March 2005

A Joint publication of PSS Gmbh, Germany and PSS USA.

GPC Streamliner

Focus: Streamline Lab. Operations

PSS present here a variety of solutions that will streamline your Lab. operations and address the common problems of analytical laboratories: more samples to test lesser staff and increased pressure to achieve efficient workflows. This issue's topics pursue three objectives:

1 Speed up the separations to reduce analysis costs
2 Escalate the amount of information acquired by combining different methods, and
3 Restructure the laboratory to expand the capacity of existing resources

Integrate all polymer characterization methods into one GPC software user interface WinGPC Unity (pages 1 and 2).
Learn about optimum GPC Systems in the discussions of GPC - Tips and Trick. This section will provide useful hints to ease your daily laboratory work

Obtain absolute Molecular Weights as well as additional structural information like radius of gyration using the Multi-Angle Light Scattering Detector SLD 7000, used for proteins page 3.

Find out on page 4, when linear columns are more appropriate than HighSpeed columns, as you read about optimum Column sets.

PSS headquarters offers courses, seminars and in-house training.

WinGPC Unity

Controlled Growth - Selective Upgrading

WinGPC Unity is the first macromolecular chromatography data system (MCDS) to integrate all fields of polymer liquid chromatography into one consistent user interface, thereby simplifying logical and transparent access to your experimental data. WinGPC Unity software modules allow producer-independent data recording with all kinds of detectors, to ease seamless integration of new functionality into your habitual software environment.
You can selectively upgrade your system to add the appropriate detection modules. With such controlled growth you assuredly elucidate any and all property distributions.

Copolymer Characterization

Block copolymers are often analyzed with GPC, which separates according to hydrodynamic volume, as well as HPLC. Under the right analytical conditions, copolymers are separated according to their composition. Using two detectors with different comonomer response instead of only one detector, the composition of the copolymer is measured by combination of the two

» Continues on Page 2

In This Issue

Feature

WinGPC Unity Brochure Available

WinGPC Unity

» From Page 1

detector signals. WinGPC Unity has two .modules "Copolymer" or "Chemical heterogeneity" that allow the characterization of Copolymers.

Product De-formulation

When the composition of samples is highly complex, WinGPC Unity allows the combination of different separation methods for their characterization. The combination of methods increases the peak capacity, making the separation more efficient, compared to using only a single separation method.

The Figure below displays a 2D plot, a projection on two single separation methods (HPLC and GPC). It demonstrates the power of 2D chromatography, showing well-resolved peaks the single methods alone cannot. Calibration of both systems allows the quantification and molar mass determination of all compounds.

WinGPC Unity offers other modules for investigation of high-performance products:

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- Light Scattering and Viscosity modules for characterization of branched samples
- End Group Analysis module for determination of end group functionality or heparin quality (according to DAB).
- Identification and quantification of additives or low molecular substances with WinGPC Unity in HPLC mode.

Advanced literature

D. Held: New "Unity" in GPC; LaborPraxis 5/04 (in English)

P. Kilz, D. Held: Recording and Processing Chromatographic Data; LaborPraxis 9/02 (in English)

D. Held: Software Solutions for the GPC Analysis; GIT Separation 1/02 (in English)

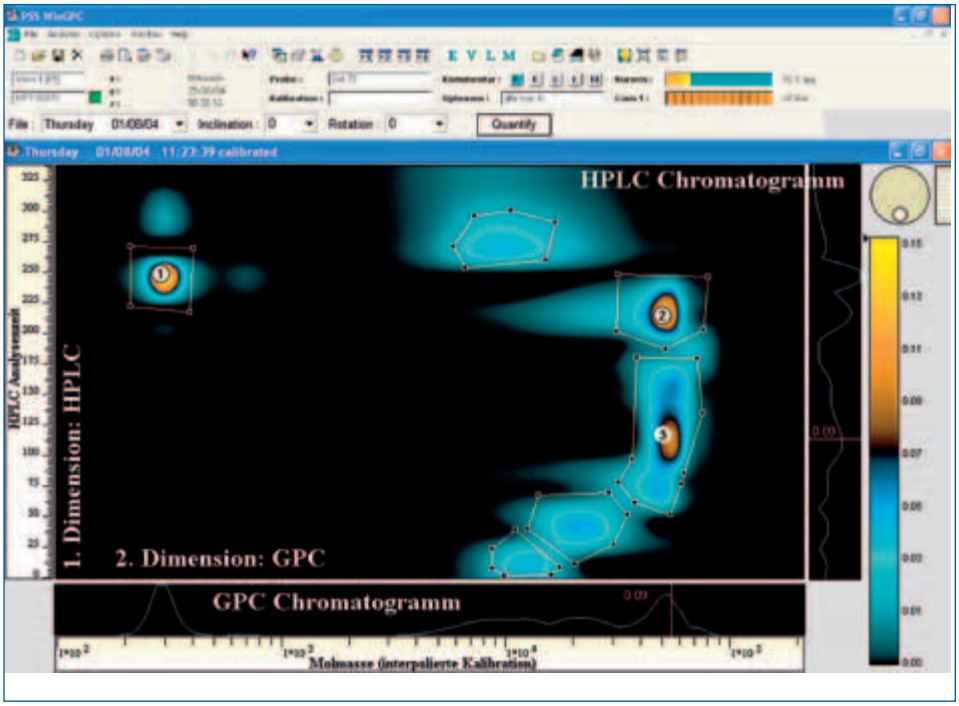

***Figure**: WinGPC Unity 2D chromatography module: GPC separation only: coelution of Peak 2 and 3; HPLC separation only: peak 1 and 2 not separated. With 2D: all peaks are well resolved*

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Announcement

New Staff at Mainz

Begining April 2004 Martina Adler joined our WinGPC Unity software team Her specialties are 2D chromatography and coupled GPC Methods (light scattering, viscometry and FTIR).

Price list and Part numbers

Begining October 2004 the product part numbers and prices for columns and polymer standards were updated. Current information is available at http://www.polymer.de.

Seminar offered in February Integrated Solutions for Lab. Efficiency

This February, PSS offered the seminar participants a platform to discuss current trends in liquid chromatography and strategies for implementing new methods and technologies. One goal was to create a forum where information about problems and solutions can be exchanged.

The seminar had lecture and workshop modules. New trends and developments in liquid chromatography were presented in a series of lectures by our special guest speaker Dr. Harald Pasch, Chief of the analytical department at the "Deutschen Kunststoff Institut" in Darmstadt, Germany.

Topics:

- Better understanding of molecular structures with modern chromatographic methods
- Strategies and approaches for introducing time optimized analysis in laboratories
- Selection, evaluation and optimization of polymer characterization methods
- Usage of hidden synergistics and existing structures for effective task management

The workshops discussed topics like column and eluent selection and the targeted analysis of copolymers and branched products

Light Scattering

For Protein Aggregates

Multi-angle laser light scattering (MALLS) is a powerful absolute characterization method for polymers and biopolymers, providing important information like weight average molar mass M_W, M_Z-average radius of gyration $<R_g>z$ and the second virial coefficient A_2. Integration of a MALLS detector into a GPC system makes additional distributional information available, giving access to M_n, M_W, M_Z and the distribution of R_g. GPC sample preparation is also less tedious than sample preparation for batch measurements, making this method very attractive for samples like proteins and viruses.

The PSS SLD 7000 fulfills all of these requirements and can easily be integrated into an existing lab infrastructure:

- Small and handy in dimension for a trouble-free integration into existing GPC/HPLC systems
- Seamless combination with other detectors and evaluation methods is possible.
- Offers analog processing of data from other detectors and cost effective expandability.

The data recording and analysis software also has to be very flexible. In the event that a sample produces an unexpected weak light scattering signal due to a low dn/dc value, a conventional data evaluation using the concentration signal with calibration curve should be possible. The results of the concentration detector are automatically determined in WinGPC Unity and no additional measurements on other GPC systems are required.

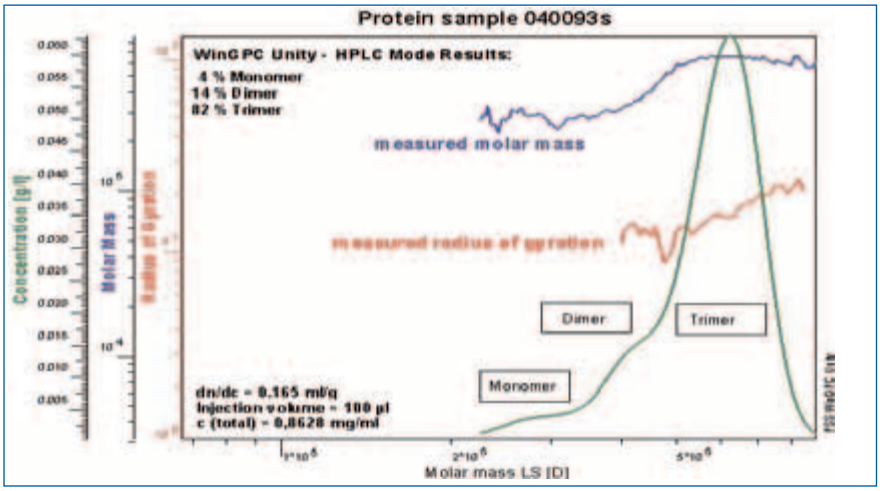

Fig: *Mono-, Di- and Trimer Protein Aggregates tested with SLD 7000 MALLS Detector and WinGPC Unity Light Scattering Module*

Comprehensive Protein Characterization

Shown above is the GPC-MALLS measurement of a protein consisting of monomer, dimer and trimer aggregates. In particular, complex protein oligomers are easily detectable by light scattering. A PSS SLD 7000 was used to measure the scattered light simultaneously at 7 different angles. The concentration detector was a PSS DN2010 which operates at the same wavelength as the SLD 7000. Average molecular weights and radii of gyration were derived from a Zimm plot and the monomer, dimer and trimer content was estimated with WinGPC Unity in HPLC mode. The absolute concentration of the sample was determined using the refractive index increment dn/dc and the RI detector constant. All results are available within one experiment.

Easy Handling during Daily Lab Work

A light scattering detector used in analytical labs should be reliable and easy to handle.

Advanced Literature:

1. P. Kilz, U.Ehmcke: SEC Analysis of Polymers with Light Scattering Detection; GIT International 2/04
2. P. Kilz, H. Pasch: Coupled LC Techniques in Molecular Characterization; in: Encyclopedia of Analytical Chemistry (R.A.Meyers, ed.), Vol 9, pp 7495-7543, Wiley, Chichester 2000

Author

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Innovation

SDV Lux and SUPREMA Lux: Columns for GPC Light Scattering

Ultra pure packing materials suitable for separations that yield excellent signal to noise ratio during on-line light scattering measurements.
The columns are available packed with single porosity or linear (mix bed) gels.

For organic eluents:
SDV Lux; Particle Size: 5µm and 10µm;
Separation Range: 100 - 4.000.000 D.

For aqueous eluents:
SUPREMA Lux; Particle size: 10µm;
Separation Range: 1.000 - >7.000.000 D.

Inverse GPC: a Characterization Method for Porous Materials

Porous materials are often used to remove unwanted substances in medical technology (dialysis) or for enriching desired compounds in biotechnology (proteins).They also play an important role as base materials for catalysts.
For product optimization procedures or quality control, the knowledge of the exact pore size is of crucial importance.
PSS uses the Inverse GPC separation technique for characterization of pore size and pore size distribution. The porous material becomes the stationary phase for chromatography of a series of special probe molecules with known average molecular size. From the retention time (elution volume) of the particle standards, the PSS PoroCheck software calculates the average pore size and the pore size distribution of the material. Additionally, other important parameters like pore volume, specific surface or selectivity can be derived from the experimental data.
PSS offers the measurements as a customer service and uses them for quality control of their own column packing materials.

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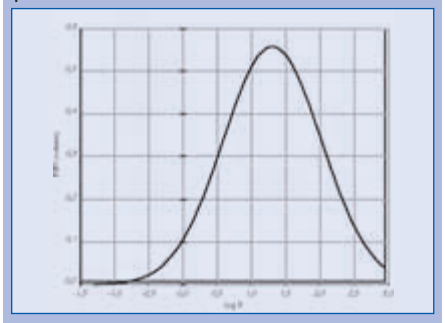

Figure: *Pore size distribution by Inverse GPC, data evaluated with PSS PoroCheck software . PSS SUPREMA column set.*

PSS GPC-Columns

Increased Polymer Characterization Efficiency with Appropriate Column Sets

Polymer analysis has to meet constantly rising quality standards to provide , fast, reliable and comprehensive results. This demands efficient laboratory organization and fast processing times. Because the heart of a GPC system resides in the GPC column, an effective selection of the appropriate column set will provide quick analysis times with the necessary resolution and accuracy

Modern polymer analysis employs columns lengths of 8x300mm for the conventional Analytical column, however, PSS HighSpeed columns (20x50mm) established a niche in the GPC market for shortening the analysis times up to 10 times. The main difference between these and conventional analytical columns is the column dimensions. Separation properties and column volumes are comparable.

Stationary phases of GPC columns fall into three categories:

1. Mono-modal columns: columns with a single pore size or very narrow pore size distribution present high resolution in the usable separation range which is narrower when compared to a linear column (at comparable elution volumes)
2. Linear (mixed bed); generally have wider linear separation range than the monomodal counterparts, presenting lower resolutions and a tendency to overload.
3. Combinations of linear or mono-modal columns will provide the best option to achieve the specific goals of efficient balance between range-resolution and analysis time. HighSpeed columns are available as linear and monomodal columns.

Follow a few guidelines for a successful combination of columns:

1. Do not mix linear and monomodal columns
2. Combine only monomodal columns from one producer
3. Choose columns that produce a flat and constant calibration curve slope over the desired separation range
4. Increased resolution responds to the equation:

$$Rs \sim L^{1/2}$$

therefore, doubling the column length would increase the resolution by a factor of 1.4 (with a consequent doubling of analysis time).

5. Be aware of unreconciled pore size distributions; they may lead to column mismatch and artificial multimodal sample distributions. To check for column mismatch, the measurement of broad calibration standards is advisable.
6. Expand the separation range (maintaining a high resolution) with combinations of different monomodal columns as shown below

Application of different columns

Product Overview	Linear Column
Product Screening	HighSpeed Column
Maximum Peak Separation	Monomodal Column
Improved Resolution	Combination of Columns with Equal Porosity
Increased Separation Range	Combination of Columns with Different Porosity

Recommended combinations of monomodal columns

	Separation Range[D]	Porosity for Organic Applications [Å]	Porosity for aqueous Porosity [Å]
Oligomer	100 – 6 x 10^4	2 x 100	2 x 100
Medium Molar mass	100 – 4 x 10^6	10^3 + 10^5 + 10^6	100 + 1000 + 3000
High Molar mass	1000 – 3 x 10^7	10^5 + 10^7	3000 + 10000 + 30000

References:

T.Hofe: A Combination of Columns or a Linear-Column- A Discussion of a Concept; GIT 11/04 (in German)

T.Hofe, G.Reinhold: GPC Analysis in minutely strokes; GIT 5/00 (in German)

Author

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Upcoming Events

Short Courses

March 15, 2005
Advances in 2-D Chromatography
at: ACS National Meeting and Exposition. San Diego, CA, USA

April 25.-27,2005
GPC-Course in Mainz
Intensivkurs für praktische und theoretische Kenntnisse der GPC

August30, 2005
Advances in GPC and 2-D Chromatography
at: ACS National Meeting and Exposition. Washington DC, USA

Shows and Exhibits

March 14.-16, 2005
Spring ACS National Meeting and Exposition. San Diego, CA, USA Booths 1719/1721

August 29-31, 2005
Fall ACS National Meeting and Exposition. Washington DC, USA
Booths 426/428

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Address

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Please send me information on

- ☐ Light Scattering Detector
- ☐ Dn/Dc Detector
- ☐ Viscometer
- ☐ RI Detector
- ☐ UV Detector
- ☐ GPC Pump, Degaser, Column Oveg Etc
- ☐ LC Spectroscopy Coupling Techniques
- ☐ GPC Software
- ☐ Particle Size Analysis
- ☐ GPC Columns Organic
- ☐ GPC Columns Aqueous
- ☐ GPC Standards / CRM
- ☐ Analytical Services
- ☐ Training
- ☐ Other, explain
- ☐ My application (polymer, elutent etc.):

Please tick requested information

For our Database

To update our Database, please provide the following informatiom

Area of work

- ☐ Analytic/consulting
- ☐ Automobile
- ☐ Chemical
- ☐ Imaging/Printing
- ☐ Biotechnology
- ☐ Rubber
- ☐ Adhesives
- ☐ Electric/Electronics
- ☐ Fibers
- ☐ Feed & Food
- ☐ Fine and specialty
- ☐ Chemistry
- ☐ Forensics
- ☐ Glass/Ceramics
- ☐ Cosmetics
- ☐ Plastics Production
- ☐ Plastics Processing
- ☐ Lacquers & Paints
- ☐ Medical
- ☐ Petroleum
- ☐ Paper / Wood
- ☐ Pharmaceutical
- ☐ Textile & Leather
- ☐ Environment
- ☐ Detergents/Surfactants
- ☐ Military / Aerospace

Work Environment

- ☐ Analytical lab.
- ☐ R&D
- ☐ QC
- ☐ Purchasing

Position

- ☐ Lab. Manager
- ☐ Department Head
- ☐ Professor
- ☐ Purchasing Agent
- ☐ Lab. Assistant
- ☐ Student

Purpose of Request:

- ☐ General information
- ☐ Intended procurement

Acquisition schedule

PSS PROTEEMA

New columns for protein separation:
Particle size 5 [µm]

- ☐ Precolumn 8.00 x 50.00 mm
- ☐ Analytical 8.00 x 300 mm
- ☐ 100[Å]
- ☐ 300[Å]
- ☐ 1000[Å]

- ☐ Provide Application
- ☐ Please send me information
- ☐ I would like a quotation

GPC-Tips and Tricks

Optimum GPC is Interaction-Free

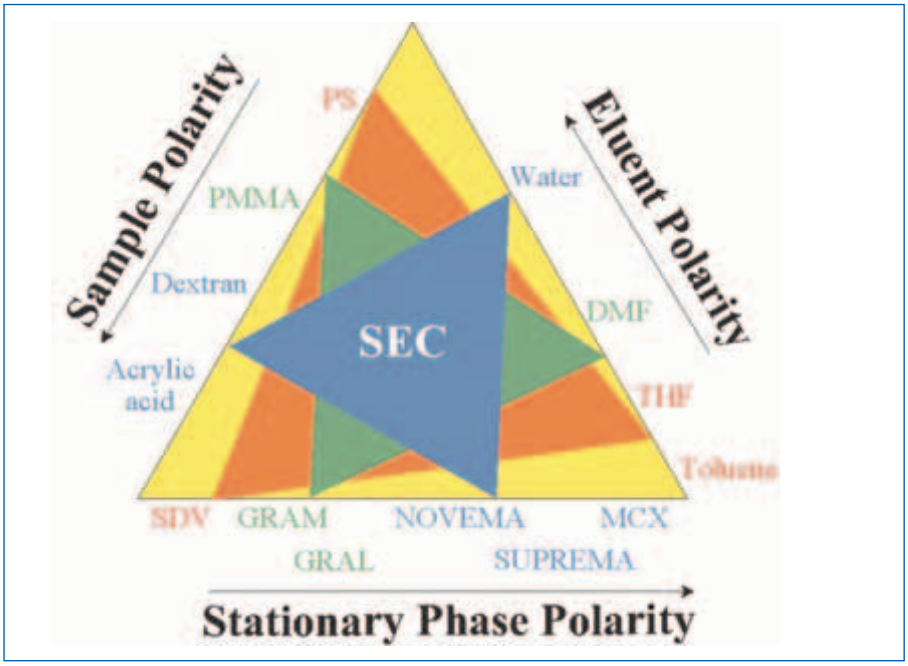
The secret for an interaction-free Gel Permeation Chromatography or Size Exclusion Chromatography is to maintain a similar polarity for sample, mobile phase and stationary phase. The higher the polarity of polymer and mobile phase is, the more polar the stationary phase has to be. The challenge in GPC is to find such a system. It is the only way that you can correlate the elution volume with the correct molar mass.

How do I find the optimum chromatographic system for my polymer?

PSS developed a Magic Triangle tool to guide you to find the appropriate system to set up interaction-free chromatography.
Your polymer determines the analytical conditions and the solvent system, whether aqueous or organic. Thepolymer sample has to be completely soluble in the chosen eluent.
The choice of organic solvents range from hydrophobic toluene, through THF and CHCl3 to very polar eluents like DMF and DMAc. The addition of low molecular electrolytes (LiBr or LiCl) to solvents with high polarity is often needed to get a proper GPC separation of polymers like PAA, PMA copolymers, Vinylpyridine etc.

Aqueous eluents also, often need the addition of low molecular electrolytes in order to get reproducible and interaction-free chromatography.

The polarity of the eluent system defines the polarity of the column packing material, which has to be of similar strength.
PSS offers a wide selection of stationary phases of increasing polarity; starting with the non-polar (hydrophobic) SDV (styrene-divinylbenzene) that is specific for organic mobile phases. As you move toward increasing polarity there are polyester phases with hydroxy functional groups, GRAM, NOVEMA (suitable for cationic systems, and SUPREMA (the preferred stationary phase for aqueous systems of anionic or neutral nature) The PSS MCX, an ion exchange phase, is the most polar phase available at PSS. It is specific for hydrophilic solvents.



PSS Magic Triangle

Key to Successful GPC Separation

Build Equilateral Triangles of Similar Polarity for Sample, Eluent (mobile phase) and Stationary Phase

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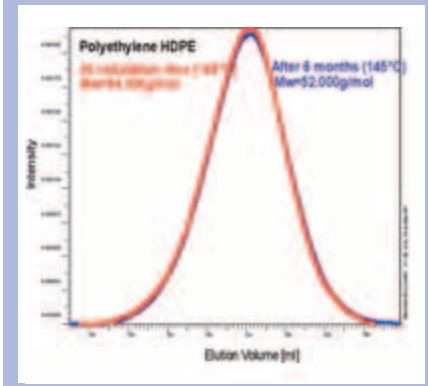
Application

PSS Polefin Columns for GPC Analysis of High Density Polyethylene (HDPE)

Rugged durability at high temperature.

No change was observed on the separation ability and resolution of the Polefin columns after six months of continuous expossure to the high temperature conditions required for HDPE. polyethylene testing.



Six Months stability of a PSS POLEFIN column set. (GPC testing of broad High Density Polyethylene Standard).

Analytical Conditions:

Eluent:	Trichlorobenzene
Columns:	PSS POLEFIN 10µm 8 x 50mm
	PSS POLEFIN 103 Å, 10µm, 8x300mm
	PSS POLEFIN 105 Å, 10µm, 8x300mm
	PSS POLEFIN 106 Å, 10µm, 8x300mm
Calibration kit:	PSS Polyethylen-Kit (Mp=100-170.000 D;
Data recording:	PSS WinGPC
Detector:	RI
Flow rate:	1.00 ml/min
Concentration:	1 g/l
Inject volume:	25µl
Temperature:	145°C

Sample Preparation : Filtration through membrane filter (pore size 0.45 µm).

Results: A Mw = 54000 D and polydispersity D = 2.9 was found for a polyethylene standard upon installation of the column set. After half a year continous running time at 145°C the same standard was measured, resulting on Mw = 52000 D and a comparable polydispersity, well within the range of statistical error (± 5%).

Application #10273. Visit www.polymer.de to search our Knowledge Bank - Application Database