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Liquid Chromatography Polymer Testing *using the DaqBook*

Materials Development

Application Note #86

Application Summary

Researchers are continually generating new molecular structures to customize polymer materials according to customer performance requirements. One key step in the process involves determining the material's molecular weight distribution. Molecular weight affects the viscosity of the molten polymer during processing steps such as extrusion or injection molding, and performance parameters such as brittleness and toughness of the resulting articles.

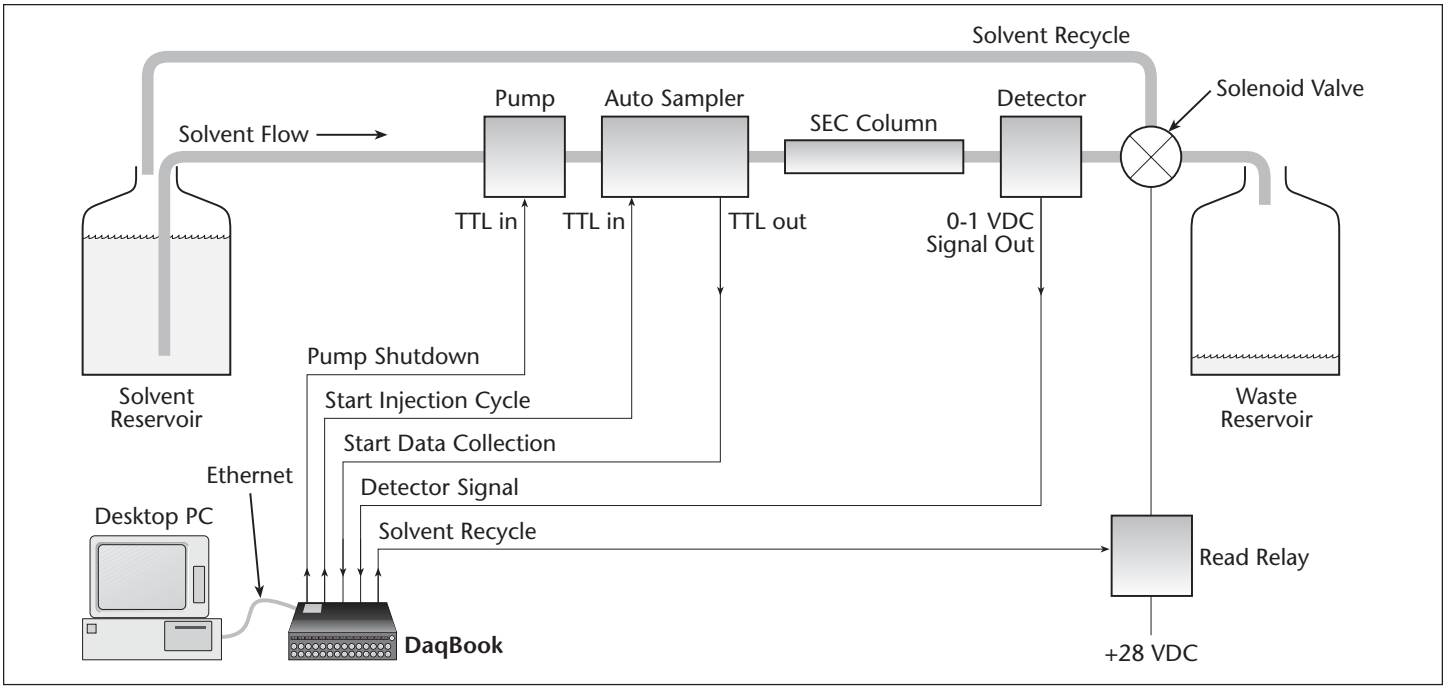
Molecular weight distribution measurements are needed here because polymer molecules are different than the molecules in many other ordinary materials and chemicals. For ordinary compounds, all of the molecules are identical, with the same molecular weight and unique properties such as melting and boiling points, and vapor pressure. Changing the chemical structure, and thus molecular weight, produces an entirely new compound with different properties.

But polymers are different. Polymer molecules consist of a number of smaller molecules, or monomers, reacted together end-to-end to form a chain of repeat units. A polymer sample contains a mixture of molecules, with molecular weights differing by even numbers of repeat units. The molecular weights of individual chains in a statistical sample typically follow a bell curve (or another) distribution.

The test setup for determining the distribution requires a chromatography system and a means to automatically run a large number of samples. The test system controls pumps, solenoid valves, and an automatic sampler while simultaneously collecting the data from the output of the chromatograph detector and sending it to a desktop computer. The controller and data collection system must be highly reliable, flexible, easy to set up, and adaptable to a wide variety of instrument configurations.

Potential Solution

George Caflisch is a Senior Research Associate at Eastman Chemical Co., Kingsport. Tenn., and was recently given the task to upgrade the existing data acquisition and control system in his lab. The commercial control and monitor system was aging and increasingly suffering failures. Replacing it with a newer version of the same type was not appealing because Caflisch really needed a more flexible system that he could better customize the data acquisition portion and interface it with a sample submission and tracking



The SEC chromatography system used by Eastman Chemical Company in their Kingsport, Tenn., facility for developing new polymers includes an IOtech DaqBook for data acquisition and control. The DaqBook TTL output circuits control the system's solvent pump, automatic sampler, and solvent recycler. The DaqBook also connects to a personal computer via an Ethernet connection, and monitors the chromatography detector output signal, which is proportional to the concentration of polymer molecules over time. The data is stored and used to construct a graph of molecular weight distributions of the polymer in order to determine its properties.

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system. In addition, he needed to improve the method of operating the on/off functions of the system.

IOtech's Solution

After some investigation, Caflisch decided to use an IOtech DaqBook®. The test procedure that Caflisch uses is called size-exclusion chromatography, SEC. Each chromatograph system consists of a DaqBook connected to a computer through an Ethernet connection, a pump, an automatic sampler, a set of size-exclusion columns, and a differential refractive index or UV absorbance detector (or both). The DaqBook fits his application much better than the existing commercial unit because it can handle the chromatography system detector's one-volt analog signal directly, and the DaqBook's TTL inputs and outputs adapt handily to controlling the pumps, valves, and automatic sampler.

"I was able to program the system to load a list of samples to be run, command the DaqBook to send a TTL-level signal to the automatic sampler to execute the programmed injection sequence, and monitor the signal in the ADC while the sample is going through," says Caflisch. "The IOtech TTL logic circuits also operate a solenoid valve that recycles the solvent back to the reservoir when polymer is not eluting from the column, and shut the system down when all of the samples have run." The detector signal changes slowly enough that it is sufficient to record only one point per second. In order to eliminate any 60-Hz noise that might enter the system the ADC is scanned at 1000 Hz and each data point is averaged over sixteen scans. Caflisch uses two eight-channel DaqBooks, each of which controls four chromatographs.

Each polymer sample to be analyzed is first dissolved and this solution is loaded into the automatic sampler, which injects a small amount of it into the flowing solvent stream of the chromatograph on command. It then passes through an SEC column, a tube filled with beads of cross-linked polystyrene. The beads contain small pores of various sizes in a range similar to the dimensions of the polymer molecules in solution. On their journey from input to output, the polymer molecules diffuse into and out of the pores. Because the smaller molecules can fit into more of the pores, they spend relatively more time there and thus travel more slowly through the column than the larger ones. Therefore, the polymer molecules are separated by molecular weight, with the larger ones eluting from the column before the smaller. The detector signal is proportional to the concentration of polymer coming out at a given time so the DaqBook can plot the concentration of polymer molecules exiting vs. time. A calibration curve is used to calculate the molecular weight from elution time and convert the time-base plot into molecular weight on the x-axis and the mass fraction with a particular weight on the y-axis.

Conclusion

Eastman Chemical Company, Kingsport, Tenn., a developer of new and custom polymers, uses a DaqBook to control a size-exclusion chromatography system and measure its detector output data to ultimately plot a distribution of molecular weights. The DaqBook's TTL input and output circuits control valves, start injection cycles and receive commands to start and stop the data collection function.

DaqBook/2000 Series

The DaqBook/2000® series of portable data acquisition devices can synchronously measure analog inputs, frequency inputs, and digital inputs. The 16-bit/200-kHz DaqBooks come equipped with built-in signal I/O capability, which can be further expanded and enhanced with over 40 DBK series expansion and signal conditioning options.

The DaqBook/2000 series includes a built-in 10/100BaseT Ethernet interface capable of transferring acquired data back to the PC at the full 200 Kreading/s measurement rate of the DaqBook. Multiple DaqBooks can be attached to a single PC via an Ethernet hub or switch, and are capable of being synchronized and of transferring data continuously at full speed into the PC. Up to 10 DaqBooks can be transferring 200 Kreading/s back to a PC concurrently, with no loss in data.

Features

- Analog input, analog output, frequency input, timer output, and digital I/O; all in one compact and portable enclosure
- Built-in Ethernet connection provides continuous streaming to the PC with no data loss
- 16-bit, 200-kHz A/D converter
- Operates from -30˚ to +70˚C
- Powerable from 10 to 30 VDC, or with included AC adapter
- Synchronous analog, digital, and frequency measurements
- Trigger modes include analog, digital, frequency, and software
- Virtually infinite pre-trigger buffer
- 4 channels of 16-bit, 100-kHz analog output (models /2001 and /2020)
- DaqBook/2020 offers convenient front panel connectors for thermocouple, voltage and frequency measurements all in one box
- DaqBooks attach to over 40 DBK signal conditioning options to assemble a low-cost system, customized to your particular application
- Signal conditioning and expansion options for thermocouples, strain gages, accelerometers, isolation, RTDs, etc. — over 40 DBK I/O expansion options are available
- DaqView™ software included for effortless data logging;
- Includes support for Visual Basic®, C/C++, ActiveX/COM, LabVIEW®, MATLAB®, and DASYLab®