

GPC Streamliner

Focus: Green GPC/SEC



In times of limited resources and increasing costs intelligent solutions are demanded which are efficient and preserve the environment. Green chemistry (or sustainable chemistry) is a concept encouraging the design of products and processes that reduce or eliminate hazardous substances. This idea is certainly convertible for GPC/SEC. PSS has therefore invented new solutions for GPC/SEC, allowing you to fulfill the legitimate demands of modern LC.

PSS will present these solutions at ACHEMA 2009. Please visit us in hall 5.1 booth C39. Entrance tickets are also available from PSS/Germany.

This GPC Streamliner has a focus on Green GPC/SEC and will show some of our latest approaches:

- Experience the PSS micro columns: especially developed for the use in semi-micro GPC/SEC systems with lowest eluent consumption – save

60% solvent with every inject

- Learn more about the new possibilities of the WinGPC Unity SystemPilot 2.0: new system control options for Waters, Dionex, Shimadzu systems together with the existing controls for PSS, Agilent and Tosoh systems
- Discover the new possibilities of modern high temperature analysis in the PSS contract analysis laboratory
- See the new features and options in our WinGPC Unity software in terms of precision and accuracy

In case you cannot come to ACHEMA:

The PSS/Tosoh EcoSEC tour includes a seminar in Eindhoven/NL. Use this opportunity to see Green GPC/SEC with the EcoSEC semi micro system live. Participation is free-of-charge.

WinGPC SystemPilot 2.0: New Functionality, New System Controls

The PSS WinGPC Unity software module SystemPilot was introduced at the end of 2005 and was a great success. This first version supported Agilent 1100 systems and facilitated the daily work through comfortable system control and sequence management in WinGPC Unity. The functionality was extended with time and instrument control for PSS SECurity systems, Agilent 1200 and 1120 series and Tosoh EcoSEC semi-micro GPC systems was added.

Now the new SystemPilot generation adds even more controls for systems from different manufacturers. According to the PSS company policy such implementations are only done when authorized and supported by the manufacturers. This ensures stable and secure solutions decreasing the workload in the lab.

PSS is proud to present the new SystemPilot 2.0 with Waters, Shimadzu and Dionex system control. As before, mixed configurations can be used without any problems. Systems from different manufacturers – no matter if controlled or not – can be operated in one WinGPC multi instrument version. Molar mass sensitive detectors, like viscosity or light scattering

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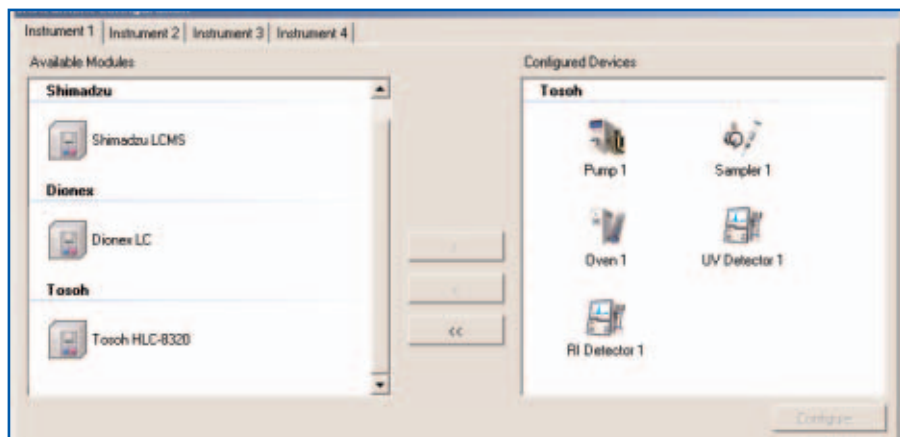


ACHEMA 2009
Frankfurt am Main/Germany
11.-15. 5. 2009

Please visit us

Hall 5.1
Booth C39

WinGPC Unity SystemPilot 2.0: New Functionality, New System Controls



detectors from different vendors, are integrated as well. This shows again the universal applicability of the WinGPC Unity Macromolecular Chromatography System (MCDS).

Important instrument parameters e.g. system pressure or detector wavelength at the time of sample injection are recorded and can be documented on an analysis report in combination with the WinGPC ReportDesigner^{plus}.

For labs in regulated environments PSS recommends the use of the optional WinGPC Unity Compliance Pack. It comprises access control and different user levels as well as detailed Audit Trails for a compliant environment. The SystemPilot adds an encrypted 'Instrument Audit Trail' where the instrument communication is automatically logged.

Besides new controls, the PSS software team has also worked on additional features and functions for the new SystemPilot. Typical end actions (e.g. reduce flow rate, switch off UV lamp or switch to recycling mode) were already implemented in the very first SystemPilot sequence manager. In addition, the new SystemPilot 2.0 allows method and parameter changes within a sequence. E.g. the UV detector wavelength, the flow rate or the column set in use can be changed conveniently in-between two injections. This enables automated measurements with different instrument parameters in just one run.

Since many customers use WinGPC not only for GPC/SEC measurements but work with the integrated HPLC mode, the new SystemPilot supports gradient control for the most important pump systems. Users will benefit from this approach especially when complex polymers are analyzed.

SystemPilot

- Instrument control
- Sequence control
- End actions

- PSS SECcurity
- Agilent
- Tosoh

SystemPilot 2.0

- Instrument control
- Sequence control
- End actions
- new** Method / parameter changes
- new** Gradient support

- PSS SECcurity
- Agilent
- Tosoh
- new** Waters
- new** Shimadzu
- new** Dionex

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Announcements

EcoSEC - Ecological & Economical SEC

PSS and Tosoh Bioscience invite GPC/SEC users to the EcoSEC tour, that has recently started in Germany and Switzerland.

PSS presents new developments in GPC/SEC to fulfill requirements in terms of competitive & ecological analysis. Besides the instrument highlights for the EcoSEC semi-micro GPC system, PSS presents lectures with the topics "GPC/SEC techniques", "viscometry, light scattering and triple detection" and "alternative methods for accurate molar masses". A live-info with EcoSEC and its advanced molar mass sensitive detectors follows in the afternoon.

Next event:

16.06.2009 Eindhoven/The Netherlands - Email info@polymer.de for more information. Find the program and registration form at www.polymer.de



Viscometry and Light Scattering Hands-on Training

Because of many inquiries PSS has expanded its training portfolio and has organized a hands-on training course with the topic "Viscometry and Light scattering" in Mainz/Germany.

This seminar covers all aspects of advanced detection methods and instrumentation, used for the determination of molar masses, sizes, and structures of soluble macromolecules and biopolymers. Lectures that provide the theoretical background are complemented with hands-on training on experimental aspects and data processing regarding (multi angle) light scattering and viscometry techniques. Small workgroup discussions with an experienced polymer chemist and a trouble-shooting session ensure that all aspects of molar mass sensitive detection are efficiently covered, including specific applications and questions.

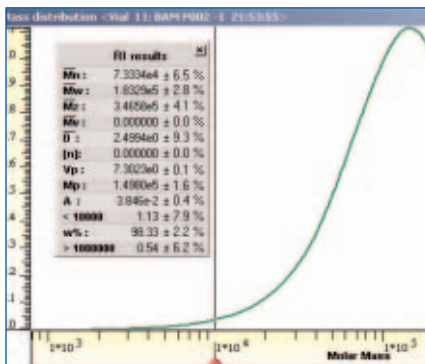
The next Hands-on-Training takes place:
29.06. – 30.06.2009 in Mainz/Germany.

Registration and program: www.polymer.de
or Email info@polymer.de for more information.

How accurate are my GPC/SEC results?

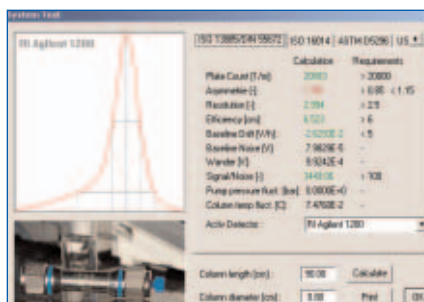
WinGPC users are looking forward to the next WinGPC release: the WinGPC Unity 7.4 preview is shown at the AICHEM 2009 Exhibition and demonstrates that PSS introduces features which are required in any modern lab.

GPC/SEC results play an important role in the registration and accreditation of polymeric products or formulations with organizations like FDA, REACH, etc. Obviously, the accuracy and uncertainty of the results is an often discussed issue. The next WinGPC release will open up novel pathways to quantify the influence of experimental factors on final results in GPC tests. Baseline quality, noise and drift of detector signals, calibration type and quality, among others, are factors which contribute to GPC/SEC result uncertainty. This will be analyzed automatically and the influence on the results will be quantified in WinGPC. Therefore, WinGPC will allow to determine e.g. mass fractions < 500 Da (or any other limit) which are crucial parameters for toxicity evaluation in FDA approvals, REACH registrations, etc. with better confidence and reliability. The uncertainty of GPC/SEC results is not only calculated for conventional GPC/SEC assays, but also for hyphenated methods (e.g. viscometry, LS, Triple detection). Moreover, instrument performance (pump pulsation, column temperature variation, etc) are included in error propagation calculations if WinGPC controls the systems directly via the WinGPC SystemPilot.



However, result accuracy and uncertainty are just one aspect of reliable GPC/SEC results: system suitability and instrument performance must be qualified additionally in many GPC/SEC assays for internal or external audits and reviews. These tests can be performed automatically with the next WinGPC release which will save time, effort and headaches. It will allow users to deliver results more quickly with complete traceability and 21CFR11 compliance. The PSS WinGPC software incorporates pre-defined tests and experimental requirements for major GPC/SEC standards (e.g. ISO, EN, ASTM) and regulatory bodies (e.g. FDA, USP, EP, JP) for quick and easy application

in every lab. Additionally, WinGPC users can create their own system suitability criteria and test their instruments quickly and easily according to their own requirements including comprehensive test result printouts. If GPC/SEC systems are used for analyses in regulated laboratories (biopharm, food, cosmetics, etc), instrument and method qualification have to be performed, verified and documented. All necessary procedures are available and are seamlessly integrated into the PSS EasyValid Validation Kit which allows quick, easy and comprehensive qualifications also for beginners.



Highlights of the next WinGPC revision:

- direct instrument control of systems and modules from Dionex, Shimadzu and Waters, together with support of the next instrument generations from Agilent and Tosoh
- all GPC/SEC results (molar masses, mass fractions, viscosities, radii, Mark-Houwink constants, etc) are reported with result uncertainties
- completely revamped WinGPC system test contains tests according to various GPC/SEC standards (ISO/EN/ASTM/DIN) and regulations (USP, EP, JP, DAB); passed/failed tests are automatically color coded for easy review
- user specific test requirements can be defined and will be checked simultaneously with external system suitability requirements
- additional performance tests (drift, noise, T stability, etc) and instrument checkouts (detector linearity, reproducibility, flow stability, etc) are also included
- all system suitability tests are seamlessly integrated in the PSS EasyValid instrument qualification procedures; verification results are generated with full FDA compliance.

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Innovations

New WinGPC Release

The PSS software team has released the new WinGPC Unity release version 7.3 build 6807 end of march 2009.

The significant changes of this version are the following:

- The ReportDesigner was completely overhauled and offers many new workflow possibilities.
- New systems were integrated in the device control of the SystemPilot.
- All versions of the operating system Windows Vista are supported.
- All current ServicePacks of Windows Vista and Windows XP are supported.

All changes and new features and functions are explained in detail in the corresponding Change Control Document. Clients with a Premium Software support contract obtain the release free of charge and have already obtained the Change Control Document in advance. Upon request, we also send you the Change Control Document, please contact: info@polymer.de.



News on the Homepage

Have you already noticed it? Improvements are available for NetCommunity users: Now you can find the current prices for columns, standards and contract analysis services in the Internet after login. Simply use the search engine, where you can look for keywords, products and part numbers.

Even more is new for customers in Germany, Austria or Switzerland. A lot of the pages on the PSS Homepage are now also available in German. Pages currently only available in English will be placed in German at your disposal in an ongoing process. The button for the change between the two languages German/English can be found in the center of the homepage.



Advanced polyolefin analysis

Polyolefins and their copolymers are insoluble in typical GPC/SEC eluents at room temperature. Common solvents for these products are e.g. 1,2,4-trichlorobenzene, 1,2-dichlorobenzene and decaline. The analysis has to be performed at temperatures between 130 °C and 160 °C. This is a typical high temperature (HT) GPC experiment. For faster and better results for polyolefin analysis PSS has invested in a new high temperature GPC system.

This system has the following advantages:

Sample preparation

Older autosamplers heat up the samples for hours before injection. This can lead to uncontrolled degradation and therefore to a bad reproducibility. The new system allows to program an individual temperature program for every sample with automated solvent delivery and gentle preparation. Filled samples (e.g. with fiber glass, carbon black, talcum) can be used directly without elaborate preparation, due to the automated filtering system with backflush cleaning. This saves time and costs.

Detection systems

Polyolefins have a very low dn/dc value, so the RI signal can be very noisy due to low signal intensity. For reliable measurements the analysis has to be interrupted for hours or days to achieve good baseline stability through purging.

IR detection is a powerful alternative for polyolefins. Detection of the CH-valence vibration with the IR4-detector gives a linear concentration signal with a good signal/noise ratio, without consuming too much eluent and time during the process of purging.

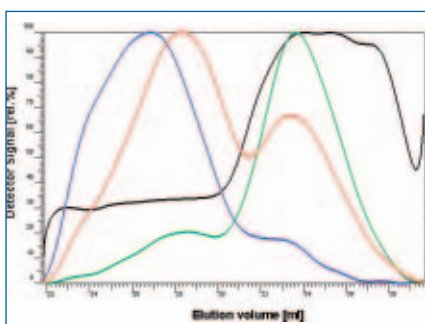


Fig 1: elugram with multi detection of an ethylene-vinyl acetate copolymer
blue: Viscosity signal; red: CH₂ signal; green: C=O signal; black: relative amount of vinyl acetate

The vinyl acetate content in the oligomeric part is around 40% and in the high molar mass area around 5%.

GPC conditions: Temperature: 140 °C, Flow rate: 1 ml/min, Eluent: 1,2-dichlorobenzene, Injection volume: 200 µl, columns: PSS POLE-FIN 10 µm 100 Å + 1 000 Å + 100 000 Å + 1 000 000 Å (each 8x300mm) and precolumn

Molar mass determination

Molar masses are commonly determined based on a Poly(styrene) calibration. Light scattering detectors can be used, but the dn/dc value of polyolefins are often unknown and therefore the advantage of light scattering detection is lost. Alternatively a viscosity detector can be used, providing good data from oligomers up to ultrahigh molar masses. True molar masses can be obtained by universal calibration, if a proper GPC/SEC method can be established.

Structure analysis

By using intelligent detector combinations, structural information is monitored in addition to the molar mass distribution.

Fig 1 shows a typical elugram for a bimodal ethylene vinyl acetate (EVA) copolymer. The combination of the molar mass sensitive viscosity signal and the concentration signal of the CH₂-vibration gives a true molar mass distribution. In addition two different types of structure information are available:

Chain branching

Long chain branching influences e.g. the rigidity of a material. By creating a Mark-Houwink-plot long chain branching can be monitored. A Mark-Houwink-plot is created by plotting the measured intrinsic viscosity vs. the measured molar mass. The slope of this plot gives information about the chain branching. α decreases when the number of branches increases.

Copolymer content

The ratio of IR-vibrations gives information about the comonomer content. For α -olefin copolymers the content of CH₃-groups is detectable. By switching to a carbonyl-sensitive chip, copolymers with C=O groups can be analyzed. This means that the vinyl acetate content can be directly measured with excellent correlation.

Summary

- Higher reproducibility through gentle sample preparation
- Simple analysis of filled samples through integrated hot filtration
- More precise and faster determination of the molar masses through sensitive IR detection
- True molar masses and long chain branching through combination with viscosity detection
- Determination of copolymer content through dual detection with different IR-vibrations

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Upcoming events

Training

08.10. – 09.10.2009

GPC training course, Mainz/Germany

Come to Mainz, Germany to obtain the skills for modern analysis of macromolecules, molar mass and property distributions, using gel permeation chromatography (GPC), a.k.a. size exclusion chromatography (SEC). The theoretical lectures and practical sessions provide the fundamental aspects of the GPC/SEC separation technique, with a balanced view of both, the advantages and limitations of GPC/SEC (stand-alone or hyphenated with light scattering, viscometry, and other techniques). The small group practical sessions (5:1 student-instructor ratio), provide you with choices between beginner vs. advanced user.

16.06.2009

EcoSEC Roadshow in Eindhoven/The Netherlands – This seminar is free of charge

29.06. – 30.06.2009

Hands-on training course: Viscometry and Light Scattering, Mainz/Germany

Shows and Exhibits

11.05. – 15.05.2009

ACHEMA 2009 in Frankfurt/Germany
Please visit us in Hall 5.1 Booth C39.

07.06. – 09.06.2009

Frontiers in Polymer Science in Mainz/Germany
Poster: "Comparison of the 2D-Chromatography (LAC-SEC) and LCCC-NMR coupling for the elucidation of copolymer composition"

08.07. – 10.07.2009

Advances in Polymer Science & Technology in Linz/Austria – Talk: "New development in HT-SEC with multidetection"

12.07. – 17.07.2009

European Polymer Congress EPF'09 in Graz/Austria
Talk: "Non-invasive studies of proteins by SEC coupling methods"

13.09. – 15.09.2009

Bayreuther Polymersymposium 2009 in Bayreuth/Germany
Booth

Further shows and exhibits

<http://www.polymer.de/infocenter/upcoming-events>

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Green GPC/SEC with PSS Micro columns

The PSS column department allocates many resources to research and design. The constant improvement of GPC/SEC columns in order to fulfill customer needs is one of our major goals. In the past, PSS has driven GPC/SEC forward by inventing stationary phases of different polarities to minimize the hydrophilic and hydrophobic interaction (see and compare also the PSS Magic Triangle). Also new column types (e.g. the PSS HighSpeed columns) were developed to save time and to make use of lab resources in the most effective way.

PSS has now used the experience gained in more than 20 years of column development to work on a new concept for micro GPC/SEC columns. The big advantage of micro columns is that they allow to save solvent without sacrificing the resolution. Compared to analytical columns, an analysis with micro columns requires only 40% of the solvent (see also Table 1).

Unfortunately, micro columns can not be used with standard LC instrumentation without losing many of the advantages of micro GPC/SEC. Micro columns need optimized detector hardware with small detector cells and short, optimized capillary connections with low inner diameter. Until now this was a major drawback for this method since conventional analytical HPLC systems (used as GPC/SEC systems) do not fit these requirements. Until 2008 there was no appropriate (semi)-micro GPC/SEC system available in Europe or the US.

This situation has changed with the launch of the Tosoh EcoSEC GPC System by PSS and Tosoh Bioscience. For the first time, there is appropriate GPC/SEC instrumentation available, to make full use of all advantages of micro columns.

Table 1 gives an overview of PSS column types with different dimensions and their recommended applications.

The new concept for the optimization of the micro GPC/SEC columns was established taking into account important customer demands: PSS wanted to develop robust columns providing comparable resolution as corresponding analytical columns. A method transfer should be easy. Particle size and porosities should be optimized and adapted in a way that all applications from an analytical column can be easily transferred. Micro columns should be available for all important GPC/SEC solvents, the surface chemistry should be the same as in analytical and HighSpeed columns.

The results of this approach were PSS micro columns with the following specifications:

- Almost all PSS column materials (compare Table 1) are available, so that existing applications can be easily transferred
- Micro columns are available as single porosity columns, linear columns or in column combinations
- Column materials are only optimized in terms of micro performance, the surface chemistry has been kept for 1:1 application transfer
- Shear degradation is not observed up to molar masses of 2 Million Da
- Plate counts above 80.000 N/m are achieved, the resolution is comparable to analytical columns

PSS columns for all applications

Type <i>Stationary phase</i>	Dimension [mm]*	Solvent consumption/ column [ml]	Analysis time/column [min]	Instrument options
PSS Analytical <i>all PSS materials</i>	8 x 300	12.5	12.5	All HPLC/GPC/SEC systems, e.g. PSS SECcurity, EcoSEC and many more
PSS HighSpeed <i>all PSS materials</i>	20 x 50	12.5	2	Most HPLC/GPC/SEC systems, e.g. PSS SECcurity
PSS Preparative Sample: up to 1.5 mg/Inject <i>all PSS materials</i>	20 x 300	78.1	12.5	Most HPLC/GPC/SEC systems, e.g. PSS SECcurity
PSS Micro <i>SDV, PFG</i>	4.6 x 250	3.5	10	Semi-micro GPC/SEC system: EcoSEC

* other dimensions on request

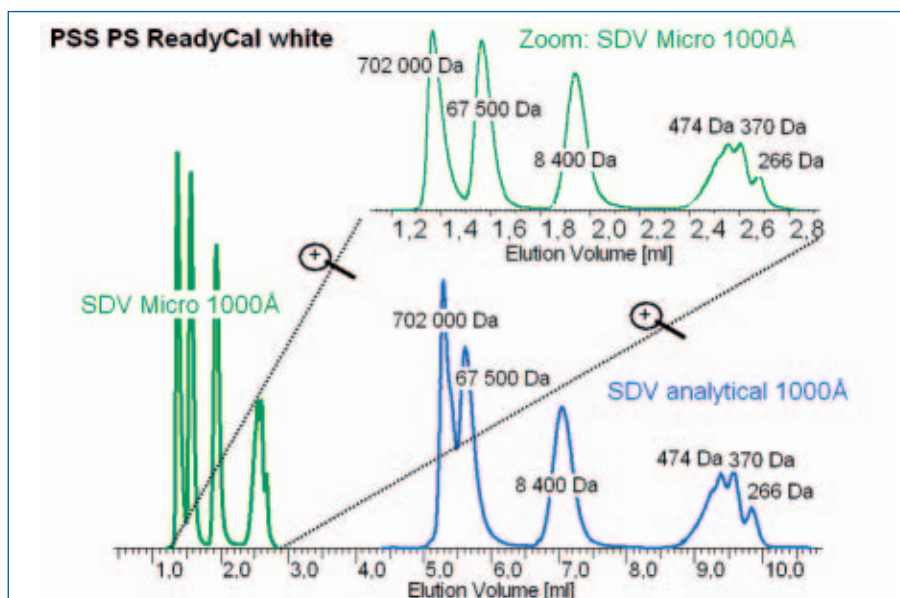


Fig. 1: Comparison of Poly(styrene) ReadyCals (4 molar masses with good oligomer separation) analyzed on an analytical and a micro column (SDV 1000 Å). For a better comparison the elution volume profile of the micro column is magnified.

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