To facilitate your planning, here is the booth’s personnel schedule:

**Tuesday, 23/03/2010:**
Dr. Daniela Held, Thomas Fickinger and Jochen Leinweber

**Wednesday, 24/03/2010:**
Dr. Daniela Held, Thomas Fickinger and Jochen Leinweber
In the afternoon: Dr. Hans-Ulrich Ehmcke, Friedhelm Gores, Dr. Thorsten Hofe, Kirsten Oleschko

**Thursday, 25/03/2010**
Thomas Fickinger, Friedhelm Gores, Dr. Thorsten Hofe and Kirsten Oleschko

**Friday, 26/03/2010**
Dr. Hans-Ulrich Ehmcke, Thomas Fickinger and Friedhelm Gores

We look forward to your visit!

Jochen Leinweber

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**In this issue**

1. **Trade show ANALYTICA 2010**
2. **Validation/Result Evaluation**
3. **New service: LC-MS-ESI-MS Coupling**
4. **System Solutions for R&D and QC**
5. **U-GPC Making GPC/SEC faster**
6. **Review 25 years PSS, lecture Prof. Wilhelm**

**ANALYTICA 2010**

Analytica 2010 is so special for PSS this year, not only to show new products, but also to celebrate our 25th anniversary with our customers, partners and friends. This anniversary year all visitors to the PSS booth (Hall A2, 423) are treated with a glass of sparkling wine. Consider PSS’ new developments and solutions, get a copy of the PSS GPC Streamliner jubilee edition “25 years PSS” and discuss with us the latest trends and projects. Also, inquire about our new educational curriculum created in response to participants’ requests and excellent evaluations of previous successful seminar series.

Finally, don’t forget to pre-register for the free-of-charge European WinGPC User-meeting scheduled for September 30, 2010 in Mainz. All WinGPC users are invited to participate. Program and registration information are available at our booth (Hall A2, 423) or at [www.polymer.de](http://www.polymer.de).

This Streamliner issue provides a news preview of the PSS booth: solutions for validation/uncertainty estimation of results; new detection solutions from the contract analysis department, and resource saving solutions for R&D and QC; the U-GPC topic is also explored in this issue.

As always, you are invited to get your Analytica tickets through PSS and/or make appointments in advance.

Just contact [info@polymer.de](mailto:info@polymer.de) to get your ticket up front.

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**Thursday, 25/03/2010**
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We look forward to your visit!

Jochen Leinweber
In addition, many analytical standards and regulations require the validation and verification of the analytical system, software and methods.

In recent years, PSS has invested in the development of products and tools for the validation of the products and the establishment of meaningful practices that assist in these additional tasks, which nowadays are time-consuming and resource-intensive in the laboratories.

PSS offers both, products and services, designed specifically for GPC/SEC validation procedures with the following advantages:

Typical GPC/SEC results, as molar mass distributions and averages, are obtained.

Typical GPC/SEC solvents can be used which eliminate the need to change solvents and avoid the risk to destroy the separation columns by using inappropriate solvents.

**New Solutions for the Validation and Qualification of GPC/SEC Systems and Results**

**New PSS Products for certified laboratories at Analytica 2010:**

**PSS WinGPC Unity 7.4**

GPC/SEC software boasts integrated system suitability tests based on signal quality, quality of calibration and system stability as well as optional reporting of uncertainty measurements.

For the first time, random error can be quantified for all methods used in and with GPC/SEC. WinGPC 7.4 now determines not only typical GPC/SEC results, but also reports uncertainty values for each result. In addition, it provides user assistance for the assessment of error sources for each sample, while graphically displaying its contribution to the uncertainty.

Other additional tools in WinGPC 7.4 determine flow stability, reproducibility of detector signals and injections as well as detector and injector linearity. A significantly enhanced system test - according to various international standards and regulations - now automatically calculates detector drift, signal to noise ratio, and if necessary, temperature and pressure stability.

**PSS EasyValid for Aqueous Systems**

PSS EasyValid Validation Kit is the only holistic test for vendor-independent qualification of GPC/SEC systems including data acquisition and data processing. It consists of a separation column, calibration and test substances and reference materials. It is used as a system suitability test to verify the complete setup by measuring true GPC/SEC results, the molar mass averages. In addition, the comprehensive user documentation describes further meaningful individual module tests in accordance with typical GPC/SEC standards (e.g. DIN EN ISO).

PSS EasyValid, previously available as a kit for organic solvents, originated a separate kit for aqueous solvents, to release operators of aqueous GPC/SEC systems from the annoying but necessary task of solvent change.

We will be happy to discuss other services, products and features of WinGPC 7.4. Talk to us!

Peter Kilz

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"A true value would be obtained by a perfect measurement. True values are by nature indeterminate."

Unfortunately, this statement has many implications for the work of a laboratory analyst. It implies not only the necessity to produce results and numbers, but also, to evaluate properly and to verify results. As a consequence of the above statement, many users are looking for solutions providing the estimation of the measurement uncertainty.
New Service: 
**Absolute Molar Masses and Structure Determination with LC-ESI-MS-coupling**

The PSS analytical department now offers contract analysis for Electro spray ionization - time of flight - mass spectrometry (ESI-ToF-MS) coupled with GPC/SEC or HPLC separation.

LC-ESI-ToF-MS is a new addition to the PSS characterization portfolio; a powerful technique for the determination of absolute molar masses, structure, and for the deconvolution of complex mixtures. Modern polymeric materials are complex mixtures of the polymer itself -with all its distributions- and different additives. A one-step characterization is not sufficient to answer important questions for such mixtures, even when using multiple coupling techniques such as light scattering, viscometry, and DAD. However, adding mass spectrometry identification methods will.

ESI-ToF-MS is an ideal method for characterization of synthetic and biopolymers because of the high mass accuracy and resolution. Determination of complex polymer systems structure is also possible when combined with the GPC/SEC, HPLC separation techniques.

**Technique**

ESI-MS direct coupling to a standard liquid chromatography system (HPLC, GPC/SEC) without any modifications constitutes a major advantage of this technique. In ESI-ToF-MS the dissolved sample passes through a sprayer needle, where a high voltage is applied. Charged droplets are formed.

The solvent volatilizes the ionized molecules and move to the high vacuum area of the mass analyzer. Many charged states are possible in the polymer chain. State of the art instrumentation enables the analysis of molar masses up to 20 000 g/mol.

The absence of any kind of matrices is an added benefit of ESI-ToF-MS eliminating the need for extensive optimization of matrices – e.g. required in MALDI-ToF-MS - or the measurement artifacts created by the matrix. Consequently, the ESI-MS approach is straightforward.

**Application example**

1) Polymer Analysis

ESI/MS measures absolute molar masses for every fragment. Molar mass differences between fragments are the molar mass of the repeat unit (monomer) in a polymer chain. This allows the identification of polymer compounds.

ESI-MS is an ideal tool for analyzing copolymers, specifically for the differentiation between a true copolymer (showing one distribution) and a blend of homopolymers (several distributions). In addition end groups are detectable.

**Figure 1** shows typical MS-spectra of a PMMA standard. The absolute molar mass of each fraction varies; the repeat unit is 100 g/mol, which is expected for the MMA monomer. An additional distribution with lower intensity is detected.

This additional distribution shows a shift of 68 g/mol and can be correlated to the different end groups of the polymer.
Application example

2) Additive Analysis

Mass spectrometry is particularly suitable for identification of unknown substances. Because of the gentle ionization conditions, molar masses of single molecule peaks are detectable. Fragmentation is also possible, when experimental parameters are manipulated.

An advantage of LC-ESI-MS coupling is that the different additives that may be present in a polymeric material can be separated and identified. Figure 2 shows a GPC/SEC-ESI-MS coupling of a five additives mixture. This mixture consists of UV-protector, antioxidants, process stabilizers, and steric hindered amines (HALS) and is typical for a technical product.

The MS spectrum of the GPC/SEC-peak at 18.25 minutes is very complex. Main peak components are the process stabilizers, which typically elute at this elution time. In addition, side signals of earlier eluting UV-protector and antioxidants can be identified. Also visible is a steric hindered amine which elutes over a broad range of elution times; this HALS is an oligomer (\(M_w = 1\ 700 \text{ g/mol}\)). MS shows several signals with different molar masses.

Conclusion:
In combination with GPC/SEC, ESI-MS is a very powerful analytical tool for the determination of absolute molar masses and structure analysis. Additive and monomer identification is possible adding another tool for the deformation of complex mixtures.

System Solutions for R&D and QC

The proper selection of a suitable GPC/SEC system - able to sustain future expansion and development - will result in a cost-effective purchase and a long-term trouble-free operation.

In practice, it is not easy to decide which GPC/SEC instrument and when to purchase it because there seems to be little or no significant performance differences or operation costs reflected in the product information brochures of many different systems (and components) available in the marketplace. The old saying “more is better” does not apply to this decision; hopefully you define upfront the most important criteria for the specific lab, prior to spending time and money on systems, methods and/or detectors, that add operational costs but lack added value.

Here we present a few considerations that apply to every lab, beginning with a non-negotiable criterion: all components of the new GPC/SEC system must work with all the typical GPC/SEC solvents.

Second, determine the following:

- Will the device be used solely as GPC/SEC system (isocratic mode)?
- Is the HPLC (gradient mode) also required?
- Will it run routinely without major changes?
- Should the device be flexible and accommodate many configuration options?
Later, it is important to understand the critical task/resource for the application:

- Resolution?
- Low solvent consumption?
- Quick analysis time?
- High loading and/or semi-preparative separations?

The answers to these questions already point to two system possibilities, both suitable for certified laboratories and upgradeable with all current GPC/SEC detectors (e.g. light scattering and/or viscometry):

1. An integrated all-in-one GPC/SEC system for routine operations, e.g. EcoSEC, where the initial configuration is done during installation and user modifications are restricted.
2. Or a modular, customizable system, such as PSS SECCurity, with different individual components that can be easily exchanged even between different systems.

Fig. 1 portrays areas of application and advantages of each system. They were prepared with your needs in mind. We would be happy to advise you personally at PSS in Mainz, at Analytica in Munich, or even at your lab.

Talk to us!

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**New at Analytica:**

**SECCurity ELS1400**
- seamless integration
- highest sensitivity

**Thomas Fickinger**

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<table>
<thead>
<tr>
<th>Description</th>
<th>EcoSEC</th>
<th>SECCurity compact</th>
<th>SECCurity</th>
</tr>
</thead>
<tbody>
<tr>
<td>All-in-one compact system</td>
<td>Beginner GPC/SEC-System with isocratic pump, manual injector, RI</td>
<td>Modular system for GPC/SEC and HPLC</td>
<td></td>
</tr>
<tr>
<td>Application Range</td>
<td>Semi micro to analytic GPC/SEC</td>
<td>Analytic to semi preparative GPC/SEC</td>
<td>Analytic to semi preparative GPC/SEC, HPLC</td>
</tr>
<tr>
<td>Additional Detectors</td>
<td>UV, ELSD, MALLS, Viscometer, (also Triple)</td>
<td>UV, MALLS, Viscometer, (also Triple)</td>
<td>UV, DAD, ELSD, MALLS, Viscometer, (also Triple)</td>
</tr>
<tr>
<td>Advantages</td>
<td>Easy-to-use integrated system: Highest resolution in semi micro mode 66% solvent savings (semi micro operation) Highest baseline stability through thermostatted column compartment Unique Dual-flow RI for fast set-up times Easy IQ/OQ, complete IQ/OQ also as service from PSS</td>
<td>Inexpensive starter package: Highest resolution in analytical mode 66-85% time savings in HighSpeed mode</td>
<td>Universal solution for changing needs: Highest resolution in analytical mode 66-85% time savings in HighSpeed mode Ideal for fractionations in semi preparative mode Easy IQ/OQ, complete IQ/OQ also as service from PSS Full flexibility, upgradeable to HPLC and/or 2D Chromatography</td>
</tr>
</tbody>
</table>

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Ultra-HPLC (UHPLC) is a powerful new form of HPLC that attains higher sample throughput (i.e., reduced analysis time) without sacrificing efficiency. This is achieved by using sub-2 µm particle size column packing at increased linear velocities.

**So the question for GPC/SEC users is:**
Can this approach be easily transferred to speed up GPC/SEC measurements to save time (and solvent)?

**Unfortunately the answer is NO.**
This approach will not work! But... there are other solutions shown below!

**Why is U-GPC not possible?**
Reviewing major differences between HPLC and GPC/SEC will lead us to understand that different alternatives are required for faster and more efficient GPC/SEC:

- GPC/SEC size exclusion separation is based on entropy effects. Interactions must be avoided. HPLC separation is based on enthalpy effects and dominated by the interaction between stationary phase and sample. Figure 1 shows in a nice example the differences between the two methods.

- While in HPLC the resolution is optimized varying the choices of the stationary phase, solvent gradient, temperature, etc., in GPC/SEC the resolution is mainly limited to one parameter: the accessible pore volume. The larger the accessible pore volume, the better the GPC/SEC separation is.

Just decreasing the particle size and GPC column dimensions does not work for two reasons:

- Small particles with too large pores would be unstable.
- Too small particles produce excessive shear forces which are complicated when analyzing high molecular weight macromolecules. This can even destroy the samples.

Using smaller particles would only be successful when analyzing low molar masses (see PSS solution 4).

**Here are PSS’ solutions to speed-up GPC/SEC**

**1. Solvent and Time Savings: Overlaid Injection**

When compared with standard injection, this approach saves 30-45% solvent and time for every sample - without any loss in resolution. Overlaid injection can be performed without hardware modification for all systems, column dimensions, and methods, if the analysis software supports it.

Users of PSS WinGPC software have used this feature for a long time, even from the first versions of the PSS GPC/SEC software.

Figure 2 shows the Overlaid injection approach. A separate WinGPC application note describes this in more detail.

Just contact us to get a free pdf copy of the application note.

**2. Time Savings: HighSpeed Columns**

PSS HighSpeed columns have an ID of 20 mm and a length of 50 mm, in contrast with an inner diameter (ID) of 8 mm and a column length of 300 mm of analytical columns (see also Figure 3).

HighSpeed columns require the same amount of solvent as analytical columns, the optimum flow-rate is 6.25 mL/min. Therefore, the analysis itself is more than 6 times faster, thus, the analysis time with a HighSpeed column is approximately 2 minutes (compared with about 15 minutes for an analytical column). PSS HighSpeed columns can be used with all conventional GPC/SEC/HPLC systems without any hardware modification.

The molecular weight/hydrodynamic volume range of samples investigated by GPC/SEC is much more diverse than that of HPLC samples. In GPC/SEC all types, from monomers/oligomers with a few 100 Daltons to macromolecules of several million Daltons, can be present even within the same sample!
3. Solvent Savings: Micro Columns

PSS micro columns have an ID of 4.6 mm and a length of 250 mm. The optimum flow rate for these columns is 0.33 mL/min. Micro columns allow savings of up to 65% solvent.

The analysis time per column is only slightly shorter than that of an analytical column. In contrast to High-Speed columns, using micro columns require dedicated equipment with low band broadening effects and dead volumes (e.g. EcoSEC).

4. Improved Resolution for Proteins and water-soluble Oligomers

PSS manufactures various column materials with smaller particle sizes for use in appropriate applications like low molecular weight samples and for solvents with low viscosity.

PSS SDV columns with a particle size of 3µm are available, with a maximum porosity of 100 000 Å, since they are not suitable for high molecular weights.

NEW and close to their final release, are the PSS SUPREMA columns with 5µm particle size for aqueous GPC/SEC. These materials are ideal for the analysis of low molecular weight samples and for proteins at a pH > 7. Figure 4 shows the excellent separation of 2 proteins on 2 SUPREMA 5µ columns with 100 Å porosity.
## PSS Events 2010 – English Training*

### GPC/SEC Training
- **GPC/SEC Theory & Practice**
  - October 14 - 15, Mainz, Germany
- **GPC/SEC Training**
  - 1 day training, 3 New England locations
  - June 14 - 18
- **Hands-on Visco/LS**
  - June 28 - 29, Mainz, Germany
- **Webinars GPC/SEC Basic Training**
  - April 21 - 22
  - July 19 - 20
  - October 13 - 14
  - November 16 - 17

### Software Training
- Single day booking available.
  - WinGPC ReportDesigner
    - August 30, Mainz, Germany
  - WinGPC Basic Training
    - August 31, Mainz, Germany
  - WinGPC Visco/LS
    - September 1, Mainz, Germany
  - WinGPC SystemPilot
    - September 02, Mainz, Germany
  - WinGPC Compliance Pack
    - September 03, Mainz, Germany

### User meetings
- **European WinGPC User Meeting**
  - September 30, Mainz, Germany
    - (Free-of-charge)

### Conferences/Trade Shows
- **March 21 - 25**
  - 239th ACS National Meeting & Exposition,
    - San Francisco, California/USA, Booth 826
- **March 23 - 26**
  - ANALYTICA, Munich, D, Hall A2, Booth 423
- **April 07 - 10**
  - POLYCHAR 18, Siegen, D
- **August 22 - 26**
  - 240th ACS National Meeting & Exposition,
    - Boston, Massachusetts/USA, Booth 437
- **September 21 - 24**
  - ILMAC, Basel/CH, Hall 1.1, Booth F94
- **September 28 - October 01**
  - HET Instruments, Amsterdam/NL

*All seminars in english language

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Freiburger Macromolecular Symposium:
25 years PSS and presentation of the low-field NMR detector for GPC/SEC-NMR coupling

PSS wants to thank Prof. Wilhelm for his colorful and competent lecture, all his co-workers for the support and especially Markus Cudaj and Gisela Guthausen for their excellent work in this project.

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