Absolute molecular weight

Molecular conformation

Molecular size

Solution viscosity

Viscotek
HT-GPC

High temperature GPC for Advanced Polyolefin Characterisation

Malvern

Introducing the Viscotek High Temperature GPC

The Viscotek HT-GPC system brings advanced triple detection to demanding applications to provide:

Absolute Molecular Weight, Polydispersity and Structure

- Absolute molecular weight without extrapolation, correction or column calibration
- Molecular size, intrinsic viscosity, concentration, structure and branching information in a single experiment
- Automated sample preparation including in-line sample filtration
- Innovative triple detector technology for use up to 160ºC

Select from four detector configurations:

Conventional Calibration	RI (refractive index) detection with column calibration
Universal Calibration	RI & viscometer detection for MW plus branching data
Light Scattering	RI & light scattering detection for absolute MW measurement without column calibration
Triple Detection	RI & Viscometer & light scattering for absolute MW measurement and branching data

An advanced system that offers unique benefits

- Automated sample preparation - The Vortex autoprep/autosampler takes care of sample preparation at up to 170°C with no user intervention needed, making the system safe, accurate and productive

- Automated sample filtration - Automation reduces the number of preparation steps as only the sample injected on the column is filtered, which avoids bulk filtration
  The sample filter is automatically back-flushed during analysis – ready for the next sample

- Detection - The flexibility of four different detector configurations; from conventional column calibration up to full triple detection with the unique 7º Low Angle Light Scattering detector (LALS), for absolute molecular weight measurements without column calibration

- Software - intuitive and powerful - The OmniSEC software controls all the functions of the HT-GPC and provides the simplicity of two clicks from data to results but also has the power to handle more complex calculations such as branching analysis

Advanced Polyolefin Characterization

Applications

Polymers
- Polyolefins
- Olefinic copolymers
- Polythiophene
- PEEK
- Delrin
- Nylon and PET
- Polyimides
- Polyacetals
- Modified Starch

Common Solvents
- TCB
- ODCB
- Chlorophenol
- m-Cresol
- DMSO
- DMAc
- DMF
- NMP

Polymer branching

The molecular weight, and molecular weight distribution, intrinsic viscosity and the presence of long chain branching of polyolefins influences physical properties such as melt viscosity and mechanical strength. The capabilities of the Viscotek High Temperature Triple Detection GPC (HT-GPC) system are ideal for characterising these parameters without the need for column calibration.
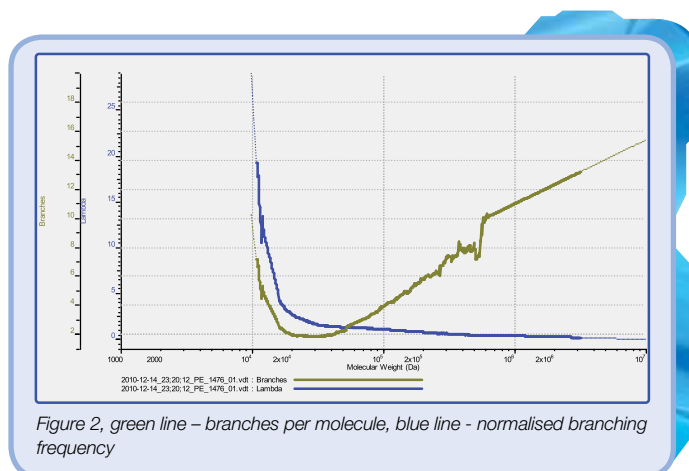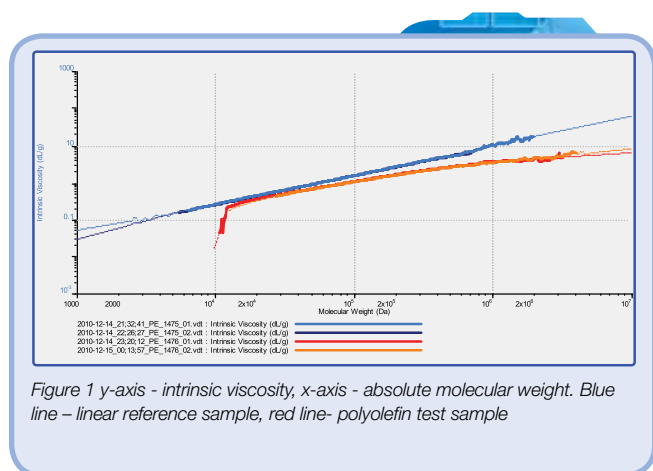

Figure 1 y-axis - intrinsic viscosity, x-axis - absolute molecular weight. Blue line – linear reference sample, red line- polyolefin test sample

Figure 2, green line – branches per molecule, blue line - normalised branching frequency

Figure 1 shows the Mark-Houwink plots for two Polyethylene samples. Sample 1 is a linear reference polymer The plot of sample 2 shows it is branched. At lower molecular weights the plot is parallel to the reference polymer, indicating a linear molecular structure but as molecular weight increases, so does the curvature in the plot indicating an increasing level of branching.

Figure 2 plots an estimate of the number of branches per molecule by applying the Zimm-Stockmayer branching calculations, and shows an average of almost five branches per molecule at low molecular weights for this sample, reaching 8 at the highest molecular weights. The plot normalized to the molecular weight gives an average branching frequency of 6 branches per 10,000 ethylene units.

Automated sample filtering - Carbon filled Polyethylene

Polyethylene pellets filled with approximately 10% carbon black were dissolved in stabilized TCB (1,2,4-Trichlorobenzene) and measured seven times sequentially. After each injection and before entering the GPC column, the sample was filtered automatically to remove the carbon filler. During each measurement, the filter was automatically cleaned by back-flushing, in preparation for the next sample. The whole system, including columns, detectors and inter-detector tubing as well as the filter was maintained at a constant temperature, in this application, at 150 °C.

Injection	Sample Id	Mw	IV	Rh	Recovery %
1	Carbon PE	141,759	0.95	11.27	89.28
2	Carbon PE	141,757	0.94	11.38	88.83
3	Carbon PE	140,812	0.93	11.22	88.79
4	Carbon PE	140,959	0.91	11.15	89.35
5	Carbon PE	140,947	0.94	11.27	89.16
6	Carbon PE	140,429	0.94	11.17	89.17
7	Carbon PE	141,650	0.94	11.27	88.51

The table demonstrates the excellent repeatability of the molecular weight, and the consistent recovery shows the reliability of the automated filtering process.

Advanced Polyolefin Characterization

HT-GPC module

The Viscotek high temperature GPC module provides a temperature controlled environment for the columns, injector and up to three detectors for the most comprehensive and accurate macromolecular characterization.

The module can be configured with:

- Up to 7 analytical columns
- HT-RI, for high temperature dn/dc or concentration measurement
- HT-Viscometer, for viscosity, shape, branching and Mark-Houwink plot
- HT-LS with Low and Right Angle Light Scattering (LALS & RALS)

Pump

- Dual piston optimized for HT-GPC
- Extremely low pulsation and stable eluent flow, to improve detector performance
- Unique user programmable soft start/stop to maximize the lifetime of columns
- Compatible with any mobile phase used for HT-GPC

Vortex module

- Autoprep/autosampler for sample preparation at up to 170°C
- No user intervention needed, improving safety accuracy and productivity
- Handles up to 30 vials of 20 or 40 mL
- Automatic wash after each injection avoids sample cross-contamination

HT-RI

Accurate measurement of the polymer concentration is essential for good molecular weight and structure information.

- Designed for minimal thermal drift, to optimise concentration and dn/dc measurements
- Light source is wavelength matched with the light scattering detector for accurate MW measurements
- Continuously flowing reference to reduce equilibration time and increase stability
- Unique design allows the RI to be placed in series between the light scattering and viscometer for full signal on all detectors

Viscotek HT-GPC

HT-Viscometer

The high temperature differential viscometer detector provides a measurement of intrinsic viscosity and allows for the determination of molecular size and branching structure.

- Over 25 years experience of HT-viscometer design and branching calculations
- High sensitivity, unrivalled baseline stability and the widest application flexibility
- Digital Inert Transducer (DIT) technology for faster, more sensitive response
- Unrivalled chemical compatibility enables use with any GPC solvent

HT-LS (LALS and RALS) Dual light scattering detectors

- Unique 7° Low Angle (LALS) detector measures the absolute MW directly without data fitting, extrapolation or correction
- 90° Right Angle (RALS) detector provides the best signal to noise ratio and the highest sensitivity for small molecules
- The OmniSEC software automatically selects the appropriate detector data to maximise the accuracy of MW even for mixtures of large and small molecules
- Flow cell volume of just 18µL ensures minimal band broadening

Columns and Standards

- Choose from a selection of columns designed for your molecular weight range to increase separation efficiency
- Standards are available for triple detection, universal and conventional calibration

Aftersales Support

The quality of Malvern's aftersales support is one of the key reasons for our continuing success in today's demanding laboratory and process markets.

- Access to a worldwide network of highly trained and experienced teams of technical support professionals
- Ensures systems are operating and used at optimum efficiency

OmniSEC software

The software is designed to give powerful yet easy to use instrument control, data acquisition, analysis and reporting. It is at the heart of the HT-GPC providing an integrated solution that helps maximize the information and efficiency of your experiments. Simply load your samples, and the software controls high temperature heating, stirring and sequencing, enabling truly automatic unattended operation.

OmniSEC provides support for:

- Multiple detectors (Single/Dual/Triple)
- Absolute molecular weight
- Molecular conformation/branching
- Intrinsic viscosity
- Universal calibration
- Conventional calibration
- Mark-Houwink plot

OmniSEC contains many features to improve productivity and convenience:

- Two clicks from raw data to results
- Manual or fully automatic baseline setting
- Automatic determination of dn/dc, concentration, recovery
- Proven asymmetric band broadening corrections
- Fully automatic process control/LIMS mode
- Interactive branching view based on Zimm Stockmayer
- Built-in, intuitive report designer
- Full export capability of raw data, results and intermediate calculations
- Integrated help system
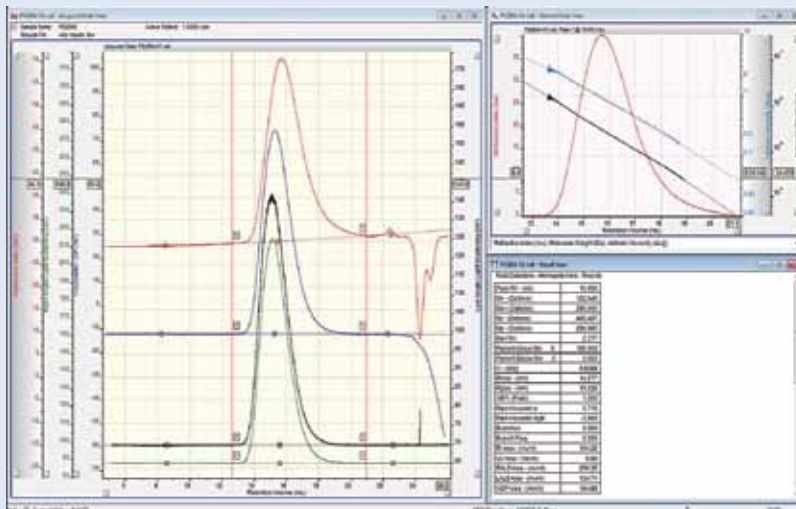
With automatic peak and baseline setting, accurate and robust results are calculated in two clicks of the mouse

Viscotek HT-GPC

The Viscotek HT-GPC delivers

You asked for:	We give you:
High Temperature GPC System	A complete modular high temperature system designed specifically for polyolefins but applicable to other polymers soluble only at high temperature
System automation	Full instrument automation for unattended operation, including in-line sample filtration to improve precision, save time and promote operator safety
Absolute Molecular Weight	A low angle light scattering detector, (LALS) measuring absolute molecular weight without assumptions, corrections or extrapolation for robust and reliable results
Intrinsic Viscosity	A sensitive differential bridge viscometer with DIT technology resistant to aggressive solvents for the measurement of intrinsic viscosity and branching
Molecular Conformation	Triple detection to give advanced information on structure, conformation, aggregation, branching, copolymer composition and hydrodynamic radius (Rh) to less than 1 nm
Easy to use, yet powerful software	Software that puts you in control, with calibration and manual or automated analysis to suit your requirements
Support	The Malvern worldwide support structure, application notes, on-demand presentations, and live help desk

Specifications

Oven/Detector module

Temperature control	30ºC to 160ºC all detector cells and columns
Detector configuration	All detector cells in series
Column capacity	Up to 7 standard 30cm GPC/SEC columns
Interface	6 channel digital interface to OmniSEC software
Inputs	2 analog inputs for auxillary detectors

Light scattering detector:

Measuring principle	RALS, 90º angle; LALS, 7º angle, with high efficiency optics
Light Source	Temperature controlled laser diode, 10mW, 660nm
Cell	18µL
Data rate	100Hz collection, DSP with 5Hz to OmniSEC

Refractive index detector:

Measuring principle	Deflection
Light Source	LED, 660nm
Cell	45° quartz glass, 12µL volume
Purging	Continuous dual flow
Data rate	100Hz collection, DSP with 5Hz to OmniSEC

Viscometer detector:

Measuring principle	4 capillary differential Wheatstone bridge configuration
Transducers	Digital Inert Transducer technology with over-pressure protection
Measuring volume	18µL
Purging	3 modes, manual, automatic and programmable
Data rate	100Hz collection, DSP with 5Hz to OmniSEC

Sample/Solvent modules

Vortex Autosampler

Capacity	30 vials, 40mL, (20mL option)
Temperature range	30ºC to 170ºC
Srirring speed	Variable, 0 to 300 rpm
Sample Filtration	Automatic self-cleaning in-line sample filtration
Sample Preparation	Optional semi-automated sample preparation

Degasser

Channels/volume	Two channels, 8mL per channel
Performance	<0.5ppm oxygen at 0.5mL/min

Pump

Flow rate range	0.01 to 9.99mL/min
Pulsation	Less than 1% (measured with viscometer)
Pressure Reading	MPa or PSI
Soft start/stop	User programmable in mL/min/min

OmniSEC software

Acquisition	6 channels, 5Hz, unlimited acquisition time
Calculation modules	Conventional, Universal, Light Scattering, Triple, Copolymer, FIPA
Results output	Mn, Mw, Mz, Mp, Mw/Mn, IV η, Rh, Rg, Branching Number, Branching Frequency, Number of Arms, Weight Fraction, Concentration, MH-a, MH-k, dn/dc, dA/dc. A2
Minimum PC specification	Windows XP or higher, 100 GB HD, 2GB RAM, 1.8 GHz processor, Graphics 1024x768, 16-bit colour depth

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detailed specifications at www.malvern.com/viscotek

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