatographic output of each detector can be plotted on a chart recorder to monitor system performance and collected on a data system (Waters 845 or 860) which can be used to calculate sample parameters.

The full-scale range of the differential pressure transducer used in the viscometer detector is 5 kilopascals (KPA) or 0.73 psi. At a flow rate of 1 ml/min with THF at 35°C, the pressure drop across the capillary due to pure solvent (Po) is ~50% of full scale, i.e. ~ 2.50 KPA. During elution of a polymer under typical chromatographic conditions (e.g. Dow 1683 polystyrene, Mw = 250K, 0.1% solution, injecting 100 µl per 30cm column), this signal increases by a maximum of ~1% of the background signal (Po) and then returns to its initial value after sample elution is complete. The transducer is activated by a signal from the viscometer electronics panel and generates a 0-5 volt output signal corresponding to a differential pressure across the viscometer capillary of 0-5 KPA. This transducer output is displayed on the viscometer electronics panel. A safety circuit has also been incorporated into the viscometer electronics to trigger alarms (audible and visual) and stop the pump if a maximum pressure drop (operator selectable) is reached or exceeded.

In summary, the viscometer detector has been completely integrated within the chromatographic system and, except for the 150CV nameplate, the only external evidence that a viscometer detector is included in the 150CV is the viscometer electronics control panel in the lower left corner on the front of the instrument. This system integration is especially important when operating at high temperature since it eliminates the need for additional temperature-controlled zones that would be required if the viscometer were located in an external module.

References

1. LAH 0436 6/90.