PD2040 High Temperature GPC Laser Light Scattering Detector

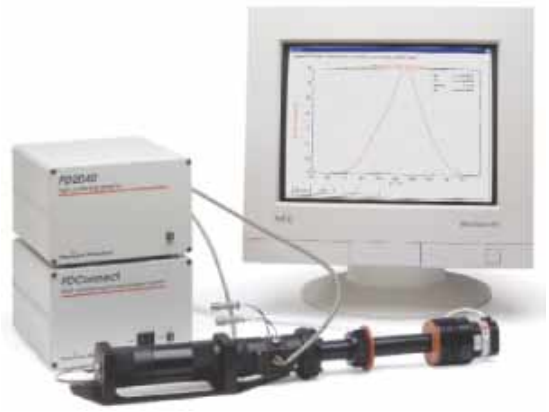
The PD2000 family of high performance molecular characterization detectors, measure absolute values of molecular weight and size. Typical applications are polymers, proteins, antibodies, polysaccharrides and other macromolecules used in the plastics, biotechnology, pharmaceutical and food industries. These laser light scattering detectors are easily added to any HPLC/GPC/SEC system and are ideal research, quality control and process monitoring tools. Available in single, dual and high temperature versions, the PD2000 Series provides cost effective critical path data for any macromolecular characterization application. The PD2040 High Temperature GPC Laser Light Scattering Detector is specifically engineered to provide high performance multi-angle measurements for polymers that are soluble at temperatures ranging from room temperature to 250 degrees C. The PD2040 couples to any commercially available high temperature GPC system (such as those from Polymer Laboratories and Waters Corporation) by direct placement of the light scattering cell inside the system oven compartment and laser outside the oven using a patented coupling design.

Introduction

The PD2040 High Temperature GPC Laser Light Scattering Detector is designed to provide absolute molecular weight distributions (Mw, Mn, Mz etc.), radius of gyration (Rg) and branching data for polymers that are soluble only at elevated temperatures. The unit is installed inside the oven compartment of commercially available high temperature GPC systems and operates from room temperature to 250 degrees C. The PD2040 is used in conjunction with Precision Detectors?new Discovery32?software. The system provides absolute molecular weight distributions from less than 10^3 to over 10^7 daltons and Rg in the 12 nm to 150 nm range.

Key Performance Features for the PD2040

- Unique alloy constructed axial platform with high performance 15 degree low angle operates inside high temperature GPC ovens to couple directly to GPC columns and other detectors. This minimizes inter-detector volumes for accurate data and insures sample integrity at GPC system temperature.
- Operates from room temperature to 250 degrees C and is compatible with typical GPC solvents for high temperature applications.
- 10 ul flow cell minimizes sample band-broadening and creates laminar flow within the cell.
- Cell design and focused laser beam (to 0.01 ul) eliminates stray light sources and minimizes deleterious GPC system effects (mobile phase and column particulates).
- Shielded fiber optic provides high efficiency transfer of detector signals to the electronics module located outside the GPC system.
- Discovery32?software provides an easy to use analyst interface with the system for acquiring, analyzing and reporting GPC data.



The PD2040 High Temperature GPC Laser Light Scattering Detector with Discovery32?software provides absolute molecular weight distributions and radius of gyration measurements for polymers soluble at temperatures up to 250 degrees C.

Designed for Unparalleled Sensitivity and Stability for Room Temperature to 250 degrees C GPC Environments

The PD2040 platform design is optimized for direct high temperature measurements within the column oven compartments of modern GPC systems. The patented axial optical platform is constructed of specialty alloys that provide high sensitivity and stability of the laser light scattering signals under the thermal stresses present in GPC systems up to 250 degrees C. These materials of construction and the ultra-small 10 ul flow cell provide close coupling of the laser light scattering platform to the GPC columns and other detectors (eg. refractive index and viscometer), providing absolute minimum inter-detector volumes for accurate data. The detector incorporates two fixed angle detectors at 90 degrees and a high-performance low angle of 15 degrees. The annular radius of the 15 degree scattering is collected via a mushroom lens. This unique optical system provides unparalleled signal-to-noise ratio at low angle to provide accurate molecular weight data when the polymer exhibits non-isotropic scattering. The scattering signals are collected via fiber optics to the electronics module located outside and transmitted to the GPC oven. The signal-to-noise ratio of each detector is optimized by focusing the laser beam to an optical volume of only 0.01 ul within the 10 ul flow cell. This minimizes the probability of any on-sample scattering? such as a particle from the mobile phase or column shedding, from entering the beam. By collecting data at a rapid rate (100 points/second) it is now possible to apply noise rejection algorithms on Precision Detectors?proprietary DSP-based data-collection board, producing high quality signals at averaged collection intervals-even below 1 point per second. The optical efficiency of the cell is also critical to the measurement. The PD2040 cell design, uses separate windows for collection in a matte black coated cell and unique eam dump? This virtually eliminates stray light from entering the collection optics thus minimizing the signal-to-noise ratio. This is very important as many solvents used in high temperature applications (eg. 1,2,4 trichlorobenzene) have an intrinsic Rayleigh scattering intensity almost 30 times that of water and 8 times that of tetrahydrofuran. The key issue here is to have the detectors respond only to signals from the 0.01 ul optical volume produced by the sample in solution and not from any stray light source.

Waters Alliance GPC 2000 Series

Polymer Laboratories, Inc. GPC 220

The PD2040 High Temperature Laser Light Scattering Detector is easily configured inside the column oven of commercially available high temperature GPC systems. Shown here are the Waters Alliance GPC 2000 Series and Polymer Labs GPC 220.

Metallocene Catalyzed Polyolefin Analysis at High Temperature Using PD2040 and Discovery32 Software

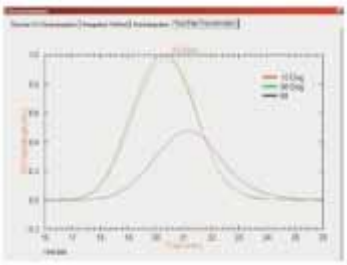


Figure 1: Laser light scattering and refractive index chromatograms show excellent sensitivity and stability at elevated temperatures.

Mw vs Elution Time

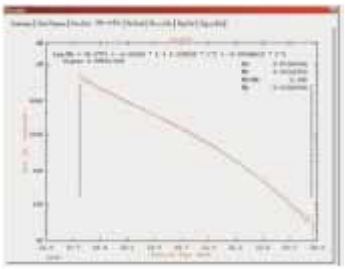


Figure 2: Discovery32 plot of molecular weight vs. retention time with corresponding calculation of Mw, Mn, polydispersity and Mp (99.65kD, 41.13kD, 2.423 and 92.26kD).

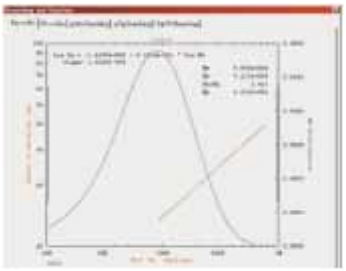
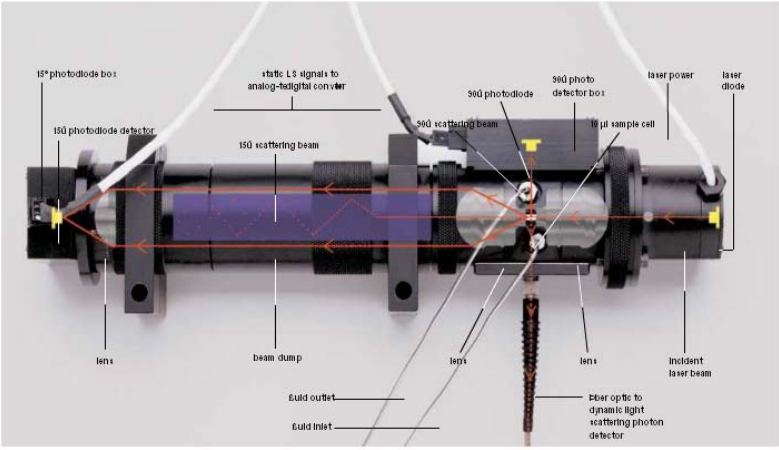


Figure 3: Plot of Radius of gyration (Rg) vs. Mw yields branching data. The exponent of 0.512 indicates the degree of branching. A linear polyolefin would have a value of 0.56.



PD2040 high-temperature light scattering platform ?cut-away image showing laser beam and scattering signal paths excluding the extension/inner and heat sink.

Light scattering technology

Light scattering technology has become a popular detection technique in both the lab and plant environment because the answers achieved are absolute and not based on analysis by comparison with calibration curves. PDI's modern designs are popular because they are convenient to use and very stable. The science behind light scattering was first described by Lord Rayleigh at the end of the 19th century but it was not until the 1970s, when polarized lasers became readily available that light scattering technology was applied to practical instruments for laboratory use. At that time, a member of our design team was applying static and dynamic light scattering to instrument designs and was awarded some of the first patents in the field. Today, the analysis of both static and dynamic light scattering signals is well understood. High-performance diode lasers, high-speed digital signal processors (DSP) and other electronic devices are readily available at economical prices. Precision Detectors applies these devices and advanced light scattering technology to the design of the most stable and versatile instruments for lab and plant use.

Rayleigh (static) light scattering

When a polarized, monochromatic laser beam passes through a solvent containing molecules or particles, the excess light scattered at an angle θ to the incident beam (I_{LS}) over that scattered by the solvent alone (the base-line) is:

$$I_{LS} = c\, M_w \left(\frac{dn}{dc}\right)^2 P(\theta)\, K(\theta)$$

The science behind light scattering was where c is the concentration of the sample in the solvent, Mw is the molecular weight of the sample, (dn/dc) is the change in refractive index with concentration for the sample/solvent pair, P(θ) is the ratio of the scattered intensity at angle ?to that at angle 0 and K(θ) is the optical constant for detection at angle θ.

A refractometer or other concentration detector following the light scattering detector in the eluant stream detects the concentration signal.

Mw is obtained by dividing the light scattering signal by the concentration signal at each elution slice. The radius of gyration (Rg) is obtained from the Debye function using the dissymmetry method. Precision software also calculates Mn , Mz and other averages. The key characteristic of PDI's Rayleigh scattering detectors is their ability to collect the highest-quality signals at angles of 90?and 15?to the incident laser beam. light scattering detection system is its high sensitivity and its unique ability to operate in flow mode with an SEC system calculating R h at each elution slice after chromatographic separation.

Dynamic (quasi-elastic) light scattering

The diffusion coefficient of particles (or molecules) undergoing Brownian movement is related to their hydrodynamic radius (Rh) as described in the Stokes-Einstein equation:

$$D = \frac{kT}{6\pi\eta R_h}$$

where k is Boltzman's constant, T is the temperature in degrees Kelvin, η is the solvent viscosity and D is the diffusion coefficient. The diffusion coefficient (D) is calculated from the reduction time of the autocorrelation function of the scattered light. Rh is then calculated. The key characteristic of PDI's dynamic light scattering detection system is its high sensitivity and its unique ability to operate in flow mode with an SEC system calculating Rh at each elution slice after chromatographic separation.

Specifications:

Sample Cell Volume	10 µL
Light Scattering Volume	0.01µL
Rayleigh Scattering Angles	90 and 15 degrees (total annular collection)
Molecular Weight Range	1000 to above 10 7 daltons
Radius of Gyration Range	12 to 150 nm
Laser Configuration	30mW at 685 nm (100mW at 809 nm available)
Operating Temperature	ambient to 250 degrees C
Inter-Cell Band-Spreading	Δt less than 1 second
Raw Data Sample Rate	100 per second
Signal Preprocessing	software selectable, on-board particle noise rejection
Signal Smoothing	none, or operator-selectable, Fast Fourier Transform
Electronic Module Size	6.75" width, 8.25" long, x 4.5" high (17.1cm, 21cm 11.4cm)
Electronic Module Weight	5.5 pounds (2.5 kg)
Light Scattering Platform Weight	7 pounds., (3.3 kg)
Power Requirement	30VA, 100-240V, 50-60Hz

Precision Detectors
...the molecular and particle characterization people

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