



# GPC Streamliner

## Focus: GPC/SEC Know-how



The GPC/SEC market is consolidating further in 2008. To complete their product range many manufacturers are expanding their program to do GPC/SEC analysis even sacrificing expertise and know-how for the sake of earnings; companies are rationalizing away the need for competent representatives, are reducing customer support and neglecting product improvement. Not PSS; we are going the opposite direction. We have deliberately maintained our 23 year old core focus on high expertise for "GPC/SEC and dedicated coupling techniques". PSS boasts a competent knowledgeable community of representatives that guarantee a reliable partnership with you now and in the future. Today we offer routine applications or high-end solutions and services to fit your needs.

In 2007, PSS introduced the SECcurity GPC System, a high-performance system for analytical and semi-preparative GPC/SEC; biopolymer analysis applications were developed; and, for the first time in autumn 2007, a GPC column seminar was offered with great success. The participants praised the direct contact with the column developers as well as the opportunity to exchange experiences.

PSS' 2008 contribution to expanding the GPC/SEC market will stand out at the Analytica Expo 2008 in Munich, with the first **Semi-micro GPC/SEC system, EcoSEC** available in Europe; PSS has secured the exclusive distribution of this Tosoh Bioscience product in Europe. There is ample development potential for the GPC/SEC field. Please visit us at the Analytica.

## Novel Integrated Semi-micro GPC/SEC System

Less solvent consumption, limited sample amounts or very previous samples have been limitations in the successful application of GPC/SEC methodologies. Because no GPC/SEC instruments were commercially available which met the demands for miniaturization, low dead volume optimization, RI detection with a micro-flow cell and highest sensitivity, many samples could not be analyzed in the best way.

Micro bore GPC/SEC columns have been sold for several years with limited applicability and usefulness, because their separation efficiency, resolution and detection limits are adversely affected by the characteristics of conventional analytical instruments.

Now the compact EcoSEC system will be introduced by PSS at the Analytica 2008 exposition. It is the first commercial semi micro GPC/SEC instrument available to the European market, but with proven technology since its introduction in Japan. PSS secured the exclusive distribution in Europe, in cooperation with Tosoh Bioscience (see figure 1).

EcoSEC was developed specifically for semi micro GPC/SEC applications. The standard configuration

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## Trade Show-Highlight:



**analytica2008**

01-04 APRIL, NEW MUNICH TRADE FAIR CENTRE

21<sup>ST</sup> INTERNATIONAL TRADE FAIR FOR INSTRUMENTAL ANALYSIS, LABORATORY, TECHNOLOGY AND BIOTECHNOLOGY WITH analytica Conference

Please visit us:

Hall A2, Booth 451



## Novel Integrated Semi-micro GPC/SEC System



Fig. 1: Semi micro GPC/SEC system EcoSEC

includes pump, autosampler, thermostated column compartment and an integrated RI detector. Salient technical features are the thermostat-controlled pumps to guarantee constant steady flows and the highly sensitive dual flow RI detector with ultimate baseline stability. Additional optional elements may be integrated to the configuration, i.e., UV detector, a column switching valve and molar mass sensitive detectors like the PSS ETA2010 viscometer or the PSS SLD7000 multi-angle light scattering detector. The semi micro GPC/SEC is a highly attractive unit especially for applications with limited sample amounts (biopolymers) or those which require expensive solvents (like HFIP); the solvent consumption is extremely low. The system can however also be used for analytical work which makes it even more attractive. Easily transfer of existing methods to the semi micro scale is simple by switching to the PSS micro bore columns.

Tosoh decided to ship each EcoSEC unit with the widely used PSS WinGPC Unity software for ease of

use and versatility. PSS incorporated the EcoSEC instrument control into the WinGPC Unity SystemPilot software module (see figure 2) which allows seamless integration of the EcoSEC system with existing systems.

**Interested? See the EcoSEC at Analytica 2008 in Munich. (PSS booth 451, Hall A2).**

PSS has specialized and is known for their expertise regarding Gel Permeation Chromatography (GPC/SEC), the recognized method to determine the molar mass distribution of synthetic polymers and biopolymers. 20+ years of growing GPC/SEC expertise and innovation hold PSS outstanding reputation and leadership to foresee and put in place advanced solutions that move forward the study of macromolecules.

**Author:**

Dr. Martina Adler  
 Tel.: +49-(0)6131-96239-42  
 E-Mail: MAdler@polymer.de

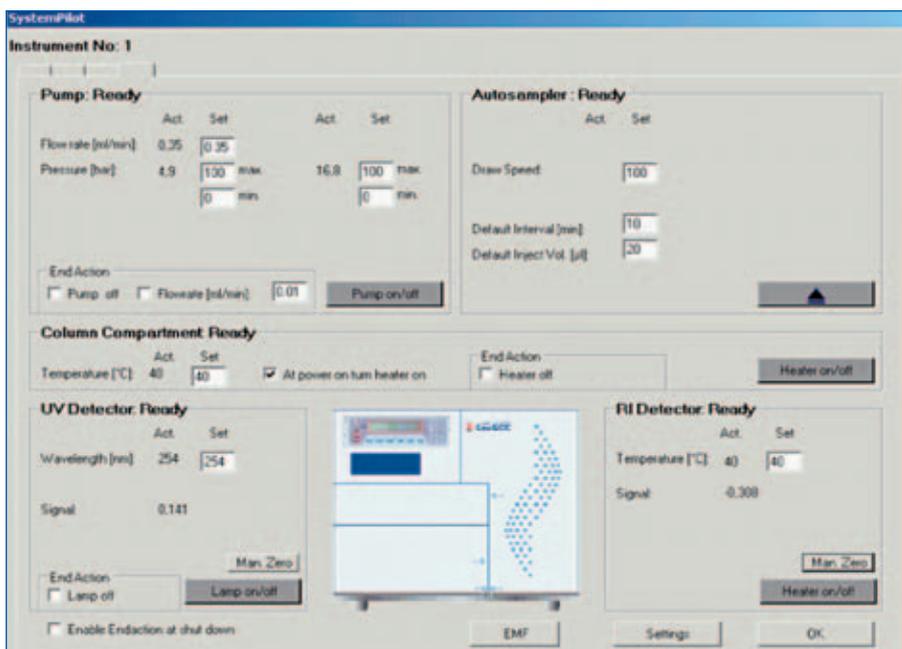
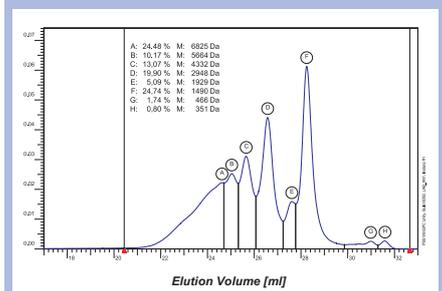


Fig. 2: Integrated system control out of the WinGPC Unity software

## Announcements

### Increased capacity fosters particle size classification service

In response to the high demand on carrier material, PSS has installed a highly modern, computer-controlled multi process classification instrument for the stationary phase gels. This instrument effectively increases throughput of narrow particle size distribution about 15 times, and places PSS in the position to offer a gel classification service. Moreover, with this acquisition PSS can be more responsive to customer demands for specific carrier materials, and sustain increased growth on column technology development and production in the future.



### REACH: Polymer or monomer?

Under REACH requirements, it is necessary to document the polymeric character of a substance. Per definition, a polymer is a material features a sequence of minimum three monomer units linked with covalent bonds (3 plus 1 rule). Furthermore, not any one molar mass species in a homogenous mixture can exceed 50 %.

An established analytical method for the determination of a polymeric substance and therefore for the evaluation of the "polymeric status" is the size exclusion chromatography (GPC/SEC), which is also favored in the REACH guidelines.

PSS offers this analytical service. GPC/SEC measurements are carried out in our service laboratories, normally done according DIN/EN guidelines or OECD rules. In the scope of work you receive an analysis report and the documentation



## Modern Biopolymer analysis

More and more in our every-day lives people are confronted with new products and properties within the biopolymer world, the world of polysaccharides, proteins, peptides, etc. The work with these molecules lay in the borderline between biology and polymer chemistry: The challenge of understanding and controlling the structure-property-relationships of biopolymers has a bearing on the application of macromolecular analysis with GPC/SEC and GPC/SEC-coupling methods, which are recognized for molecular analysis in polymer science.

GPC/SEC is an established and promising method for molecular investigation of macromolecules concerning dimensions/topology, structure, composition and molar mass (molecular weight), provided that a GPC separation phase optimized for their properties successfully achieve the separation.

PSS fosters the investigation of Biopolymers with the development of modern separation phases to study the full gamut of biopolymer compounds:

Polyanions: hyaluronic acid, lignin, lignin sulfonates  
Neutral polymers: cellulose, starch, starch derivatives, dextrans and other polysaccharides  
Biodegradable polymers: polylactides

The broad diversity of bio-polymers mandates separate GPC columns available for aqueous or organic eluents, which cover the full molar mass range from low-molecular to ultra-high molecular size.

Amylose and amylopectin are examples of high molar mass biopolymers; these may be separated on PSS SUPREMA columns up to > 10 000 000 Da.

Proteins demand a maximum resolution on a narrow range of molar mass, e. g. to separate monomers from dimers and trimers. For this application PSS PROTEE-MA columns are best suited.

Biodegradable biopolymers like polylactides are soluble only in halogenated organic solvents like chloroform or trifluoroethanol because of their semi-crystalline character. For this reason they are separated on special functionalized silica columns (PSS PFG).

Sometimes any one concentration detector is not sufficient to characterize all the separated products. In those cases coupling of GPC/SEC with molar mass sensitive detectors, like MALLS detector PSS SLD7000 or online-viscometer PSS ETA2010, allows the determination of the "real" molar mass, structure (degree of branching, stiff rods, rigid globes/balls) and dimensions (hydrodynamic volume or radius of gyration) of the polymer in solution.

The GPC-MALLS coupling set-up permits the straight forward determination of all important protein parameters, including the quantification of associates.

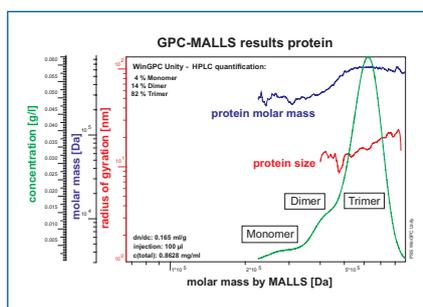


Fig. 1: Results of protein analytics with GPC-MALLS coupling.

Another example of GPC biopolymer-analysis is the hyaluronic acid. Hyaluronic acid is a glycosaminoglycan an important component of the epithelium (greatly hygroscopic) and of the synovial fluid (thixotropic property).

PSS has developed a reproducible and robust method for GPC analysis of hyaluronic acid in water with PSS SUPREMA columns (Fig. 2).

Hyaluronic acids from human or streptococcus origin have different peak profiles and UV/RI ratios. The two hyaluronic acids can be differentiated by overlay of the two GPC curves.

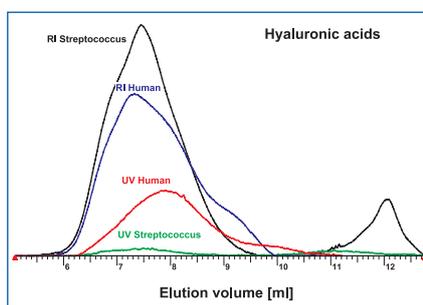


Fig. 2: GPC analysis of hyaluronic acids on PSS SUPREMA columns: human:  $A(UV/RI) = 0.396$  V/mL; streptococcus:  $A(UV/RI) = 0.046$  V/mL.

These two examples illustrate that PSS is well positioned for new challenges in biopolymer analytics. With our customers in mind, we continue to foresee and develop effective concepts and solutions for tomorrow's challenges.

### Author:

Dr. Thorsten Hofe  
Tel.: +49-(0)6131-96239-60  
E-Mail: THofe@polymer.de



## Innovations

### WinGPC Unity SystemPilot Module

The new WinGPC module SystemPilot adds comprehensive and convenient system control e.g. for Agilent 1100 and 1200 series to the overall functionality. The module fits seamlessly in any existing workstation, network or server installation. Mixed configurations, with instruments or detectors from other vendors, are fully supported to allow one data system for all GPC/SEC applications in the lab.

The SystemPilot provides all important functions for traceability of measurements and for documentation of system performance. Full compliance for recorded data and for controlled access to the SystemPilot is achieved with the optional WinGPC Unity module Compliance Pack. User levels can be defined there to restrict SystemPilot functionality to certain user groups.

Part number: 400-1010



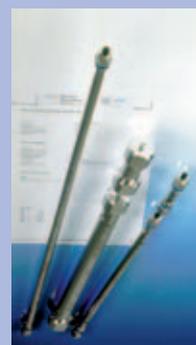
### Support for GPC/SEC columns

PSS offers widespread range of support options to column customers: phone or email column troubleshooting, column check service, column selection service and complete method development.

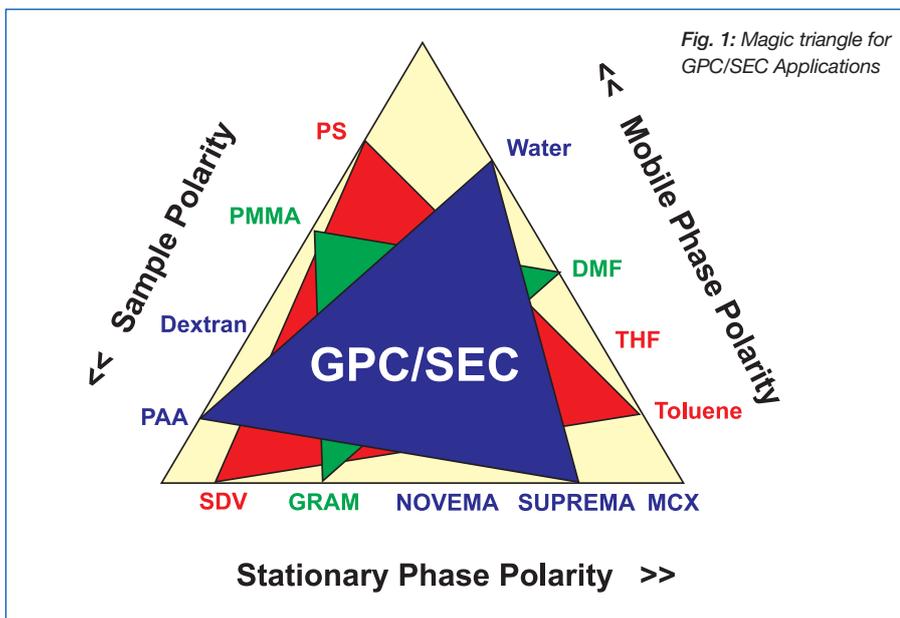
The column check service consists on testing the column plate number, asymmetry, resolution, and when necessary cleaning, repair and issue of a new quality certificate. For irreparable column, PSS offers a cost-effective refill service.

PSS also offers a column selection service, where customer samples are tested in plausible column materials. The customer receives a report with column recommendation.

For the method development builds on the column selection service. The report includes validation, reproducibility test and optimization of the measurement.



## GPC/SEC Column Selection and Method Development Column Users Meeting



The first PSS GPC/SEC seminar on column selection and method development took place in Mainz, on 13. November 2007

40 participants from Germany, Austria and Switzerland took the opportunity for interactive knowledge exchange with other users and the GPC/SEC specialists from PSS.

Some of our GPC/SEC users motivated us to arrange such a dedicated meeting. There was a massive response for the seminar; So much that we had to turn down some users with the promise to schedule another seminar at a later date. We will keep you informed.

The seminar was lead by Dr. Thorsten Hofe, head of the PSS column production, and Dr. Günter Reinhold, responsible for the development of new GPC/SEC-separation materials at PSS.

Initially, all the steps for the production of a GPC/SEC column were presented: from the gel synthesis (che-

mical, physical and technical) with the different requirements for the separation materials over the purification and testing of the materials to the packing of the columns with subsequent individual quality control.

Strategies for method development were presented (see Fig. 1) and the discussion included general problems and questions like how to avoid interaction between the sample and the column material and how to check the column mismatch.

The lectures reviewed special applications like protein separation, GPC light scattering coupling and fast GPC with PSS HighSpeed columns.

The afternoon workshops were specific for either organic or aqueous applications; the aspects were discussed in more depth with practical examples.

The very positive feedback from participants showed, that this seminar was wished by many people and that PSS pursuit of modern, effective and new high-tech carrier materials corresponds to the requirements of users.

PSS thanks all participants for the active attendance and the lively discussion.

We look forward to seeing you at the 2009 GPC/SEC column user meeting in Mainz, Germany.



Fig. 2: GPC/SEC column user meeting 2007

### Author:

Thomas Fickinger  
Tel.: +49-(0)6131-96239-31  
E-Mail: TFickinger@polymer.de



## Upcoming events

### GPC Training courses in Mainz, Germany

09. – 10.10.2008

The GPC Theory and Praxis takes place in Mainz, Germany; it provides theoretical lectures and practical sessions for modern analysis of macromolecules using gel permeation chromatography (GPC), also known as size exclusion chromatography (SEC). It covers the fundamental aspects of the 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). It equips you to produce and analyze data; to qualify the condition of the analytical system and the separation columns; to optimize the application conditions and the work flow in the lab. You will receive practical advice to get reproducible and accurate separations. The number of participants is limited to a maximum of 5 people per instructor. You may choose to a group working with aqueous or organic solvents and with beginners or advanced users.

### WinGPC User Meeting in Mainz, Germany

08.10.2008

Lectures and workshops  
This seminar is free of charge

### Shows and Exhibits

01.04. – 04.04.2008

Analytica 2008; München  
Please visit PSS Booth #451 in Hall A2,  
03.04.2008: 3 pm – 3.30 pm; Hall A3  
Speaker: Peter Kilz: "Micro Separations utilizing a novel integrated GPC/SEC system"  
Forum "Innovations & Technology";  
Block "Instrumentelle Analytik"

17.08.-21.08.2008

ACS Fall Meeting; Philadelphia, USA  
Please visit PSS Booth #1834

14.09. – 17.09.2008

2nd International Conference on  
Polyolefin Characterization; Valencia/Spain

### Publisher:

PSS Polymer Standards Service GmbH  
Postfach 3368 • D-55023 Mainz • Germany  
Tel.: +49 (0)6131-96239-0  
Fax: +49 (0)6131-96239-11  
E-Mail: info@polymer.de  
Web: www.polymer.de

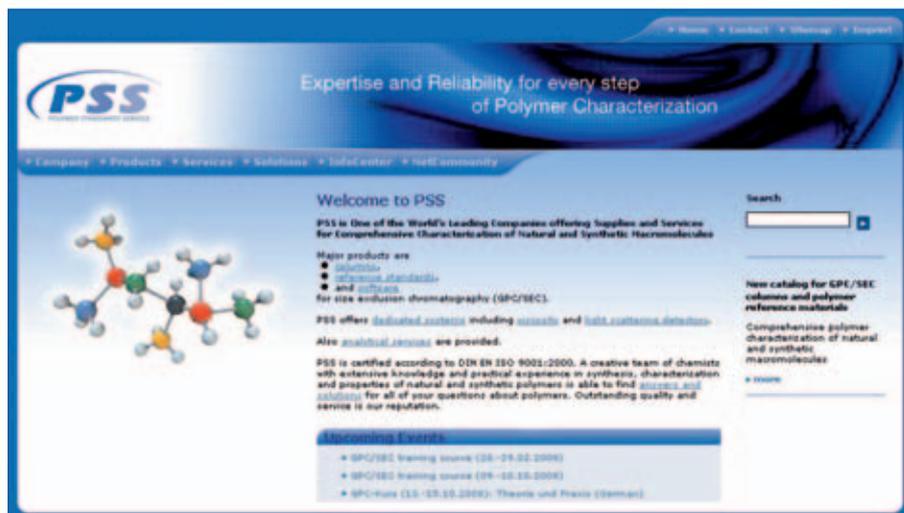
### PSS Polymer Standards Service-USA, Inc

43 Jefferson Blvd. Ste 3  
Warwick, RI 02888 • USA  
Tel.: +1-401-780-8884  
Fax: +1-401-780-8824  
E-Mail: pssusa@polymer.de  
Web: www.pssgpcshop.com

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# GPC/SEC InfoCenter www.polymer.de Total Makeover



In November 2007, a fresh new format for the PSS homepage went online, to provide an easy to navigate fully searchable GPC/SEC information resource, showing PSS' solemn commitment to the success of macromolecular liquid chromatographers. The comprehensive **Products** section covers every material need

- Polymer standards, kits, special polymers  
With Certificates and MSDS
- GPC/SEC Columns  
With specification, calibration curves and separation range
- Software for GPC/SEC and pore size determination  
With modules and expandability
- GPC/SEC systems and molar mass sensitive detectors  
From simple GPC/SEC instruments to fully validated turnkey systems
- Contract analysis (for polymer testing)  
From molar mass determination to a complete product deformation
- GPC/SEC courses and support  
Theory and practice GPC course, maintenance contract, software support.

PSS' unrivaled expertise is evident through the **Services** tab, which features (but is not limited to) column selection, method development, contract analysis and the highly successful PSS GPC/SEC training courses. Visit [www.polymer.de](http://www.polymer.de); you may get a solution to the GPC/SEC challenges.

The brand new **Solutions** tab shows products and tools to apply the theoretical concepts of GPC/SEC tasks, like molar mass determination, copolymer ana-



lysis, 2-dimensional chromatography, membrane characterization and HighSpeed GPC/SEC. Furthermore you can find validated system solutions, to work under regulatory guidelines: FDA, ICH and GxP.

The **InfoCenter** offers GPC/SEC tips&tricks and practical advice for daily use.

Registered users of the free **PSS NetCommunity** will enjoy the ability to download fully detailed application notes with measuring parameters, and articles with method information, theoretical background, GPC/SEC tips&tricks and WinGPC applications; all is accessible through the PSS publication database. The PSS column application database helps you search for appropriate column material and column set(s) for specific applications.

Visit [www.polymer.de](http://www.polymer.de)

### Author:

Jochen Leinweber  
Tel.: +49-(0)6131-96239-30  
E-Mail: [JLeinweber@polymer.de](mailto:JLeinweber@polymer.de)



## Application

### GPC/SEC analysis of Mono-, Di-, Tri-acylglyceride and Glycerol according to European Pharmacopoeia<sup>1)</sup>

Mono-, Di-, Triacylglyceride are widely used as emulsifiers in food and pharmaceutical industries. Some paints and adhesives may also contain different glyceride samples.

For pharmaceutical industry quality control, the European Pharmacopoeia (Ph. Eur. 2000) has the specific procedure used herein to determine the contents of Mono-, Di-, Tri-acylglyceride and Glycerol.

#### Sample preparation

Each sample is dissolved in 5 ml THF to a homogeneous solution (concentration below).

#### Experimental according to Ph. Eur. 2000

SEC/GPC analysis was performed on a SECcurity GPC1200 System, under the following analytical conditions:

Sample:	Mixture of Mono-, Di-, Tri-acylglyceride and Glycerol
Column:	PSS SDV, 5 µm, 100 Å, 8 x 600 mm + precolumn
Solvent:	THF
Flow-rate:	1 ml / min
Calibration standards:	0.5 g/l, 1 g/l, 2 g/l 4g/l Glycerol reference solutions
Concentration:	40 g/l
Inject volume:	40 µl
Detector:	RI
Data acquisition:	PSS WinGPC Unity
Sample Concentration:	5.0 g/l
Injection volume:	20 µl
Temperature:	25° C

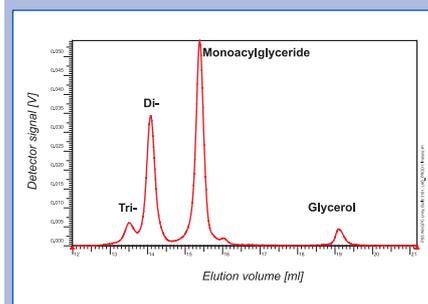


Fig. 1: Technical sample of a Glyceride mixture

#### Results and Conclusion

The content of Glycerol is based on a Glycerol reference solution calibration curve. Various Glycerol concentrations were injected and the correlated areas and peak heights are calculated. The content of Mono-, Di-, Tri-acylglyceride and Glycerol is calculated by a normalization process, relative to the calculated Glycerol content.

<sup>1)</sup> European Pharmacopoeia, 495, page 882 ff

## PSS Distributors

### Austria

CP-Analytica GmbH  
Am Pulverturm 17  
2130 Mistelbach  
phone: +43-(0)2572-4381  
fax: +43-(0)2572-20791  
email: info@cp-analytica.at  
Web: www.cp-analytica.at  
contact: Josef Massong

### Benelux

Bester bv  
Touwslagerij 9  
1185 ZP Amstelveen  
phone: +31-(0)20-6400046  
fax: +31-(0)20-6470411  
email: info@bester.nl  
Web: www.bester.nl  
contact: Rudi J. P. Goedknecht

### China

Chance International Group Ltd.  
Rm. 1903, Yimao Building,  
No. 1399, Jinqiao Road  
200129 Shanghai  
phone: +86-(0)20-50550642  
fax: +86-(0)20-50310742  
email: info@chanceint.com  
Web: www.chanceint.com  
contact: Candy Ni

### Czech Republic

Scitech spol. s.r.o.  
Nad Šárkou 75  
160 00 Praha 6  
phone: +420-(0)2-24311850  
fax: +420-(0)2-24311850  
email: scitech@scitech.cz  
Web: www.scitech.cz  
contact: Dr. Pavel Drašar

### Finnland

Krotek Oy  
Rantatie 11  
33250 Tampere  
phone: +358-(0)3-2535218  
fax: +358-(0)3-2535144  
email: krotek@sci.fi  
Web: www.krotek.fi  
contact: Pekka Kippo

### France

Blossom Products  
5/7, place Marcel Rebuffat  
Villejust  
91971 Courtaboeuf Cedex fr  
phone: +(33) (0)1-60-10-37-74  
fax: +(33) (0)1-60-14-07-94  
email: jpg@blossom-partners.fr  
Web: www.blossom-products.com  
contact: Jean-Pierre Grenotton

### Greece

Analytical Instruments S. A.  
9 Tzavella str.  
152 31 Chalandri  
phone: +30-(0)210-674 89 73  
fax: +30-(0)210-674 89 78  
email: contact@analytical.gr  
Web: www.analytical.gr  
contact: Katerina Aravantinou

### India

Chromline Equipment (I) Pvt. Ltd.  
152-D, 1st floor, Udyog Bhavan,  
Sonavala Road, Goregaon (East)  
Mumbai - 400 063  
phone: +91-(0)22-26860816  
fax: +91-(0)22-26860306  
email: mail@chromlineindia.com  
Web: www.chromlineindia.com  
contact: Rajendra Barabde

### Ireland

Brennan & Company  
61 Birch Avenue  
Stillorgan Industrial Park  
Stillorgan, Co. Dublin  
phone: +353-(0)1-295 2501  
fax: +353-(0)1-295 2333  
email: enquiries@brennanco.ie  
Web: www.brennanco.ie  
contact: Mogan Burgess

### Israel

Bargal Analytical Instruments &  
Software Ltd.  
Galil Street  
Airport City 69719  
phone: +972-(0)3-9796533  
fax: +972-(0)3-9796538  
email: bargal@bargal.co.il  
Web: www.bargal.co.il  
contact: Dr. Arie Gillon

### Italy

SRA Instruments Italia S.r.l.  
Viale Assunta 101  
20063 Cernusco sul Naviglio (Mi) it  
phone: +39-02-92143258  
fax: +39-02-92470901  
email: miliazza@srainstruments.com  
Web: www.srainstruments.com  
contact: Armando Miliazza

### Japan

S.A.S. Corporation  
3-16-4 Kinugaoka, Hachioji  
192 Tokyo  
phone: +81-426-465662  
fax: +81-426-465692  
email: sas@fa2.so-net.ne.jp  
Web: www.sascorp.jp  
contact: Otohiko Sato

### Korea

Dong-il Shimadzu Corp  
D.I Bldg. 3F 90-1 NonHyun-Dong 135-818  
Kang nam-Ku - SEOUL  
phone: +82-2-5405541  
fax: +82-2-5412163  
email: wpyyo@e-shimadzu.co.kr  
Web: www.e-shimadzu.co.kr  
contact: Pyo Woo Young or Yun Kyung Kim

### Norway

Instrument Teknikk A.S.  
Østensjøveien 36  
0667 Oslo  
phone: +47-(0)-23 19 46 40  
fax: +47-(0)67-23 19 46 41  
email: it@instrument-teknikk.no  
Web: www.instrument-teknikk.no  
contact: Bosse Emilsson

### Poland

Anchem Sp z.o.o.  
ul. Rakowiecka 36  
02-532 Warszawa  
phone: +48-(0)22-6462660  
fax: +48-(0)22-6462685  
email: info@anchem.pl  
Web: www.anchem.pl  
contact: Mariusz Malczewski

### Portugal

Elnor Equipamentos Tecnicos e de  
Laboratorio SA  
R. Frei Jeronimo Brito Melo, 835  
4465-642 Leca do Balio  
phone: +351-(0)22-9050400  
fax: +351-(0)22-9050499  
email: info@elnor.com  
Web: www.elnor.pt  
contact: Adão Araújo

### Slovakia

Scitech spol. s.r.o.  
Nad Šárkou 75  
160 00 Praha 6  
phone: +420-(0)2-24311850  
fax: +420-(0)2-24311850  
email: scitech@scitech.cz  
Web: www.scitech.cz  
contact: Dr. Pavel Drašar

### Slovenia

Kobis d.o.o.  
Kidri čeva 11  
1236 Trzin  
phone: +386-(0)1-5636080  
fax: +386-(0)1-5636089  
email: info@kobis.si  
Web: www.kobis.si  
contact: Marko Prezelj

### Spain

Ingenieria Analitica S.L.  
Avda. Cerdanyola, 73  
08190 Sant Cugat del Vallès, Barcelona  
phone: +34-90-2456677  
fax: +34-90-2466677  
email: inf@ingenieria-analitica.com  
Web: www.ingenieria-analitica.com  
contact: Marc Gibert

### Sweden

Dalco ChromTech AB  
Tingsvägen 19  
191 61 Sollentuna  
phone: +46-(0)8-59496969  
fax: +46-(0)8-59496968  
email: info@dalcochromtech.se  
Web: www.dalcochromtech.se  
contact: Jörgen Persson

### Turkey

Ant Teknik Cihazlar Paz.ve Dis Tic. Ltd  
Burhaniye Mah. Beybostani Sok. No: 37/1  
34676 Beylerbeyi/Istanbul  
phone: +90 216 422 67 00  
fax: +90 216 422 39 54  
email: antteknik@antteknik.com  
Web: www.antteknik.com  
contact: Ali Kemal Karak

### United Kingdom

Kromatek Ltd.  
18 Oak Industrial Park Chelmsford Road  
Great Dunmow, CM6 1XN gb  
phone: +44-(0)1371-876500  
fax: +44-(0)1371-873237  
email: inbox@kromatek.co.uk  
Web: www.kromatek.co.uk  
contact: Nigel Hopkinson